Addendum 1
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Standard Practice for Field Testing Water-Based Drilling Fluids

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Addendum 1 to Standard Practice for Field Testing Water-Based Drilling Fluids

1. Delete Section 10.2 Alternative Alkalinity Method

2. Replace Tables A-1 and A-2 with the following:

Table A-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.

Table A.2—Dräger Tube or Equivalent—Identification, Sample Volumes, and Tube Factors for Various Carbonate Ranges

Carbonate Range (mg/L)	Sample Volume (cm^3)	Dräger Tube Identification (See tube body)	Tube Factor (Used in Calculation)
25 to 750	10.0	CO_2 100/a	2.5[a]
50 to 1500	5.0	CO_2 100/a	2.5[a]
100 to 3000	2.5	CO_2 100/a	2.5[a]
250 to 7500	1.0	CO_2 100/a	2.5[a]

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[a] Tube factor 2.5 applies to tubes marked CO_2 100/a (Cat. No. 8101811) with 100 to 3000 scale.

3. Insert the following Appendices J, K, and L:

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

J.1 Description

J.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.

J.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.

J.2 Safety Considerations

CAUTION: The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded end caps and those with set screw secured end caps. Among them they have a total of five different pressure ratings. For safety's sake, it is imperative that the operator know the maximum operating pressure of his equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of the possible limits.

On all PPAs with a threaded end cap, manufacturers have modified the hydraulic pressurization system with a means of pressure relief. These might be available on the earlier models using a set screw secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator of the equipment should familiarize himself with this portion of the apparatus and ensure that it is working properly

Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective parts, create the possibility of cell leakage or failure, which could result in serious injury, or equipment damage.

The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the equipment during normal operation. These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical shorts can occur and create the risk of fire, injury and equipment damage. These devices should be used only on grounded circuits.

Following are suggestions that must be observed to ensure safe operation and maintenance of the PPA.

J.2.1 SAFE OPERATION OF THE HYDRAULIC PRESSURIZATION SYSTEM

J.2.1.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

a. Attempting to disconnect pressure hose from cell at quick coupler.
b. Attempting to remove cell from heating jacket.
c. Moving the PPA.
d. Refilling the hydraulic pump.
e. Performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

J.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.

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

J.2.2 SAFE PNEUMATIC PRESSURIZATION OF BACK PRESSURE RECEIVER

J.2.2.1 Always use either nitrogen or carbon dioxide to pressurize the back pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. 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.

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

J.2.2.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

J.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.

J.2.3 SAFE HEATING

J.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.

J.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.

J.2.4 SAFE ELECTRICAL OPERATION

J.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.

J.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.

J.2.5 SAFE TEST CELL MAINTENANCE

The filtration cell is a pressure vessel and should be considered to be a source of potential danger. The following safety precautions should be followed to ensure safe operation.

a. Cell material should be compatible with the test samples.

b. Never use cells that show signs of severe pitting or stress cracking.

c. Never use cells, cell caps, or retainer rings that show any sign of deformation, or damage. Inspect all threads carefully for signs of damage.

d. Use only undamaged, hardened steel, set screws. Others are unsafe.

J.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. 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.

J.3.1 PERMEABILITY PLUGGING APPARATUS

The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands as closely as possible. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top, and the filtrate collected from the top. The cell pressure is applied by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic oil/sample contamination is prevented by redundant O-ring seals on the piston.

J.3.1.1 Test pressures are limited by the safety limits of the cell as specified by the manufacturer (See preceding Caution); usually either 1800 or 2000 psi (12,420 or 13,800 kPA) at some defined temperature. If back pressure is used in the test, the test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

Note: Cell damage caused by excessive pressure can be categorized as follows: end cap bending, end cap compression, cylinder shear, and cylinder stress. End cap bending can be detected visually or by measurement. End cap compression can be detected by the distortion of the set screw holes or seats, which become oval rather than round. Caps showing signs of damage must not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set screw holes, must not be used.

J.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.

J.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 500°F (260°C). The cell temperature can be measured using a metal stem thermometer. Insert the thermometer into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 watts.

J.3.2 FILTER MEDIUM

J.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.

J.3.2.2 The filter medium can be any porous material such as ceramic, sintered metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness is 0.25 in. (6.4 mm) but, with adapters, thicker disks can be used. A new disk is required for each test. Disks should be soaked in the base fluid for five to ten minutes before use. Vacuum saturation should be used for filter media with low porosity and permeability.

J.3.2.3 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores, too, have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 0.25 in. (6.4 mm) thick. With modification of the cylinder, 1.00 in. (25.4 mm) cores can also be used.

J.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.

J.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.

J.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed, and then weigh the amount of sand necessary to obtain that height. The sand bed should be saturated with the base fluid before the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, resin coated sand can be placed in the cell, heated for 1–3 hours at 300°F (149°C), cooled, and then inverted for the test.

J.3.2.7 There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with water to further classify the disk

Note: Procedure for ceramic disk comparison—Install disk in a PPA cell and fill the cell with water. Using the air permeability equipment, with the upper cell valve closed, adjust the pressure on 30 psi test gauge to 4.0 to 4.5 psi. Open the valve on top of the cell and adjust pressure to 2.0 psi ±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.

J.3.3 Timer: 30-minute interval.

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

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

J.3.6 High speed mixer.

J.4 Procedure—High Temperature, High Pressure

J.4.1 PREHEATING THE HEATING JACKET

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will turn on when the heating jacket temperature has reached the thermostat setting. Readjust the thermostat to 10°F (5.6°C) over the desired test temperature.

J.4.2 LOADING THE FILTRATION CELL

WARNING: The filtration cell is a pressure vessel. The following safety precautions should be followed to ensure safe operation: The cell material should be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting, must not be used. Use only undamaged, hardened steel, set screws. The use of damaged, or common, mild steel, set screws is hazardous.

As received from the manufacturer, the PPA is 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.

J.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.

J.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.

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

J.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.

J.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 mud for five minutes immediately before loading the cell.

J.4.2.6 Turn the cell upright and fill with approximately 275 ml of mud. This allows for expansion while heating. Do not exceed this amount.

J.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.

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

Note: The disk should be soaked in water for at least five minutes immediately before use. Disks should never be reused.

J.4.2.9 Position the top end cap in the cell, tighten the set screws snugly, and close the valve on the top end cap

CAUTION: Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell must be controlled by bleeding off the excess periodically.

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

J.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.

J.4.3.1 Refer to Table J.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.

J.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 J.4.3.4.

J.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.

J.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.

J.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.

J.4.3.6 Refer to Table J.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.

J.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.

Table J.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.

J.4.4 CONDUCTING THE FILTRATION TEST

J.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 minute, and plot cumulative filtrate volumes vs. the square root of time.

J.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.

J.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.

J.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 from a drain hose attached to it. If a hose must be used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

J.4.4.5 After 30 minutes, close the filtrate valve and drain all of the filtrate from the back pressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder should be recorded.

J.5 Test Conclusion and Disassembly

J.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.

J.5.2 The pressurized cell assembly should be allowed to cool in the heating jacket. (See above.) When these tests are run with sufficient frequency to justify it, a cooling stand, station, or bath can be provided to expedite the cooling process. There is a cell handling tool available which should be used any time a hot cell is to be handled.

Note: Extreme care must be exercised in cooling hot cells. This procedure, as recommended, makes it difficult to perform more than one test in an eight hour work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and equipment. Safety must be the primary consideration in these designs.

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

J.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.

J.5.5 Release the back pressure by turning the T-screw on the regulator counter-clockwise until it turns freely.

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

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

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

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

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

CAUTION: If there are indications that the cell is still pressurized, and the lower end cap does not include a screen, the following procedure can be used to determine the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is NOT at the bottom, there is no pressure. If the piston is at the bottom, there could be pressure remaining in the cell. Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

If there are indications 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.

J.5.11 Loosen the six set screws securing the end cap and, using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter may free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool in its place, and use it to remove the cap.

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

J.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 make-up water, then measure and record its thickness and remarks concerning its composition.

J.5.14 Dispose of the remaining mud and wash the cell with fresh water. 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.

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

J.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.

J.5.17 Clean the parts for re-use.

J.6 Data Reporting

J.6.1 FILTRATE REPORTING

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

J.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 J.4.4.1.

J.6.3 CALCULATIONS

Where:

EV is the effluent volume (filtrate) in ml using a 3.5 in^2 disk.

$EV_{7.5}$ is the filtrate volume in ml after 7.5 minutes.

EV_{30} is the filtrate in ml after 30 minutes.

PPT Value, ml = $2 \times EV_{30}$

Spurt Loss, ml = $2 \times [EV_{7.5} - (EV_{30} - EV_{7.5})]$

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

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.

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

K.1 Description

K.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.

K.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.

K.2 Safety Considerations

CAUTION: The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded end caps and those with set screw secured end caps. Among them they have a total of five different pressure ratings. For safety's sake, it is imperative that the operator know the maximum operating pressure of his equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of the possible limits.

On all PPAs with a threaded end cap, manufacturers have modified the hydraulic pressurization system with a means of pressure relief. These might be available on the earlier models using a set screw secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator of the equipment should familiarize himself with this portion of the apparatus and ensure that it is working properly

Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective parts, create the possibility of cell leakage or failure, which could result in serious injury or equipment damage.

The sample cell is hot during operation. The operator 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.

K.2.1 SAFE OPERATION OF THE HYDRAULIC PRESSURIZATION SYSTEM

K.2.1.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

a. Attempting to disconnect pressure hose from cell at quick coupler.
b. Attempting to remove cell from heating jacket.
c. Moving the PPA.
d. Refilling the hydraulic pump.
e. Performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

K.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.

K2.1.3 Make sure, when assembling the cell, that the O-rings in the end caps are properly seated.

K.2.2 SAFE PNEUMATIC PRESSURIZATION OF BACK PRESSURE RECEIVER

K.2.2.1 Always use either nitrogen or carbon dioxide to pressurize the back pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. 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.

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

K.2.2.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

K.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.

K.2.3 SAFE HEATING

K.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.

K.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.

K.2.4 SAFE ELECTRICAL OPERATION

K.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.

K.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.

K.2.5 SAFE TEST CELL MAINTENANCE

The filtration cell is a pressure vessel and should be considered to be a source of potential danger. The following safety precautions should be followed to ensure safe operation.

a. Cell material should be compatible with the test samples.

b. Never use cells bodies 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.

K.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 manufacturer's recommendations concerning maximum temperature, pressure, and sample size be followed. Failure to observe these limitations 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 lower pressure limit should 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.

K.3.1 PERMEABILITY PLUGGING APPARATUS

The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands as closely as possible. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top, and the filtrate collected from the top. The cell pressure is applied by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic oil/sample contamination is prevented by redundant O-ring seals on the piston.

K.3.1.1 Test pressures are limited by the safety limits of the cell as specified by the manufacturer; usually 3000, 4000, or 5000 psi (20,700, or 27,600, or 34,500 kPA) at 500°F (260°C). The back pressure receiver may be used at pressures as high as 750 psi (5171 kPa). If back pressure is used in the test, the test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

K.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.

K.3.1.3 The PPA cell is encased in a thermostatically controlled aluminum chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 500°F (260°C). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 watts.

The PPA can be used 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.

K.3.2 FILTER MEDIUM

K.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 millidarcies to 100 darcies. 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.

K.3.2.2 The filter medium can be any porous material such as ceramic, sintered metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness is 0.25 in. (6.4 mm) but, with adapters, thicker disks can be used. A new disk is required for each test. Disks should be soaked in the base fluid for five to ten minutes before use. Vacuum saturation should be used for filter media with low porosity and permeability.

K.3.2.3 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores, too, have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 0.25 in. (6.4 mm) thick. With modification of the cylinder, 1.00 in. (25.4 mm) cores can also be used.

K.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.

K.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.

K.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed, and then weigh the amount of sand necessary to obtain that height. The sand bed should be saturated with the base fluid before the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, resin coated sand can be placed in the cell, heated for 1–3 hours at 300°F (149°C), cooled, and then inverted for the test.

K.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 water to further classify the disk.

Note: Procedure for ceramic disk comparison—Install disk in a PPA cell and fill the cell with water. Using the air permeability equipment, with the upper cell valve closed, adjust the pressure on 30 psi test gauge to 4.0 to 4.5 psi. Open the valve on top of the cell and adjust pressure to 2.0 psi ±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.

K.3.3 Timer: 30-minute interval.

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

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

K.3.6 High speed mixer.

K.4 Procedure—High Temperature, High Pressure

K.4.1 PREHEATING THE HEATING JACKET

Connect the power cord to the proper voltage as indicated on the nameplate. Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will turn on when the heating jacket temperature has reached the thermostat setting. Readjust the thermostat to 10°F (5.6°C) over the desired test temperature.

K.4.2 LOADING THE FILTRATION CELL

WARNING: The filtration cell is a pressure vessel. The following safety precautions should be followed to ensure safe operation: The cell material should be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting, must not be used.

As received from the manufacturer, the PPA will be equipped with valves that are rated to 500°F (260(C). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 500°F (260°C) or more.

K.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.

K.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.

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

K.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.

K.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.

K.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.

K.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.

K.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.

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

Note: The disk should be soaked In water for at least five minutes before use. Disks should never be reused.

K.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.

K.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 bleeding off the excess periodically.

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

K.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.

K.4.3.1 Refer to Table J.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.

K.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 K.4.3.4.

K.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.

K.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.

K.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.

K.4.3.6 Refer to Table J.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.

K.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.

K.4.4 CONDUCTING THE FILTRATION TEST

K.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 the filtrate at 1, 5, 7.5, 15, 25, and 30 minutes, and plot cumulative filtrate volumes vs. the square root of time.

K.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.

K.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.

K.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, its length should be minimized to reduce the error caused liquid retention on its internal surface.

K.4.4.5 After 30 minutes, close the filtrate valve and drain all of the filtrate from the back pressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder should be recorded.

K.5 Test Conclusion and Disassembly

K.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. 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.

K.5.2 The pressurized cell assembly should be allowed to cool in the heating jacket. (See above.) When these tests are run with sufficient frequency to justify it, a cooling stand, station, or bath can be provided to expedite the cooling process. There is a cell handling tool available which should be used any time a hot cell is to be handled.

K.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.

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

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

K.5.6 Remove its locking pin and the back pressure receiver.

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.

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

K.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 pressure remains in the cell, and the lower end cap does not include a screen, the following procedure can be used to determine the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is NOT at the bottom, there is no pressure. If the piston is at the bottom, there could be pressure remaining in the cell. Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

If there are indications 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.

K.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.

K.5.10 Threaded caps are removed using spanner wrenches. It may be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over-tightening, or insufficient cleaning. It may be necessary to use a suitable holding tool such as a soft jaw vice, chain wrench, strap wrench, or another similar device to secure the cell while the cap is unscrewed.

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

K.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 gently with water, then measure and record its thickness and remarks concerning its composition.

K.5.13 Empty the cell and wash it with fresh water. 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.

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

K.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.

K.5.16 Clean the parts for re-use.

K.6 Data Reporting

K.6.1 FILTRATE REPORTING

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

K.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 K.4.4.1.

K.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 is the effluent volume (filtrate) in ml using a 3.5 in^2 disk.

$EV_{7.5}$ is the filtrate volume in ml after 7.5 minutes.

EV_{30} is the filtrate in ml after 30 minutes.

Note: Observe that all three of these parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard API low pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant, 2, can be modified as necessary to accommodate tests made utilizing other filtration areas.

K.6.4 FILTER CAKE REPORTING

K.6.5 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 L—WATER-BASED DRILLING MUD REPORT FORM

DRILLING MUD REPORT

WATER 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)	/	/	/	/
Retort Solids (% Vol.)				
Retort Liquid (% Vol.) Oil/Water	/	/	/	
Sand Content (% Vol.)				/
Methylene Blue Capacity ☐ lb/bbl equiv ☐ cm^3/cm^3 mud				/
pH ☐Strip ☐Meter @ ________°F				
Alkalinity Mud (P_m), cm^3 N/50 Acid/cm^3				
Alkalinity Filtrate (P_f/M_f), cm^3 N/50 Acid/cm^3	/	/	/	
Chloride (mg/L)				
Total Hardness as Calcium (mg/L)				

MUD PROPERTY SPECIFICATIONS:

Weight	Viscosity	Filtrate

RECOMMENDED TOUR TREATMENT:

REMARKS:

PRODUCTS

SOLIDS EQUIPMENT:

MUD VOLUME	SOLIDS ANALYSIS	MUD RHEOLOGY & HYDRAULICS	COST ANALYSIS

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

API RECOMMENDED PRACTICE 13B-1
SECOND EDITION, SEPTEMBER 1997

American
Petroleum
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Exploration and Production Department

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FOREWORD

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

Additional publications under jurisdiction of this committee include Specification 13A, which covers specifications and test procedures for barite, hematite, bentonite, nontreated bentonite, attapulgite, and sepiolite, starch, technical-grade low-viscosity CMC, technical-grade high-viscosity CMC, and OCMA grade bentonite. Also included are Recommended Practice 13B-2, Bulletin 13C, Bulletin 13D, Recommended Practice 13E, Recommended Practice 13G, Recommended Practice 13I, Recommended Practice 13J, and Recommended Practice 13K.

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

1 Scope

The purpose of this recommended practice is to provide standard procedures for the testing of water-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 Recommended Practice 13G, Second Edition, May 1982). 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

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

API

RP 13B-2	*Recommended Practice Standard Procedure for Field Testing Oil-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*

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).

3.2 EQUIPMENT

The following equipment is needed:

a. Any instrument of sufficient accuracy to permit measurement within ±0.1 pound per gallon (or 0.5 pound per cubic foot, 0.01 gram per cubic centimeter, 10 kilograms per cubic meter) may be used. The mud balance (see 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, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (Attachments for extending the range of the balance may be used when necessary.)

b. Thermometer: 32–220°F (0–105°C).

3.3 PROCEDURE

This test procedure should be followed:

a. Set the instrument base on a flat, level surface.

b. Measure the temperature of the mud and record it on the "Drilling Mud Report" form.

c. 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. Ensure 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 for air removal).

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

e. 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.

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

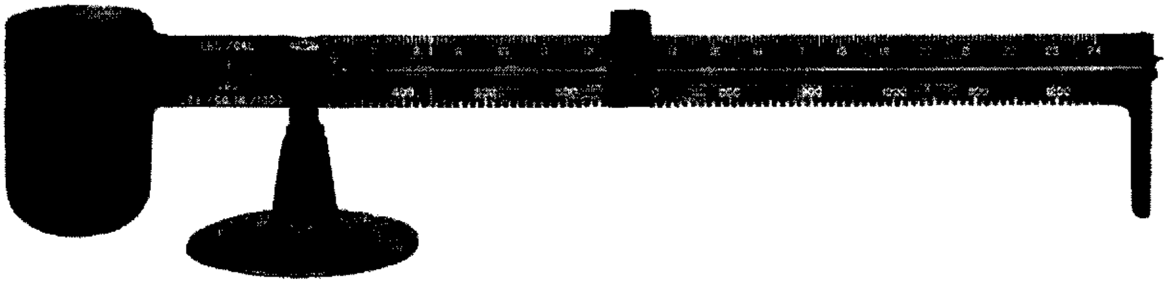
3.4 PROCEDURE—CALIBRATION

The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 pounds per gallon or 62.3 pounds per cubic foot (1000 kilograms per cubic meter) 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.

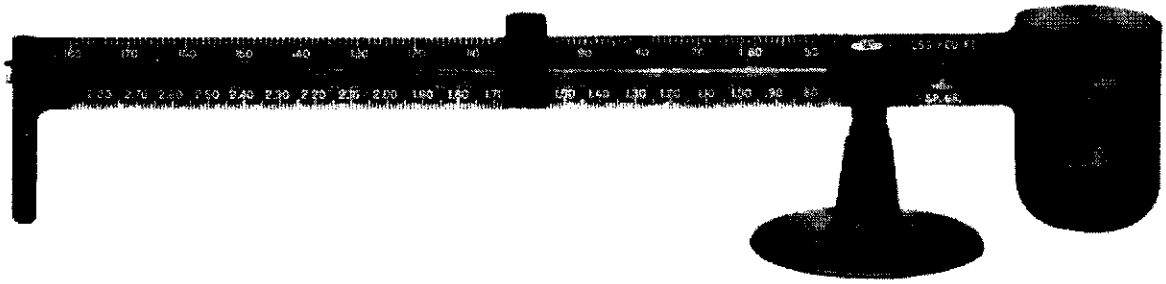
3.5 CALCULATION

The following are the steps for calculation:

a. Report the mud weight to the nearest 0.1 pound per gallon or 0.5 pound per cubic foot (0.01 gram per cubic meter, 10 kilograms per cubic meter).



