Sulfur Determination in Ultra Low Sulfur Steel, Nickel, and Superalloys

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Instrument: CS844/CS844ES

Introduction

Sulfur is known to degrade a high temperature alloy's ability to form a protective alumina scale on its surface, which protects it from thermal oxidation, and also has a detrimental effect on the mechanical properties of the alloy. Even ultra-low levels of sulfur can limit an alloy's useful operating life in critical applications like aerospace engine components. The accurate determination of ultra-low levels of sulfur in steel, nickel, and super alloys is an important quality control metric when these alloys are utilized in such high temperature applications. The LECO CS844ES with a detection limit of approximately 0.1 µg of sulfur expands the capability of the combustion technique to levels previously limited to GD-MS applications. The following application note outlines the settings and steps required to determine ultra-low levels of sulfur in steel, nickel, and superalloys.

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. 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 may be difficult to remove all traces of the solvent through drying. Refer to ASTM E1806 for additional sampling and sample preparation information.

Accessories

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

**For optimal precision, ceramic crucibles must be preheated in a muffle or tube furnace (LECO TF4) at ≥ 1250 °C for a minimum of 15 minutes or at ≥ 1000 °C for a minimum of 1 hour. Crucibles must be handled with clean tongs to avoid contamination. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and then are transferred to a desiccator for storage. Crucibles should be reheated if not used within four hours.*

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. Linear calibration curves are recommended. Refer to the operator's instruction manual for more details.

Method Parameters

Analysis Parameters	*Regular Mode*	*ES Mode*
Purge Time	15 s	15 s
Analysis Delay	20 s	80 s
Sample Cool Time	10 s	10 s
Furnace Mode	Constant	Constant
Furnace Power	100%	100%
Analysis Mode	Normal	Enhanced Sensitivity

Element Parameters	*Regular Mode*	*ES Mode*
Integration Delay	0 s	0 s
Starting Baseline	2 s	7 s
Use Comparator	No	No
Integration Time	55 s	130 s
Use Endline	Yes	Yes
Ending Baseline	2 s	7 s
Range Select	Auto	Auto
Range Lower Limit	800	800
Range Upper Limit	950	950

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Login a minimum of three blank replicates.
   b. Weigh 1.000 ± 0.005 g of 502-173 LECOCEL II HP accelerator into a preheated 528-018HP Crucible.
   c. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.
   d. Repeat steps 2b through 2c a minimum of three times.
   e. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Login a minimum of three standard replicates.
   b. Weigh ~1.0 g of a suitable calibration/drift standard into a preheated 528-018HP Crucible.
   c. Enter the standard mass and identification into the software.
   d. Add 1.000 ± 0.005 g of 502-173 LECOCEL II HP accelerator into the crucible, on top of the standard.
   e. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable), and initiate the analysis sequence.
   f. Repeat steps 3b through 3e a minimum of three times for each calibration/drift standard utilized.
   g. Calibrate/Drift correct by following the procedure outlined in the operator's instruction manual.

4. Sample Analysis.
   a. Login a Sample with the appropriate number of replicates.
   b. Weigh 1.0 ± 0.2 g of a sample into a preheated 528-018HP Crucible.
   c. Enter the sample mass and identification into the software.
   d. Add 1.000 ± 0.005g of 502-173 LECOCEL II HP accelerator into the crucible, on top of the sample.
   e. Place the crucible on the furnace pedestal or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.

*Typical Results**

	Regular Mode		ES Mode	
Name	*Mass(g)*	*Sulfur (ppm)*	*Mass(g)*	*Sulfur (ppm)*
502-704 1001	0.9998	1.01	1.0007	1.10
Steel Chip LCRM	1.0108	1.08	0.9941	1.07
@ 1.0 ± 0.2 ppm	1.0132	1.12	1.0007	1.09
	1.0002	1.07	0.9963	1.05
	1.0121	1.03	0.9980	1.01
	Avg =	***1.06***	***Avg =***	***1.06***
	s =	***0.043***	***s =***	***0.036***
Nickel Wire	1.2025	0.54	1.1997	0.63
	1.2030	0.44	1.2023	0.57
	1.2027	0.52	1.2050	0.56
	1.2011	0.51	1.2020	0.50
	1.2025	0.60	1.2049	0.53
	Avg =	***0.52***	***Avg =***	***0.56***
	s =	***0.058***	***s =***	***0.048***
Low Alloy Silicon Steel	0.9931	3.56	1.0002	3.74
	1.0002	3.62	1.0045	3.90
	0.9999	3.81	0.9983	3.91
	0.9994	3.62	0.9947	3.76
	1.0009	3.68	1.0004	3.84
	Avg =	***3.66***	***Avg =***	***3.83***
	s =	***0.094***	***s =***	***0.078***
Nickel Superalloy	1.0492	2.00	1.0925	2.03
	1.0624	2.04	1.0479	2.08
	1.1864	1.90	1.1756	1.97
	1.0929	2.23	1.1507	1.94
	1.0940	1.99	1.1331	2.04
	Avg =	***2.03***	***Avg =***	***2.01***
	s =	***0.121***	***s =***	***0.058***
Single Crystal Nickel Alloy	1.0586	0.81	1.0130	0.78
	0.9375	0.78	1.0131	0.87
	1.0435	0.54	1.0492	0.71
	1.1693	0.74	0.9976	0.83
	1.1831	0.56	1.0541	0.66
	Avg =	***0.69***	***Avg =***	***0.77***
	s =	***0.13***	***s =***	***0.09***

**Note: Results based on a linear full regression calibration utilizing 502-348 (Lot #1034) High Purity Steel @ 9 ± 2 ppm and NIST 861 Nickel Based Superalloy @ 0.561 ± 0.078 ppm.*

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