Determination of Trace Elements in Silicon Powders by Inductively Coupled Plasma Quadrupole Mass Spectrometry With a Membrane Desolvation Sample Introduction System

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ABSTRACT

An inductively coupled plasma quadrupole mass spectrometer, equipped with a desolvation sample introduction system, was successfully used for the accurate determination of B, Al, P, Ti, Fe, and Cu in silicon powder samples. The effects of the operating conditions of the pneumatic nebulization membrane desolvation sample introduction system were optimized to achieve the best signal-to-background ratio for the elements studied. To avoid the vaporization loss of boron during sample dissolution, mannitol was added to the sample. The method alleviated the effects of polyatomic interferences at m/z 27, 31, 47, 48, 56, and 57 caused by $^{28}\text{Si}^+$ spread, $^{14}\text{N}^{16}\text{OH}^+$, $^{28}\text{Si}^{19}\text{F}^+$, $^{29}\text{Si}^{18}\text{F}^+$, $^{28}\text{Si}^{28}\text{Si}^+$, and $^{28}\text{Si}^{29}\text{Si}^+$, respectively. The detection limits

were better than 0.02, 0.007, 0.1, 0.004, 0.1, and 0.002 ng mL⁻¹ for B, Al, P, Ti, Fe, and Cu, respectively. The method was applied to the determination of B, Al, P, Ti, Fe, and Cu in four solar-grade silicon powder samples. The accuracy of the procedure was verified by analyzing certified reference materials NIST SRM 1643e Trace Element in Water for B, Al, Fe, Cu and NIST SRM 2711 Montana Soil for Al, P, Ti, Fe, Cu.

The concentrations of B, Al, P, Ti, Fe, and Cu were determined by external calibration with Rh as the internal standard. Good agreement was obtained between the experimental results and the reference values. The precision between the sample replicates was better than 6% for all of the determinations.

studied in this work. Furthermore, it is difficult to determine low concentrations of analytes in silicon powder without the help of matrix-matched standards. The quantification of trace elements using SIMS is not straightforward and involves complicated calculations, and GD-MS analysis requires the calculation of relative sensitivity factors (RSF).

Inductively coupled plasma mass spectrometry (ICP-MS) is a powerful technique for trace multi-elemental and isotopic analysis. This technique combines the characteristics of the ICP for atomization and ionization with the sensitivity of mass spectrometry. Although ICP-MS is a powerful technique for the measurement of trace elements, it is not always readily applicable for all elements in every possible sample. The determination of B, Al, P, Ti, Fe, and Cu in silicon by quadrupole ICP-MS suffers from high background. With a large concentration of silicon in the sample, ICP-MS suffers from severe matrix suppression and a multitude of silicon-induced spectral interferences. As shown in Table I, the isotopes of Al, P, Ti, Fe, and Cu are interfered by various background ions. Furthermore, since boron is quite volatile, it might be lost during sample dissolution. Another issue with trace ppt level B analysis is background from the torch. Hence, the determination of B, Al, P, Ti, Fe, and Cu in silicon powder by quadrupole ICP-MS is not an easy task. Mannitol [$\text{C}_6\text{H}_8(\text{OH})_6$] has been used to minimize memory

INTRODUCTION

Silicon is the most important material for use in the photovoltaic industry. More than 90% of the annual photovoltaic cell production is based on silicon, and the challenge is to reduce manufacturing costs. Metallurgical grade (MG) silicon (97–99% purity) has been purified to the upgraded metallurgical grade (UMG) silicon, known as solar grade silicon, where impurity concentration has a crucial influ-

ence on the energy efficiency. As a quality check, the silicon samples need to be analyzed for trace elements for which a sensitive and accurate method is required. Several methods such as total reflection X-ray fluorescence spectrometry (TXRF) (1–3), secondary ion mass spectrometry (SIMS) (4), laser-induced breakdown spectrometry (LIBS) (5,6), and glow discharge mass spectrometry (GD-MS) (7) have been reported for trace element determination in silicon. However, the sensitivity of XRF is not sufficient for the determination of the lighter elements, such as B and Al,

