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Underbalanced Drilling Test Kit

Part No. 144-82

Instruction Manual

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Ver. 1.1

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Table of Contents

Components	2
Chloride Ion Determination	3
Mud Alkalinity Determination	5
Filtrate Alkalinity Determination	6
Total Hardness Determination	8

Components

Equipment:

- #110-10 Plastic Marsh Funnel Viscometer
- #110-20 OFI Plastic Measuring Cup; 1,000 mL
- #152-00 Hamilton Beach Mixer with Container; Single Spindle; 110V
- #152-40 Stainless Steel Container for Mixers; 30 oz.
- #153-25-2 Hand-Crank Centrifuge for 15 mL Tubes; 4-Place Head and Shields

Supplies:

- #147-53 pH Strips; 0 - 14; Package of 100; Qty: 5
- #153-19 Pyrex 8080 Centrifuge Tube; 15 mL; Qty: 4
- #153-26 Polyethylene Titration Dish
- #153-28 4" Stirring Rod; Polyethylene
- #153-29 Glass Tip Syringe; 2 cc; Qty: 2
- #153-29-1 Glass Tip Syringe; 5 cc; Qty: 2
- #153-29-2 Glass Tip Syringe; 10 cc; Qty: 2
- #153-34 Glass Pipet; 1 mL \times 1/100 mL; Qty: 2
- #153-36 Glass Pipet; 2 mL \times 1/10 mL; Qty: 2
- #153-38 Glass Pipet; 5 mL \times 1/10 mL; Qty: 2
- #153-40 Glass Pipet; 10 mL \times 1/10 mL; Qty: 4
- #153-42 10 mL Fast-Release Pipet Filler
- #153-52 Hydrometer Set in Plastic Carrying Case

Reagents:

- #205-02 Versenate Hardness Indicator Solution; 2 oz.
- #205-04 *Versenate Hardness Buffer Solution; 2 oz.; UN #2672
- #205-06 Versenate Hardness Titration Solution; 1 mL - 2 EPM; 8 oz.
- #205-08 Versenate Hardness Titration Solution; 1 mL - 20 EPM; 4 oz.
- #206-02 Deionized Water; 16 oz.
- #215-00 Potassium Chromate Solution; 2 oz.
- #220-00 Phenolphthalein Solution; 2 oz.
- #230-04 *Sulfuric Acid; N/50; 16 oz.; UN #2796
- #245-00 Bromocresol Green Methyl Red Indicator Solution; 2 oz.
- #265-00 Silver Nitrate Solution; .001g; 0.0282N; 8 oz.
- #265-13 Silver Nitrate Solution; 0.1g; 0.282N; 4 oz.
- #285-00 Calcium Carbonate; 2 oz.

Case:

- #144-82-03 Case with Custom Foam Inserts

Chloride Ion Determination

This test measures the soluble chloride ion concentration in the mud filtrate. The chloride can come from sodium chloride, calcium chloride, or potassium chloride. Also, for the titration to work correctly the pH of the filtrate needs to be only weakly basic (pH = 8.3). This is the reason for the first step in the procedure. There are two chemical reactions taking place simultaneously during the titration:

1. $\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl}$
2. $2 \text{Ag}^+ + \text{CrO}_4^{2-} \rightarrow \text{Ag}_2\text{CrO}_4$

The first reaction, the formation of silver chloride, accounts for the appearance of the white specs or milky appearance during the titration. The formation of the silver chromate, which is red, will not start until all the chloride ions are tied up as silver chloride. The silver nitrate will then react with the chromate from the potassium chromate indicator to form silver chromate. So, for the above two reactions to occur, the filtrate needs to be weakly basic (pH = 8.3). High pH will precipitate silver oxide.

Procedure

1. Pipette 1.0 mL of filtrate or sample to be tested into titration dish.
2. Add 2 - 3 drops of phenolphthalein indicator to the filtrate.
3. If a pink color appears, titrate with N/50 sulfuric acid until the color changes from pink to that of the original sample. If no pink color appears, the test can be continued.
4. If needed, add 25 - 50 mL of distilled water. This serves to dilute the dark color of a deeply colored filtrate. It does not influence the test in any way if there are no chlorides present in the distilled water. If any water other than distilled water is used, the chloride in it must be accounted for by titration before adding the filtrate to it.
5. Add 10 - 15 drops of potassium chromate indicator to give the filtrate a bright yellow color.
6. Add silver nitrate from a pipette dropwise, stirring continuously with a stirring rod, until the sample just turns from yellow to an orange-red.

