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DATE 16/01/25	Exp 1: Introduction; Design and Preparation of WBM	SHEET NO. 1

Aim: Design and Preparation of WBM drilling fluids

Defination: Drilling fluid also called drilling mud, used to aid the drilling of oil and natural gas wells and on exploration drilling rigs.

Drilling fluid composition: oil-Based muds (<5% water), Water-Based muds, Foams, Gas fluids, Reverse emulsions (5 to 50% water)

Drilling fluid additives:

⇒ Water Base:
- → Base fluid: clear water or brines
- → viscosifiers: Clay (bentonite, hectorite), Polymers
- → Filtration control agents
- → Shale stabilizers
- → weighting agents: Low gravity solids (~2.7 sg), high gravity solids (~4.2 sg)

⇒ Oil Base:
- → Base fluid: isomeric olefins, diesel, esters etc.
- → Emulsifiers
- → water (or Brine)
- → viscosifiers: organophilic clays, Polymers
- → Filtration control agents
- → weighting agents: Low gravity solids, High gravity solids

Equipment used : Hamilton Beach Mixture

Standard operating Procedure of Hamilton Beach Mixture:

i) Formulate the required amount of water, Salt, viscosifier, Weighting material, Additives and LGS as req.

ii) Clean/wipe the mechanical stirrer properly before inserting into mixing container.

iii) Measure all liquid components using measuring cylinder and solid components using properly balanced weighting balance for accuracy.

iv) Add the required amount of water into mixing container.

v) Add the req. amount of salt into water and mix @ low speed for 90 sec.

vi) Add the req. amount of viscosifier, Additives, LGS and weighting material in proper order with a time interval of 5 min after every addition of single component.

vii) continue the mixing until uniformity is obtained

viii) once uniformity is obtained stop mixing and remove the contents to separate container

ix) Rinse and clean the mechanical stirrer properly.

x) Make ~~sure~~ RPM kept to zero before turning the mechanical stirrer OFF and then power OFF main switch.

Calculation and Results

Given data,

→ Water based mud → P_P = 1lb-bbl (350ml)

→ Fluid density = 11ppg = 1.32 g/ml

→ Salt = 75000ppm = ~~75g/L~~ = 7.5%

→ Barite density = 4.2 SG

Formula used:

→ $\frac{P_f}{8.345}^{\to(ppg)} = V_{Brine}\rho_{Brine} + V_{add}\rho_{add} + V_{Barite}\rho_{Barite}$ | $\frac{V_{Br}}{V_W} = \frac{100/\rho_{Br}}{(100 - salt\_wt\%)}$

$V_f = V_{Br} + V_{Barite} + V_{add}$.

$M_{Br} = M_W + M_{salt}$

From additive details: —

$V_{add} = \frac{1}{1} + \frac{2}{1.2} + \frac{15}{2.6} = 8.4358$ ml/350ml.

From salt table, for NaCl | $\rho_{add} = \frac{1+2+15}{\left(\frac{8.4358}{350}\right)}$

For weight % = 7%. Density = 1.045 kg/L

weight % = 8%. " = 1.056 kg/L

$\therefore \rho_{Brine} = 1.052$ kg/L for 7.5%.

$\frac{V_{Br}}{V_W} = \frac{100/1.052}{(100-7.5)} = 1.0276$

$V_f = 350 = V_{Brine} + 8.4358 + V_{Barite}$

$\therefore V_{Brine} + V_{Barite} = 341.56$ — (i)

$V^f_{Brine} + V^f_{Barite} = 0.9758$

Now, $V_{Brine} \times 1.052 + \frac{8.4358}{\cancel{350}} \times \frac{15+2+1}{8.4358} + V_{wt} \times 4.2 = \left(\frac{\cancel{11}}{8.345}\right) 350 \times 1.3$

~~$\Rightarrow 1.052 V^f_{Br} + 4.2 V^f_{wt} = 1.147 \Rightarrow 1.052 (0.9758 - V^f_{BWt}) + 4.2 V^f_{wt}$~~ ~~= 1.147~~

$\therefore 462 = 1.052 V_{Br} + 4.2 V_{Br} + 18$ — (ii)

On solving (i), (ii) ——

$\boxed{V_{wt}^{f} = \frac{0.1204}{3.148} = 0.038 \quad \frac{V_{Br}}{V_w} = 1.0276 \Rightarrow V_w^f = 0.913 \quad V_{Br}^{f} = 0.0379}$

$V_{wt}^{f} = \cancel{13.3\,ml}$ 26.899 ml $\quad V_{Br} = \cancel{318.668\,ml}$ 314.66 ml $\quad V_{add} = 8.4358$ ml

Mass of Barite $\Rightarrow$ ~~13.3 × 4.2 = 55.8 g~~ 112.976 g.

Mass of water $\Rightarrow$ ~~$V_w^f \times 350 \times \rho_w = 0.913 \times 350 \times 1$~~

$\hookrightarrow \frac{314}{1.0276 \times 350} \times \frac{350}{1} \times 1 = 305.56g \quad = \cancel{319.45\,g}$

Salt to be added $= M_{Brine} - M_{water}{}^{2} = 331.029 - 305.56$

$= \cancel{\rho_{Brine}\left(350 \times V_{Br}^{f}\right) - 319.45}$

$= 25.48g$

Results:

Mass of Barite: 112.97g
Mass of NaCl: 25.48g
Mass of Water: 305.56g

<u>Precautions:</u>

1) Continue to mixing until uniformity is obtained in the solution
2) Rinse and clean the mechanical stirrer properly
3) Make sure to turn off the stirrer after use.

DATE 16/01/25 Exp: 2 | Mud Balance SHEET NO. 5

Working Principle of Mud balance:

→ A cup of known volume and mass is attached to one end of the beam.

→ Engineers fill the cup with drilling mud.

→ The mass of the filled cup is balanced on the other end of the beam by a fixed countermass and a rider that can move freely along the graduated scale.

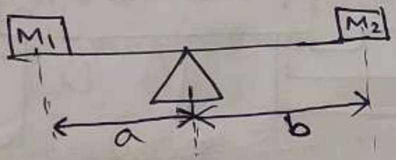
$T_1 = F_1 a$, $T_2 = F_2 b$

$M_1 \times a = M_2 \times b$

F_1 = input force to the lever

F_2 = output force

a, b = ⊥'r distance between the forces and the fulcrum.



Theory: Density is defined as weight per unit volume. It is expressed either in pounds per gallon (lb/gal) or pounds per cubic foot (lb/ft^3), or in kg per cubic meter, or as specific gravity (SG). Pressure exerted by a static mud column depends on both the density and the depth. Mud density is used to control subsurface pressures and stabilize the wall bore.

Procedure:

1) Remove lid from the cup, and completely fill the cup with the mud to be tested

2) Put the lid on the top of the cup and rotate until firmly seated, making sure mud is expelled through the hole in the lid.