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effects (8) and reduce vaporization loss in sample pretreatment for boron determination (9). Chang et. al. (10) utilized an electrodialyzer equipped with an anion permeable membrane to separate matrix elements in the form of SiO_4^- along with other anions such as NO_3^- and F^- . For the analysis of silicon-based samples by ICP-MS, the matrix-based interferences are alleviated using octapole or dynamic reaction cell™ (11,12). The introduction of a membrane desolvation sample introduction system is also an effective method for alleviating metal oxide ion spectral interferences in the ICP-MS analysis (13).

The aim of the present work is to develop an easy and accurate method using pneumatic nebulization membrane desolvation quadrupole ICP-MS to determine trace amounts of B, Al, P, Ti, Fe, and Cu in solar-grade silicon powder samples. The optimization of the pneumatic nebulization membrane desolvation ICP-MS technique and its analytical figures of merit, as well as its application to the determination of B, Al, P, Ti, Fe, and Cu in silicon powder samples, are described.

EXPERIMENTAL

Instrumentation

An ELAN® 6100 DRC™ II ICP-MS instrument (PerkinElmer®, Inc. Shelton, CT, USA) was used for the analyses. The samples were introduced by a membrane desolvation sample introduction system (Aridus-I, Cetac, Omaha, NB, USA). Solutions of 0.1% (m/v) dissolved silicon powder #4 solution, 1 ng mL⁻¹ of Ce in 1% (v/v) HNO_3 and 1% (v/v) HNO_3 (to be treated as the blank) were introduced into the nebulization system successively. The aerosol generated was then transported to ICP-MS for the determination of the ion signals. The conditions of the pneumatic nebulization membrane desolvation sample introduction system were selected to give the best signals for the elements studied while minimizing the spectral interferences effectively. The operating conditions of the membrane desolvation ICP-MS system used in this work are summarized in Table II.

Reagents

Suprapur® HNO_3 (70% m/m) and HF (35% m/m) were obtained from Merck (Darmstadt, Germany). Mannitol and Si, Al, Cu, Fe, P, Rh, and Ti ICP standard solutions were also from Merck. A boron standard solution was obtained from High

Purity Standards, Charleston, SC, USA. All standards and sample solutions were prepared using high purity de-ionized water (Milli-Q™ reagent water system, Millipore Corporation, Bedford, MA, USA). In order to minimize the boron and silicon backgrounds, polypropylene vessels were used throughout this work. The polypropylene vessels used to prepare the sample solutions were soaked overnight in 10% HNO_3 , followed by cleaning in an ultrasonic bath and rinsing with deionized water.

Sample Preparation

The applicability of the method to real samples was demonstrated by the determination of B, Al, P, Ti, Fe, and Cu in four solar-grade silicon powder samples (100 mesh,

TABLE II
Instrumental ICP-MS
Operating Conditions

PerkinElmer ELAN 6100 DRC	
<u>Plasma Conditions</u>	
RF Power	1300 W
Plasma argon flow rate	15 L min ⁻¹
Auxiliary argon flow rate	1.33 L min ⁻¹
Carrier argon flow rate	0.97 L min ⁻¹
<u>Data Acquisition</u>	
Dwell time	100 ms
Scan mode	Peak-hopping
Sweeps per reading	20
Isotopes monitored	¹¹ B, ²⁷ Al, ³¹ P, ⁴⁷ Ti, ⁴⁸ Ti, ⁴⁹ Ti, ⁵⁶ Fe, ⁵⁷ Fe, ⁶³ Cu, ⁶⁵ Cu
<u>Aridus-I Sample Introduction</u>	
<u>Settings</u>	
Sweep gas flow rate	1.2 L min ⁻¹
Membrane temperature	140 °C
N ₂ gas flow rate	0 mL min ⁻¹
Spray chamber temperature	90 °C
Solution flow rate	60 µL min ⁻¹

