Sulfur in Biomass and Biofuel

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruSpec® S Module

Sample Preparation

A representative, uniform sample is required.

Accessories

528-203 Crucibles, 502-321 Com-Cat™

Calibration Samples

LECO Sulfur in Oil samples; 502-420, 502-419, 502-418, 502-417, or other suitable reference materials.

*Method Parameters**

External Sulfur Analysis Parameters

Furnace Temperature	1350°C
Analysis Stabilize Comparator	2
Manual Load Baseline Delay Time (seconds)	5
Baseline Time (seconds)	1
Auto Detect Data Missed Time (seconds)	4
Endline Time seconds	5
Minimum Analysis Time (seconds)	60
Comparator Level	0.30 %
Conversion Factor	1.00
Significant Digits	5

System Configuration

Gas Conservation Timeout (minutes)	15
Auto Increment Sample Name	Disable
Nominal Mass	1.0000
Lance Delay Time (seconds)	20
Lance Limits	50000

**Refer to TruSpec/Sulfur Module Operator's Instruction Manual for Method Parameter definitions.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing a three to five ~0.25 gram coal samples.
3. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Add ~1 gram of 502-321 Com-Cat into a 528-203 Crucible.
   c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on the display, click OK, and then slide crucible into the combustion tube until it reaches crucible stop. *Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.*
   d. Repeat steps 3a through 3c a minimum of three times.
   e. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate/Drift Correct.
   a. Weigh ~0.1 gram calibration sample into a 528-203 Crucible containing ~0.5 gram of 502-321 Com-Cat, enter mass and sample identification into Sample Login (F3).
   b. Cover with ~0.5 gram 502-321 Com-Cat.
   c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, click OK, then slide crucible into the combustion tube until it reaches crucible stop. *Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.*
   d. Repeat steps 4a through 4c a minimum of three times for each calibration/drift sample used.
   e. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
5. Analyze Samples/Biofuels.
   a. Weigh ~0.1 gram sample into 528-203 Crucible containing ~ 0.5gram of 502-321 Com-Cat, enter mass and sample identification into Sample Login (F3).
   b. Cover with ~0.5 gram of 502-321 Com-Cat.
   c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, click OK, and then slide crucible into the combustion tube until it reaches crucible stop. *Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.*
6. Analyze Samples/Biomass.
   a. Weigh ~0.15 gram sample into 528-203 Crucible and enter mass and sample identification into Login (F3).
   b. Add ~1 gram of 502-321 Com-Cat to the crucible and thoroughly mix with sample.
   c. Initiate the analysis sequence (F5). When "Load Sample into Furnace" message appears on screen, click OK and then slide crucible into the combustion tube until it reaches crucible stop. *Alternately, place crucible onto the appropriate position of carousel if equipped with autoloader system.*

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Organic Application Note

Delivering the Right Results

Typical Results

Sample	*Mass*	*% S*
Wood Chips #1	0.1545	0.138
	0.1555	0.134
	0.1551	0.153
	0.1548	0.137
	0.152	0.134
	***X* =**	***0.139***
	***s* =**	***0.008***
Wood Chips #2	0.1553	0.061
	0.1539	0.053
	0.1531	0.058
	0.1523	0.054
	0.1555	0.057
	***X* =**	***0.057***
	***s* =**	***0.003***
Biofuel	0.0985	0.01
	0.0971	0.009
	0.1017	0.012
	0.0989	0.01
	0.0995	0.011
	***X* =**	***0.010***
	***s* =**	***0.001***

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