3) Wipe the mud from the outside of cup by using clean tissue paper

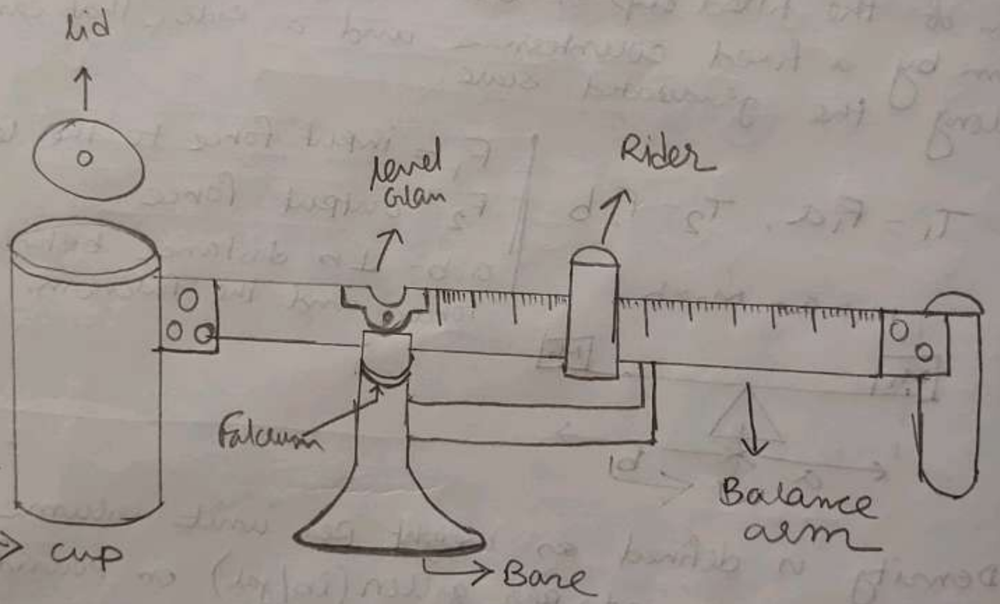


Fig: Mud Balance device

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4. Place the balance arm on the base, with the knife-edge resting on the fulcrum.

5. Move the ride until the graduated arm is level, indicated by the level bubble present on the beam. (Bubble should be in centre of the markings and steady).

6. At the left-hand edge of the rider, note down the density value with temperature without disturbing the rider.

Precautions:

1) Use separate dustbin to throw mud.
2) Clean everything after use.

Calibration Procedure: The mud balance calibration can be checked using fresh water. At (21C) fresh water should give a reading of 1.00 on the specific gravity scale psi/1000ft, 8.34 on the lb/gal scale, and 62.3 on the lb/cu ft. scale.

→ if the Mud Balance doesn't give the correct reading for water then replacing the lid on the balance cup with a new lid can cause the Mud Balance to be out of calibration.

Calibration frequency: once a month.

Conclusion:

The mud balance experiment is essential for determining the density of drilling mud, which plays a crucial role in wellbore stability and controlling formation pressures. This experiment reinforces the importance of proper mud weight management for overall well integrity and operational success.

Result: The density of ~~Mud was~~ WBM was found to be 11ppg from the given exp.

Assignment 01 (A)

1) V_W = vol. of water req. V_{Br} = vol. of Brine

W_S = Mass of salt in weight %

ρ_{Br} = density of brine

Brine's total weight (W_{Br}) per unit vol. $\Rightarrow W_{Br} = \rho_{Br} \times V_{Br}$

weight of salt : W_S = Salt_wt % $\times W_{Br}$

$\therefore W_S = \text{Salt\_wt\%} \times (\rho_{Br} \times V_{Br})$

weight of water: $W_W = (100 - \text{Salt\_wt\%}) \times W_{Br}$

$W_W = (100 - \text{Salt\_wt\%}) \times (\rho_{Br} \times V_{Br})$

~~We~~ Also, $W_W = V_W \times 8.345$

$\Rightarrow V_W \times 8.345 = (100 - \text{Salt\_wt\%}) \times (\rho_{Br} \times V_{Br})$

Now, $\dfrac{V_{Br}}{V_W} = \dfrac{1}{\rho_{Br}} \dfrac{100}{(100 - \text{Salt\_wt\%})} \times 8.345$

$$\therefore \boxed{\frac{V_{Br}}{V_W} = \frac{\dfrac{100}{\rho_{Br}/8.345}}{100 - \text{Salt\_wt\%}}}$$

2)	Dispersed System	Non-dispersed System
Defination	A system where fine solids are intentionally dispersed into the fluid using chemical dispersants.	System where fine solids are not dispersed but allowed to settle or be removed.
Key Additives	Dispersants (e.g. Lignosulfonates, tannis) to break down and evenly distribute particles.	Minimal chemical additives, typically uses viscosifiers (bentonite)
Fluid Characteristics	Higher viscosity due to dispersed fine solids	lower viscosity, as fine solids are not dispersed
Solid Control	Solids are dispersed to prevent sedimentation	Solids are controlled via mechanical removal or settling.
Applica-tions	Used for wells with high temp. and high Pressure (HTHP) or in reactive clay formations.	Used in simple, low-cost wells with minimal reactive clays.

3) Arrangement of additives in typical order of addition for WBM formation :

Salt → ~~weighting Material~~ viscosifier → ~~Viscosifier~~ weighting Material

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Assignment 01 (B)

1) N/m^3 not a unit of density, unit of specific density.

2) Density of substance changes with temperature, so it is necessary to mention the temp at which specific gravity is calculated.

3) True; because it can lead to incosistencies in the measurement.

4) Hydrostatic Pressure (Psi) = Mud weight (ppg) × TVD (ft) × 0.052

= 11 × 9000 × 0.052 = 5148 Psi

DATE 23/01/25 | EXP 3: Digital PH Meter | SHEET NO. 10

Aim: To determine the pH of a given fluid using pH meter.

Theory:

pH of the solution is the logarithm of the reciprocal of the (H^+) concentration in gm/lit.

The acidity and the alkalinity of the drilling fluid can be measured by the conc. of the (H^+) ion in the fluid.

$$PH = -\log[H^+] = \log\frac{1}{[H^+]}$$

on the scale 7 is neutral, less than 7 is acidic and greater than 7 is alkaline.

Procedure:

1) Before measurement, rinse pH electrode and ATC probe with distilled water to remove any impurities stuck onto the bodies of probes.
2) Wipe pH electrode and ATC Probe with clean dry tissue Paper
3) Power on the meter using ON/OFF key. Press 'MODE' key to select your desired mode (pH) of operation (pH, mV, Ion, or temperature)
4) Dip and stir both probes gently into given test sample, swirl gently and wait for the reading to stabilize. Note the reading. freeze the display if desired.
5) Note the value of pH with temp.
6) Rinse probe with distilled water before taking next reading or storage.

