

Free Moisture and LOI in Hydrated Lime

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Instrument: TGA701

Introduction

Thermogravimetric analysis (TGA) is an analytical technique in which changes in physical and chemical properties of materials are measured as a function of temperature and/or time. TGA is commonly used to determine selected characteristics of materials that exhibit mass loss or gain due to decomposition, oxidation, or loss of volatile materials such as moisture. Macro TGA systems use gram-size samples to allow more accurate mass change measurements in heterogeneous materials. The automation capability of the TGA701 allows up to 19 samples to be analyzed simultaneously in one analysis batch.

Hydrated lime is made from the hydration of quicklime by replacing oxides, produced from the kiln firing of limestone, with hydroxides that chemically combine with water to form a hydrate. Hydrated lime has several uses including as a flocculant in the wastewater treatment process as well as multiple uses in the food industry.

The free moisture in hydrated lime is water absorbed but not bound chemically in the structure of the material. The hydrated water can be liberated by heating the sample above the decomposition temperature of Calcium Hydroxide ($\text{Ca}(\text{OH})_2$). Further heating of the sample will liberate absorbed CO_2 from the sample. The measure of liberated hydrated water and CO_2 is commonly referred to as Loss on Ignition (LOI) and is a quality control process parameter classifying the degree of calcination of the lime.

Sample Preparation

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

Accessories

621-331 Ceramic Crucibles, 611-844 Spoon

Sample Mass ~1.0 g

Analysis Time ~2 hours

Method General Parameters

Crucible Type	Ceramic
Crucible Density	3.00
Cover Density	3.00
Sample Type	Lime
Sample Density	1.5



System Parameters

Significant Digits	F6
Barometric Pressure	760
Cover Open Half	800
Cover Open Full	450
Furnace Over Temp.	1200
Increment Sample Names	Disable
Crucible Locations	Auto Find
Temperature Check	False
Alarm On Errors	True
Cover Option	Half Open
Carousel Option	Weigh Position
Batch Mode	False
Enable Lid Dispenser	False

Balance Parameters

Readability	4
Balance Environment	Stable
Balance Constancy	Absolute Weight (g)
Minimum Weight	0.8000
Maximum Weight	1.2000
Zero Deviation	0.0005
Retake Initial Weights	False

Method Step Parameters

Parameter	Free Moisture	LOI @ 600 °C	LOI @ 1000 °C
Covers	No	No	No
Start Temp. (°C)	25	120	600
End Temp. (°C)	120	600	1000
Ramp Rate	15	20	20
Ramp Time (h:m)	0:06	0:24	0:20
Hold Time (h:m)	0:00	0:00	0:20
Total Time (h:m)	0:06	0:24	0:40
Max Time (h:m)	0:00	0:00	0:00
Atmosphere	Nitrogen	Nitrogen	Nitrogen
Flow Rate	Medium	Medium	Medium
Window	3	3	3
Comparator	0.0005 g	0.0005 g	0.0005 g
Final Weight	At Constancy	At Constancy	At Constancy

Method Equation Parameters

*See page 2

Procedure

1. Create and/or select a method, using the parameters for the method provided, following the procedure outlined in the TGA701 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGA701 Instruction Manual.

*Method Equation Parameters

Equation Name	Equation Text
Free Moisture	$(([\text{Initial Mass}] - [\text{Free Moisture Mass}]) / [\text{Initial Mass}]) * 100$
LOI @ 600 °C	$(([\text{Free Moisture Mass}] - [\text{LOI @ 600 °C Mass}]) / [\text{Initial Mass}]) * 100$
LOI @ 1000 °C	$(([\text{LOI @ 600 °C Mass}] - [\text{LOI @ 1000 °C Mass}]) / [\text{Initial Mass}]) * 100$

Typical Results

Results represent three separate batches using two different TGA701 instruments with the same operator.

Instrument 1

Sample	Mass (g)	% Free Moisture	% LOI @ 600 °C	% LOI @ 1000°C	Batch
Hydrated Lime	1.0207	0.77	23.1	1.26	1
n = 21	1.0144	0.77	23.0	1.35	1
	1.0574	0.76	23.1	1.30	1
	1.0015	0.77	23.0	1.38	1
	1.0060	0.76	23.0	1.32	1
	1.0204	0.76	23.1	1.30	1
	1.0051	0.77	23.1	1.28	1
	1.0093	0.76	23.0	1.32	2
	1.0149	0.78	23.1	1.29	2
	1.0423	0.77	23.1	1.25	2
	1.0311	0.76	23.1	1.28	2
	1.0178	0.76	23.0	1.33	2
	1.0276	0.77	23.1	1.27	2
	1.0040	0.77	23.1	1.34	2
	1.0693	0.78	23.1	1.33	3
	1.0811	0.77	23.0	1.32	3
	1.1516	0.77	23.0	1.29	3
	1.0234	0.78	23.0	1.29	3
	1.0275	0.75	23.0	1.31	3
	1.0124	0.77	23.1	1.26	3
	1.0327	0.77	23.1	1.26	3
Avg =		0.77	23.0	1.30	
s =		0.01	0.03	0.03	

Instrument 2

Sample	Mass (g)	% Free Moisture	% LOI @ 600 °C	% LOI @ 1000°C	Batch
Hydrated Lime	1.0069	0.76	23.1	1.28	1
n = 21	1.0367	0.75	23.1	1.25	1
	1.0373	0.75	23.1	1.24	1
	1.0044	0.76	23.1	1.25	1
	1.0139	0.78	23.1	1.22	1
	1.0526	0.78	23.0	1.25	1
	1.0101	0.76	23.0	1.28	1
	1.0019	0.75	23.1	1.27	2
	1.0226	0.74	23.1	1.26	2
	1.0082	0.76	23.0	1.26	2
	1.0106	0.75	23.1	1.23	2
	1.0195	0.76	23.0	1.25	2
	1.0235	0.75	23.1	1.21	2
	1.0313	0.75	23.1	1.23	2
	1.1078	0.78	23.1	1.26	3
	1.0452	0.76	23.1	1.24	3
	1.0414	0.76	23.1	1.26	3
	1.0056	0.78	23.1	1.22	3
	1.0332	0.77	23.0	1.26	3
	1.0836	0.76	23.1	1.24	3
	1.0356	0.79	23.0	1.23	3
Avg =		0.76	23.1	1.25	
s =		0.01	0.02	0.02	

