

Sulfur in Hydrocarbons*

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CHN628 Series w/Sulfur Add-On Module (S628)



Introduction

Sulfur content in hydrocarbons can be seen as detrimental and in some cases beneficial depending upon the specific form and use of the hydrocarbon product or material. Sulfur analysis for hydrocarbons is often used to predict performance, handling, and processing properties, as well as determine regulatory requirements if the hydrocarbon material will be used as a fuel.

Reference

ASTM D1552 Standard Test Method for Sulfur in Petroleum Products

Accessories

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

Calibration Samples

LECO or other suitable hydrocarbon reference materials.

Method Parameters**

Furnace Temperature	1350°C
Element Parameters	Sulfur
Manual Load Baseline Delay Time	3 seconds
Minimum Analysis Time	90 seconds
Comparator Level	0.30%
Endline Time	1 second
Conversion Factor	1.00
Significant Digits	5
IR Analysis Stabilize Comparator	2.00
IR Baseline Time	1 second
Auto Detect Data Missed Time	5 seconds

System Parameters

Gas Conservation Timeout	15 minutes
Auto Increment Sample Name	Disable
Lance Delay Time	20 seconds
Lance Limits	50000

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 Ceramic Boat.
 - c. Initiate the analysis sequence (F5). When "Load Sample into Furnace" message appears on the display, slide the ceramic boat into the combustion tube until it reaches the boat stop.
 - d. Repeat steps 3a through 3c a minimum of five times.
 - e. Set the blank using at least five results following the procedure outlined in the operator's instruction manual.
 - f. The standard deviation of the last five blanks for sulfur should be less than or equal to 0.0005% (5 ppm) @ 1 g. Additional blanks beyond the recommended five may need to be analyzed in order to achieve the recommended precision.
4. Calibrate.
 - a. Add ~0.5 g of 502-321 Com-Cat into a 528-203 Ceramic Boat and create a trough in the Com-Cat with a spatula, then tare the balance.
 - b. Weigh ~0.1 to 0.13 g of a hydrocarbon calibration sample (Residual Fuel Oil, White Mineral Oil, #2 Diesel Fuel, etc.) into the trough created in the Com-Cat.
 - c. Enter sample mass and identification into Sample Login (F3).
 - d. Cover the sample in the trough with ~0.5 g of Com-Cat.
 - e. Initiate the analysis sequence (F5). When "Load Sample into Furnace" message appears on the display, slide the ceramic boat into the combustion tube until it reaches the boat stop.
 - f. Repeat steps 3a through 3e a minimum of five times.
 - g. Calibrate or drift-correct the instrument following the procedure outlined in the operator's instruction manual.
 - h. Verify the calibration by analyzing ~0.1 to 0.13 g of a hydrocarbon material different (typically a lower concentration of sulfur) than the material used for calibration.

* Applicable to samples boiling above 177° C (350° F). (Refer to ASTM D1552 for details)

** Refer to CHN628 Series Sulfur Add-On Module Operator's Instruction Manual for Method Parameter Definitions.

5. Analyze Samples.
 - a. Add ~0.5 g of 502-321 Com-Cat into a 528-203 Ceramic Boat and create a trough in the Com-Cat with a spatula, then tare the balance.
 - b. Weigh ~0.1 to 0.13 g of a sample into the trough created in the Com-Cat.
 - c. Enter sample mass and identification into Sample Login (F3).
 - d. Cover the sample in the trough with ~0.5 g of Com-Cat.
 - e. Initiate the analysis sequence (F5). When "Load Sample into Furnace" message appears on the display, slide the ceramic boat into the combustion tube until it reaches the boat stop.

Note: Light hydrocarbons such as diesel fuel will tend to evaporate while waiting to be analyzed. It is advisable to not pre-weigh too many samples in advance.

Typical Results

(Based on a single standard calibration with hydrocarbon calibration materials)

Sample	Mass (g)	% Sulfur
Residual Fuel Oil		
2.15% Sulfur	0.1040	2.15
	0.1053	2.16
	0.1060	2.13
	0.1138	2.17
	0.1105	2.17
	0.1016	2.20
	0.1063	2.14
	0.0994	2.18
	0.1019	2.19
	0.1013	2.17
	X=	2.17
	s=	0.02

White Mineral Oil		
0.101% Sulfur	0.1068	0.102
	0.1014	0.100
	0.1099	0.100
	0.1096	0.100
	0.1018	0.099
	0.1099	0.099
	0.1087	0.100
	0.1124	0.101
	0.1096	0.100
	0.1063	0.099
	X=	0.100
	s=	0.001

#2 Diesel Fuel Oil		
0.031% Sulfur	0.1070	0.030
	0.1096	0.030
	0.1070	0.031
	0.1066	0.031
	0.1058	0.030
	0.0988	0.030
	0.1088	0.032
	0.1134	0.031
	0.1041	0.031
	0.1029	0.031
	X=	0.031
	s=	0.001

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