TABLE I
Isotopes of Interest and Most Abundant Polyatomic Interferences

Isotope	Abundance (%)	Interferences
²⁷ Al	100	²⁸ Si spread
³¹ P	100	³⁰ SiH, ¹⁴ N ¹⁶ OH, ¹⁵ N ¹⁶ O
⁴⁷ Ti	7.32	²⁸ Si ¹⁹ F, ²⁹ Si ¹⁸ O, ¹⁵ N ¹⁶ O ¹⁶ O
⁴⁸ Ti	73.98	²⁹ Si ¹⁹ F, ¹⁴ N ¹⁶ O ¹⁸ O
⁴⁹ Ti	5.46	³⁰ Si ¹⁹ F
⁵⁴ Fe	5.82	⁴⁰ Ar ¹⁴ N, ³⁸ Ar ¹⁶ O
⁵⁶ Fe	91.66	⁴⁰ Ar ¹⁶ O, ²⁸ Si ²⁸ Si
⁵⁷ Fe	2.19	⁴⁰ Ar ¹⁷ O, ²⁸ Si ²⁹ Si
⁶³ Cu	69.17	⁴⁷ Ti ¹⁶ O, ²⁸ Si ³⁵ Cl, ²⁸ Si ¹⁷ O ¹⁸ O, ⁴⁰ Ar ²³ Na
⁶⁵ Cu	30.83	⁴⁷ Ti ¹⁸ O, ²⁸ Si ³⁷ Cl, ²⁹ Si ¹⁸ O ¹⁸ O

99.9–99.99% purity) obtained from ITRI (Industrial Technology Research Institute, Hsinchu, Taiwan). The procedure was initially applied to the analysis of reference material NIST SRM 1643e Trace Element in Water and the accuracy of the procedure was verified by analyzing the silicon-rich reference material SRM 2711 Montana Soil (National Institute of Standards and Technology, Gaithersburg, MD, USA).

A simple sample dissolution procedure was used in this study as follows: 0.1 g silicon powder was transferred into a 10-mL polypropylene flask; then 0.1 mL 0.5% (m/v) mannitol and 1 mL concentrated HNO_3 were added successively, followed by five 0.2-mL aliquots of concentrated HF (1 mL total). The digest was diluted to the mark with pure water, followed by a 1:10 dilution for ICP-MS analysis. The B, Al, P, Ti, Fe, and Cu concentrations in the sample were quantified by external calibration with 1 ng mL⁻¹ Rh as the internal standard. For the spike recovery study, 0.1 g powders of Sample #4 were spiked with 10 $\mu\text{g g}^{-1}$ of B, Ti and Cu, 50 $\mu\text{g g}^{-1}$ of P, and 100 $\mu\text{g g}^{-1}$ of Al and Fe (procured from Merck), dried, and then dissolved using the procedure described above.

For studying the effects of various Aridus parameters on the minimization of silicon-induced spectral interferences, a 0.1% (m/v) silicon powder solution of Sample #4 was used as the model solution. A 1-ng mL⁻¹ Ce solution was used to study the effect of Aridus conditions on the reduction of oxide ion interferences. The SRM 2711 Montana Soil reference sample was dissolved using the method described in a previous paper (14).

RESULTS AND DISCUSSION

In the preliminary experiments, a microwave-assisted vapor phase dissolution method described in a

previous paper was tested to determine B, Al, P, Ti, Fe, and Cu in silicon powder (15). These experiments showed that the determined concentrations of B and P were lower than the GD-MS values provided by ITRI, even when mannitol was added to the silicon powder before sample dissolution. This could be due to the vaporization loss of B and P when the sample was heated in a closed system at elevated temperatures. Hence, conventional dissolution was attempted.

Mannitol was used to reduce the vaporization loss during acid dissolution. The effect of the amount of mannitol added to the silicon powder on the recovery of boron was studied. The experiments showed that the recovery was quantitative when 0.1 g powder was spiked with more than 500 μg of mannitol before sample dissolution. In addition to the improvement of boron recovery, the addition of mannitol, HNO_3 , and HF could also minimize the memory effect of boron. In this study, a suitable amount of mannitol (500 μg mannitol in 0.1-g silicon powder) was added before sample dissolution.