FRONT VIEW



BACK VIEW

Figure 1—Mud Balance

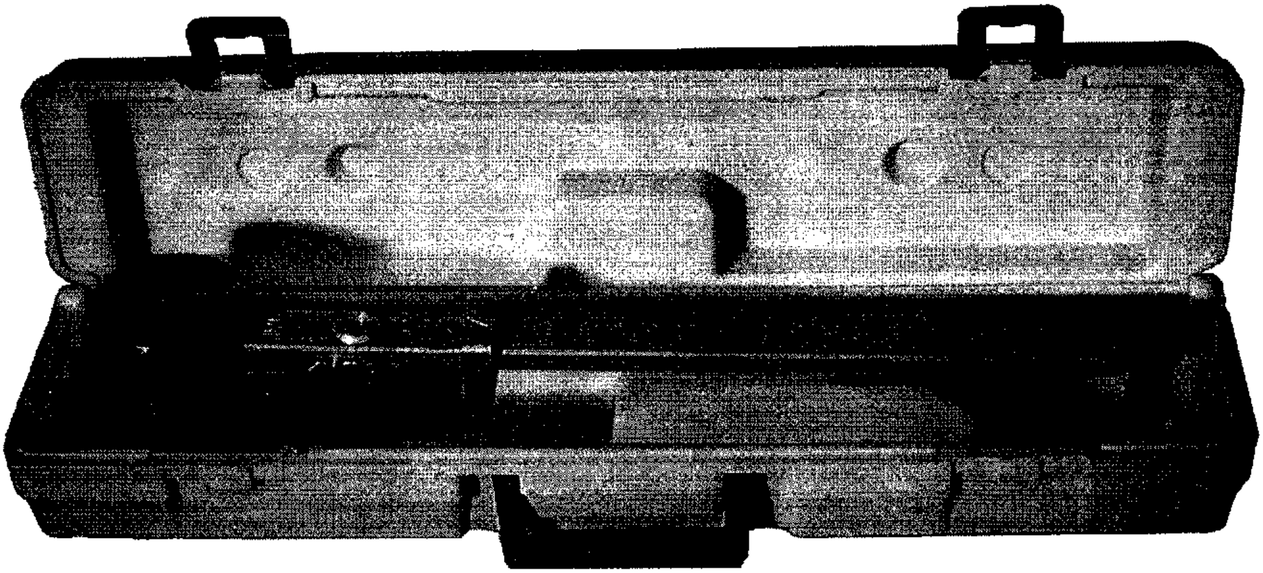


Figure 2—Mud Balance and Case

b. To convert the reading to other units, use the following:

$$Density = g/cm^3 = \frac{lb/ft^3}{62.3} = \frac{lb/gal}{8.345}$$

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

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

3.6 ALTERNATE MUD WEIGHT METHOD

3.6.1 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 cut 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.

3.6.2 Equipment

The following equipment is needed for the alternate mud weight method:

a. Measurement device: Any instrument of sufficient accuracy to permit measurement within ±0.1 pound per gallon (or 0.5 pound per cubic foot, 0.01 gram per cubic meter, 10 kilograms per cubic meter) may be used. The pressurized mud balance (see 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).

3.6.3 Procedure

This test procedure should be followed for the alternate mud weight method:

a. Fill the sample cup to a level slightly below the upper edge of the cup [approximately 0.25 inch (6.4 millimeters)].
b. Place the lid on the cup with the attached check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry will be expelled through the check valve. When the lid has been placed on the cup, pull the check valve up in the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.
c. 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. Then draw the piston rod upward, thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with a fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean-up of the plunger mechanism.
d. 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 newtons) force or greater should be maintained on the piston rod (see Figure 4).
e. 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.
f. The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place the 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 pounds per gallon, specific gravity, pounds per square inch/1000 feet and pounds per cubic feet.
g. To release the pressure inside the cup, reconnect the empty plunger assembly and push downward on the cylinder housing.
h. Clean the cup and rinse thoroughly with water. For best operation in water-based slurries, the valve should be greased frequently with waterproof grease.

3.6.4 Procedure—Calibration

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 8.3 pounds per gallon or 62.3 pounds per cubic feet (1000 kilograms per cubic meter) 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.

3.6.5 Calculation

The following is the calculation procedure:

a. Report the mud weight to the nearest 0.1 pound per gallon or 0.5 pound per cubic foot (0.01 gram per cubic centimeter, 10 kilograms per cubic meter).

Figure 3—Pressurized Fluid Density Balance

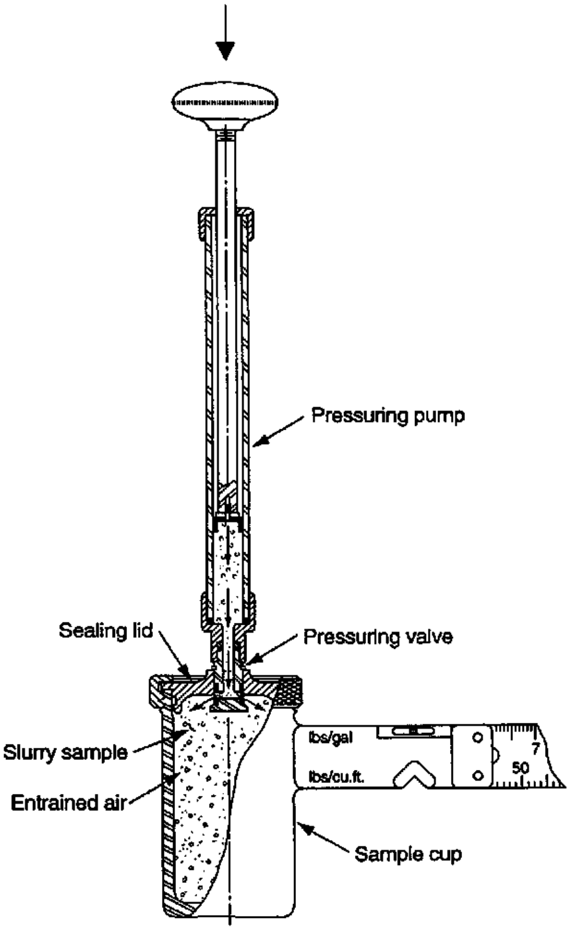


Figure 4—Typical Pressurized Fluid Density Balance Diagram

b. To convert the reading to other units, use the following:

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

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

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

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

Table 1—Density Conversion

1	2	3	4
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	112.5	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

---

[a] Same as specific gravity (sg).

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 called *rheology*. An in-depth discussion of rheology is made in API Bulletin 13D.

4.2 MARSH FUNNEL

4.2.1 Equipment

The following equipment is used:

a. Marsh funnel: a marsh funnel (see Figure 5) is calibrated to out-flow one quart (946 cubic centimeters) 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:

1. Funnel Cone:
   Length .... 12.0 inches (305 millimeters).
   Diameter .... 6.0 inches (152 millimeters).
   Capacity to the bottom
   of the screen .... 1500 cubic centimeters.
2. Orifice:
   Length .... 2.0 inches (50.8 millimeters).
   Inside Diameter .... 3/16-inch (4.7 millimeters).
3. Screen: .... 12 mesh.

The screen has 1/16-inch (1.6 millimeters) openings and is fixed at a level 3/4 inch (19.0 millimeters) below the top of the funnel.

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

4.2.2 Procedure

This procedure should be followed:

a. Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill the funnel until the fluid reaches the bottom of the screen.
b. Remove finger and start the stopwatch. Measure the time for the mud to fill to the one-quart (946-cubic centimeter) mark of the cup.
c. Measure the temperature of fluid in °F (°C).
d. Report the time to the nearest second as marsh funnel viscosity. Report the temperature of the fluid to the nearest °F (°C).

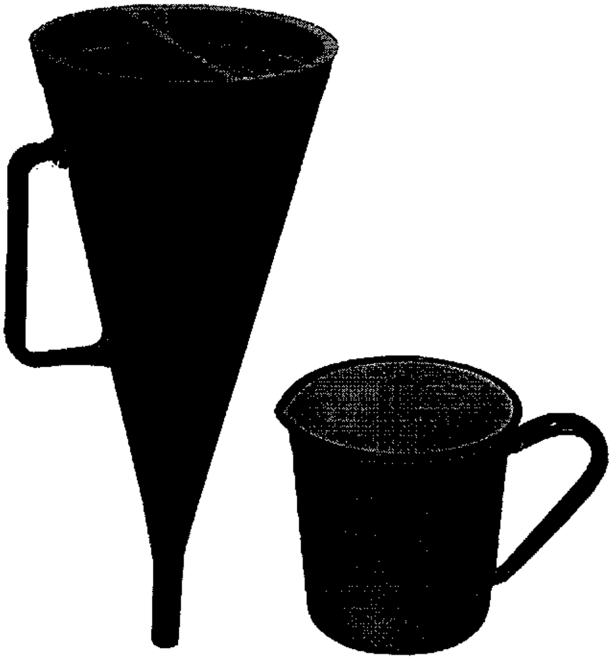


Figure 5—Marsh Funnel and Cup

4.3 DIRECT-INDICATING VISCOMETER

4.3.1 Equipment

The following equipment is used:

a. Direct-indicating viscometers: These 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 (revolutions per minute). 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 revolutions per minute and 600 revolutions per minute.

Specifications—Direct-indicating viscometer:

1. Rotor Sleeve:

Inside diameter........1.450 inches (36.83 millimeters).
Total length ....3.425 inches (87.00 millimeters).
Scribed line ....2.30 inches (58.4 millimeters).

These dimensions are above the bottom of the sleeve.

There are two rows of ⅛-inch (3.18-millimeter) holes spaced 120 degrees (2.09 radians) apart, around the rotor sleeve just below the scribed line.

2. Bob:

Diameter ....1.358 inches (34.49 millimeters).
Cylinder length ......1.496 inches (38.00 millimeters).

The bob is closed with a flat base and a tapered top.

3. Torsion spring constant: 386 dyne-centimeters/degree deflection.

4. Rotor speeds:

High speed...............600 revolutions per minute.
Low speed...............300 revolutions per minute.

b. Types of viscometers used to test drilling fluids:

1. Hand-cranked instrument (see Figure 6) has speeds of 300 revolutions per minute and 600 revolutions per minute. 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 revolutions per minute and 600 revolutions per minute. 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 revolutions per minute. The 3-revolutions per minute 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-revolution per minute and 625 revolutions per minute. The 3-revolutions per minute speed is used to determine gel strength.

c. Stopwatch.

d. Suitable container, for example, the cup provided with the viscometer.

e. Thermometer: 32°F–220°F (0°C–105°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.

This procedure should be followed:

a. Place a sample in the container and immerse the rotor sleeve exactly to the scribed line. Make measurements in the field with minimum delay (within five minutes, if possible) and at a temperature as near as is practical to that of the mud at the place of sampling [not to differ more than 10°F (6°C)]. State the place of sampling on the report.

b. Record the temperature of the sample.

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

d. Shift to 300 revolutions per minute and wait for the dial reading to reach steady value. Record the dial reading for 300 revolutions per minute.

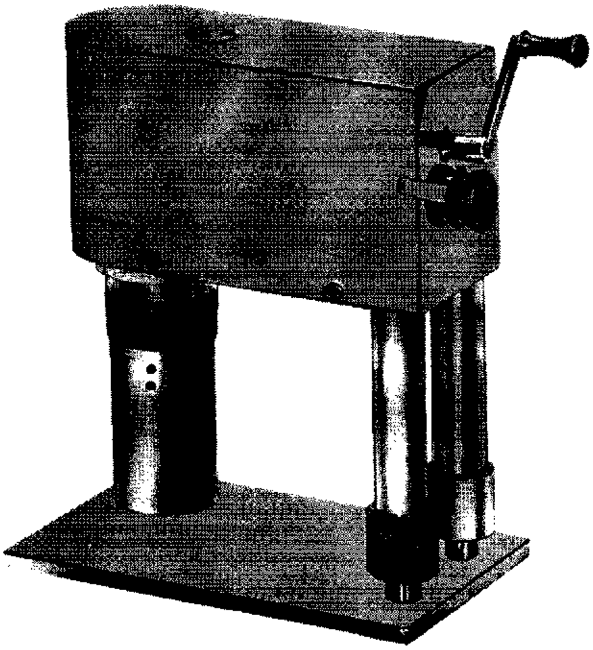


Figure 6—Hand-Crank Viscometer

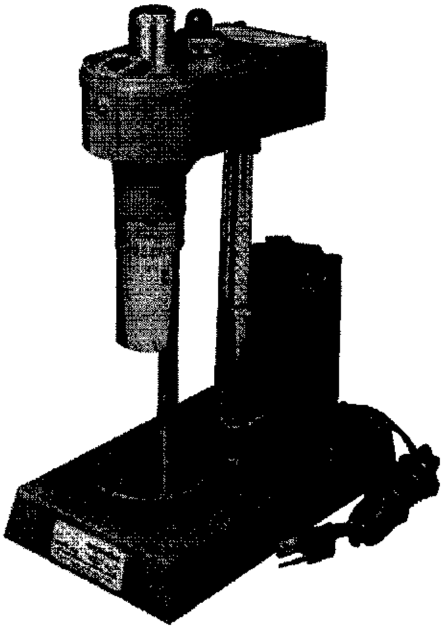


Figure 8—115-Volt Motor-Driven Viscometer

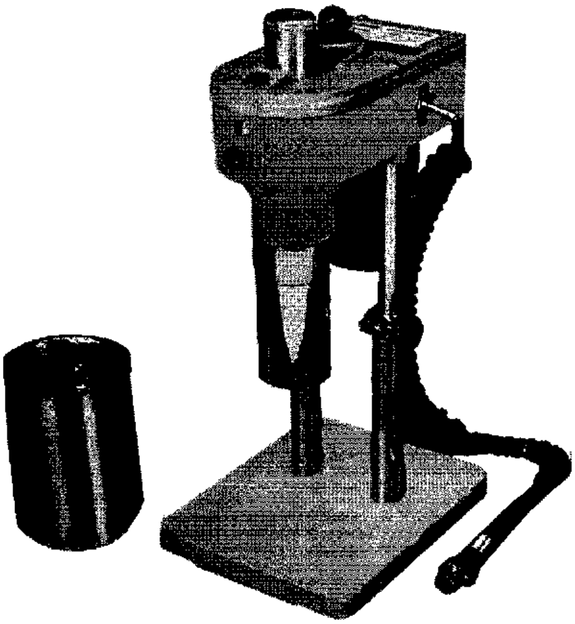


Figure 7—12-Volt Motor-Driven Viscometer

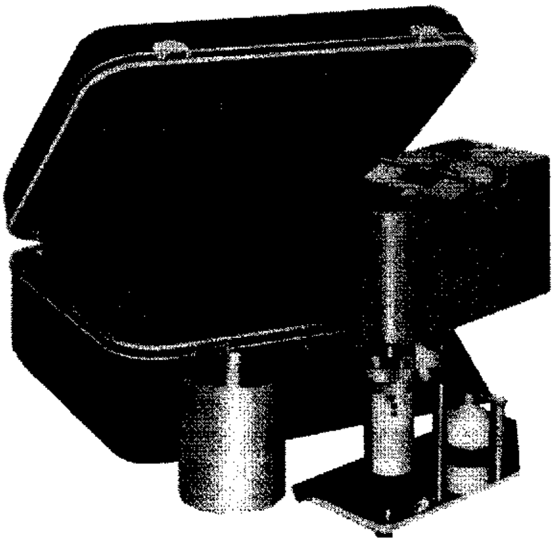


Figure 9—Variable Speed Viscometer

e. Stir the drilling fluid sample for 10 seconds at high speed.

f. Allow the drilling fluid sample 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-revolutions per minute speed, the maximum reading attained after starting rotation at 3 revolutions per minute is the initial gel strength. Record the initial gel strength (10 sec gel) in pounds per 100 square feet (pascals).

g. Restir the drilling fluid sample 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, Item f, and report the maximum reading as 10-minute gel in pounds per 100 square feet (pascals).

4.3.3 Calculation

The following calculation should be used:

$$\textit{Plastic Viscosity}, \text{cP} = \begin{bmatrix} 600\ rpm \\ reading \end{bmatrix} - \begin{bmatrix} 300\ rpm \\ reading \end{bmatrix}$$

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

$$\textit{Apparent Viscosity}, \text{cP} = \frac{600\ rpm\ reading}{2}$$

5 Filtration

5.1 DESCRIPTION

Measurement of the filtration behavior and wall-cakebuilding characteristics of a mud are fundamental to drilling-fluid control and treatment, as are the characteristics of the filtrate, such as oil, water, or emulsion content.

These characteristics are affected by the types and quantities of solids in the fluid and their physical and chemical interactions, which, in turn, are affected by temperature and pressure. Therefore, tests are run at both low pressure/low temperature and high pressure/high temperature, and each requires different equipment and techniques.

5.2 LOW-TEMPERATURE/LOW-PRESSURE TEST

5.2.1 Equipment—Low-Temperature/Low-Pressure Test

The following equipment is needed for the low-temperature/low-pressure test:

a. A filter press: This consists mainly of a cylindrical mud cell having an inside diameter of 3 inches (76.2 millimeters) and a height of at least 2.5 inches (64.0 millimeters). This chamber is made of materials resistant to strongly alkaline solutions and is so fitted that a pressure medium can be conveniently admitted into, and bled from, the top. The arrangement is also such that a sheet of 9-centimeter filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is 7.1 ±0.1 cubic inches (4580 ±60 cubic millimeters). Below the support is a drain tube for discharging the filtrate into a graduated cylinder.

Sealing is accomplished with gaskets, and the entire assembly supported by a stand. Figures 10 and 11 depict standard units by different manufacturers.

Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges, or means for utilizing hydraulic pressure.

To obtain correlative results, one thickness of the proper 90-millimeter filter paper, Whatman No. 50, S&S No. 576, or the equivalent, must be used.

Note: The mini-press or half-area press does not directly correlate with the results obtained when using the standard-sized press.

Note: The API low-temperature/low-pressure filter press must have a filter area of 4520 square millimeters to 4640 square millimeters, which is a diameter of 75.86 millimeters to 76.86 millimeters. The filter press gasket is the determining factor of the filter area. It is recommended that a 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.

b. Timer: 30-minute interval.

c. Graduated cylinder (TC): 10-cubic centimeter or 25-cubic centimeter.

5.2.2 Procedure—Low-Temperature/Low-Pressure Test

This procedure should be followed for the low-temperature/low-pressure test:

a. Be sure each part of the cell, particularly the screen, is clean and dry, and that the gaskets are not distorted or worn. Pour the mud sample into the cell to within 0.5 inch (13 millimeters) to the top (to minimize CO_2 contamination of filtrate) and complete the assembly with the filter paper in place.

b. Place a dry graduated cylinder under the drain tube to receive the filtrate. Close the relief valve and adjust the regulator so that a pressure of 100 ±5 pounds per square inch (690 ±35 kilopascals) is applied in 30 seconds or less. The test period begins at the time of pressure application.

c. At the end of 30 minutes, measure the volume of filtrate. Shut off the flow through the pressure regulator and open the relief valve carefully. Report the time interval, if other than 30 minutes.

d. Report the volume of filtrate in cubic centimeters (to 0.1 cubic centimeter) as the API filtrate, and the initial mud temperature in °F (°C). Save the filtrate for appropriate chemical testing.

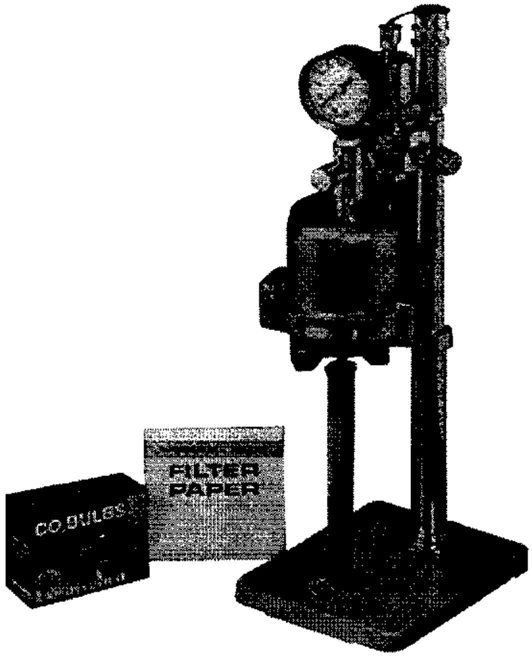
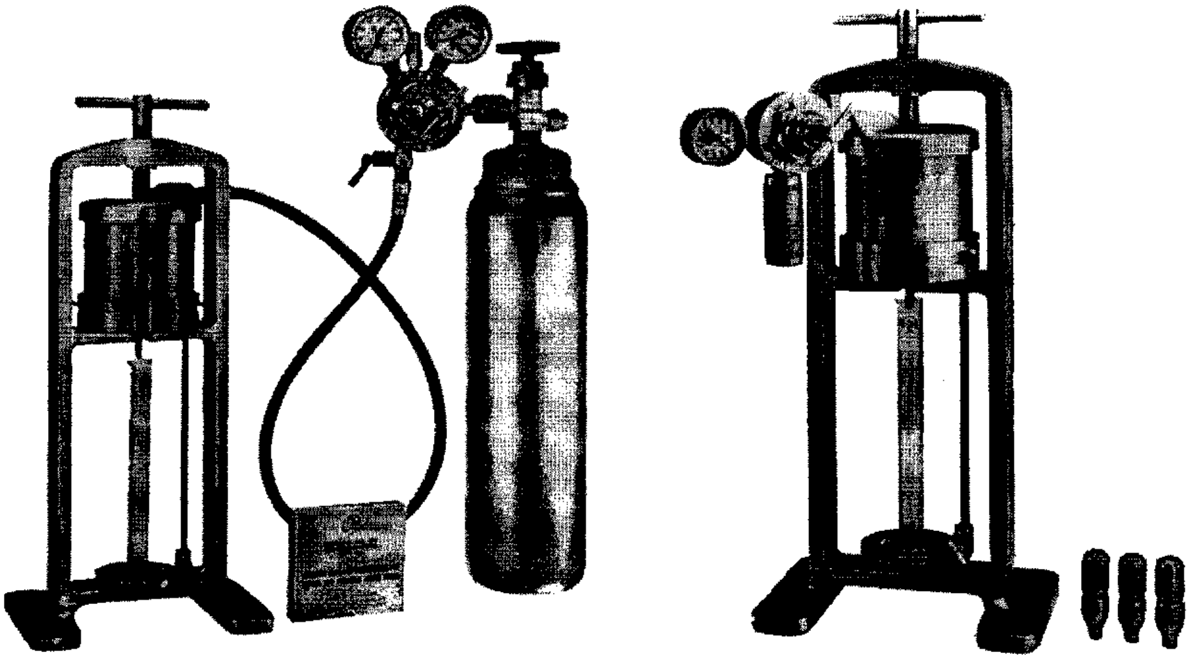


Figure 10—Filter Press With CO_2 Cartridge Pressurization



WITH NITROGEN PRESSURIZATION

WITH CO_2 CARTRIDGE PRESSURIZATION

Figure 11—Filter Press

e. Remove the cell from the frame, first making certain that all pressure has been relieved. Using extreme care to save the filter paper with a minimum of disturbance to the cake, disassemble the cell and discard the mud. Wash the filter cake on the paper with a gentle stream of water.
f. Measure and report the thickness of the filter cake to the nearest 1⁄32 inch (0.8 millimeter).
g. Although cake descriptions are subjective, one may use such notations as *hard, soft, tough, rubbery, firm,* and the like, to convey important information of cake quality.

