

Sulfur in Plant, Feed, Grain, and Flour

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruSpec[®] S Module

Sample Preparation

A representative, uniform sample is required. Samples should be ground to pass a No. 100 (150 micron) sieve.

Accessories

528-203 Crucibles, 502-321 Com-Cat[™].

Calibration Samples

NIST, or other suitable reference materials. LECO 502-055 Orchard Leaves, 502-082 Tobacco Leaves, 502-273 Alfalfa, 502-274 Wheat Flour, and 502-277 Barley. Additional calibration materials can be found in LECO "Supplies for Organic Analysis" catalog (form no. 203-828).

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)	5
Endline Time seconds	1
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 three to five ~0.25 g coal samples.
3. Determine blank.
 - a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
 - b. Add ~1 g 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 ~0.15 to 0.35 g of calibration sample into a 528-203 Crucible, enter mass and sample identification into Sample Login (F3).
 - b. Add ~1 g of 502-321 Com-Cat to the crucible and thoroughly mix with the 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.
 - 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.
 - a. Weigh ~0.15 to 0.35 g of sample into 528-203 Crucible and enter mass and sample identification information into Sample Login (F3).
 - b. Add ~1 g of 502-321 Com-Cat to the crucible and thoroughly mix with the 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.

Typical Results*

Sample	Mass	% S
NIST 8436	0.1871	0.195
Durum	0.1872	0.193
Wheat Flour	0.1874	0.191
0.193% S	0.1887	0.192
	0.1892	0.193
	X =	0.193
	s =	0.001

LECO	0.2005	0.341
502-273	0.2057	0.340
Alfalfa	0.2021	0.339
0.339% S	0.2001	0.339
	0.2007	0.337
	X =	0.339
	s =	0.001

LECO	0.1549	0.204
502-274	0.1549	0.206
Wheat Flour	0.1537	0.205
0.205% S	0.1524	0.205
	0.1522	0.207
	X =	0.205
	s =	0.001

Sample	Mass	% S
Distillers	0.1548	0.521
Grain #1	0.1505	0.516
	0.1533	0.522
	0.1534	0.521
	0.1512	0.522
	X =	0.520
	s =	0.002

Distillers	0.1518	0.786
Grain #2	0.1543	0.800
	0.1503	0.790
	0.1528	0.795
	0.1506	0.795
	X =	0.793
	s =	0.005

*Results based on multipoint, multi-standard calibration using LECO Plant Tissue calibration samples.



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