Calculation

1. Chloride Concentration (mg / L) = $\frac{1000^* \times \text{Silver Nitrate Volume (mL)}}{\text{Filtrate Sample Volume (mL)}}$
2. Chloride Concentration (ppm) = $\frac{\text{Chloride Concentration (mg / L)}}{\text{Specific Gravity of Filtrate}}$
3. Salt (NaCl) Concentration (ppm) = Chloride Concentration (ppm) \times 1.65

*Use 1,000 as the multiplier for 0.0282N (.001gram) chloride ion solution, and 10,000 for the 0.252N (.01 gram) AgNO₃.

Remarks

1. The chloride test may be run on the same samples used in the P_f determination if the M_f test was not performed.
2. Avoid contact with silver nitrate. Wash immediately with water if silver nitrate gets on skin or clothing.
3. The end point of the reaction is when the silver chromate is first formed. It is reddish in color. When using the weak silver nitrate, the end point is approached very gradually. Therefore, the formation of the silver chromate can be seen by a color change from yellow to orange-red. If the strong silver nitrate is used, the end point is approached much more rapidly. Hence the early formation of the silver chromate and its orange-red color may be missed due to the larger amounts of silver nitrate being added. So the color change will go from yellow to red, as soon as the red color is seen, the titration is complete.
4. White lumps of silver chloride form when titrating high concentrations of salt. This should not be taken for the end point.
5. If the chloride ion concentration is less than about 10,000 ppm, the weak silver nitrate solution should be used. If it is greater than 10,000, the strong silver nitrate solution should be used.

Mud Alkalinity Determination

This test measures the alkalinity of the whole mud (P_m). When used along with the filtrate alkalinity determination, the amount of excess lime present in lime muds can be determined.

Procedure

1. Measure 1 mL of a freshly stirred sample of mud into a titration dish using a syringe.
2. The syringe should have a capacity of at least 3 mL. Draw at least two mL and preferably three mL of mud into the syringe. Remove any air bubbles from the sample. Then inject a one mL sample by pushing the plunger from the 3 mL graduation to the 2 mL graduation or the 2 mL graduation to the 1 mL graduation, but never from 1 mL to the bottom of the syringe.
3. Dilute the mud in the dish with 25 - 50 mL of distilled water.
4. Add no more than 4 or 5 drops of phenolphthalein.
5. If the sample does not change color, report the P_m as 0.
6. If the sample turns pink, titrate rapidly with N/50 sulfuric acid until the pink color disappears.

Calculations

Report the phenolphthalein alkalinity of the mud, (P_m) as the number of mL of N/50 sulfuric acid added to discharge all the pink color.

Remarks

1. If the mud sample is deeply colored from chemical thinners and the color change is hard to see, use $\frac{1}{2}$ mL of mud and report the P_m as the volume of N/50 sulfuric acid added to discharge the pink color doubled.
2. If 0.1N sulfuric acid is used, the P_m is reported as volume of N/50 sulfuric acid added to 1 mL of mud times 5.

Filtrate Alkalinity Determination

These tests are designed to measure the alkalinity contributions from soluble mud constituents. The P_f and M_f test together can be used to get an estimate of the hydroxide ion (OH^-), the bicarbonate ion (HCO_3^-) and the carbonate ion (CO_3^{2-}) concentration. Also the P_f can be used with the P_m to determine the excess amount of lime present in a lime base mud.

Procedure (P_f)

1. Pipette 1 mL of filtrate into a clean titration dish. (Do not dilute with distilled water.)
2. Add more than 2 - 3 drops of phenolphthalein. If no color develops, the P_f is 0 (and pH is less than 8.3). If a pink color develops, add N/50 sulfuric acid through a pipette until the color changes pink to that of the original sample.

Calculations (P_f)

1. The P_f is the amount of N/50 sulfuric acid required to discharge the pink color.

2. Excess lime content (lb/bbl) = $\frac{P_m - P_f}{4}$

Procedure (M_f)

1. To the same sample used for the P_f determination, add 2 -3 drops of brom cresol green-methyl red indicator.
2. Again add N/50 sulfuric acid until the color changes from a blue-green to a light pink color.