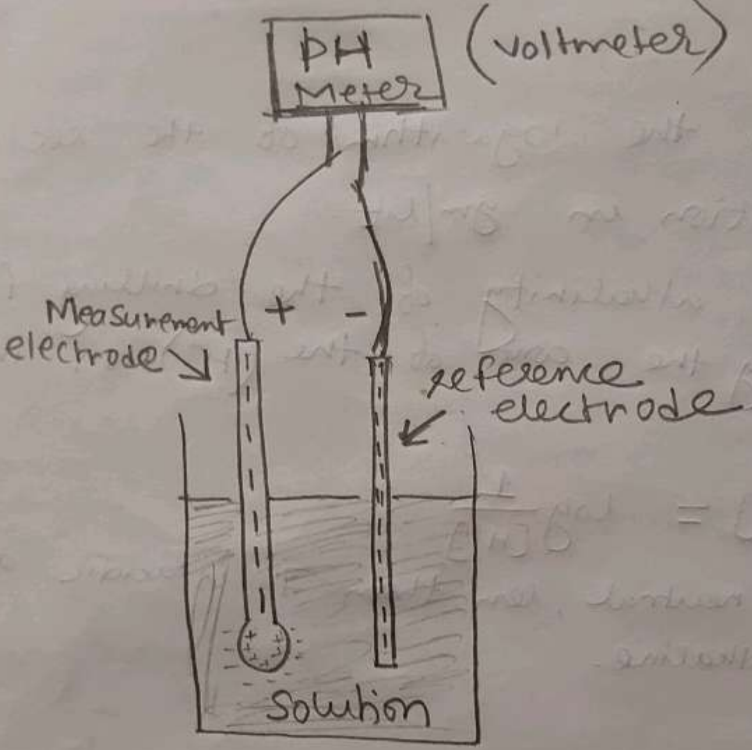

fig: pH measurement

Working Principle of pH Meter:

~~1) The H^+ ions present~~

1) The working principle of pH meter is based on the relation between the electric voltage and hydrogen ion concentration.

2) The pH meter consists of glass (also called as measurement electrode) and reference electrode.

3) Both electrode are connected to a voltmeter

4) The measurement electrode consists of a glass membrane, which is sensitive to hydrogen ion concentration.

5) The reference electrode is standard and has constant Potential.

6) The acidic solution is rich in H^+ ion ~~con~~ concentration

7) when pH Probe is dipped in an acidic solution and Switch on the voltmeter

8) The H^+ ions move close to the glass membrane of the sensitive glass bulb (external side of the bulb)

9) A similar reaction occurs inside the bulb, which is filled with a buffer solution of neutral pH (which has constant no. of hydrogen ions).

10) The H^+ ions present inside the bulb also moves close to glass membrane. (internal side of bulb)

11) Hence, this causes the difference in the concentration of hydrogen ions across the membrane

12) when hydrogen ion conc. inside bulb is less than Outside solution (test solution), then given solution is acidic and pH < 7.

13) when hydrogen ion conc. inside glass bulb is similar to the outside solution, then given solution is natural pH hence the pH is 7.

14) when hydrogen ion concentration inside the glass bulb is more than the outside solution (test sol.), the given solution is alkaline and pH > ~~7~~.

Calculation and Results

1) pH = 7.16 for Distilled water @ 24.5°C

2) pH = 7 for buffer solution (Given)
observed pH = 7.04 @ 24.7°C

3) for pH = 10 buffer solution
observed pH = 9.8 @ 24.8°C

4) for WBM, observed pH = 7.30 @ 24.7°C

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Precautions:

1) Don't use the electrode as a stirring glass rod when adjusting pH.
2) Do the calibrations before testing the each sample using standard buffer solutions.
3) Avoid temp. fluctuations and never keep the pH meter exposed to direct sunlight
4) Always do the calibration by attaching ATC to the electrode for best accuracy.
5) Glass electrode shouldn't be left out of the storage solution Prolonged intervals as the glass membrane get dehydrated resulting in slower response or response failure.

Discussion

The pH of the mud sample was found to be 7.30 at 24.7°C, indicating that it is slightly alkaline. This suggests that the presence of basic components in the sample, making it more neutral but leaning towards alkalinity. Proper pH balance in drilling mud is ~~ex~~ important for maintaining the drilling efficiency and Preventing corrosion or, sedimentation of the equipment.

Assignment

1) Full form of ATC in the pH measurement equipment:
Automatic Temp. Compenzation

2) pH meter shouldn't be exposed to direct sunlight because:
Effect on accuracy of the equipment.

3) During calibration of pH meter buffer sol. of pH 7 should be taken first.

4) $pH = -\log[H^+]$ | given: $[H^+] = 10.45 \times 10^{-3}$ M

$\therefore pH = -\log(10.45 \times 10^{-3}) = -\log(10.45) + 3$

$= -1.02 + 3 = 1.98$

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DATE 30/01/25 | Expt: 6 Speed Viscometer (FANN-35S) | SHEET NO. 15

Aim: To determine the viscosity of a given fluid sample using 6 speed viscometer.

~~Procedure~~

~~1) Ensure that the bob~~

Theory: FANN-35S 6-speed viscometer is used to determine the viscosity of a given fluid sample by measuring resistance to rotational motion at different speeds (RPM). Viscosity is a key property in fluid mechanics, particularly in drilling fluid analysis, where it impacts flow behaviour and performance.

Working Principle

The rotor rotates inside the fluid sample, and the torque on the ~~bob~~ bob (attached to the bob shaft) is measured.

The dial readings at different speeds (600 RPM, 300 RPM, etc) help determine viscosity characteristics using standard eqn^s.

→ Apparent viscosity (AV): 600 RPM Reading / 2

→ Plastic viscosity (PV): (600 RPM Reading) − (300 RPM Reading)

→ Yield Point (YP) = (300 RPM Reading) − (PV)

P.R.E.

