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Standard Procedure for Field Testing Oil-Based Drilling Fluids

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Addendum 1 to Standard Procedure for Field Testing Oil-Based Drilling Fluids

1. In the Title of Appendix C, replace "ELECTROHYGROMETER" with "CHENEVERT METHOD"

2. Replace Table I-1with the following:

Table I-1—Dräger Tube or Equivalent—Identification, Sample Volumes, and Tube Factors to Be Used for Various Sulfide Ranges

Sulfide Range (mg/L)	Sample Volume (cm^3)	Dräger Tube Identification (See tube body)	Tube Factor (Used in Calculation) (New tubes)
1.2 to 24	10.0	H_2S 100/a	0.12[a]
2.4 to 48	5.0	H_2S 100/a	0.12[a]
4.8 to 96	2.5	H_2S 100/a	0.12[a]
30 to 1050	10.0	H_2S 0.2%/A	1500[b]
60 to 2100	5.0	H_2S 0.2%/A	1500[b]
120 to 4200	2.5	H_2S 0.2%/A	1500[b]

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[a] Tube factor 0.12 applies to tubes marked H_2S 100/a (Cat. No. CH-291-01) with 100 to 2000 scale. For old tubes with 1–20 scale, use a tube factor of 12.

[b] Tube factor 1500 applies to tubes marked H_2S 0.2%/A (Cat. No. CH-281-01) with 0.2 to 7.0 scale. For old tubes with 1–17 cubic centimeters scale, use tube factor 600 times ratio: Batch Factor/0.40.

3. Add the following Appendices, L, M, and N.

APPENDIX L—HIGH-TEMPERATURE/HIGH-PRESSURE FILTRATION TESTING OF OIL-BASED MUDS USING THE PERMEABILITY PLUGGING APPARATUS AND CELLS EQUIPPED WITH SET SCREW SECURED END CAPS

L.1 Description

L.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.

L.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.

L.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 the 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 can create the possibility of cell leakage or failure, resulting in serious injury or equipment damage.

The sample cell is hot during operation. The operator must 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. Use these devices only on grounded circuits.

Following are suggestions to ensure safe operation and maintenance of the PPA.

L.2.1 SAFE OPERATION OF THE HYDRAULIC PRESSURIZATION SYSTEM

L.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 the pressure hose from cell at quick coupler.
b. Attempting to remove the 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.

L.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.

L.2.1.3 Make sure, when assembling the cell, that the end cap set screws are properly aligned and tightened.

L.2.2 SAFE PNEUMATIC PRESSURIZATION OF BACK PRESSURE RECEIVER

L.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. CO_2 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.

L.2.2.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

L.2.2.3 Repair or replace leaking pressurization systems, hydraulic or pneumatic. Gauges, fittings, and hoses must be kept in good condition, and leaks 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.

L.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.

L.2.3 SAFE HEATING

L.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.

L.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.

L.2.4 SAFE ELECTRICAL OPERATION

L.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.

L.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.

L.2.5 SAFE TEST CELL MAINTENANCE

The filtration cell is a pressure vessel. Consider it to be a source of potential danger. The following safety precautions listed 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.

L.3 Equipment—Permeability Plugging Apparatus—Using Cells 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. Attach the operating manual, or this procedure, to the apparatus. Before using the PPA, these instructions must be read by anyone who is unfamiliar with the equipment.

L.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 and the pressure applied to the bottom of the cell. The filter medium is at the top end of the cell, through which the filtrate is collected. Pressure is applied to the cell 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.

L.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 is 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 de 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 must be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set screw holes, must not be used.

L.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 CO_2 pressurizing source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

L.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum well 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 wall of the cell. 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.

L.3.2 FILTER MEDIUM

L.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 downhole.

L.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. but, with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, soak the disk for five to ten minutes in a sample of the base oil prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

L.3.2.3 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores 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.

L.3.2.4 Resin coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand should 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.

L.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.

L.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 prior to 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.

L.3.2.7 There is an 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 fresh water to further classify the disk.

Note: *Procedure for ceramic disk comparison*—Install a 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 ± 0.1 psi. After opening valve at the bottom of the cell, readjust pressure with the upper valve to 2.0 psi ± 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.

L.3.3 Timer: 30-minute interval.

L.3.4 Thermometer: up to 500°F (260°C).

L.3.5 Graduated cylinder (TC): 25 ml or 50 ml.

L.3.6 High speed mixer.

L.4 Procedure—High Temperature, High Pressure

L.4.1 PREHEATING THE HEATING JACKET

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the midscale 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.

L.4.2 LOADING THE FILTRATION CELL

WARNING: The filtration cell is a pressure vessel. The following safety precautions must be followed to ensure safe operation: The cell material must 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.

L.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.

L.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 is at or near the bottom end of the cell, then unscrew the wrench from the piston.

L.4.2.3 Fill the space above the piston with hydraulic oil to just above the end face.

L.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.

L.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.

L.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.

L.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.

L.4.2.8 Install the O-ring and set the selected disk, or other filtering medium, on top of it.

Note: The disk should be soaked in the base oil for at least five minutes before use. Never reuse disks.

L.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 periodically bleeding off the excess.

L.4.2.10 Unless it was installed at step L.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.

L.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.

L.4.3.1 Refer to Table L.1 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.

L.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 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 CO_2 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 CO_2 assembly and the filtrate drain valve are closed.
d. Set the back pressure assembly aside. It will be installed at Step L.4.3.4.

L.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.

L.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 CO_2 pressurizing unit on top of the receiver and lock it in place with the other retaining pin.

L.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.

L.4.3.6 Refer to Table L-1 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.

L.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.

L.4.4 CONDUCTING THE FILTRATION TEST

L.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.

L.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.

L.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 used, it should be operated at about one stroke per second.

L.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 that the filtrate be recovered directly from the back pressure receiver, not through a drain hose attached at the discharge valve. If a hose must be used, minimize its length to reduce the error caused liquid retention within the hose.

L.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. Record the total volume of filtrate in the graduated cylinder.

L.5 Test Conclusion and Disassembly

L.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.

Table L-1—Starting Cell Pressures and Back-Pressures for Various Test Temperatures

Temperature Range, °F	Heating Pressure or Back Pressure, psi
Less than 200	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

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.

L.5.2 Allow the pressurized cell assembly 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.

L.5.3 Close the valve between the cell and the back pressure receiver.

L.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.

L.5.5 Release the back pressure by turning the T-screw on the regulator counterclockwise until it turns freely.

L.5.6 Bleed the pressure from the back pressure receiver by opening the pressure relief valve on the CO_2 unit.

L.5.7 Open the drain valve on the back pressure receiver and collect the last few drops of filtrate in the graduated cylinder.

L.5.8 After removing the locking pin securing it, remove the CO_2 assembly from the top nipple adapter.

L.5.9 After removing its locking pin, remove the back pressure receiver.

L.5.10 The cell may be opened after its contents have been allowed to cool.

CAUTION: If it is suspected that the cell is still pressured, and the lower end cap does not include a screen, 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. 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 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 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.

L.5.11 Loosen the six set screws securing the 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.

L.5.12 The end cap must be removed with the cell in the vertical position and the filtration end of the cell facing upwards.

L.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 the base oil, then measure and record its thickness and remarks concerning its composition.

L.5.14 Dispose of the mud and wash the inside of the cell with a suitable solvent. 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.

L.5.15 Remove the bottom end cap using the procedure outlined in section L.5.11.

L.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.

L.5.17 Clean the parts for reuse.

L.6 Data Reporting

L.6.1 FILTRATE REPORTING

Report the actual cumulative filtrate volume, in mL, collected through each of the selected time periods.

L.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 more accurately define the spurt loss, collect and record the filtrate more frequently, and plot the data as described in the NOTE following Section L.4.4.1.

L.6.3 CALCULATIONS

PPT Value, mL = 2 x EV_{30}

Spurt Loss, mL = 2 x $[EV_{7.5} - (EV_{30} - EV_{7.5})]$

Static Filtration Rate, $R_S = 2 \times [(EV_{30} - EV_{7.5})] / 2.739$

where:

EV = effluent volume (filtrate) in mL using a 3.5 in^2 disk.

$EV_{7.5}$ = filtrate volume in mL after 7.5 minutes.

EV_{30} = 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.

L.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 M—HIGH TEMPERATURE/HIGH PRESSURE FILTRATION TESTING OF OIL-BASED MUDS USING THE PERMEABILITY PLUGGING APPARATUS AND CELLS EQUIPPED WITH THREADED END CAPS

M.1 Description

M.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.

M.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 as high as 5000 psi (34,500 kPA) and temperatures from ambient to 500°F (260°C). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

M.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 the 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 must 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.

M.2.1 SAFE OPERATION OF THE HYDRAULIC PRESSURIZATION SYSTEM

M.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 the pressure hose from cell at quick coupler.
b. Attempting to the 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.

M.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.

M.2.1.3 When assembling the cell, make sure that the end cap O-rings are in good condition and are properly lubricated.

M.2.2 SAFE PNEUMATIC PRESSURIZATION OF BACK PRESSURE RECEIVER

M.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. CO_2 is normally supplied in small cartridges containing gas 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.

M.2.2.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

M.2.2.3 Leaking pressurization systems, either hydraulic or pneumatic, must be repaired or replaced. Keep gauges, fittings, and hoses in good condition and correct leaks when found. 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.

M.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.

M.2.3 SAFE HEATING

M.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.

M.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.

M.2.4 SAFE ELECTRICAL OPERATION

M.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.

M.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.

M.2.5 SAFE TEST CELL MAINTENANCE

The filtration cell is a pressure vessel and is to be considered 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 cell bodies that show signs of stress cracking or severe pitting.
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.

M.3 Equipment—Permeability Plugging Apparatus—Using Cells 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.

M.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 and the pressure applied to the bottom of the cell. The filter medium is at the top end of the cell, through which the filtrate is collected. Pressure is applied to the cell 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.

M.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 (5171kPa). 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.

M.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 CO_2 pressurizing source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

M.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum well 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 insert 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 either in the field or in a laboratory environment. A stainless steel carrying case with fold down working shelf is available for convenience in field operations.

M.3.2 FILTER MEDIUM

M.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 downhole.

M.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. but, with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, soak the disk for five to ten minutes in a sample of the base oil prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

M.3.2.3 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores 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.

M.3.2.4 Resin coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand should 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.

M.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.

M.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 prior to 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.

M.3.2.7 There is an 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 fresh 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 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 ± 0.1 psi. After opening the valve at the bottom of the cell, readjust pressure with upper valve to 2.0 psi ± 0.1 psi. Measure the 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.

M.3.3 Timer: 30-minute interval.

M.3.4 Thermometer: up to 500°F (260°C).

M.3.5 Graduated cylinder (TC): 25 ml or 50 ml.

M.3.6 High speed mixer.

M.4 Procedure — High Temperature, High Pressure

M.4.1 PREHEATING THE HEATING JACKET

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the midscale 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.

M.4.2 LOADING THE FILTRATION CELL

WARNING: The filtration cell is a pressure vessel. The following safety precautions must 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.

M.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.

M.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 routinely replaced 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 is at or near the bottom end of the cell, then unscrew the wrench from the piston.

M.4.2.3 Lubricate the end face of the cell bore, the horizontal area at the end of the bore, with antiseize compound and fill the space above the piston with hydraulic oil to just above the end face.

M.4.2.4 Lubricate the threads with high temperature grease and then, while pushing on the back pressure ball on the nipple of the end cap to permit the air to escape, 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.

M.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.

M.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—or in a specially constructed stand.

Note: For improved consistency in test results, stir drilling fluid for five minutes immediately before loading the cell.

M.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.

M.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.

M.4.2.9 Install the O-ring and set the selected disk, or other filtering medium, on top of it.

Note: The disk should be soaked in the base oil for at least five minutes before use. Disks should never be reused.

M.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.

M.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 pin 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 periodically bleeding off the excess.

M.4.2.12 Unless it was installed at step M.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.

M.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.

M.4.3.1 Refer to Table L-1 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.

M.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 counter-clockwise enough to 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 CO_2 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 CO_2 assembly and the filtrate drain valve are closed.

d. Set the back pressure assembly aside. It will be installed at Step M.4.3.4.

M.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.

M.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 CO_2 pressurizing unit on top of the receiver and lock it in place with the other retaining pin.

M.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.

M.4.3.6 Refer to Table L-1 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.

M.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.

M.4.4 CONDUCTING THE FILTRATION TEST

M.4.4.1 Use a laboratory timer and collect and record volume of filtrate 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 the 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.

M.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. If a manually actuated pump is used, it should be operated at about one stroke per second until the desired pressure is reached.

M.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.

M.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 that the filtrate be recovered directly from the back pressure receiver, not through a drain hose attached to it. If a hose must be used, minimize its length to reduce the error caused liquid retention within the hose.

M.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. Record the total volume of filtrate in the graduated cylinder.

M.5 Test Conclusion and Disassembly

M.5.1 Disconnect the heating jacket from the power source.

CAUTION: Do not proceed with disassembly until the temperature of the sample in the cell has been reduced to less than 100°F (37.8°C). 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.

M.5.2 Allow the pressurized cell assembly 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.

M.5.3 Isolate the back pressure assembly from its pressure source by turning the T-screw on the back pressure regulator counter-clockwise until it turns freely.

M.5.4 Open the bleed valve on the CO_2 unit to release the pressure in the back pressure receiver.

M.5.5 Remove locking pin and remove the CO_2 pressuring assembly from the top nipple adapter.

M.5.6 Remove locking pin and remove the back pressure receiver.

M.5.7 Open the valve on the hydraulic pump to release cell pressure, then disconnect the hydraulic quick coupler.

M.5.8 Open the filtration valve. This will bleed 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 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 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.

M.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 the tool 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.

M.5.10 Threaded end 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.

M.5.11 Reposition the cell as required so that the filter end is up, then unscrew the top cap.

M.5.12 Remove the filter disk. Use a small knife, small screwdriver, 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 the base oil, then measure and record its thickness and remarks concerning its composition.

M.5.13 Empty the cell and wash it with a suitable solvent. 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.

M.5.14 Remove the bottom end cap using the procedure outlined in sections "M.5.11" and "M.5.12" above, except that the cell position is reversed, and the two-pin spanner wrench is used.

M.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.

M.5.16 Clean the parts for reuse.

M.6 Data Reporting

M.6.1 FILTRATE REPORTING

Report the actual cumulative filtrate volume, in mL, collected through each of the selected time periods.

M.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 Section M.4.4.1.

M.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, RS = $2 \times [(EV_{30} - EV_{7.5})] / 2.739$

where:

EV = effluent volume (filtrate) in mL using a 3.5 in^2 disk.

$EV_{7.5}$ = filtrate volume in mL after 7.5 minutes.

EV_{30} = 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.

M.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 N—OIL-BASED DRILLING MUD REPORT FORM

DRILLING MUD REPORT

OIL MUD REPORT NO.

Date ______________ 20 ______

Depth ______ ☐ MD ☐ TVD ____

Spud Date ___________________

Present Activity ____________________

API Well No.	State		County			Well						S/T

OPERATOR:	CONTRACTOR:	RIG NO:
Report for	Report for	Section, Township, Range

Well Name and No	Field or Block No	County, Parish, Offshore Area	State/Province

BIT DATA	DRILLING STRING	CASING	CIRCULATION DATA

MUD PROPERTIES:				
Sample from	☐F.L. Pit☐	☐F.L. Pit☐	☐F.L. Pit☐	☐F.L. Pit☐
Time Sample Taken				
Flowline Temperature (°F)				
Weight ☐(ppg) ☐(lb/cu ft) ☐(sp gr) _______°F				
Funnel Viscosity (sec/qt) API @ _________ °F				
Plastic Viscosity cp @ ___________°F				
Yield Point (lb/100 ft^2)				
Gel Strength (lb/100 ft^2) 10 sec/10 min	/	/	/	/
Filtrate API (cm^3/30 min)				
API HTHP Filtrate (cm^3/30 min) @ _________°F				
Cake Thickness (32nd in. API/HTHP)	/	/	/	/
Electrical Stability (v), Meter Type ______________				
Retort Solids (% Vol.)				/
Retort Liquid (% Vol.) Oil/Water	/	/	/	
Oil/Water Ratio	/	/	/	
Alkalinity (V_{SA}), (cm^3 H_2SO_4)				
Chloride Whole Mud Cl_{OM} (mg/L)				
Calcium Whole Mud Ca_{OM} (mg/L)				/

MUD PROPERTY SPECIFICATIONS:

Weight	Viscosity	Filtrate

RECOMMENDED TOUR TREATMENT:

REMARKS:

PRODUCTS	SOLIDS EQUIPMENT:

MUD VOLUME	SOLIDS ANALYSIS	MUD RHEOLOGY & HYDRAULICS	COST ANALYSIS

REPRESENTATIVE ______________________________ PHONE ____________________ WAREHOUSE PHONE ____________________

5/00—7C

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Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluids

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American
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Institute

Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluids

Exploration and Production Department

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FOREWORD

This recommended practice is under the jurisdiction of the API Committee on Standardization of Drilling Fluid Materials.

Additional publications under jurisdiction of this committee: Spec 13A, *Specification for Drilling Fluid Materials,* covers specifications and test procedures for barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, starch, technical-grade low viscosity CMC, technical-grade high viscosity CMC and OCMA grade bentonite.

RP 13B-1	*Recommended Practice for Standard Procedure for Field Testing Water-Based Drilling Fluids*
Bul 13C	*Bulletin on Drilling Fluids Processing Equipment*
Bul 13D	*Bulletin on the Rheology of Oil Well Drilling Fluids*
RP 13E	*Recommended Practice for Shale Shaker Screen Cloth Designation*
RP 13G	*Recommended Practice for Drilling Mud Report Form*
RP 13I	*Recommended Practice for Laboratory Testing of Drilling Fluids*
RP 13J	*Recommended Practice for Testing Heavy Brines*
RP 13K	*Recommended Practice for Chemical Analysis of Barite*

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Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

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Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluid

1 Scope

The purpose of this recommended practice is to provide standard procedures for the testing of oil-based 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.

This recommended practice is organized to follow the tests as listed on the API Drilling Mud Report form (API RP 13G, Third Edition, February 1992). Additional tests are given in the Appendix of this recommended practice.

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

2 References

API

RP 13B-1	*Recommended Practice for Standard Procedure for Field Testing Water-Based Drilling Fluids*
Bul 13D	*Bulletin on the Rheology of Oil Well Drilling Fluids*
RP 13G	*Recommended Practice for Drilling Mud Report Form*

ASTM[1]

D611-82	*Standard Test Method for Aniline Point and Mixed Aniline Point for Petroleum Products and Hydrocarbon Solvents*
Spec E1	*Standard Specification for ASTM Thermometers*

IP[2]

2/76(1976)

[3]Clark, E. C. W. and Grew, D. N., *J. Phys. Chem. Ref. Data,* 1977, Vol. 6, No. 2, pp. 385–407.

[4]Staples, B. R. and Nuttall, *J. Phys. Chem. Ref. Data,* 1985, Vol. 14, No. 2, pp. 489–610.

[5]Stokes, R. H. and Robinson, R. A., "Standard Solutions for Humidity Control at 25°C," *Ind. Eng. Chem.* 41, 2013, 1949.

[6]Watkins, T. E. and Nelson, M. D., "High Temperature Gelation of Drilling Fluids," *AIME Transactions,* Vol. 193, 1953, pp. 213–218.

3 Mud Weight (Density)

3.1 DESCRIPTION

This test procedure is a method for determining the weight of a given volume of liquid. Mud weight may be expressed as pounds per gallon (lb/gal), pounds per cubic foot (lb/ft^3), grams per cubic centimeter (g/cm^3), or kilograms per cubic meter (kg/m^3).

3.1.1 Equipment

Following is the list of equipment needed:

a. Any instrument of sufficient accuracy to permit measurement within ±0.1 lb/gal (or 0.5 lb/ft^3, 0.01 g/cm^3, 10 kg/m^3) may be used. The mud balance (Figures 1 and 2) is the instrument generally used for mud weight determinations. The mud balance is designed such that the mud cup, at one end of the beam, is balanced by a fixed counterweight at the other end and a sliding rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (Attachments for extending the range of the balance may be used when necessary.)

b. Thermometer: 32–220°F (0–105°C).

3.1.2 Procedure

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

3.1.2.2 Measure the temperature of the mud and record on the Drilling Mud Report form.

3.1.2.3 Fill the clean, dry cup with mud to be tested; put the cap on the filled mud cup and rotate the cap until it is firmly seated. Insure that some of the mud is expelled through the hole in the cap in order to free any trapped air or gas (see Appendix D of API RP 13B-1 for Air Removal).

3.1.2.4 Holding cap firmly on mud cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

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

3.1.2.6 Read the mud weight at edge of the rider toward the mud cup. Make appropriate corrections when a range extender is used.

3.1.3 Procedure—Calibration

The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft^3 (1000 kg/m^3) at 70°F (21°C). If it does not, adjust the

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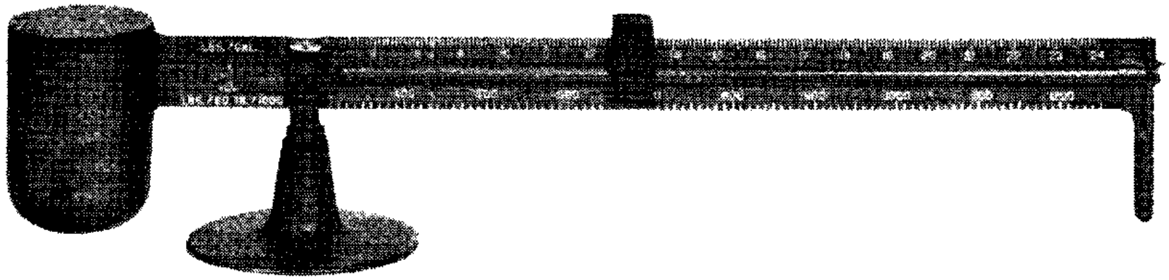
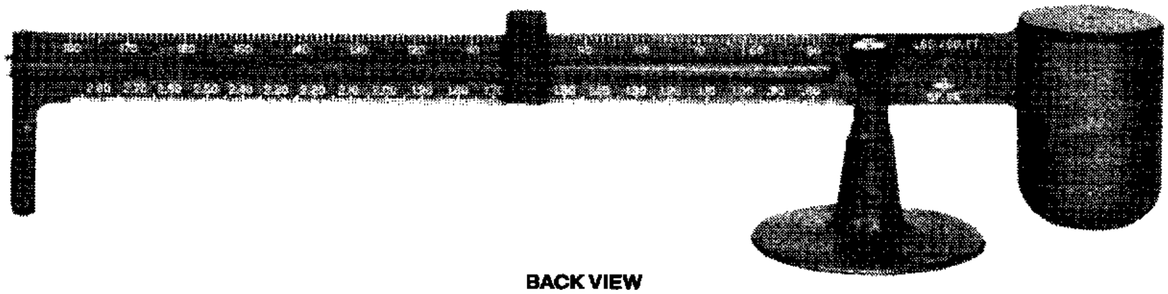
[2] Institute of Petroleum, 61 New Cavendish Street, London W1M 8AR, England.



FRONT VIEW



BACK VIEW

Figure 1—Mud Balance

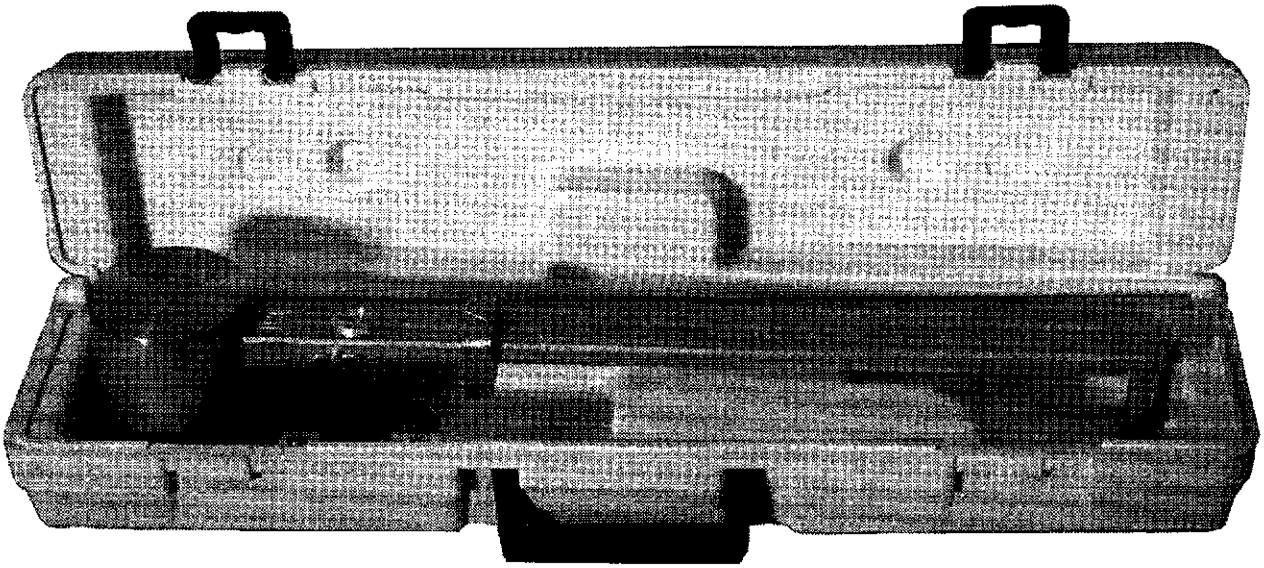


Figure 2—Mud Balance and Case

balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

3.1.4 Calculation

3.1.4.1 Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft^3 (0.01 g/cm^3, 10 kg/m^3).

