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

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS744

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 CS744 utilizes an 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.

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 (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. After preheating, crucibles must be handled using clean tongs only, do not use fingers.

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. Single or multipoint calibration curves can be utilized. Refer to the operator's instruction manual for details.

Reference Method

ASTM E1019

Method Parameters

Analysis Parameters

Purge Time:	10 seconds
Analysis Delay:	20 seconds
Sample Cool Time:	0 seconds
Furnace Power:	100%

Element Parameters	*Carbon*	*Sulfur*
Integration Delay:	0 seconds	0 seconds
Starting Baseline:	2 seconds	2 seconds
Use Comparator:	No	No
Integration Time:	50 seconds	55 seconds
Use Endline:	Yes	Yes
Ending Baseline:	2 seconds	2 seconds

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 (773-579) scoop (~1.2 g) of LECOCEL II or LECOCEL II HP accelerator to the preheated 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. Instrument calibration/drift correction.
   a. Login a minimum of 3 Standard reps for each calibration/drift reference material to be used for calibration/drift.
   b. Weigh ~1.0 g of a calibration/drift reference material into the preheated crucible and enter the mass and reference material identification into the standard login.
   c. Add 1 (773-579) scoop (~1.2 g) of LECOCEL II or LECOCEL II HP accelerator on top of the reference material.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   e. Repeat steps 3b through 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 operators instruction manual.
4. Sample Analysis.
   a. Login a Sample with the desired number of reps.
   b. Weigh ~1.0 g of sample into the preheated crucible and enter the mass and sample identification into the sample login.
   c. Add 1 (773-579) scoop (~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.
   e. Repeat steps 4a through 4d as necessary.

Inorganic Application Note

Delivering the Right Results

Typical Results

Low Sulfur Reference Materials

Description	*Mass (g)*	*% Carbon*	*% Sulfur*
501-506*	1.0	0.812	0.0041
0.814% Carbon		0.818	0.0039
0.0038% Sulfur		0.817	0.0034
Steel Ring		0.820	0.0038
		0.828	0.0038
	X=	***0.819***	***0.0038***
	s=	***0.006***	***0.0002***
JK NR 21*	1.0	0.175	0.0107
0.175% Carbon		0.175	0.0107
0.0110% Sulfur		0.175	0.0109
Steel Milling		0.175	0.0108
		0.174	0.0107
	X=	***0.175***	***0.0108***
	s=	***0.001***	***0.0001***
NIST SRM 2166*	1.0	0.0144	0.0024
0.0150% Carbon		0.0142	0.0024
0.00216% Sulfur		0.0144	0.0023
Steel Chip		0.0143	0.0024
		0.0147	0.0023
	X=	***0.0144***	***0.0023***
	s=	***0.0002***	***0.00004***
LECO 501-675*	1.0	0.053	0.0278
0.052% Carbon		0.053	0.0279
0.0272% Sulfur		0.053	0.0280
Steel Pin		0.050	0.0278
		0.052	0.0281
	X=	***0.052***	***0.0279***
	s=	***0.002***	***0.0001***
NIST SRM 362*	1.0	0.1618	0.0364
0.0160% Carbon		0.1626	0.0349
0.0360% Sulfur		0.1618	0.0360
Steel Chip		0.1625	0.0366
		0.1622	0.0361
	X=	***0.1622***	***0.0360***
	s=	***0.0004***	***0.0006***
NIST SRM 179*	1.0	0.0275	0.0261
0.027% Carbon		0.0273	0.0251
0.026% Sulfur		0.0271	0.0262
Steel Chip		0.0273	0.0254
		0.0271	0.0256
	X=	***0.0273***	***0.0257***
	s=	***0.0002***	***0.0005***

Description	*Mass (g)*	*% Carbon*	*% Sulfur*
LECO 501-504*	1.0	0.428	0.0113
0.429% Carbon		0.428	0.0121
0.0111% Sulfur		0.428	0.0120
Steel Ring		0.426	0.0121
		0.428	0.0119
	X=	***0.428***	***0.0119***
	s=	***0.001***	***0.0003***
NIST SRM 344*	1.0	0.0677	0.0189
0.069% Carbon		0.0677	0.0184
0.019% Sulfur		0.0680	0.0184
Steel Chip		0.0680	0.0187
		0.0684	0.0186
	X=	***0.0680***	***0.0186***
	s=	***0.0003***	***0.0002***
LECO 502-348*	1.0	0.0016	0.0012
0.0015% Carbon		0.0016	0.0011
0.0011% Sulfur		0.0016	0.0013
Steel Pin		0.0016	0.0010
		0.0017	0.0013
	X=	***0.0016***	***0.0012***
	s=	***0.0001***	***0.0001***

High Sulfur Reference Materials

Description	*Mass (g)*	*% Carbon*	*% Sulfur*
NIST SRM 368**	1.0	0.0871	0.1344
0.090% Carbon		0.0868	0.1327
0.1324% Sulfur		0.0873	0.1329
Steel Chip		0.0875	0.1288
		0.0874	0.1331
	X=	***0.0872***	***0.1324***
	s=	***0.0003***	***0.0021***
NIST SRM 8k**	1.0	0.0790	0.0768
0.0806% Carbon		0.0794	0.0765
0.0775% Sulfur		0.0795	0.0749
Steel Chip		0.0800	0.0751
		0.0800	0.0741
	X=	***0.0796***	***0.0755***
	s=	***0.0004***	***0.0011***

**Calibrated with NIST SRM 16f for Carbon, NIST SRM 362 for Sulfur.*
***Calibrated with NIST SRM 16f for Carbon, NIST SRM 368 for Sulfur.*

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