5.3 HIGH-TEMPERATURE/HIGH-PRESSURE TEST

5.3.1 Equipment—High-Temperature/High-Pressure Test

CAUTION: All manufacturers' equipment are not capable of the same temperatures and pressures. Rigid adherence to manufacturers' recommendations as to sample volumes, temperatures, and pressures is essential. Failure to do so could result in serious injury.

The following equipment is used for the high-temperature/high-pressure test:

a. The high-temperature/high-pressure filter press: This consists of a controlled pressure source (CO_2 or nitrogen), regulators, a mud cell able to contain working pressures from 600 pounds per square inch to 1300 pounds per square inch, a system for heating the cell, a pressurized collection cell able to maintain proper back pressure (see Table 2) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. Figures 12 and 13 show available units. The mud cell has a thermometer well, oil-resistant gaskets, a support for the filter medium, and a valve on the filtrate delivery tube to control flow from the cell. It may be necessary to replace the gaskets frequently.

CAUTION: Nitrous oxide cartridges should not be used as pressure sources for high-temperature, high-pressure (HTHP) 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:
  1. Whatman No. 50 or equivalent filter paper for temperatures to 400°F (204°C).
  2. Dynalloy X-5 or equivalent porous disc for temperatures above 400°F (204°C). A new disc is required for each test.
c. Timer: 30-minute interval.
d. Thermometer: up to 500°F (260°C).
e. Graduated cylinder (TC): 25-cubic centimeter or 50-cubic centimeter.
f. High-speed mixer.

5.3.2 Procedure—Temperature to 300°F (149°C)

Follow this procedure for the high-temperature/high-pressure test to 300°F (149°C).

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain desired temperature.
b. Stir the mud sample for 10 minutes with a high-speed mixer. Pour the mud sample into the mud cell, being careful not to fill closer than 0.5 inch (13 millimeters) from the top to allow for expansion. Install the filter paper.
c. Complete the assembly of the cell and, with both top and bottom valves closed, place it in the heating jacket. Transfer the thermometer to the well in the mud cell.
d. Connect the high-pressure collection cell to the bottom valve and lock in place.
e. Connect a regulated pressure source to the top valve and collection cell, and lock in place.
f. Keeping the valves closed, adjust the top and bottom regulators to 100 pounds per square inch (690 kilopascals). Open the top valve, applying 100 pounds per square inch (690 kilopascals) to the mud. Maintain this pressure until the desired temperature is stabilized. The time of heating of the sample in the filter cell should never exceed a total of one hour.
g. When the sample reaches the selected temperature, increase the pressure of the top pressure unit to 600 pounds per square inch (4140 kilopascals) and open the bottom valve to start filtration. Collect the filtrate for 30 minutes, maintaining the selected temperature within ±5°F (±3°C). If back pressure rises above 100 pounds per square inch (690 kilopascals) during the test, cautiously reduce the pressure by drawing off a portion of the filtrate. Record the total volume, temperature, pressure, and time.
h. Correct the filtrate volume to a filter area of 7.1 square inches (4580 square millimeters). If the filter area is 3.5 square inches (2258 square millimeters), double the filtrate volume, and report that volume.
i. At the end of the test, close the top and bottom valves on the mud cell. Bleed the pressure from the regulators.

CAUTION: Pressure in the mud cell will still be approximately 500 pounds per square inch (3450 kilopascals). Keep the cell upright and cool it to room temperature before disassembling. Bleed the pressure from the cell before disassembling.

j. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off the regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from the cell contents, and open the cell. Discard the mud, and retrieve the filter cake. Wash the filter cake on the paper with a gentle stream of water.
k. Measure and report the thickness of the filter cake to the nearest 1⁄32 inch (0.8 millimeter).

Table 2—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure		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
		LIMIT of "Normal" Field Testing			
[a]350	177	135	932	160	1104
[a]400	204	247	1704	275	1898
[a]450	232	422	2912	450	3105

[a] Do not exceed equipment manufacturers' recommendations for maximum temperatures, pressures, and volumes.

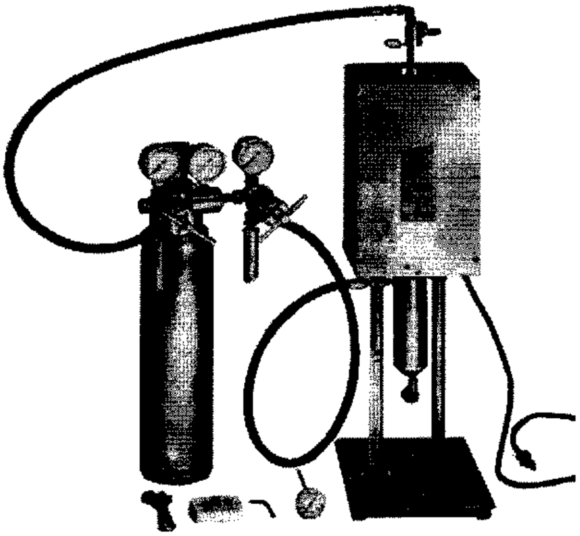


Figure 12—High-Temperature Filter Press, I

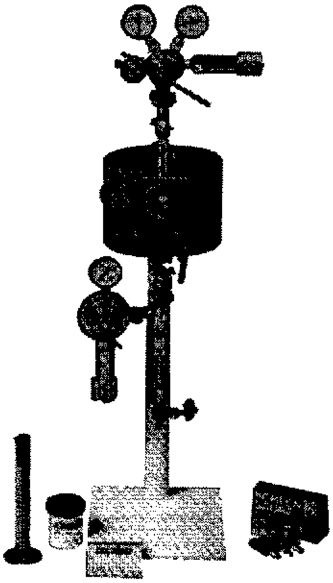


Figure 13—High-Temperature Filter Press, II

5.3.3 Procedure—Temperature Above 300°F (149°C)

CAUTION: Not all the manufacturers' equipment can be used above 300°F (149°C). Know the pressure/temperature rating of equipment in use. Failure to do so could result in serious injury.

Testing at high temperature and high pressure calls for added safety precautions. All pressure cells should be equipped with manual relief valves. Heating jackets should be equipped with both an overheat safety fuse and thermostatic cutoff. Vapor pressure of the liquid phase of muds becomes an increasingly critical design factor as test temperatures are raised. Water vapor pressures at various temperatures are shown in Table 2.

Follow this procedure for high-temperature/high-pressure tests above 300°F (149°C):

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain the correct temperature.
b. Stir the mud sample for 10 minutes with a high-speed mixer. Pour the mud sample into the mud cell, being careful not to fill the cell closer than 1.5 inches (38 millimeters) from the top to allow for expansion. Install the proper filter medium (see 5.3.1, Item b).
c. Complete the assembly of the cell, and with the top and bottom valves closed, place the mud cell in the heating jacket. Transfer the thermometer to the well in the mud cell.
d. Connect the high-pressure collection cell to the bottom valve, and lock in place.

e. Connect the regulated pressure source to the top valve and the collection cell, and lock in place.

f. With the top and bottom valves closed, apply the recommended back pressure (Table 2) for the test temperature to both top and bottom. Open the top valve, applying the same pressure to the mud while heating. Maintain this pressure until the test temperature is reached and stabilized.

g. When the temperature of the sample reaches the test temperature, increase the pressure on the top by 500 pounds per square inch (3450 kilopascals) over the back pressure being held, and open the bottom valve to begin filtration. Collect the filtrate for 30 minutes, holding the test temperature within ±5°F (±3°C) and maintaining the proper back pressure. If the back pressure should begin to rise, it can be reduced by cautiously drawing off a small portion of the filtrate. Time of heating of the sample in the filter cell should never exceed a total of one hour.

h. After the test period close both top and bottom valves on the pressure cell and bleed pressure from the regulators. Allow a minimum of 5 minutes for the filtrate to cool to avoid vaporizing, then cautiously drain and record the total volume. Also record the temperature, pressures, and time. Be sure to allow sufficient time for all the filtrate to drain from the receiver.

CAUTION: Pressure inside the filter cell will still be approximately 500 pounds per square inch (3450 kilopascals). Keep the cell upright and cool to room temperature before disassembly. Bleed pressure from cell before disassembling. Failure to do so could result in serious injury.

i. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off the regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from the cell contents and open the cell. Discard the mud, and retrieve the filter cake. Wash the filter cake on the paper with a gentle stream of water.

j. Measure and report the thickness of the filter cake to the nearest $1/32$ inch (0.8 millimeter).

6 Water, Oil, and Solids

6.1 DESCRIPTION

The retort instrument provides a means for separating and measuring the volumes of water, oil, and solids contained in a sample of water-based mud. In the retort, a known volume of a whole water mud sample is heated to vaporize the liquid components which are then condensed and collected in a graduated receiver. Liquid volumes are determined directly from reading the oil and water phases in the receiver. The total volume of solids (suspended and dissolved) is obtained by the difference (total sample volume – liquid volume). Calculations are necessary to determine the volume of suspended solids since any dissolved solids will be retained in the retort. The relative volumes of low-gravity solids and weighting material can also be calculated. Knowledge of the solids concentration and composition is considered basic to viscosity and filtration control in water-based muds.

6.2 EQUIPMENT

The following equipment is used to separate and measure the volumes of water, oil, and solids.

a. Retort instrument (Figures 14 and 15):

Retorts of two sizes (10-cubic centimeter and 20-cubic centimeter) are commonly available. Specifications of these retorts are as follows:

1. Sample cup:

Item	Sample Cup Size	
Total volume	10-cm^3	20-cm^3
Precision	±0.05 cm^3	±0.10 cm^3

2. Liquid condenser: sufficient mass to cool the oil and water vapors below their vaporization temperature prior to leaving the condenser.
3. Heating element: sufficient wattage to raise the temperature of the sample above the vaporization point of the liquid components within 15 minutes without causing solids boil-over.
4. Temperature control: a temperature control is desirable. It should be capable of limiting the temperature of the retort to 930° ±70°F (500° ±20°C).

b. Liquid receiver:

1. Graduated cylinder, or tube.
2. Material: transparent and inert to oil, water, salt solution, and temperatures up to 90°F (32°C).
3. Specifications of graduation marks:

	Graduate Size	
Precision	10-cm^3	20-cm^3
	±0.10 cm^3	±0.20 cm^3
Graduation	0.10 cm^3	0.20 cm^3
Calibration method for marks	TC (to contain at 20°C)	
Scale to read	cm^3 and/or volume percent	

c. Fine steel wool (in other words, No. 000).

Note: Liquid Steel Wool or similar products are not recommended for this application.

d. High-temperature silicone grease (used for a thread seal and a lubricant).

e. Pipe cleaners.

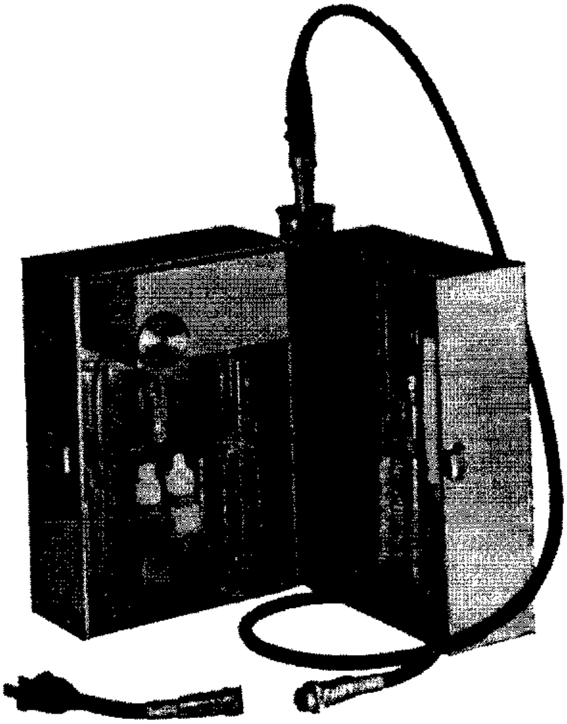


Figure 14—Retort for Liquid and Solid Content

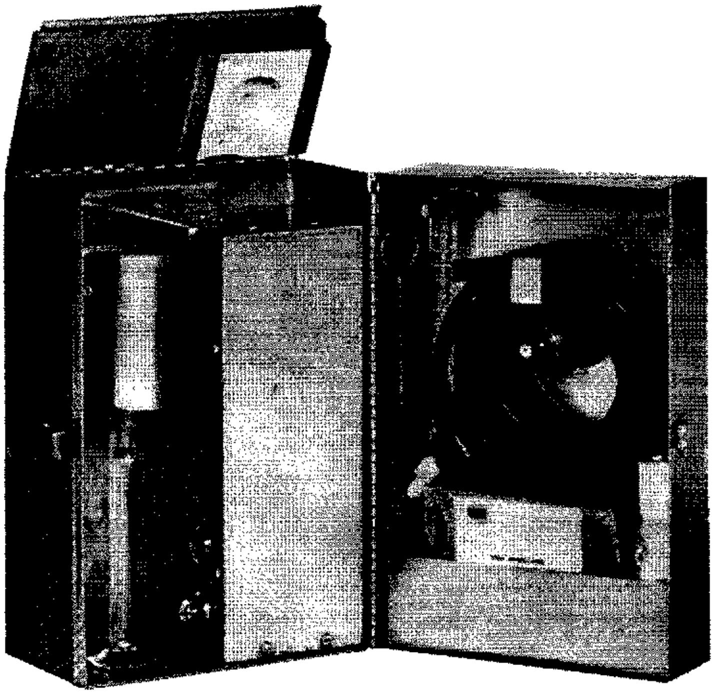


Figure 15—Retort for Liquid and Solid Content

f. Putty knife or spatula with the blade shaped to fit the inside dimensions of the sample cup of the retort.
g. Defoaming agent.

6.3 PROCEDURE

Note: This procedure will vary slightly depending on the type of retort used. See the manufacturer's instructions for the complete procedure.

Proceed as follows:

a. Be sure the retort sample cup, condenser passage, and liquid receiver are clean, dry, and cooled from previous use.
    1. The inside of the sample cup and lid must be thoroughly cleaned prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool.
    2. In addition, the condenser passage should be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency and cause erroneous liquid readings in a test.

b. Collect a representative sample of water-based mud and allow it to cool to approximately 80°F (26°C). Screen the sample through the 20-mesh screen on the marsh funnel to remove lost circulation material, large cuttings, or debris.
c. If the mud sample contains gas or air, add 2 drops to 3 drops of defoaming agent to about 300 cubic centimeters of mud, and stir slowly for 2 minutes to 3 minutes to release gases.
d. Lubricate the threads on the sample cup and condenser tube with a light coating of silicone grease. This prevents vapor loss through the threads and also facilitates disassembly of the equipment and clean-up at the end of the test.
e. Lightly pack a ring of steel wool into the chamber above the sample cup. Use only enough steel wool to prevent a boiling over of solids into the liquid receiver. (This is determined from experience.)
f. Fill the retort sample cup with the gas-free water mud described in Item c.
g. Carefully place the lid on the sample cup and allow an overflow of the sample through the hole in the lid to insure that the correct volume of the sample is in the cup.
h. With the lid held tightly in place, wipe the overflow from the sample cup and lid. Be sure that the sample cup threads are still covered with silicone grease after wiping, and that the hole in the lid is not plugged.
i. Screw the retort cup onto the retort chamber with its condenser.
j. Place a clean, dry liquid receiver under the condenser discharge tube.
k. Heat the retort and observe the liquid falling from the condenser. Continue heating for ten minutes beyond the time that no more condensate is being collected.
l. Remove the liquid receiver from the retort. Notice if solids are in the liquid that was recovered. If so, whole mud has boiled over from the sample cup and the test must be repeated.
m. Read the volumes of water and oil in the liquid receiver after it has cooled to ambient temperature. Record the volumes (or volume percentage) of water and oil collected.

6.4 CALCULATION

This calculation should be used:

a. Using the measured volumes of oil and water and the volume of the original whole mud sample (10-cubic centimeter or 20-cubic centimeter), calculate the volume percent of water, oil, and total solids in the mud as follows.

Volume percent water (V_w):

$$V_w = \frac{100\ \mathit{volume\ of\ water}, \mathrm{cm})^2}{\mathit{volume\ of\ sample}, \mathrm{cm})^3}$$

Volume percent oil (V_o):

$$V_o = \frac{100\ (\mathit{volume\ of\ water}, \mathrm{cm})^3}{\mathit{volume\ of\ sample}, \mathrm{cm}^3}$$

Volume percent retort solids (V_s):

$$V_s = 100 - (V_w + V_o)$$

Note: The volume percent retort solids above is only the difference between water plus oil, and the total sample volume (10-cubic centimeter or 20-cubic centimeter). This difference is both suspended solids (weighting material and low gravity) and dissolved materials (for example, salt). This volume percent retort solids is the suspended solids only if the mud is an untreated, freshwater mud.

b. Additional calculations are required to find the volume percent suspended solids and relate them to the relative volumes of low-gravity solids and weighting material. To make these calculations, an accurate mud weight and chloride concentration are needed.

$$V_{ss} = V_s - V_w \quad \frac{C_s}{1,680,000 - 1.21C_s}$$

Where:

V_{ss} = volume percent suspended solids.
C_s = chloride concentration, milligrams per liter.

Volume percent low-gravity solids are calculated as follows:

$$V_{lg} = \frac{1}{\rho_b - \rho_{lg}}[100\rho_f + (\rho_b - \rho_f)V_{ss} - 12W_m - (\rho_f - \rho_o)V_o]$$

Where:

V_{lg} = volume percent low-gravity solids.
W_m = mud weight, pounds per gallon.
ρ_f = density of filtrate, grams per cubic meter.

Note: ($\rho_f = 1 + 0.00000109\ C_s$) is based on sodium chloride.

ρ_b = density of weighting material, grams per cubic meter.
ρ_{lg} = density of low gravity solids, grams per cubic meter. (Use 2.6 if unknown.)
ρ_o = density of oil, grams per cubic meter. (Use 0.84 if unknown.)

Volume percent weighting material (V_b) is calculated as follows:

$$V_b = V_{ss} - V_{lg}$$

Concentration of low gravity solids, weighting material, and suspended solids can be calculated as follows:

$$C_{lg} = 3.49\ (\rho_{lg})\ (V_{lg})$$
$$C_b = 3.49\ (\rho_b)\ (V_b)$$
$$C_{ss} = C_{lg} + C_b$$

Where:

C_{lg} = low gravity solids concentration, pounds per barrel.
C_b = weighting material concentration, pounds per barrel.
C_{ss} = suspended solids concentration, pounds per barrel.

7 Sand

7.1 DESCRIPTION

The sand content of mud is the volume percent of particles larger than 74 microns. It is measured by a sand-screen set (see Figure 16).

7.2 EQUIPMENT

Use the following equipment to measure the sand content of mud:

a. 200-mesh sieve, 2.5 inches (63.5 millimeters) in diameter.
b. Funnel to fit sieve.
c. Glass-measuring tube marked for the volume of mud to be added. The tube is graduated from 0 percent to 20 percent in order to read the percentage of sand directly.

7.3 PROCEDURE

Follow this procedure to measure the sand content of mud:

a. Fill the glass measuring tube with mud to the "mud" mark. Add water to the next mark. Close the mouth of the tube and shake it vigorously.
b. Pour the mixture onto the clean, wet screen. Discard the liquid passing through the screen. Add more water to the tube. Then shake, and again pour it onto the screen. Repeat this process until the tube is clean. Wash the sand retained on the screen to free it of any remaining mud.
c. Put the funnel upside down over the top of the sieve. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass tube. Wash the sand into the tube by playing a fine spray of water through the screen. Allow the sand to settle. From the graduations on the tube, read the volume percent of the sand.
d. Report the sand content of the mud in volume percent. Report the source of the mud sample, in other words, above the shaker, suction pit, and so forth. Coarse solids other than sand will be retained on the screen (for example, lost circulation material), and the presence of such solids should be noted.

Figure 16—Sand-Content Set

8 Methylene Blue Capacity

8.1 DESCRIPTION

The methylene blue capacity of drilling fluid is an indication of the amount of reactive clays (bentonite and/or drill solids) present as determined by the methylene blue test (MBT). The methylene blue capacity provides an estimate of the total cation exchange capacity (CEC) of the drilling fluid solids. Methylene blue capacity and cation exchange capacity are not necessarily equivalent, the former normally being somewhat less than the actual cation exchange capacity.

Methylene blue solution is added to a sample of drilling fluid (which has been treated with hydrogen peroxide and acidified) until saturation is noted by formation of a dye "halo" around a drop of solids suspension placed on filter paper. Variations of the procedure used on the drilling fluid can be performed on drill solids and commercial bentonite to allow an estimate of the amount of each type of solid present in the fluid (see Recommended Practice13I).

Drilling fluids frequently contain substances in addition to reactive clays that adsorb methylene blue. Pretreatment with hydrogen peroxide (see 8.3, Item b) is intended to remove the effect of organic materials such as lignosulfonates, lignites, cellulosic polymers, polyacrylates, and the like.

8.2 EQUIPMENT

The following equipment is needed to perform the methylene blue test:

a. Methylene blue solution: 3.20 grams reagent grade methylene blue ($C_{16}H_{18}N_3SCl$)/L (1 cubic centimeter = 0.01 milliequivalent) (CAS # 61-73-4).