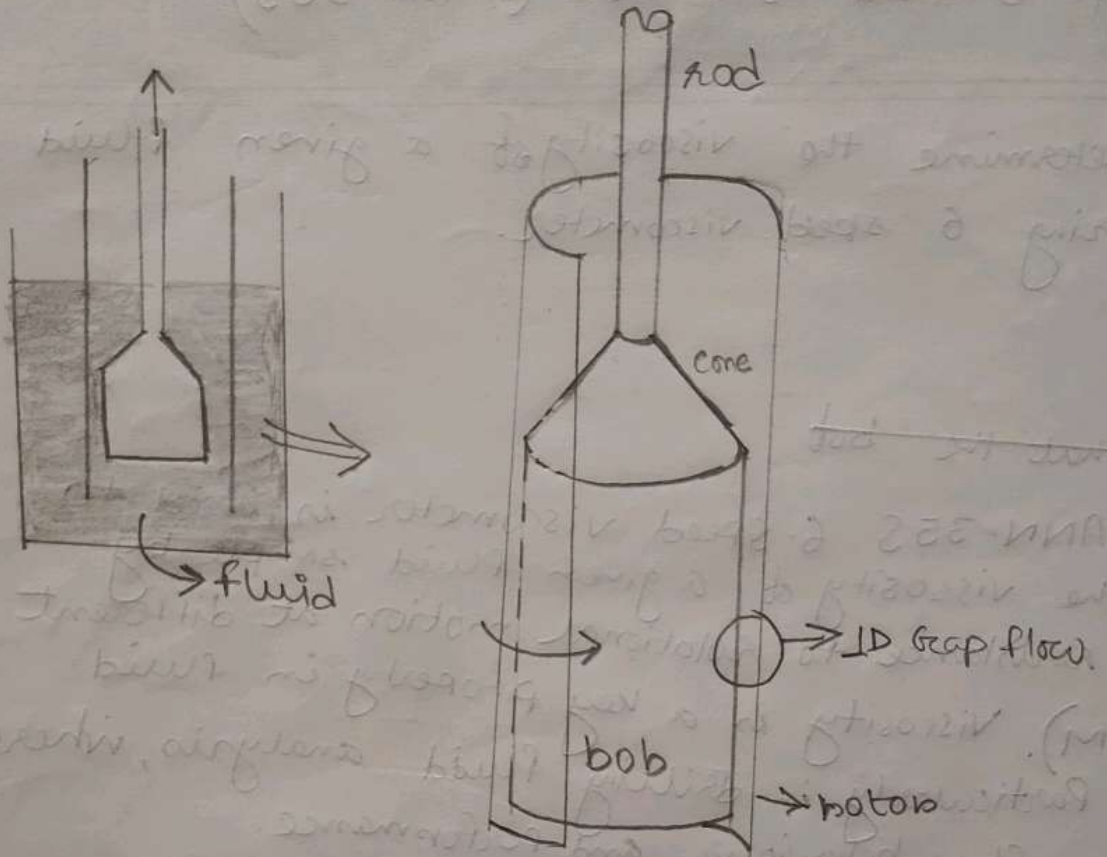

fig: Schematic of FANN-35S Viscometer

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SHEET NO. 16

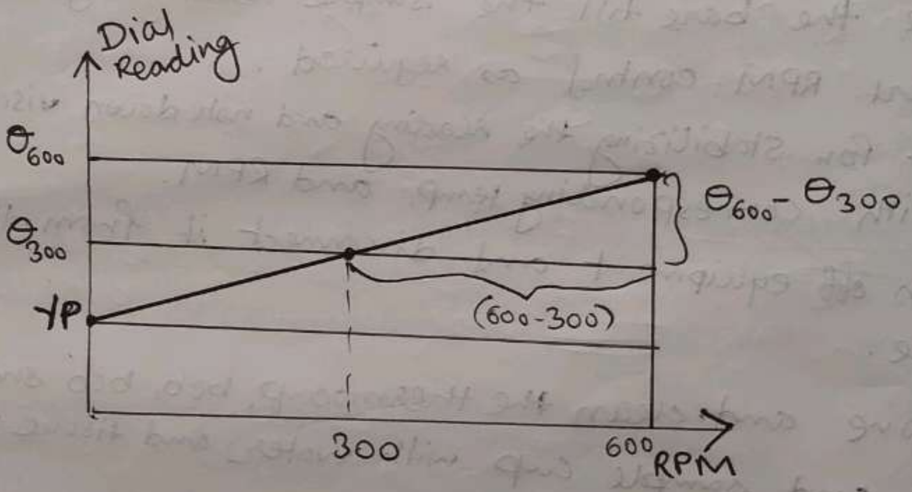
Procedure

1) Ensure the bob and sleeve is attached with the bob shaft and rotor.
2) Connect the instrument to a power source and turn it on.
3) Plug the FANN 35 motor to 110 volt voltage transformer
4) Fill the sample cup to 75% capacity (170ml)
5) If temp. control is required, used a thermocup and thermo-meter
6) Put the sample cup in the base of the instrument and raise the base till the sample touch the given marker
7) Adjust RPM control as required.
8) Wait for stabilizing the reading and note down viscosity in cp with corresponding temp. and RPM.
9) Turn off equipment and disconnect it from the power source.
10) Remove and clean the thermocup, bob, bob shaft, sleeve and sample cup with water and tissue paper.

Observation

@ T = 26°C

$\dot{\gamma}\,(s^{-1})$	RPM	Dial Reading	τ (Pa)	μ (Pa.s)
1021.8	600	98	50.078	0.05
510.9	300	71	36.281	0.07
340.6	200	60	30.66	0.09
170.3	100	45	22.995	0.135
10.218	6	19	9.709	0.95
5.109	3	17	8.687	1.7



Calculation and lab Exercise

→ Derive formular for Yield Point:

$YP(lb/100ft^2) = (300 \cdot RPM)_{DR}$ − Plastic Viscosity

→ from the plot,

~~slope of the line = m =~~ $\frac{\theta_{600} - \theta_{300}}{\cancel{1}/\cancel{300}} = PV$ (in CP)

eqn of line: $\frac{y - \theta_{300}}{\theta_{600} - \theta_{300}} = \frac{x - 300}{600 - 300}$

$\therefore y - \theta_{300} = \frac{PV(x - 300)}{300}$

$\therefore Y.P - \theta_{300} = \frac{PV(0 - 300)}{300} = -PV$

$\therefore \boxed{YP = \theta_{300} - PV}$ (Proved)

→ $\tau(Pa) = D.R \times 0.511$ ↳ spring constant $\dot{\gamma}(sec^{-1}) = RPM \times 1.703$

$\mu(Pa.s) = \frac{\tau}{\dot{\gamma}}$

sample calculation for obs 1: RPM = 600, DR = 98

$\therefore \boxed{\tau = 50.078\ Pa}$

$\dot{\gamma} = 600 \times 1.703 = \boxed{1021.8\ sec^{-1}}$ $\mu = \frac{50.078}{1021.8} = \boxed{0.05\ Pa.s}$

$\boxed{AP = \frac{98}{2} = 49\ CP}$ $\boxed{PV = 98 - 71 = 27\ cP}$ $\boxed{YP = 44\ lb/100ft^2}$

Results

Apparent viscosity = 49 cP   Yield Point = 44 lb/100 ft^2

Plastic viscosity = 27 cP

Precautions

~~Discussion~~

1) Always ensure that the fluid temp. doesn't go beyond 180°F
2) Don't move "Gear Shift knob" if "viscometer Switch" is off.
3) Don't try to rotate the Thermo-cup knob beyond marked position.
4) The rotor, bob and splash guard should be cleansed and dried after each operation.

Conclusion

i) The Fann-35S viscometer accurately determines fluid viscosity at varying rates.

ii) Plastic viscosity indicates particle interaction and friction, while Yield Point reflects the fluid's ability to suspend solids

iii) This experiment highlights the importance of proper temp. control, equipment calibration, and fluid sample Preparation to ensure accurate viscosity readings.

iv) Future improvements could include automated data logging for efficiency and minimizing ~~sea~~ manual reading error.

Assignment

1) Temp. 2) To Protect bob shaft 3) Rotor

4) $P.V. = (DR)_{600} - (DR)_{300} = 36 - 12 = 24\ Pa$

$YP = (DR)_{300} - PV = 12 - 24 = -12\ lb/100ft^2$

DATE 05/02/25 | Exp: 5 | Marsh funnel Viscometer | SHEET NO. 20

Aim: To measure the viscosity of drilling fluid using Marsh Funnel Viscometer.

Theory: Marsh funnel Viscometer is a sample device used to measure the viscosity of drilling fluids. It operates on the principle that the time taken for a known volume of fluid to flow through a standardite funnel correlates with its viscosity.

The key theoretical aspects include:

1) Fluid flow: Rate at which fluid flows through the funnel is inversely proportional to its viscosity.

2) Effective Viscosity: The viscosity measured is an effective viscosity, which is calculated using given formula:

$$\boxed{\mu = \rho(t-25)}$$

μ = effective viscosity in centipoise
ρ = density in g/cm^3
t = quart funnel time in seconds

3) Temp. sensitivity: The viscosity is a function of temp, which is why temp. measurement is part of Procedure.

