Organic Application Note

Sulfur in Hydrocarbons[1]

Instrument
TruSpec S Module

Method Reference
ASTM D1552

Sample Preparation
A representative, uniform sample is required in order to obtain suitable results.

Accessories
528-203 Ceramic Boats, 502-321 Com-Cat™

Calibration Samples
LECO, NIST, or other suitable hydrocarbon reference materials.

Method Parameters*
External Sulfur Analysis Parameters

Furnace Temperature	1350°C
Analysis Stabilize Comparator	2
Manual Load Baseline Delay Time	3 seconds
Baseline Time	1second
Auto Detect Data Missed Time	5 seconds
Endline Time	1 second
Minimum Analysis Time	90
Comparator Level	0.30 %
Conversion Factor	1.00
Significant Digits	5

System Configuration

Gas Conservation Timeout	15 minutes
Auto Increment Sample Name	Disable
Nominal Mass	1.0000
Lance Delay Time	20 seconds
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 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 Ceramic Boat.
   c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on the display, slide ceramic boat into the combustion tube until it reaches boat stop. Alternately, place crucible unto the appropriate position of carousel if equipped with autoloader system.

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[1]*Applicable to samples boiling above 177°C (350°F).*

d. Repeat steps 3a through 3c a minimum of five times.
e. Set the blank following the procedure outlined in the operator's instruction manual.

4. Calibrate/Drift Correct.
   a. Add ~0.5 g of 502-321 Com-Cat to a 528-203 Ceramic Boat and spread evenly.
   b. Weigh ~0.1 to 0.13 g of hydrocarbon calibration sample into the Com-Cat, enter mass and sample identification into Sample Login (F3).
   c. Cover with ~0.5 g Com-Cat.
   d. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches boat stop. Alternately, place crucible unto the appropriate position of carousel if equipped with autoloader system.
   e. Repeat steps 4a through 4d a minimum of five times for each calibration/drift sample used.
   f. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

5. Analyze samples.
   a. Add ~0.5 g of 502-321 Com-Cat to a 528-203 Ceramic Boat and spread evenly.
   b. Weigh ~0.1 to 0.13 g sample into the Com-Cat and enter mass and identification information into Sample Login (F3).
   c. Cover with ~0.5 g Com-Cat.
   d. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide ceramic boat into the combustion tube until it reaches boat stop. Alternately, place crucible unto the appropriate position of carousel if equipped with autoloader system.

   Note: Light hydrocarbons may evaporate while in the carousel and it is advisable to minimize the time they spend in a carousel position by not pre-weighing the samples too far in advance.

Typical Results*

Sample	*Mass (g)*	*% S*
LECO	0.1027	0.506
502-420	0.1073	0.493
Mineral Oil	0.0957	0.506
0.503% S	0.1015	0.507
		X = 0.503
		s = 0.007

Sample	*Mass (g)*	*% S*
LECO	0.0996	1.02
502-391	0.0997	1.01
Residual	0.0983	1.02
Fuel Oil	0.0973	1.02
1.034% S		***X = 1.02***
		s = 0.01

Sample	*Mass (g)*	*% S*
LECO	0.1039	0.103
502-419	0.1061	0.102
Mineral Oil	0.1172	0.103
0.102 % S	0.0979	0.101
		X = 0.102
		s = 0.001

Sample	*Mass (g)*	*% S*
LECO	0.0986	3.02
502-393	0.1012	2.99
Residual	0.0985	3.04
Fuel Oil	0.0978	3.00
3.013% S		***X = 3.01***
		s = 0.02

**Results based on multipoint, multi-standard calibration using NIST and LECO hydrocarbon reference materials.*

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