

Guidelines for Free Lime calibration in clinker
Collection of clinker samples and analysis

1. The customer should collect 5 to 8 clinker samples from his kiln (100g per sample). In order to obtain a wide range of free lime concentrations it is advised to collect clinker samples during a kiln start-up or a kiln stoppage. This allows to get a really wide range of concentrations of free lime, typically from 2.5% down to 0.5% or lower.
2. After collecting the clinker samples, each of them should be stored separately in a small plastic bag or plastic container and kept tight until the installation of the instrument.
3. After installation of the ARL 9900 instrument, the customer should grind the clinker samples with his own preparation machines and preparation conditions. For each sample the resulting powder will be split into two portions.
 - a. the first portion will be used by the customer to determine the free lime content by wet chemical analysis. For improved accuracy, it is recommended that the wet chemical analysis is done three times on the same clinker powder. The average of the three concentration values is taken as “known” value for calibration of the integrated XRD system.
 - b. the second portion will be pressed into a pellet and measured for the free lime peak on the ARL 9900 instrument. The resulting intensity will be used in the MVR program to adjust the basic calibration curve done in the factory with synthetic standards (in case such a basic calibration has been ordered).

In conclusion, clinker/cement samples for free lime determination should be prepared carefully in order to achieve correct and reliable results. Particular care should be taken to protect them from exposure to air or humid atmosphere to avoid hydration of free lime into calcium hydroxide as this will affect the calibration results and quantitative analysis of free lime.

Note 1: Although there is a standard method (ASTM) of doing wet chemical analysis, different customers use different methods (ASTM method takes too much time for a production lab).

Note 2: After grinding the samples to a particle size of about 50 microns they must be kept in a dessicator or air tight package, because any absorption of humidity will produce transformation of a part of the free lime to calcium hydroxide: $\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2$

Note 3: The wet chemistry analysis by titration of ethylene glycol will provide the sum of both free lime (CaO) and calcium hydroxyde (Ca(OH)_2) present in the clinker sample. The XRD analysis on the contrary determines only and specifically the content of free lime (CaO). Hence there can be a discrepancy if the wet chemistry and XRD calibration are not done during the same day on clinker samples that are carefully kept from hydrating.

Setting-up samples

One setting-up sample is required for maintenance of the calibration curve over time. No standard samples are delivered with this calibration.