

Ultra-Low Carbon and Sulfur Analysis in Steel, Nickel-Base, and Cobalt-Base Alloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS-444

Approval

Carbon/Sulfur combustion method - ASTM E1019

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 acetone and dried. Care must be taken to remove all traces of the solvent. If a sample is porous, it is advisable not to use solvents as it will be difficult to remove all traces.

Accessories

528-018 Crucibles (preheated); 502-173 LECOCEL® II HP; 502-231 Low C/S Fe Chip*

Calibration Standard

NIST or other suitable standard

Program Settings

Pre-Analyze Purge: 5 seconds
Pre-Analyze Delay: 25 seconds
C IR Cell Range: Low
C Minimum Timeout: 45 seconds
S Minimum Timeout: 55 seconds
C Comparator Level: 0.5%
S Comparator Level: 0.5%

Procedure

1. Preheat ceramic crucibles in a muffle or tube furnace at least 1250°C for not less than 15 minutes, or at 1000°C for not less than 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and then placed in a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked.
2. Prepare the instrument for operation as outlined in the operator's instruction manual.
3. Determine blank as follows:
 - a. Enter a 1.000 g weight into weight stack.
 - b. Add 1.000 g (± 0.005 g) of 502-173 LECOCEL II HP to a preheated crucible.
 - c. Place the crucible on furnace pedestal and analyze.
 - d. Repeat steps 3a through 3c a minimum of 3 times.
 - e. Enter blank following routine outlined in operator's instruction manual.



4. Calibrate as follows:
 - a. Weigh ~ 1.000 g calibration standard into a preheated 528-018 Crucible and enter the weight into weight stack.
 - b. Add 1.000 g (± 0.005 g) of 502-173 LECOCEL II HP to the crucible.
 - c. Place the crucible on furnace pedestal and analyze.
 - d. Repeat steps 4a through 4c a minimum of three times and calibrate the instrument following the auto calibration procedure as outlined in the operator's instruction manual.
 - e. Verify the calibration by analyzing the calibration standard again. It should fall within the expected tolerance. If not, repeat steps 4a through 4d again.
5. Analyze samples as follows:
 - a. Weigh ~ 1.000 g sample into a preheated 528-018 Crucible and enter the weight into weight stack.
 - b. Add 1.000 g (± 0.005 g) of 502-173 LECOCEL II HP to the crucible.
 - c. Place crucible on furnace pedestal and analyze.

**NOTE: For some samples which are difficult to burn, it may be necessary to use ~ 0.75 g 502-231 Iron Chip and 1.0 g LECOCEL II HP to obtain a suitable combustion. (Generally, a peak plate current of 380 to 400+ milliamps obtained 15 to 25 seconds into the analysis indicates a good combustion.) The blank is determined by analyzing ~ 0.75 g 502-231 Iron Chip and 1.0 g LECOCEL II accelerators following steps 3b through 3e. Only 0.5 g samples should be analyzed, otherwise the burn will be too vigorous.*

Typical Results

Sample	Weight (g)	ppm C	ppm S
LECO 502-254	1.1089	7.8	5.6
High Purity Iron	1.1013	8.0	4.3
@ 8 ±2 ppm C	1.0058	7.6	4.9
5 ±1 ppm S	1.0003	7.6	5.5
	1.0150	7.4	4.7
	1.0075	8.4	4.9
	1.0674	8.2	4.9
	1.0231	8.2	4.1
	1.0102	7.3	4.4
	1.0152	7.1	5.1
		$\bar{\chi} = 7.8$	$\bar{\chi} = 4.8$
		$s = 0.43$	$s = 0.49$

Nickel Base	1.0020	32.0	3.1
Chips	1.0068	31.0	3.0
	0.9971	30.0	3.1
	0.9967	30.0	2.5
	0.9949	31.0	1.9
	0.9974	29.0	2.2
	1.0006	32.0	1.8
	0.9968	29.0	1.9
	0.9970	29.0	1.7
	1.0282	31.0	2.6
		$\bar{\chi} = 30.4$	$\bar{\chi} = 2.4$
		$s = 1.2$	$s = 0.56$

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