Application Note

Instrument: TGA801

Thermogravimetric Analysis of Flue Gas Desulfurization (FGD)

LECO Corporation; Saint Joseph, Michigan USA

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 either 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.

Flue Gas Desulfurization (FGD) gypsum is a byproduct of some industrial coal fired flue gas scrubbing systems and is a widely used product in the building material and cement industries. Gypsum ($CaSO_4 \cdot 2H_2O$), calcium sulfite ($CaSO_3 \cdot ½\ H_2O$), limestone ($CaCO_3$), free moisture, and moisture in hydrates can all be present in the FGD solids material. Determination and monitoring of these constituents is important for quality control and characterization of the FGD gypsum product.

Sample Preparation

Samples must be of uniform consistency to produce suitable results. Typically, samples should be ground to a fineness of <0.5 mm (>35 mesh).

Accessories

621-331 Ceramic Crucibles, 611-844 Spoon

Sample Mass ~1.0 g

Analysis Time ~4 h

General Parameters

Crucible Type	Ceramic
Minimum Crucible Weight	20.0000
Maximum Crucible Weight	30.0000
Crucible Density	3.00
Lid Density	3.00
Sample Type	Solids
Sample Density	~2.6
Minimum Sample Weight	0.8
Maximum Sample Weight	1.2

Method Step Parameters

Parameter	*Step 1*	*Step 2*	*Step 3*	*Step 4*	*Step 5*	*Step 6*
Step Type	Custom	Custom	Custom	Custom	Custom	Custom
Step Name	Free Moisture	$CaSO_4*2H_2O$	$CaSO_3*.5H_2O$	$CaSO_3*.5H_2O$-2	$Ca(OH)_2$	CO_2
Cooling Option	Active	Active	Active	Active	Active	Active
Crucible Lids	No	No	No	No	No	No
Start Temperature	25.0 °C	50.0 °C	240.0 °C	400.0 °C	400.0 °C	550.0 °C
End Temperature	50.0 °C	240.0 °C	400.0 °C	400.0 °C	550.0 °C	950.0 °C
Ramp Rate	1 °C/min	17 °C/min	17 °C/min	1 °C/min	10 °C/min	25 °C/min
Hold Time	15 min	15 min	5 min	15 min	25 min	15 min
Maximum Time	180 min	180 min	180 min	180 min	180 min	180 min
Atmosphere	Nitrogen	Nitrogen	Nitrogen	Oxygen	Nitrogen	Nitrogen
Flow Rate	7.0 L/min	7.0 L/min	7.0 L/min	7.0 L/min	7.0 L/min	7.0 L/min
Final Weight	At Constancy	At End of Step	At End of Step	At End of Step	At End of Step	At End of Step
Constancy Window	9 min	–	–	–	–	–
Constancy Level	0.0005 g	–	–	–	–	–

Method Equation Parameters*

Parameter	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9
Calculation Type	Custom	Custom	Custom	Custom	Custom	Custom	Custom	Custom	Custom
Measurement Type	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio	Mass Ratio
Calculation Name	Free Moisture	$CaSO_4$* $2H_2O$	CaSO3* $.5H_2O$	$Ca(OH)_2$	CO_2	$CaCO_3$	Ash	Calculated Ash	Calculated Fly Ash
Enable Calibration	Disabled	Disabled	Disabled	Disabled	Disabled	Disabled	Disabled	Disabled	Disabled
Calculation	(((Initial Mass - Free Moisture Mass))/Initial Mass)	(((((Free Moisture Mass-$CaSO_4$* $2H_2O$ Mass))/Initial Mass))*100)) *((((100/((100-Free Moisture))))/20.93)))	((((($CaSO_4$* $2H_2O$ Mass-$CaSO_3$* $.5H_2O$ Mass))/Initial Mass))*100)) *((((100/((100-Free Moisture))))/6.98)))	(((($CaSO_3$* $.5H_2O$-2 Mass - $Ca(OH)_2$ Mass))/Initial Mass)	(((($Ca(OH)_2$ Mass - CO_2 Mass))/Initial Mass)	(CO_2 * 2.274)	(CO_2 Mass/Initial Mass)	(((((($CaSO_4$* $2H_2O$/100))*79.08))+ ((((($CaSO_3$* $.5H_2O$/100))*105.53)))+ ((((Ca(OH)$_2$/100))*75.68)))+ ((((CaCO$_3$/100))*56.03)))	(Ash - Calculated Ash)

Due to the complexity of the method calculations, please contact LECO Service for import files.

Procedure

1. Create and/or select a method, using the table of 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

Name	Description	Initial Mass (g)	% Free Moisture	% $CaSO_4$* $2H_2O$	% $CaSO_3$* $.5H_2O$	% $Ca(OH)_2$	% $CaCO_3$	% Ash	% Calculated Ash	% Calculated Fly Ash
NIST SRM 2429	FGD Gypsum	1.0098	0.00	97.59	2.59	0.06	0.45	79.14	80.21	<0.01
Gypsum Purity		1.0106	0.02	97.54	2.62	0.07	0.38	79.15	80.17	<0.01
97.4%		1.0186	0.01	97.63	2.58	0.06	0.38	79.15	80.19	<0.01
		1.0020	0.03	97.56	2.70	0.06	0.40	79.13	80.27	<0.01
		1.0118	0.05	97.53	2.51	0.05	0.36	79.15	80.02	<0.01
		1.0248	0.04	97.59	2.41	0.08	0.33	79.15	79.96	<0.01
		1.0175	0.02	97.64	2.31	0.07	0.37	79.13	79.91	<0.01
		1.0327	0.05	97.57	2.55	0.06	0.42	79.11	80.13	<0.01
		1.0349	0.00	97.72	2.63	0.06	0.43	79.12	80.34	<0.01
		1.0060	0.04	97.62	2.39	0.06	0.44	79.11	80.01	<0.01
		Avg =	***0.03***	***97.60***	***2.53***	***0.06***	***0.40***	***79.13***	***80.12***	***<0.01***
		s =	***0.02***	***0.06***	***0.12***	***0.01***	***0.04***	***0.02***	***0.14***	***<0.01***

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