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

$$Specific\ gravity = g/cm^3 = \frac{lb/ft^3}{62.43} = \frac{lb/gal}{8.345} \qquad (1)$$

$$kg/m^3 = (lb/ft^3)\ (16) = (lb/gal)\ (120) \qquad (2)$$

$$Mud\ gradient,\ psi/ft = \frac{lb/ft^3}{144}, \frac{lb/gal}{19.24}, \text{ or } \frac{kg/m^3}{2309} \qquad (3)$$

A list of density conversions may be found in Table 1.

3.2 ALTERNATE MUD WEIGHT METHOD

3.2.1 Description

The density of a mud containing entrained air or gas can be determined more accurately by using the pressurized fluid density balance as described in this section. The pressurized fluid density balance is similar in operation to the conventional mud balance, the difference being that the slurry sample can be placed in a fixed volume sample cup under pressure.

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

3.2.2 Equipment

Following is a list of equipment needed:

a. Any instrument of sufficient accuracy to permit measurement within ±0.1 lb/gal (or 0.5 lb/ft^3, 0.01 g/cm^3, 10 kg/m^3) may be used. The pressurized mud balance (Figures 3 and 4) is the instrument generally used for pressurized balance mud weight determinations. The pressurized mud balance is designed such that the mud cup and screw on lid, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (see Figures 3 and 4).

b. Thermometer: 32–220°F (0–105°C).

Table 1—Density Conversion

Pounds per Gallon (lb/gal)	Pounds per Cubic Foot (lb/ft^3)	Grams per Cubic Centimeter (g/cm^3)[a]	Kilograms per Cubic Meter (kg/m^3)
6.5	48.6	0.78	780
7.0	52.4	0.84	840
7.5	56.1	0.90	900
8.0	59.8	0.96	960
8.3	62.3	1.00	1000
8.5	63.6	1.02	1020
9.0	67.3	1.08	1080
9.5	71.1	1.14	1140
10.0	74.8	1.20	1200
10.5	78.5	1.26	1260
11.0	82.3	1.32	1320
11.5	86.0	1.38	1380
12.0	89.8	1.44	1440
12.5	93.5	1.50	1500
13.0	97.2	1.56	1560
13.5	101.0	1.62	1620
14.0	104.7	1.68	1680
14.5	108.5	1.74	1740
15.0	112.2	1.80	1800
15.5	115.9	1.86	1860
16.0	119.7	1.92	1920
16.5	123.4	1.98	1980
17.0	127.2	2.04	2040
17.5	130.9	2.10	2100
18.0	134.6	2.16	2160
18.5	138.4	2.22	2220
19.0	142.1	2.28	2280
19.5	145.9	2.34	2340
20.0	149.6	2.40	2400
20.5	153.3	2.46	2460
21.0	157.1	2.52	2520
21.5	160.8	2.58	2580
22.0	164.6	2.64	2640
22.5	168.3	2.70	2700
23.0	172.1	2.76	2760
23.5	175.8	2.82	2820
24.0	179.5	2.88	2880

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[a] Same as specific gravity (sg).

3.2.3 Procedure

3.2.3.1 Fill the sample cup to a level slightly below the upper edge of the cup [approximately ¼ inch (6.4 mm)].

3.2.3.2 Place the lid on the cup with the attached check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the

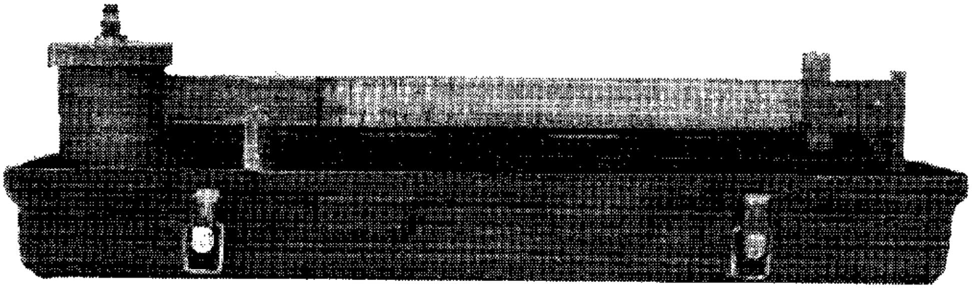

Figure 3—Pressurized Fluid Density Balance

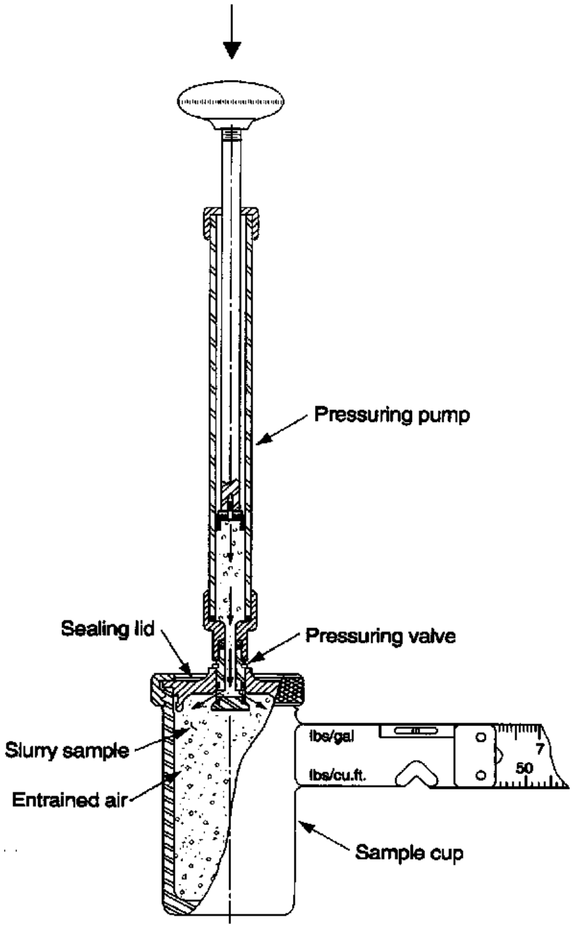

Figure 4—Typical Pressurized Fluid Density Balance Diagram

cup. Any excess slurry will be expelled through the check valve. When the lid has been placed on the cup, pull the check valve up in the closed position, rinse off the cup and threads with oil, and screw the threaded cap on the cup.

3.2.3.3 The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submersing its end in the slurry with the piston rod in the completely inward position. The piston rod is then drawn upward thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean up of the plunger mechansm.

3.2.3.4 Push the nose of the plunger onto the mating O-ring surface of the cap valve. Pressurize the sample cup by maintaining a downward force on the cylinder housing in order to hold the check valve down (open) and at the same time forcing the piston rod inward. Approximately 50 pounds (225 N) force or greater should be maintained on the piston rod (see Figure 4).

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

3.2.3.6 The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place instrument on the knife edge as illustrated. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the attached bubble is centered between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of lb/gal, specific gravity, psi/1000 ft and lb/ft³.

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

3.2.3.8 Clean the cup and rinse thoroughly with base oil.

3.2.4 Procedure—Calibration

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft³ (1000 kg/m³) at 70°F (21°C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

3.2.5 Calculation

3.2.5.1 Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft³ (0.01 gm/cm³, 10 kg/m³).

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

$$Density = g/cm^3 = \frac{lb/ft^3}{62.43} = \frac{lb/gal}{8.345} \quad (4)$$

$$kg/m^3 = (lb/ft^3)(16) = (lb/gal)(120) \quad (5)$$

$$Mud/gradient = \frac{lb/ft^3}{144}, \frac{lb/gal}{19.24}, = \frac{k/gm^3}{2309} \quad (6)$$

A list of density conversions may be found in Table 1.

4 Viscosity and Gel Strength

4.1 DESCRIPTION

The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

a. Marsh funnel—a simple device for indicating viscosity on a routine basis.
b. Direct-indicating viscometer—a mechanical device for measurement of viscosity at varying shear rates.

Viscosity and gel strength are measurements that relate to the flow properties of muds. The study of deformation and flow of matter is rheology. An in-depth statement of rheology is made in API Bulletin 13D: ***The Rheology of Oil-Well Drilling Fluids.***

4.2 MARSH FUNNEL

4.2.1 Equipment

Following is a list of equipment needed:

a. Marsh Funnel: A Marsh funnel (see Figure 5) is calibrated to outflow one quart (946 cm³) of fresh water at a temperature of 70 ±5°F (21 ±3°C) in 26 ±0.5 seconds. A graduated cup is used as a receiver. Specifications for the Marsh funnel are as follows:

1. Funnel Cone	
Length	12.0 in. (305 mm)
Diameter	6.0 in. (152 mm)
Capacity to bottom of screen	1500 cm³
2. Orifice	
Length	2.0 in. (50.8 mm)
Inside Diameter	3⁄16 in. (4.7 mm)
3. Screen	12 mesh

Has 1⁄16 in. (1.6 mm) openings and is fixed at a level 3⁄4 in. (19.0 mm) below top of funnel.

b. Graduated cup: one-quart.
c. Stopwatch.
d. Thermometer: 32–220 ±2°F (0–105 ±1°C).

4.2.2 Procedure

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

Figure 5—Marsh Funnel and Cup

4.2.2.2 Move finger covering orifice and start stopwatch. Measure the time for mud to fill to one-quart (946 cm^3) mark of the cup.

4.2.2.3 Measure temperature of fluid in degrees F (C).

4.2.2.4 Report the time to nearest second as Marsh funnel viscosity. Report the temperature of fluid to nearest degree F (C).

4.3 DIRECT-INDICATING VISCOMETER

4.3.1 Equipment

Following is a list of equipment needed:

a. Direct-indicating viscometers are rotational types of instruments powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity (rpm). The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 rpm and 600 rpm.

Specifications for the direct-indicating viscometer are as follows:

1. Rotor Sleeve
   Inside diameter .... 1.450 in. (36.83 mm)
   Total length .... 3.425 in. (87.00 mm)
   Scribed line .... 2.30 in. (58.4 mm) above the bottom of sleeve. Two rows of 1/8 in. (3.18 mm) holes spaced 120 degrees (2.09 radians) apart, around rotor sleeve just below scribed line.
2. Bob
   Diameter .... 1.358 in. (34.49 mm)
   Cylinder length .... 1.496 in. (38.00 mm)
   Bob is closed with a flat base and a tapered top.
3. Torsion Spring Constant
   386 dyne-cm/degree deflection
4. Rotor Speeds
   High speed .... 600 rpm
   Low speed .... 300 rpm

The following are types of viscometers used to test drilling fluids:

1. Hand-cranked instrument (Figure 6) has speeds of 300 rpm and 600 rpm. A knob on the hub of the speed-change lever is used to determine gel strength.
2. The 12-volt, motor driven instrument (Figure 7) also has speeds of 300 and 600 rpm. A governor-release switch permits high shearing before measurement, and a knurled hand-wheel is used to determine gel strength.
3. The 115-volt instrument (Figure 8) is powered by a two-speed synchronous motor to obtain speeds of 3, 6, 100, 200, 300, and 600 rpm. The 3-rpm speed is used to determine gel strength.
4. The variable speed 115-volt or 240-volt instrument (Figure 9) is motor powered to obtain all speeds between 1 and 625 rpm. The 3-rpm speed is used to determine gel strength.

b. Thermostatically controlled viscometer cup: (Figures 10 and 11).

c. Stopwatch.

d. Thermometer: 32–220 ±2°F (0–105 ±1°C).

4.3.2 Procedure

CAUTION: Maximum operating temperature is 200°F (93°C). If fluids above 200°F (93°C) are to be tested, a solid metal bob or a hollow metal bob, with a completely dry interior, should be used. Liquid trapped inside a hollow bob may vaporize when immersed in high temperature fluid and cause the bob to explode.

4.3.2.1 Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume in the cup for the displacement of the viscometer bob and sleeve. The bob and sleeve will displace approximately 100 cm^3 of drilling fluid. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of sampling. Test should be made at either 120 ±2°F (50 ±1°C) or 150 ±2°F (65 ±1°C). The place of sampling should be stated on the report.

4.3.2.2 Heat or cool the sample to the selected temperature. Intermittent or constant shear at the 600 rpm speed should be used to stir the sample while heating or cooling to obtain a uniform sample temperature. After the cup temperature has reached the selected temperature, immerse the thermometer into the sample and continue stirring until the

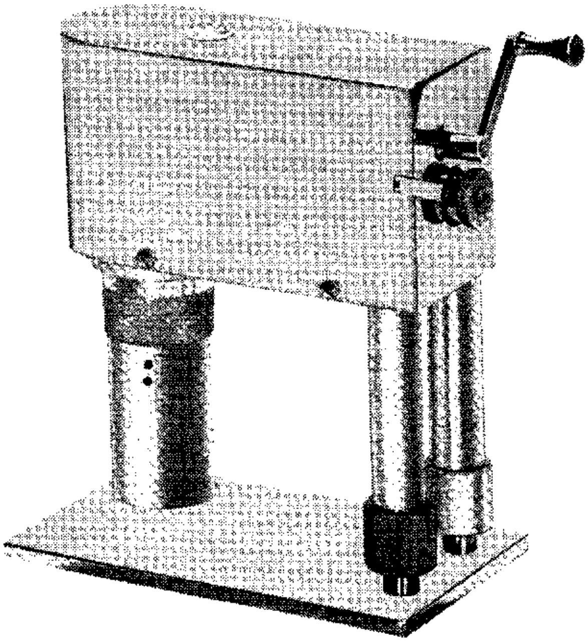

Figure 6—Hand-Crank Viscometer

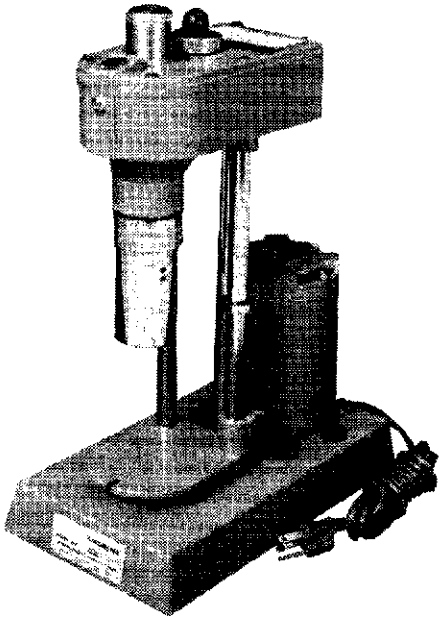

Figure 8—115-Volt Motor-Driven Viscometer

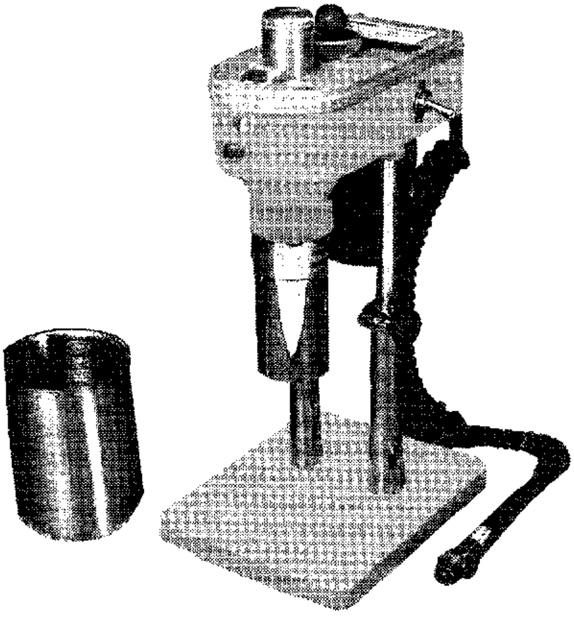

Figure 7—12-Volt Motor-Driven Viscometer

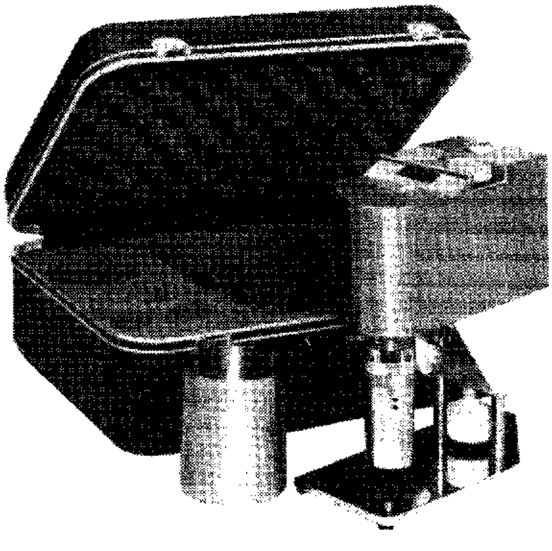

Figure 9—Variable Speed Viscometer

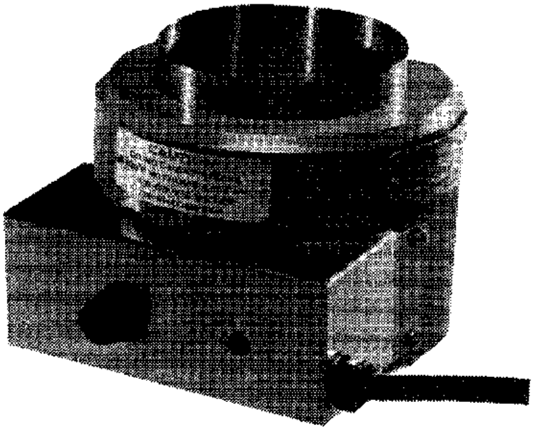

Figure 10—Heated Jacket With Cup

sample reaches the selected temperature. Record the temperature of the sample.

4.3.2.3 With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.

4.3.2.4 Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.

4.3.2.5 Stir drilling fluid sample for 10 seconds at high speed.

4.3.2.6 Allow mud to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft² (Pa).

4.3.2.7 Restir the mud at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in 4.3.2.6 and report the maximum reading as 10 minute gel in lb/100 ft² (Pa).

4.3.3 Calculation

$$Plastic\ Viscosity,\ \text{cP} = \begin{bmatrix} 600\ rpm \\ reading \end{bmatrix} - \begin{bmatrix} 300\ rpm \\ reading \end{bmatrix} \quad (7)$$

$$Yield\ Point,\ \text{lb/100 ft}^3 = \begin{bmatrix} 300\ rpm \\ reading \end{bmatrix} - Plastic\ Viscosity \quad (8)$$

$$Apparent\ Viscosity,\ \text{cP} = \frac{600\ rpm\ reading}{2} \quad (9)$$

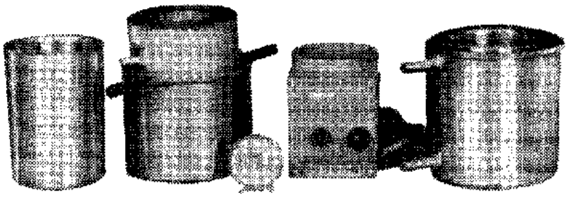

Figure 11—Thermocup

5 Filtration

5.1 DESCRIPTION

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

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

5.1.3 Filtration tests are performed at both ambient (low) temperature and at high-temperature conditions to provide data for comparison purposes. Two filtration procedures are given—one procedure is for testing up to 350°F (177°C) and one procedure for testing from 351°F (177°C) up to 450°F (232°C). Use only the filtration equipment and procedure specified for the temperature required.

Note 1: No API low-temperature filtration test procedure for oil-based drilling fluids is specified herein, but can be performed much like the water-based drilling fluid test, found in API RP 13B-1.

Note 2: The API Low Temperature/Low Pressure filter press must have a filter area of 4520 to 4640 square millimeters, which is a diameter of 75.86 to 76.86 millimeters. The filter press gasket is the determining factor of the filter area. It is recommended that the filter press gasket used be tested by a conical gauge that has the maximum (76.86 millimeters) and the minimum (75.86 millimeters) marked on it. Any filter press gasket found out of these ranges (either larger or smaller than the markings) will be discarded.

5.1.4 Equipment for testing filtration up to and including 350°F (177°C) can be either the 175-, 250-, or 500-cm³ units. For testing above 351°F (178°C) only the 500-cm³ unit can be used and must be equipped with a thermocouple to measure the temperature of drilling fluid in the cell and also a porous stainless steel filter media must be used.

5.2 HIGH-TEMPERATURE/HIGH-PRESSURE TEST UP TO AND INCLUDING 350°F (177°C)

5.2.1 Equipment

Filtration testing equipment may not be rated to perform safely at temperatures and pressures called for in this procedure. It is essential to know and to follow the manufacturer's recommendations regarding maximum operating temperature, pressure, and sample volumes. Failure to do so could result in injury.

a. The high-temperature/high-pressure filter press apparatus consists of:

1. A filter cell to contain working pressures up to 1300 psi (8970 kPa) at temperature.
2. A pressurized gas source, such as carbon dioxide or nitrogen with regulators.

Note: Nitrogen is preferred.

3. A heating system to heat to 350°F (177°C).
4. A high-pressure filtrate collection vessel maintained at proper back pressure (see Table 2) to avoid flashing or evaporation of the filtrate.

CAUTION: Do no use nitrogen oxide cartridges as pressure sources for high-temperature/high-pressure filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

Figures 12 and 13 are photographs of two filtration units used for testing up to 350°F (177°C).

b. Filter cell: the filter cell contains a thermometer well. It is fitted with a removable end, a filter-media support and with oil-resistant seals. Valve stems on each end of the cell can be opened or closed during the test.

c. Filter medium: Whatman No. 50 or S&S 576, or equivalent, filter paper.

d. Timer: to measure 30-minute interval. Mechanical or electronic.

e. Thermometer: to measure up to 500°F (260°C) with 5-inch (12.5-cm) or longer stem.

f. Receivers: 10-cm^3 and 20-cm^3 long, slender graduated cylinders, as used for oil, water and solids content. (See Section 6 and Figure 15).

g. Receiver (optional): 25-cm^3 glass graduated cylinder (TC).

h. Field Mixer: cup type, to operate at 10,1000 to 15,000 rpm.

i. Ruler: to measure filter cake thickness in $\frac{1}{32}$-inch increments.

5.2.2 Procedure—Temperatures to 350°F (177°C)

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

Note: If the filtration unit is equipped with a thermocouple to measure drilling fluid temperature inside the cell (test temperature) then that temperature should be monitored and maintained during the filtration test. Results may differ from this standard procedure, which uses the cell wall temperature as the test temperature. Under "Comments" section, note if the thermocouple method was used.

Table 2—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure of Water		Minimum Back Pressure	
°F	°C	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
350	177	135	932	160	1104

5.2.2.2 Stir the drilling fluid sample for 5 minutes using the field mixer. Pour the fluid sample into the filter cell, leaving at least 1 inch (2.5 cm) space in the cell, to allow for fluid expansion. Install the filter paper in the cell.

5.2.2.3 Complete the assembly of the filter cell. With upper and lower valve stems closed, place the cell in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

5.2.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place.

Note: Be sure the collection vessel is completely free of water or oil.

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

5.2.2.6 While keeping the two valve stems closed, adjust the upper pressure regulator to a pressure 100 psi (690 kPa) higher than the "Minimum Back Pressure" value, as shown in Table 2. Next, set the lower regulator to the pressure "Minimum Back Pressure" shown in Table 2 for the test temperature. Maintain this pressure until the test temperature is reached.

Note: If time required to reach test temperature exceeds one hour, the heater may be defective and validity of the test is questionable.

5.2.2.7 When the sample reaches the selected test temperature, open the lower valve stem and immediately increase the pressure on the upper regulator to 500 psi (3450 kPa) higher than the back pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ±5°F (±3°C) during the test. If the back pressure rises above the selected back pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back pressure.

5.2.2.8 Collect the filtrate in the long, slender graduated glass cylinder (or graduated cylinder). Read the 30-minute total (water plus oil) filtrate volumes. Also read volumes of solid and water phases, if present.

Note: A long, slender, glass cylinder, rather than a conventional graduated cylinder, is recommended because the long cylinder allows more accurate detection and measurements of volumes of oil, water and solids in the filtrate. Heating of the cylinder near an emulsion interface can improve separation of water, solids and oil in the filtrate.

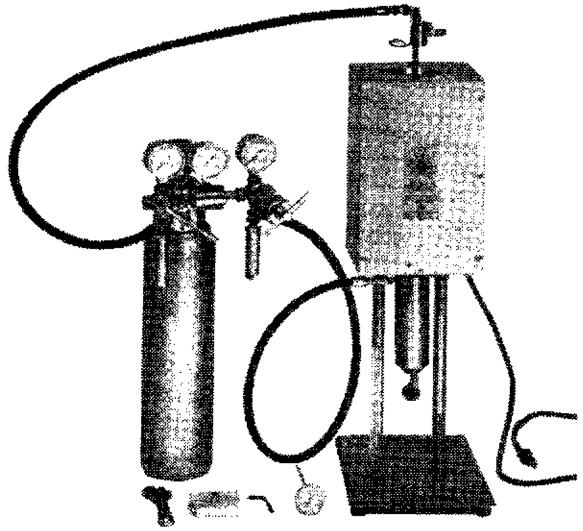

Figure 12—High Temperature Filter Press

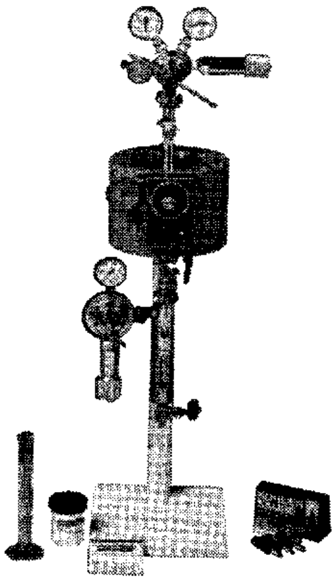

Figure 13—High Temperature Filter Press

5.2.2.9 Immediately after collecting the 30-minute filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the gas pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 125°F (52°C). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION: Pressure in the filter cell can be high and dangerous, even after the cell is cooled. Be careful not to open the cell with pressure trapped inside.

