

SEMI PV59-0115 (Reapproved 0121) TEST METHOD FOR DETERMINATION OF TOTAL CARBON CONTENT IN SILICON POWDER BY INFRARED ABSORPTION AFTER COMBUSTION IN AN INDUCTION FURNACE

This Standard was technically approved by the Photovoltaic Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 10, 2020. Available at www.semiviews.org and www.semi.org in January 2021; originally published January 2015.

1 Purpose

1.1 The purpose of this Test Method is to standardize analytical protocols for the determination of total carbon content in photovoltaic (PV) silicon raw materials which may affect the properties and performances of PV solar product or its processing. This will facilitate the comparison of test results among worldwide laboratories used for monitoring or qualifying PV silicon raw materials.

2 Scope

2.1 This Test Method can be used to analyze the total carbon content in silicon powder as the raw material for producing solar-grade polycrystalline silicon. It may also be used for determining the total carbon content in single and multicrystalline silicon wafers and slugs if they were broken into small pieces or powder without contamination.

2.2 The Test Method is for use with commercially available carbon analyzers which are based on combustion in an induction furnace principle and use infrared detector as the measuring technique.

2.3 The detection range of the method described is between 0.001% (m/m) and 1.0% (m/m).

2.4 Some impurities or carbon from a lower grade accelerator can affect the accuracy of analysis data. It is recommended to use only high purity accelerator.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Not all the information needed to determine the carbon content in PV silicon raw materials is included in this test method. For instance, determination of the optimum temperatures for measuring the carbon content in a variety of silicon samples is not described in this Test Method.

3.2 The limit of detection is determined by the instrumental blank value limitations, and may vary with instrumentation and preparation techniques.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI PV17 — Specification for Virgin Silicon Feedstock Materials for Photovoltaic Applications

4.2 ASTM Standards¹

ASTM E50 — Standard Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

ASTM E122 — Standard Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

ASTM E135 — Standard Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>

4.3 ISO Standards²

ISO 5725-2:1994 — Accuracy (Trueness and Precision) of Measurement Methods and Results – Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method

ISO 9556 — Steel and Iron – Determination of the Total Carbon Content – Infrared Absorption Method After Combustion in an Induction Furnace

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Terminology in this Test Method is consistent with Terminology in ASTM E135. Required terminology specific to this Test Method, not covered in ASTM E135, is indicated in § 5.2.

5.2 Definitions

5.2.1 *reference material* — material accepted as suitable for use as a calibration/sensitivity reference standard by all parties concerned with the analyses.

5.2.2 *silicon powder* — the designation is given to silicon solid materials in accordance with SEMI PV17.

5.2.3 *steel or iron reference material* — steel or iron sample known carbon content (% m/m) with certification.

5.2.4 *test portion* — a portion of the test sample to be tested for carbon content by this method.

5.2.5 *test sample* — material (silicon) to be analyzed for carbon content by this method. Generally the test sample is selected from a large batch of silicon raw materials and is intended to be representative of the batch.

6 Summary of Test Method

6.1 The principle of test method is the same with the standard ISO 9556.

6.2 *Combustion* — Combustion of a test portion with accelerator at a high temperature in a high-frequency induction furnace in a current of pure oxygen.

6.3 *Transformation* — Transformation of carbon into carbon dioxide and/or carbon monoxide.

6.4 *Measurement* — Measurement by infrared absorption of the carbon dioxide and/or carbon monoxide carried by a current of oxygen.

7 Apparatus

7.1 *High-Frequency Induction Furnace and Infrared Carbon Analyzer*

7.2 *Electronic Balance(s)* — 0.1 mg accuracy for weighing test portion, steel or iron reference materials and accelerators; 0.01 mg accuracy for weighing anhydrous sodium carbonate in ¶ 12.2.3.1.

7.3 *Micropipette* — 100 µL, limit of error shall be less than 1 µL.

7.4 *Tin Capsule* — About 6 mm in diameter, 13 to 18 mm in height, 0.4 mL in volume, of known low carbon content less than 0.0005% (m/m).

7.5 *Ceramic Crucible* — Capable of withstanding combustion in an induction furnace. Ignite crucibles in an electric furnace in air or in a current of oxygen for not less than 2 hours at 1000°C and store in a desiccator before use.

