Carbon and Sulfur in Potassium Heptafluorotantalate Materials

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS400/CS-444

Sample Preparation

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.

Accessories

528-018 Crucibles (preheated); 502-173 LECOCEL® II HP, 502-231 Iron Chip, 502-351 Halogen Trap Material, 773-579 Metal Scoop, 781-420 Manual Cleaner, 769-641 Fluorine/Chlorine Trap Kit

Calibration Standard

NIST (steel) or other suitable standard

Sample Weight ~0.1 g

Program Settings

Pre-Analyze Purge:	15 seconds
Pre-Analyze Delay:	25 seconds
C Minimum Timeout:	50 seconds
C Comparator Level:	0.5%
S Minimum Timeout:	60 seconds
S Comparator Level:	1.0%

NOTE: The oxides of Potassium Heptaluorotantalate produced during combustion may cause a flow restriction with the auto cleaner, therefore it is necessary to use the MANUAL CLEANER. The down stream pressure on the furnace will decrease if there is a restriction from these oxides.

Procedure

1. Install a 769-641 Trap in the measure line between the furnace and determinator, following instructions found in the trap kit. Pack the trap with 502-351 Halogen Trap Material or equivalent.
2. Preheat ceramic crucibles in a muffle or tube furnace at 1250°C for not less than 15 minutes or at 1000°C for not less than 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes and placed in a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked.
3. Prepare instrument as outlined in the operator's instruction manual.
4. Determine the blank.
   a. Enter 1.000 g weight into the weight stack.
   b. Add 1.000 g (±0.005 g) of 502-173 LECOCEL II HP and 1.000 g (±0.005 g) of 502-231 Iron Chip to a preheated 528-018 Crucible.
   c. Place the crucible on furnace pedestal and analyze.
   d. Repeat steps 4a through 4c a minimum of three times.
   e. Enter blank following routine outlined in operator's instruction manual.
5. Calibrate.
   a. Weigh ~0.5 g (steel) calibration standard into a preheated 528-018 Crucible and enter weight into the weight stack.
   b. Add 1.000 g (±0.005 g) of 502-173 LECOCEL II HP and 1.000 g (±0.005 g) 502-231 Iron Chip to the preheated crucible.
   c. Place the crucible on furnace pedestal and analyze.
   d. Repeat steps 5a through 5c a minimum of three times and calibrate the instrument following the auto calibration procedure as outlined in the operator's instruction manual.
   e. Verify the calibration by analyzing the calibration standard again. It should fall within the expected tolerance. If not, repeat steps 5a through 5d.
6. Analyze samples.
   a. Weigh ~0.1 g sample into a preheated 528-018 Crucible and enter weight into the weight stack.
   b. Add 1.000 g (±0.005 g) of 502-173 LECOCEL II HP and 1.000 g (±0.005 g) 502-231 Iron Chip to the preheated crucible.
   c. Place the crucible on furnace pedestal and analyze.

Typical Results

Sample	*Weight*	*% Carbon*	*% Sulfur*
K_2TaF_7	0.1071	0.0046	0.0174
	0.1059	0.0053	0.0204
	0.1058	0.0054	0.0185

Inorganic Application Note

LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
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