5.2.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits stem. Carefully disassemble the cell. Be sure no pressure is trapped before dislodging the cap.

5.2.2.11 Pour the liquid from the cell.

5.2.2.12 Remove the filter cake on the paper. Measure the filter cake thickness at its center.

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

5.2.3 Calculation

5.2.3.1 Mathematically calculate the volume of filtrate for high-temperature/high-pressure filtration.

$$\text{Filtrate vol., cm}^3 = 2\,(\text{volume collected in 30 minutes, cm}^3) \quad (10)$$

Record the filtrate volume.

Note: Filtrate volumes (oil, water, solids) collected in the glass cylinder must be mathematically corrected to correspond to a standard filter area of 7.1 in² (4580 mm²). High-pressure filter cells usually have half the standard filter area, 3.5 in² (2258 mm²), thus all the observed volumes must be doubled before reporting.

5.2.3.2 Under "Comments" section of the report, record the doubled volumes of water or solids observed in the 30-minute filtrate.

5.2.3.3 Report the cake thickness to the nearest 1/32 inch (0.80 mm) on the Oil Mud Report Form.

5.3 HIGH-TEMPERATURE/HIGH-PRESSURE TEST 351°F (177°C) UP TO AND INCLUDING 450°F (232°C)

5.3.1 Equipment

CAUTION: Not all filtration testing equipment is rated to perform safely at temperatures and pressures called for in this procedure. It is essential to know and to follow the manufacturer s recommendations regarding maximum safe operating temperature, pressure and sample volumes. Failure to do so could result in injury. The 175- and 250-cm³ filtration cells are not recommended for use at these higher temperatures and pressures.

a. The high-temperature/high-pressure filter press apparatus consists of:

1. A filter cell to contain working pressures up to 2250 psi (14,490 kPa) at a temperature of 450°F (232°C). The filter cell is equipped with an internal thermocouple to monitor temperature of a drilling fluid sample near its center in the cell. It is fitted with oil-resistant seals. Valve stems on each end of the cell can be opened or closed during a test.

2. A pressurized nitrogen gas source, with regulators.
3. A heating system to heat up to 450°F (232°C).
4. A high-pressure filtrate collection vessel maintained at proper back pressure to avoid flashing or evaporation of the filtrate. See Table 3.

CAUTION: Do not use nitrous oxide cartridges as pressure sources for high-temperature/high-pressure filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

b. Filter medium: Dynalloy® X-5, or equivalent, porous stainless steel disk to fit and seal in the cells.

c. Timer: to measure 30-minute intervals. Mechanical or electronic.

d. Thermometer: to measure up to 500°F (260°C) with 8-inch (20.3-cm) or longer stem.

e. Receivers: 10-cm^3 and 20-cm^3 long, slender graduated glass cylinders, as used for oil, water and solids content (see Section 6 and Figure 15).

f. Receiver (optional): 25-cm^3 glass cylinder (TC).

g. Field Mixer: cup type, to operate at 10,000 to 15,000 rpm.

h. Ruler: to measure filter cake thickness in 1/32-inch (0.80 mm) increments.

Note: Dynalloy is a registered trademark of Memtec America Corporation.

5.3.2 Procedure—Temperatures from 351°F (177°C) to 450°F (232°C)

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

5.3.2.2 Stir the drilling fluid sample for 5 minutes using the field mixer. Pour the fluid sample into the filter cell, leaving at least 1 inch (2.5 cm) space in the cell to allow for drilling fluid expansion. Install the porous stainless steel filter disk in the cell.

5.3.2.3 Complete the assembly of the filter cell. Install the thermocouple into the cell to monitor the temperature of the fluid near its center in the cell. With upper and lower valve stems closed, place the cell in the heating jacket. Connect the thermocouple to the temperature readout instrument and determine that it is reading correctly.

5.3.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place.

Note: Be sure the filtrate collection vessel is free of residual water or oil.

5.3.2.5 Connect the pressure-regulated gas source to the upper valve. Connect a similar source to the lower collection vessel and lock these connections in place.

Table 3—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure of Water		Minimum Back Pressure	
°F	°C	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
350	177	135	932	160	1104
Limit of Normal Field Testing					
350	177	135	932	160	1104
400	204	247	1704	275	1898
450	232	422	2912	450	3105

5.3.2.6 While keeping the two valve stems closed, adjust the upper pressure regulator to a pressure 100 psi higher than the "Minimum Back Pressure" value, as shown in Table 3. Next, set the lower regulator to the pressure "Minimum Back Pressure" shown in Table 3 for the test temperature.

5.3.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, open the lower valve stem and immediately increase the pressure on the upper regulator to 500 psi (3450 kPa) higher than the back pressure. This will start the filtration process.

Note: Start the filtration process immediately upon reaching test temperature, as read by the thermocouple in the fluid. This will minimize time during which solids settling is most likely to occur in the heated fluid.

5.3.2.8 Start the timer. Maintain the test temperature to within ±5°F (±3°C) during the test. If the back pressure rises above the selected back pressure during the test, cautiously draw off an collect a portion of the filtrate to reduce the back pressure.

5.3.2.9 Collect the filtrate in the long, slender glass cylinder, or the graduated cylinder. Read the 30-minute total (water plus oil) filtrate volume. Also read volumes of solid and water phases, if present.

5.3.2.10 Immediately after collecting the 30-minute filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the gas pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 125°F (52°C). Keep the cell upright during cooling, depressurization, and disassembly.

CAUTION: Pressure in the filter cell can be high and dangerous, even after the cell is cooled. Be careful not to open the cell with pressure trapped inside.

5.3.2.11 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits stem. Carefully disassemble the cell. Be sure no pressure is trapped before dislodging the cap.

5.3.2.12 Pour the liquid from the cell.

5.3.2.13 Remove the filter cake on the Dynalloy disk. Measure the filter cake thickness at its center.

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

5.3.3 Calculation

5.3.3.1 Mathematically calculate the volume of filtrate for high-temperature/high-pressure filtration.

$$\textit{Filtrate vol., cm}^3 = 2\ (\textit{volume collected in } 30 \textit{ minutes, cm}^3) \quad (11)$$

Record the filtrate volume on the Oil Mud Report Form.

Note: Filtrate volumes (oil, water, solids) collected in the glass cylinder must be mathematically "corrected" to correspond to a standard filter area of 7.1 in^2 (4580 mm^2). High-pressure filter cells usually have half the standard filter area, 3.5 in^2 (2258 mm^2), thus all the observed volumes must be doubled before reporting.

5.3.3.2 Under "Comments" section of the report, record the doubled volumes of water or solids observed in the 30-minute filtrate.

5.3.3.3 Report the cake thickness to the nearest 1/32 inch (0.80 mm) on the Oil Mud Report Form.

6 Oil, Water, and Solids Content

6.1 DESCRIPTION

6.1.1 A retort test measures water and oil released from an oil mud sample when heated in a calibrated and properly operating "Retort" instrument.

6.1.2 Knowledge of water, oil and solids content is fundamental to proper control of mud properties such as: oil/water ratio, rheology, density, filtration and salinity of aqueous phase. Knowledge of solids in an oil mud is essential to evaluation of solids control equipment.

6.1.3 In a retort test, a known volume of oil mud is heated in a retort instrument to vaporize the liquid components. These vapors are then condensed and collected in a precision graduated receiver. Volume percent retort solids is calculated by subtracting the total liquid volume from the starting volume of oil mud.

6.2 EQUIPMENT

Following is a list of equipment needed:

a. Retort instrument: The recommended oil mud retort instruments are 10-cm^3 or 20-cm^3 volume with an external heating jacket (see Figure 14).

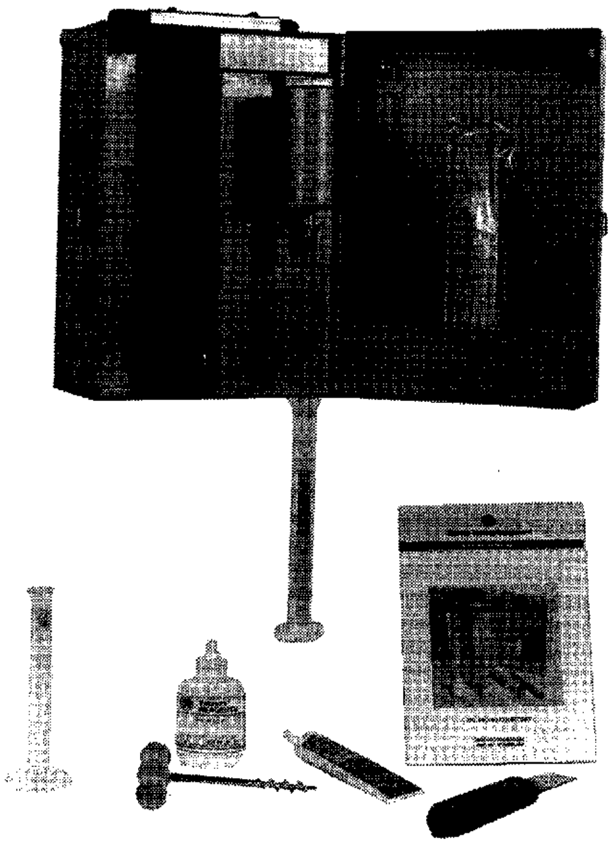

Figure 14—Retort for Liquid and Solid Content

Note: A 50-cm^3 retort, although not specified, is available.

Specifications for retort instruments are as follows:

1. Retort assembly: retort body, cup and lid.
   (a) Material: 303 stainless steel, or equivalent.
   (b) Volume: retort cup with lid.

Cup volume:	10-cm^3	20-cm^3
Precision:	±0.05 cm^3	±0.1 cm^3

Note: *Verification of retort cup volume.* The retort cup volume with lid should be verified gravimetrically. The procedure and calculations are in 6.5.

2. Condenser: capable of cooling the oil and water vapors below their liquification temperature.
3. Heating jacket: nominal 350 watts.
4. Temperature controller: capable of limiting temperature of retort to 930 ±70°F (500 ±38°C).

b. Liquid receiver: specially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops (see Figure 15).

Specifications for liquid receivers are the following:

Total volume:	10-cm^3	20-cm^3
Precision (0 to 100%):	±0.05 cm^3	±0.05 cm^3
Outside diameter:	10 mm	13 mm
Well thickness:	1.5 ± 0.1 mm	1.2 ±0.1 mm

Figure 15—Liquid Receivers

Frequency of graduation marks:		
(0 to 100%):	0.10 cm^3	0.10 cm^3
Calibration:	To contain "TC" at 68°F (20°C)	
Scale:	cm^3 or volume percent	
Material:	Pyrex®,[3] or equivalent	

Note: Verification of receiver volume. The receiver volume should be verified gravimetrically. The procedure and calculations are in 6.5.

c. Fine steel wool (No. 000): for packing retort body.

Note: "Liquid Steel Wool" is not recommended.

d. Thread sealant/lubricant: high temperature lubricant, e.g., Never-Seez®,[4] or equivalent.

e. Pipe cleaners: to clean condenser and retort stem.

f. Brush: to clean receivers.

g. Retort spatula: to clean retort cup.

h. Corkscrew: to remove spent steel wool.

i. Syringe (10-cm^3 or 20-cm^3): to fill retort cup.

j. Screen, 12-mesh, or Marsh funnel (See 4.2).

6.3 PROCEDURE

6.3.1 Clean and dry the retort assembly and condenser.

6.3.2 Collect and prepare the oil mud sample as follows:

a. Collect a representative mud sample and pour it through a 12-mesh Marsh funnel screen. Record sample temperature.

Note: Sample temperature should be within 10°F (5°C) of the temperature at which mud density was determined.

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

Note: Air or gas entrapment in retort sample will result in erroneously high retort solids due to reduced mud sample volume.

6.3.3 Using a clean syringe, fill retort cup slowly to avoid air entrapment. Lightly tap side of cup to expel air. Place lid on the cup. Rotate lid to obtain a proper fit. Be sure a small excess of mud flows out of the hole in the lid. Wipe excess mud from lid; avoid wicking out mud.

6.3.4 Pack retort body with steel wool.

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

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

6.3.7 Place the clean and dry liquid receiver below condenser outlet.

Note: The length of the receiver may require that it be angled out from the retort and perhaps supported off the edge of the work table.

6.3.8 Turn on the retort. Allow it to run a minimum of 45 minutes.

Note: If mud boils over into receiver, the test must be rerun. Pack the retort body with a larger amount of steel wool (see 6.3.4) and repeat the test.

6.3.9 Remove the liquid receiver and allow it to cool. Read and record: (a) total liquid volume, (b) oil volume, and (c) water volume in the receiver.

Note: If an emulsion interface is present between the oil and water phases, heating the interface may break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. *Carefully* heat the receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool. Read the water volume at the lowest point of the meniscus (see Figure 16).

6.3.10 Turn off the retort. Allow it to cool before cleaning.

6.4 CALCULATION

The measured volumes (cm^3) of oil and water are converted into volume percents based on volume of whole mud (cm^3) in the retort cup.

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[3] Pyrex® is a registered trademark of Corning Glass Works.

[4] Never-Seez® is a registered trademark of Never-Seez Compound Corp.

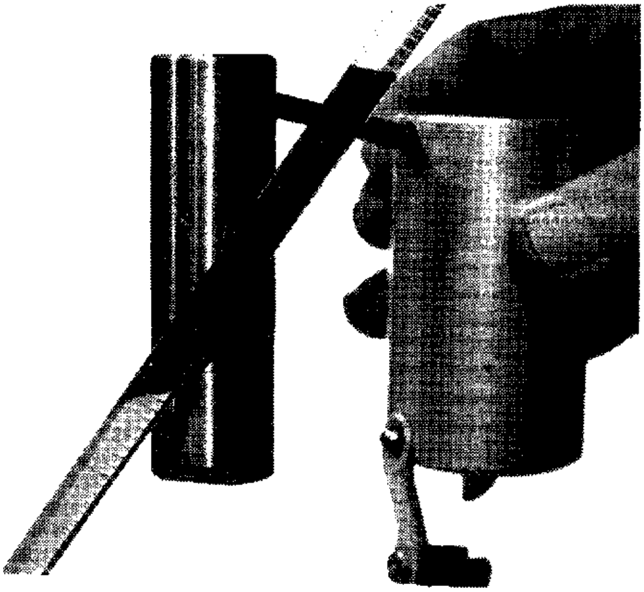


Figure 16—Heating the Emulsion Band on Condenser

Note: The nominal retort cup volume, V_{RC}, is usually assumed to be correct, either 10-cm³ or 20-cm³. However, it should be verified (see 6.5) that the retort cup meets volumetric specifications (see 6.2.a, 1.b).

6.4.1 Calculate volume percent oil, V_o:

$$V_o = \frac{100\,(oil\ volume,\,\mathrm{cm}^3)}{V_{RC}} \qquad (12)$$

6.4.2 Calculate volume percent water, V_w:

$$V_w = \frac{100\,(water\ volume,\,\mathrm{cm}^3)}{V_{RC}} \qquad (13)$$

6.4.3 Calculate volume percent retort solids V_s:

$$V_s = 100 - (V_w + V_o) \qquad (14)$$

Note: The calculated "retort solids" overestimates the volume of suspended solids because of dissolved salts. A volumetric correction for salinity can be made using known volume factors for salt solutions (see 9.5).

6.5 PROCEDURE—VERIFICATION OF RETORT CUP AND LIQUID RECEIVER VOLUMES

6.5.1 Description—This procedure is used to verify that the retort cup and the liquid receiver meet specifications stated in 4.2.b.

6.5.2 Following is a list of equipment needed:

a. Distilled water.

b. Glass thermometer: to measure ambient temperature ±0.1°F (±0.1°C).

c. Toploading balance: precision of 0.01 g (see Figure 17).

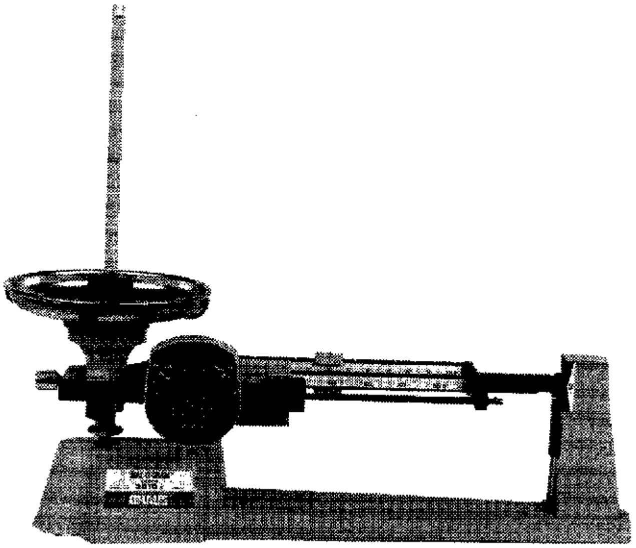


Figure 17—Weighing the Receiver

6.5.3 Procedure—Retort Cup:

6.5.3.1 Allow retort cup, lid and distilled water to reach ambient temperature. Record the temperature.

6.5.3.2 Place the clean, empty retort cup and lid on the balance and tare to zero.

6.5.3.3 Fill retort cup with distilled water. Place lid on the cup. Rotate lid to obtain proper fit. Be sure a small excess of water flows out of hole in lid. Wipe excess water from lid; avoid wicking out water.

6.5.3.4 Place filled retort cup with lid on the previously tared balance. Record weight of water, W_w, grams.

6.5.4 Calculation—Retort Cup:

Calculate retort cup volume, V_{RC}, using density of water at ambient temperature (see Table 4).

$$V_{RC} = \frac{W_w,\,\mathrm{g}}{Density\ of\ Water,\ \mathrm{g/cm}^3} \qquad (15)$$

6.5.5 Procedure — Liquid Receiver Volume:

6.5.5.1 Allow the receiver and distilled water to reach ambient temperature. Record the temperature.

6.5.5.2 Place the clean, empty receiver with its base on the balance and tare to zero.

6.5.5.3 While the receiver is on the balance, fill it with distilled water to the various graduations marks: 2, 4, 6, 8, 10 cm³, and 4, 8, 12, 16, 20 cm³ for the 10-cm³ and 20-cm³ receivers, respectively. Using a pipette or syringe, carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

Table 4—Density of Water

°F	°C	Density, g/cm³
59.0	15.0	0.9991
59.9	15.5	0.9991
60.8	16.0	0.9990
61.7	16.5	0.9989
62.6	17.0	0.9988
63.5	17.5	0.9987
64.4	18.0	0.9986
65.3	18.5	0.9985
66.2	19.0	0.9984
67.1	19.5	0.9983
68.0	20.0	0.9982
68.9	20.5	0.9981
69.8	21.0	0.9980
70.7	21.5	0.9979
71.6	22.0	0.9977
72.5	22.5	0.9976
73.4	23.0	0.9975
74.3	23.5	0.9974
75.2	24.0	0.9973
76.1	24.5	0.9971
77.0	25.0	0.9970
77.9	25.5	0.9969
78.8	26.0	0.9968
79.7	26.5	0.9966
80.6	27.0	0.9965
81.5	27.5	0.9964
82.4	28.0	0.9962
83.3	28.5	0.9961
84.2	29.0	0.9959
85.1	29.5	0.9958
86.0	30.0	0.9956
86.9	30.5	0.9955
87.8	31.0	0.9953
88.7	31.5	0.9952
89.6	32.0	0.9950
90.5	32.5	0.9949
91.4	33.0	0.9947
92.3	33.5	0.9945
93.2	34.0	0.9944
94.1	34.5	0.9942
95.0	35.0	0.9940

6.5.5.4 Record the weights for each incremental volume, IV, of water at the specific graduation mark, W_{IV}, grams.

6.5.6 Calculation for liquid receiver:

Calculate volume of the receiver at each mark, V_{MARK}, using density of water from Table 4, based on the measured ambient temperature.

$$V_{MARK} = \frac{W_{IV}, \text{g}}{\textit{Density of Water}, \text{g/cm}^3} \qquad (16)$$

7 Chemical Analysis of Oil Muds

7.1 DESCRIPTION

7.1.1 The whole mud alkalinity test procedure is a titration method which measures the volume of standard acid required to react with the alkaline (basic) materials in an oil mud sample. The alkalinity value is used to calculate the pounds per barrel unreacted "excess" lime in an oil mud. Excess alkaline materials, such as lime, help to stabilize the emulsion and also neutralize carbon dioxide or hydrogen sulfide acidic gases.

7.1.2 The whole mud chloride test procedure is a titration method which measures the volume of standard silver nitrate required to react with the chloride (and other halide) ions to form insoluble silver chloride (or silver halide) salts. The test procedures can utilize the same sample as the alkalinity test provided the sample is acidic (pH below 7.0). The chloride value reported in the whole mud can be assigned to the water phase up to the saturation point. The water soluble salt concentration is related to the effectiveness of an oil mud in controlling shale through the "aqueous phase activity" concept. The water phase salinity value is also needed to adjust the retort water value to obtain a corrected solids content for the oil mud.

7.1.3 The whole mud calcium test is a titration method which measures the volume of a standard calcium-chelating agent (EDTA, Versenate, or equivalent) required to react with all the calcium (and other water soluble alkaline earth ions) released from the oil mud into the aqueous phase when a mixture of solvents is used to extract the oil mud. The calcium measured in this test can come from the calcium chloride, $CaCl_2$, and lime CaO or $Ca(OH)_2$, used to prepare the oil muds, but some calcium could come from drilled-up gypsum or anhydrite ($CaSO_4$). The calcium analysis results can be used, along with the chloride and water content tests, to calculate the $CaCl_2$ and $NaCl$ salinity of the mud's aqueous phase.

Note: This calcium analysis excludes Magnesium (Mg^{+2}) ion. No such API procedure now exists. If Mg^{+2} is expected, special calcium and magnesium ion titration and calculation procedures should be arranged by the mud engineer and/or operator.

7.1.4 Section 9, "Lime, Salinity and Solids Calculations," gives procedures for the calculations based on these chemical analyses.

7.2 EQUIPMENT

Following is a list of equipment needed:

a. Solvent: Propylene glycol normal propyl ether (PNP) (CAS #1569-01-3).

b. Titration vessel: pint jar or 400-cm³ beaker.

c. Phenolphthalein indicator solution: (CAS #77-09-8) 1 g/100 cm³ of 50% isopropanol/water solution.

d. Sulfuric acid solution (CAS #7664-93-9): standardization 0.1 normal (N/10).

e. Potassium chromate indicator solution (CAS #7789-00-6): 5 g/100 cm³ of water.

f. Silver nitrate reagent (CAS #7761-88-8): containing 47.91 g/L (equivalent to 0.01 g chloride-ion/cm³) (0.282 N), stored in an amber or opaque bottle.

g. Distilled or deionized water.

h. Disposable syringe: two 5-cm³.

i. Graduated cylinder (TC): one 25-cm³.

j. Serological (graduated) pipettes (TD): two 1-cm³ and two 10-cm³.

Note: One pair of pipettes for sulfuric acid and one pair for silver nitrate.

k. Magnetic stirrer with 1.5-inch (38-mm) stirring bar (coated).

l. Calcium buffer solution: 1N sodium hydroxide (NaOH) (CAS #1310-73-2) prepared from fresh "Certified ACS" grade NaOH, which contains less than 1% by weight sodium carbonate.

Note: Calcium buffer solution should be kept in a closed, sealed bottle to minimize absorption of CO_2 from air.

m. Calcium indicator: Calver II or hydroxy naphthol blue (CAS #63451-35-4).

n. EDTA solution (Versenate, or equivalent) (CAS #139-33-3): 0.1 molar, EDTA: disodium (ethylenediamine) tetraacetate dihydrate, standardized (1 cm³ = 10,000 mg/L $CaCO_3$, 1 cm³ = 4000 mg/L calcium).

Note: This EDTA solution is ten times more concentrated than that used in water-based fluid testing.

7.3 PROCEDURE—WHOLE MUD ALKALINITY

7.3.1 Add 100 cm³ of PNP solvent to a 400-cm³ beaker or pint jar.

7.3.2 Fill a 5-cm³ syringe with whole mud to past the 3 cm³ mark.

7.3.3 Displace 2.0 cm³ of whole mud into the beaker or pint jar.

7.3.4 Swirl the oil mud and solvent until the mixture is homogenous.

7.3.5 Add 200 cm³ of distilled (or deionized) water.

7.3.6 Add 15 drops of phenolphthalein indicator solution.

7.3.7 While stirring rapidly with a magnetic stirrer, slowly titrate with 0.1 N sulfuric acid until pink color just disappears. Continue stirring and if no pink color reappears within one minute, stop stirring.

Note: It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to more clearly see the color in the aqueous phase.

7.3.8 Let sample stand for five minutes. If no pink color reappears, the end point has been reached. If a pink color returns titrate a second time with sulfuric acid. If pink color returns, titrate a third time. If after the third titration, pink color returns, call this the end point.