8 Reagents and Materials

8.1 *Water Free from Carbon Dioxide* — Boil water for 30 minutes, insert a rubber stopper equipped with a soda-lime drying tube into the neck of the flask bottle, cool to room temperature and bubble with oxygen (see ¶ 8.2) for 15 minutes. Prepare just before use.

8.2 *Oxygen* — 99.5% (m/m) minimum.

² International Organization for Standardization, ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; Telephone: +41.22.749.01.11, <http://www.iso.org>

8.3 *Pure Iron* — Of known low carbon content less than 0.0005% (m/m), particle size less than 1.25 mm, recommended particle size from 0.42 to 0.85 mm (40 ASTM mesh to 20 ASTM mesh).

8.4 *Anhydrous Sodium Carbonate* — Reference Material. Dry anhydrous sodium carbonate (minimum assay: 99.9%) at $285^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 2 hours and cool in a desiccator before use.

8.5 *Sodium Carbonate Standard Solution* — Reference Material Solution, corresponding to 2.50 mg of carbon per milliliter. Weight, to the nearest 0.1 mg, 5.5152 g of anhydrous sodium carbonate (see ¶ 8.4), dissolve in about 200 mL of water free from carbon dioxide (see ¶ 8.1), transfer to a 250 mL one-mark volumetric flask quantitatively, dilute to the mark with water free from carbon dioxide and mix.

8.6 *Tin Accelerator* — High-purity tin particles specified for carbon/sulfur analysis, minimum assay: 99.8%, particle size from 0.42 to 0.85 mm (40 ASTM mesh to 20 ASTM mesh), known carbon content less than 0.0005% (m/m).

8.7 *Tungsten Accelerator* — Minimum assay: 99.9%, particle size from 0.42 to 0.85 mm (40 ASTM mesh to 20 ASTM mesh), known carbon content less than 0.0001% (m/m).

8.8 *Iron-Tungsten-Tin Mixture Accelerator* — Particle size from 0.42 to 0.85 mm (40 ASTM mesh to 20 ASTM mesh), known carbon content less than 0.0003% (m/m).

8.9 *Steel or Iron Reference Materials Series* — Known carbon contents covering from 0.001% to 1.0% (m/m).

9 Sampling

9.1 No general sampling procedure is included as part of this Test Method, because the most suitable sampling plan will vary considerably depending upon individual conditions.

9.2 For referee purposes, a sampling plan shall be agreed upon before conducting the test. See ASTM E122 for suggested choices of sampling plans.

NOTE 1: The test sample to be analyzed is intended to be representative of the status of silicon powder being put into production and it should not be contaminated in sampling procedure. If the test sample may be contaminated it should be cleaned with appropriate cleaning agent such as anhydrous ethanol or isopropyl alcohol before being analyzed.

10 Safety Precautions

10.1 It is assumed that trained laboratory personnel are performing the analysis described under this test method that is familiar with general laboratory safety procedures.

10.2 The risks related to combustion analysis are mainly burns in preigniting the ceramic crucibles and in the fusions. Use crucible tongs at all times and suitable containers for the used crucibles. Normal precautions for handling oxygen cylinders shall be taken. Oxygen from the combustion process should be removed effectively from the apparatus since a high concentration of oxygen in a confined space can present a fire hazard.

10.3 Other safety care in the test process, see ASTM E50.

11 Preparation of Apparatus

11.1 The instrument shall be prepared for operation in accordance with the instrument manufacturer's instructions.

11.2 A minimum of three warm-up specimens are analyzed prior to calibrating the instrument in order to ensure that there are no leaks in the system and the instrument is ready for measurements.

12 Calibration and Standardization

12.1 *Blank Test* — Prior to the determination, carry out the following blank tests in duplicate.

12.1.1 *Blank Specimen*

12.1.2 *Blank Specimen Using Tin Capsule* — Weigh the mass m_{tc} (in grams) of one tin capsule (see ¶ 7.4). Transfer it to the ceramic crucible (see ¶ 7.5), press the tin capsule lightly against the bottom of the ceramic crucible, add $(0.4 - m_{tc})$ gram of the tin accelerator (see ¶ 8.6), 0.3 g of the pure iron (see ¶ 8.3), and cover with 1.5 g of the tungsten accelerator (see ¶ 8.7).