

Determination of Carbon in Nitrides

LECO Corporation; Saint Joseph, Michigan USA

Instrument: C744

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 crushed to a uniform mesh size prior to analysis.

Accessories

528-018 or 528-018HP Crucible (preheated*); 502-492 Copper; 502-231 Iron Chip Accelerator; 773-579 Metal Scoop; 761-929 Tongs.

*Ceramic crucibles must 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 then transferred to a desiccator for storage. Baked crucibles must be handled with clean tongs to avoid contamination.

Calibration

LECO LRM[®] or LCRM[®], NIST, JK, JSS, JCRM, BCS or other suitable Reference Material providers offer a variety of materials which can be used for calibration. During development of this application note JK Nr 21, steel chip CRM was used for calibration. Calibration was verified using JCRM Silicon Nitride CRM's.

Method Parameters

General Parameters

| | |
|------------------|------|
| Purge Time | 15 s |
| Analysis Delay | 20 s |
| Sample Cool Time | 10 s |
| Furnace Power | 100% |

Element Parameters

| | Carbon |
|-------------------|--------|
| Integration Delay | 0 s |
| Starting Baseline | 2 s |
| Use Comparator | No |
| Integration Time | 55 |
| Use Endline | Yes |
| Ending Baseline | 2 s |



Procedure

1. Prepare the instrument and crucibles 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 502-492 Copper and ~ 1 g of 502-231 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 reps.
 - b. Weigh ~ 0.5 g of a steel calibration/drift Reference Material or ~ 0.15 to 0.25 g of a nitride calibration/drift Reference Material into the crucible and enter the mass and standard identification of the standard.
 - 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 to be used 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 ~ 0.15 to 0.25 g nitride 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.
 - e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Typical Results

| Sample | Mass (g) | % Carbon |
|-------------|---------------|--------------|
| **JCRM R007 | 0.1508 | 0.133 |
| Si3N4 | 0.1504 | 0.131 |
| Certified | 0.1502 | 0.131 |
| @ 0.136% | 0.2492 | 0.131 |
| Carbon | 0.2508 | 0.131 |
| | Avg. = | 0.132 |
| | s = | 0.001 |

| | | |
|-------------|---------------|--------------|
| **JCRM R008 | 0.1470 | 0.097 |
| Si3N4 | 0.1481 | 0.098 |
| Certified | 0.1502 | 0.097 |
| @ 0.097% | 0.2481 | 0.097 |
| Carbon | 0.2454 | 0.098 |
| | Avg. = | 0.097 |
| | s = | 0.001 |

| Sample | Mass (g) | % Carbon |
|---------|---------------|--------------|
| ZrN | 0.1504 | 0.049 |
| Reagent | 0.1504 | 0.049 |
| Grade | 0.1495 | 0.049 |
| | 0.1494 | 0.049 |
| | 0.1504 | 0.052 |
| | 0.1496 | 0.049 |
| | 0.1508 | 0.049 |
| | 0.1497 | 0.050 |
| | 0.1491 | 0.049 |
| | 0.1503 | 0.049 |
| | Avg. = | 0.049 |
| | s = | 0.001 |

| Sample | Mass (g) | % Carbon |
|---------|---------------|--------------|
| AlN | 0.1509 | 0.113 |
| Reagent | 0.1492 | 0.114 |
| Grade | 0.1502 | 0.113 |
| | 0.1511 | 0.113 |
| | 0.2012 | 0.116 |
| | 0.1987 | 0.116 |
| | 0.2013 | 0.115 |
| | 0.2502 | 0.115 |
| | 0.2485 | 0.115 |
| | 0.2500 | 0.114 |
| | Avg. = | 0.114 |
| | s = | 0.001 |

| | | |
|---------|---------------|--------------|
| BN | 0.2027 | 0.070 |
| Reagent | 0.1997 | 0.064 |
| Grade | 0.2000 | 0.060 |
| | 0.2007 | 0.064 |
| | 0.2012 | 0.062 |
| | 0.1999 | 0.064 |
| | 0.1999 | 0.062 |
| | 0.2014 | 0.064 |
| | 0.2009 | 0.066 |
| | 0.1998 | 0.062 |
| | Avg. = | 0.064 |
| | s = | 0.003 |

*Calibrated with JK Nr 21 steel chips using linear forced through origin calibration.

**Certified Reference Material by The Ceramic Society of Japan.

