

# Carbon Determination in Tungsten Carbide

LECO Corporation; Saint Joseph, Michigan USA

## Instrument: WC600

### Sampling and Sample Preparation

Typically, samples should be in the form of fine powder (- 200 mesh)

### Accessories

528-018 or 528-018HP Ceramic Crucibles (preheated)\*, 619-880 Crucible Covers (optional), 763-266 LECOCEL and 501-263 Copper Chip accelerator; Five place (0.01 mg) balance (recommended).

\*Ceramic crucibles and covers are baked in a muffle or tube furnace (LECO TF10) 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/covers are not used within four hours, they should be re-baked.

### Calibration

LECO 502-123 Tungsten Carbide (WC). NIST and BCS are certified bodies that have tungsten carbide reference materials (SRM/CRM) available as well.

### Method Parameters

Purge Time (seconds)	50
Delay Time (seconds)	30
Sample Cool Time (seconds)	20
Furnace Low Power (%)	90
Furnace High Power (%)	90
Furnace Ramp Rate (%)	0

### Carbon

Minimum Timeout (seconds)	90
Comparator Level	100.00
Significant Digits	5
Integration Delay	0

### Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
  - a. Enter 1.0000 g mass into Sample Login.
  - b. Add 1.2 g ( $\pm 0.010$  g) of LECOCEL and 1.8 g ( $\pm 0.010$  g) of copper accelerator to crucible.
  - c. Place the crucible on the furnace pedestal (or appropriate auto-loader position if so equipped) and initiate 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 0.25 g ( $\pm 0.005$  g) WC calibration sample (to the nearest 0.01 mg) into crucible and enter mass into Sample Login.
  - b. Add 1.2 ( $\pm 0.010$  g) of LECOCEL followed by 1.8 ( $\pm 0.010$  g) of copper accelerator on top of sample (take care to evenly cover sample with accelerator).
  - c. Place the crucible on the furnace pedestal (or appropriate auto-loader position if so equipped) and initiate 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 0.25 g ( $\pm 0.005$  g) WC sample (to the nearest 0.01 mg) into crucible and enter mass into Sample Login.
  - b. Follow steps 3b through 3c.

*Note: Using a 619-880 Ceramic Cover in conjunction with the 528-018 or 528-018HP Crucibles may help reduce splatter of the products of combustion onto the combustion tube. This in-turn can improve the usable life of the combustion tube. The 619-880 Covers must be baked off using the same procedure as used for the 528-018 Crucibles.*

## Typical Results

Sample	Mass g	C %	Sample	Mass g	C %
NIST	0.24995	6.1023	Production	0.25198	5.9912
SRM 276b	0.25042	6.1001	Tungsten	0.25011	5.9935
Tungsten	0.25091	6.0986	Carbide	0.25196	5.9889
Carbide	0.24945	6.1035		0.25218	5.9968
@ 6.10% C	0.25156	6.1058		0.25216	5.9955
	0.25081	6.0974		0.25055	5.9992
	0.24972	6.1034		0.25101	5.9981
	0.25223	6.0957		0.25118	5.9956
	0.24973	6.0955		0.25048	5.9981
	0.25056	6.0978		0.25174	5.9931
	<b>X =</b>	<b>6.1000</b>		<b>X =</b>	<b>5.9950</b>
	<b>s =</b>	<b>0.00356</b>		<b>s =</b>	<b>0.00331</b>
LECO	0.24989	6.1954	BS 107	0.25035	6.1355
501-123	0.25368	6.1954	Tungsten	0.24978	6.1431
Tungsten	0.25102	6.2030	Carbide	0.24952	6.1304
Carbide	0.25338	6.2015	@ 6.14% C	0.25026	6.1457
@ 6.20% C	0.25287	6.1948		0.25238	6.1353
	0.25001	6.2023		0.25129	6.1409
	0.25162	6.1979		0.25122	6.1403
	0.25084	6.2033		0.25223	6.1479
	0.25221	6.2026		0.24971	6.1399
	0.25299	6.2040		0.25113	6.1410
	<b>X =</b>	<b>6.2000</b>		<b>X =</b>	<b>6.1400</b>
	<b>s =</b>	<b>0.00371</b>		<b>s =</b>	<b>0.00515</b>

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