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

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Instrument: CS844

Summary

The determination of the amount of carbon and sulfur in iron, steel, nickel-, and cobalt-base 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 typically considered a contaminant in these materials and its level influences such parameters as conductivity and brittleness. The CS844 utilizes an induction-based 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.

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 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.

Inorganic Application Note

d. Repeat steps 3b and 3c a minimum of three times for each calibration/drift standard intended for calibration/drift.
e. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.

4. Sample Analysis
   a. Login a Sample with appropriate number of reps.
   b. Weigh a ~1.0 g sample into the crucible and enter the mass and sample identification of the sample.
   c. Add ~1.2 g of LECOCEL II or LECOCEL II HP accelerator on top of the sample.
   d. 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
	***X* =**	***0.825***	***0.0110***
	***s* =**	***0.003***	***0.0004***

Sample	Mass (g)	C %	S %
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
	***X* =**	***0.0185***	***0.0093***
	***s* =**	***0.0004***	***0.0001***

Sample	Mass (g)	C %	S %
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
	***X* =**	***0.054***	***0.307***
	***s* =**	***0.001***	***0.002***

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
	***X* =**	***0.00150***	***0.00090***
	***s* =**	***0.00005***	***0.00006***

Sample	Mass (g)	C %	S %
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
	***X* =**	***0.00070***	***0.00100***
	***s* =**	***0.00005***	***0.00003***

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