Inorganic Application Note

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

Instrument

CS600-Series

Sampling and 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 Ceramic Crucibles (preheated)*; LECOCEL II (501-008) or LECOCEL II HP (502-173) accelerator.

**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 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 calibration samples 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 ranges are calibrated. Single or multipoint calibration curves can be utilized. Refer to the operator's instruction manual for details.

Method Parameters

Purge Time (seconds):	1**
Delay Time (seconds):	8**
Furnace Low Power (%):	100
Furnace High Power (%):	100
Furnace Ramp Rate:	0

	Carbon	*Sulfur*
Minimum Timeout (seconds):	40	40
Comparator Level:	1.00	1.00
Significant Digits:	4 or 5	4 or 5
Integration Delay:	0	0

***For optimum analytical performance on samples with low carbon and sulfur content (≤0.05%), a purge time of 10 seconds and a delay time of 20 seconds is recommended.*

CS600

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Enter 1.0000 g weight into weight stack.
   b. Add ~1.5 g of accelerator to crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped), and analyze.
   d. Repeat steps 2a through 2c a minimum of five times.
   e. Enter blank following procedure outlined in the operator's instruction manual.
3. Calibrate.
   a. Weigh ~1.0 g calibration sample into crucible and enter weight into weight stack.
   b. Add ~1.5 g of accelerator on top of sample.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped), and analyze.
   d. Repeat steps 3a through 3c a minimum of five times for each calibration sample intended for calibration.
   e. Calibrate using the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1.0 g sample into crucible and enter weight into weight stack.
   b. Add ~1.5 g of accelerator on top of sample.
   c. Place crucible on furnace pedestal (or appropriate autoloader position if so equipped), and analyze.

Note: Some "solid" samples, notably nickel and/or cobalt-base alloys, are difficult to combust and may require additional accelerator. Likewise, reducing the sample to small chunks, chips, or pieces will facilitate combustion. For additional information on Carbon and Sulfur Determination using an Induction Furnace IR detection instrument, request LECO's Application Tips CD (209-050-055).

Typical Results

Sample	Weight (g)	C %	S %
LECO	1.0	0.605	0.0176
501-505		0.605	0.0172
Steel Ring		0.605	0.0169
0.607 % C		0.607	0.0171
0.0172% S		0.607	0.0174
		0.605	0.0176
		0.606	0.0178
		0.604	0.0173
		0.606	0.0173
		0.605	0.0168
	X =	***0.606***	***0.0173***
	s =	***0.001***	***0.0003***

Sample	Weight (g)	C %	S %
LECO	1.0	0.00089	0.00062
502-348		0.00089	0.00067
Low C and S		0.00094	0.00068
Steel Pin		0.00095	0.00060
0.0009 % C		0.00085	0.00064
0.0007 % S		0.00085	0.00061
		0.00090	0.00062
		0.00090	0.00060
		0.00090	0.00065
		0.00089	0.00055
	X =	***0.00090***	***0.00062***
	s =	***0.00003***	***0.00004***

Sample	Weight (g)	C %	S %
LECO	1.0	0.0375	0.0223
501-501		0.0380	0.0221
Steel Ring		0.0375	0.0222
0.0377 % C		0.0377	0.0222
0.0220 % S		0.0376	0.0223
		0.0376	0.0222
		0.0373	0.0221
		0.0376	0.0222
		0.0377	0.0223
		0.0377	0.0222
	X =	***0.0376***	***0.0222***
	s =	***0.0002***	***0.0001***

Sample	Weight (g)	C %	S %
LECO	1.0	0.0414	0.0025
501-933		0.0414	0.0027
Ni-Base Alloy		0.0413	0.0027
Chip Sample		0.0414	0.0028
0.042 % C		0.0415	0.0027
0.003 % S		0.0413	0.0028
		0.0414	0.0028
		0.0415	0.0028
		0.0412	0.0027
		0.0414	0.0029
	X =	***0.0414***	***0.0027***
	s =	***0.0001***	***0.0001***

Delivering the Right Results

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