Water Based Drilling Fluids

When Hardness Titration solution (EDTA) is added to water or mud filtrate that contains both Calcium and Magnesium, the EDTA first combines with the Calcium. Calcium alone can be determined with EDTA when the pH of the sample is sufficiently high so that magnesium is precipitated out as the Hydroxide, and an indicator specific to Calcium is used. Several indicators will give color changes when all of the Calcium has been complexed by the EDTA at a pH in the range of 12 to 13. An end point obscured by dark organic components may be treated by oxidizing with Sodium Hypochlorite.

Equipment:

#153-14	Graduated Cylinder, 50 ml x 1.0 ml, glass
#153-34	Pipette, 1.0 ml x 1/100 ml, glass
#153-36	Pipette, 2.0 ml x 1/10 ml, glass
#153-38	Pipette, 5.0 ml x 1/10 ml, glass
#153-40	Pipette, 10.0 ml x 1/10 ml, glass
#153-51	Beaker, 250 ml, glass
#168-01	Hot Plate, with Thermostat, 115 volt
	(Required if the filtrate is colored)
	or
#168-01-1	Hot Plate, with Thermostat, 230 volt
	Reagents:
#205-12	Titration sol'n (EDTA), Ver. Hard., 0.01M, 400 Mg/L, 16 oz
#205-14	Calcium Buffer sol'n (NaOH, 1N), 2 oz
#206-02	Distilled Water, 16 oz
#210-00	Calver® II Indicator Powder, 10 gram
#230-25	Acetic Acid, Glacial, 8 oz (UN2789)
#261-00	Sodium Hypochlorite sol'n, 8 oz (UN1791)
#261-50	Masking Agent, 1:1:2 mixture by volume of
	Triethanolamine: Tetraethylenepentamine : Water, 16 oz

Procedure:

1. Add at least 1.0 mL of sample to the Beaker.
   If the filtrate is colorless or is only slightly colored, omit steps 2 - 5.

2. Add 10.0 ml of Sodium Hypochlorite solution and mix.

3. Add 1.0 ml of Glacial Acetic Acid and mix.

4. Boil the sample for 5 minutes and maintain the sample volume by adding distilled water as required during boiling. Boiling removes excess Chlorine and this may be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continue boiling. A sufficiently boiled sample will have a pH of 5.0.

5. Cool the sample and wash the sides of the beaker with distilled water.

6. With distilled water, dilute the sample to approximately 50 ml.

7. Add 10 to 15 ml of Calcium Buffer solution, or sufficient Buffer solution to produce a pH of 12 - 13.

8. Optional: 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) is a suitable masking agent.

9. Add sufficient Calver® II Indicator powder (0.1 to 0.2 grams) to produce a pink to wine-red color if Calcium is present. Too much indicator will obscure the endpoint. Note: Adding several drops of Methyl Orange Indicator solution may improve the visibility of the endpoint.

10. While stirring, titrate with Hardness Titration solution (EDTA), to the end point. Calcium indicators produce a Red-to-Blue change and the endpoint is best described as that point where additional Titration solution will produce no further Red-to-Blue color change. The Titration solution volume will be used in the calculation procedure.

Calculation:

$$\textit{Calcium, mg/L} = \frac{\textit{400 (Titration sol'n, ml)}}{\textit{Sample Volume, ml}}$$

Oil Based Drilling Fluids:

Equipment:

#153-53 Magnetic Stirrer, with Stirring Bar
#153-64 Syringe, 5 ml, disposable,
#154-75 Scoop, brass, 4 inch length
#297-05 Mason Jar, with Lid, 16 oz

Reagents:

#205-17-3 Titration sol'n. (EDTA), 4000 mg/L Ca^{+2}, 0.1M, 200 EPM, 16 oz
#206-04 Distilled Water, gal
#210-00-1 Calver® II Indicator powder, 100 gram
#260-05 Sodium Hydroxide Buffer sol'n, 1.0 N, 8 oz (UN1824)
#280-30 Arcosolv PNP, gal. (UN1993)

Procedure:

1. Add 100 mls of Arcosolv PNP solvent to a 16 oz jar
2. Fill a new 5 ml syringe with whole mud past the 3 ml mark.
3. Displace 2.0 mls of oil mud into the jar with the Arcosolv solution.
4. Cap the jar and shake vigorously by hand for 1 minute.
5. Add 200 mls of distilled water to the jar.
6. Add *6.0 mls of Sodium Hydroxide Buffer sol'n, 1 N, to the mixture.
7. Add *0.7 to 1.0 gram of Calver® II Indicator powder. (Note: 5 scoops = 0.7 grams)
8. Recap the jar tightly and shake vigorously again for 2 minutes. Set the jar aside for a few seconds to allow the upper and lower phases to separate. If a reddish color appears in the aqueous (lower) phase, this indicates that calcium is present.
9. Agitate by placing the jar on a magnetic stirrer and drop in a stirring bar.
10. Titrate very slowly, adding Titration solution (EDTA) drop by drop from a pipette, while stirring only fast enough to agitate the aqueous (lower) phase without remixing the upper and lower phases. A distinct color change from the reddish color to a blue-green occurs at the end point. Note the volume of Titration solution added.
11. Use the mls of titration solution required to reach the end point to calculate the whole mud calcum.

Calculation:

$$Ca_{oil\ mud} = \frac{Ca,\ mg}{oil\ mud\ sample,\ L} = \frac{4000\ (0.1\ M\ \ EDTA,\ mls)}{2.0\ mls}$$

Quantities have been revised since the last API issue - "R.P. 13B-2**", Third Edition, Dated Feb. 1998.*

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