Procedure:

i) Hold the funnel in an upright Position just above measuring cup.
ii) Block the orifice outlet with your index finger
iii) Pour drilling mud through the screen until fluid level reaches.

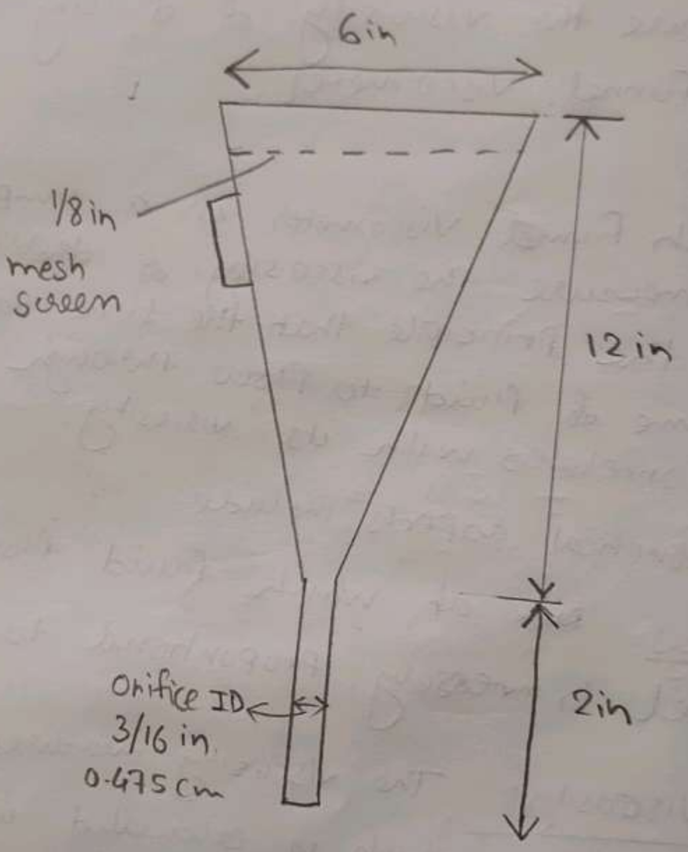


fig: Schematic of Marsh Funnel.

iv) Wait for 15 sec. and ensure your stopwatch is working Properly.

v) Remove your index finger from the orifice outlet and start the stopwatch simultaneously.

vi) Measure the time it takes for drilling mud to fill the marked portion (946ml or 1 quart) of measuring cup.

vii) Measure temp. of fluid in °C or °F.

viii) Record the viscosity in seconds and temp.

Calculations and Result

$$\mu = \rho(t-25)$$

$$\mu = 1.44 \times (44.5 - 25)$$

$$= 28.08 \ \frac{g \cdot s}{c.c.} = 2808 \ cP$$

Observation

$\rho = 12$ ppg $= 1.44\ g/c.c$

$t = 44.5$ sec

$T = 25°C$

Precautions

i) Clean and dry the funnel and cup throughly after each use.

ii) Don't bend or flatten the orifice in the bottom.

Assignment

1) 1500 cm^3

2) Mesh size ≃ 12 inch

3) ~~Shift mt~~ All of the above

4) $\mu = \rho(t-25)$ | ρ = density, t = quart funnel time

i) $\mu_1 = 1.4(135-25) = 154 \frac{g}{cm^3}$ sec.

ii) $\mu_2 = 2.8 \times (110-25) = 238 \frac{g}{cm^3}$ sec.

iii) $\mu_3 = 2.5 \times (28-25) = 7.5 \frac{g}{cm^3}$ sec.

DATE 13/02/25 Ex6: LPLT filter Press SHEET NO. 23

Aim: To determine the fluid ~~viscosity~~ loss using ~~LTLP~~ LPLT API filtration apparatus.

Theory: The LTLP filter press is used to measure the filtration characteristics of drilling fluids under low temperature and low pressure conditions. The test simulates the formation of filter cake on the wellbore well and the invasion of mud filtrate into the formation. In this process while applying a pressure of 100 psi to a mud sample for 30 mins, during which the liquid phase (filtrate) passes through a standardized filter paper. The volume of the filtrate collected and the thickness of the filter cake formed are key indicators of the mud's ability to control the fluid loss and maintain wellbore stability.

Procedure:

1) Prepare the sample according to API specifications. Stir the test sample atleast 10 mins with a high speed mixer.
2) Assemble the filtration cell first to fit the neoprene gasket into the base cap.
3) Fill the cell with approximately 400 ml of mud sample, leaving 1-1.5cm of void space.
4) place the assembly on the frame
5) Install a CO_2 cartridge in the barrel and tighten it.
6) Tighten T-screw to fix assembly in the frame.
7) Position a dry graduated cylinder under the filter tube.

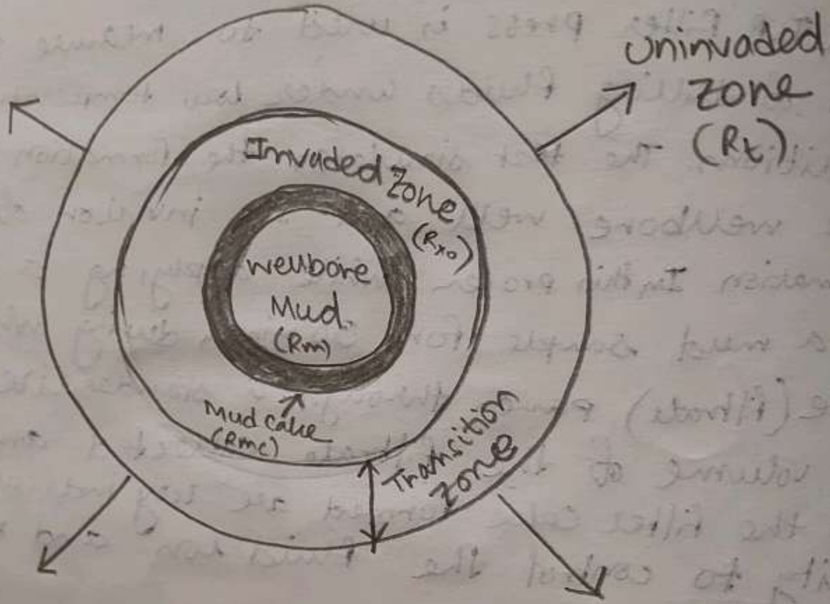


Fig: Top view of Well Bore
(Mud Filtrate Invasion)

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8) Ensure th
9) Run the
10) Carefully
Cake thic

Observation

time(min)
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Result

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8) Ensure the bleeder valve is closed.

9) Run the exp for 30 mins, measuring the filtrate vol in mL.

10) Carefully remove the filter paper and measure the filter cake thickness in mm.

Observation

time (min)	vol. (ml)
7.5	1.7
10	1.9
20	2.7
30	3.5

Result

~~Total fluid loss = 3.5 ml~~

The ~~total~~ fluid loss has been shown in the above table. We have calculated the last two observation by extrapolation. ~~eqn of time:~~

Also, the mud cake thickness = 1mm.