Calculations (M_f)

1. Report the M_f alkalinity of the filtrate as the total volume of N/50 sulfuric acid required to reach the end point, including the volume required to reach the P_f end point.
2. An estimation of hydroxide ion, bicarbonate ion, and carbonate ion concentrations can be determined using the following table.

Test Results

M_f Calculations

If $P_f = M_f$
(Indicates OH^- only)

$$P_f \times 340 = \text{mg/L OH}^-$$

If $2P_f - M_f > 0$
(Indicates OH^- and CO_3^{2-})

$$\begin{aligned} (2P_f - M_f) \times 340 &= \text{mg/L OH}^- \\ 2(M_f - P_f) \times 600 &= \text{mg/L CO}_3^{2-} \end{aligned}$$

If $2P_f = M_f$
(Indicates CO_3^{2-} only)

$$M_f \times 600 = \text{mg/L CO}_3^{2-}$$

If $2P_f - M_f < 0$
(Indicates CO_3^{2-} and HCO_3^-)

$$\begin{aligned} 2P_f \times 600 &= \text{mg/L CO}_3^{2-} \\ (M_f - 2P_f) \times 1,220 &= \text{mg/L HCO}_3^- \end{aligned}$$

If P_f is very low or unreadable
(Indicates HCO_3^- only)

$$M_f \times 1220 = \text{mg/L HCO}_3^-$$

Nomenclature

P_f = Phenolphthalein end point of the filtrate sample using N/50 sulfuric acid

M_f = Methyl orange or brom cresol green-methyl red end point of filtrate sample using N/50 Sulfuric Acid

OH^- = Hydroxide ion

CO_3^{2-} = Carbonate ion

HCO_3^- = Bicarbonate ion

Total Hardness Determination

The test measures the amount of soluble magnesium and calcium ions in the mud filtrate. When measured simultaneously, the result is called total hardness. The calcium ion concentration can be determined separately and when it is subtracted from the total hardness, the magnesium ion concentration can be determined. A different buffer-indicator combination not included in this kit would be needed to perform this test. The buffer is calcium buffer solution, #205-14; the indicator is calcium indicator powder, #210-00.

Procedure

1. Add 50 mL of deionized water to a titration dish.
2. Add 20 to 40 drops (1 - 2 mL) of hardness buffer solution to the dish.
3. Add 10 to 15 drops (0.5 - 0.75 mL) of hardness indicator solution to the mixture.
4. If a wine-red to a purple color develops, the deionized water used in step 1, contains hardness. Add total hardness titrating solution drop wise while stirring until the water turns to a bright blue color.
5. Using the pipette, add 1 mL of filtrate into the titration dish and stir. A wine red to purple color will again develop if calcium and/or magnesium is present in the filtrate solution.
6. Add total hardness titrating solution, stirring continuously, until the sample again turns a bright blue color.

Calculations

The total hardness is calculated as:

1. EPM Calcium and Magnesium = $2 \times$ Titration Solution Volume (mL)

If the solution strength is 1 mL = 2 EPM Hardness

2. EPM Calcium and Magnesium = $20 \times$ Titration Solution Volume (mL)

If the solution strength is 1 mL = 20 EPM Hardness

3. Usually the magnesium ion content is negligible so the total hardness is reported as being all calcium. Therefore:

ppm Calcium = EPM Total Hardness \times 20

EPM = Equivalent Parts Per Million

Remarks

1. If the mud filtrate is deeply colored, a 0.5 mL sample can be used instead of a 1.0 mL sample. The total hardness is determined as before except that the results have to be doubled to account for using 0.5 mL of mud filtrate instead of the required 1.0 mL.
2. This test actually measures several metal ions simultaneously: calcium, magnesium, lead, iron, aluminum, zinc, and manganese. Usually the results are reported as being all from calcium and/or magnesium since the quantity of the other metal ions is usually negligible.
3. At least 25 mL of distilled water must be used. Also, the buffer must be mixed into the water before the indicator is added.
4. Before the filtrate is added, the water with the buffer and indicator must be blue. If it is not, total hardness titrating solution must be added until it is.
5. When zeroing the distilled water, it is very important to add *just enough* total hardness solution until the water turns blue. Any excess that is added will influence the results of the total hardness determination.