The determination of phosphorus is difficult since its only isotope ($^{31}\text{P}^+$) suffers from severe spectral interference caused by $^{14}\text{N}^{16}\text{OH}^+$ and $^{30}\text{SiH}^+$, especially when HNO_3 is used for sample dissolution. The DRC ICP-MS with O_2 as the reaction gas has been used to minimize the $^{14}\text{N}^{16}\text{OH}^+$ interference on phosphorus determination by converting the $^{31}\text{P}^+$ to $^{31}\text{P}^{16}\text{O}^+$ in the reaction cell and measuring at m/z 47 (16). The method was tested for the determination of phosphorus in silicon powder. However, from the experiment it was found that background interference was more serious when the DRC mode was used, due to the interferences caused by the $^{28}\text{Si}^{19}\text{F}^+$ and/or $^{30}\text{Si}^{16}\text{OH}^+$ at m/z 47. Hence, the ICP-MS operated in DRC mode could not be used in

this work. As an alternative, membrane desolvation sample introduction was attempted.

Selection of Aridus Conditions

As shown in Table I, the determination of Al, P, Ti, Fe, and Cu in solutions with a high silicon content by quadrupole ICP-MS suffers from high background problems. In order to evaluate the significance of these interferences, experiments were performed to check the interferences caused by silicon-induced ions. Solutions containing 10 ng mL⁻¹ Al, Ti and Fe, 100 $\mu\text{g mL}^{-1}$ Si in 1% (v/v) HF and 1% (v/v) HNO_3 and 1% (v/v) HNO_3 (to be treated as blank) were prepared. The solutions were introduced into the ICP-MS by conventional cross-flow pneumatic nebulization with a Scott-type spray chamber. The ion signals at the masses of interest were determined, and the results are shown in Figure 1. As shown, a solution containing 100 $\mu\text{g mL}^{-1}$ Si in 1% (v/v) HF and 1% (v/v) HNO_3 produced a background signal equivalent to 4.5 times of the signal of 10 ng mL⁻¹ Ti at m/z 47 and 26% of the signal of 10 ng mL⁻¹ Ti at m/z 49. To minimize the interference, after evaluation, a sample introduction device with membrane desolvation (Aridus-D) was selected for this study. Various parameters of the Aridus, including nebulizer gas flow rate, sweep gas flow rate, membrane temperature, and N_2 gas flow rate, were studied to achieve maximum ion signals and minimum background interferences. Both of the ion signals of the elements studied and the ratios of $^{47}\text{Ti}/^{49}\text{Ti}$ and CeO/Ce were monitored. The $^{47}\text{Ti}/^{49}\text{Ti}$ ratio will be larger than 1.33 (the natural isotopic abundance ratio of $^{47}\text{Ti}/^{49}\text{Ti}$) when the determination of $^{47}\text{Ti}^+$ and $^{49}\text{Ti}^+$ is interfered by $^{28}\text{Si}^{19}\text{F}^+$ and $^{30}\text{Si}^{19}\text{F}^+$, respectively. A $^{47}\text{Ti}/^{49}\text{Ti}$ ratio close to 1.33 and a small value of CeO/Ce indicates the minimization of spectral interferences. In this study, 0.1% (m/v) solution of silicon pow-

der #4 and 1 ng mL⁻¹ Ce solution were used as the model solutions.

The effects of nebulizer gas flow rate on the Ti signal and ⁴⁷Ti/⁴⁹Ti ratio are shown in Figure 2. As shown, the Ti signal increased with an increase of nebulizer gas flow rate due to the increase of nebulization efficiency. Meanwhile, the ⁴⁷Ti/⁴⁹Ti ratio increased with the nebulizer gas flow rate when the gas flow rate was less than 0.89 L min⁻¹. The effect of nebulizer gas flow rate on oxide formation was also studied. As shown in Figure 3, the Ce ion signal increased with an increase in gas flow rate, while the CeO/Ce ratio decreased slightly with the nebulization gas flow rate. A nebulization gas flow rate of 0.97 L min⁻¹ was selected for this study.

