

Organic Application Note

Sulfur and Carbon in Hydrocarbons¹

®

Instrument

SC632

Sample Preparation

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

Accessories

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

Calibration Samples

NIST, LECO, or other suitable hydrocarbon reference materials

Method Parameters*

Description

| | |
|---------------------|-------------------|
| Nominal Mass | 1.0000 |
| Furnace Temperature | 1350°C |
| Lance Delay Time | 20 seconds |
| Elements to Analyze | Sulfur and Carbon |

Element Parameters

| | Sulfur | Carbon |
|---|-----------|-----------|
| IR Analysis Stabilize Comparator | 2.00 | 2.00 |
| Manual Load Baseline Delay Time (seconds) | 3 | 3 |
| IR Baseline Time (seconds) | 1 | 2 |
| Auto Detect Data Missed Time (seconds) | 5 | 5 |
| Endline Time (seconds) | 1 | 1 |
| Minimum Analysis Time (seconds) | 90 | 60 |
| Maximum Analysis Time (seconds) | 360 | 360 |
| Comparator Level (%) | 0.30 | 1.00 |
| Conversion Factor | 1.00 | 1.00 |
| Significant Digits | 5 | 5 |
| Range Selection | Automatic | Automatic |
| Automatic Range Switch Level | 3500.0 | 4000.0 |
| Automatic Range Switch-Back Level | 3000.0 | 3500.0 |

System Parameters

| | |
|-----------------------------|-----------|
| Gas Conservation Timeout | 5 minutes |
| Auto Increment Sample Name | Disable |
| Lance Limits | 50000 |
| Furnace Standby Temperature | 1050 |

*Refer to SC632 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 ~1.0 g of 502-321 Com-Cat™ into a 528-203 Ceramic Boat.



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

SC632

- c. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on the display, slide the crucible into the combustion tube until it reaches the boat stop. Alternately, place the crucible onto the appropriate position of the carousel if equipped with an autoloader system.
- 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, spreading evenly.
- b. Weigh 0.1 to 0.13 g sample onto the Com-Cat™, and enter mass and sample identification information into Sample Login (F3).
- c. Cover the sample with ~0.5 g Com-Cat™.
- d. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide the crucible into the combustion tube until it reaches the boat stop. Alternately, place the crucible onto the appropriate position of the carousel if equipped with an autoloader system.
- e. Repeat steps 4a through 4d a minimum of five times for each calibration/drift sample used.
- f. Calibrate/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, spreading evenly.
- b. Weigh ~0.1 to 0.13 g sample onto the Com-Cat™, and enter mass and identification information into Sample Login (F3).
- c. Cover the sample with ~0.5 g Com-Cat™.
- d. Initiate the analysis sequence (F5), when "Load Sample into Furnace" message appears on screen, slide the crucible into the combustion tube until it reaches the boat stop. Alternately, place the crucible onto the appropriate position of the carousel if equipped with an 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 | % C | Sample | Mass g | % S | % C |
|----------|------------|-------------|--------------|---|------------|--------------|--------------|
| LECO | 0.1054 | 4.49 | 82.21 | LECO | 0.1150 | <0.01 | 86.24 |
| 502-394 | 0.1136 | 4.50 | 82.22 | 501-439 | 0.1066 | <0.01 | 85.81 |
| 4.49% S | 0.1005 | 4.46 | 82.28 | 86.10% C | 0.1136 | <0.01 | 85.96 |
| Residual | 0.1071 | 4.49 | 82.42 | Paraffin | 0.1058 | <0.01 | 86.11 |
| Fuel Oil | 0.1155 | 4.52 | 82.53 | Oil | 0.1051 | <0.01 | 86.37 |
| | X = | 4.49 | 82.33 | | X = | — | 86.10 |
| | s = | 0.03 | 0.14 | | s = | — | 0.22 |
| LECO | 0.1095 | 3.04 | 83.76 | LECO | 0.1100 | 0.098 | 84.12 |
| 502-393 | 0.1148 | 3.02 | 83.91 | 502-427 | 0.1079 | 0.095 | 83.89 |
| 3.01% S | 0.1046 | 2.99 | 83.87 | 0.1003% S | 0.1102 | 0.097 | 83.73 |
| Residual | 0.1190 | 3.01 | 83.85 | Diesel | 0.1072 | 0.098 | 83.97 |
| Fuel Oil | 0.1128 | 2.99 | 83.82 | Fuel Oil | 0.1137 | 0.097 | 83.70 |
| | X = | 3.01 | 83.84 | | X = | 0.097 | 83.88 |
| | s = | 0.02 | 0.06 | | s = | 0.001 | 0.17 |
| LECO | 0.1086 | 2.04 | 85.06 | | | | |
| 502-392 | 0.1083 | 2.02 | 85.67 | | | | |
| 2.05% S | 0.1024 | 2.03 | 85.00 | | | | |
| Residual | 0.1115 | 2.02 | 84.81 | *Results based on single-standard calibration using | | | |
| Fuel Oil | 0.1112 | 2.03 | 85.12 | LECO 502-394 Residual Oil @ 4.49% S and | | | |
| | X = | 2.03 | 84.93 | 501-439 Paraffin Oil @ 86.10% C. | | | |
| | s = | 0.01 | 0.19 | | | | |



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