

## Instrument: CS844

## Carbon and Sulfur in Iron, Steel, Nickel-Base, and Cobalt-Base Alloys

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### Summary

The determination of the amount of carbon and sulfur in iron, steel, nickel, and cobalt alloys represents two of the most important quality metrics for these materials. Carbon is the primary interstitial alloying constituent in these materials, and the level greatly influences properties such as hardness, ductility, weldability, and wear resistance. Sulfur is either considered a contaminant or desired additive in these materials as its level influences malleability. The CS844 utilizes a high-efficiency induction furnace that quickly breaks down these materials and allows very rapid determination of carbon and sulfur, helping to increase the efficiency of the production operation. The new infrared detection system and high-efficiency furnace provide the precision necessary for material certification.

### Sample Preparation

Surface contamination on the sample can cause significant errors in the analytical data; therefore, care must be taken to ensure a clean, representative sample is analyzed. Solid samples should be abraded with a clean file, rinsed in acetone, and dried with warm air prior to analysis. Samples that cannot be abraded due to irregular shapes should be rinsed in a suitable solvent such as acetone, and dried with warm air. Care must be taken to remove all traces of the solvent. If a sample is porous, refrain from using solvents, as it will be difficult to remove all traces of the solvent by drying. Refer to ASTM E1806 for additional sampling and sample preparation information.

### Accessories

528-018 or 528-018HP Crucible (preheated\*); LECOCEL II (501-008) or LECOCEL II HP (502-173) Accelerator; Metal Scoop (773-579); Tongs (761-929).

\*Ceramic crucibles are baked in a muffle or tube furnace (LECO TF-10) at 1250°C for a minimum of 15 minutes, or at 1000°C for 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and then are transferred to a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked.

### Calibration

There are several suitable reference materials available from LECO. Likewise, NIST, JK, JSS, and BCS are certified bodies that have a variety of certified reference materials (SRM/CRM) available as well. It is important that both high- and low-carbon and sulfur ranges are calibrated. Linear calibration curves are recommended. Refer to the operator's instruction manual for details.

### Reference Method

ASTM E1019

### Method Parameters

#### Analysis Parameters

|                   |          |
|-------------------|----------|
| Purge Time        | 15 s     |
| Analysis Delay    | 20 s     |
| Sample Cool Time  | 0 s      |
| Furnace Mode      | Constant |
| Furnace Power     | 100%     |
| Furnace Ramp Rate | 0        |

#### Element Parameters

|                    | Carbon | Sulfur |
|--------------------|--------|--------|
| Integration Delay  | 0 s    | 0 s    |
| Starting Baseline  | 2 s    | 2 s    |
| Use Comparator     | No     | No     |
| Integration Time   | 50     | 55     |
| Use Endline        | Yes    | Yes    |
| Ending Baseline    | 2 s    | 2 s    |
| Significant Digits | 5      | 5      |
| Range Select       | Auto   | Auto   |
| Range Low          | 800    | 800    |
| Range High         | 950    | 950    |

### Procedure

1. Prepare the instrument and crucibles as outlined in the operator's instruction manual.
2. Determine the instrument blank.
  - a. Login a minimum of 3 Blank reps.
  - b. Add ~1.2 g of LECOCEL II or LECOCEL II HP accelerator to the crucible.
  - c. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
  - d. Repeat steps 2b through 2c a minimum of three times.
  - e. Set the blank by following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct
  - a. Login a minimum of 3 Standard reps.
  - b. Weigh ~1.0 g of a calibration/drift standard into the crucible and enter the mass and standard identification of the standard.
  - c. Add ~1.2 g of LECOCEL II or LECOCEL II HP on top of the standard.
  - d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable) and initiate analysis.
  - e. Repeat steps 3b and 3d a minimum of three times for each calibration/drift standard intended for calibration/drift.
  - f. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.