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

$$\textit{Weight of sample to be taken}, \text{g} = \frac{3.20}{\textit{weight of dried sample}}$$

b. Hydrogen peroxide: 3 percent solution (CAS #7722-88-5).
c. Dilute sulfuric acid: approximately 5 newtons.
d. Syringe (TD): 2.5-cubic centimeter or 3-cubic centimeter.
e. Erlenmeyer flask: 250-cubic centimeter.
f. Burette (TD): 10-cubic centimeter, micropipette: 0.5-cubic centimeter, or graduated pipette: 1-cubic centimeter.
g. Graduated cylinder (TD): 50-cubic centimeter.
h. Stirring rod.
i. Hot plate.
j. Whatman No. 1 filter paper, or equivalent.

8.3 PROCEDURE

Follow this procedure to perform the MBT:

a. Add 2.0 cubic centimeters of drilling fluid (or suitable volume of drilling fluid to require from 2 cubic centimeters–10 cubic centimeters of methylene blue solution) to 10 cubic centimeters of water in the Erlenmeyer flask. To assure that exactly 2.0 cubic centimeters are being added, use the following procedure:

1. The syringe should have a capacity of more than 2 cubic centimeters—generally 2.5 cubic centimeters or 3 cubic centimeters. By using a larger syringe, it is not necessary to remove the air trapped in the syringe.
2. The air or gas entrained in the drilling fluid must be removed (see Appendix D). Stir the drilling fluid to break the gel and quickly draw the drilling fluid into the syringe. Then, slowly discharge the syringe back into the drilling fluid, keeping the tip submerged.
3. Again, draw the drilling fluid into the syringe until the end of the plunger is at the last graduation on the syringe (for example, at the 3-cubic centimeter line on a 3-cubic centimeter syringe.)
4. Deliver 2.0 cubic centimeters of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 cubic centimeters from the last graduation on the syringe. Thus, in a 3-cubic centimeter syringe, it would be at the 1-cubic centimeter line.

b. Add 15 cubic centimeters of 3 percent hydrogen peroxide and 0.5 cubic centimeter of sulfuric acid. Boil gently for 10 minutes, but do not allow to boil to dryness. Dilute to about 50 cubic centimeters with water.

c. Add methylene blue to the flask in increments of 0.5 cubic centimeter. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous testing, larger increments (1 cubic centimeter to 2 cubic centimeters) can be used at the beginning of the titration. After each addition of methylene blue solution, swirl the contents of the flask for about 30 seconds. While the solids are still suspended, remove one drop of liquid with the stirring rod and place the drop on the filter paper. The initial endpoint of the titration is reached when dye appears as a blue or turquoise ring surrounding the dyed solids as shown in Figure 17.

d. When the blue tint spreading from the spot is detected, shake the flask an additional 2 minutes and place another drop on the filter paper. If the blue ring is again evident, the final endpoint has been reached. If the blue ring does not appear, continue as before (see Item c) until a drop taken after 2 minutes shows the blue tint.

8.4 CALCULATION

Report the methylene blue capacity (MBT) of the drilling fluid, calculated as follows:

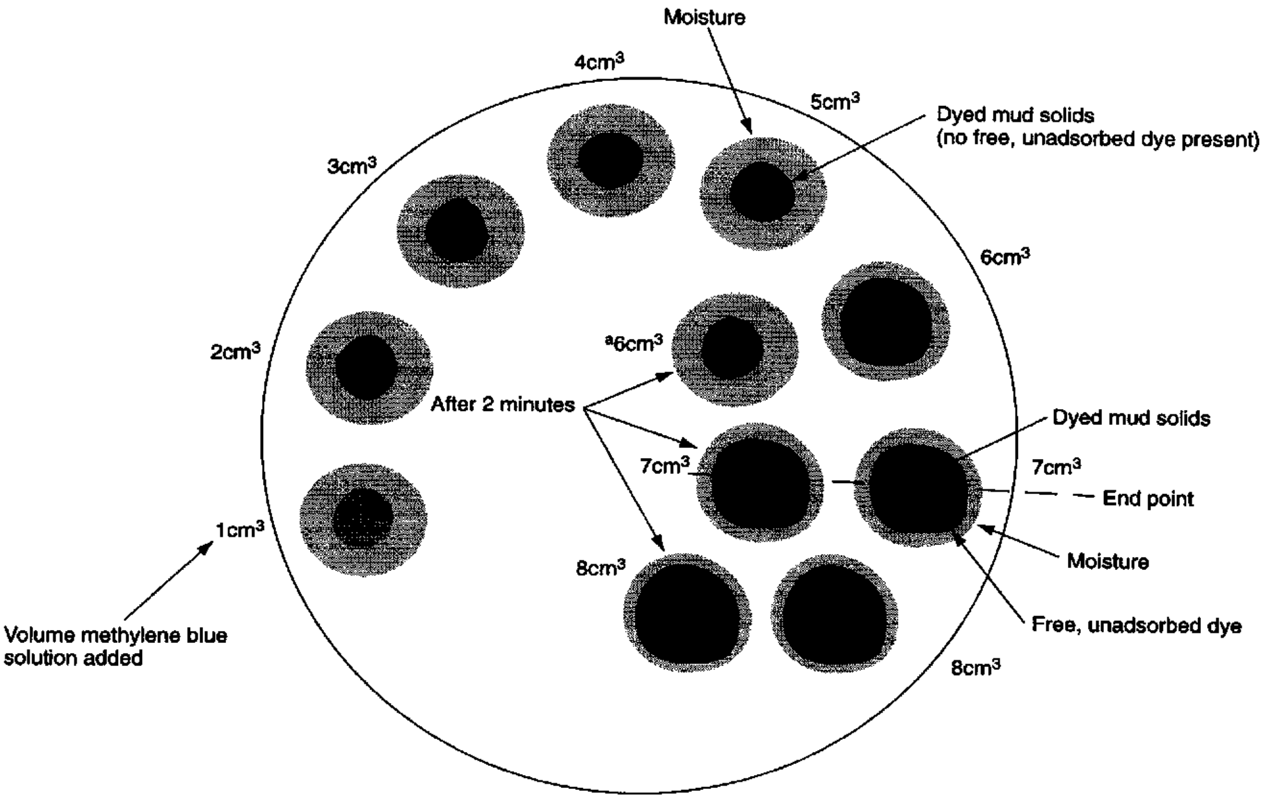
$$\begin{matrix}\textit{Methylene blue}\\ \textit{capacity}, \text{cm}^3/\text{cm}^3\end{matrix} = \frac{\textit{methylene blue}, \text{cm}^3}{\textit{drilling fluid}, \text{cm}^3}$$

Alternately, the methylene blue capacity can be reported as pounds per barrel bentonite equivalent (based on bentonite with a cation exchange capacity of 70 meq/100 grams) calculated as follows:

$$\textit{Bentonite equivalent}, \text{lb/bbl} = \frac{5\ (\textit{methylene blue}, \text{cm}^3)}{\textit{drilling fluid}, \text{cm}^3} \quad (1)$$

$$\textit{Bentonite equivalent}, \text{kg/m}^3\textit{g}, = \begin{matrix}2.85\ (\textit{bentonite}\\ \textit{equivalent}, \text{lb/bbl})\end{matrix} \quad (2)$$

Note: The pounds per barrel bentonite equivalent from (Equations 1 or 2) is not equal to the amount of commercial bentonite in the drilling fluid. Reactive clays in the drill solids contribute to this quantity as well as commercial bentonite. See Recommended Practice 13I for additional information on estimating the amount of commercial bentonite and drill solids present.



[a]Free dye detected immediately after adding sixth cm³ is adsorbed after two minutes and indicates that end point has not quite been reached.

Figure 17—Spot Tests for End Point of Methylene Blue Titration

9 pH

9.1 DESCRIPTION

Field measurement of drilling-fluid (or filtrate) pH and adjustments to the pH are fundamental to drilling fluid control. Clay interactions, solubility of various components and contaminants, and effectiveness of additives are all dependent on pH, as is the control of acidic and sulfide corrosion processes.

The term *pH* denotes the negative logarithm of the hydrogen ion, H^+, activity in aqueous solutions (activity and concentration are equal only in dilute solutions):

$$pH = -log\,[H^+]$$

For pure water at 75°F (24°C), the hydrogen ion activity $[H^+]$ is 10^{-7} moles/liter and pH = 7. This system is termed *neutral* because the hydroxyl ion activity $[OH^-]$ is also 10^{-7} moles/liter. In aqueous systems at 24°C the ion product, $[H^+] \times [OH^-]$, is 10_{-14} (a constant). Consequently, an increase in H^+ denotes a like decrease in $[OH^-]$. A change in pH of one unit indicates a ten-fold change in both $[H^+]$ and $[OH^-]$. Solutions with pH less than 7 are termed *acidic*, and those with pH greater than 7 are termed *basic* or *alkaline*.

The recommended method for pH measurement of drilling fluid is with a glass electrode pH meter. This method is accurate and gives reliable pH values, being free of interferences if a high-quality electrode system is used with a properly designed instrument. Rugged pH instruments are available that automatically temperature-compensate the slope and are preferred over the manually adjusted instruments.

Note: Color matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. These methods are reliable only in very simple water muds. Mud solids, dissolved salts and chemicals, and dark-colored liquids cause serious errors in pH paper values. Readability is normally about 0.5 pH unit.

9.2 EQUIPMENT

The following equipment is needed:

a. pH meter: millivolt range potentiometer calibrated to show pH units for measuring the potential between a glass-membrane electrode and a standard "reference" electrode. The instrument is (preferred) to be water-, shock-, and corrosion-resistant, and portable. Specifications are as follows:

1. The pH range: 0 to 14.
2. Electronics type: solid state (preferred).

3. Power source: batteries (preferred).
4. Operating temperature range: 32°F–150°F (0°C–66°C).
5. Readout: digital (preferred).
6. Resolution: 0.1 pH unit.
7. Accuracy: ±0.1 pH unit.
8. Repeatability: 0.1 pH unit.
9. Adjustments:
   (a) Temperature compensation of electrode system.
   (b) Slope of electrode system (preferred).
   (c) Calibration setting of readout. (An instrument with the preceding internal temperature compensation is preferred.)

b. Electrode system: A combination system of a glass electrode for sensing H^+ ions and a standard-voltage reference electrode, constructed as a single electrode (preferred). The body of this probe should be constructed of durable material. A flat-end probe is preferred for better protection and easier cleaning of the electrode. A waterproof connection to the meter is recommended. Specifications are as follows:

1. Glass pH electrode response range: 0-to-14 pH unit.
2. Electrodes: a glass electrode and a silver/silver chloride electrode in combination, having a ceramic or a plastic single or double junction.

Note: Use a double junction electrode for measuring liquids containing sulfide or bromide ions to avoid damaging the (silver) reference electrode system.

3. Electrolyte in reference electrode: KCl gel.
4. Glass composition suitable for low-sodium ion error.
5. Sodium ion error at pH = 13 or at 0.1 mole Na+ ion, an error less than 0.1 pH unit.

c. Buffer solutions: three solutions to calibrate and set the slope of the pH meter prior to sample measurement.

1. The pH = 4.0: potassium hydrogen phthalate at 0.05 molar in water. Gives 4.01 pH at 75°F (24°C).
2. The pH = 7.0: potassium dihydrogen phosphate at 0.02066 molar and disodium hydrogen phosphate at 0.02934 molar in water. Gives 7.00 pH at 75°F (24°C).
3. The pH = 10.0: sodium carbonate at 0.025 molar and sodium bicarbonate at 0.025 molar in water. Gives 10.01 pH at 75°F (24°C).

Note: Buffers may be obtained from supply houses as pre-made solution, dry-powder packages, or a given formula, but must duplicate National Bureau of Standards primary or secondary buffers. Shelf life of all buffers is not to exceed six months before disposal. The date of preparation of buffer should be shown on bottles used in the field. Bottles should be kept tightly stoppered.

d. Distilled or deionized water in spray bottle.
e. Soft tissues to blot electrodes.
f. Thermometer: glass, 32°F–220°F (0°C–150°C).
g. Accessory equipment:

1. Soft-bristle test tube brush to clean electrode.
2. Mild liquid detergent: Ivory®, or equivalent.
3. Electrode storage vial to keep electrodes moist.
4. Sodium hydroxide: 0.1 molar (approximately) to recondition electrode.
5. Hydrochloric acid: 0.1 molar (approximately) to recondition electrode.
6. Ammonium bifluoride: 10 percent solution (approximately) to recondition electrode.

CAUTION: This is a strong and toxic acid.

7. Hydrofluoric acid: ACS reagent grade.

CAUTION: This is a strong acid.

9.3 PROCEDURE—pH MEASUREMENT

Proceed as follows to measure pH:

a. Obtain a sample of the fluid to be tested. Allow it to reach 75 ±5°F (24 ±3°C).
b. Allow the buffer solution to also reach the same temperature as the fluid to be tested.

Note: For accurate pH measurement the test fluid, buffer solution, and reference electrode must all be at the sample temperature. The pH of the buffer solution indicated on the container label is the correct pH only at 75°F (24°C). If one is attempting to calibrate at another temperature, the actual pH of the buffer at this temperature must be used. Tables of buffer pH values at various temperatures are available from the suppliers and should be used in the calibration procedure.

c. Clean the electrodes by washing them with distilled water, and blot dry.
d. Place the probe into the pH 7.0 buffer.
e. Turn on the meter; wait 60 seconds for the reading to stabilize (See 9.4, Item a if the meter reading is not stable).
f. Measure the temperature of pH 7 buffer solution.
g. Set this temperature on the temperature knob.
h. Set the meter reading to 7.0 using the calibration knob.
i. Rinse the probe with distilled water and blot it dry.
j. Repeat the operations in 9.3, Items f through i, using either a pH 4.0 or pH 10.0 buffer. Use pH 4.0 if an acidic sample is to be tested, or pH 10.0 if an alkaline sample is to be tested. Set the meter to number 4.0 or 10.0 respectively, using the slope adjustment knob. (If no slope knob exists, use the temperature knob to set 4.0 or 10.0 on the meter.)
k. Check the meter with the pH 7 buffer again. If it has changed, reset it to 7.0 with the calibration knob. Repeat Items f through k. If the meter does not calibrate properly, recondition or replace the electrodes as given in 9.4, Item a through 9.4, Item f.

Note: Discard and do not reuse the sample of buffer solutions used in calibration. The meter should be fully calibrated every day, as in Items b through k, using two buffers. Check with the pH 7 buffer every three hours.

l. If the meter calibrates properly, rinse the electrode with distilled water, and blot it dry. Place the electrode in the sample to be tested and stir gently. Allow 60 seconds to 90 seconds for the reading to stabilize.

m. Record the sample pH to the nearest 0.1 pH unit and the temperature of the sample tested on the drilling-mud report form.
n. Carefully clean the electrode in preparation for the next usage. Store in a vial of pH 4 buffer. NEVER let the probe tip become dry.
o. Turn the meter off and close the cover to protect the instrument. Avoid storing the instrument at extreme temperatures [below 32°F (0°C) or above 120°F (49°C)].

9.4 PROCEDURE—CARE OF THE ELECTRODE

To care for the electrode, follow this procedure:

a. Cleaning the electrode will be necessary periodically, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Clean the electrode with a soft-bristle brush and a mild detergent.
b. Reconditioning the electrode may be necessary if plugging becomes severe, as indicated by slow response, drifting of readings or if the slope and calibration cannot be mutually set.
c. Recondition the electrode by soaking it for 10 minutes in 0.1 M HCl, followed by rinsing in water and soaking for 10 minutes in 0.1 M NaOH, and rinsing it again.
d. Check the electrode for response by performing a calibration (see 9.3, Items b through k).
e. If the electrode continues to perform poorly, soak it for two minutes, only, in 10 percent $NH_4F•HF$ solution. Repeat the steps in 9.3, Items b through k to check for calibration capability.

CAUTION: This is strong and toxic acid.

f. Replace the electrode system if the preceding steps fail to recondition it.

10 Chemical Analysis

10.1 ALKALINITY AND LIME CONTENT

10.1.1 Description

Alkalinity is the acid-neutralizing power of a substance. In drilling-fluid testing, alkalinity measurements can be made on either the whole mud (designated with a subscript m) or on the filtrate (designated with a subscript f). The data collected from the alkalinity test can also be used to estimate the concentrations of hydroxyl (OH^-), carbonate (CO_3^{-2}), and bicarbonate (HCO_3) ions in the drilling fluid.

Knowledge of the mud and filtrate alkalinities is important in many drilling operations to insure proper control of the mud chemistry. Mud additives, particularly some deflocculants, require an alkaline environment to function properly. In information about the source and nature of the alkalinity exists. Alkalinity arising from hydroxyl ions is generally accepted as being beneficial while alkalinities resulting from carbonates and/or bicarbonates may have adverse effects on the mud performance.

The ions that are primarily responsible for filtrate alkalinities are the hydroxyl (OH^-), carbonate (CO_3^{-2}), and bicarbonate (HCO_3^-) ions. It is important to realize that the carbonate species can change from one form to another form by changing the solution pH. The interpretation of filtrate alkalinities involves calculating differences between the titration values obtained by the following procedures. It is for this reason that special attention to accurate measurement of the various reagents is important in all steps of the procedure. In addition, it is important to realize that the following calculations are only estimates of the concentrations of the reported ionic species based on theoretical chemical equilibrium reactions.

The composition of mud filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components may be misleading. Any particular alkalinity value represents all of the ions which will react with the acid in the pH range over which that particular value was tested. Inorganic ions that may contribute to the alkalinity, in addition to the hydroxyl, carbonate, and bicarbonate ions, are as follows: borates, silicates, sulfides, and phosphates. Perhaps a more serious problem in drilling fluids and anionic organic thinners, filtrate reducers, and their degradation products may contribute to a large portion of the alkalinity value, as well as masking the endpoint color change. These organic materials make a particularly large contribution to the M_f alkalinity and thus render the test highly inaccurate in muds treated with organic thinners. However, for simple bentonite-base mud systems containing no organic thinners, the phenolphthalein (P_f) and the methyl orange (M_f) alkalinities can be used as guidelines to determine both the presence of carbonate/bicarbonate contamination and the treatment necessary to alleviate the contamination. If organic thinners are present in large amounts, the P_f/M_f test is suspect and the P_1/P_2 method should be used.

10.1.2 Equipment

The following equipment is needed:

a. Sulfuric acid solution: standardized 0.02 Normal (N/50) (CAS #7664-93-9).
b. Phenolphthalein indicator solution: 1 gram per 100 cubic centimeters of a 50 percent alcohol/water solution (CAS #518-51-4).
c. Methyl orange indicator solution: 0.1 gram per 100 cubic centimeters of water (CAS #547-58-0).
d. A pH meter (optional).

Note: A pH meter is more accurate than an indicator solution.

e. Titration vessel: 100-cubic centimeter to 150-cubic centimeter, preferably white.

f. Serological (graduated) pipettes (TD): one 1-cubic centimeter and one 10-cubic centimeter.
g. Volumetric pipette (TD): 1-cubic centimeter.
h. Hypodermic syringe (TD): 1-cubic centimeter.
i. Stirring rod.

10.1.3 Procedure—Filtrate Alkalinity: P_f, M_f

To measure filtrate alkalinity: P_f, M_f, proceed as follows:

a. Measure one or more cubic centimeters of filtrate into the titration vessel. Add two or more drops of the phenolphthalein indicator solution. If the indicator turns pink, add 0.02 normal (N/50) sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink color just disappears. If the sample is so colored that the indicator color change is masked, the endpoint can be taken when the pH drops to 8.3, as measured with a pH meter. (Refer to Section 9 for proper pH measurement.)
b. Report the phenolphthalein alkalinity of the filtrate, P_f, as the number of cubic centimeters of 0.02 normal acid required per cubic centimeter of filtrate.
c. To the sample which has been titrated to the P_f endpoint, add two or three drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the color of the indicator changes from yellow to pink. The endpoint can also be taken when the pH of the sample drops to 4.3 as measured by a pH meter. (Refer to Section 9 for proper pH measurement.)
d. Report the methyl orange alkalinity of the filtrate, M_f, as the total cubic centimeters of 0.02 normal acid per cubic meter of filtrate required to reach the methyl orange endpoint (including that amount required for the P_f endpoint).

10.1.4 Procedure—Mud Alkalinity: P_m

To measure the mud alkalinity: P_m, proceed as follows:

a. Measure 1.0 cubic centimeter of mud into the titration vessel using a syringe or volumetric pipette. Dilute the mud sample with 25 cubic centimeters to 50 cubic centimeters of distilled water. Add 4 drops to 5 drops of phenolphthalein indicator solution and, while stirring, titrate rapidly with 0.02 normal (N/50) standard sulfuric acid solution until the pink color disappears. If the endpoint color change cannot be seen, it can be taken when the pH drops to 8.3 measured by a pH meter. (Refer to Section 9 for proper pH measurement.)

Note: If cement contamination is suspected, the titration must be performed as rapidly as possible and the endpoint reported as the first disappearance of the pink color.

b. Report the phenolphthalein alkalinity of the mud, P_m, as the number of cubic centimeters of 0.02 normal (N/50) acid required per cubic centimeter of mud.