7.3.9 Use the cm³ of 0.1 N sulfuric acid solution required to reach the end point (see 7.3.8) to calculate the whole mud alkalinity, V_{SA}.

7.4 CALCULATION — WHOLE MUD ALKALINITY

$$\begin{aligned} Alkalinity\ (whole\ mud),\ V_{SA} &= \frac{0.1\ N\ sulfuric\ acid,\ \text{cm}^3}{mud\ sample,\ \text{cm}^3} \\ &= \frac{0.1\ N\ sulfuric\ acid,\ \text{cm}^3}{2.0\ \text{cm}^3} \end{aligned} \quad (17)$$

7.5 PROCEDURE—WHOLE MUD CHLORIDE

7.5.1 Perform the alkalinity procedures in 7.3.1 through 7.3.8.

Note: Make sure the mixture to be titrated for chloride is acidic (below pH 7.0) by adding 10-20 drops, or more, of 0.1 N sulfuric acid.

7.5.2 Add 10–15 drops potassium chromate indicator solution.

7.5.3 While stirring rapidly with a magnetic stirrer, slowly titrate with 0.282 N silver nitrate reagent until a salmon pink color remains stable for at least one minute.

Note 1: Additional potassium chromate indicator solution may be required during titration process.

Note 2: It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to more clearly see the color in the aqueous phase.

7.5.4 Use the cm³ of 0.282 N silver nitrate reagent required to reach the end point (see 7.5.3) to calculate the whole mud chloride.

7.6 CALCULATION—WHOLE MUD CHLORIDE

$$\begin{aligned} Chloride\ (whole\ mud),\ Cl_{OM} &= \frac{Cl,\ mg}{oil\ mud\ sample,\ L} \\ &= \frac{10{,}000\ (0.282\ N\ silver\ nitrate,\ \text{cm}^3)}{2.0\ \text{cm}^3} \end{aligned} \quad (18)$$

7.7 PROCEDURE—WHOLE MUD CALCIUM

7.7.1 Add 100 cm³ of 50/50 xylene/isopropanol solvent to a pint jar.

7.7.2 Fill a new 5-cm³ syringe with whole oil mud past the 3 cm³ mark.

7.7.3 Displace 2.0 cm³ of oil mud into the pint jar.

7.7.4 Cap the jar tightly and shake vigorously by hand for 1 minute.

7.7.5 Add 200 cm³ distilled or deionized water to the jar.

7.7.6 Add 3.0 cm³ 1 N sodium hydroxide buffer solution.

7.7.7 Add 0.1–0.25 g Calver II indicator powder.

7.7.8 Recap the jar tightly, shake vigorously again for 2 minutes. Set jar aside for a few seconds to allow upper and lower phases to separate. If reddish color appears in aqueous (lower) phase, this indicates that calcium is present.

7.7.9 Place jar on magnetic stirrer and drop in a stir bar.

7.7.10 Titrate very slowly, adding EDTA (Versenate, or equivalent) solution drop by drop from the pipette, while stirring only fast enough to agitate the aqueous (lower) phase without remixing the upper and lower phases. A distinct color change from the reddish color to a blue-green occurs at the end point. Note the volume of EDTA added.

7.7.11 Use the cm³ of EDTA (Versenate, or equivalent) required to reach the end point in 7.7.10 to calculate the whole mud calcium.

7.8 CALCULATION—WHOLE MUD CALCIUM

Calcium (whole mud),

$$Ca_{OM'} = \frac{Ca,\ mg}{oil\ mud\ sample,\ L} = \frac{4000(0.1\ M\ EDTA, \mathrm{cm}^3)}{2.0\ \mathrm{cm}^3} \quad (19)$$

8 Electrical Stability Test

8.1 DESCRIPTION

8.1.1 The electrical stability (ES) of an oil-based drilling fluid mud is a property related to its emulsion stability and oil-wetting capability. ES is determined by applying a voltage-ramped, sinusoidal electrical signal across a pair of parallel flat-plate electrodes immersed in the mud. The resulting current remains low until a threshold voltage is reached, whereupon the current rises very rapidly. This threshold voltage is referred to as the ES of the mud and is defined as the voltage (in peak volts-measured when the current reaches 61 µA).

Note: The sine wave specified for the voltage ramp results in more efficient energizing of the fluid and generates considerably lower ES values (often less than half) than the spiky waveform of the instrument described in previous editions of RP 13B-2. The symmetry of the sinusoidal signal also inhibits buildup of solids on the electrode faces and enhances reproducibility. To further enhance reproducibility, the instrument specifications include automatic voltage ramping at a fixed ramp rate.

8.1.2 Chemical composition and shear history of a mud control the absolute magnitude of ES in a complex fashion. Consequently, interpreting the oil-wet state of a mud from single ES measurements is not appropriate. *Only trends* in ES should be used in making treatment decisions.

8.2 EQUIPMENT

Following is a list of equipment needed:

a. Electrical Stability Meter (see Figures 18 and 19):

Specifications for the electrical stability meter are the following:

Waveform	Sine, <5% total harmonic distortion.
AC Frequency	340 ±10 Hz.
Output Units	Peak volts.
Ramp Rate	150 ±10 V/sec, automatic operation.
Minimum Output Range	3–2000 V (peak).
Trip Current	61 ±5 µA (peak).

b. Electrodes:

Specifications for electrodes are the following:

Housing	Material resistant to oil mud components up to 220°F (105°C).
Material	Corrosion-resistant metal.
Diameter	0.125 ±0.001 in. (3.18 ±0.03 mm).
Spacing (gap)	0.061 ±0.001 in. (1.55 ±0.03 mm) at 72°F (22°C).

c. Calibration Resistors/Diodes:

Specifications for calibration resistors/diodes are the following:

Number	Two (Low and High).
Types	Standard resistor or Zener diodes.
Range	Give Voltage (ES) reading of (1) Low 500–1000 V (2) High >1900 V.
Accuracy	±2% of expected voltage, temperature corrected by manufacturer's table (if needed and provided).

Note: The standard resistors are used to check the ratio of readout voltage to trip current. The Zener diodes check the readout voltage directly. Optimum confidence in the performance of the ES meter is obtained from tests with both resistors and diodes.

d. Thermometer: 32-220°F (0-105°C), with 1% accuracy.

e. Screen: 12-mesh, or Marsh funnel (see Section 4).

f. Thermostatically-controlled viscometer cup (see Section 4).

8.3 PROCEDURE—EQUIPMENT CALIBRATION/ PERFORMANCE TEST

8.3.1 Inspect the electrode probe and cable for evidence of damage.

8.3.2 Ensure that the entire electrode gap is free of deposits and the connector to the instrument is clean and dry.

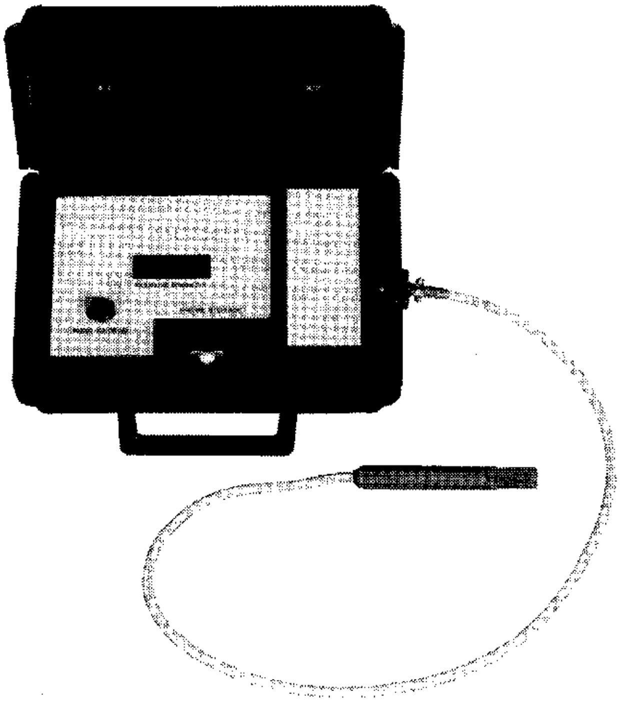


Figure 18—Electrical Stability Meter

Figure 19—Electrical Stability Meter

8.3.3 Disconnect the electrode probe (if possible) and run a voltage ramp test, following instructions in the ES Meter operating manual. If the meter is working properly, the ES reading should reach the maximum voltage permitted by the instrument.

8.3.4 Reconnect the electrode probe to the ES Meter and repeat the voltage ramp in air. Again, the ES reading should reach the maximum permitted voltage; if it does not, the electrode probe and connector may need to be recleaned or replaced.

8.3.5 Repeat the voltage ramp test with the electrode probe in tap water. The ES reading should not exceed 3 volts. If ES does exceed 3 volts, reclean the electrode probe or replace it.

8.3.6 Check the accuracy of the ES meter with the standard resistors and/or Zener diodes (see note under 8.2.c). The ES readings should fall within 2.5% (combined uncertainty of meter and resistor/diodes) of the expected values. If any of the ES readings fall outside this range, the instrument should be returned to the supplier for adjustment or repair.

Note: These procedures (see 8.3) do not check waveform, AC frequency, or ramp rate. Therefore, have the ES meter and electrode probe checked by a qualified technician periodically to ensure that both units meet all specifications.

8.4 PROCEDURE—ELECTRICAL STABILITY MEASUREMENTS

8.4.1 Verify daily equipment calibration/performance (see 8.3).

8.4.2 Place the oil mud sample, which has been screened through a 12-mesh screen or a Marsh funnel, in a viscometer cup maintained at 120 ±5°F (50 ±2°C). Record the mud temperature on the Drilling Mud Report Form.

8.4.3 Clean the electrode probe body thoroughly by wiping with a clean paper towel. Pass the towel through the electrode gap a few times. Swirl the electrode probe in the base oil used to formulate the mud. If the base oil is not available, another oil or a mild solvent (such as isopropanol) is acceptable. Clean and dry the electrode probe as before.

Note: Do not use detergent solutions or aromatic solvents, such as xylene, to clean the electrode probe or cable.

8.4.4 Hand-stir the 120°F sample with the electrode probe for approximately 10 sec to ensure that the composition and temperature of the mud are uniform. Position the electrode probe so that it does not touch the bottom or sides of the container, and be sure that the electrode surfaces are completely covered by the sample.

8.4.5 Begin the voltage ramp. Follow the procedure described in the ES meter operating manual. Do not move the electrode probe during the voltage ramp.

8.4.6 At the conclusion of the ramp test, note the ES value displayed on the readout device.

8.4.7 Repeat 8.4.3 through 8.4.6 with the same mud sample. The two ES values should not differ by more than 5%. If they differ by more than 5%, check the meter or electrode probe for malfunction.

8.4.8 Record the average of the two ES measurements on the API Drilling Mud Report Form.

9 Lime, Salinity, and Solids Calculations

9.1 DESCRIPTION

9.1.1 Equations in this procedure allow calculations of the following components for an oil-based mud:

a. Lime content, as $Ca(OH)_2$ in the whole mud.
b. $CaCl_2$ and/or NaCl in the whole mud.
c. $CaCl_2$ and/or NaCl in the water-phase.
d. Low-density solids (NaCl salt and/or drill solids).
e. High-density solids.

9.1.2 Data used in the equations are from oil-based mud tests specified in Sections 3, 6 and 7 as follows:

a. Mud weight, *MW* (see 3.1.3 or 3.2.3).
b. Whole mud alkalinity, V_{SA} (see 7.3).
c. Whole mud calcium, Ca_{OM}, (see 7.7).
d. Whole mud chloride, Cl_{OM}, (see 7.5).
e. Retort oil, V_O, and water, V_w, (see 6.3).

9.1.3 Data known or assumed are as follows:

a. Density of weighting material, ρ_{HDS}.
b. Density of drilled solids, ρ_{LDS}.
c. Density of base oil, ρ_O.

9.1.4 These calculations do *not* utilize a "lime correction" for adjusting the whole mud calcium content as determined by the Versenate titration.

9.1.5 Be sure to use units requested in the equations. Also, carefully note the units asked for on the API Oil Mud Report Form (Recommended Practice 13G). Units for all input data and results for each equation are indicated and are either SI, metric, or common oilfield units.

Note: Results required on the API Oil Mud Report Form are shown enclosed in a "box" in this section.

9.1.6 Example calculations can be found in Appendix E.

9.2 EQUIPMENT

It is recommended that a hand-held calculator be used to quickly and correctly perform the mathematical operations detailed in this procedure.

9.3 WHOLE MUD CALCULATIONS

9.3.1 Whole Mud Lime and Calcium Content

The total lime content $[Ca(OH)_2]$ is:

$$\text{Lime}_{OM}, \text{lb/bbl} = 1.295(V_{SA}) \tag{20}$$

Note: Lime_{OM} is often referred to as "whole mud alkalinity" or as "excess lime."

Where:
Lime_{OM} = $Ca(OH)_2$, lb/oil mud, bbl
V_{SA} = cm^3 of 0.1 N sulfuric acid/cm^3 of mud

The total calcium content is:

$$Ca_{OM} = 4000(V_{EDTA}) \tag{21}$$

Where:
Ca_{OM} = mg Ca^{++}/L of mud
V_{EDTA} = cm^3 0.1 Molar EDTA/cm^3 of mud

9.3.2 Whole Mud Total Chloride Content

The total chloride content is:

$$Cl_{OM} = 10{,}000\ (V_{SN}) \tag{22}$$

Where:
Cl_{OM} = mg Cl^-/L of mud
V_{SN} = cm^3 0.282 N silver nitrate/cm^3 of mud

9.3.3 Whole Mud Calcium Chloride and Sodium Chloride Content

9.3.3.1 An oil-based mud will probably contain at least one and perhaps both calcium chloride ($CaCl_2$), and sodium chloride (NaCl). Use the Ca_{OM} and Cl_{OM} titration data and proceed below to determine if both NaCl and $CaCl_2$ are present.

9.3.3.2 The calcium chloride content is:

$$CaCl_{2_{OM}} = 2.774\ (Ca_{OM}) \tag{23}$$

Where:
Ca_{OM} = mg $CaCl_2$/L of mud

9.3.3.3 The pounds per barrel of calcium chloride is:

$$CaCl_2, \text{lb/bbl} = 0.000971\ (Ca_{OM}) \tag{24}$$

9.3.3.4 The chloride ion associated with $CaCl_2$ based on the Ca_{OM} analysis is:

$$Cl_{CaCl_2} = 1.77\ (Ca_{OM}) \tag{25}$$

Where:
Cl_{CaCl_2} = mg Cl/L of mud from $CaCl_2$

9.3.3.5 Check to see which numerical value is *larger:*

a. If Cl_{CaCl_2} is larger than or equal to CL_{OM}, then assume that *only $CaCl_2$ is present in the mud* and *no NaCl* is present. Proceed to 9.3.3.6, Equation 26.
b. If Cl_{OM} is larger than Cl_{CaCl_2}, then assume that *both $CaCl_2$ and NaCl exist in the mud.* Proceed to 9.3.3.8.

9.3.3.6 The calcium chloride content is:

$$CaCl_{2_{OM}} = 1.57\ (Cl_{OM}) \tag{26}$$

Where:

$CaCl_{2_{OM}}$ = mg $CaCl_2$/L of mud

Note: Use this result in place of the value calculated in Equation 23 when no NaCl is present.

9.3.3.7 The pounds per barrel of $CaCl_2$ is:

$$CaCl_2,\ lb/bbl = 0.00035\ (CaCl_{2_{OM}}) \qquad (27)$$

Note: Use this result in place of the value calculated in Equation 24 when no NaCl is present.

At this point proceed directly to 9.4.

9.3.3.8 The $CaCl_2$ content has already been determined in Equations 23 and 24. The amount of chloride ion associated with NaCl is:

$$Cl_{NaCl} = Cl_{OM} - Cl_{CaCl_2} \qquad (28)$$

Where:

Cl_{NaCl} = mg Cl/L of mud from NaCl

9.3.3.9 The NaCl content is:

$$NaCl_{OM} = 1.65\ (Cl_{NaCl}) \qquad (29)$$

Where:

$NaCl_{OM}$ = mg NaCl/L of mud

9.3.3.10 The pounds per barrel of NaCl is:

$$NaCl,\ lb/bbl = 0.00035\ (NaCl_{OM}) \qquad (30)$$

9.4 AQUEOUS PHASE SALT CONTENT CALCULATIONS

The calculations above have involved only the whole mud salt content. In order to determine salt concentrations in the aqueous phase of the mud, the titration data in the following calculations must be combined with the retort data described in 6.3.

9.4.1 The following Equations 31 and 32 calculate the amount of NaCl and $CaCl_2$ in the aqueous phase of an oil-based mud. Weight percent $CaCl_2$ is:

$$W_c = \frac{100\ (CaCl_{2_{OM}})}{CaCl_{2_{OM}} + NaCl_{OM} + (V_w)} \qquad (31)$$

Where:

W_c = Wt% $CaCl_2$ in the brine

V_W = Volume percent of retorted water

The weight percent of NaCl is:

$$W_N = \frac{100\ (NaCl_{OM})}{CaCl_{2_{OM}} + NaCl_{OM} + (V_w)} \qquad (32)$$

Where:

W_N = Wt % NaCl in the brine

9.4.2 Check Figure 20 to determine if the weight percent of sodium chloride, W_N, is outside the limit of mutual solubility for NaCl/$CaCl_2$ brine solutions at 25°C. If it is, *undissolved NaCl* is present in the oil-based mud. The aqueous phase salinities as calculated in Equations 31 and 32 are incorrect because all the NaCl is not actually in solution.

To arrive at the correct $NaCl_{OM}$, it must be systematically lowered by using a "Fraction" method involving Equations 31 and 32 until the calculated W_N and W_C fall within the range shown on Figure 20. The following steps in 9.4.2.1 to 9.4.2.6 perform an iteration solution approximating the correct value of $NaCl_{OM}$ and W_N and W_C. Example D in Appendix E gives an example of how to do this calculation.

9.4.2.1 Calculate the "Fraction" below. It will be less than 1.00 even when there is a large amount of insoluble NaCl present in the mud. No more than three "trial" calculations are required.

$$\text{Fraction} = \frac{\text{NaCl, wt \% [Figure 20]}}{W_N\ \text{[Equation 31]}}$$

9.4.2.2 Multiply the $NaCl_{OM}$ values used in Equation 30 and 31 by the "Fraction" to obtain a new trial value for $NaCl_{OM}$.

$$\text{New } NaCl_{OM} = \text{Fraction}\ (W_N)$$

9.4.2.3 Use the new $NaCl_{OM}$ in the denominator of Equation 30 to calculate a new W_C and using the new $NaCl_{OM}$ in the denominator and numerator of Equation 31, calculate a new W_N.

9.4.2.4 Read a new value of $NaCl_{OM}$ from Figure 20 using the new W_C from 9.4.2.3 on the graph.

9.4.2.5 Calculate a second new "Fraction" using $NaCl_{OM}$ found from Figure 20 from 9.4.2.4 (above) and W_N calculated from 9.4.2.3 (above).

9.4.2.6 Calculate second new W_C and W_N, as 9.4.2.3. Record these values when the Fraction becomes 0.95 or larger.

Note 1: When the "Fraction" becomes 0.95 or larger no more calculations of new values for $NaCl_{OM}$ and W_C and W_N are needed. This usually requires no more than three cycles of this calculation.

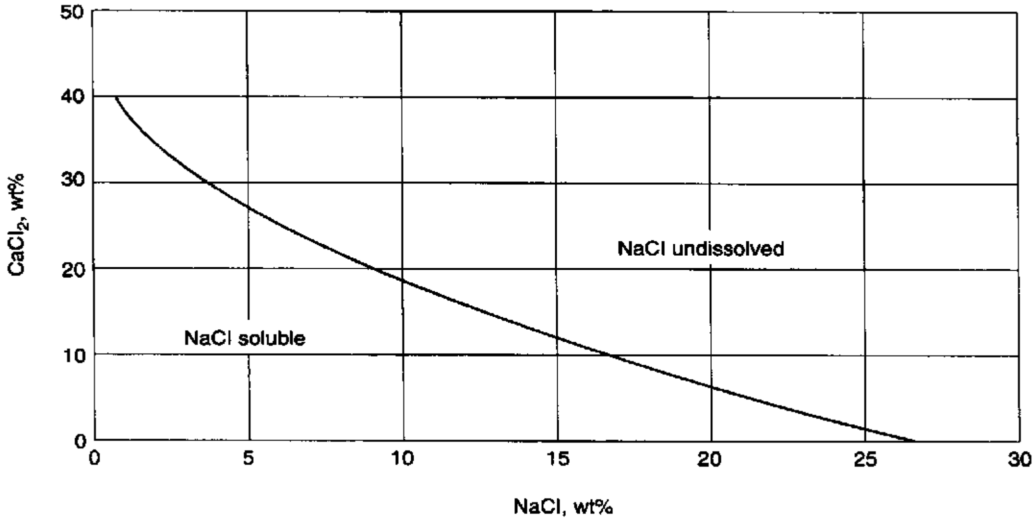
Note 2: Undissolved sodium chloride salt has a lower specific gravity than most drill solids. If it is present in the oil mud, it will make the solids calculations in error. No technique is provided in this procedure to compensate for that type of error.

9.4.3 The $CaCl_2$ concentration in parts per million is:

$$CaCl_2,\ ppm = 10{,}000\ (W_c) \qquad (33)$$

9.4.4 The NaCl concentration in parts per million is:

$$NaCl,\ ppm = 10{,}000\ (W_N) \qquad (34)$$



Max $W_N = 26.432 - 1.0472\,(W_C) + 7.98191 \times 10^{-3}\,(W_C)^2 + 5.2238 \times 10^{-5}\,(W_C)^3$

Figure 20—Maximum Concentrations of NaCl in $CaCl_2$ Brine at 25°C

9.4.5 The density of the brine is:

$$\rho_B = 0.99707 + 0.006504\,(W_N) + 0.007923\,(W_C) + 0.00008334\,(W_N)\,(W_C) + 0.00004395\,(W_N)^2 + 0.00004964\,(W_C)^2 \qquad (35)$$

Where:

ρ_B = Brine density, g/cm^3

Note: Densities of single salt solutions are found in Table 5 for $CaCl_2$ and Table 6 for NaCl.

9.4.6 $CaCl_2$ concentration in mg/L is:

$$CaCl_2\text{, mg/L} = 10{,}000\,(W_C)\,(\rho_B) \qquad (36)$$

9.4.7 NaCl concentration in mg/L is:

$$\text{NaCl, mg/L} = 10{,}000\,(W_N)\,(\rho_B) \qquad (37)$$

9.5 SOLIDS CALCULATIONS

9.5.1 Corrected Solids Content

Determine the corrected solids content in the mud as follows:

Note: The water content must be adjusted for the volume of dissolved salt(s) in the aqueous phase of the mud.

a. The volume percent of brine is:

$$V_B = \frac{100\,(V_W)}{(\rho_B)[100 - (W_N + W_C)]} \qquad (38)$$

Where:

V_B = Volume % of brine

b. The volume percent of corrected solids is:

$$V_S = 100 - (V_O + V_B) \qquad (39)$$

Where:

V_S = Volume % corrected solids

V_O = Volume % of retorted oil

9.5.2 Oil/Water Ratio Calculations

The oil/water ratio (OWR) is calculated as follows:

$$O_R = \frac{100\,(V_O)}{(V_O + V_W)} \qquad (40)$$

Where:

O_R = Oil % in the ratio

$$W_R = \frac{100\,(V_W)}{(V_O + V_W)} \qquad (41)$$

Where:

W_R = Water % in the ratio

$$OWR = O_R/W_R \qquad (42)$$

Note: *OWR* is written as a ratio.

9.5.3 Oil/Brine Ratio Calculations

The oil/brine ratio (OBR) is calculated as follows:

$$O_R = \frac{100\,(V_O)}{(V_O + V_B)} \qquad (43)$$

as before:

O_R = Oil % in the ratio

$$B_R = \frac{100\,(V_B)}{(V_O + V_B)} \qquad (44)$$

Table 5—Calcium Chloride Brine Table (see note)
(Properties at 20°C—Except Water Activity at 25°C)

W_C Lot %	$CaCl_2$ mg/L	Chloride mg/L	Final Volume	Water[a] Activity	Density g/cm³	Density lb/gal
0	0	0	1.000	1.000	0.998	8.330
2	20,330	12,990	1.004	0.996	1.0166	8.484
4	41,340	26,410	1.008	0.989	1.0334	8.624
6	63,030	40,280	1.013	0.979	1.0505	8.766
8	85,420	54,590	1.018	0.967	1.0678	8.911
10	108,540	69,360	1.024	0.951	1.0854	9.058
12	132,400	84,600	1.030	0.933	1.1033	9.207
14	157,050	100,360	1.037	0.912	1.1218	9.361
16	182,510	116,630	1.044	0.888	1.1407	9.519
18	208,780	133,410	1.051	0.862	1.1599	9.679
20	235,920	150,750	1.060	0.832	1.1796	9.844
22	263,930	168,650	1.069	0.800	1.1997	10.011
24	292,820	187,100	1.078	0.765	1.2201	10.182
26	322,660	206,170	1.089	0.727	1.2410	10.356
28	353,420	225,820	1.100	0.686	1.2622	10.533
30	385,140	246,090	1.113	0.643	1.2838	10.713
32	417,890	267,020	1.126	0.597	1.3059	10.898
34	451,620	288,570	1.141	0.548	1.3283	11.047
36	486,430	310,780	1.156	0.496	1.3512	11.276
38	522,310	333,740	1.173	0.441	1.3745	11.470
40	559,280	357,370	1.192	0.384	1.3982	11.668

Note: The calcium ion concentration can be determined by subtracting the "Chloride" ion value from the "$CaCl_2$" value, for example:
Ca^{++} mg/L = $CaCl_2$, mg/L–Cl, mg/L.