A sweep gas is needed to remove the SiF₄ and the solvent vapor after they penetrate the membrane. The effect of the sweep gas flow rate on the ion signals and the background interferences are shown in Figure 4. It can be seen that the Ti⁺ signal increased with an increase of sweep gas flow when the flow rate was less than 1.2 L min⁻¹. The increase of the ion signal could be due to the removal of the major matrix component which minimized the ion suppression effect. When the sweep gas flow rate was larger than 1.2 L min⁻¹, the Ti⁺ signal decreased and the ⁴⁷Ti/⁴⁹Ti ratio increased. The high sweep gas flow rate might prohibit the penetration of SiF₄ vapor through the membrane and cause non-spectral and spectral interferences. As shown in Figure 5,

a similar behavior was detected for the Ce ion signal and CeO/Ce ratio. As can be seen, the Ce ion signal decreased and the CeO/Ce ratio increased when the sweep gas flow rate was larger than 1.2 mL min⁻¹. Hence, 1.2 mL min⁻¹ was selected for the following experiments.

The effects of nitrogen gas flow rate on the Ti ion signal and ⁴⁷Ti/⁴⁹Ti ratio are shown in Figure 6. As shown, the Ti signal decreased significantly with an increase in the N₂ gas flow rate. In contrast, the ⁴⁷Ti/⁴⁹Ti ratio increased slightly with the N₂ gas flow rate. Although not shown, the CeO/Ce ratio also increased slightly with the N₂ gas flow rate. In this study, no N₂ gas was added (i.e., N₂ flow = 0).

Fig. 1. Ion signals of 10 ng mL⁻¹ of Al, Ti, and Fe and background signals of 100 µg mL⁻¹ Si in 1% v/v HF and 1% v/v HNO₃ solution; (a) ion signal and (b) relative signal.

Fig. 2. Effects of nebulizer gas flow rate on Ti ion signal and ⁴⁷Ti/⁴⁹Ti ratio. Membrane temperature = 160 °C; Sweep gas flow rate = 1.2 L min⁻¹; N₂ gas flow rate = 0 mL min⁻¹.

Fig. 3. Effects of nebulizer gas flow rate on Ce ion signal and CeO/Ce ratio. Membrane temperature = 160 °C; Sweep gas flow rate = 1.2 L min⁻¹; N₂ gas flow rate = 0 mL min⁻¹.

Fig. 4. Effect of the sweep gas flow rate on Ti ion signal and ⁴⁷Ti/⁴⁹Ti ratio. Nebulization gas flow rate = 0.97 L min⁻¹; Membrane temperature = 160 °C; N₂ gas flow rate = 0 mL min⁻¹.

Fig. 5. Effect of the sweep gas flow rate on Ce ion signal and CeO/Ce ratio. Nebulization gas flow rate = 0.97 L min⁻¹; Membrane temperature = 160 °C; N₂ gas flow rate = 0 mL min⁻¹.

Fig. 6. Effects of nitrogen gas flow rate on Ti ion signal and ⁴⁷Ti/⁴⁹Ti ratio. Nebulization gas flow rate = 0.97 L min⁻¹; Membrane temperature = 160 °C; Sweep gas flow rate = 1.2 L min⁻¹.

The effect of membrane temperature on the Ti ion signal and the $^{47}\text{Ti}/^{49}\text{Ti}$ ratio is shown in Figure 7. As shown, the increase of membrane temperature did not affect the $^{47}\text{Ti}/^{49}\text{Ti}$ ratio; however, the signal of Ti increased slightly with the membrane temperature, which could be due to the more effective removal of the Si matrix. It was also found that the membrane temperature did not affect the CeO/Ce ratio when the temperature was higher than 110 °C. Furthermore, the boron signal decreased slightly when the membrane temperature was higher than 130 °C. The decrease of boron ion signal could be due to the vaporization of boron. After evaluation, 140 °C was selected. A summary of the optimized operating conditions of the membrane desolvation ICP-MS is given in Table II (see page 2).

