Thermogravimetric Analysis of Flue Gas Desulfurization (FGD)

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TGA701

Organic Application Note

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 ($CaSO_3 \cdot \frac{1}{2} H_2O$), 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.

Accessories

621-331 Ceramic Crucibles, 529-048 Ceramic Covers, 611-844 Spoon

Sample Mass

~1.0 g

Analysis Time

~4 hours

Method General Parameters

Crucible Type	Ceramic
Crucible Density	3.00
Cover Density	3.00
Sample Type	Solids
Sample Density	1.50

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 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 Step Parameters**

Parameter	**Free Moisture**	**$CaSO_4$*$2H_2O$**	**$CaSO_3$*$0.5H_2O$**	**$CaSO_3$*$0.5H_2O$-2**	**$Ca(OH)_2$**	**CO_2**
Covers	No	No	No	No	No	No
Start Temp.	25	50	240	400	400	550
End Temp.	50	240	400	400	550	950
Ramp Rate	1	17	17	0	10	25
Ramp Time	0:25	0:11	0:09	0:00	0:15	0:16
Hold Time	0:15	0:15	0:05	0:15	0:25	0:15
Total Time	0:40	0:26	0:14	0:15	0:40	0:29
Max Time	0:00	0:00	0:00	0:00	0:00	0:00
Atmosphere	Nitrogen	Nitrogen	Nitrogen	Oxygen	Nitrogen	Nitrogen
Flow Rate	Medium	Medium	Medium	Medium	Medium	Medium
Window	3	0	0	0	0	0
Comparator[†]	0.0005 g	100.0000 g	100.0000 g	100.0000 g	100.0000 g	100.0000 g
Final Weight	At Constancy	At End of Step	At End of Step	At End of Step	At End of Step	At End of Step

*Method Equation Parameters***

Free Moisture
(([Initial Mass]-[Free Moisture Mass])/[Initial Mass])*100

$CaSO_4$*2H_2O
(((((([Free Moisture Mass]-[$CaSO_4$*2H_2O Mass])/[Initial Mass])*100)*(100/(100-[Free Moisture])))/20.93)*100

$CaSO_3$*0.5H_2O
(((((([$CaSO_4$*2H_2O Mass]-[$CaSO_3$*0.5H_2O Mass])/[Initial Mass])*100)*(100/(100-[Free Moisture])))/6.98)*100

$Ca(OH)_2$
(([$CaSO_3$*0.5H_2O-2 Mass]-[$Ca(OH)_2$ Mass])/[Initial Mass])*100

CO_2
(([$Ca(OH)_2$ Mass]-[CO_2 Mass])/[Initial Mass])*100

Ash
([CO_2 Mass]/[Initial Mass])*100

$CaCO_3$
[CO_2]*2.274

Calculated Ash
((([$CaSO_4$*2H_2O]/100)*79.08)+((([$CaSO_3$*0.5H_2O]/100)*105.53)+((([$Ca(OH)_2$]/100)*75.68)+((([$CaCO_3$]/100)*56.03)

Calculated Fly Ash
[Ash]-[Calculated Ash]

NOTES

- The factors 20.93 and 6.98 in the above equations represent the % moisture in $CaSO_4$•2$H_2$0 and $CaSO_3$•0.5$H_2$0 respectively
- The factor 2.274 in the above equation converts the CO_2 mass loss to the mass of $CaCO_3$
- The factors 79.08, 105.53, 75.68 and 56.03 in the above equation convert the various calcium compounds to the oxide and sulfate forms in the ash

Typical Results

Name	Initial Mass	Free Moisture	$CaSO_4$*2H_2O	$CaSO_3$*.5H_2O	$Ca(OH)_2$	%$CaCO_3$	Ash	Calculated Ash	Calculated Fly Ash
NIST SRM 2429	1.0411	0.01	97.57	2.68	0.08	0.31	79.18	80.22	<0.01
Gypsum Purity	1.0648	0.01	97.59	2.47	0.06	0.30	79.19	79.99	<0.01
97.4%	1.0024	0.00	97.60	2.44	0.08	0.30	79.20	79.98	<0.01
	1.0103	-0.01	97.61	2.46	0.08	0.30	79.19	80.02	<0.01
	1.0267	0.01	97.59	2.59	0.08	0.28	79.19	80.13	<0.01
	Avg.	***0.00***	***97.59***	***2.53***	***0.08***	***0.30***	***79.19***	***80.07***	***<0.01***
	Std Dev	***0.01***	***0.01***	***0.10***	***0.01***	***0.01***	***0.01***	***0.10***	***-***

Delivering the Right Results

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