#### 4. Sample Analysis

- Login a Sample with appropriate number of reps.
- Weigh a ~1.0 g sample into the crucible and enter the mass and sample identification of the sample.
- Add ~1.2 g of LECOCEL II or LECOCEL II HP accelerator on top of the sample.
- Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

### Typical Results

| Sample     | Mass (g)   | C %          | S %           |
|------------|------------|--------------|---------------|
| LECO       | 1.0        | 0.825        | 0.0112        |
| 501-506    |            | 0.823        | 0.0113        |
| Steel Ring |            | 0.822        | 0.0107        |
| 0.826% C   |            | 0.826        | 0.0110        |
| 0.0110% S  |            | 0.822        | 0.0111        |
|            |            | 0.824        | 0.0113        |
|            |            | 0.827        | 0.0113        |
|            |            | 0.825        | 0.0106        |
|            |            | 0.831        | 0.0103        |
|            |            | 0.821        | 0.0110        |
|            | <b>X =</b> | <b>0.825</b> | <b>0.0110</b> |
|            | <b>s =</b> | <b>0.003</b> | <b>0.0004</b> |

|            |            |               |               |
|------------|------------|---------------|---------------|
| LECO       | 1.0        | 0.0186        | 0.0092        |
| 501-501    |            | 0.0189        | 0.0093        |
| Steel Ring |            | 0.0186        | 0.0093        |
| 0.0186% C  |            | 0.0187        | 0.0092        |
| 0.0093% S  |            | 0.0181        | 0.0094        |
|            |            | 0.0183        | 0.0093        |
|            |            | 0.0190        | 0.0092        |
|            |            | 0.0186        | 0.0093        |
|            |            | 0.0180        | 0.0092        |
|            |            | 0.0181        | 0.0094        |
|            | <b>X =</b> | <b>0.0185</b> | <b>0.0093</b> |
|            | <b>s =</b> | <b>0.0004</b> | <b>0.0001</b> |

|           |            |              |              |
|-----------|------------|--------------|--------------|
| LECO      | 1.0        | 0.056        | 0.308        |
| 502-449   |            | 0.055        | 0.309        |
| Steel Pin |            | 0.055        | 0.310        |
| 0.054% C  |            | 0.052        | 0.305        |
| 0.306% S  |            | 0.055        | 0.309        |
|           |            | 0.055        | 0.306        |
|           |            | 0.055        | 0.305        |
|           |            | 0.052        | 0.307        |
|           |            | 0.054        | 0.309        |
|           |            | 0.055        | 0.305        |
|           | <b>X =</b> | <b>0.054</b> | <b>0.307</b> |
|           | <b>s =</b> | <b>0.001</b> | <b>0.002</b> |

| Sample    | Mass (g)   | C %            | S %            |
|-----------|------------|----------------|----------------|
| LECO      | 1.0        | 0.00149        | 0.00083        |
| 502-348   |            | 0.00142        | 0.00096        |
| Steel Pin |            | 0.00161        | 0.00094        |
| 0.0015% C |            | 0.00153        | 0.00086        |
| 0.0009% S |            | 0.00148        | 0.00082        |
|           |            | 0.00156        | 0.00095        |
|           |            | 0.00148        | 0.00096        |
|           |            | 0.00147        | 0.00088        |
|           |            | 0.00148        | 0.00097        |
|           |            | 0.00148        | 0.00084        |
|           | <b>X =</b> | <b>0.00150</b> | <b>0.00090</b> |
|           | <b>s =</b> | <b>0.00005</b> | <b>0.00006</b> |

|           |            |                |                |
|-----------|------------|----------------|----------------|
| LECO      | 1.0        | 0.00074        | 0.00100        |
| 501-952   |            | 0.00068        | 0.00103        |
| UHP Iron  |            | 0.00065        | 0.00096        |
| Powder    |            | 0.00081        | 0.00103        |
| 0.0007% C |            | 0.00065        | 0.00096        |
| 0.0010% S |            | 0.00069        | 0.00101        |
|           |            | 0.00070        | 0.00102        |
|           |            | 0.00069        | 0.00100        |
|           |            | 0.00067        | 0.00098        |
|           |            | 0.00072        | 0.00100        |
|           | <b>X =</b> | <b>0.00070</b> | <b>0.00100</b> |
|           | <b>s =</b> | <b>0.00005</b> | <b>0.00003</b> |