Precaution

1) Before beginning a test, make sure each part of the cell is clean and dry, particularly the screen.
2) Be careful while adjusting the pressure
3) Maximum allowable pressure is 100 psi. Don't operate beyond of this pressure.
4) Should not ~~explore~~ expose the carbon catridges to heat.

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Assignment

1) Residue: solid portion after filtration

2) filtration area = 7.1 inch^2

3) Correct sequence:
Neoprene gasket → Mesh screen → Filter paper → Neoprene gasket

4) Law behind filtration: Darcy's law.

5) Amount of filtrate obtained after 30 mins: $11\ cm^3$
Thickness of filter cake : 2.5 mm

Darcy's Law: $\frac{dV_f}{dt} = \frac{KA\Delta P}{\mu h_{mc}}$

V_f = vol. of filtrate in cm^3
t = time in second
K = average permeability of mud cake in Darcy

$A = 45\ cm^2$ | $\Delta P = 100\ psig = 6.8\ atm$

Amount of filtrate obtained in 30 min = $11\ cm^3$.
Thickness of filter cake = 2.5 mm. = h_{mc}

$$K = \frac{11 \times 1.5 \times 2.5}{45 \times 30 \times 60 \times 6.8} = 7.47 \times 10^{-6}\ \text{Darcy}$$

$= 7.37 \times 10^{-18}\ m^2$

1 Darcy $= 9.8 \times 10^{-13}\ m^2$

P.R.E.

DATE

Ex7: Total Hardness Determination and Chlorides Using Titration

SHEET NO. 26

Aim: Determination of Total Hardness (Ca) and Chlorides in drilling fluid using Titration Method

Theory: Hardness of water or mud filtrate is due to the primarily to the presence of Calcium and magnesium ions. The harder the water, the more difficult for many chemicals to function, particularly bentonite clay. Hardness decreases the efficiency of most polymers. Excessive hardness will cause flocculation of clays in the mud, effecting filtration rates.

Procedure:

Total Hardness Determination:

i) Add 25ml deionized water to titration vessel, then add 1ml (Approx. 10 drops) of Versenate Hardness Buffer solution.

ii) Add 3 drops of versenate Hardness Indicator solution. If Calcium and/or magnesium are present in the deionized water, a wine red color will develop. otherwise, will remain blue.

iii) While stirring, titrate with EDTA (Standard Versenate) until the solution color changes from wine-red to blue.

iv) Note mL of EDTA and calculate Hardness, mg/L

v) Add 1mL filtrate to deionized water. If Ca^{2+}/Mg^{2+} ion are present wine red color will develop. While stirring, titrate with EDTA until color indicator changes to blue.

Observation

~~Total hardness of given filtrate is~~

Amount of EDTA Sol. used for Total Hardness determination: 1.2mL

$\therefore$ Total Hardness (calcium) of given filtrate = 1.2 × 4000
= 4800 mg/L

Now, to find the chlorides present in given drilling fluid –

Apparatus and Chemical / Reagents Required

i) Burette ii) Pipette iii) Wash Bottle iv) Spatula
v) Conical flask vi) Beaker vii) Silver Nitrate Sol.
viii) Potassium Chromate ix) Mud filtrate sample

Significance of the Exp:

→ Chlorides (mg/L) is determined by the salty of water-based mud
→ Chlorides act as a contaminant in freshwater mud system.
→ Salt contamination can cause an increase in viscosity, gel strengths, and fluid loss.
→ If you see changes in the chloride content, it can be indications of drilling in a saline formation, or to take the influx of water in reservoirs.

Procedure

1) Add 1ml of the filtrate to the titration vessel
2) Add 2-3 drops of phenolphthalein to the test sol.
3) If the pink is seen add 0.2N sulphuric acid until pink colour disappears
4) If no pink colour is seen skip the H_2SO_4 addition.
5) Add 5-10 drops of Potassium Chromate to titration vessel.
6) While stirring, titrate with 0.282/0.1 $AgNO_3$ sol. till the first colour disappears

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Observation

Amount of $AgNO_3$ sol. used for titrating filtrate : 10 mL

Calculation

if using 0.282N $AgNO_3$:

Chlorides (mg/L) = 10 × 10,000 = 10 × 10 mg/L

if using 0.1 $AgNO_3$: chlorides (mg/L) = $\frac{10 \times 10{,}000}{2.82}$ = 3546 mg/L

Results

→ Amount of Chlorides present in given filtrate sol = 3546 mg/L

→ Total hardness of a given filtrate sol. = 4800 mg/L

→ Amount of calcium present in given filtrate = 4800 mg/L

Precautions

i) Make sure that there should not be any leakage from burette during titration

ii) Remove the air bubbles from the nozzle of the burette

iii) Use your index finger while pipetting the sol.

iv) Keep your eye in level with the liq. surface while taking the burette reading or while reading the pipette or measuring flask etc.

Asgn: Total Hardness

1) The hardness of mud filtrate is due to the presence of ~~chlorides of~~ Calcium and Magnesium ~~and~~

2) Temporary hardness in water is due to the Presence of carbonates ~~chlorides~~ ~~and~~ of calcium.

3) Permanent hardness of water is removed by all methods except Boiling Process.

4) ~~They~~ Type of hardness: Temporary Hardness, Permanent Hardness.

Temporary hardness is caused by: Presence of dissolved bicarbonates and carbonates of calcium and magnesium.

Permanent hardness is caused by: Presence of sulfates, chlorides, or nitrates of Ca^{2+} and Mg^{2+}.

5) Indicator used in determination of total hardness: Versenate Hardness Indicator.

Asgn Chloride

1) Test is performed on Water Based Mud system

2) The chloride concentration is determined by titration with $AgNO_3$ solution.

3) End point of titration is detected by Potassium Chromate (K_2CrO_4) indicator.

Asgn: Numerical

filtrate vol. = 1 ml

vol. of $AgNO_3$ = 2.1 ml

Normality of $AgNO_3$ = 0.282 N

for chlorides (mg/L): $2.1 \times 10{,}000 = 21000$ mg/L

$$\text{Total Hardness} = \frac{\text{ml of EDTA} \times 4000}{\text{ml of filtrate}} = 0.9 \times 4000 = 3600 \text{ mg/L}$$

$$[Ca^{2+}] = \frac{40}{110} y = 3600 \Rightarrow y = \frac{3600 \times 110}{40} = 9{,}900 \text{ ppm}$$

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$$[Cl^-] = \frac{35}{58} x + \frac{70}{110} y = 21000$$

$$\Rightarrow \frac{35}{58} x + \frac{70}{110} \times 9900 = 21000 \Rightarrow x = 24360 \text{ ppm}$$

$\therefore$ NaCl amount = 24360 ppm
$CaCl_2$ " = 9900 ppm

Total Cl^- = 21000 ppm
Ca^{2+} = 3600 ppm

DATE 06/03/25 | Exp : 8 | Mud filtrate Alkalinity Test | SHEET NO. 31

Aim: To find the alkalinity in the mud filtrate by titration method.

Theory: Alkalinity in drilling mud filtrate is primarily due to the presence of carbonate (CO_3^{2-}), and bicarbonate (HCO_3^-) ions. It plays a crucial role in maintaining pH, preventing corrosion, and ensuring stability of drilling fluids. The alkalinity measurement is typically conducted through acid-base titration, where the filtrate is titrated using phenolphthalein (P) and methyl orange (M) indicators to determine different forms of alkalinity.

