Carbon in Mold Powder

LECO Corporation; Saint Joseph, Michigan USA

Instrument: C744 or Equivalent

Introduction

This application note outlines the system parameters and process steps to set up the LECO CS/C744 elemental analyzer to determine the amount of carbon in mold powder. Mold powders are used to lubricate the mold and to help control the mold heat transfer of a continuous casting steel production process. The liquid steel melts the mold powder, creating a layer of liquid mold slag, which subsequently creates a thin slag film that solidifies into a glassy and crystalline phase. The properties of this phase are responsible for the lubricity and thermal performance of the mold powder. Lubricity relates to the speed at which you can cast, and the thermal performance can be tailored to minimize surface cracking of the steel and to protect the mold itself.

These mold powders are typically a mixture of SiO_2, CaO, Al_2O_3, Na_2O, F and C with lesser amounts of metal oxides such as MgO, MnO, Li_2O and Fe_2O_3 depending on the formulation. One of the critical performance metrics is the total carbon content of the mold powder. Carbon content is one of the parameters used to control the melting rate of the mold powder, which directly relates to lubricity and production speed. The rapid rate of analysis and precise results make the CS/C744 the industry choice for this application. This application note is similar in purpose to ASTM E2050-12a Standard Test Method for Determination of Total Carbon in Mold Powders by Combustion.

Sample Preparation

Samples should be a uniform fine powder.

Accessories

528-018 or 528-018HP Ceramic Crucibles*; 501-008 LECOCEL II or 502-173 LECOCEL II HP Accelerator; 501-077 or 502-231 Iron Chip Accelerator; 773-579 Metal Scoop.

**For best precision, ceramic crucibles should be baked in a muffle or tube furnace (LECO TF10) at a minimum of 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 transferred to a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked. After baking, handle crucibles with clean tongs only. Do not use fingers.*

NOTE: Analyzing mold powder on the CS/C744 requires the installation of a fluorine trap using the LECO 619-592-149 Trap Kit.

Calibration

LECO 502-491 Ore Tailings, LECO 502-030 Synthetic Carbon, or other suitable reference materials.

Method Parameters

Analysis Parameters

Purge Time:	15 s
Analysis Delay:	20 s
Sample Cool Time:	5 s
Furnace Power:	100%

Element Parameters	*Carbon*
Integration Delay:	0 s
Starting Baseline:	2 s
Use Comparator:	No
Integration Time:	50 s
Use Endline:	Yes
Ending Baseline:	2 s

Procedure

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 one (773-579) scoop (~1.2 g) of LECOCEL II or LECOCEL II HP accelerator to the crucible.
   c. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator to the crucible.
   d. Place the crucible on the pedestal (or appropriate autoloader position if applicable).
   e. Initiate the analysis by pressing the Analyze button.
   f. Repeat steps 2b through 2e a minimum of three times.
   g. Set the Blank according to the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Login a minimum of three Standard reps for each calibration/drift reference material to be used for calibration/drift.
   b. Weigh ~0.1 to 0.25 g of a calibration/drift reference material into the crucible and enter the mass and reference material identification into the standard login.
   c. Add one (773-579) scoop (~1.2 g) of LECOCEL II or LECOCEL II HP accelerator on top of the reference material.
   d. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator on top of the reference material.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   f. Repeat steps 3b through 3e a minimum of three times for each calibration/drift reference material intended for calibration/drift.
   g. Calibrate/drift correct by following the procedure outlined in the operators instruction manual.

Inorganic Application Note

Delivering the Right Results

4. Sample Analysis.
   a. Login a sample with the desired number of reps.
   b. Weigh ~0.1 to 0.25 g of sample into the crucible and enter the mass and sample identification into the sample login.

   Note: Sample mass is dependent upon carbon concentration. Higher carbon concentration should use lower sample weights.

   c. Add one (773-579) scoop (~1.2 g) of LECOCEL II or LECOCEL II HP accelerator on top of the sample.
   d. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator on top of the sample.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   f. Repeat steps 4a through 4e as necessary.

Typical Results

Description	*Mass (g)*	*% Carbon*
Mold Powder A	0.1197	16.8
	0.1215	16.7
	0.1194	16.7
	0.1205	16.8
	0.1211	16.5
	$\bar{\chi}$=	***16.7***
	***s*=**	***0.1***

Description	*Mass (g)*	*% Carbon*
Mold Powder B	0.2496	3.70
	0.2481	3.67
	0.2496	3.67
	0.2531	3.70
	0.2480	3.70
	$\bar{\chi}$=	***3.69***
	***s*=**	***0.01***

Description	*Mass (g)*	*% Carbon*
Mold Powder C	0.1022	19.9
	0.0995	20.2
	0.1011	20.3
	0.1040	20.1
	0.1030	20.3
	$\bar{\chi}$=	***20.2***
	***s*=**	***0.1***

Description	*Mass (g)*	*% Carbon*
Mold Powder D	0.2414	7.86
	0.2396	7.89
	0.2407	7.87
	0.2395	7.86
	0.2401	7.82
	$\bar{\chi}$=	***7.86***
	***s*=**	***0.02***

Calibrated with NIST SRM 276b Tungsten Carbide using a linear force through origin calibration.

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