Carbon in Refractory and Reactive Metals and Their Alloys[†]

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS600

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, it is advisable to avoid using solvents, as it will be difficult to remove all traces of the solvent by drying.

Method Reference   ASTM E1941

Accessories

528-018 or 528-018HP Ceramic Crucibles and 619-880 Ceramic Crucible Covers (preheated)*; 502-492 High Purity Copper Accelerator and 502-231 High Purity Iron Chip Accelerator

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/covers are removed from the furnace, allowed to cool for 1 to 2 minutes, and transferred to a desiccator for storage. If the crucibles/covers are not used within four hours, they should be re-baked.

Calibration Samples

NIST or other suitable reference materials.
Note: Due to limited availability of refractory/reactive reference materials, steel calibration samples can be used to calibrate.

Method Parameters

Purge Time (seconds)	10
Delay Time (seconds)	20
Furnace Low Power (%)	100
Furnace High Power (%)	100
Furnace Ramp Rate	0

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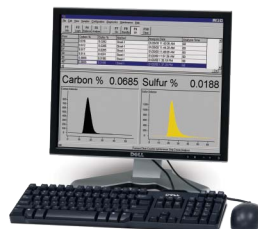
Procedure

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 (F3) using Blank as the sample name.
   b. Add ~1.0 g of iron chip and ~1.5 g of copper accelerator to a prepared crucible.
   c. Place a prepared crucible lid on the crucible.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate Analyze (F5).
   e. Repeat steps 2a through 2d a minimum of three times.
   f. Set blank following procedure outlined in operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~0.5 g calibration/drift sample into a prepared crucible, enter mass and sample identification into Sample Login (F3).
   b. Add ~1.0 g of iron chip accelerator and ~1.5 g of copper accelerator on top of sample.
   c. Place a prepared crucible lid on the crucible.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate Analyze (F5).
   e. Repeat steps 3a through 3d a minimum of three times for each calibration/drift sample intended for calibration/drift.
   f. Calibrate/drift correct using the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~0.5 g sample into prepared crucible, enter mass and sample identification into Sample Login (F3).
   b. Add ~1.0 g of iron chip accelerator ~1.5 g of copper accelerator on top of sample.
   c. Place a prepared crucible lid on the crucible.
   d. Place crucible on furnace pedestal (or appropriate autoloader position if so equipped) and initiate Analyze (F5).

[†]Ti, Zr, W, Ta, Mo, Nb, Hf, and Re

Typical Results*

Sample	Mass g	C %	Sample	Mass g	C %
Titanium	0.5353	0.0247	NIST	0.4983	0.0156
Wire	0.5215	0.0246	SRM 360a	0.5032	0.0158
	0.5106	0.0252	Zircaloy-2	0.5056	0.0158
	0.5377	0.0245	Chip	0.5061	0.0157
	0.4677	0.0244	(C not certified)	0.4983	0.0154
	0.4969	0.0246		0.5094	0.0156
	0.4590	0.0246		0.5015	0.0156
	0.4981	0.0245		0.5032	0.0156
	0.5281	0.0245		0.5099	0.0154
	0.4285	0.0246		0.5091	0.0160
	X =	*0.0246*		*X =*	*0.0157*
	s =	*0.0002*		*s =*	*0.0002*
NIST	0.5005	0.0250	Tantalum	0.5029	0.0022
SRM 173a	0.4989	0.0251	Powder	0.4962	0.0020
Titanium alloy	0.5031	0.0251		0.5024	0.0022
Chip	0.4973	0.0249		0.5069	0.0021
@ 0.025% C	0.4988	0.0251		0.4974	0.0021
	0.5041	0.0249		0.4977	0.0022
	0.5010	0.0249		0.5060	0.0024
	0.4986	0.0249		0.5092	0.0022
	0.4996	0.0250		0.5066	0.0022
	0.5023	0.0251		0.5004	0.0022
	X =	*0.0250*		*X =*	*0.0022*
	s =	*0.0001*		*s =*	*0.0001*

*Based on single-standard calibration using NIST SRM 173a