Titration Process and Interpretation:

Phenolphthalein Alkalinity (P_f): Measured when pH is above 8.5. It indicates the presence of CO_3^{2-}.

Methyl Orange Alkalinity (M_f): Measured when pH is between 4.3 and 8.5. It represents the presence of CO_3^{2-} and HCO_3^-.

Chemical Rxn involved:

1) Reaction of sodium carbonate with sulfuric Acid at higher pH (>8.5):

$$Na_2CO_3 + H_2SO_4 \rightarrow NaHSO_4 + NaHCO_3$$

The change in color of phenolphthalein from Pink to colorless indicates the conversion of all carbonate into bicarbonates.

2) Reaction of Sodium Bicarbonate with Sulfuric Acid (4.3 < pH < 8.5)

$$NaHCO_3 + H_2SO_4 \rightarrow NaHSO_4 + H_2CO_3$$

This conversion reaction is confirmed with the change in color of the filtrate in methylene blue titration test.

Apparatus : pipette, burette, wash bottle, spatula, beaker, conical flask

Chemical/Reagents: mud, filtrate, distilled water, phenolphthalein indicator, methyl orange indicator, sulphuric acid

Procedure:

→ Filtrate Alkalinity (P_F):

1) Transfer 1ml of LPLT filtration filtrate into the titration vessel using appropriate pipette.
2) Add 3 drops of phenolphthalein indicator sol.
3) if sol. turns pink, titrate (drop by drop) with 0.02 N sulphuric acid until pink color just disappears.
4) Report the phenolphthalein alkalinity of filtrate P_F, as the number of cm^3 of 0.02N acid req. per cm^3 of filtrate.
5) Filtrate (liq. only) P_F alkalinity can indicate the presence of carbonate ions in most mud systems.

→ Filtrate Alkalinity (M_F):

1) To the same sample that has been titrate to the P_F endpoint, add 3 drops methyl orange indicator solution.
2) Titrate with 0.02N sulphuric acid (drop by drop) while stirring until the indicator changes from pale yellow to pinkish orange.
3) Report the methyl orange Alkalinity, M_F as the total cm^3 of 0.02N acid per cm^3 of filtrate req. to reach the methyl orange endpoint. The M_F is recorded as the total vol. of acid req. per cm^3 of filtrate. (The total vol. indicates the cm^3 of acid in the P_F titration plus the cm^3 of acid M_F titration).

Observation

For filtrate 1

Amount of 0.02N sulphuric acid reqq. for filtrate Alkalinity (P_F): 0.8 ml. = T_1

Amount of 0.02N sulphuric acid req. for filtrate Alkalinity (M_F):

$$T_1 + T_2 = (0.8 + 0.7) \text{ ml} = 1.5 \text{ ml}$$

For filtrate 2

$P_F = 0 = T_1$ $\qquad$ $T_2 = 1.1$

$M_F = 1.1$

Calculation :

For filtrate 1: $P_F = 0.8$ $\qquad$ $M_F = 1.5 \approx 2P_F$

$\therefore$ Carbonate (CO_3^{2-}) = $1200 \times P_F = 1200 \times 0.8 = 960$ ppm

only Carbonate is Present.

For filtrate 2: $P_F = 0$, $\quad M_F = 1.1$

$\therefore$ $[CO_3^{2-}] = 0$ $\qquad$ $[HCO_3^-] = 1220 \times M_F$

$= 1220 \times 1.1 = 1342$ ppm

Precautions:

i) Make sure there shouldn't be any leakage from burette

ii) Remove air bubbles from nozzle of ~~bottle~~ burette.

iii) Use your index finger while pipetting the sol.

Assignment

Gr: C

$T_2 = 0.7 mL$

$T_1 = 0.8 mL$

1) $P_F = 0.8$ $M_F = 0.8 + 0.7 = 1.5$

2) ~~$P_H = 2$~~ $M_F \cong 2P_F$

$\therefore [HCO_3^-] = 0$ only carbonate (CO_3^{2-}) will be there.

3) $[CO_3^{2-}] = 1200 \times P_F = 960$ ppm

4) $[HCO_3^-] = 0$ ppm.

5) So, there is no bicarbonate present in the given filtrate solution.. There is carbonate only, which can increase alkalinity, which may help control corrosion in drilling equipment but can also lead to scale formation in pipes and equipment.

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DATE 20/03/25 | Exp 9: Cation Exchange Capacity | SHEET NO. 35

Aim: To determine the cation exchange capacity of a given drilling fluid sample using Methylene Blue Test.

Theory:

> Methyelene Blue capacity gives an estimate of the total cation exchange capacity (CEC) of the solids in the drilling fluid.

> Methylene Blue is a cationic dye, which strongly attracts to negatively charged sites on clays.

> The test, therefore, is a measure of the reactive clay concentration of a water-based drilling fluid.

> Increasing MBTs usually an indication of an increase in low gravity solids concentration.

Procedure:

i) Pipette 10ml of deionised water into the Erlenmeyer flask

ii) Use syringe to accurately measure 2ml of freshly agitated drilling fluid. Transfer this portion into flask.

iii) Boil this mixture on hotplate for 10 min. Don't boil to dryness.

iv) Add deionized water to dilute this mixture to approx. 50 ml.

v) Dip a stirring rod into the flask while the solids are suspended, and then remove it. Place a drop of the liquid on filter paper. The initial endpoint is reached when dye appears as a blue ring surrounding the dyed solid.

vi) When blue ring spreading from the spot appears, shake the flask an additional 2mins. Place another drop on the filter paper. If the blue ring appears again, the final endpoint has been reached.

If blue ring doesn't appear, continue the titration until a drop taken after two minutes shows the blue tint.

Observation

→ Amount of Drilling fluid = 2 ml.
→ Amount of Methylene Blue indicator = 2.5 ml.

Calculation:

→ CEC = Amount of Methylene Blue (ml) / Amount of drilling fluid (ml)
= 2.5/2 = 1.25

→ Bentonite, lb/bbl drilling fluid = 5 * Methylene Blue capacity
= 5*2.5 = 12.5 ml.

Precautions

1) Make sure there is no leakage from burette during titration.
2) Remove the air bubbles from nozzle of burette
3) Use your index finger while pipetting the solution.

Assignment | Grp → C

1) Negatively Charged
2) Hydrogen peroxide used to remove organic materials, such as polymers, and thinning agents, which would react with MB dye.
3) Temperature
4) Cation Exchange Capacity (CEC) : $\frac{3\,ml}{5\,ml} = 0.6$

Aim: To measure the sand content in a given drilling fluid using sand content kit.

Procedure

i) Fill the glass measurement tube up to "mud line" with mud
ii) Use wash bottle to add water to next scribed mark
iii) Place thumb over mouth of glass measure tube and shake vigorously
iv) Pour mixture onto the clean 200 mesh screen.
v) Add more water into the glass measuring tube and shake
vi) Again pour onto 200 mesh screen
vii) Repeat this procedure until glass measure tube is clean
viii) After that wash with water the retained sand present at top of 200 mesh screen.
ix) Place funnel on top of screen assembly
x) Slowly invert assembly and insert tip of funnel into glass measuring tube mouth.
xi) Spraying a fine spray of fresh water at the back side of the 200 mesh screen that retained sand back into the glass measuring tube mix with water.
xii) Allow clean sand to settle.
xiii) Read the record vol. percent sand from graduations on the glass measuring tube.

