

SEMI PV64-0715 (Reapproved 0121) TEST METHOD FOR DETERMINING B, P, Fe, Al, Ca CONTENTS IN SILICON POWDER FOR PV APPLICATIONS BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY

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 July 2015.

1 Purpose

1.1 Hydrochlorination is a key process for the production of polysilicon. Hydrochlorination consumes a large amount of silicon powder. The contents of B, P, Fe, Al, Ca in silicon powder may affect the purity level of produced trichlorosilane significantly. Therefore, it is necessary to develop a method to measure the contents of B, P, Fe, Al, and Ca in silicon powder.

1.2 This Test Method can facilitate a unifying of protocols and test results among worldwide laboratories used for monitoring or qualifying B, P, Fe, Al, and Ca in silicon powder.

2 Scope

2.1 This Standard specifies a test method for determining the B, P, Fe, Al, Ca contents in silicon powder by inductively coupled plasma optical emission spectrometry (ICP-OES).

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 The element detection range of this Test Method is shown in Table 1, with reference to SEMI C10.

Table 1 Element Detection Range

<i>Element</i>	<i>B</i>	<i>P</i>	<i>Fe</i>	<i>Al</i>	<i>Ca</i>
Mass Percent/%	0.0005~0.25	0.0005~0.50	0.02~1.00	0.02~0.50	0.02~1.00

4 Referenced Standards and Documents

4.1 SEMI Standards and Documents

SEMI C10 — Guide for Determination of Method Detection Limits

SEMI C28 — Specification for Hydrofluoric Acid

SEMI C35 — Specification and Guideline for Nitric Acid

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

SEMI PV49 — Test Method for the Measurement of Elemental Impurity Concentrations in Silicon Feedstock for Silicon Solar Cells by Bulk Digestion, Inductively Coupled-Plasma Mass Spectrometry

4.2 ASTM Standards¹

ASTM D1193 — Standard Specification for Reagent Water

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>

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

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

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 ICP-OES — inductively coupled plasma optical emission spectrometry

5.2 Definitions

5.2.1 *acid blank* — a solution of acid used to establish the background spectrum and trace metal contamination of the acid mixture used in the procedure.

5.2.2 *analytical line* — the optical emission line used for analyzing the specimen.

5.2.3 *Certified Reference Material (CRM)* — reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation issued by a certifying body.

5.2.4 *method blank* — a solution of acid prepared using the preparation method without a specimen or test sample used to establish the contribution of trace metal contamination from the laboratory environment, reagents or laboratory ware to the background.

5.2.5 *silicon powder* — silicon solid materials in accordance with SEMI PV17.

6 Summary of Test Method

6.1 Use mannitol to form complex reaction with boron in the sample (the recovery of boron is 90% to 110%). Remove the silicon matrix with hydrofluoric acid and nitric acid by smoking reaction. Dissolve the residue with nitric acid solution, and use ICP-OES to measure the B, P, Fe, Al, and Ca contents in the prepared solution.

6.2 Key Points of Test Accuracy Control

6.2.1 Drop the nitric acid slowly in the process of sample dissolving, and control the reaction rate to avoid boiling; the sample dissolution temperature shall not exceed 140°C, to prevent over evaporation.

6.2.2 Repeat the standard curve before each sample measurement, and ensure the linear correlation coefficient is no less than 0.9995.

6.2.3 The purity of argon gas shall be over than 99.99% (v/v).

6.2.4 Prevent contamination and sample losses during the sample treatment.

6.2.5 Flush the injection pipe thoroughly, since there is a memory effect when the boron content is high.

6.2.6 Mannitol has the best complex reaction with the boron when the boron content is 0.0005% to 0.25% (m/m).

6.2.7 The process of sample dissolving shall be carried out in a polytetrafluoroethylene (PTFE) or other hydrofluoric acid corrosion-resistant plastic volumetric flask.

6.2.8 Flasks for different reagents or samples shall not be cross-used; and flasks for different concentration of the same reagent or sample shall not be cross-used. When it is necessary, perform a blank analysis test.

² International Organization for Standardization, ISO Central Secretariat, 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland; Telephone: +41.22.749.01.11, Fax: +41.22.733.34.30, <http://www.iso.org>