Figure 8 shows the reduction of spectral interferences in the determination of Al, Ti, and Fe when the

Aridus was employed as a sample introduction device. As shown, the background at m/z 47 reduced significantly when the Aridus was used as the sample introduction device. The remaining background signals detected at various isotopes could be due to the trace contamination of the analytes in the reagents used for the preparation of the simulated matrix solution. Furthermore, from the experiment it was found that the background ion signal at m/z 31 and 57 was reduced from 13,200 and 8490 c s⁻¹ to 1540 and 695 c s⁻¹, respectively, when a blank solution containing 5 µg mL⁻¹ mannitol in 1% HNO₃ and 1% HF was introduced. These experiments demonstrated the effectiveness of the Aridus sample introduction system for the minimization of spectral interferences in ICP-MS analysis. Under the optimum conditions, calibration curves (six points) in the range of 5–300 ng mL⁻¹ for P and 1–50 ng mL⁻¹ for B, Al, Ti, Fe, and

Cu were linear with a coefficient of determination (r^2) better than 0.9991. The detection limit estimated from the calibration curve was better than 0.02, 0.007, 0.1, 0.004, 0.1, and 0.002 ng mL⁻¹ for B, Al, P, Ti, Fe, and Cu, respectively. The detection limits obtained in this work are low enough to determine various trace elements in solar-grade silicon.

Determination of B, Al, P, Ti, Fe, and Cu in Reference Materials and Silicon Powder

In order to prove that the proposed method is suitable for practical analysis, the concentrations of B, Al, P, Ti, Fe, and Cu were determined in NIST SRM 1643e Trace Element in Water and NIST SRM 2711 Montana Soil reference samples, and four silicon powder samples obtained from ITRI. The concentrations of B, Al, P, Ti, Fe, and Cu present in these samples were quantified by the external

Fig. 7. Effect of membrane temperature on Ti ion signal and the $^{47}\text{Ti}/^{49}\text{Ti}$ ratio. Nebulization gas flow rate = 0.97 L min⁻¹; Sweep gas flow rate = 1.2 L min⁻¹; N₂ gas flow rate = 0 mL min⁻¹.

Fig. 8. Ion signals of 10 ng mL⁻¹ of Al, Ti and Fe and background signals of 100 µg mL⁻¹ Si in 1% v/v HF and 1% v/v HNO₃ solution when (a) conventional cross-flow nebulizer and (b) Aridus was used as the sample introduction device, respectively.

calibration method with 1 ng mL⁻¹ Rh as the internal standard. The analysis results of the reference samples are listed in Table III. Agreement of the obtained results with the certified values of the standard reference materials was good.

The developed method was applied to the determination of B, Al, P, Ti, Fe, and Cu in four solar-grade silicon powder samples. The results are listed in Table IV. As shown, the Al, Ti, and Fe results of the conventional pneumatic nebulization method were high, most likely due to the interferences caused by the silicon-induced ions. The membrane desolvation ICP-MS results of B, P, Fe, and Cu were in good agreement with the GD-MS reference values, although the Al and Ti results were higher than the GD-MS values, possibly due to inhomogeneity or RSF values. However, the DRC will alleviate the interferences associated with the determination of Al, Fe, and Cu. However, the aim of the present work is the development of a simpler procedure and hence DRC was not used. (As mentioned in the text, the GD-MS values are given by ITRD). Furthermore, the membrane desolvating ICP-MS results of B, Al, P, Fe, and Cu determination were in good agreement with those obtained by USS-ETV-ICP-MS (17) in accordance with the Student's *t*-test for a confidence level of 95%. The spiked recovery was determined using the procedure described in the experi-

mental section. The recovery was in the 94–107% range for the elements studied. Hence, the proposed membrane desolvation ICP-MS method is suitable for the determination of B, Al, P, Ti, Fe, and Cu in silicon powder.