10.1.5 Calculation—P_f, M_f

The concentrations of hydroxyl, carbonate, and bicarbonate ions can be estimated as follows in Table 3:

Table 3—Concentrations, mg/L

Item	OH^-	CO_3^{-2}	HCO_3^-
$P_f = 0$	0	0	$1220M_f$
$2P_f < M_f$	0	$1200P_f$	$1220(M_f - 2P_f)$
$2P_f = M_f$	0	$1200P_f$	0
$2P_f > M_f$	$340(2P_f - M_f)$	$1200(M_f - P_f)$	0
$P_f = M_f$	$340M_f$	0	0

10.1.6 Procedure—Estimated Lime Content

To estimate the lime content proceed as follows:

a. Determine the P_f and P_m of the filtrate and mud as described in 10.1.3 and 10.1.4.
b. Determine the volume fraction of water in the mud, F_w, using the value for volume percent water from the liquid and solids determination (Section 6) in the following equation:

$$F_w = \frac{Volume\ percent\ water}{100}$$

10.1.7 Calculation—Estimated Lime Content

Report the lime content of the mud in pounds per barrel (lb/bbl) or in the SI equivalent, kilograms per cubic meter (kg/m³) from the following equations:

$$Estimated\ Lime,\ \text{lb/bbl} = 0.26\,(P_m - F_w P_f)$$

$$Estimated\ Lime,\ \text{kg/m}^3 = 0.742\,(P_m - F_w P_f)$$

10.2 ALTERNATE ALKALINITY METHOD

10.2.1 Description

The P1/P2 back-titration method was mainly developed in an attempt to overcome the limitations of the P_f/M_f alkalinity method (see 10.1.1 through 10.1.5). The P1/P2 method also has limitations. A comparison of the generally accepted advantages and disadvantages of the two alkalinity measurements are listed in Table 4.

Table 4—Filtrate Alkalinity Methods Comparison

Method	Advantages	Disadvantages
P_f/M_f	Traditional method	Interference in the M_f titration
	2 titrations, 1 sample	Bicarbonate result normally too high
P1/P2	Eliminates interference in M_f titrations	3 titrations with 3 samples Caustic measurement critical Uses a toxic material ($BaCl_2$)

Note: The total carbonate concentration in a drilling fluid can also be determined by use of the Garrett gas train as described in Appendix A.

10.2.2 Equipment—P1/P2 Alkalinity Method

The following equipment is needed for the P1/P2 alkalinity method:

a. Hydrochloric acid solution: standardized 0.02 normal (N/50) (CAS #7647-01-0).
b. Sodium hydroxide solution: 0.1 normal (N/10) (CAS #1310-73-2).
c. Barium chloride solution: 10 percent, neutralized to pH 7 with NaOH (CAS # 10361-37-2).
d. Phenolphthalein indicator solution: 1 gram per 100 cubic centimeters of a 50-percent alcohol-water solution (CAS #518-51-4).
e. Deionized water.
f. pH paper strips: 6–12 range.
g. pH meter, optional (as discussed in Section 7).
h. Titration vessel: 100-cubic centimeter to 150-cubic centimeter, preferably white.
i. Volumetric pipette (TD): one 1-cubic centimeter and one 2-cubic centimeter.
j. Burette (TD): automatic student type, 25-cubic centimeter.
k. Graduated cylinders (TC): one 25-cubic centimeter and one 5-cubic centimeter or 10-cubic centimeter.
l. Stirring rod.

10.2.3 Procedure—P1/P2 Alkalinity Method

Follow this procedure for the P1/P2 alkalinity method:

a. Determine the P_f alkalinity as described in 10.1.2, 10.1.3, Items a and b.
b. Using a volumetric pipette, measure 1.0 cubic centimeter of filtrate into the titration vessel. Add 25 cubic centimeters of deionized water to the titration vessel.
c. Using a volumetric pipette, add 2.0 cubic centimeters of 0.1 normal (N/10) sodium hydroxide solution and stir well. Measure the pH with the high-range pH paper (or pH meter). If the pH is 11.4 or greater, proceed to 10.1.10, Item d. If the pH is less than 11.4 add 2.0 cubic centimeters more of 0.1 normal sodium hydroxide solution; then proceed to 10.1.10, Item d.

Note: Exact measurement of the sodium hydroxide is necessary to avoid serious errors.

d. Using the small graduated cylinder, measure 3 cubic centimeters of barium chloride and add to the titration vessel. Add 2 drops to 4 drops of phenolphthalein indicator solution while stirring.

CAUTION: Do not use your mouth to pipette the barium chloride solution. *It is extremely poisonous.*

e. Immediately titrate the mixture with the standard 0.02 normal hydrochloric acid to the first disappearance of the pink color (or to a pH of 8.3 with a pH meter). The color may reappear after a short time: do not continue the titration.
f. Report the alternate alkalinity, P1, as the cubic centimeter of 0.02 normal acid to reach the phenolphthalein endpoint.
g. Determine the blank alkalinity, P2. Omit the filtrate, but otherwise repeat the procedure described in Items b through f for determining P1, using *exactly* the same quantities of water and reagents in preparing the sample.
h. Report the blank alkalinity, P2, as the cubic centimeter of 0.02 normal acid needed to titrate the reagent mixture to the phenolphthalein endpoint.

10.2.4 Calculation—P1/P2 Alkalinity Method

The procedure given in the previous paragraphs is intended to reduce the major interferences in the P_f/M_f alkalinity test, and thus provide a better estimate of hydroxyl, carbonate, and bicarbonate concentrations. Calculating these concentrations does not make them true values; the composition is theoretical, based on water chemistry carbonate equilibriums.

Within the limitations outlined, the various ionic concentrations can be calculated as follows in milligrams per liter (mg/L):

When P1 > P2

$$\text{OH}^-,\ \text{mg/L} = 340\ (\text{P1} - \text{P2})$$
$$\text{CO}_3^{-2},\ \text{mg/L} = 1200\ [P_f - (\text{P1} - \text{P2})]$$

When P1 < P2

$$\text{HCO}_3^-,\ \text{mg/L} = 1220\ (\text{P2} - \text{P1})$$
$$\text{CO}_3^{-2},\ \text{mg/L} = 1200\ P_f$$

10.3 CHLORIDE

10.3.1 Description

The chloride test measures the chloride-ion concentration in the mud filtrate.

10.3.2 Equipment

The following equipment is needed to perform the chloride test:

a. Silver nitrate solution containing 4.7910 grams per liter (g/L) (equivalent to 0.001 grams chloride-ion/cubic centimeters), stored in an amber or opaque bottle (CAS #7761-88-8).
b. Potassium chromate indicator solution: 5 grams per 100 cubic centimeters of water (CAS #7789-00-6).
c. Sulfuric or nitric acid solution: standardized 0.02 normal (N/50) (Sulfuric acid CAS #7664-93-9 or nitric acid CAS #7697-37-2).
d. Phenophthalein indicator solution: 1 gram per 100 cubic centimeters of 50 percent alcohol/water solution (CAS #518-51-4).
e. Calcium carbonate: precipitated, chemically pure grade (CAS #471-34-1).
f. Distilled water.

g. Serological (graduated) pipettes (TD): one 1-cubic centimeter and one 10-cubic centimeter.
h. Titration vessel: 100 cubic centimeter to 150-cubic centimeter, preferably white.
i. Stirring rod.

10.3.3 Procedure

To perform the chloride test, proceed as follows:

a. Measure one or more cubic centimeters of filtrate into the titration vessel. Add 2 drops to 3 drops of phenolphthalein solution. If the indicator turns pink, add acid drop-by-drop from pipette, while stirring, until the color is discharged. If the filtrate is deeply colored, add an additional 2 cubic centimeters of 0.02 normal (N/50) sulfuric or nitric acid and stir. Then add 1 gram of calcium carbonate and stir.
b. Add 25 cubic centimeters to 50 cubic centimeters of distilled water and 5 drops to 10 drops of potassium chromate solution. Stir continuously while adding standard silver nitrate solution drop-by-drop from the pipette until the color changes from yellow to orange-red and persists for 30 seconds. Record the number of cubic centimeters of silver nitrate solution required to reach the endpoint. If over 10 cubic centimeters of silver nitrate solution are used, repeat the test with a smaller sample of filtrate.

Note: If the chloride-ion concentration of the filtrate exceeds 10,000 milligrams per liter, a silver nitrate solution equivalent to 0.01 gram chloride-ion per cubic centimeter may be used. The factor 1,000 in Equation 3 is then changed to 10,000.

10.3.4 Calculation

Report the chloride-ion concentration of the filtrate in milligrams per liter, calculated as follows:

$$Chloride\text{, mg/L} = \frac{1000\ (silver\ nitrate\text{, cm}^3)}{filtrate\ sample\text{, cm}^3} \qquad (3)$$

To convert units:

$$Chloride\text{, ppm} = \frac{Chloride\text{, mg/L}}{Specific\ Gravity\ of\ Filtrate}$$

$$Salt\ \text{(NaCl), mg/L} = (1.65)\ Chloride\text{, mg/L}$$

Refer to Table 1 for filtrate specific gravity.

Refer to Table 5 for conversion of the percent of salt in salt water to milligrams per liter or parts per million.

10.4 TOTAL HARDNESS AS CALCIUM

10.4.1 Description

The hardness of water or mud filtrate is due primarily to the presence of calcium and magnesium ions. When EDTA (ethylenediaminetetraacetic acid or its salt) is added to the water, it combines both with the calcium and magnesium, and the endpoint is determined with a suitable indicator. The total hardness of the water is expressed as milligrams per liter calcium. An endpoint obscured by dark components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

Table 5—Conversion of Milligrams per Liter Chloride to Weight Percent Salt (NaCl) or Parts per Million Salt Solution Temperature at 68°F (20°C)

Milligrams per Liter Chloride	Weight Percent Salt	Milligrams per Liter Salt	Parts per Million Salt
3,040	0.5	5,020	5,000
6,100	1	10,050	10,000
12,300	2	20,250	20,000
18,600	3	30,700	30,000
24,900	4	41,100	40,000
31,599	5	52,000	50,000
37,900	6	62,500	60,000
44,200	7	73,000	70,000
51,200	8	84,500	80,000
57,600	9	95,000	90,000
64,900	10	107,100	100,000
71,800	11	118,500	110,000
79,000	12	130,300	120,000
86,100	13	142,000	130,000
93,400	14	154,100	140,000
100,900	15	166,500	150,000
108,200	16	178,600	160,000
115,800	17	191,000	170,000
123,500	18	203,700	180,000
131,200	19	216,500	190,000
139,200	20	229,600	200,000
147,300	21	256,100	210,000
155,200	22	256,100	220,000
163,600	23	270,000	230,000
169,400	24	279,500	240,000
171,700	25	283,300	250,000
188,700	26	311,300	260,000

Note: It is quite common for many laboratories analyzing water samples to report milligrams per liter of salt as parts per million. If it is known that an analysis is reported in this way, the milligram per liter salt column should be used in converting to percent salt rather than the parts per million salt column.

10.4.2 Equipment

The following equipment is needed:

a. EDTA solution (Versenate): 0.01 molar: disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cubic centimeter = 1000 milligrams per liter $CaCO_3$, 1 cubic centimeter = 400 milligrams per liter Ca^{+2}) (CAS #139-33-3).
b. Buffer solution: 67.5 grams ammonium chloride (CAS #12125-02-9) and 570 cubic centimeters ammonium hydroxide (CAS #1336-21-6) (15N) diluted to 1000 cubic centimeters with distilled water.
c. Hardness indicator solution: 1 gram per liter Calmagite® or equivalent, 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid in distilled water (CAS #3147-14-6).

d. Acetic acid: glacial (CAS #64-19-7).

CAUTION: Avoid skin contact.

e. Titration vessel: 150-cubic centimeter beaker.
f. Serological (graduated) pipettes (TD): one 5-cubic centimeter and one 10-cubic centimeter.
g. Volumetric pipettes (TD): one 1-cubic centimeter, one 2-cubic centimeter and one 5-cubic centimeter.
h. Hot plate (required if the filtrate is colored).
i. Masking agent: 1:1:2 mixture by volume of triethanolamine: tetraethylenepentamine: water (optional).
j. pH paper strip.
k. Sodium hypochlorite solution: 5.25 percent sodium hypochlorite in deionized water (in other words, Clorox® or equivalent).

CAUTION: Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure the sodium hypochlorite is fresh as it will deteriorate with time.

l. Deionized or distilled water.

Note: The deionized water and sodium hypochlorite solution should be tested for hardness by using 50.0 cubic centimeters of the deionized water in 10.4.3, Item f, and 10 cubic centimeters of the hypochlorite in 10.4.3, Item b, without the test sample, and continuing with Items g and h. If the procedure is then repeated with the test sample utilizing 50.0 cubic centimeters of the deionized water and 10 cubic centimeters hypochlorite in 10.4.3, Items f, g, and h, the hardness of the test sample can be determined by subtracting the hardness of the deionized water and hypochlorite.

10.4.3 Procedure

To measure total hardness of water, proceed as follows:

a. Measure 1.0 cubic centimeters or more of sample into a 150-cubic centimeter beaker. (If the filtrate is clear, or is only lightly colored, omit the following steps b through e).
b. Add 10 cubic centimeters hypochlorite (Clorox® or equivalent), and mix.
c. Add 1 cubic centimeter glacial acetic acid, and mix.
d. Boil the sample for five minutes. Maintain the sample volume by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. Verify the absence of chlorine by immersing a strip of pH paper in the sample. If the paper is bleached white, continue boiling.

CAUTION: Work in an adequately ventilated area.

e. Cool the sample and wash the sides of the beaker with deionized water.
f. Dilute the sample to 50 cubic centimeters with deionized water. Add about 2 cubic centimeters of hardness buffer and swirl to mix.

Note: The presence of soluble iron may interfere with the endpoint determination. If this is suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) has proven to be a suitable masking agent. One cubic centimeter of the mixture is used per titration.

g. Add sufficient hardness indicator (2 drops to 6 drops) and mix. A wine-red color will develop if calcium and/or magnesium is present.
h. While stirring, titrate with EDTA solution to the proper endpoint. Calcium indicators will produce a red-to-blue change. The endpoint is best described as that point where additional EDTA produces no further red-to-blue color change. The EDTA volume will be used in the calculation in 10.4.4.

10.4.4 Calculation

The following calculation should be used to determine total hardness as calcium:

$$\textit{Total hardness as calcium}, \text{ mg/L} = \frac{400(\,\textit{EDTA volume}, \text{ cm}^3)}{\textit{volume sample}, \text{ cm}^3}$$

APPENDIX A—CHEMICAL ANALYSIS OF WATER-BASED DRILLING FLUIDS

A.1 Calcium

A.1.1 DESCRIPTION

When EDTA (ethylenediaminetetraacetic acid or its salt) is added to water or mud filtrate containing both calcium and magnesium, the EDTA combines first with calcium. Calcium can be determined with EDTA when the pH of the sample is sufficiently high that magnesium is precipitated as the hydroxide, and when an indicator specific for calcium is used. Several indicators will give color changes when all of the calcium has been complexed by EDTA at a pH of 12–13. An end point obscured by dark organic components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

A.1.2 EQUIPMENT

The following equipment is needed to measure calcium:

a. EDTA solution (Versenate) (CAS #139-33-3): 0.01 molar, EDTA: disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cubic centimeter = 1000 miligrams per liter $CaCO_3$, 1 cubic centimeter = 400 milligrams per liter calcium).
b. Calcium buffer solution: 1N sodium hydroxide (NaOH) (CAS #1310-73-2).
c. Calcium indicator: Calver® II or hydroxy naphthol blue (CAS #63451-35-4).
d. Acetic acid: glacial (CAS #64-19-7).

CAUTION: Avoid skin contact.

e. Titration vessel: 150-cubic centimeter beaker.
f. Serological (graduated) pipettes (TD): two 10-cubic centimeter and one 1-cubic centimeter.
g. Volumetric pipettes (TD): one 1-cubic centimeter, one 2-cubic centimeter, and one 5-cubic centimeter.
h. Hot plate (required if the filtrate is colored).
i. Masking agent: 1:1:2 mixture by volume of triethanolamine: tetraethylenepentamine: deionized water (optional).
j. pH paper.
k. Graduated cylinder (TC): 50-cubic centimeter.
l. Sodium hypochlorite solution: 5.25 percent sodium hypochlorite in deionized water (i.e., Clorox® or equivalent).

CAUTION: Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure the sodium hypochlorite is fresh as it will deteriorate with time.

m. Deionized or distilled water.

Note: The deionized water and sodium hypochlorite solution should be tested for calcium by using 50.0 cubic centimeters of the deionized water as described in A.1.3, Item f and 10 cubic centimeters of the hypochlorite noted in A.1.3, Item b, without the test sample, and continuing with the steps in A.1.3, Items g and h. If the procedure is then repeated with the test sample utilizing 50.0 cubic centimeters of the deionized water and 10 cubic centimeters of the hypochlorite noted in A.1.3, Items f, g, and h, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and hypochlorite.

A.1.3 PROCEDURE

Proceed as follows to measure calcium:

a. With a volumetric pipette, add 1.0 cubic centimeter or more of the sample to a 150-cubic centimeter beaker. This sample volume will be used in the calculation shown in A.1.4. If the filtrate is colorless or is only slightly colored, omit steps A.1.3, Items b through e.
b. With serological pipette, add 10 cubic centimeters hypochlorite solution and mix.
c. With serological pipette, add 1 cubic centimeter glacial acetic acid and mix.
d. Boil the sample for five minutes. Maintain the sample by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required. A sufficiently boiled sample will show a pH of 5.0.

CAUTION: Work in an adequately ventilated area.

e. Cool the sample and wash the sides of the beaker with deionized water.
f. Dilute the sample to approximately 50 cubic centimeters with deionized water. Add 10 cubic centimeters to 15 cubic centimeters of NaOH buffer solution, or sufficient NaOH to produce a pH of 12–13.

Note: The presence of soluble iron may interfere with the endpoint determination. If this is suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) is a suitable masking agent. Add 1.0 cubic centimeters of the mixture after A.1.3, Item f.

g. Add sufficient calcium indicator (0.1 grams to 0.2 grams) to produce a pink to wine-red color if calcium is present. Too much indicator will obscure the endpoint.

Note: The addition of several drops of methyl orange along with the calcium indicator may improve the visibility of the endpoint.

h. While stirring, titrate with standard EDTA to the proper endpoint. Calcium indicators will produce a red-to-blue change. The endpoint is best described as that point where additional EDTA produces no further red-to-blue color change. The EDTA volume will be used in the calculation in. A.1.4.

A.1.4 CALCULATION

$$\mathit{Calcium}, \text{mg/L} = \frac{400\ (\mathit{EDTA\ volume}, \text{cm}^3)}{\mathit{volume\ sample}, \text{cm}^3}$$

A.2 Magnesium

A.2.1 DESCRIPTION

The magnesium content of the mud filtrate can be calculated by subtracting the calcium ion content from the total hardness. This gives the magnesium content in terms of calcium, which is converted to magnesium by multiplying the value by the ratio of atomic weights (24.3/40 = 0.6).

A.2.2 PROCEDURE

Proceed as follows:

a. Determine the total hardness as calcium (see 10.4.3 through 10.4.4).
b. Determine the calcium content (see A.1.1 through A.1.4).

A.2.3 CALCULATION

$$\mathit{Magnesium}, \text{mg/L} = (0.6)\ (\mathit{Total\ Hardness}, \text{mg/L} - \mathit{Calcium}, \text{mg/L})$$

A.3 Calcium Sulfate

A.3.1 DESCRIPTION

The calcium sulfate content of mud is determined by using the EDTA method as described in A.1.1 through A.1.4 to determine the total calcium in a mud filtrate and the whole mud. The total and undissolved calcium sulfate contents of the mud can then be calculated.

A.3.2 EQUIPMENT

The following equipment is needed to test for calcium sulfate:

a. EDTA solution (Versenate): 0.01 molar EDTA: disodium (ethylenediamine) tetraacetate dihydrate standardized, (1 cubic centimeter = 1000 milligrams per liter $CaCO_3$, 1 cubic centimeter = 400 milligrams per liter Ca^{-2}) (CAS #139-33-3).
b. Buffer solution: 1N sodium hydroxide (NaOH) (CAS #1310-73-2).
c. Calcium indicator: Calver® II or equivalent, or hydroxy naphthol blue (CAS #63451-35-44).
d. Acetic acid: glacial (CAS #64-19-7).

CAUTION: Avoid skin contact.

e. Titration vessel: 150-cubic centimeter beaker.
f. Serological (graduated) pipettes (TD): two 10-cubic centimeter and one 1-cubic centimeter.
g. Volumetric pipettes (TD): one 1-cubic centimeter, one 2-cubic centimeter, one 5-cubic centimeter, one 10-cubic centimeter.
h. Hot plate (required, if filtrate is colored).
i. Masking agent: 1:1:2 mixture by volume of triethanolamine:tetraethylenepentamine:deionized water (optional).
j. pH paper.
k. Graduated cylinder (TC): 50-cubic centimeter.
l. Mud retort, as described in Section 10.
m. Sodium hypochlorite solution: 5.25 percent sodium hypochlorite in deionized water (in other words, Clorox® or equivalent).

CAUTION: Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure the sodium hypochlorite is fresh as it will deteriorate with time.

n. Deionized or distilled water.

Note: The deionized water and sodium hypochlorite solution should be tested for calcium by using 10.0 cubic centimeters of the deionized water mentioned in A.3.3, Item a, and 10 cubic centimeters of the hypochlorite also listed in A.3.3, Item a, without the test sample, and continuing with Items a through Items c. If the procedure is then repeated with the test sample utilizing 10.0 cubic centimeters of the deionized water and 10 cubic centimeters of the sodium hypochlorite listed in A.3.3, Items a, b, and c, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and sodium hypochlorite.