[a] B. R. Staples and R. L. Nuttall[4]. See References.

Where:

B_R = Brine % in the ratio

$$OBR = O_R/B_R \tag{45}$$

Note: *OBR* is written as a ratio.

9.5.4 Solids Concentration and Densities

The solids in an oil-based mud consist of low density solids, such as drill solids, and high density solids, such as barite or hematite. Densities for materials commonly occurring in the oil-based mud are found in Table 7.

The average density of the suspended solids is:

$$\rho_S = \frac{[100\,(MW)] - [(V_O)(\rho_O)] - [(V_S)(\rho_S)(8.345)]}{8.345\,(V_S)} \tag{46}$$

Where:

ρ_S = Average solids density, g/cm³
ρ_O = Oil density, lb/gal
MW = Mud weight, lb/gal

9.5.5 Volume Percent and Concentrations of High- and Low-Density Solids

9.5.5.1 The volume percent high-density solids is:

$$V_{HDS} = \frac{\rho_S - \rho_{LDS}}{\rho_{HDS} - \rho_{LDS}}\,(V_S) \tag{47}$$

Where:

V_{HDS} = Volume percent high-density solids
ρ_{HDS} = Density high-density solids, g/cm³
ρ_{LDS} = Density low-density solids, g/cm³

9.5.5.2 The volume percent low-density solids is:

$$V_{LDS} = V_S - V_{HDS} \tag{48}$$

Where:

V_{LDS} = Volume percent low-density solids

9.5.5.3 The concentration of high-density solids is:

$$M_{HDS} = 3.5\,(\rho_{HDS})\,(V_{HDS}) \tag{49}$$

Where:

M_{HDS} = High-density solids, lb/bbl

Table 6—Sodium Chloride Brine Table (see note)
Table (Properties at 20°C — Except Water Activity at 25°C)

W_C Lot %	$NaCl_2$ mg/L	Chloride mg/L	Final Volume	Water[a] Activity	Density g/cm³	Density lb/gal
0	0	0	1.000	1.000	0.998	8.330
2	20,250	12,310	1.006	0.989	1.0143	8.464
4	41,140	24,960	1.013	0.976	1.0286	8.584
6	62,590	37,980	1.020	0.964	1.0431	8.705
8	84,620	51,350	1.028	0.950	1.0578	8.827
10	107,260	60,680	1.036	0.935	1.0726	8.951
12	130,510	79,200	1.045	0.919	1.0876	9.076
14	154,390	93,680	1.054	0.901	1.1028	9.203
16	178,910	108,570	1.065	0.882	1.1182	9.331
18	204,100	123,840	1.076	0.861	1.1339	9.462
20	229,960	139,540	1.087	0.839	1.1498	9.595
22	265,520	155,660	1.099	0.815	1.1660	9.730
24	283,800	172,210	1.113	0.788	1.1825	9.868
26	311,820	189,210	1.127	0.759	1.1993	10.008

Note: The sodium ion concentration can be determined by subtracting the "Chloride" ion value from the "NaCl" value, for example:
Na^+, mg/L = NaCl, mg/L–Cl, mg/L.

[a] E. C. W. Clarke and D. N. Grew[3]. See References.

Table 7—Commonly Used Densities

Mud Component	Density, g/cm³	Density, lb/gal	Density, kg/m³
Fresh Water	1.00	8.345	1000
Diesel Oil, Typical	0.84	7.00	840
Low Viscosity Oil, Typical	0.80	6.70	800
Low Density Solids	2.65	22.10	2650
High Density Solids			
Barite	4.20	35.00	4200
Hematite	5.05	42.10	5050

9.5.5.4 The concentration of low-density solids is:

$$M_{LDS} = 3.5\,(\rho_{LDS})\,(V_{LDS}) \qquad (50)$$

Where:

M_{LDS} = Low-density solids, lb/bbl

APPENDIX A—SHEAR STRENGTH MEASUREMENT USING SHEAROMETER TUBE

A.1 Description

A.1.1 Experience has shown that some drilling muds tend to develop excessive shear strength under static conditions, especially at elevated temperatures. Excessive shear strength results in high pump pressures to "break circulation," and may therefore result in loss of circulation. High shear strength may also cause difficulties in logging, perforating, and other "down hole" operations.

A.1.2 The following technique can be used to determine the tendency and estimate the extent to which the mud will develop excessive shear strength. This shear strength measurement is normally made on a static heat-aged mud sample. Aging temperatures are therefore selected to be near the estimated bottom-hole temperature of the well. Aging cells or vessels meeting the pressure and temperature requirements for the test are required.

A.2 Equipment

Following is a list of equipment needed:

a. Stainless steel shearometer tube. Specifications are the following:

Length	3.5 in. (89 mm)
Outside diameter	1.4 in. (36 mm)
Wall thickness	0.008 in. (0.2 mm)

Note: A slight outside taper on the bottom of the shear tube has been found to improve reproducibility of the test results.

b. Platform for weights.
c. Set of gram weights.
d. Ruler, in.

A.3 Procedure

A.3.1 The shear tube and platform are placed and balanced carefully on the surface of the aged sample cooled to room temperature. It may be necessary to shift the weights on the platform to assure the tube's initial penetration into the mud is vertical. If a crust develops on the heat aged sample, this crust should be gently broken before placing the shear tube in place for test.

A.3.2 Sufficient gram weights are placed carefully on the platform to start the downward movement of the shear tube. Unless too much weight is added, the tube will stop its downward travel at the point where the shear strength of the aged mud against the surface of the tube is sufficient to support the applied weight (see Figure A-1). It is desirable to submerge at least one half the length of the tube.

A.3.3 Record the total weight in grams which includes the platform and weights. Measure the portion of the tube submerged in the fluid in inches. The length of the tube submerged can be most accurately determined by measuring the length of the nonsubmerged portion while the tube is at its maximum penetration depth. A small ruler held at the mud surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

A.4 Calculation

$$\textit{Sheer strength (S)}, = \frac{3.61\,(Z + W)}{L} - 0.256\,A \qquad \text{(A-1)}$$

Where:

S = shear strength, lb/100 ft^2
Z = weight of shear tube, g
W = total shear weight, g (sum of platform and weights)
L = submerged length of shear tube, in.
A = mud weight, lb/gal

Note: From Watkins and Nelson (see Reference section).

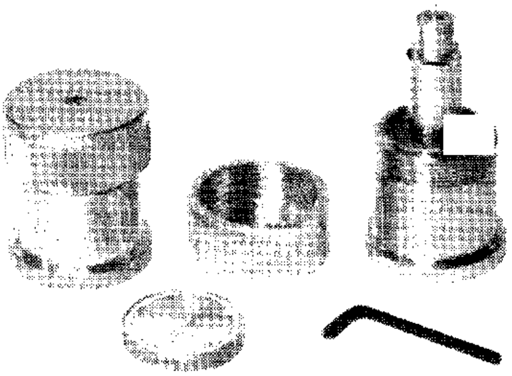


Figure A-1—Shear Strength Apparatus

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APPENDIX B—OIL AND WATER CONTENT FROM CUTTINGS FOR PERCENTAGE GREATER THAN 10%

B.1 Description

B.1.1 This procedure is specifically intended to measure the amount of oil from cuttings generated during a drilling operation. It is a retort test which measures all oil and water released from a cuttings sample when heated in a calibrated and properly operating "Retort" instrument.

B.1.2 In this retort test a known *weight* of cuttings is heated in the retort chamber to vaporize the liquids associated with the sample. The oil and water vapors are then condensed, collected and measured in a precision graduated receiver.

Note: Obtaining a representative sample requires special attention to the details of sample handling (location, method, frequency). The sampling procedure in a given area may be specified by local or governmental rules.

B.2 Equipment

Following is a list of Equipment needed:

a. Retort instrument—The recommended retort instrument has a 50-cm³ volume with an external heating jacket (see Figure B-1). Retort specifications are the following:

1. Retort assembly—retort body, cup and lid:
   a) Material: 303 stainless steel or equivalent.
   b) Volume: Retort cup with lid.

Cup volume:	50-cm³
Precision:	±0.25-cm³

2. Condenser—capable of cooling the oil and water vapors below their liquification temperature.
3. Heating jacket—nominal 350 watts.
4. Temperature control — capable of limiting temperature of retort to 930 ±70°F (500 ±38°C).

b. Liquid receiver (10-cm³, 20-cm³ or 50-cm³)—the 10-cm³ and 20-cm³ receivers are specially designed cylindrical glassware with rounded bottom to facilitate cleaning and funnel-shaped top to catch falling drops (see Figure B-2).

1. Receiver specifications are the following:

Total volume:	0-cm³	20-cm³	50-cm³
Precision: (0 to 100%)	±0.05 cm³	±0.05 cm³	±0.5 cm³ nom.
Outside diameter:	10-mm	13-mm	—
Wall thickness:	1.5 ± 0.1mm	1.2 ± 0.1mm	—
Frequency of graduation marks (0 to 100%)	0.10 cm³	0.10 cm³	1.0 cm³
Calibration:	To contain "TC" @ 20°C		
Scale:	cm³	cm³	cm³

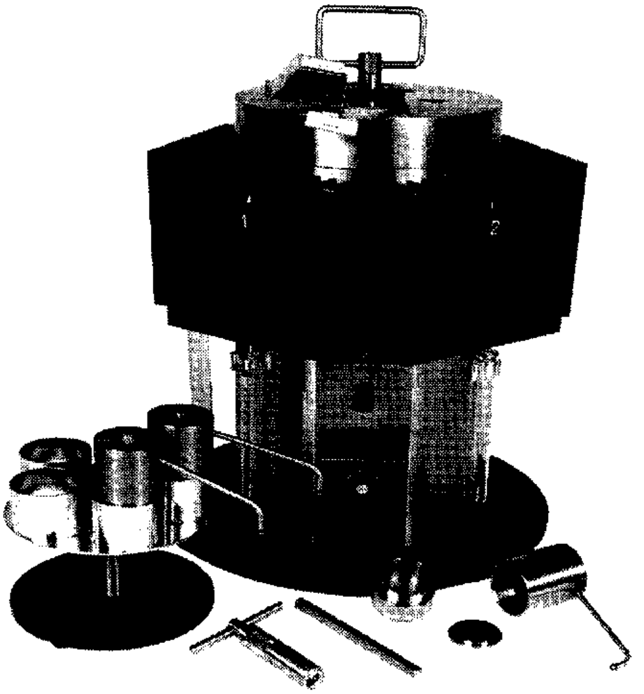

Figure B-1—Retort Instrument

Note: Verification of receiver volume. The receiver volume should be verified gravimetrically. The procedure and calculations are in B.5.

2. Material—Pyrex or equivalent glass.

c. Toploading balance—capable of weighing 2000 g and precision of 0.1 g.

d. Fine steel wool (No. 000)—for packing retort body.

e. Thread sealant lubricant: high temperature lubricant, e.g., Never-Seez or equivalent.

f. Pipe cleaners—to clean condenser and retort stem.

g. Brush—to clean receivers.

h. Retort spatula—to clean retort cup.

i. Corkscrew—to remove spent steel wool.

B.3 Procedure

B.3.1 Clean and dry the retort assembly and condenser.

B.3.2 Pack the retort body with steel wool.

B.3.3 Apply lubricant/sealant to threads of retort cup and retort stem.

B.3.4 Weigh and record the total mass of the retort cup, lid, and retort body with steel wool. This is mass, *A*, grams.

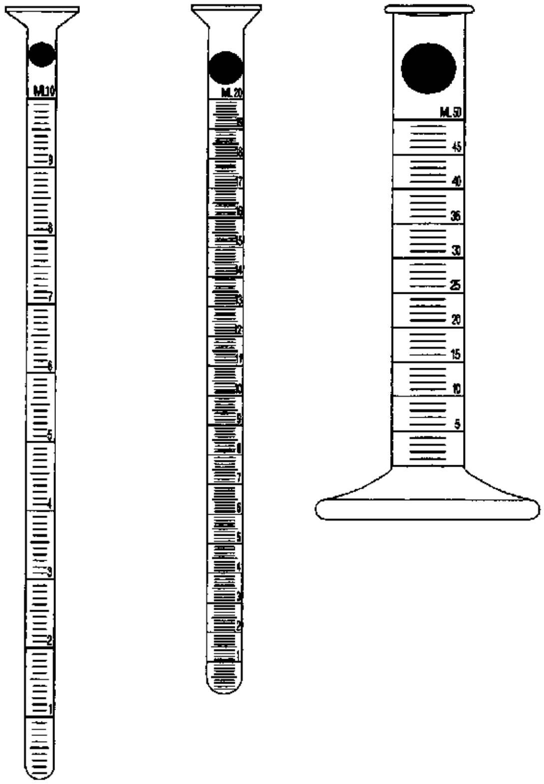

Figure B-2—Liquid Receivers

B.3.5 Collect a representative cuttings sample (see Note B.1).

B.3.6 Partially fill the retort cup with cuttings and place the lid on the cup.

B.3.7 Screw the retort cup (with lid) onto the retort body, weigh and record the total mass. This is mass, *B*, grams.

B.3.8 Attach the condenser. Place the retort assembly into the heating jacket.

B.3.9 Weigh and record the mass of the clean and dry liquid receiver. This is mass, *C*, grams. Place the receiver below condenser outlet.

B.3.10 Turn on the retort. Allow it to run a minimum of 1 hour.

Note: If solids boil over into receiver, the test must be rerun. Pack the retort body with a greater amount of steel wool (B.3.2.) and repeat the test.

B.3.11 Remove the liquid receiver. Allow it to cool. Record the volume of water recovered. This is *V*, cm^3.

Note: If an emulsion interface is present between the oil and water phases, heating the interface may break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. *Carefully* heat the receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquids. After the emulsion interface is broken, allow the liquid receiver to cool. Read the water volume at the lowest point of the menicus. (See Figure B-3)

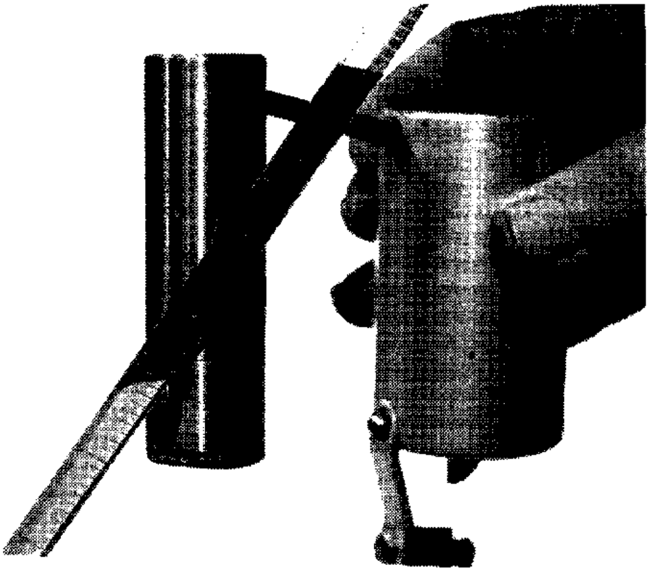

Figure B-3—Heating the Emulsion Band on Condenser

B.3.12 Weigh and record the mass of the receiver and its liquid contents (oil plus water). This is mass, *D*, grams.

B.3.13 Turn off the retort. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser.

B.3.14 Weigh and record the mass of the cooled retort assembly without the condenser. This is mass, *E*, grams.

B.3.15 Clean the retort assembly and condenser.

B.4 Calculations

B.4.1 Calculate the mass of oil from the cuttings as follows:

a. Mass of the wet cuttings sample M_W, equals the mass of the retort assembly retort assembly, *A*.

$$M_W = B - A \qquad \text{(B-1)}$$

b. Mass of the dry retorted cuttings, M_d, equals the mass of the cooled retort assembly, *E*, minus the mass of the empty retort assembly, *A*.

$$M_d = E - A \qquad \text{(B-2)}$$

c. Mass of the oil, M_O, equals the mass of the liquid receiver with its contents, *D*, minus the sum of the mass of the dry receiver, *C*, and the mass of the water (*V*).

$$M_O = D - (C + V) \qquad \text{(B-3)}$$

Note: Assuming the density of water is 1 g/cm^3, the volume of water is equivalent to the mass of the water.

B.4.2 Mass balance requirement:

The sum of M_d, M_o, and V should be within 5% of the mass of the wet sample.

$$\frac{M_d + M_o + V}{M_w} = 0.95 \text{ to } 1.05$$

The procedure should be repeated if this requirement is not met.

B.4.3 Reporting oil from cuttings:

Mass of oil, g, per kg of wet cuttings sample

$$= \frac{M_o}{M_w} \times 1000$$

Mass of oil (g) per kg of dry retorted cuttings

$$= \frac{M_o}{M_d} \times 1000$$

Note: When reporting oil from cuttings in terms of the dry (retorted) cuttings, the oil content may be higher than that calculated from the wet cuttings sample due to interstitial water extracted during retorting. Also, a cutting containing a higher percentage of water than another cutting containing a lesser amount of water, will appear to give a higher oil retention weight for the same reasons.

B.5 Verification of Liquid Receiver Volume

B.5.1 Description:

This procedure is used to verify that the liquid receiver meets specifications stated in B.2.b.

B.5.2 Equipment:

Following is a list of equipment needed.

a. Distilled water.
b. Glass thermometer—to measure ambient temperature ±0.1°F (±0.1°C).
c. Toploading balance—precision of 0.1 g.
d. Syringe or pipette—10-cm³ or larger.

B.5.3 Procedure:

B.5.3.1 Allow receiver and distilled water to reach ambient temperature. Record temperature.

B.5.3.2 Place the clean, empty receiver with its base on the balance and tare to zero.

B.5.3.3 While the receiver is on the balance, fill it to the various graduation marks (2, 4, 6, 8, 10-cm³ for the 10-cm³ receiver, 4, 8, 12, 16, 20-cm³ for the 20-cm³ receiver and 10, 20, 30, 40, 50-cm³ for the 50-cm³ receiver) with distilled water. Using a pipette or syringe, carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

B.5.3.4 Record weights for the incremental volumes, IV, of water at the specific graduation marks, W_{IV}, grams.

B.5.4 Calculation:

Calculate volume of the receiver at each mark, V_{MARK}, using density of water from Table B-1.

$$V_{MARK} = \frac{W_{IV}, \text{g}}{Density\ of\ Water, \text{g/cm}^3} \tag{B-4}$$

Table B-1—Density of Water

°F	°C	Density, g/cm³
59.0	15.0	0.9991
59.9	15.5	0.9991
60.8	16.0	0.9990
61.7	16.5	0.9989
62.6	17.0	0.9988
63.5	17.5	0.9987
64.4	18.0	0.9986
65.3	18.5	0.9985
66.2	19.0	0.9984
67.1	19.5	0.9983
68.0	20.0	0.9982
68.9	20.5	0.9981
69.8	21.0	0.9980
70.7	21.5	0.9979
71.6	22.0	0.9977
72.5	22.5	0.9976
73.4	23.0	0.9975
74.3	23.5	0.9974
75.2	24.0	0.9973
76.1	24.5	0.9971
77.0	25.0	0.9970
77.9	25.5	0.9969
78.8	26.0	0.9968
79.7	26.5	0.9966
80.6	27.0	0.9965
81.5	27.5	0.9964
82.4	28.0	0.9962
83.3	28.5	0.9961
84.2	29.0	0.9959
85.1	29.5	0.9958
86.0	30.0	0.9956
86.9	30.5	0.9955
87.8	31.0	0.9953
88.7	31.5	0.9952
89.6	32.0	0.9950
90.5	32.5	0.9949
91.4	33.0	0.9947
92.3	33.5	0.9945
93.2	34.0	0.9944
94.1	34.5	0.9942
95.0	35.0	0.9940

APPENDIX C—AQUEOUS-PHASE ACTIVITY BY ELECTROHYGROMETER

C.1 Description

C.1.1 This procedure describes the measurement of relative humidity in a closed air space above an oil emulsion drilling fluid using an electrohygrometer, and to relate humidity to the activity of the emulsified water. Activity is a measure of the chemical potential or reaction availability of a chemical species, in this case water. Hole conditions in water sensitive shales can be correlated with changes in the activity of emulsified water in oil-based drilling fluids.

C.1.2 The abundance of charged particles, surfaces, and ions in shales reduces the activity of water and increases a shale's tendency to absorb water. Water will migrate through a semi-permeable membrane from a region of lower salinity (higher activity) to one of higher salinity (lower activity). The oil films surrounding emulsified water droplets in an oil-based drilling fluid constitute semi-permeable membranes through which water may pass by osmosis.

C.1.3 Water transfer from an oil-based drilling fluid can be prevented by dissolving salt in the emulsified water. Salt lowers the activity of the water phase, thus reducing the tendency of water to be absorbed by shales. Water transfer to shale is eliminated when the activity of the emulsified water is reduced to a value equal to or lower than that of the exposed shales. Hole condition and cuttings texture can guide proper activity adjustment.

C.1.4 The most efficient commonly available salt for reducing the activity of water in oil-based drilling fluids is calcium chloride. Table C-1 gives calcium chloride solution activity versus percent concentration. Brine having a given activity will retain that activity when emulsified in oil. The aqueous-phase activity of an oil-based drilling fluid will be changed by water evaporation or water addition or by incorporation by solution of other water soluble salts.

Note: A common industry practice for combatting shale hydration has been based on a partial ion analysis of the water phase of the oil-based drilling fluid. This method is prone to error because it relies on substantial water dilution, several titrations, and a retort analysis for each test. Furthermore, insufficient data exists on the activity of mixed salt systems to allow ion analyses to be related accurately to aqueous-phase activity when multiple salts are present. The use of electrohygrometers as described here is a way to determine activity regardless of the composition of the water phase. Table C-1 should not be used to obtain an activity value of an oil-based drilling fluid based on a titration for calcium chloride or a calcium chloride value based on an activity measurement.

C.2 Equipment

Following is a list of equipment needed:

a. Portable relative humidity sensor and indicator, also called an electrohygrometer. This instrument may also be equipped with a temperature probe, in which case a separate thermometer is unnecessary. Specifications for an electrohygrometer are the following (see Figure C-2):

   1. Relative humidity sensor probe suitable for insertion in the air space of a jar containing the sample, as shown in Figure C-1.
   2. The instrument should indicate within 10% of the true activity within 30 minutes, when measuring the brine activity standards described below. The true activity, a_w, of the standards was obtained from the chemical literature.
   3. Linearity response of the instrument such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0.95.

b. Thermometer, 32–220°F (0–105°C).

c. Saturated solutions of the following salts:

Table C-1—Water Activity Versus $CaCl_2$ at 77°F (25°C)

Anhydrous $CaCl_2$, wt%	Water Activity, a_w
9.33	0.95
14.95	0.90
19.03	0.85
22.25	0.80
24.95	0.75
27.40	0.70
29.64	0.65
31.73	0.60
33.71	0.55
35.64	0.50
37.61	0.45
39.62	0.40
41.83	0.35
44.36	0.30

Note: From Stokes and Robinson[5] (see References).

Salt	a_w	Salt per 100 cm^3 water
Calcium Chloride	0.295	100 grams
Calcium Nitrate	0.505	200 grams
Sodium Chloride	0.753	200 grams
Potassium Nitrate	0.938	200 grams

Each salt solution should be prepared by adding the indicated grams of salt to 100 cm^3 of distilled or deionized water, stirring at temperatures between 150–200°F (66–93°C) for 30 minutes, then cooling to a temperature between 75–80°F (24–72°C). Solid salt *must* be visible in the liquid after equilibration.

If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride.

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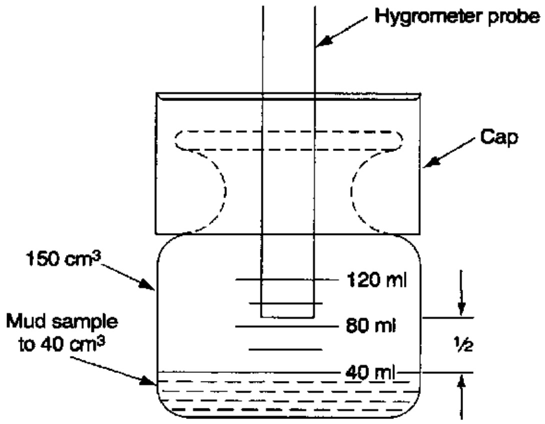

Figure C-1—Jar Containing Oil-based Drilling Fluid Sample and Relative Humidity Probe in Cap: RH Detector in Air Space Above the Sample

Salts of reagent grade purity are recommended. However, if necessary, field grade NaCl or $CaCl_2$ can be substituted.

d. A small wide-mouth jar, approximately 150-cm^3 capacity, which will give a liquid surface area of approximately 38-cm^2. The container should have a flexible elastomer cap, which when bored through will form an airtight seal around the shaft of a sensor probe (see Figure C-1). An example of such a container is the Fleaker®, manufactured by Dow Corning, or equivalent.

e. An insulating cover made of synthetic foam for the wide-mouth jar. The cover should fit snugly and should insulate the side and bottom of the jar from temperature fluctuations. Alternatively, the jar can be kept in a constant temperature water bath.

f. Graph paper—linear.

