

Section IV

Free Lime Rapid Methods

The present version of ASTM Method for Chemical Analysis of Hydraulic Cement (C 114-85) contains two methods for the determination of free lime in cement and clinker. Method A is a modified Franke method wherein the free lime extracted in an ethylacetate-isobutyl alcohol solvent is titrated with perchloric acid. Method B uses a glycerin-alcohol (ethanol SDA 2B or isobutyl alcohol are specified, but could be replaced by absolute alcohol or ethanol SDA 3A) solvent with a strontium nitrate accelerator to extract free lime, which is then titrated with ammonium acetate. Both of these are rapid methods in which the sample and solvent are boiled for 15 to 20 min, filtered, and titrated. The latter method is a modification of the classical alcohol-glycerol extraction (often referred to as "ASTM," but which last appeared in the 1980 *Annual Book of ASTM Standards*) in which the extraction was boiled for 2 h or longer. Thus, "ASTM" is now much faster than before and is no longer the lengthy procedure that chemists and operators used to curse. References to "ASTM" in the methods that follow refer, of course, to the old method.

The three methods that follow are variations on a theme. They are all based on rapid ethylene glycol extraction with a subsequent titration for free lime as published by Schlapfer and Bukowski in 1933 (see Method 2 for references). It may also interest some to note that current Method A in C 114 is based on the 1941 work of Franke while Method B, modified from the old "ASTM" method, is based on the 1926 publication of Lerch and Bogue.

These methods, as with those in the *Annual Book of ASTM Standards*, do not distinguish between calcium oxide and calcium hydroxide. Consequently, reference to "free lime" is probably correct only when dealing with fresh clinker. In all other cases, "free lime" is undoubtedly a mixture of calcium oxide and hydroxide or calcium hydroxide alone. This has been mentioned many times but is worth repeating again here.

Editor