A.3.3 PROCEDURE

This procedure should be followed to determine calcium sulfate:

a. Add 5 cubic centimeters of whole mud to 245 cubic centimeters of deionized water. Stir the mixture for 15 minutes and filter it through a standard API filter press. Collect only clear filtrate. Into a 150-cubic centimeter beaker, add 10 cubic centimeters of clear filtrate with the 10-cubic centimeter volumetric pipette and titrate to the EDTA endpoint as described in A.1.1 through A.1.4. Call this volume of EDTA V_t.
b. Titrate 1 cubic centimeter of the original mud filtrate (obtained as described in 5.1 through 5.3) to the EDTA endpoint. Call this volume of EDTA V_f.
c. Retort the mud. Determine the volume fraction of water in the mud, F_w, by using the value for volume percent water from the liquid and solids determination and the following equation:

$$F_w = \frac{\mathit{Volume\ percent\ water}}{100}$$

A.3.4 CALCULATION

The calcium sulfate content of the mud in pounds per barrel is calculated by using the following equation:

$$\mathit{Total\ calcium\ sulfate}, \text{lb/bbl} = 2.38(V_t)$$

The (excess) undissolved calcium sulfate content of the mud in pounds per barrel may be calculated by using the subsequent equation:

$$\mathit{Excess\ calcium\ sulfate}, \text{lb/bbl} = 2.38(V_t) - 0.48(V_f F_w)$$

A.4 Formaldehyde

A.4.1 DESCRIPTION

This procedure is for the determination of the paraformaldehyde content of the drilling fluid. Sodium sulfite is reacted with a filtrate sample (neutralized to a phenolphthalein endpoint) which is then titrated with the acid to the phenolphthalein endpoint again. A blank must be run to subtract the contribution to alkalinity attributable to sodium sulfite. The difference in two titrations is the amount of paraformaldehyde present in the drilling fluid.

A.4.2 EQUIPMENT

The following equipment is needed:

a. Phenolphthalein indicator: 1 gram per 100 cubic centimeters of 50 percent alcohol/water (CAS #518-51-4).
b. Sodium hydroxide solution: 0.02 normal (N/50) (CAS #1310-73-2).
c. Sulfuric acid solution: 0.02 normal (N/50) (CAS #7664-93-9).
d. Sodium sulfite solution: 4 grams per 100 cubic centimeters of distilled water (CAS #7757-83-7).

Note: This solution deteriorates rapidly. If older than 30 days, it should be replaced with a fresh solution.

e. Titration vessel: a test tube or a casserole dish.
f. Serological (graduated) pipette (TD): 10-cubic centimeter.
g. Volumetric pipettes (TD): one 1-cubic centimeter and one 3-cubic centimeter.

A.4.3 PROCEDURE

Follow this procedure:

a. Pipette 3 cubic centimeters of the mud filtrate into the casserole or test tube. Add 2 drops of phenolphthalein indicator solution. If the sample remains colorless, add sodium hydroxide solution drop-by-drop, with agitation, until a faint pink color develops. Then add sulfuric acid drop-by-drop to dispel the color.
b. If, upon the first addition of phenolphthalein, the filtrate becomes colored, add sulfuric acid dropwise until the color is just dispelled.
c. To the neutralized filtrate, add 1 cubic centimeter of sodium sulfite solution; a red color will develop.
d. After approximately 30 seconds, titrate with sulfuric acid until the sample is a very faint pink. Record the amount of acid used in cubic centimeters. This is the amount of sulfuric acid which will be used for the calculation, and noted as V_f.
e. Repeat the foregoing steps in Items c through d using distilled water instead of mud filtrate. Record the amount of acid used in cubic centimeters. This will be used for the calculation in A.4.4, and noted as V_b.

A.4.4 CALCULATION

Formaldehyde content, lb/bbl = $(0.07)(V_f\ V_b)$

A.5 Sulfide

A.5.1 DESCRIPTION

The concentration of soluble sulfides in a drilling fluid can be determined by this method. Soluble sulfides include H_2S and the sulfide (S^{-2}) and bisulfide (HS^-) ions. Mud filtrate is acidified in a Garrett gas train, converting all sulfides to H_2S, which is evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is passed through a Dräger tube which responds to H_2S by darkening along its length. The darkened length is proportional to the total sulfide in the mud filtrate. The low-range Dräger tube turns from white to brownish-black, and the high-range Dräger tube turns from pale blue to jet-black. No common mud contaminant will cause these color changes.

Lead-acetate paper disks can be accommodated in the Garrett gas train to determine the presence or absence of sulfide. If the presence of sulfide is indicated by a darkening of the lead-acetate paper, a Dräger tube should be used for quantitative analysis.

A.5.2 EQUIPMENT

The following equipment is needed:

a. The Garrett gas train apparatus, as shown in Figure A-1, consists of the following: a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter, and a Dräger tube. Specifications of the Garrett gas train is as follows:

Body:	
Chamber 1:	
Depth	3.54 inches (90 millimeters)
Diameter	1.52 inches (38 millimeters)
Chambers 2 & 3:	
Depth	3.54 inches (90 millimeters)
Diameter	1.18 inches (30 milligrams)
Passages between chambers:	
Diameter	0.08 inch (2.0 millimeters)
Material:	
Lucite® or equivalent transparent material or glass that is inert to acid, sulfides, and hydrogen sulfide gas.	
Dispersion Tube:	
Stem:	
Diameter	0.315 inch (8.0 millimeters)
Length	approximately 5.9 inches (150 millimeters)
Dispersion frit: (bell-shaped ASTM 4-5.5 fine):	
Diameter	1.18 inches (30 millimeters)
Pyrex® or equivalent glass.	

Flow meter: floating ball-type preferred, capable of measuring 300 cubic centimeters per minute of CO_2 gas.

Flexible tubing: type that is inert to hydrogen sulfide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and rigid tubing: type that is inert to hydrogen sulfide and acid.

b. Carrier gas: type that is inert to hydrogen sulfide, acid, and Dräger tube reagents. Nitrogen is preferred, but carbon dioxide is acceptable. (Avoid air or other oxygen-containing gases.)

c. Dräger H_2S analysis tubes:

1. Low range: marked H_2S 100/a (No. CH-291-01).
2. High range: marked H_2S 0.2 percent/A (No. CH-281-01).

d. Lead-acetate paper disk (see the Note on A.5.3, Item *p*).

e. Sulfuric acid (CAS #7664-93-9): approximately 5N, reagent grade.

f. Defoamer in a dropper bottle.

g. Hypodermic syringes: one 10-cubic centimeter (for acid), and one 2.5-cubic centimeter, one 5-cubic centimeter and one 10-cubic centimeter (for a sample).

h. Hypodermic needles: two 1.5-inch (38-millimeter) 21-gauge needles.

A.5.3 PROCEDURE

Follow this procedure:

a. Be sure the gas train is clean and dry and on a level surface, with the top removed.

Note: Moisture in the train can cause the ball in the flowmeter to float erratically and may affect the accuracy of the Dräger tube reading.

b. Add 20 cubic centimeters of deionized water to Chamber 1.

c. Add 5 drops defoamer to Chamber 1.

d. See Table A-1 for the sample volume and type of Dräger tube required for the expected sulfide range. Select the proper type Dräger tube. Break the tip from each end of the tube.

e. Install the Dräger tube with the arrow pointing downward into the bored receptacle. Likewise, install the flowmeter tube with the word TOP upward. Be sure the o-rings seal around the body of each tube.

f. Install the top on the gas train and hand-tighten all screws evenly to seal the o-rings.

g. With the regulator backed off, connect the carrier gas to the dispersion tube of Chamber 1 using flexible tubing. If a CO_2 cartridge is used, install and puncture the cartridge and connect it to the dispersion tube as shown in Figure A-1.

h. Attach the flexible tubing from Chamber 3 outlet to the Dräger tube, as shown in Figure A-1.

Note: Use only latex rubber or inert plastic tubing. Do not clamp the flexible tubing; unclamped tubing provides pressure relief in the event of over pressurization.

i. Adjust the dispersion tube in Chamber 1 to approximately ¼ inch (5 millimeters) above the bottom.

j. Gently flow the carrier gas for a 30-second period to purge air from the system. Check for leaks. Shut off the carrier gas.

k. Collect a sufficient volume of solids-free filtrate for analysis. (If a low concentration of soluble sulfides is to be detected, a large volume of filtrate is required. Use Table A-1 as a guide.)

l. Inject a measured volume of the solids-free filtrate sample into Chamber 1 through the rubber septum, using a hypodermic syringe and needle.

m. Slowly inject 10 cubic centimeters of sulfuric acid solution into Chamber 1 through the rubber septum using the hypodermic syringe and needle.

n. Immediately restart the carrier gas flow. The flow rate should be maintained between 200 cubic centimeters and 400 cubic centimeters per minute.

Note: One CO_2 cartridge should provide about 15 minutes to 20 minutes of flow at this rate.

o. Observe changes in the appearance of the Dräger tube. Note and record the maximum darkened length (in units marked on the tube) before the front starts to smear. Continue flowing for a total of 15 minutes even though the front may attain a diffuse and feathery coloration. In the high-range tube, an orange color (caused by SO_2) may appear ahead of the black front if sulfites are present in the sample. The orange SO_2 region should be ignored when recording darkened length.

Note: For best Dräger-tube accuracy, the darkened length should fill more than half the tube's length; therefore, the filtrate sample volume must be carefully selected.

p. A lead-acetate paper disk fitted under the o-ring of Chamber 3 can be substituted for the Dräger tube in the gas train. The lead-acetate paper will show qualitatively the presence or absence of sulfides in the sample. A dark discoloration of the paper is a positive indication of sulfides. After the positive indication, the Dräger tube should be used on a separate sample for quantitative analysis.

q. To clean the gas train, remove the flexible tubing and remove the top. Take the Dräger tube and flow-meter out of the receptacles, and plug the holes with stoppers to keep them dry. Wash out the chambers with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passages between the chambers. Wash, rinse, and blow-out the dispersion tube with a dry gas. Rinse the unit with deionized water and allow it to drain dry.

A.5.4 CALCULATION

Using the measured sample volume, the Dräger tube's maximum darkened length, and the tube factor from Table A-1, calculate the sulfide in the sample:

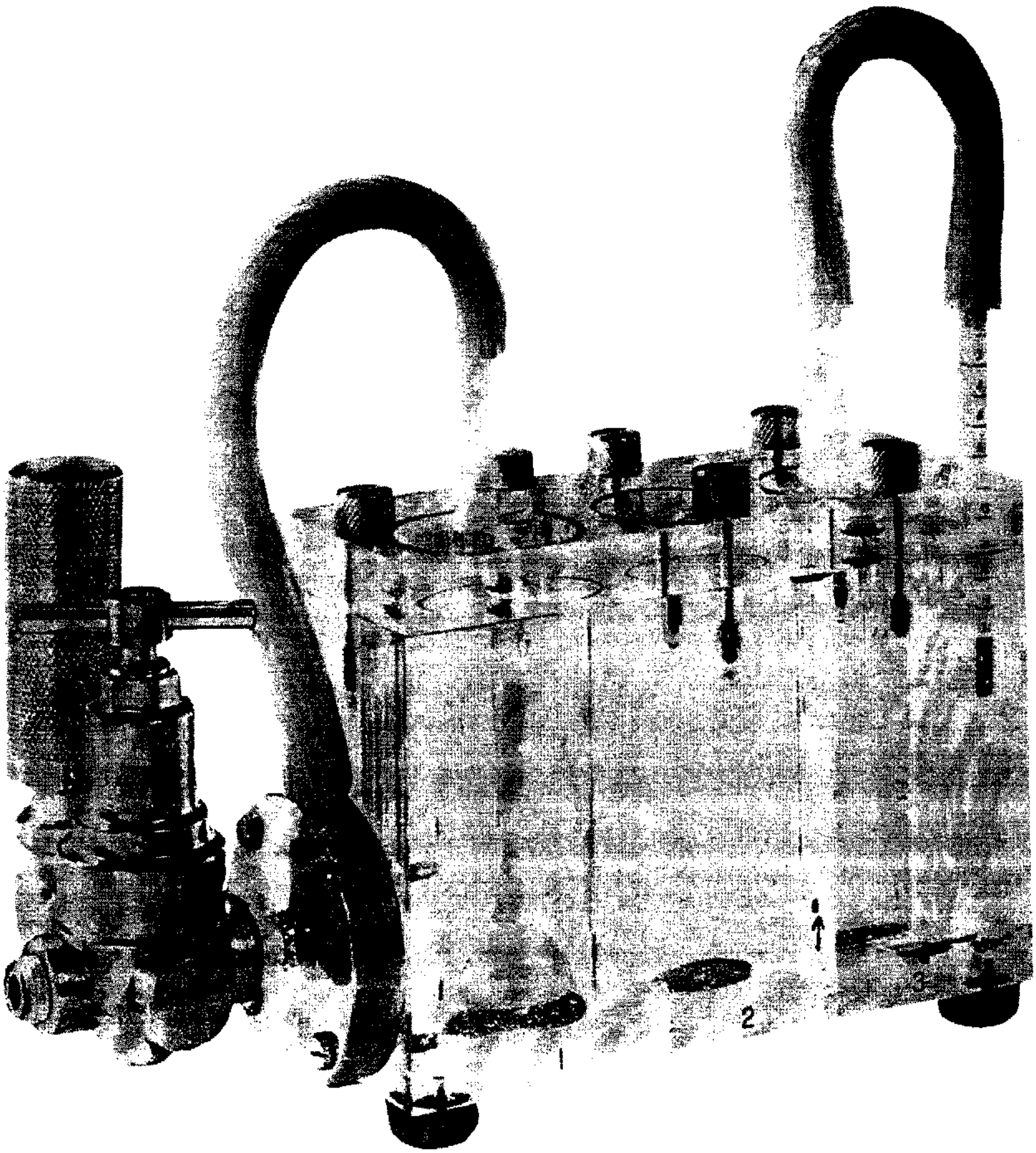


Figure A-1—Analysis of Soluble Sulfides

Table A-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]
2.4–48	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]

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[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 percent/A (Catalog No. CH-281-01), with a scale from 0.2 to 7.0. For old tubes with 1–17 cubic centimeters scale, use tube factor 600 times ratio: batch factor/0.40.

$$Sulfide,\ \text{mg/L} = \frac{(Darkened\ Length)\ (Tube\ Factor)}{(Sample\ Volume,\ \text{cm}^3)}$$

Note: Calculate darkened length in units marked on the tube.

A.6 Carbonate

A.6.1 DESCRIPTION

The concentration of soluble carbonates in a drilling-fluid filtrate can be determined by this method. Total soluble carbonates include CO_2 and the carbonate (CO_3^{-2}) and bicarbonate (HCO_3^-) ions. Mud filtrate is acidified in a Garrett gas train, converting all carbonates to CO_2, which is then evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is collected in a one-liter gas bag (to allow CO_2 to mix uniformly) and subsequently drawn through a Dräger tube at a fixed flow rate. The Dräger tube responds to CO_2 by progressively staining purple along its length. A reaction between CO_2 and a hydrazine chemical causes a crystal violet indicator to turn purple. The stain length is proportional to the total carbonate concentration in the filtrate.

A.6.2 EQUIPMENT

The following equipment should be used:

a. Garrett gas train apparatus, as shown in Figure A-2, consisting of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter, and a Dräger tube. Specifications of the Garrett gas train:

Body:
  Chamber 1:
    Depth ....3.54 inches (90 millimeters)
    Diameter............1.52 inches (38 millimeters)
  Chambers 2 & 3:
    Depth ....3.54 inches (90 millimeters)
    Diameter............1.18 inches (30 millimeters)
  Passages between chambers:
    Diameter.............0.08 inch (2.0 millimeters)
  Material:
    Lucite® or equivalent transparent material or glass which is inert to acid, carbonates, and carbon dioxide gas.
Dispersion tube:
  Stem:
    Diameter............0.315 inch (8.0 millimeters)
    Length................ approximately 5.9 inches (150 millimeters)
  Dispersion frit:...... (bell-shaped ASTM 4-5.5 fine)
    Diameter............1.18 inches (30 millimeters)
    Pyrex® or equivalent glass.

Flexible tubing: type that is inert to carbon dioxide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and rigid tubing: type that is inert to carbon dioxide and acid.

b. Carrier gas: nitrogen (N_2) bottle with low-pressure regulator (preferred), or N_2O gas cartridges (for example, WHIP-PETS #561241 by Walter Kidde and Company Inc., Belleville, New Jersey) or equivalent.

CAUTION: Nitrous oxide cartridges should not be used as pressure sources for high-temperature, high-pressure (HTHP) 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 a Garrett gas train carbonate analysis.

c. Dräger CO_2O analysis tube: marked CO_2 0.01 percent/a (No. CH-308-01).

d. Dräger one-liter ALCOTEST gas bag No. 7626425 or equivalent.

e. Dräger MULTIGAS DETECTOR hand-operated vacuum pump Model 31 or equivalent.

f. Stopcock (2-way bore), 8-millimeter glass with Teflon® plug.

g. Sulfuric acid (CAS #7664-93-9): approximately 5N, reagent grade.

h. Defoamer in a dropper bottle.

i. Hypodermic syringes: one 10-cubic centimeter (for acid) and one 1.0-cubic centimeter, one 5-cubic centimeter, and one 10-cubic centimeter (for a sample).

j. Hypodermic needles: two 1.5-inch (38-millimeter) 21-gauge needles.

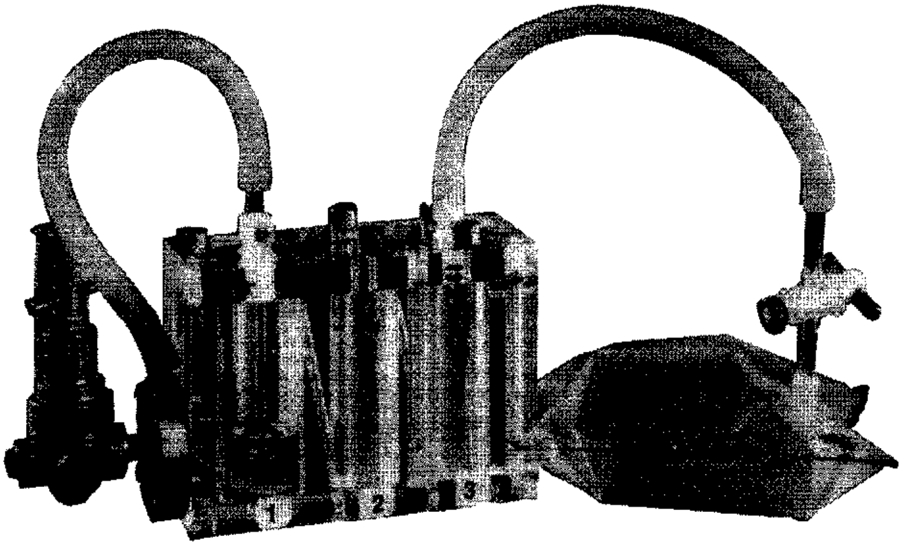


Figure A-2 Step 1—Freeing Carbonates as CO_2 Gas

Note: Nitrogen is preferred over N_2O as the carrier gas. Because N_2O cools upon expansion and chills the diaphragm in the regulator, prolonged N_2O flow will cause the regulator to perform erratically.

A.6.3 PROCEDURE

Follow this procedure:

a. Be sure that the gas train is clean and dry and on a level surface, with the top removed.

Note: If CO_2 has been used as the carrier gas in the previous test (in other words, sulfide analysis), the regulator, tubing, and dispersion tube should be purged with carrier gas at this time.

b. Add 20 cubic centimeters deionized water to Chamber 1.

c. Add 5 drops of defoamer to Chamber 1.

d. Install the top on the gas train and hand-tighten evenly to seal all of the o-rings.

e. Adjust the dispersion tube to approximately 0.25 inch (5 millimeters) off the bottom.

f. With the regulator backed off, connect the carrier gas supply to the glass dispersion tube of Chamber 1, using flexible tubing.

g. Flow carrier gas through the train for one minute to purge air from the system. Check for leaks in the gas train unit.

h. Fully collapse the gas bag and simultaneously check the system for leaks. To do this, connect the gas bag and stopcock to the hand pump, as shown in Figure A-3. (Use a discarded Dräger tube as the connection, and start with the bag essentially empty.) Fully depress and release the hand pump. When the bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is detected, check the pump and all connections. To check the pump alone, insert a sealed Dräger tube into the pump opening and depress the bellows. It will remain depressed if the pump does not leak.

i. With the bag fully collapsed, install flexible tubing from the stopcock and bag onto the outlet of Chamber 3, as seen in Figure A-2.

j. Inject a measured volume of solids-free filtrate into Chamber 1 through the septum, using a hypodermic syringe and needle. See Table A-2.

k. Slowly inject 10 cubic centimeters sulfuric acid solution into Chamber 1 through the rubber septum using a clean syringe and needle. Gently shake the gas train to mix the acid with the sample in Chamber 1.

l. Open the stopcock on the gas bag. Restart the gas flow and allow the gas bag to fill steadily during a 10-minute interval. When the bag is firm to the touch (do not burst it), shut off the flow and close the stopcock. Immediately proceed to the next step.

m. Break the tip off each end of the Dräger tube.

n. Remove the tubing from the Chamber 3 outlet and reinstall it onto the upstream end of the Dräger tube. (Observe that an arrow on the tube indicates gas flow direction.) Attach the Dräger hand pump to the downstream end of the Dräger tube, as shown in Figure A-3.

o. Open the stopcock on the bag. With steady hand-pressure, fully depress the hand pump. Release the pump so that the gas flows out of the bag and through the Dräger tube. Operate the pump and count the strokes until the bag is empty. (Ten strokes should empty the bag. More than ten strokes indicates leakage has occurred, and the test results will not be correct.)

p. Observe a purple stain on the Dräger tube if CO_2 is present in the gas bag, and record the stain length in units marked on the Dräger tube. (Include the faint blue tinge in the purple stain length reading.)