C.3 Procedure—Calibration of Hygrometer

C.3.1 Prepare each known relative humidity standard. A total volume of 40-cm^3 is sufficient in each sample container. These solutions must be kept firmly capped to prevent spillage, evaporation or contamination.

C.3.2 Desiccate the probe with anhydrous calcium chloride in a sample container. (Calcium sulfate, Drierite®, or silica gel also can be used.)

C.3.3 Place the sensor probe (which is in the jar cap) in the air space above the salt. Wait 10–15 minutes. A reading indicating that the probe is dry is 24% or lower relative humidity (RH).

Note: *Keep a distance of ½ inch (12 mm) between sensing probe and desiccant and maintain same distance for salt solutions and drilling fluid samples. Never allow the probe to directly contact any samples or solutions.*

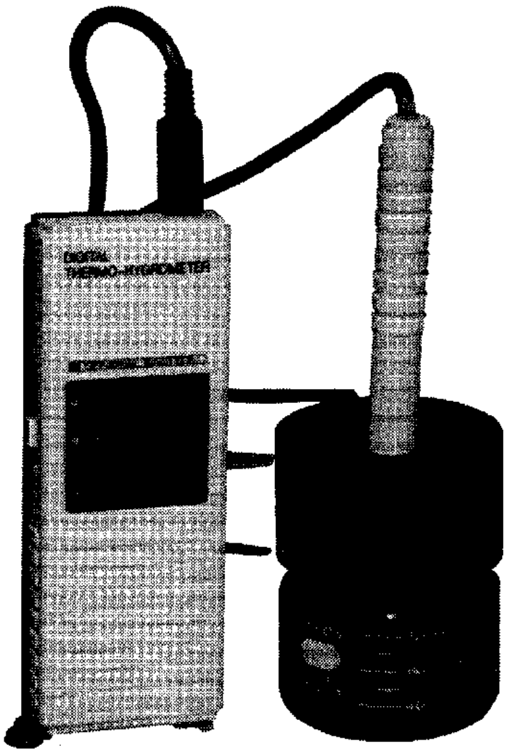

Figure C-2—Digital Electrohygrometer and Mud Sample Jar

C.3.4 Transfer probe and the jar cap to the first (lowest a_w) standard solution and allow 30 minutes for equilibrium to be reached.

C.3.5 Remove probe from the first solution and place into the container with the second lowest known solution.

C.3.6 Proceed as in C.3.3 and measure the relative humidity for each solution, proceeding from the lowest to the highest a_w.

C.3.7 Allow each standard to equilibrate the same length of time. Record temperature and percent relative humidity, % RH.

C.3.8 When complete data are obtained, plot % RH versus a_w on graph paper.

Note: Temperature of the standards and drilling fluid samples should be maintained at 75–77°F (24–25°C).

C.4 Procedure—Measuring Oil-Based Drilling Fluid (Activity (a_{wm})

C.4.1 Desiccate probe for 10–15 minutes as in C.3.2.

C.4.2 Place sample of room temperature fluid (40-cm^3) into sample container.

C.4.3 Insert probe into air space ½ inch (12 mm) above the fluid surface. Sample temperature should be between 75–77°F (24–25°C), or the same temperature at which the standards were measured.

C.4.4 Turn on hygrometer, wait 30 minutes (the same time period used for standard calibration), and record % RH and temperature.

Note 1: Examine the fluid to assure that separation has not occurred. Separation of an oil layer will lead to erroneous readings.

Note 2: Make sure that sample container and cap are clean and free of salt before each new sample is poured into container.

C.5 Calculation—Drilling Fluid Activity (a_{wm})

Using the graph plotted in C.3.8, enter the % RH obtained in C.4.4. Read from the graph the drilling fluid activity, a_{wm}, and record.

APPENDIX D—ANILINE POINT

D.1 Description

D.1.1 This test method covers the determination of the aniline point of petroleum products and hydrocarbon solvents, such as diesel oils and mineral oils used in preparation of oil-based drilling fluids. This method is suitable for transparent liquid samples having 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. This procedure is similar to "Method A" of ASTM: D611-82 (1982) and IP: 2/76 (1976).

D.1.2 The aniline point is useful in characterizing pure hydrocarbons. For example, aromatic hydrocarbons exhibit the lowest aniline points and paraffinic hydrocarbons exhibit the highest values. Cycloparaffins and olefins exhibit values that lie between those of paraffins and aromatics. In a homologous series, the aniline point increases with increasing molecular weight. The aniline point for mixtures of hydrocarbons, such as diesel oils and mineral oils serves as a guideline for judging the aromatic hydrocarbon content of an oil and for comparing oils.

D.1.3 When testing base oils for use in oil muds, low aniline point values (assumed to be related to higher aromatic content) warns of potential damage to the elastomeric ("rubber") components which are in contact with the oil-based mud, such as: pump swabs, drillpipe rubbers, BOP rubbers and hoses. High aniline point values (assumed to represent lower aromatic content) indicate lowered risk of elastomer damage but may indicate oils of less solvency and lowered ability to disperse asphaltic materials in oil muds.

D.1.4 Definition: *aniline point* is the lowest temperature at which equal volumes of aniline and hydrocarbon samples are completely miscible.

D.2 Equipment

Following is a list of equipment needed:

a. Aniline: (CAS #62-53-3) pure, water-free and not excessively dark colored. (Store in a dark, cool place.)

CAUTION: Aniline is extremely toxic and should not be pipetted by mouth. Aniline is also toxic by absorption through the skin and should be handled carefully while wearing proper plastic gloves. Dispose of aniline and its mixtures in safe and proper manner according to federal, state and local environmental and health regulations.

b. Calcium sulfate CAS #7778-18-9 or sodium sulfate CAS #7757-82-6 (anhydrous): drying agent.

c. Test tube: One 25-mm diameter, 150-mm length, made of heat resistant glass.

d. Jacket: approximately 37-mm to 42-mm diameter and 150-mm length, made of heat resistant glass.

e. Thermometer: 77 to 221°F (25 to 105°C). Refer to ASTM Spec. E.1, thermometer 34 C. 34 F.

f. Cork stoppers: (1) to fit inside test tube to hold thermometer; (2) to fit inside jacket to hold the test tube. *(Do not use rubber stoppers).*

g. Wire stirrer: 14 gauge wire bent with a 19-mm diameter loop, used to stir mixture in test tube. (See Figure D-1.)

h. Heating bath: a hot air bath, a nonvolatile, transparent liquid bath (oil or silicone—not water) or an infrared heating lamp (250 to 375 W) to provide a means for heating the test tube and sample.

i. Cooling bath: a cool air bath or a transparent liquid bath (not water) to provide a means for cooling the test tube and sample below room temperature.

Note: Water should not be used as a heating or cooling medium because aniline is hygroscopic. Moist aniline will cause erroneously high results.

j. Pipette (TD): one 10-cm^3 ±0.04-cm^3.

k. Plastic pipette suction bulb.

l. Safety goggles.

m. Plastic gloves: material impervious to aniline.

D.3 Procedure

D.3.1 Dry the oil sample by adding approximately 10 volume % drying agent in the oil. Shake vigorously for 3 to 5 minutes. Filter or centrifuge to remove the drying agent.

D.3.2 Clean and dry the test tube, jacket, thermometer and wire stirrer.

D.3.3 Pipette 10-cm^3 aniline (using suction bulb) into test tube. Likewise, pipette 10-cm^3 of oil sample into the test tube.

D.3.4 Assemble the thermometer and stirrer as shown in Figure D-1. Position the thermometer in the test tube so that the immersion mark is at the top of the liquid. Be sure that the thermometer bulb does not touch the sides of the tube. Center the test tube in the jacket tube.

D.3.5 While at room temperature, stir the mixture rapidly, using an "up-and-down" motion of the stirrer. Avoid forming air bubbles. Observe if mixture is miscible (clear) or immiscible (cloudy). If the mixture is clear go to D.3.12, for procedure using cooling.

Note: For most base oils the aniline-oil mixture will be immiscible (cloudy) at room temperature.

D.3.6 If the mixture is cloudy, apply heat to the jacket tube to increase temperature of mixture in test tube at a rate of 2 to 5°F/min. (1 to 3°C/min.).

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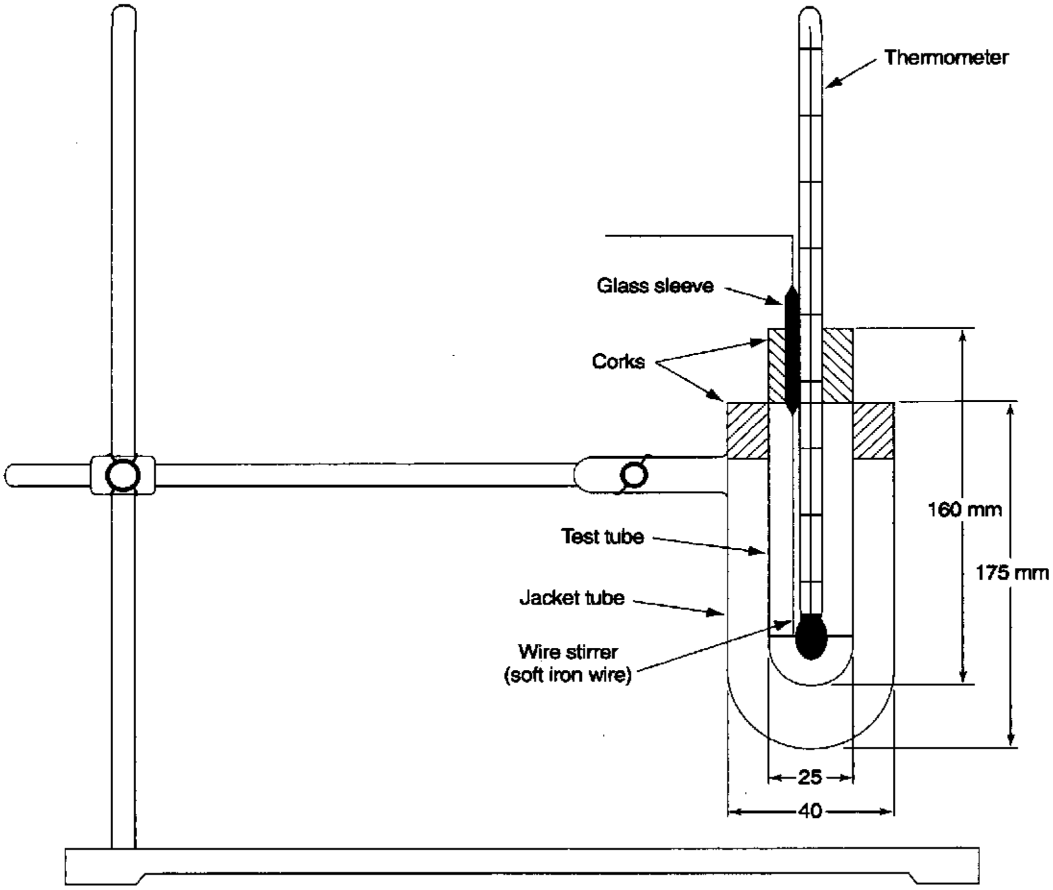

Figure D-1—Aniline Point Apparatus

D.3.7 Continue heating while stirring until the mixture becomes completely miscible (clear). Remove the mixture from the heating source.

Note: Do not heat an aniline/oil mixture beyond 200°F. To do so could be unsafe.

D.3.8 Continue stirring while the clear mixture cools at a rate of 1 to 1.8°F/min. (0.5 to 1.0°C/min.). Notice when the cloudiness first reappears. Record as "aniline point" the temperature at which the mixture becomes cloudy throughout.

Note: The true aniline point is characterized by a turbidity that is so cloudy as to obscure the thermometer bulb in reflected light.

D.3.9 Continue cooling the mixture while stirring to a temperature of 2.0 to 3.5°F (1 to 2°C) below the first appearance of cloudiness, as noted in D.3.8.

D.3.10 Heat the mixture again (as given in D.3.7) until it again becomes miscible. Remove the heat and cool until cloudiness returns, to obtain another observation of the aniline point temperature.

D.3.11 Repeat the observations of aniline point several times until a value has been determined within ±1°F (±0.5°C).

D.3.12 If the aniline-oil mixture was completely miscible at ambient temperature (D.3.6), substitute a cooling bath for the heating bath. Allow the mixture to cool slowly while stirring rapidly at the rate specified in D.3.6. Determine the aniline point as described in D.3.8–11.

APPENDIX E—LIME, SALINITY, AND SOLIDS CALCULATIONS

E.1 Description

The following example calculations are designed to show how the equations in Section 9 are used to determine the amount of salt and solids present in a *diesel* oil-based mud containing *barite* weighting material. Four different cases are presented:

a. The mud contains *only* $CaCl_2$ salt.
b. The mud contains *only* NaCl salt.
c. The mud contains *both* $CaCl_2$ and NaCl salts.
d. The mud contains *both* $CaCl_2$ and *excess* NaCl salt.

Note: A significant figure to one decimal place is considered accurate enough for all field calculations. Two decimal places are used in these calculations for the Average Density of the Suspended Solids, High-Density Solids, Low-Density Solids and the Brine Density.

E.2 Test data used in examples.

For physical and chemical properties see Table E-1.

E.3 Example Calculations.

E.3.1 Example A—The mud contains *only* $CaCl_2$ salt.

Following are the example calculations:

a. Total Lime Content.

$$\text{Lime}_{OM} = 1.295\ (2.6) = 3.4 \text{ lb/bbl} \tag{20}$$

b. Total Calcium Content.

$$Ca_{OM} = 4000\ (10.0) = 40{,}000 \text{ mg/L of mud} \tag{21}$$

c. Total Chloride Content.

$$Cl_{OM} = 10{,}000\ \ (5.8) = 58{,}000 \text{ mg/L of mud} \tag{22}$$

d. Calcium Chloride Content.

$$CaCl_{2_{OM}} = 2.774\ (40{,}000)\ 110{,}960 \text{ mg/L of mud} \tag{23}$$

$$CaCl_2 = 0.000971\ (40{,}000) = 38.8 \text{ lb/bbl} \tag{24}$$

e. Chloride Content associated with the $CaCl_2$.

$$Cl_{CaCl_2} = 1.77\ (40{,}000) = 70{,}800 \text{ mg/L of mud} \tag{25}$$

f. Sodium Chloride Content check.

$$Cl_{NaCl} = 58{,}000 - 70{,}800 = -12{,}800 \text{ mg/L of mud} \tag{28}$$

Note: The negative number indicates that *no NaCl* is present in this mud; therefore the calcium chloride content must be recalculated.

g. Calcium Chloride Content.

$$CaCl_{2_{OM}} = 1.57\ (58{,}000) = 91{,}060 \text{ mg/L of mud} \tag{26}$$

$$CaCl_2 = 0.00035\ (91{,}060) = 31.9 \text{ lb/bbl} \tag{27}$$

h. Calcium Chloride Weight Percent.

$$W_c = \frac{100\ (91{,}060)}{91{,}000 + 0 + 10{,}000\ (25.0)}\ 26.7\% \text{ by wt.} \tag{31}$$

i. Calcium Chloride, parts per million.

$$CaCl_2 = 10{,}000\ (26.7) = 267{,}000 \text{ ppm} \tag{33}$$

j. Density of the Calcium Chloride Brine using only the $CaCl_2$ value.

$$\rho_B = 0.99707 + 0.007923\ (26.7) + 0.00004964$$

$$(26.7)^2 = 1.24 \text{ g/cm}^3 \tag{35}$$

k. Calcium Chloride, mg/L.

$$CaCl_2 = 10{,}000\ (26.7)\ (1.24) = 331{,}808 \text{ mg/L} \tag{36}$$

l. Volume Percent Brine.

$$V_B = \frac{100\ (25.0)}{(1.24)\ [100 - 26.7]} = 27.5\% \text{ by volume} \tag{38}$$

m. Volume Percent Corrected Solids.

$$V_S = 100 - (59.6 + 27.5) = 13.0\% \text{ by volume} \tag{39}$$

n. Oil/Water Ratio Calculations.

$$O_R = \frac{100(59.5)}{59.5 + 25.0} = 70.4\% \text{ ratio oil} \tag{40}$$

$$W_R = \frac{100(25.0)}{59.5 + 25.0} = 29.6\% \text{ ratio water} \tag{41}$$

$$OWR = 70.4/29.6 \tag{42}$$

o. Oil/Brine Ratio Calculations.

$$O_R = \frac{100(59.5)}{59.5 + 27.5} = 68.4\% \text{ ratio oil} \tag{43}$$

$$B_R = \frac{100(27.5)}{59.5 + 27.5} = 31.6\% \text{ ratio brine} \tag{44}$$

$$OBR = 68.4/31.6 \tag{45}$$

Table E-1—Physical and Chemical Properties For Examples

Property	Mud A	Mud B	Mud C	Mud D
1) Mud Density, lb/gal	11.0	11.0	11.0	11.0
2) Oil Content (V_o), volume %	59.5	59.5	59.5	59.5
3) Water Content (V_w), volume %	25.0	25.0	25.0	25.0
4) EDTA (V_{EDTA}), cm³/cm³ mud	10.0	0.0	8.4	8.4
5) Silver Nitrate (V_{SN}), cm³/cm³ mud	5.8	4.4	7.0	10.0
6) Sulfuric Acid (V_{SA}) cm³/cm³ mud	2.6	2.0	2.6	3.0
7) Oil Density, lb/gal	7.0	7.0	7.0	7.0
8) Low-Density Solids, g/cm³	2.65	2.65	2.65	2.65
9) High-Density Solids (barite), g/cm³	4.20	4.20	4.20	4.20

p. Average Density of Suspended Solids.

$$\rho_S = \frac{[100(11.0)] - [(59.5)(7.0)] - [(27.4)(1.24)(8.345)]}{8.345(13.0)} \qquad (46)$$

$$= 3.68 \text{ g/cm}^3$$

q. Volume High-Density Solids.

$$V_{HDS} = \frac{3.68 - 2.65}{4.2 - 2.65} = 8.6\% \text{ by volume} \qquad (47)$$

r. Volume Low-Density Solids.

$$V_{LDS} = 1.30 - 8.6 = 4.4\% \text{ by volume} \qquad (48)$$

s. Concentration High-Density Solids.

$$M_{HDS} = 3.5\ (4.20)\ (8.6) = 126.4 \text{ lb/bbl} \qquad (49)$$

t. Concentration Low-Density Solids.

$$M_{LDS} = 3.5\ (2.65)\ (4.4) = 40.8 \text{ lb/bbl} \qquad (50)$$

E.3.2 Example B—The mud contains *only NaCl* salt.

Following are the example calculations:

a. Total Lime Content.

$$\text{Lime}_{OM} = 1.295\ (2.0) = 2.6 \text{ lb/bbl} \qquad (20)$$

b. Total Calcium Content.

$$\text{Ca}_{OM} = 4000(0.0) = 0.0 \text{ mg/L of mud} \qquad (21)$$

c. Total Chloride Content.

$$\text{Cl}_{OM} = 10{,}000\ (4.4) = 44{,}000 \text{ mg/L of mud} \qquad (22)$$

d. Calcium Chloride Content.

$$\text{CaCl}_{2_{OM}} = 2.774\ (0.0) = 0.0 \text{ mg/L of mud} \qquad (23)$$

$$\text{CaCl}_2 = 0.000971\ (0.0) = 0.0 \text{ lb/bbl} \qquad (24)$$

e. Chloride Content associated with CaCl_2.

$$\text{Cl}_{\text{CaCl}_2} = 1.77\ (0.0) = 0.0 \text{ mg/L of mud} \qquad (25)$$

f. Sodium Chloride Content check.

$$\text{Cl}_{\text{NaCl}} = 44{,}000 - 0 = 44{,}000 \text{ mg/L of mud} \qquad (28)$$

Note: The positive number indicates that NaCl is present in this mud; therefore the sodium chloride content must be calculated.

g. Sodium Chloride Content.

$$\text{NaCl}_{OM} = 1.65\ (44{,}000) = 72{,}600 \text{ mg/L of mud} \qquad (29)$$

$$\text{NaCl, lb/bbl} = 0.00035\ (72{,}600) = 25.4 \text{ lb/bbl} \qquad (30)$$

h. Weight Percent Sodium Chloride.

$$W_N = \frac{100(72{,}600)}{72{,}600 + 0 + 10{,}000(25.0)} = 22.5\% \text{ by wt.} \qquad (32)$$

i. Sodium Chloride Content, parts per million

$$\text{NaCl} = 10{,}000\ (22.5) = 225{,}000 \text{ ppm} \qquad (34)$$

j. Density of the Sodium Chloride Brine using only the NaCl value.

$$\rho_B = 0.99707 + 0.006504(22.5) + 0.00004395(22.5)^2 \qquad (35)$$

$$= 1.17 \text{ g/cm}^3$$

k. Sodium Chloride, mg/L.

$$\text{NaCl} = 10{,}000\ (22.5)\ (1.17) = 263{,}250 \text{ mg/L} \qquad (37)$$

l. Volume Percent Brine.

$$V_B = \frac{100(25.0)}{(1.17)[100 - 22.5]} = 27.6\% \text{ by volume} \qquad (38)$$

m. Volume Percent Corrected Solids.

$$V_S = 100 - (59.5 + 27.6) = 12.9\% \text{ by volume} \qquad (39)$$

n. Oil/Water Ratio Calculations.

$$O_R = \frac{100\ (59.5)}{59.5 + 25.0} = 70.4\% \text{ ratio oil} \qquad (40)$$

$$W_R = \frac{100\ (25.0)}{59.5 + 25.0} = 29.6\%\text{ ratio water} \quad (41)$$

$$OWR = 70.4/29.6 \quad (42)$$

o. Oil/Brine Ratio Calculations.

$$O_R = \frac{100\ (59.5)}{59.5 + 27.6} = 68.3\%\text{ ratio oil} \quad (43)$$

$$B_R = \frac{100\ (27.6)}{59.5 + 27.6} = 31.7\%\text{ brine water} \quad (44)$$

$$OWR = 68.3/31.7 \quad (45)$$

p. Average Density Suspended Solids.

$$\rho_S = \frac{[100(11.0)] - [(59.5)(7.0)] - [(27.6)(1.17)(8.345)]}{8.345(12.9)} \quad (46)$$

$$= 3.85\text{ g/cm}^3$$

q. Volume High-Density Solids.

$$V_{HDS} = \frac{3.68 - 2.65}{4.2 - 2.65}(12.9) = 10.0\%\text{ by volume} \quad (47)$$

r. Volume Low-Density Solids.

$$V_{LDS} = 12.9 - 10.0 = 2.9\%\text{ by volume} \quad (48)$$

s. Concentration High-Density Solids.

$$M_{HDS} = 3.5(4.20)(10.0) = 147.0\text{ lb/bbl} \quad (49)$$

t. Concentration Low-Density Solids.

$$M_{LDS} = 3.5(2.65)(2.9) = 26.9\text{ lb/bbl} \quad (50)$$

E.3.3 Example C—The mud contains *both* $CaCl_2$ and NaCl salts.