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SHEET NO. 38

Observation and Result

Sand content was found to be = 0.01

Assignment

1) Excessive sand content may harm: All of the above
2) Top most line-marking of glass measuring tube: Mud line
water line
3) Water is used for cleaning purpose only ~~if~~ in case of water based mud.
4) All of the above

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DATE | Exp10: Retort Kit | SHEET NO.

Aim: To determine the volumes of water, oil and solids in drilling fluid using Retort kit.

Working Principle : The Retort Kit

-> This instrument is used to separate water, oil and solid components in drilling fluid samples, and determine their volume percentage of the drilling fluid

-> It heats a known vol. of drilling fluid sample in the sample cup, evaporating the liquid components in a distiller, condenser to cool it, collecting in a graduated measuring cylinder.

-> Obtain the contents of water, oil and solid components in the drilling fluid sample

Components of Retort Kit :

-> Graduated cylinder, 50ml
-> Spatula
-> Corkscrew
-> Square Bar retort Wrench
-> Pipe Cleaners
-> Steel wool
-> Drill Twist
-> Brushes - wire and stainless steel
-> High Temp. Lubricant
-> Wetting Agent.

Working Procedure : Retort Kit

-> Clean and dry the retort assembly and condenser. Condenser Passage should be cleaned using a pipe cleaner

-> Mix the mud sample throughly to ensure homogeneity, being careful not to entrain any air or gas entrapment will cause erroneously high retort solids due to reduced mud sample volume.

-> Use a clean syringe to take a sample of the mud to be tested

-> Fill retort cup slowly to avoid air entrapment. Lightly tap side of cup to excel air. Place lid on the cup. Rotate lid to obtain a proper fit. Be sure a small excess of mud flows out of the hole in the lid. Wipe off any ~~acean~~ excess mud without wicking any of the samples from inside the cup. Wipe the cup clean with rough cloth.

-> Pack retorts body with steel wool.

-> Place the retort assembly inside the retort cup. Apply Never-Seez (high temperature lubricant) to the threads of the retort cup. with lid in place, hand tighten retort cup onto retort body.

-> Apply never-seez to threads of stem and attach condenser.

-> Plug cord into 110-volt outlet. Allow the retort to run for a min^m of 45 mins.

-> Unplug retort and allow cooling of retort cup to reach less than 100°F. Disassemble and clean retort assembly and condenser.

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Observation

Total liq. volume in the receiver = 35 ml

oil volume = 0 ml

water volume = 35 ml

Calculation

1) vol. of retort cup, $V_{rc} = \dfrac{\text{Weight of water (g)}}{\text{Density of water } (g/cm^3)} = 50$ ml

~~= 35 ml.~~

2) vol. Percent of water, $V_W = \dfrac{100 \times 35}{50} = 70\ \%$

3) vol. Percent of oil, $V_o = \dfrac{100 \times 0}{V_{rc}} = 0\ \%$

4) vol percent of solids, $V_S = 100 - (V_W + V_o) = 30\ \%$

Now, $\cancel{V_o}^{0} + V_{Bn} + V_{LGS} + V_{WE} = 1$

$\Rightarrow V_{Bn} + V_{LGS} + V_{WE} = 1$

$\cancel{\rho_o V_o}^{0} + \rho_{Bn} V_{Bn} + \rho_{LGS} V_{LGS} + \rho_{WE} V_{WE} = MW = 15$ (Given)

$\dfrac{V_{Br}}{V_W} = \dfrac{100/\rho_{Br}}{100 - wt\%}$

Given, MW = 15 PPg

Salinity = 150,000 PPm (15%)

Salt table, $\rho_{Br} = 1.116\ g/c.c.$

$\therefore V_{Br} = 0.7 \times \left(\dfrac{100/1.116}{100-15}\right) = 0.738$

$\therefore V_{LGS} + V_{WE} = 1 - 0.738 = 0.262$ — (1)

And, $\rho_{Br} V_{Br} + \rho_{LGS} V_{LGS} + \rho_{wt} V_{wt} = MW = 15$

$\Rightarrow 1.116 \times 0.738 + \rho_{LG} 2.7 V_{LGS} + 4.2 V_{wt} = \frac{15}{8.345}$ — (2)

Solving (1) and (2) —

$V_{LGS} = 0.084 \quad V_{wt} = 0.178$

~~AS~~ $ASG = \frac{\rho_{LGS} V_{LGS} + \rho_{wt} V_{wt}}{V_{LGS} + V_{wt}} = 3.714$

RESULTS:

→ vol. percent of brine = 0.738 = 73.8 %.

→ vol. percent of oil = 0 %.

→ vol. percent of LGS = 8.4 %

→ vol. percent of HGS = 17.8 %.

→ ASG of given sample = 3.714

PRECAUTIONS:

→ The assembly should be cooled to a temp. less than 100°F (37.8°C) from any previous usage

→ The threads on the retort should be visually inspected before use of any sign of damage

→ The steel wool should be changed out after every test to prevent solids from building up

→ Regular retort should ~~be~~ NEVER be used for drilling fluids containing formate brines

Assignment

Group C

1) Never-seez
2) Average specific Gravity
3) ~~True~~ False
4) Retort sample cup
5) Condenser : cools water and oil vapors into liq. for measurement.

Pipe Cleaner: Removes residue from the condenser passage to ensure unobstructed vapor flow.

Numerical

WBM → 16 lbm/gal ~~having NaCl brine~~

Brine salinity → 150000 ppm NaCl (15 wt%)

Retort water content : 33 ml (66% of 50 ml sample)

$\rho_{Lgs} = 2.7$ $\rho_{Barite} = 4.2$

$\rho_{Br} = 1.116\ g/cm^3$ (from salt table)

$$V_w = \frac{\text{vol. of water}}{\text{vol}_{rc}} = \frac{33}{50} = 0.66$$

$$\frac{V_{Br}}{V_w} = \frac{100/\rho_{Br}}{100 - wt\%} = \frac{100/1.116}{100-15} = 0.696$$

Using, $\cancel{V_o}^{0} + V_{Br} + V_{Lgs} + V_{wt} = 1$ [$V_o = 0$ ∵ WBM]

$V_{Lgs} + V_{wt} = 0.304$ — (i)

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$$\rho_0 \cancel{V_0}^{0} + \rho_{Br} V_{Br} + \rho_{LGS} V_{LGS} + \rho_{wt} V_{wt} = MW$$

$$\Rightarrow 1.116 \times 0.696 + 2.7 \times V_{LGS} + 4.2 V_{wt} = \frac{16}{8.345} \quad \text{—} (2)$$

Solving (1) and (2) ———

$V_{LGS} = 0.0908 \qquad V_{wt} = 0.2132$

$\therefore \; V_{LGS} = 0.0908 \qquad V_{HGS} = V_{wt} = 0.2132 \qquad V_{brine} = 0.696$