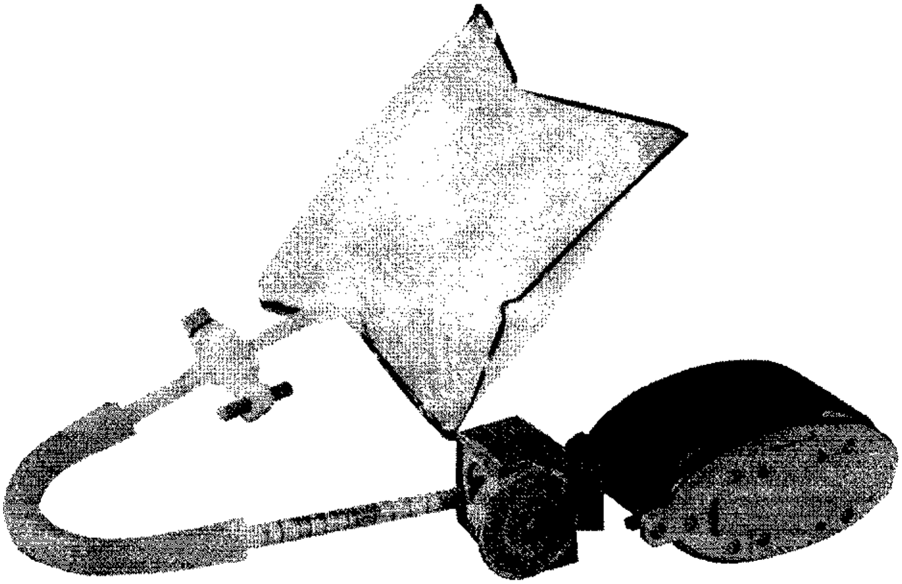


Figure A-3 Step 2—Analyzing CO_2 With Dräger Tube Analysis of Soluble Carbonate

Note: For best Dräger tube accuracy, the stain length should fill more than half the tube length; therefore, the sample volume must be carefully selected.

q. To clean the gas train, remove the flexible tubing and remove the top. Using a brush, wash out the chambers with warm water and mild detergent. Use a pipe cleaner to clean the passages between the chambers. Wash, rinse, and then blow out the dispersion tube with dry gas. Rinse the unit with deionized water and allow it to drain dry. Be sure to periodically replace the disposable gas bag to avoid leaks and contamination in the bag. (Bag replacement is suggested after ten analyses.)

A.6.4 CALCULATION

Using the measured sample volume, the Dräger tube stain length, and tube factor of 2.5 (see Table A-2), calculate total soluble carbonates ($CO_2 + CO_3^{-2} + HCO_3^{-}$) in the filtrate sample using this equation:

$$\mathit{Carbonate}, \text{mg/L} = \frac{2.5\ (\mathit{Stain\ Length})}{\mathit{Sample\ Volume}, \text{cm}^3}$$

A.7 Potassium Above 5000 Milligrams per Liter

A.7.1 DESCRIPTION

Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the properties of the drilling fluid. This procedure is used to measure the potassium ion content in mud filtrates at levels above 5000 milligrams per liter or 3.5 pounds per barrel KCl. Potassium is precipitated in a centrifuge tube as the perchlorate salt, and then the precipitate volume is measured. The potassium ion content is read from a prepared standard curve.

A.7.2 EQUIPMENT

The following equipment should be used:

a. Sodium perchlorate solution (CAS #7601-89-0): 150.0 grams $NaClO_4$/100 cubic centimeters distilled water.

Note: Sodium and potassium perchlorates are explosive in the dry state if heated or if in contact with organic reducing agents. The perchlorates are not hazardous if kept wet. They will decompose harmlessly in water.

b. Standard potassium chloride solution (CAS #7447-40-7): 14.0 grams KCl made up to 100 cubic centimeters with deionized or distilled water.

c. Centrifuge, horizontal-swing rotor head (manual or electric) capable of producing approximately 1800 revolutions per minute (see Figure A-4).

Note: A fairly constant 1800 revolutions per minute can be obtained with a manual centrifuge as follows:

1. Determine the number of revolutions of the rotor per each turn of the crank; in other words, move the crank very slowly and count the number of revolutions of the rotor head during one turn of the crank. For example, 15 revolutions of the rotor per one turn of the crank.
2. Calculate the number of crank turns required to obtain 1800 revolutions of the rotor head. In the example, to obtain 1800 revolutions of the head would require 120 turns of the crank (1800/15). Thus the crank must be turned 120 times in one minute to obtain the rate of 1800 revolutions per minute. At this rate, in 5 seconds the handle must be turned 10 times (120/60) (5). By counting the crank turns in 5 seconds and adjusting the rate to obtain the required number of turns, a constant 1800 revolutions per minute should be obtained in 15 seconds to 20 seconds. The interval used to adjust to the 1800 revolutions per minute should be included in the centrifuge time of the sample.

Table A-2—Dräger Tube or Equivalent, Identification, Sample Volumes, and Tube Factors to Be Used for Various Carbonate Ranges

Carbonate Range (mg/L)	Sample Volume (cm^3)	Dräger Tube Identification (see tube body)	Tube Factor (used in calculation)
25–750	10.0	CO_2 100/a	2.5[a]
50–150	5.0	CO_2 100/a	2.5[a]
250–7500	2.5	CO_2 100/a	2.5[a]

[a] A tube factor of 2.5 applies to new tubes, CO_2 100/a (Catalog No. 8101811), with a scale from 100 to 3000. For old tubes with 0.01 to 0.3 percent scale, use a tube factor of 25,000.

d. Clinical centrifuge tube: 10-cubic centimeter Kolmer type (do not substitute), for example, Corning #8360 (see Figure A-5).
e. Volumetric pipettes (TD): one 0.5-cubic centimeter, one 1.5-cubic centimeter, one 2.5-cubic centimeter, and one 3-cubic centimeter.
f. Hypodermic syringe or serological (graduated) pipette (TD): 10-cubic centimeter.
g. Distilled or deionized water.

A.7.3 PROCEDURE—STANDARD CALIBRATION CURVE

A standard calibration curve is required for each type of centrifuge. A minimum of three points (3.5 pounds per barrel, 10.5 pounds per barrel, and 17.5 pounds per barrel KCl) is required to obtain an accurate graph.

a. Samples can be prepared by using the standard potassium chloride solution (0.5 cubic centimeters of standard potassium chloride solution is equivalent to 3.5 pounds per barrel KCl). To obtain 3.5 pounds per barrel, 10.5 pounds per barrel, and 17.5 pounds per barrel KCl, use 0.5 cubic centimeter, 1.5 cubic centimeters, and 2.5 cubic centimeters of the standard potassium chloride solution, respectively.
b. Dilute the sample to the 7.0-cubic centimeter mark with distilled water and agitate.
c. Add 3.0 cubic centimeters of standard sodium perchlorate solution. *Do not agitate.*
d. Centrifuge at a constant speed (approximately 1800 revolutions per minute) for one minute and read the precipitate volume immediately.
CAUTION: Counterbalance the centrifuge tube with another tube and liquid of the same weight.

e. Clean the centrifuge tube immediately after use to facilitate ease of cleaning.
f. Plot the volume of precipitate (cubic centimeters) versus potassium chloride content (pounds per barrel) on rectangular graph paper as shown in Figure A-6.

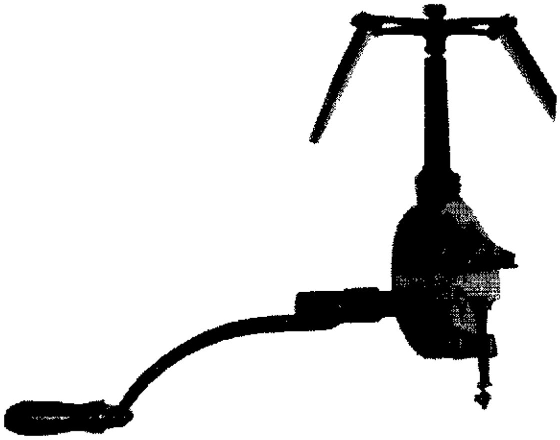


Figure A-4—Manual Centrifuge With Horizontal-Swing Rotor Head

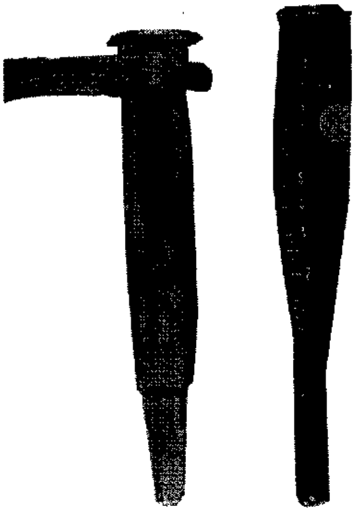
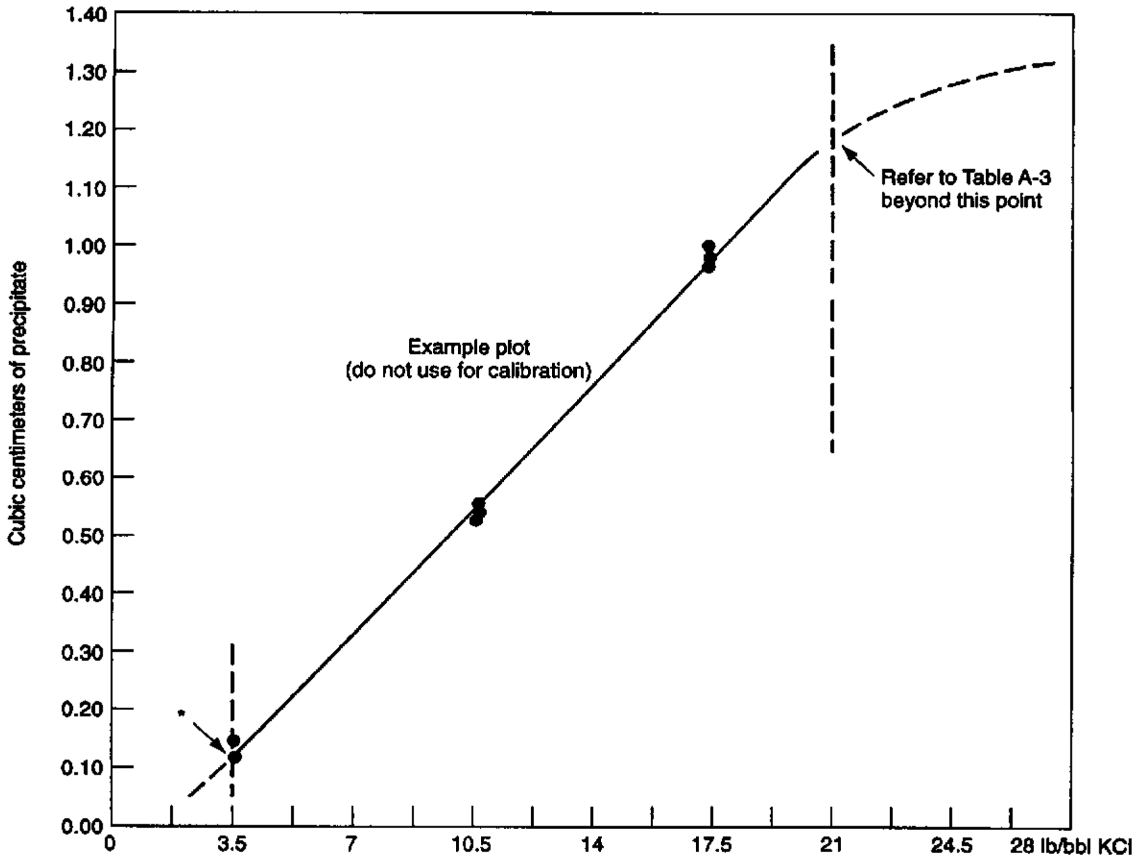


Figure A-5—10-cm³ Clinical Centrifuge Tube Kolmer Type

A.7.4 PROCEDURE — SAMPLE TESTING

Follow this procedure to perform sample testing:

a. Measure the appropriate volume of filtrate into the centrifuge tube (see Table A-3 for range).
b. Dilute to 7.0 cubic centimeters if necessary with distilled water, and agitate.
c. Add 3.0 cubic centimeters of standard sodium perchlorate solution. (*Do not agitate.*) If potassium is present, precipitation occurs at once.
d. Centrifuge at constant speed (approximately 1800 revolutions per minute) for one minute. Read the precipitate volume immediately and then record it.



Note: *Do not use this procedure for concentrations less than 3.5 lb/bbl of KCl.

Figure A-6—Example Plot Calibration Curve for Potassium Chloride

Note: Counterbalance the centrifuge tube with another tube and liquid of the same weight.

e. Add 2 drops to 3 drops of the sodium perchlorate solution to the tube. If precipitate still forms, the total amount of potassium was not measured. (See Table A-3 and use the next smaller filtrate volume. Repeat the steps in Items a through e.)

f. Determine the potassium chloride concentration by comparing the precipitate volume measured with the standard calibration curve as prepared in A.7.3. Report the potassium concentration as pounds per barrel KCl (kilograms per cubic meter). The potassium concentration may also be reported as milligrams per liter potassium ion. If the filtrate potassium chloride concentration from the standard calibration curve exceeds an 18-pounds per barrel reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in Table A-3, and repeat the steps in A.7.4, Items a through e.

A.7.5 CALCULATION

The following calculations apply:

$$\textit{KCl in filtrate}, \text{lb/bbl} = \frac{7}{\text{cm}^3 \textit{filtrate used}} (\textit{value from standard curve})$$

$$K^+ \textit{ in filtrate}, \text{mg/L} = 1500\ (\textit{KCl in filtrate}, \text{lb/bbl})$$

A.8 Potassium Below 5000 Milligrams per Liter

A.8.1 DESCRIPTION

This procedure is used to measure the potassium ion content in mud filtrates at levels below 5000 milligrams per liter. Potassium ion is precipitated as the tetraphenylborate salt by adding an excess of standard sodium tetraphenylborate (STPB) solution. The unreacted STPB is then determined by titration with a quaternary ammonium salt (QAS), hexadecyltrimethyl ammonium bromide, using bromophenol blue as an indicator. The endpoint is a purple-blue to light-blue color change. The potassium ion concentration in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

Table A-3—Filtrate Volumes to Be Used at Various KCl Concentrations

Concentration Range KCl (lb/bbl)	K+ (mg/L)	Filtrate Volume to Use (cm^3)
3.5–18	5,250–27,000	7.0
18–35	27,000–52,500	3.5
35–70	52,500–105,000	2.0
over 70	over 105,000	1.0

A.8.2 EQUIPMENT

The following equipment is needed:

a. Standard sodium tetraphenylborate solution (STPB) (CAS #143-66-8): 8.754 grams in 800 cubic centimeters deionized water. Add 10 grams to 12 grams aluminum hydroxide, stir 10 minutes, and filter it. Add 2 cubic centimeters of 20 percent NaOH solution to the filtrate, and dilute it to one liter with deionized water.
b. Quaternary ammonium salt solution (QAS) (CAS #57-09-0): 1.165 grams hexadecyltrimethyl ammonium bromide/500 cubic centimeters deionized water.
c. Sodium hydroxide solution (CAS #1310-73-2): 20 grams per 80 cubic centimeters deionized water.
d. Bromophenol blue indicator (CAS #115-39-9): 0.04 gram tetrabromophenolsulfonphthalein/3 cubic centimeters 0.1N NaOH. Dilute it to 100 cubic centimeters with deionized water.
e. Deionized or distilled water.
f. Serological (graduated) pipettes (TD): one 2-cubic centimeter graduated in 0.01-cubic centimeter subdivisions, two 5-cubic centimeter and two 10-cubic centimeter.
g. Graduated cylinders: two 25-cubic centimeter (TD) and two 100-cubic centimeter (TC).
h. Beakers: two 250-cubic centimeter.
i. Funnel.
j. Filter paper.

A.8.3 PROCEDURE

Follow this procedure:

a. Place the proper amount of filtrate into a 100-cubic centimeter graduated cylinder, using Table A-4 to determine the sample size. Be sure to use a pipette to measure the amount of filtrate
b. Add 4 cubic centimeters of 20 percent NaOH solution (measured with a 5-cubic centimeter pipette), 25 cubic centimeters of STPB solution (measured with a 25-cubic centimeter graduated cylinder), and enough deionized water to bring the level of the solution to the 100 cubic centimeter mark.
c. Mix and allow it to stand ten minutes.
d. Filter into a 100-cubic centimeter graduated cylinder. If the filtrate is cloudy, the solution must be refiltered.
e. Transfer 25 cubic centimeters of the above filtrate (measured with a 25-cubic centimeter graduated cylinder) into a 250-cubic centimeter beaker.
f. Add 10 drops to 15 drops of bromophenol blue indicator.
g. Titrate with QAS solution until the color changes from purple-blue to light blue.

Table A-4—Filtrate Volumes to Be Used at Low KCl Concentrations

Concentration Range KCl (lb/bbl)	K+ (mg/L)	Filtrate Volume To Use (cm^3)
0.2–1.3	250–2,000	10.0
1.3–2.7	2,000–4,000	5.0
2.7–6.7	4,000–10,000	2.0

Note: It is important to check the concentration of QAS solution versus the STPB solution at monthly intervals. To determine the equivalent QAS, dilute 2 cubic centimeters of the STPB solution in a titration vessel with 50 cubic centimeters deionized water. Add 1 cubic centimeter of 20 percent NaOH solution and 10 drops to 20 drops of the bromophenol blue indicator. Titrate with the QAS solution until the color changes from purple-blue to light blue.

$$Ratio\ of\ QAS\ to\ STPB = \frac{QAS,\ \mathrm{cm}^3}{2}$$

If the ratio is other than 4.0 ±0.5, calculate a correction factor to be used in the calculation of milligrams per liter K^+.

$$Correction\ factor\ (CF) = \frac{8}{QAS,\ \mathrm{cm}^3}$$

A.8.4 CALCULATION

The following calculation should be used:

$$K^+\ in\ filtrate,\ \mathrm{mg/L} = \frac{1000\ (25 - QAS,\ \mathrm{cm}^3)}{mud\ filtrate,\ \mathrm{cm}^3}$$

If a correction factor is necessary, the following applies:

$$K^+\ in\ filtrate,\ \mathrm{mg/L} = 1000 \left[\frac{25 - (CF)\ (QAS,\ \mathrm{cm}^3)}{mud\ filtrate,\ \mathrm{cm}^3}\right]$$

$$KCl\ in\ filtrate,\ \mathrm{lb/bbl} = \left[\frac{K^+\ in\ filtrate,\ \mathrm{mg/L}}{1500}\right]$$

APPENDIX B—SHEAR STRENGTH MEASUREMENT USING SHEAROMETER TUBE

B.1 Description

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.

The following technique can be used to determine the tendency and to 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 thus 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.

B.2 Equipment

The following equipment is needed:

a. Stainless steel shearometer tube:

Length	3.5 inches (89 millimeters).
Outside diameter	1.4 inches (36 millimeters).
Wall thickness	0.008 inch (0.2 millimeter).

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 inches.

B.3 Procedure

This procedure should be followed:

a. 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 ensure that 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 putting the shear tube in place for the test.

b. 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 B-1). It is desirable to submerge at least one-half the length of the tube.

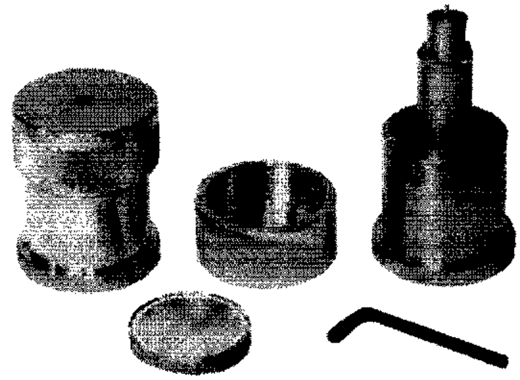


Figure B-1—Shear Strength Apparatus

c. 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.

B.4 Calculation

The following calculation[3] should be used:

$$\text{Shear strength } (S)\text{, lb/100 ft}^2 = \frac{3.61(Z+W)}{L} - 0.256\,A$$

Where:

S = shear strength, pounds per 100 square feet.
Z = weight of shear tube, grams.
W = total shear weight, grams (sum of platform and weights).
L = submerged length of shear tube, inches.
A = mud weight, pounds per gallon.

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

APPENDIX C—RESISTIVITY

C.1 Description

Control of the resistivity of a mud and mud filtrate may be desirable to better evaluate formation characteristics from electric logs.

C.2 Equipment

The following equipment should be used:

a. Direct reading resistivity meter or similar resistivity meter. Follow the manufacturer's instructions for current source, calibration, measurement, and calculations.
b. Calibrated resistivity cell.
c. Thermometer: 32°F to 220°F (0°C to 105°C).

C.3 Procedure

Follow this procedure:

a. Fill the clean, dry resistivity cell with freshly stirred mud or mud filtrate.

CAUTION: No air or gas should be entrained in the sample.

b. Connect the cell to the meter.
c. Measure resistance in ohm-meters (direct reading) or ohms (not direct reading). The meter or the manufacturer's instructions will indicate the type of reading.
d. Measure the temperature of the sample to the nearest degree °F (°C).
e. Clean the cell. Scrub it with a brush and detergent if necessary. Rinse with distilled water and allow it to dry.

C.4 Calculation

The following calculation applies:

a. Report the mud resistivity R_m or filtrate resistivity R_{mf} in ohm-meters to the nearest 0.01 ohm-meter.
b. Report the sample temperature in °F (°C). If the reading is in ohms, convert it to ohm-meters as follows:

Resistance, ohm-meter = (*R*, ohms) (*cell constant*, m^2/m)

APPENDIX D—PROCEDURE FOR REMOVAL OF AIR OR GAS FROM MUD PRIOR TO TESTING

D.1 Description

The majority of drilling muds require no special equipment to remove entrained air or gas prior to testing. Usually, gentle agitation, together with a few drops of an appropriate defoamer, are all that is necessary. Stirring with a spatula or pouring back and forth is sufficient in most cases. When a mud is encountered that retains air or gas after the preceding steps have been taken, the following procedure can be taken to deaerate the mud.