Following are the example calculations:

a. Total Lime Content.

$$Lime_{OM} = 1.295\ (2.6) = 3.4\text{ lb/bbl} \quad (20)$$

b. Total Calcium Content.

$$Ca_{OM} = 4000(8.4) = 33{,}600\text{ mg/L of mud} \quad (21)$$

c. Total Chloride Content.

$$Cl_{OM} = 10{,}000\ (7.0) = 70{,}000\text{ mg/L of mud} \quad (22)$$

d. Calcium Chloride Content.

$$CaCl_{2_{OM}} = 2.774\ (33{,}600) = 93{,}206\text{ mg/L of mud} \quad (23)$$

$$CaCl_2 = 0.000971\ (33{,}600) = 32.6\text{ lb/bbl} \quad (24)$$

e. Chloride Content associated with $CaCl_2$.

$$Cl_{CaCl_2} = 1.77\ (33{,}600) = 59{,}472\text{ mg/L of mud} \quad (25)$$

f. Sodium Chloride Content check.

$$Cl_{NaCl} = 70{,}000 - 59{,}472 = 10{,}528\text{ mg/L of mud} \quad (28)$$

Note: The positive number indicates that *NaCl is present* in this mud; therefore the Sodium Chloride Content must be calculated.

g. Calcium Chloride Content.

$$CaCl_{2_{OM}} = 1.57\ (59{,}472) = 93{,}371\text{ mg/L of mud} \quad (26)$$

$$CaCl_2 = 0.00035\ (93{,}371) = 32.7\text{ lb/bbl} \quad (27)$$

h. Sodium Chloride Content.

$$NaCl_{OM} = 1.65\ (10{,}528) = 17{,}371\text{ mg/L of mud} \quad (29)$$

$$NaCl = 0.00035\ (17{,}371) = 6.1\text{ lb/bbl} \quad (30)$$

i. Weight Percent Calcium Chloride.

$$W_C = \frac{100(93{,}371)}{93{,}371 + 17{,}371 + 10{,}000(25.0)} = 25.9\%\text{ by wt.} \quad (31)$$

j. Weight Percent Sodium Chloride.

$$W_N = \frac{100(93{,}371)}{93{,}371 + 17{,}371 + 10{,}000(25.0)} = 4.8\%\text{ by wt.} \quad (32)$$

Note: Checking Figure 20 confirms that all of the sodium chloride is in solution.

k. Calcium Chloride Content, parts per million.

$$CaCl_2 = 10{,}000\ (25.9) = 259{,}900\text{ ppm} \quad (33)$$

l. Sodium Chloride Content, parts per million.

$$NaCl = 10{,}000\ (4.8) = 48{,}000\text{ ppm} \quad (34)$$

m. Density of Calcium Chloride Brine using only the $CaCl_2$ value.

$$\rho_B = 0.99707 + 0.006504\ (4.8) + 0.007923\ (25.9) \quad (35)$$

$$+ 0.00008334\ (4.8)\ (25.9) + 0.00004395\ (4.8)^2$$

$$+ 0.00004964\ (25.5)^2 = 1.28\text{ g/cm}^3$$

n. Calcium Chloride, mg/L.

$$CaCl_2 = 10{,}000\ (25.9)\ (1.28) = 331{,}520\text{ mg/L} \quad (36)$$

o. Sodium Chloride, mg/L.

$$NaCl = 10{,}000\ (4.8)\ (1.28) + 61{,}440\text{ mg/L} \quad (37)$$

p. Volume percent Brine.

$$V_B = \frac{100(25.0)}{(1.28)[100 - (25.9 + 4.8)]} = 28.2\%\text{ by volume} \quad (38)$$

q. Volume percent Corrected Solids.

$$V_S = 100 - (59.5 + 28.2) = 12.3\%\text{ by volume} \quad (39)$$

r. Oil/Water Ratio Calculations.

$$O_R = \frac{100(59.5)}{59.5 + 25.0} = 70.4\%\text{ ratio oil} \quad (40)$$

$$W_R = \frac{100\ (25.0)}{59.5 + 25.0} = 29.6\%\ \text{ratio water} \qquad (41)$$

$$OWR = 70.4/29.6 \qquad (42)$$

s. Oil/Brine Calculations.

$$O_R = \frac{100\ (59.5)}{59.5 + 28.2} = 67.8\%\ \text{ratio oil} \qquad (43)$$

$$B_R = \frac{100\ (28.2)}{59.5 + 28.2} = 32.2\%\ \text{ratio brine} \qquad (44)$$

$$OBR = 67.8/32.2 \qquad (45)$$

t. Average Density of Suspended Solids.

$$\rho_S = \frac{[100(11.0)] - [(59.5)(7.0)] - [(28.2)(1.28)(8.345)]}{8.345(12.3)} \qquad (46)$$

$$= 3.75\ \text{g/cm}^3$$

u. Volume High-Density Solids.

$$V_{\text{HDS}} = \frac{3.72 - 2.65}{4.2 - 2.65}\ (12.3) = 8.5\%\ \text{by volume} \qquad (47)$$

v. Volume Low-Density Solids.

$$V_{\text{LDS}} = 12.3 - 8.5 = 3.8\%\ \text{by volume} \qquad (48)$$

w. Concentration High-Density Solids.

$$M_{\text{HDS}} = 3.5(4.20)(8.5) = 125.0\ \text{lb/bbl} \qquad (49)$$

x. Concentration Low-Density Solids.

$$M_{\text{LDS}} = 3.5(2.65)(3.8) = 35.3\ \text{lb/bbl} \qquad (50)$$

E.3.4 Example D—The mud contains *both* $CaCl_2$ and *excess* NaCl salts.

Following are the example calculations:

a. Total Lime Content.

$$\text{Lime}_{\text{OM}} = 1.295\ (3.0) = 3.9\ \text{lb/bbl} \qquad (20)$$

b. Total Calcium Content.

$$\text{Ca}_{\text{OM}} = 4000(8.4) = 33{,}600\ \text{mg/L of mud} \qquad (21)$$

c. Total Chloride Content.

$$\text{Cl}_{\text{OM}} = 10{,}000\ \ (10.0) = 100{,}000\ \text{mg/L of mud} \qquad (22)$$

d. Calcium Chloride Content.

$$\text{CaCl}_{2_{\text{OM}}} = 2.774\ (33{,}600) = 93{,}206\ \text{mg/L of mud} \qquad (23)$$

$$\text{CaCl}_2 = 0.000971\ (33{,}600) = 32.6\ \text{lb/bbl} \qquad (24)$$

e. Chloride Content associated with $CaCl_2$.

$$\text{Cl}_{\text{CaCl}_2} = 1.77\ (33{,}600) = 59{,}472\ \text{mg/L of mud} \qquad (25)$$

f. Sodium Chloride Content check.

$$\text{Cl}_{\text{NaCl}} = 100{,}000 - 59{,}472 = 40{,}528\ \text{mg/L of mud} \qquad (28)$$

Note: The positive number indicates that *both* $CaCl_2$ and NaCl are present in the mud; therefore, the Calcium Chloride Content must be recalculated with the Cl_{CaCl_2} value.

g. Calcium Chloride Content.

$$\text{CaCl}_{2_{\text{OM}}} = 1.57\ (59{,}472) = 93{,}371\ \text{mg/L of mud} \qquad (26)$$

$$\text{CaCl}_2 = 0.00035\ (93{,}371) = 32.7\ \text{lb/bbl} \qquad (27)$$

h. Sodium Chloride Content.

$$\text{NaCl}_{\text{OM}} = 1.65\ (40{,}528) = 66{,}871\ \text{mg/L of mud} \qquad (29)$$

$$\text{NaCl} = 0.00035\ (66{,}871) = 23.4\ \text{lb/bbl} \qquad (30)$$

i. Weight Percent of Calcium Chloride.

$$W_C = \frac{100(93{,}371)}{93{,}371 + 66{,}871 + 10{,}000(25.0)} = 22.8\%\ \text{by wt.} \qquad (31)$$

j. Weight Percent of Sodium Chloride.

$$W_N = \frac{100(66{,}871)}{93{,}371 + 66{,}871 + 10{,}000(25.0)} = 16.3\%\ \text{by wt.} \qquad (32)$$

Note: For this example, according to Figure 20 the sodium chloride in solution cannot exceed 7.0% by weight for this amount of $CaCl_2$ in this brine. To find the true sodium chloride in solution, W_N, one must go through the calculational procedure below to arrive at the W_C and W_N that agree with the Figure 20 mutual solubility restraints. In this example one must calculate 1st, 2nd and 3rd "Fractions" and W_C and W_N values to achieve accuracy above 95%.

$$1^{\text{st}}\ \text{Fraction} = \frac{7.0\%\ [\text{NaCl, Figure 20}]}{16.3\%\ \ [W_N,\ \text{Step j above}]}\ 0.429$$

$$1^{\text{st}}\ \text{new NaCl}_{\text{OM}} = 0.429\ (66{,}871) = 28{,}687\ \text{mg/L}$$

$$1^{\text{st}}\ \text{new}\ W_C = \frac{100(93{,}371)}{93{,}371 + 28{,}687 + 250{,}000} = 25.1\%\ \text{by wt.}$$

$$1^{\text{st}}\ \text{new}\ W_N = \frac{100(28{,}687)}{93{,}371 + 28{,}687 + 250{,}000} = 7.7\%\ \text{by wt.}$$

Read from Figure 20; NaCl = 6.0% by wt.

$$2^{\text{nd}}\ \text{Fraction} = \frac{6.0\%}{7.7\%} = 0.779$$

$$2^{\text{nd}}\ \text{new NaCl}_{\text{OM}} = 0.779\ (28{,}687) = 22{,}347\ \text{mg/L}$$

$$2^{\text{nd}}\ \text{new}\ W_C = \frac{100(93{,}371)}{93{,}371 + 28{,}347 + 250{,}000} = 25.5\%\ \text{by wt.}$$

$$2^{\text{nd}}\ \text{new}\ W_N = \frac{100(22{,}347)}{93{,}371 + 28{,}347 + 250{,}000} = 6.1\%\ \text{by wt.}$$

Read from Figure 20; NaCl = 6.0% by wt.

$$3^{\text{rd}}\ \text{Fraction} = \frac{6.0\%}{6.1\%} = 0.984\ \text{(see note below)}$$

$$3^{\text{rd}}\ \text{new NaCl}_{\text{OM}} = (21{,}347)\ (0.984) = 21{,}981\ \text{mg/L}$$

$$3^{\text{rd}}\ \text{new}\ W_C = \frac{100(93{,}371)}{93{,}371 + 21{,}981 + 250{,}000} = 25.5\%\ \text{by wt.}$$

$$3^{rd}\text{ new } W_N = \frac{100(21{,}981)}{93{,}371 + 21{,}981 + 250{,}000} = 6.0\%\text{ by wt.}$$

Note: When the "Fraction" approaches 1.00 no further trials for new values are needed. (In practice, Fractions above 0.95 are satisfactory). In this example the 3^{rd} iteration "Fraction" was 0.984 and W_C and W_N were accurate enough after 2nd trial.

k. Calcium Chloride Content, parts per million.

$$\text{CaCl}_2 = 10{,}000\ (25.5) = 255{,}000\text{ ppm} \qquad (33)$$

l. Sodium Chloride Content, parts per million.

$$\text{NaCl} = 10{,}000\ (6.0) = 60{,}000\text{ ppm} \qquad (34)$$

m. Density of Calcium Chloride and Sodium Chloride Brine.

$$\begin{aligned}\rho_B &= 0.99707 + 0.006504\ (6.0) + 0.007923\ (25.5)\\ &\quad + 0.00008334\ (6.0)\ (25.5) + 0.00004395\ (6.0)^2\\ &\quad + 0.00004964\ (25.5)^2 = 1.28\text{ g/cm}^3\end{aligned}$$

n. Calcium Chloride, mg/L.

$$\text{CaCl}_2 = 10{,}000\ (25.5)\ (1.28) = 326{,}400\text{ mg/L} \qquad (36)$$

o. Sodium Chloride, mg/L.

$$\text{NaCl} = 10{,}000\ (6.0)\ (1.28) = 76{,}800\text{ mg/L} \qquad (37)$$

p. Volume Percent Brine.

$$V_B = \frac{100(25.0)}{(1.28)[100 - (25.5 + 6.0)]} = 28.5\%\text{ by volume} \qquad (38)$$

q. Volume Percent Corrected Solids.

$$V_S = 100 - (59.5 + 28.5) = 12.0\%\text{ by volume} \qquad (39)$$

r. Oil/Water Ratio Calculations.

$$O_R = \frac{100(59.5)}{59.5 + 25.0} = 70.4\%\text{ ratio oil} \qquad (40)$$

$$W_R = \frac{100\ (25.0)}{59.5 + 25.0} = 29.6\%\text{ ratio water} \qquad (41)$$

$$OWR = 70.4/29.6 \qquad (42)$$

s. Oil/Brine Ratio Calculations.

$$O_R = \frac{100\ (59.5)}{59.5 + 28.5} = 67.6\%\text{ ratio oil} \qquad (43)$$

$$B_R = \frac{100\ (28.5)}{59.5 + 28.5} = 32.4\%\text{ ratio brine} \qquad (44)$$

$$OBR = 67.6/32.4 \qquad (45)$$

t. Average Density of Suspended Solids.

$$\begin{aligned}\rho_S,\text{ g/cm}^3 &= \frac{[100(11.0)] - [(59.5)(7.0)] - [(28.5)(1.28)(8.3451)]}{8.345(12.0)}\\ &= 3.79\text{ g/cm}^3\end{aligned} \qquad (46)$$

u. Volume High-Density Solids.

$$V_{HDS} = \frac{3.79 - 2.65}{4.2 - 2.65}\ (12.0) = 8.8\%\text{ by volume} \qquad (47)$$

v. Volume Low-Density Solids.

$$V_{LDS} = 12.0 - 8.8 = 3.2\%\text{ by volume} \qquad (48)$$

w. Concentration High-Density Solids.

$$M_{HDS} = 3.5(4.20)(8.8) = 129.4\text{ lb/bbl} \qquad (49)$$

x. Concentration Low-Density Solids.

$$M_{LDS} = 3.5(2.65)(3.2) = 29.7\text{ lb/bbl} \qquad (50)$$

APPENDIX F—SAMPLING, INSPECTION, AND REJECTION

F.1 Description

The following procedure is the official sampling, inspection, and rejection method for materials conforming to API specification. It is applicable to barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, technical-grade low viscosity carboxymethylcellulose, technical-grade high viscosity carboxymethylcellulose, OCMA grade bentonite, and is to be used in the event of product property dispute.

F.2 Procedure—Powdered Material in Packages

F.2.1 The number of samples taken to be combined for the test sample (see F.4) shall be 15 samples of one pound (0.45 kg) or more per lot.

F.2.2 For each lot of 1,000 packages or less, 15 packages shall be sampled.

F.2.3 The sampling may be carried out by either of the following methods as agreed upon by contracting parties:

F.2.3.1 A sample weighing at least 1 lb (0.45 kg) shall be taken from the top of each package.

F.2.3.2 A sampling tube, capable of taking a core not less than 1 in. (2.5 cm) in diameter shall be used. The tube shall be inserted into the package being sampled so that it will take a core of material from substantially the entire length of the package.

F.3 Procedure—Powdered Material in Bulk

F.3.1 In the collection of samples from cars or storage bins containing 25 to 100 tons (22,700 to 90,900 kg), a sampling tube capable of taking a core not less than 1 in. (2.5 cm) in diameter shall be used. The tube shall be of sufficient length to permit taking a sample substantially from the top to the bottom of the mass being sampled.

F.3.2 Fifteen samples shall be taken from each lot (each car or bin shall be considered one lot). If the dimensions of the container are such that the sample cannot be taken in this manner, the sample shall be taken by a method agreed upon by the contracting parties.

F.3.3 From bins containing less than 25 tons (22,700 kg), at least one sample shall be taken from each container up to lots of 10 containers, and the total number of samples taken from each lot of 100 tons (90,900 kg) or less shall be not less than 10.

F.4 Procedure—Test Sample

F.4.1 The samples from each lot shall be combined, mixed, and quartered or riffled to furnish a test sample of 15 lb (6.8 kg) which shall be divided into 3 equal portions.

F.4.2 Each portion shall be sealed in a suitable airtight moisture-proof container. One sample shall be delivered to the purchaser, one to the supplier, and the third sample shall be retained for a reference test, if required.

F.5 Procedure—Test Results

F.5.1 Each party shall make one measurement on his test sample.

F.5.2 A control or known reference standard, such as the API Test Calibration Barite, shall be tested in the same manner, at the same time as part of the test series. If this test result is within the acceptable range, the test results can be considered valid and reported. If it is not within the acceptable range, the results are to be considered suspect, and the cause found and corrected before retesting.

F.6 Procedure—Inspection

The purchaser's inspector shall be afforded, without charge, all reasonable facilities for careful sampling and inspection. A period of six days shall be allowed for sampling and completion of acceptance test.

F.7 Procedure—Rejection

Rejection of material, based on failure to pass the test prescribed in the specifications, shall be reported to the supplier immediately upon completion of the tests and the cause for rejection shall be stated.

F.8 Procedure—Retesting

F.8.1 Either of the contracting parties may make claim for retest within one week of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest.

F.8.2 Should the contracting parties be unable to reach agreement, the third sample of material shall be delivered unopened to a mutually satisfactory referee laboratory for tests, and the results of this referee shall be binding on both parties.

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APPENDIX G—RIG SITE SAMPLING

G.1 Description

G.1.1 The following is a recommended procedure for obtaining test samples at the rig site from specific tank-truck loads. It is not the official procedure approved for sampling materials for API specification testing.

G.1.2 Upon agreement by contracting parties this procedure can be used for testing the properties of material as delivered to the rig-site. It has been used on barite and weight material with good results and should be equally applicable to similar bulk shipments of bentonite, attapulgite, and sepiolite.

G.2 Equipment—Bottom Flow Sampling (Preferred)

Following is a list of equipment needed:

a. Side-stream sampler: shown in Figure G-1.
b. Canvas bag: 5½ in. (140 mm) diameter and 12 in. (300 mm) long.
c. Container: tie-bag or other suitable container, so constructed as to retain all fines.

G.3 Procedure—Bottom Flow Sampling (Preferred)

G.3.1 Connect a side-stream sampler to the truck outlet.

G.3.2 Take three 3–5 lb (1.4–2.3 kg) samples while unloading, spaced so as to be taken when approximately ¼, ½, and ¾ of the load has been transferred.

Note: One 9–15 lb (4.1–6.8 kg) sample can be taken with the recognition that there is a chance of obtaining a nonrepresentative sample. The greatest potential for error in sampling is contamination from prior shipments of material in the same vessel. This potential error can be minimized by following the instructions closely. Do not take samples from the very top surface or from the very first material out of the bottom. A second potential for error in sampling is taking material from the side-stream sampler when the transfer hose is only partially full. This can occur when the truck is nearly empty or material flow has been interrupted. Segregation has been shown to occur in these instances.

G.3.3 Before each individual sample is taken, open the valve momentarily to flush out any material trapped in the side pipe. Tie a clean canvas bag to the sampler and fill it with one valve opening operation.

Note: Partial closing of the air-jet valve on the bottom outlet prior to taking the sample often helps to get a full sack with a minimum of dusting.

G.3.4 Carefully transfer the sample from the canvas bag to a tie-bag or other suitable container. Label this container with all pertinent information.

G.3.5 Empty the canvas bag before taking the next sample. Always use a clean or new canvas bag for each truck.

G.4 Equipment—Grab Sampling

Following is a list of equipment needed:

a. Sample scoop (shown in Figure G-2):
   1. 4–6 in. (100–150 mm) deep
   2. 12–18 in. (300–450 mm) long
b. Container: tie-bag or other suitable container, so constructed as to retain all fines.

G.5 Procedure—Grab Sampling

G.5.1 Open the top hatch carefully. Do not let the cover bump the top of the tank.

G.5.2 Remove the top surface material under the hatch to form a trench 1½ times wider than the sample scoop.

G.5.3 Scoop out a 3–5 lb (1.4–2.3 kg) sample from the bottom of this trench and transfer to a tie-bag or other suitable container. Label the container.

G.5.4 Repeat this operation at two other spots on the top of the material near the midpoint between the middle and the ends of the truck tank.

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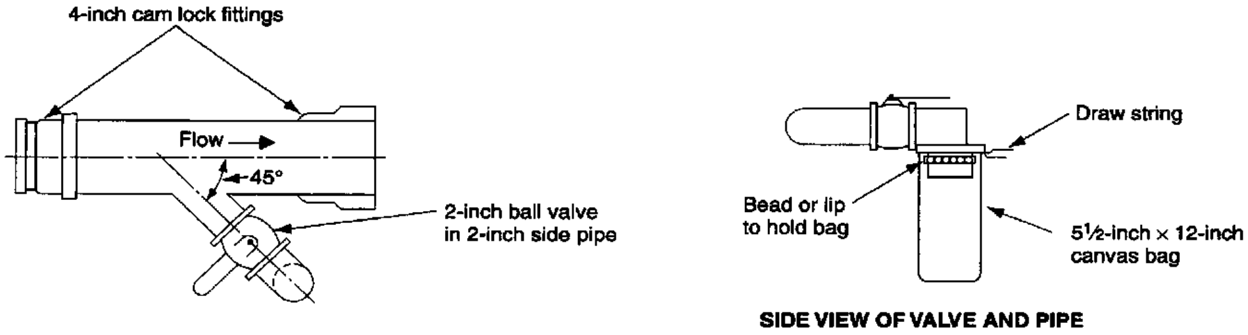


Figure G-1—Side-Stream Sampling Device

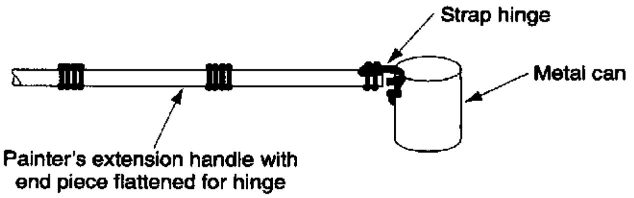


Figure G-2—Sampling Scoop

APPENDIX H—CUTTINGS ACTIVITY BY CHENEVERT METHOD

H.1 Description

H.1.1 This procedure describes the electrohygrometer measurement of the relative humidity of stagnant air in equilibrium with a freshly collected and cleaned cuttings (shale) sample taken from a wellbore. The measure activity, a number ranging from 0 to 1, is proportional to the escaping tendency of water vapor (fugacity) from the sample and relates to the water's chemical potential to react. The water being measured on cuttings by this method is primarily water associated with clay and other hydrous minerals in the shales.

H.1.2 This procedure provides specific directions on sample collection, handling, and washing. It gives details on how to test cuttings using the electrohygrometer. It closely parallels the procedure in Appendix C for measuring the activity of an oil-based mud, and allows for a reliable comparison between activity data for an oil mud and activity data for the cuttings from the same well.

H.1.3 It has been established that cuttings measured at the surface by this test probably do not represent the activity of the same shale downhole. A cutting may be altered due to changes in temperature, pressure, and stress state as it moves from the wellbore to the surface. In addition, a significant difference between the mud and the cutting activity will cause the cutting's activity to approach that of the oil mud. Measured trends of oil mud and cuttings activity from the same well will, in most cases, allow one to judge which has the greater or lesser activity value—the cutting or the oil mud. Additional discussion of oil mud activity concepts are in Appendix C.

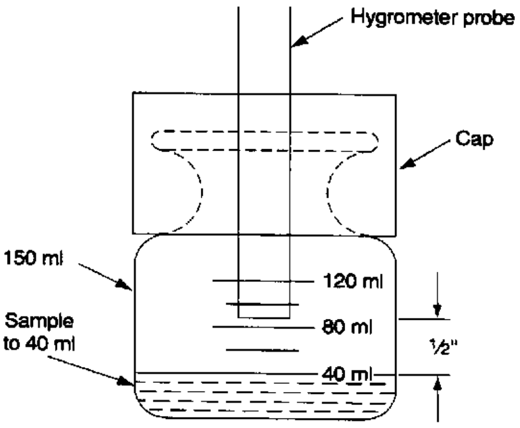
H.2 Equipment

The following is a list of equipment needed:

a. Portable relative humidity sensor and indicator, also called an electrohygrometer. If this instrument is equipped with a temperature probe, a separate thermometer is unnecessary. The specifications for equipment are as follows:

  1. A relative humidity sensor probe suitable for insertion in the air space of a jar containing the samples, as shown in Figure H-1.
  2. When measuring the brine activity standards described below, the instrument must indicate within 10 percent of the true activity within 30 minutes. The true activity, AW, of the standards was obtained from the chemical literature.
  3. The linearity response of the instrument is such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0.95.

b. Thermometer, 32–220°F (0–105°C).



Note: Probe is placed in air space above the sample.

Figure H-1—Jar Containing Cuttings Sample and Relative Humidity Probe in Cap

c. Saturated solutions of the following salts:

Salt	A_w 77°F (25°C)	Salt per 100 cm^3 water
Calcium Chloride	0.295	100 g
Calcium Nitrate	0.505	200 g
Sodium Chloride	0.753	200 g
Potassium Nitrate	0.938	200 g

Each salt solution is prepared by adding the indicated grams of salt to 100 cubic centimeters of distilled or deionized water, stirring at temperatures between 150–200°F (66–93°C) for 30 minutes. Cool to a temperature between 75–80°F (24–27°C). Solid salt must be visible in the liquid after equilibration.

If no crystals are visible, add additional crystals or grains of the same salt to induce precipitation. This is often necessary with calcium chloride.

Salts of reagent grade purity are recommended. Field grade NaCl or $CaCl_2$ can be substituted, if necessary.

d. Small wide-mouthed jar, approximately 150-cubic centimeter capacity, which has a liquid surface area of approximately 5.9 square inches (38 square centimeters). A flexible elastomer cap is fitted in the jar, which when bored through forms an airtight seal around the shaft of the sensor probe (see Figure H-1). An example of such a container is the Fleaker, or equivalent.

e. Insulating cover made of synthetic foam for the wide-mouth jar: the cover fits snugly and insulates the side and bottom of the jar from temperature fluctuations. Alternatively, keep the jar in a constant-temperature water bath.

f. Graph paper: linear.

g. Paper towels: one or more rolls.

h. Washing vessel: 500 cubic centimeter plastic bottle with screw-top lid (such as a Nalgene® bottle or equivalent) or pint jar with lid.
i. Sieve: Tyler No. 30 (28-mesh equivalent).
j. Graduated beaker: 400 cubic centimeters.
k. "Base oil" supply: 10–1.5 liters.

Note: Use the same oil as in the oil mud. Be sure no free water is present.

H.3 Procedure—Calibration of Hygrometer

H.3.1 Calibrate the hygrometer daily.

H.3.2 Prepare each relative humidity standard. A total volume of 40 cubic centimeters is sufficient in each sample container. These solutions must be kept firmly capped to prevent spillage, evaporation, or contamination.

H.3.3 Desiccate the probe with anhydrous calcium chloride in a sample container.

Note: Calcium sulfate, Drierite, or silica gel can also be used.

H.3.4 Place the sensor probe, which is already inserted through the jar cap, in the air space above the desiccant. Wait 10–15 minutes. A reading of 24 percent or lower relative humidity, RH, indicates that the probe is dry.

Note: Keep a distance of ½ inch (12 millimeters) between the sensing probe and desiccant. Maintain the same distance for the salt solutions and the cuttings samples. Never allow the probe to directly contact any samples or solutions.

H.3.5 Transfer the probe and the jar cap to the lowest AW standard solution and allow 30 minutes to reach equilibrium. Read and record percent RH.

H.3.6 Remove the probe and cap from the first solution container and place onto the container with the second-lowest known AW solution. Wait 30 minutes, then read and record percent RH.

