

Determination of Carbon and Sulfur in Tungsten Carbide

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS844 Series

Introduction

Tungsten carbide (WC) is an extremely hard and tough material with numerous industrial applications including abrasives, cutting tools, and wear parts. The determination of carbon in WC is an important quality control parameter. The carbon level can be used to confirm complete carburization of the tungsten feed stock, or to confirm the ratio of WC to the metal binder in cemented carbide products. The determination of sulfur in WC is conducted as a measure of purity, and is another important quality control parameter. The CS844 series carbon and sulfur elemental analyzer is ideally suited for the simultaneous determination of carbon and sulfur in tungsten carbide with its high precision for carbon and low level detection for sulfur.

Sample Preparation

Samples should be crushed to a uniform powder prior to analysis.

Accessories

528-018 or 528-018HP Crucible;
502-492 Copper; 502-231 Iron Chip; 763-266 or 502-860 LECOCEL; 501-078 Iron Powder;
501-636-HAZ V2O5 Accelerator; 773-579 Metal Scoop;
761-929 Tongs

For optimal sulfur precision, ceramic crucibles can be pre-baked in a muffle oven at $\geq 1000^{\circ}\text{C}$ for a minimum of 1 hour, or tube furnace (LECO TF1/TF-10) at $\geq 1250^{\circ}\text{C}$ for a minimum of 15 minutes. The crucibles are removed from the tube furnace/muffle oven, allowed to cool, and are then transferred to a desiccator for storage. Baked crucibles must be handled with clean tongs only to avoid contamination.

Calibration

501-123 Tungsten Carbide (WC) Reference Material; LECO 502-402 Steel Chip, or other suitable reference materials

Method Parameters

General Parameters

Purge Time:	10 s
Delay Time:	20 s
Sample Cool Time:	10 s
Furnace Mode:	Constant
Furnace Power:	100%

Element Parameters	Carbon	Sulfur
Integration Delay:	0 s	0 s
Starting Baseline:	2 s	2
Use Comparator:	No	No
Integration Time:	60 s	60 s
Use Endline:	Yes	Yes
Ending Baseline:	2 s	2 s
Range Select:	Auto	Auto
Range Lower Limit:	800	800
Range Upper Limit:	950	1200



Procedure for Carbon-Only Determination

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine the instrument Blank.
 - a. Login a minimum of three Blank reps.
 - b. Add ~ 1 g of copper and ~ 1 g iron chip accelerator to a preheated 528-018 or 528-018HP 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 three Standard Drift reps.
 - b. Weigh ~ 0.25 g of 501-123 Tungsten Carbide reference material, or other suitable calibration/drift material, into the crucible and enter the mass and identification of the reference material.
 - c. Add ~ 1 g of copper and ~ 1 g iron chip 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 reference material intended for calibration/drift.
 - f. 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 ~ 0.25 g of tungsten carbide sample into the crucible and enter the mass and sample identification of the sample.
 - c. Add ~ 1 g of copper and ~ 1 g iron chip accelerator on top of the sample.
 - d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Procedure for Sulfur Only or Simultaneous Carbon and Sulfur Determination

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 ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V2O5 to a preheated 528-018 or 528-018HP Crucible and thoroughly mix.
 - c. Add ~1.5 g of LECOCEL to the crucible.
 - d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
 - e. Repeat steps 2b through 2d a minimum of three times.
 - f. Set the blank by following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct
 - a. Login a minimum of three Standard Drift reps.
 - b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V2O5 to a preheated 528-018 or 528-018HP Crucible and thoroughly mix. Tare the crucible and accelerators.
 - c. Weigh ~0.25 to ~0.5 g of 502-402 Steel Chip or other suitable calibration/drift reference material into the crucible for sulfur calibration and thoroughly mix; enter the mass and identification of the reference material.
4. Sample Analysis
 - a. Login a sample with appropriate number of reps.
 - b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V2O5 to a preheated 528-018 or 528-018HP crucible and thoroughly mix. Tare the crucible and accelerators.
 - c. Weigh ~ 0.25 g tungsten carbide sample into the crucible thoroughly mix; enter the mass and sample identification of the crucible.
 - d. Add ~1.5 g of LECOCEL to crucible.
 - e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Repeat step 3b for carbon calibration; weigh ~0.25 g of 501-123 Tungsten Carbide reference material, or other suitable calibration/drift material, into the crucible and thoroughly mix; enter the mass and identification of the reference material.

Typical Results for Carbon-Only Determination

Sample	Mass (g)	% Carbon
501-123	0.2505	6.26
@ 6.26% C	0.2500	6.26
	0.2488	6.27
	0.2503	6.26
	0.2504	6.27
	0.2512	6.24
	0.2493	6.26
	0.2494	6.26
	0.2494	6.24
	0.2500	6.27
	Avg =	6.26
	s =	0.010

Typical Results for Sulfur Only or Simultaneous Carbon and Sulfur Determination

Sample	Mass (g)	% Carbon	% Sulfur
501-123	0.2490	6.26	0.0025
@ 6.26% C	0.2503	6.26	0.0025
	0.2499	6.27	0.0023
	0.2509	6.25	0.0023
	0.2495	6.27	0.0024
	0.2513	6.26	0.0023
	0.2518	6.27	0.0024
	0.2487	6.27	0.0025
	0.2503	6.29	0.0023
	0.2515	6.27	0.0022
	Avg =	6.27	0.0024
	s =	0.009	0.0001