Note: If mud density is the only property desired, the pressurized fluid density balance described in 3.6.1 through 3.6.5 may be used. This unit is also illustrated in Figure 3.

D.2 Equipment

The following equipment is needed:

a. A device that can be evacuated is necessary. The equipment shown in Figure D-1 is available. Follow the manufacturer's operating instructions.
b. Defoamer.

D.3 Procedure

Follow this procedure:

a. Fill a clean, dry reservoir about one-half full with the air-cut mud.
b. Add several drops of defoamer to the mud surface.
c. Insert the stirrer and cap; cover with a lid that has a gasket.
d. Affix the vacuum line from the pump to the instrument and hold it about 5 inches-of-mercury vacuum.
e. Increase the vacuum to 25 inches to 27 inches of mercury and proceed according to the manufacturer's instructions.
f. When mud has been deaerated, partially relieve the vacuum to about 15 inches to 10 inches-of-mercury vacuum and observe the mud for air bubbles.
g. If deaeration is not sufficient, repeat steps e and f until the air is removed.
h. With the cylinder on end, relieve the vacuum completely and remove the mud sample for testing.

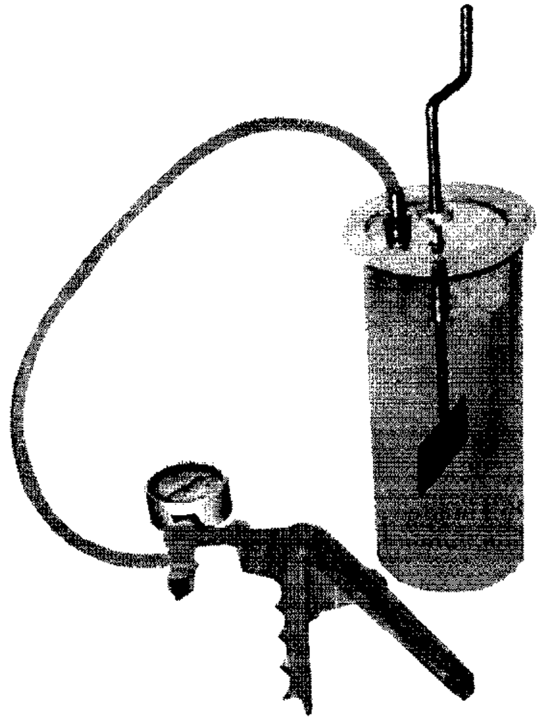

Figure D-1—Instrument for Air or Gas Removal

APPENDIX E—DRILL PIPE CORROSION RING COUPON

E.1 Description

The placement of corrosion test rings in the drill string is one of the more common techniques used to evaluate the corrosiveness of drilling-fluid environments on the drill string and other steel equipment. Removal and examination of these rings after a period of exposure downhole can be highly informative as to the corrosiveness of the drilling fluid, as well as to the type of corrosion encountered. An examination of scales and pits on the exposed rings gives clues as to the cause of the corrosion, thus aiding in choosing proper remedial action.

The ring technique is specifically designed for detection of the type of corrosion characterized by metal loss whether it be localized pitting or generalized attack. The test ring is not designed to give information relating to hydrogen embrittlement, stress corrosion cracking, or other forms of fracture formation except in the manner in which pitting may relate to these failures.

E.2 Equipment

The following equipment is needed:

a. Ring construction: The ring-type drill string corrosion coupon, or corrosion ring, should be machined to fit in the tool box recess, at the end of the pin, and should have a bore the same as that of the tool joint to minimize turbulence.

b. Ring composition: To avoid galvanic corrosion, the ring should be made from steel identical to that of the tool joint in which it is placed. Such a requirement is impractical, and use of a steel that is similar in chemical composition, such as AISI 4130, is recommended.

Note: If steel other than AISI 4130 is used, it should be identified on the report form. The rings are normally cut from tubes that have not been quenched and tempered. The similarity in composition of the 4130 steel and the tool joint should be adequate to minimize galvanic effects and provide useful data.

c. Ring marking: The rings should be stenciled with a serial number for permanent identification.

d. Ring preparation: The rings should be scrubbed with a stiff fiber bristle brush and detergent solution, rinsed with clean water and with anhydrous acetone or methanol. The rings should be allowed to dry, weighed to nearest milligram, and this weight recorded on the report form. The ring should be stored in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed envelopes or wrappers to minimize atmospheric corrosion.

E.3 Procedure

The drill pipe corrosion rings should be kept in the drill string for a minimum of 40 hours. A normal time for exposure is 100 hours. Exposure periods of less than 40 hours should not be used because initial corrosion rates may be unusually high and can give misleading data. The ring is usually placed in the tool joint at the top of the first stand above the drill collars and can be left in the drill string for more than one bit run. An additional ring can be placed in the kelly saver sub to monitor corrosion at that point. Care should be taken to ensure that the box recess is clean to prevent interference with proper make-up of the joint and to avoid damage to the ring. In some instances specially manufactured subs have been used for the ring placement in the string. During installation, the ring should be handled with clean, dry gloves.

The drill pipe corrosion coupon form should be filled out completely. Each form should have a space for ring material, drilling fluid properties, type of corrosion, location of ring in the drill string, initial weight, time, depth in, depth out, ring number, color of scale, and any other information of significance in the specific test. The form may be printed on a mailing envelope for the ring or on a separate form to be enclosed with the ring.

The drilling-fluid residue should be removed from the coupon by wiping with a cloth when the ring is pulled from the drill string. The ring should be examined for severity of corrosion or mechanical damage. If severe corrosion is evident, the cause of the corrosion should be determined promptly so remedial action can be taken. Following visual observation, the coupon should be placed in the original envelope or wrapper containing a vapor phase corrosion inhibitor for return to the laboratory.

Before proceeding with a quantitative evaluation of corrosion of the ring, the ring should be rinsed with a suitable solvent, such as acetone or petroleum ether, to remove the oil applied to the ring on location. Prior to cleaning for weighing, a spot test should be made for corrosion by-products and mineral scale. For example, the surface can be examined qualitatively for sulfides by the acid arsenite test. The rings should be cleaned with a detergent solution and a stiff fiber bristle brush. It may be necessary to dip the ring for 5 seconds to 10 seconds in inhibited 10 percent to 15 percent hydrochloric acid one or more times to remove corrosion products. The ring should be scrubbed with the detergent solution after each acid dip, and rinsed thoroughly with clean water and then with anhydrous acetone or methanol. The ring should dry prior to weighing. Very abrasive materials or strong, uninhibited acids should not be used. An ultrasonic bath can be useful in cleaning the rings.

After the preweighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of attack noted, the ring should be reweighed to the nearest milligram and the weight loss determined. If significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as kilograms per square meter per year, pounds per square foot per year, or mils per year. Formulas for calculating for corrosion rate are given in E.5.

E.4 Comments on Visual Examination

If visual corrosion is evident, it will normally be detectable as pitting corrosion. Uniform attack or general corrosion can best be determined by a weight loss measurement. Mechanical damage to the ring will most often be evidenced by cuts or dents on the outer surfaces of the ring. In some cases, the ring will exhibit a series of dents and worn spots, indicating considerable movement of the ring in the box recess.

In assessing the magnitude of the corrosion rates as calculated from weight loss measurements, it should be remembered that the rate is also influenced by the erosive effects of the drilling fluid. Since the bore of the ring is exposed to the mud pumped down the drill pipe, the loss of metal includes that removed by erosion as well as from corrosion. Loss from erosion can be substantial when the drilling fluid contains a high concentration of sand.

Examination of the ring may reveal a few deep pits with a relatively low weight loss. This condition would indicate a rather severe corrosion problem even though the calculated corrosion rate would be considered low.

E.5 Calculation

The following formulas apply to calculating the weight loss (wt. loss) corrosion rate:

a. For kilograms per square meters per year (kg/m²/yr):

$$= \left[\frac{\mathit{wt.\ loss},\ \mathrm{mg}}{1{,}000{,}000}\right]\left[\frac{10{,}000}{\mathit{area},\ \mathrm{cm}^{2*}}\right]\left[\frac{365}{\mathit{days\ exposure}}\right]$$

$$= \frac{87.60(\mathit{wt.\ loss},\ \mathrm{mg})}{\mathit{area},\ \mathrm{cm}^{2*}(\mathit{hours\ exposed}^{**})}$$

$$= \frac{13.58\ (\mathit{wt.\ loss},\ \mathrm{mg})}{\mathit{area},\ \mathrm{in}^{2*}(\mathit{hours\ exposed}^{**})}$$

b. For pounds per square feet per year (lb/ft²/yr):

$$= \left[\frac{(\mathit{wt.\ loss},\ \mathrm{mg})}{453{,}600}\right]\left[\frac{144}{\mathit{area},\ \mathrm{in}^{2*}}\right]\left[\frac{365}{\mathit{days\ exposure}}\right]$$

$$= \frac{2{,}781\ (\mathit{wt.\ loss},\ \mathrm{mg})}{\mathit{area},\ \mathrm{in}^{2*}(\mathit{hours\ exposed}^{**})}$$

c. For mils per year (mils/yr):

$$= \frac{\mathit{wt.\ loss},\ \mathrm{mg}}{\left[\frac{16{,}387}{\mathrm{cm}^3/\mathrm{in}^3}\right]\left[\frac{\mathit{specific\ gravity},}{\mathrm{g/cm}^3}\right]\left[\frac{(\mathit{area}}{\mathrm{in}^{2*})}\right]\left[\frac{(\mathit{year}}{\mathit{days}/365)}\right]}$$

For steel coupons with a specific gravity of 7.86, the formula can be reduced to the following:

$$\mathrm{mils/yr} = \frac{68.33\ (\mathit{wt.\ loss},\ \mathrm{mg})}{\mathit{area},\ \mathrm{in}^{2*}(\mathit{hours\ exposed}^{**})}$$

---

*Total surface area of the ring is used in these calculations.

**Time used is based on total time in the drill string.

Following are the conversion rates between the various units for steel coupons (specific gravity 7.86):

mils/yr = 24.62 (lb/ft²/yr)

mils/yr = 5.03 (kg/m²/yr)

lb/ft²/yr = 0.04 (mils/yr)

lb/ft²/yr = 0.20 (kg/m²/yr)

kg/m²/yr = 0.20 (mils/yr)

kg/m²/yr = 4.90 (lb/ft²/yr)

Corrosion rings available from drilling fluid service companies or corrosion test laboratories are generally supplied with a multiplication factor which includes the metal density and surface area of the ring. Thus, the weight loss divided by the exposure time need only be multiplied by the supplied factor to obtain the corrosion rate.

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, and OCMA-grade bentonite and is to be used in the event of product property dispute.

F.2 Procedure—Powdered Material in Packages

The following is the procedure applying to powdered material in packages:

a. The number of samples taken to be combined for the test sample (see F.4) shall be 15 samples of one pound (0.45 kilograms) or more per lot.
b. For each lot of 1000 packages or less, 15 packages shall be sampled.
c. The sampling may be carried out by either of the following methods as agreed upon by the contracting parties.

   1. A sample weighing at least 1 pound (0.45 kilograms) shall be taken from the top of each package.
   2. A sampling tube, capable of taking a core not less than 1 inch (2.5 centimeters) in diameter shall be used. The tube shall be inserted into the package being sampled so that it will take a core of material for substantially the entire length of the package.

F.3 Procedure—Powdered Material in Bulk

The following procedure applies to powdered material in bulk:

a. In the collection of samples from cars or storage bins containing 25 tons to 100 tons (22,700 kilograms to 90,900 kilograms), a sampling tube capable of taking a core not less than 1 inch (2.5 centimeters) 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.
b. 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.
c. From bins containing less than 25 tons (22,700 kilograms), 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 kilograms) or less shall be not less than 10.

F.4 Procedure—Test Sample

The following procedure applies to the test sample:

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

The following procedure applies to the test results:

a. Each party shall make one measurement on his test sample.
b. 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 can be 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 the 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

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.

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.

APPENDIX G—RIG SITE SAMPLING

G.1 Description

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.

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)

The following equipment is needed for bottom flow sampling:

a. Side-stream sampler: shown in Figure G-1.
b. Canvas bag: 5½ inches (140 millimeters) diameter and 12 inches (300 millimeters) long.
c. Container: tie-bag or other suitable container, constructed to retain all fines.

G.3 Procedure—Bottom Flow Sampling (Preferred)

The following procedure is used for bottom flow sampling:

a. Connect a side-stream sampler to the truck outlet.
b. Take three 3-pound to 5-pound (1.4-kilogram to 2.3-kilogram) samples while unloading, spaced so as to be taken when approximately one-fourth, one-half, and three-fourths of the load has been transferred.

Note: One 9-pound to 15-pound (4.1-kilogram to 6.8-kilogram) 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.

c. 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.

d. Carefully transfer the sample from the canvas bag to a tie-bag or other suitable container. Label this container with all pertinent information.
e. 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

The following equipment is needed for grab sampling:

a. Sample scoop: shown in Figure G-2.
   1. 4 inches to 6 inches (100 millimeters to 150 millimeters) deep.
   2. 12 inches to 18 inches (300 millimeters to 450 millimeters) long.

a. Container: tie-bag or other suitable container, so constructed as to retain all fines.

G.5 Procedure—Grab Sampling

This procedure should be followed for grab sampling:

a. Open the top hatch carefully. Do not let the cover bump the top of the tank.
b. Remove the top surface material under the hatch to form a trench one-and one-half times wider than the sample scoop.
c. Scoop out a 3-pound to 5-pound (1.4-kilogram to 2.3-kilogram) sample from the bottom of this trench and transfer it to a tie-bag or other suitable container. Label the container.
d. 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.

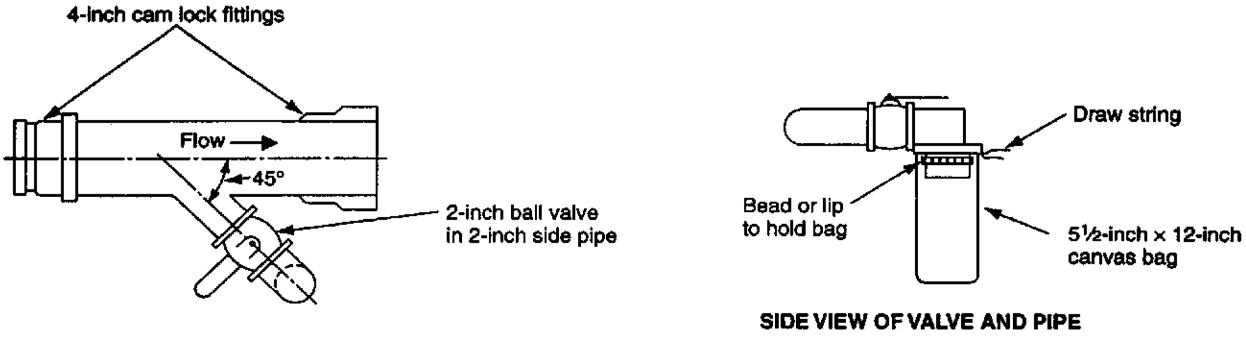

Figure G-1—Side-Stream Sampling Device

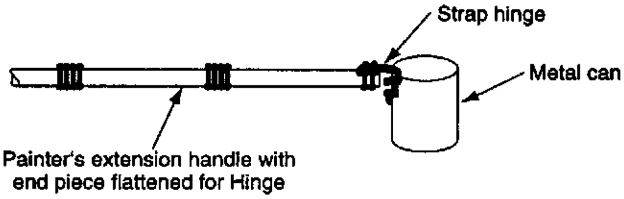

Figure G-2—Sample Scoop

APPENDIX H—CALIBRATION OF GLASSWARE, THERMOMETERS, VISCOMETERS, AND MUD BALANCES

H.1 Description

This section covers calibration of field testing equipment not covered as part of a test procedure.

H.2 Equipment

H.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.

H.2.2 THERMOMETERS

Thermometers used in field testing, especially those commonly used metal dial thermometers, should be calibrated 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.

H.2.3 VISCOMETERS

Viscometers used in field testing should be calibrated 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.

H.2.4 MUD BALANCES

Mud balances should be calibrated 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.

H.3 Calibration Procedure—Thermometer

H.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.

H.3.2 Read both thermometers and record the readings.

H.3.3 Repeat the readings at 5-minute intervals to obtain at least four sets of readings.

H.3.4 Calculate the average reading for each thermometer.

H.3.5 For adjustable thermometers, adjust to read the same as the standard thermometer. For other thermometers, go to H.3.6.

H.3.6 Calculate the thermometer correction as follows:

$$C = R1 - R2$$

Where:

$R1$ = the average reading for the standard thermometer.
$R2$ = the average reading for the working thermometer.
C = the value to add to the thermometer reading.

The following are examples:

a. Example 1—thermometer correction determination:

$$R1 = 75.0^\circ\text{F}$$

$$R2 = 75.5^\circ\text{F}$$

$$C = 75.0^\circ\text{F}\ 2\ 75.5^\circ\text{F} = 20.5^\circ\text{F}$$

or

$$R1 = 23.89^\circ\text{C}$$

$$R2 = 24.17^\circ\text{C}$$

$$C = 23.89^\circ\text{C}\ 2\ 24.17^\circ\text{C} = 20.28^\circ\text{C}$$

b. Example 2—thermometer correction application:

$$C = 20.5$$

$$R = 78.5$$

$$Rc\ (\text{corrected}) = 78.5\ 1\ (20.5) = 78.0^\circ\text{F}$$

H.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.

H.4 Calibration Procedure—Viscometer

H.4.1 Obtain a certified calibration fluid or fluids with a chart (viscosity versus temperature) to cover the range of interest; for example, 50 centipoise, 100 centipoise, and so forth. Make certain that the lot number on the chart matches the lot number on the fluid container. Each lot of standard fluid is individually certified. The viscosity will normally vary slightly from lot to lot.

H.4.2 Clean and dry the viscometer bob, rotor, and cup. Place the viscometer and fluid side-by-side on the 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.

H.4.3 Operate the viscometer in air for 2 minutes to 4 minutes to loosen bearings and gears.

Note: If the rotor wobbles excessively, correct it if possible.

H.4.4 Fill the cup to the scribed line with calibration fluid and place it on the meter stage. Move the stage until the fluid level is to the inscribed line on the rotor sleeve.

H.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 a low-speed setting until the thermometer reading is stable to within 0.2°F (0.1°C) per 30 seconds. Record the temperature reading.

H.4.6 Operate the viscometer and take readings at 600 revolutions per minute and 300 revolutions per minute. Estimate readings to the nearest 0.5 dial unit. If needed, use a magnifying glass.

H.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 the deviation (plus or minus). Divide the 600 revolutions per minute dial reading by 1.98, compare that to standard viscosity, and record the deviation.

H.4.8 Deviations exceeding 1.5 units are not acceptable. If deviation exceeds these tolerances, adjust or calibrate the viscometer.

H.4.9 Record the viscometer serial number, date, and deviation. Mark the viscometer with the date of calibration and indication of calibration status.

H.5 Calibration Procedure—Filter Press Gasket and O-Rings

H.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).

H.5.2 Check the gasket or O-ring using a gauge having these diameters.

H.5.3 Do not use gaskets or O-rings outside these tolerances for filter loss testing.

APPENDIX I—SI UNIT CONVERSION TABLE

APPENDIX I—SI UNIT CONVERSION TABLE

Property	Traditional Units	Recommended SI Units	Symbol	Conversion Factor	Example
Mechanical Properties				**Multiply by:**	
Depth	feet	meter	m	0.3048	10,000 ft = 3048 m
Hole diameter	inch	millimeter	mm	25.4	12 ¹/₄ in. = 311 mm
Pipe diameter	inch	millimeter	mm	25.4	4 ¹/₂ in. = 114 mm
Bit size	inch	millimeter	mm	25.4	12 ¹/₄ in. = 311 mm
Weight on bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary table speed	rpm	ᵃrevolutions 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	ᵃmeter/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	6¹/₂ in. = 165 mm
Rod diameter	inch	millimeter	mm	25.4	2¹/₄ in. = 57 mm
Stroke length	inch	millimeter	mm	25.4	16 in. = 406 mm
Pump output	barrel/minute	ᵃcubic meter/min.	m^3/min.	0.159	8.5 bbl/min = 1.35 m^3/min.
	gallon/minute	ᵃcubic 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.	ᵃmeter/minute	m/min.	0.3048	200 ft/min = 61 m/min.
Slip velocity	feet/min.	meter/minute	m/min.	0.3048	20 ft/min = 6.1 m/min.
Temperature	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	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	120.0	10 lb/gal = 1200 kg/m^3
	pounds/ft^3	kilogram/cubic meter	kg/m^3	16.0	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	ᵃcentipoise	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
	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 centimeter/ 30 min.	cubic centimeter/ 30 min.	cm^3/30 min.		
Filter cake	¹/₃₂ inch	millimeter	mm	0.8	³/₃₂ in. = 2.4 mm
Sand content)					
Solids content)	volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Oil content)					
Water content)					
Particle size	micron	micrometer	µm	1.0	
Chemical Properties					
Ionic Concentration	grains/gallon	ᵃmilligram/liter	mg/L	17.1	500 grains/gal = 8600 mg/L
	parts/million	ᵃmilligram/liter	mg/L	× specific gravity	100,000 ppm of NaCl × 1.0707 = 107,070 mg/L
Alkalinity P_f, M_f, $P1$, $P2$	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		

ᵃDenotes acceptable deviation from API 2564 preferred unit.

APPENDIX I—SI UNIT CONVERSION TABLE (Continued)

Property	Traditional Units	Recommended SI Units	Symbol	Conversion Factor	Example
Miscellaneous				**Multiply by:**	
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	0.0254	200 mils/year = 5.08 mm/a
Bentonite yeld	barrels/US ton	[a]cubic meter/metric ton	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	1.0	
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

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[a] Denotes acceptable deviation from API 2564 preferred unit.

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