

Thermo. Titr. Application Note No. H-010

Title: Determination of Calcium in Drilling Fluids

Scope: Determination of calcium in fluids employed in drilling oil and gas wells.

Principle: Treatment of the sample with $\text{NH}_3/\text{NH}_4\text{Cl}$ buffer, and titration to a single exothermic endpoint with tetra-sodium EDTA (Na_4EDTA).

Reagents: Na_4EDTA titrant, 1 mol/L. The Na_4EDTA solution may be prepared by adding a stoichiometric amount of A.R. NaOH to the appropriate amount of A.R. $\text{Na}_2\text{H}_2\text{EDTA}$ prior to making to volume. It may be standardized against a solution of Zn^{2+} prepared from A.R. metallic zinc. Alternatively, it may be prepared from commercial A.R. Na_4EDTA .

$\text{NH}_3/\text{NH}_4\text{Cl}$ buffer solution. Dissolve 17.5 g A.R. NH_4Cl in 172 mL A.R. conc. NH_3 soln. and make to 250 mL with deionised water.

Method: Basic Experimental Parameters:

Data rate (per second)	10
Titrant delivery rate (mL/min.)	1
No. of exothermic endpoints	1
Data smoothing factor	40

Procedure:

Weigh accurately approximately 6g of homogenized drilling fluid into a clean, dry titration vessel. Add 10mL $\text{NH}_3/\text{NH}_4\text{Cl}$ buffer and 15mL deionized water. Titrate to a single exothermic endpoint

Results:

Analysis of formulated drilling fluids for North Sea exploration:

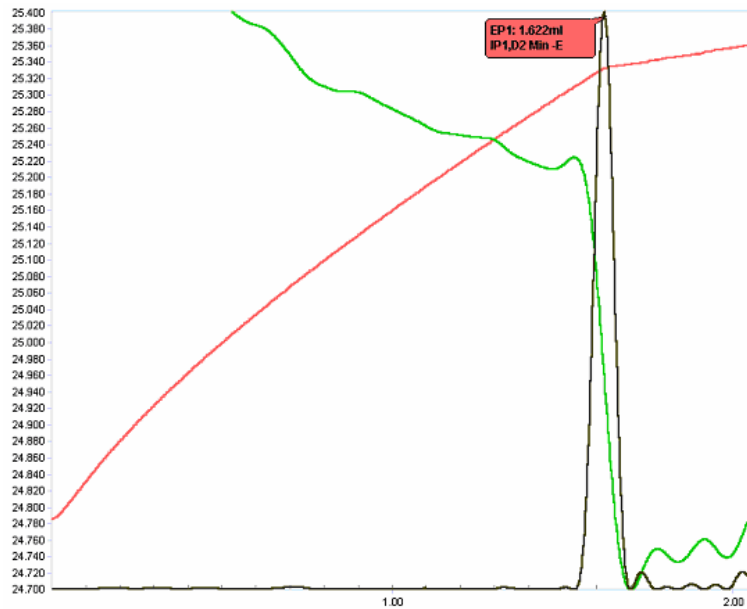
Sample I.D.	Sample Mass, g	Titre, mL Na ₄ EDTA	%CaCl ₂ w/w
Sample A	3.6033	1.076	3.01
	5.4227	1.622	3.03
Average			3.02
Sample B	6.4324	2.498	3.94
	7.1617	2.811	3.98
Average			3.96

Calculation:

$$\%CaCl_2 \text{ w/w} = \frac{((Titre - blank) \times FW \text{ CaCl}_2 \times M \text{ Na}_4\text{EDTA} \times 100)}{(sample \text{ mass, g} \times 1000)}$$

$$\%CaCl_2 \text{ w/w} = \frac{((2.498 - 0.010) \times 110.99 \times 0.9180 \times 100)}{(6.4324 \times 1000)}$$

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Green = first derivative curve

Black = second derivative curve