CONCLUSION

The use of pneumatic nebulization membrane desolvation inductively coupled plasma mass spectrometry (ICP-MS) provides a simple, rapid, and accurate technique to determine B, Al, P, Ti, Fe, and Cu routinely in solar-grade silicon samples. The effectiveness of the membrane desolvation system for the alleviation of spectral interferences was demonstrated. As a result of interference-free measurements, detection limits of better than 0.02, 0.007, 0.1, 0.004, 0.1, and 0.002 ng mL⁻¹ for B, Al, P, Ti, Fe and Cu, respectively, were achieved. An easy and simple sample dissolution method was developed. The addition of mannitol for the minimization of boron vaporization loss has also been demonstrated.

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TABLE III
Determination of B, Al, P, Ti, Fe and Cu in Standard Reference Materials by Membrane Desolvation ICP-MS^a
(n = 3)

Sample		B	Al	P	Ti	Fe	Cu
NIST SRM 2711							
Montana soil	Measured value	–	2.64±0.10 ^c	0.087±0.002 ^c	0.317±0.018 ^c	2.78±0.06 ^c	114±1 ^d
	Certified value	–	2.00~3.10 ^{b,c}	0.086±0.007 ^c	0.306±0.023 ^c	2.89±0.06 ^c	114±2 ^d
NIST SRM 1643e							
Fresh water	Measured value	155.7±0.8 ^c	147.3±1.5 ^c	–	–	102.5±3.4 ^c	22.71±0.13 ^c
	Certified value	157.9±3.9 ^c	141.8±8.6 ^c	–	–	98.1±1.4 ^c	22.76±0.31 ^c

^a Values are means of three measurements ± standard deviation.

^b Leach data range; ^c Values are in %; ^d Values are in µg g⁻¹; ^e Values are in ng mL⁻¹.

TABLE IV
Determination of B, Al, P, Ti, Fe, and Cu in Silicon Powders by ICP-MS^a (n = 3)

Sample	Method	B	Al	P	⁴⁷ Ti	⁴⁸ Ti	Fe	Cu
Sample #1	Cross-flow Nebulizer ^b	13.3±0.4	81.3±7.1	52.6±3.4	276±11	16.7±0.6	87.7±4.8	197±5
	ARIDUS ^c	14.0±0.7	76.9±4.3	43.6±1.7	14.4±0.2	13.3±0.3	82.2±4.8	203±2
	GD-MS ^d	n.a. ^e	51	n.a.	11	74	258	
Sample #2	Cross-flow Nebulizer	12.4±0.3	149±7	76.2±2.7	258±14	28.6±1.2	112±4	17.0±0.6
	ARIDUS	12.7±0.6	136±4	66.3±1.4	28.0±0.9	26.6±0.6	101±4	16.7±0.2
	GD-MS	11	110	66	22	94	22	
Sample #3	Cross-flow Nebulizer	155±5	154±11	73.9±2.6	252±9	27.5±0.9	171±5	9.8±0.4
	ARIDUS	151±7	146±8	57.6±2.6	27.8±0.3	25.1±0.6	163±2	9.8±0.2
	GD-MS	150	120	58	19	160	8.9	
Sample #4	Cross-flow Nebulizer	13.5±0.3	155±8	79.2±3.8	267±10	32.6±0.6	164±3	7.2±0.1
	ARIDUS	14.2±0.8	147±4	57.7±1.1	31.8±0.7	29.3±0.2	156±6	7.4±0.1
	GD-MS	14	110	62	24	160	8.8	

^a Values are means of three measurements ± standard deviation; all values are in µg g⁻¹.

^b Sample solution contained 0.1% m/v silicon powder, 5 µg mL⁻¹ mannitol in 1% HNO₃ and 1% HF. Solutions were introduced with conventional cross-flow nebulizer.

^c Sample solution contained 0.1% m/v silicon powder, 5 µg mL⁻¹ mannitol in 1% HNO₃ and 1% HF. Solutions were introduced with Aridus-I system.

^d GD-MS reference values were provided by ITRI.

^e Not available.

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