

## Instrument: TGA801

### Determination of Split Loss on Ignition in Cement

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#### Introduction

In the cement industry, use of the term Loss on Ignition (LOI) normally refers to a mass loss in a sample heated to up to a maximum of 1,000 °C. The cement industry utilizes the LOI method to determine the water content and/or carbonation in the cement as these reduce the quality. A high loss on ignition can indicate pre-hydration and/or carbonation, which may be caused by improper and prolonged storage or adulteration during transport or transfer by suppliers. Testing cement using the LOI method can be used as an indicator which can be employed to monitor and improve the quality of the final product.

Split Loss analysis involves determining the mass loss of a sample at two specific temperatures (Typically 550 °C and 950 °C for cement samples). Using the Split Loss method, an estimate of the carbon dioxide content of the sample can be determined by calculating the difference between the residue masses. Split Loss on Ignition Testing ensures that the cement used at a site is of the correct composition to meet the safety protocols and requirements of the customer.

Thermogravimetric analysis (TGA) is an analytical technique in which changes in sample mass due to changes in physical and chemical properties of materials is measured as a function of temperature and/or time. TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss (or gain) due to decomposition, oxidation, or loss of volatile materials such as moisture. The LECO TGA801 is a macro thermogravimetric analyzer that allows up to 19 samples to be analyzed simultaneously.

#### Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Typically, samples should be ground to a fineness of <0.5 mm.

#### Accessories

621-331 Ceramic Crucibles, 621-011-507 Double Sided Spoon

**Sample Mass** ~1.0 g

#### Method Reference

ASTM C114 Standard Test Methods for Chemical Analysis of Hydraulic Cement

**Analysis Time** ~4.5 h

#### General Method Parameters

Crucible Type	Ceramic
Minimum Crucible Weight	20.0000
Maximum Crucible Weight	30.0000
Crucible Density	3.00
Lid Density	3.00
Sample Type	Cement
Sample Density	3.1
Minimum Sample Weight	0.8000
Maximum Sample Weight	1.2000

#### Method Step Parameters

	Step 1	Step 2
Step Type	Custom	Custom
Step Name	LOI @ 550 °C	LOI @ 950 °C
Cooling Option	Active	Active
Crucible Lids	No	No
Start Temperature	25.0 °C	550.0 °C
End Temperature	550.0 °C	950.0 °C
Ramp Rate	10 °C/min	10 °C/min
Hold Time	120 min	120 min
Maximum Time	240 min	240 min
Atmosphere	Air	Air
Flow Rate	10.0 LPM	10.0 LPM
Final Weight	At End of Step	At End of Step

## Method Equation Parameters

	Step 1	Step 2
Calculation Type	Custom	Custom
Measurement Type	Mass Ratio	Mass Ratio
Calculation Name	LOI @ 550 °C	LOI @ 950 °C
Enable Calibration	Disabled	Disabled
Calculation	$((\text{Initial Mass} - \text{LOI @ 550 °C Mass}) \div \text{Initial Mass})$	$((\text{LOI @ 550 °C Mass} - \text{LOI @ 950 °C Mass}) \div \text{Initial Mass})$

## Procedure

1. Create and/or select a method, using the Method Step Parameters listed above, following the procedure outlined in the TGA801 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGA801 Instruction Manual.

## Typical Results

	Initial Mass (g)	% LOI @ 550 °C	% LOI @ 950 °C	% Total LOI
Portland Cement	1.0082	0.63	1.81	2.44
NIST SRM 633a	0.9934	0.67	1.78	2.45
LOI between 550 °C and 950 °C = 1.805% ±0.037%	1.0061	0.65	1.77	2.42
Total LOI = 2.46% ±0.084%	1.0029	0.73	1.82	2.55
	0.9979	0.64	1.76	2.40
	<b>Avg =</b>	<b>0.67</b>	<b>1.79</b>	<b>2.45</b>
	<b>s =</b>	<b>0.04</b>	<b>0.02</b>	<b>0.06</b>
Portland Cement	1.0035	1.27	0.79	2.06
NIST SRM 1888b	1.0039	1.28	0.77	2.05
LOI between 550 °C and 950 °C = 0.85% ±0.069%	0.9999	1.28	0.76	2.04
	1.0443	1.27	0.78	2.05
	1.0701	1.29	0.78	2.07
	<b>Avg =</b>	<b>1.28</b>	<b>0.78</b>	<b>2.06</b>
	<b>s =</b>	<b>0.01</b>	<b>0.01</b>	<b>0.01</b>

