LaFarge Chemical Method No. 42: Free CaO[1]

Introduction

This method specifies a longer time than the other methods, including those now in ASTM Method for Chemical Analysis of Hydraulic Cement (C 114). It has two unique features in that the heating of the extraction involves 30 min in a 110°C oven and then, after suitable preparation steps, determines actual CaO extracted by means of a complexometric titration using ethylenediaminetetraacetate (EDTA) at high pH.

Editor

Reagents, Equipment, and Supplies

- Ethylene glycol, anhydrous
- Absolute ethyl alcohol
- EDTA solution, standardized against CaO
- 2 *N* NaOH solution
- Triethanolamine: water, 1:2 by volume
- c.HCL:water, 1:1 by volume
- Chrome blue-black indicator, 2 g in solution of 50-mL triethanolamine + 50-mL water
- pH indicator paper, 11.0 to 14.0
- 110°C oven
- Vacuum pump (water) with check valve in line
- 500-cm^3 vacuum Erlenmeyer flask
- Fritted disk Pyrex Bucher funnel, fine, 65-mm diameter (Fisher Catalogue No. 10-358K)
- Magnetic stirrer
- Pyrex beakers, 100-mL capacity, with watch glass
- Plastic wash bottle for alcohol, 100 to 200 mL capacity

Table 1 illustrates the LaFarge Chemical Method No. 42 for Free calcium oxide (CaO).

[1] Canada Cement LaFarge Ltd., 506 Cathcart, Montreal 111, Quebec, Canada.

TABLE 1—*Application of complexometry to determination of free lime in cement and clinker after dissolution in glycol.*

Steps	Key Points	Explanation
1 putting free CaO into solution	material ground finer than 100-μm (No. 140) sieve	to help dissolution
	2-g material	
	100-mL beaker, dry	water liberates CaO from silicate causing high result for lime
	25-mL ethylene glycol, mix	
	cover with small watch glass	to condense glycol vapor
	30 min in 110°C oven	necessary to dissolve all the free lime
2 filtering the solution	before filtering, shake if necessary to suspend material stuck to bottom of beaker. Add a little alcohol to rinse if necessary; dry	to help clean beaker and to get better filtration
	500-mL conical suction flask, with check valve	to avoid water running back into flask
	filter through Pyrex fritted fine Bucher funnel (Fisher 10-358K) and rinse with alcohol; dry	no water in contact with material
	filter all solution under vacuum, rinse beaker and funnel with about 40-mL alcohol	no water in contact with material
	repeat further two times	
	filtrate must be completely clear	if particles pass into filtrate free CaO result will be high
	after using once or twice, clean the funnel with acid	fine fritted funnel retains very fine particles
3 preparing solution for pH test	to the flask containing filtrate, add several drops 1:1 HCL and 300-mL distilled water	to avoid hydrolysis of CaO on adding water
	conical flask placed on magnetic stirrer, no heating	better observation of color change if solution is dilute
	add 15-mL 1:2 triethanolamine from very clean test tube or other container	to complex Fe_2O_3 and Al_2O_3 possibly present, which could interfere with CaO determination
	add 40-mL 2 *N* NaOH from very clean container, to give pH of 12.5 to 13.5	if pH is less than 12.5 CaO result will be high, because of complexing of MgO
	check pH with pH paper	if pH is above 13.5, color change will not be as clear

TABLE 1—*Continued*

4 determination of CaO	indicator chrome blue-black about 8 to 9 drops added to give red-violet color	red-violet color obtained in presence of free Ca ions
	titrate with EDTA solution through violet to a clear blue color	clear blue coloration indicates disappearance of calcium ions
	read mL of EDTA used	
	add 3 or 4 drops more of EDTA solution	at least 3 drops are necessary to cause a change in color
	if blue color has not changed, first reading of mL EDTA is final reading; otherwise record reading for final addition when blue color is stable	
	if end point is not clear, check pH again	
5 calculation of free CaO	N mL of EDTA solution used for titration	1-mL EDTA equivalent to t grams CaO
	t is EDTA factor	
	$\% \text{ CaO} = \frac{Nt \times 100}{2}$	2-g material used for determination

NOTE: For very high or very low concentrations of free CaO, sample weight of material may be varied; for w grams sample, $\% \text{ CaO} = Nt \times 100/w$.