Note: Make sure the jar cap with probe is clean and dry when it is transferred between containers. Any salt solution adhering to the cap must be cleaned off before moving the cap from standard salt solution containers or the sample container. Thoroughly wipe the rim of each container dry before installing a cap.

H.3.7 Proceed as in H.3.4 and measure the relative humidity for each solution, proceeding from the lowest to the highest AW.

H.3.8 Allow each standard to equilibrate the same length of time (30 minutes). Record the temperature and percent relative humidity (%RH) for each.

H.3.9 When complete data are obtained, plot percent RH versus AW on graph paper.

Note: Maintain the temperature of the standards and drilling fluid samples between 75–77°F (24–25°C).

H.4 Procedure—Measuring Oil-Based Mud Cuttings Activity

H.4.1 Desiccate the probe for 10–15 minutes as in H.3.2.

H.4.2 Collect a fresh sample of cuttings directly from the shale shaker screen, taking care to minimize its exposure to air. Minimize the amount of oil mud adhering to the cuttings.

H.4.3 Add 300 cubic centimeters of clean, water-free, room-temperature base oil to a 500-cubic centimeter bottle or pint jar.

H.4.4 Measure approximately 100 cubic centimeters loosely packed, fresh cuttings in the 400-cubic centimeter beaker. Transfer these to the bottle containing the base oil and install the lid.

H.4.5 Gently shake the bottle by hand for 30 seconds.

H.4.6 Remove the lid and place the sieve over the mouth of the bottle. Pour off and discard the wash oil, retaining the cuttings in the bottle.

H.4.7 Again, add 300 cubic centimeters of clean, room-temperature base oil to the bottle containing the same cuttings and repeat steps in H.4.5 and H.4.6. Continue these washing steps until the oil looks clean after shaking with the cuttings, usually 2–14 washings.

H.4.8 After washing the cuttings, drain off the oil. Pour the cuttings onto 3–4 layers of absorbent paper towels. Fold the towels over the cuttings and gently blot them dry.

Note: Do not leave the cuttings open to the air any longer than absolutely necessary.

H.4.9 Quickly transfer 35–45 cubic centimeters of the washed cuttings into the 150-cubic centimeter hygrometer container (Fleaker). Place the sample container into the insulating foam jacket. Install the top.

H.4.10 Replace the container top with the top that has the precalibrated hygrometer probe inserted. Be sure the probe does not contact the cuttings but is about ½ inch (12 millimeters) above the cuttings. The cuttings sample temperature should be the same temperature at which the hygrometer was calibrated, preferably room temperature of 75–77°F (24–25°C).

H.4.11 Turn on the hygrometer, wait 30 minutes (the same time used for standard calibration), and record percent relative humidity (%RH) and temperature.

H.5 Calculation—Cuttings Activity (AWS)

Using the graph plotted in H.3.9, enter the percent relative humidity obtained in H.4.11. Read the cuttings activity (AWS) from the graph and record it along with the temperature obtained in H.4.11.

APPENDIX I—CHEMICAL ANALYSIS OF ACTIVE SULFIDES BY GARRETT GAS TRAIN METHOD

I.1 Description

I.1.1 This procedure is used to measure "active sulfides" in an oil-based drilling fluid using the Garrett Gas Train (GGT). The GGT, Figure I-1, separates gas from a liquid sample and passes the gas through a hydrogen sulfide detector, the Dräger tube, for quantitative sulfide analysis. The Dräger tube responds to H_2S by progressively darkening in length as H_2S reacts with the tube reagent. Two Dräger tubes will cover a wide range of sulfide concentrations, from approximately 1 to 4000 milligrams per liter. The low-range tube is white until H_2S turns it brownish black. The high-range tube is pale blue until H_2S turns it black. No known common mud component nor contaminant, other than H_2S, causes this discoloration in these Dräger tubes.

Note: A lead-acetate paper disk can be used in the top of Chamber 3 for a qualitative indication of the presence of H_2S in a sample.

I.1.2 Whole oil-mud, not filtrate, is analyzed in order to capture all of the "active sulfides." Active sulfides are typically the neutralization products of H_2S and lime plus any unreacted H_2S gas. Utilizing a weak acid in the GGT oil-mud test liberates H_2S gas from active sulfides but not from inert sulfides, such as zinc sulfide, copper sulfide, or lead sulfide.

I.2 Equipment

Following is a list of equipment needed:

a. The Garrett Gas Train apparatus, Figure I-1, consists of a transparent plastic gas train equipped with an inert gas supply and pressure regulator, a floating-ball flowmeter, and a Dräger tube. Specifications for the Garrett Gas Train are:

1. Body:
   - Chamber 1:
     - Depth 3.54 inches (90 millimeters)
     - Diameter 1.52 inches (39 millimeters)
   - Chambers 2 and 3:
     - Depth 3.54 inches (90 millimeters)
     - Diameter 1.18 inches (30 millimeters)
   - Passages between chambers:
     - Diameter 0.08 inches (2.0 millimeters)
   - Material:
     - Lucite®, or equivalent., transparent plastic or glass that is inert to acid, sulfides, and hydrogen sulfide gas.
2. Dispersion tube:
   - Stem:
     - Diameter 0.315 inches (8.0 millimeters)
     - Length 5.9 inches (150 millimeters)
   - Dispersion frit:
     - Diameter 1.18 inches (30 millimeters)
   - Material: Pyrex®, or equivalent, glass.

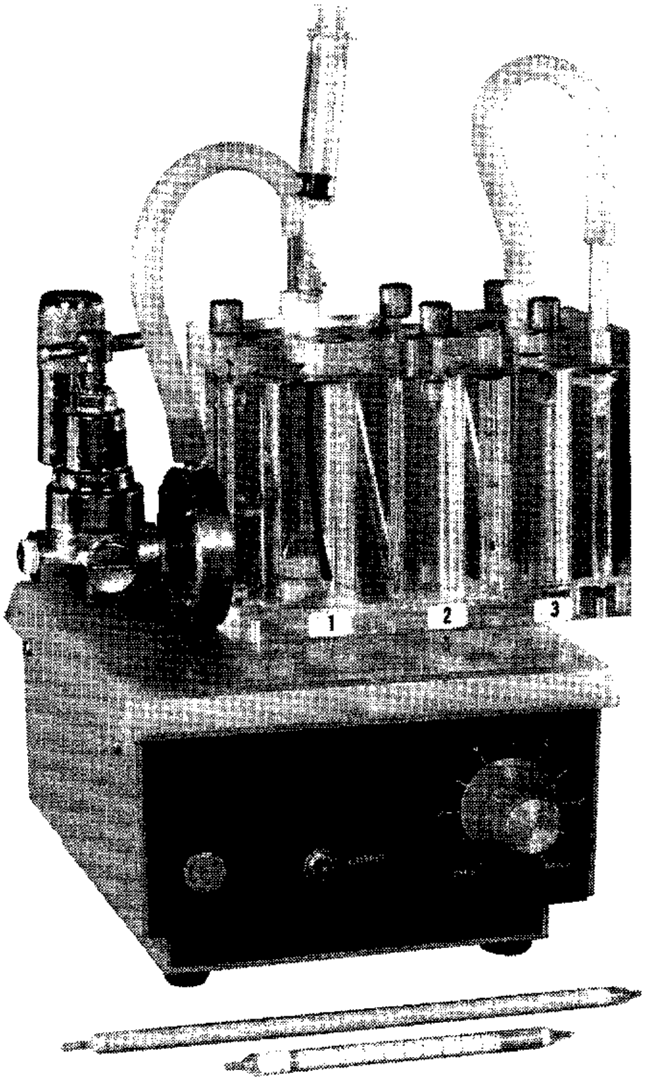


Figure I-1—Active Sulfides by Garrett Gas Train

3. Flowmeter: Floating-ball type, preferred, capable of measuring 300 cubic centimeters per minute of CO_2 gas.
4. Flexible tubing: Type inert to hydrogen sulfide and carrier gas. Latex rubber or Tygon® plastic, or equivalent, preferred.
5. Fittings and Rigid Tubing: Type inert to hydrogen sulfide and acid.

b. Carrier gas: a type inert to hydrogen sulfide, sulfide salts, citric acid, demulsifier, and isopropyl alcohol. Nitrogen or CO_2 is preferred.

CAUTION: Do not use air, N_2O, or other oxidative gases.

c. Dräger H_2S-analysis tubes:
  1. Low range—marked "H_2S 100/a" (Part Number CH-291-01).
  2. High range—marked "H_2S 0.2%/A" (Part Number CH-281-01), or equivalent, H_2S analysis tubes.

d. Citric acid/demulsifier/isopropyl alcohol solution: Prepare a 2M citric acid solution by dissolving 420 grams reagent-grade citric acid ($C_6H_8O_7H_2O$) (CAS #77-929) in deionized water to make 1000 cubic centimeters of solution. Mix 200 cubic centimeters of isopropyl alcohol (CAS #67-63-0) and 25 cubic centimeters of Dowell Schlumberger W-35 demulsifier, or equivalent, into 1000 cubic centimeters of the 2M citric acid solution.

e. Defoamer: Octanol (CAS #111-87-5) in a dropper bottle.

f. Syringe: one 10 cubic centimeter, glass or plastic that is inert to oil mud and sulfides.

g. Graduated cylinder: one 25-cubic centimeter.

h. Magnetic stirrer with a 1-inch (2.5-centimeter) stirring bar, coated with glass or PTFE (Teflon®, or equivalent).

i. Sample injection tube: 6-inch (15.2-millimeters) long PTFE (Teflon, or equivalent) needle, or equivalent, plastic that is inert to oil mud and sulfides.

j. Soft brush.

k. Pipe cleaners.

l. Acetone (CAS #67-64-1), or similar organic liquid.

I.3 Procedure

I.3.1 For Garrett Gas Train equipment other than the type shown in Figure I-1, follow the manufacturer's instructions for method of assembly and operation.

I.3.2 Pass the injection tube through the rubber septum on top of Chamber 1, being sure it seals in the septum.

I.3.3 Set the GGT body, with top removed, on the magnetic stirrer with the center of Chamber 1 over the center of the stirrer. Place the stirring bar in Chamber 1.

Note: The rubber feet must be removed from the GGT body so it will rest flat on the stirrer.

I.3.4 Add 20 cubic centimeters of the citric acid/demulsifier/isopropyl alcohol solution into Chamber 1.

I.3.5 Add 10 drops of octanol defoamer into Chamber 1.

I.3.6 Refer to Table I-1 to select the proper Dräger tube and sample volume.

I.3.7 Break the tips off both ends of the Dräger tube. Install it, with the arrow pointing downward into the bored receptacle downstream of Chamber 3.

I.3.8 Similarly, install the flowmeter tube, with "top" upward, into a receptacle downstream of the Dräger tube. Be sure the O-rings fit tightly and seal around the Dräger tube and the flowmeter tube.

I.3.9 Place the top, with the dispersion tube in place, onto the GGT body. Secure the top firmly. Visually note that all O-rings are pressing firmly against the top. Position the dispersion tube high enough in Chamber 1 to be above the liquid level.

I.3.10 Connect the regulated gas supply by a flexible hose onto the top of the dispersion tube of Chamber 1.

CAUTION: Do not use N_2O or air as carrier gas.

I.3.11 Connect flexible tubing from the outlet of Chamber 3 to the top of the Dräger tube.

CAUTION: To provide pressure relief, do not clamp these tubing connections.

I.3.12 Draw into the syringe, without a needle on it, the mud sample volume as determined in I.3.6, plus 0.5 cubic centimeters of excess mud.

Note: The excess mud compensates for the hold-up volume inside the injection tube, which is approximately 0.5 cubic centimeters.

I.3.13 Attach the syringe to the injection tube and restrain the plunger to keep it from being blown out under pressure. A rubber band can serve to restrain the plunger.

I.3.14 While keeping the dispersion tube frit above the liquid level in Chamber 1, flow the carrier gas for 15 seconds to purge air from the GGT. Avoid frothing of the solution in Chamber 1.

I.3.15 With the carrier gas flowing, check the GGT for leaks.

I.3.16 Stop the gas flow.

I.3.17 Turn on the magnetic stirrer. Adjust its speed until a vortex is formed. Carefully lower the gas dispersion tube into the liquid to a point just above the rotating stirring bar.

I.3.18 Lower the injection tube to the point where the outlet is slightly below the dispersion tube. This will allow the oil mud to enter the vortex.

I.3.19 Very slowly inject the sample from the syringe. Increase the stirrer speed to improve dispersion and to prevent oil mud from sticking to the walls of Chamber 1. Continue stirring for about 1 minute.

I.3.20 Restrain the syringe plunger again and restart carrier gas flow. Adjust the flow rate to 200 to 400 cubic centimeters per minute by keeping the flowmeter ball between the marks.

I.3.21 Observe the Dräger tube for changes, which will begin at the inlet end. Record the maximum distinct stain length, in units marked on the tube, before the front starts to "feather" or smear. Continue the gas flow for at least 15 minutes to ensure that the maximum stain length is achieved.

Note 1: A second CO_2 cartridge may be needed for the full 15 minutes of flow.

Table I-1—Dräger Tube, or Equivalent, Identification, Sample Volumes, and Tube Factors to Be Used for Various Sulfide Ranges

Sulfide Range, mg/L	Sample Volume, cm^3	Dräger Tube Identification (see tube body)	Tube Factor (used in calculation)
1.2–24	10.0	H_2S 100/a	0.12[a]
1.4–48	5.0	H_2S 100/a	0.12[a]
4.8–96	2.5	H_2S 100/a	0.12[a]
60–1020	10.0	H_2S 0.2%/A	1500[b]
120–2040	5.0	H_2S 0.2%/A	1500[b]
240–4080	2.5	H_2S 0.2%/A	1500[b]

[a] A tube factor of 0.12 applies to new tubes, H_2S 100/a (Catalog No. CH-291-01), with a scale from 100 to 2000. For old tubes with 1–20 scale, use a tube factor of 12.

[b] A tube factor of 1500 applies to new tubes, H_2S 0.2%/A (Catalog No. CH-281-01), with a scale from 0.2 to 7.0. For old tubes with 1–17 cm scale, use tube factor 600 times ratio: Batch Factor/0.40.

Note 2: The high-range tube may show an orange-colored stain ahead of the black H_2S front. Orange color indicates that SO_2 gas is coming from sulfite salts in the mud. Ignore the orange region; record only the black stain length.

I.3.22 Clean the GGT apparatus immediately after each use to minimize deterioration of the plastic. Clean the chambers and passages with warm water and mild detergent, using a soft brush and pipe cleaner. Wash the dispersion tube frits and the injection tube with an organic solvent, such as acetone, and then with water. Blow out frits with gas. Occasionally, the dispersion tube may need to be soaked in strong acid to remove calcium carbonate deposits. Rinse the entire GGT unit with deionized water and allow it to drain dry.

I.4 Calculation

Using the "Sample Volume" (not including the hold-up volume), the Dräger tube's maximum "Stain Length," and "Tube Factor" (found in Table I-1), calculate the "Active Sulfides" in the oil mud.

$$Active\ Sulfides,\ \text{mg/L} = \frac{(Stain\ Length)(Tube\ Factor)}{(Sample\ Volume,\ \text{cm}^3)}$$

Note: For best Dräger tube accuracy, the "darkened length" should fill more than half of a tube length. Therefore, select the mud "sample volume" carefully to achieve accuracy.

APPENDIX J—CALIBRATION OF GLASSWARE, THERMOMETERS, VISCOMETERS, AND MUD BALANCES

J.1 Description

This section covers calibration of field testing equipment not covered as part of a test procedure.

J.2 Equipment

J.2.1 VOLUMETRIC GLASSWARE

Calibration of pipettes, graduated cylinders, and so forth, is generally done by the glassware supplier and can be part of the purchase specification. Where deemed important, glassware users should obtain documented evidence of glassware calibration from the supplier.

J.2.2 THERMOMETERS

Calibrate thermometers used in field testing, especially commonly used metal dial thermometers, against a standard thermometer. Thermometers should be calibrated before being put into service, and then periodically, depending on the importance of the measurement and the stability of the thermometer.

J.2.3 VISCOMETERS

Calibrate viscometers used in field testing against standard viscosity fluids traceable to national standards. Viscometers should be calibrated before being put into service, and then periodically, depending on the importance of the measurements and the stability of the viscometer. While in service, viscometers should be checked at least monthly and more frequently if the viscometer indicates instability.

J.2.4 MUD BALANCES

Calibrate mud balances as specified in the test procedure using deionized water. The recommended frequency is prior to each set of measurements, or as specified by the operator, mud company, or other interested party.

J.3 Calibration Procedure—Thermometer

J.3.1 Place the thermometer to be calibrated side-by-side with a standard thermometer into a constant temperature bath (or suitable container of 1 gallon or more filled with water on a counter top in a room where temperature is relatively constant) and allow to equilibrate for 30 minutes.

J.3.2 Read both thermometers and record the readings.

J.3.3 Repeat the readings at 5-minute intervals to obtain at least four sets of readings.

J.3.4 Calculate the average reading for each thermometer.

J.3.5 For adjustable thermometers, adjust to read the same as the standard thermometer. For other thermometers, go to J. 3.6.

J.3.6 Calculate the thermometer correction as follows:

$$C_1 = R - R_2$$

Where:

R_1 = average reading for the standard thermometer.

R_2 = average reading for the working thermometer.

C = value to add to thermometer reading.

The following are examples:

a. Example 1—thermometer correction determination:

$$R_1 = 75.0°F$$

$$R_2 = 75.5°F$$

$$C = 75.0°F - 75.5°F = 0.5°F$$

or

$$R_1 = 23.89°C$$

$$R_2 = 24.17°C$$

$$C = 23.89°C - 24.17°C = 0.28°C$$

b. Example 2—thermometer correction application:

$$C = 0.5$$

$$R = 78.5$$

$$R_C \text{ (corrected)} = 78.5 + (-0.5) = 78.0°F$$

J.3.7 Mark and identify the thermometer with its correction and calibration date.

Note: It is not good practice to use thermometers whose correction exceeds twice the allowable tolerance of the measurement.

J.4 Calibration Procedure—Viscometer

J.4.1 Obtain a certified calibration fluid or fluids with chart (viscosity versus temperature) to cover the range of interest; for example, 50 centipoise, 100 centipoise, and so forth. Make certain that lot number on the chart matches lot number on the fluid container. Each lot of standard fluid is individually certified. The viscosity will normally vary slightly from lot to lot.

J.4.2 Clean and dry the viscometer bob, rotor, and cup. Place viscometer and fluid side-by-side on counter top in a room with reasonably constant temperature [<5°F (<2.7°C) per hour change]. Allow to stand at least 2 hours to equilibrate.

CAUTION: Water will contaminate the standard fluid.

J.4.3 Operate the viscometer in air for 2–4 minutes to loosen bearings and gears.

Note: If rotor wobbles excessively, correct if possible.

J.4.4 Fill cup to scribed line with calibration fluid and place on meter stage. Move the stage until fluid level is to inscribed line on rotor sleeve.

J.4.5 Place a thermometer (capable of ±0.2°F [±0.1°C]) into the fluid and hold or tape it to prevent breakage. Operate the viscometer at low speed setting until thermometer reading is stable to within 0.2°F (0.1°C) per 30 seconds. Record temperature reading.

J.4.6 Operate viscometer and take readings at 600 revolutions per minute and 300 revolutions per minute. Estimate readings to nearest 0.5 dial unit. If needed, use a magnifying glass.

J.4.7 Using the temperature-viscosity chart supplied with the calibration fluid, determine certified viscosity to the nearest 0.5 centipoise. Compare the 300 revolutions per minute dial reading to standard viscosity and record deviation (plus or minus). Divide the 600 revolutions per minute dial reading by 1.98, compare to standard viscosity, and record deviation.

J.4.8 Deviations exceeding 1.5 units are not acceptable. If deviation exceeds these tolerances, adjust or calibrate the viscometer.

J.4.9 Record the viscometer serial number, date, and deviation. Mark the viscometer with the date of calibration and indication of calibration status.

J.5 Calibration Procedure—Filter Press Gasket and O-Rings

J.5.1 Gaskets or O-rings for the low-pressure, low-temperature filter press must have an inside diameter between 2.99 inches (75.8 millimeters) and 3.03 inches (76.9 millimeters).

J.5.2 Check the gasket or O-ring using a gauge having these diameters.

J.5.3 Do not use gaskets or O-rings outside these tolerances for filter loss testing.

APPENDIX K—METRIC "SI" UNIT CONVERSION TABLE

Property	Traditional Unit	Recommended SI Unit	Symbol	Conv. Factor	Example
Mechanical Properties				**Multiply By:**	
Depth	feet	meter	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimeter	mm	25.4	12¼ in. = 311 mm
Pipe Diameter	inch	millimeter	mm	25.4	4½ in. = 114 mm
Bit Size	inch	millimeter	mm	25.4	12¼ in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	[a]revolutions per minute	r/min	1.0	45 rpm = 45 r/min
Nozzle Size	¹⁄₃₂ inch	millimeter	mm	0.79	¹⁰⁄₃₂ in = 7.9 mm
Nozzle Velocity	feet/sec	meter/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	[a]meter/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic meter	m^3	0.159	3000 bbl = 477 m^3
Liner Size	inch	millimeter	mm	25.4	6½ in. = 165 mm
Rod Diameter	inch	millimeter	mm	25.4	2¼ in. = 57 mm
Stroke length	inch	millimeter	mm	25.4	16 in. = 406 mm
Pump Output	barrel/minute	[a]cubic meter/min	m^3/min	0.159	8.5 bbl/min = 1.35 m^3/min
	gallon/minute	[a]cubic meter/min	m^3/min	0.00378	357 gpm = 1.35 m^3/min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	[a]meter/minute	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	[a]meter/minute	m/min	0.3048	20 ft/min = 6.1 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	$\frac{(^\circ F-32)}{1.8}$	80°F = 27°C
	degree centigrade	degree Celsius	°C	1.0	
Funnel Viscosity	seconds/quart	seconds/liter	s/L	Units cannot normally be converted	
	seconds/1000 cm^3	seconds/liter	s/L	1.0	
Mud Weight	pounds/gallon	kilogram/cubic meter	kg/m^3	120	10 lb/gal = 1200 kg/m^3
	pounds/ft^3	kilogram/cubic meter	kg/m^3	16	74.8 lb/ft^3 = 1200 kg/m^3
Pressure Gradient	psi/ft	kilopascal/meter	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 ft^2	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	[a]centipoise	cP		
Yield Point	lbf/100 ft^2	pascal	Pa	0.48	15 lb/100 ft^2 = 7.2 Pa
Gel Strength	lbf/100 ft^2	pascal	Pa	0.48	3 lb/100 ft^2 = 1.44 Pa
Dial Reading	lbf/100 ft^2	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 centimeter	mPa• s^n/cm^2	100	10 dynes s^n/cm^2 = 100 mPa•s^n/cm^2

Property	Traditional Unit	Recommended SI Unit	Symbol	Conv. Factor	Example
	lbf s^n/100 ft^2	millipascal $seconds^n$/ square centimeter	mPa- s^n/cm^2	479	1.2 lb s^n/100 ft^2 = 575 mPa-s^n/cm^2
API Filtrate	cubic centimetre/30 min	[a]cubic centimeter/30 min	cm^3/30 min		
Filter Cake Sand Content)	1/32 inch	millimeter	mm	0.8	3/32 in. = 2.4 mm
Solids Content) Oil Content) Water Content)	Volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Particle Size	micron	micrometer	µm	1.0	
Chemical Properties					
Ionic Concentration	grains/gallon	[a]milligram/liter	mg/L	17.1	500 grains/gal = 8600 mg/L
	parts/million	[a]milligram/liter	mg/L	× specific gravity	100,000 ppm of NaCl × 1.0707 = 107,070 mg/L
Alkalinity P_f, M_f, P_1, P_2	cubic centimeter/ cubic centimeter	cubic centimeter/ cubic centimeter	cm^3/cm^3		
MBT					
Methylene Blue Content	cubic centimeter/ cubic centimeter	cubic centimeter/ cubic centimeter	cm^3/cm^3		
Miscellaneous					
Additive Concentration	pound/barrel	kilogram/cubic meter	kg/m^3	2.85	10 lb/bbl = 28.5 kg/m^3
Corrosion Rate	lb/ft^2/yr	kilogram/square meter/ year	kg/m^2/yr	4.9	0.9 lb/ft^2/year = 4.3 kg/m^2/year
	mils/year	millimeter/year	mm/a	.0254	200 mils/year = 5.08 mm/a
Bentonite Yield	barrels/US ton	[a]cubic meter/metric tonne	m^3/t	0.175	100 bbl/ton = 17.5 m^3/t
Hydraulic HP	horsepower	kilowatt	kw	0.746	600 hhp = 450 kw
Screen Size					
Mesh	openings/inch	openings/centimeter	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperature	micron	micrometer	µm	1	
Open Area	area percent	square meter/square meter	m^2/m^2	0.01	30% = 0.3 m^2/m^2
Weight of Drill Pipe	pound/foot	kilogram/meter	kg/m	1.49	19.5 lb/ft = 29.1 kg/m
Oil Muds					
Oil Content) Water Content)	volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Emulsion Stability	volts	volts	V		
Activity	Dimensionless				
Salinity	ppm	[a]milligrams/liter	mg/L	specific gravity	250,000 ppm of $CaCl_2$ × 1.24 = 310,000 mg/L
Aniline Point	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	150°F = 66°C

Note:[a]Denotes acceptable deviation from API 2564 preferred unit.

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