

Industrial

Analysis of photovoltaic grade silicon using triple quadrupole inductively coupled plasma mass spectrometry (ICP-MS)

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Keywords

Bulk analysis, photovoltaic grade, silicon matrix, silicon wafer, trace analysis, triple quadrupole, TQ-O₂ mode

Goal

To demonstrate a robust and accurate analytical method for the determination of bulk and trace elements in photovoltaic samples using triple quadrupole ICP-MS

Introduction

Development of renewable and low-carbon energy sources has become critical in addressing global concerns related to carbon emissions and climate change. One key technology in achieving the reduction of CO₂ emissions while ensuring a stable and reliable energy supply is the use of photovoltaic (PV) technology. PV technology harnesses the natural energy from the sun and converts it into electrical power, emits zero CO₂, and enhances energy security. Additionally, PV solar cells are predominantly composed of silicon, an abundant resource on Earth, making the transition to PV energy a viable option as a primary source of electricity, aligning with the move towards climate-friendly energy resources.

However, it is important to acknowledge that PV also has certain drawbacks, including susceptibility to damage, dependence on sunlight, and associated costs. To address these concerns, it is crucial to focus on developing innovative solutions that result in durable products, increased energy efficiency, and degradation prevention.

Photovoltaic cells are typically composed of n-type and p-type solar materials. The n-type solar cell is doped with phosphorus, which introduces an additional electron compared to silicon, while the p-type solar cell is doped with boron, which has one less electron than silicon. The performance and production cost of PV cells are influenced by the characteristics of these two types of materials. Consequently, it is crucial to carefully determine the concentration of phosphorus (among other impurities) during the development of PV technology to optimize its efficiency and cost-effectiveness. Inductively coupled plasma mass spectrometry (ICP-MS) is a highly sensitive technique that can work with low sample volumes while enabling high sample throughput and robust analysis even in the most demanding sample types. However, the analysis of samples containing higher levels of silicon is a known challenge in ICP-MS. The high amount of silicon matrix can significantly affect the sensitivity of the instrument, cause intensity fluctuation of the internal standard (suppression and drift), and lead to increased system maintenance with unwanted downtime due to obstruction of the interface cones, torch, and injector, or the nebulizer. In addition, samples need to be digested or dissolved using hydrofluoric acid (HF), which can damage glassware such as the nebulizer, spray chamber, and injector unless HF-resistant materials are used.

Some elements may be more difficult to detect in a matrix containing elevated levels of silicon. Phosphorus, for example, is not only affected by abundant polyatomic interferences, but also from an isobaric interference caused by the matrix (^{31}Si interfering with the only natural isotope of Phosphorus). Even powerful interference removal technology typically used with single quadrupole ICP-MS systems, such as kinetic energy discrimination (KED) mode, is not able to resolve the isobaric interference. However, reactive gases can be used for interference removal, enabling detection of phosphorus when using triple quadrupole ICP-MS technology.

This application note focuses on the development of a fast, robust, and accurate method for the analysis of bulk and trace elements in photovoltaic grade silicon using the Thermo Scientific™ iCAP™ MTX ICP-MS with argon gas dilution (AGD). Use of triple quadrupole technology offered superior interference removal and facilitated the analysis of phosphorus, including ultra-trace levels in the silicon matrix samples.

Experimental

Experimental optimization of the instrument parameters

An iCAP MTX ICP-MS, operated together with a Thermo Scientific™ iSC-65 Autosampler, was used for all analyses. The sample introduction system consisted of a Peltier-cooled (at 2.7 °C) and dedicated HF sample introduction kit consisting of a baffled PFA cyclonic spray chamber (SavilleX, Eden Prairie, MN, USA), PFA concentric nebulizer (SavilleX), and PLUS torch¹ with a 2.0 mm i.d. removable sapphire injector. The built-in argon humidifier was used for moisturizing the nebulizer gas to prevent salt deposit on the nebulizer tip, along with integrated argon gas dilution.

To further increase uptime of the instrument, intelligent matrix handling was used, a unique feature of the Thermo Scientific™ iCAP™ MX Series ICP-MS instruments to reduce exposure to sample matrix during sample uptake and wash. Interferences on all analytes were effectively controlled using the QCell collision/reaction cell (CRC) and leveraging the superior interference removal capabilities of a triple quadrupole ICP-MS instrument. In addition to the use of O₂ as a reactive gas in triple quadrupole mode for highly interfered analytes such as phosphorus, sulfur, arsenic and selenium, the instrument was also operated in single quadrupole mode using helium and kinetic energy discrimination (KED) for interference free analysis of other analytes over the full mass range. Table 1 summarizes the instrument configuration and analytical parameters.

Table 1. Instrument configuration and operating parameters

Parameter	Value	
Nebulizer	PFA concentric nebulizer, 400 $\mu\text{L}\cdot\text{min}^{-1}$	
Peristaltic pump tubing	PVC orange-yellow tubing, 0.51 mm i.d.	
Peristaltic pump speed	25 rpm	
Humidifier	On	
Spray chamber	PFA cyclonic, cooled at 2.7 °C	
Torch	PLUS torch	
Injector	2.0 mm i.d., Sapphire	
Interface	Nickel sampler and skimmer cone	
Plasma power	1,550 W	
AGD setting	Level 5	
AGD flow	0.50 $\text{L}\cdot\text{min}^{-1}$	
Nebulizer gas	0.45 $\text{L}\cdot\text{min}^{-1}$	
QCell setting	He KED	TQ-O₂
QCell gas flow	100% He 4.2 $\text{mL}\cdot\text{min}^{-1}$	100% O ₂ 0.32 $\text{mL}\cdot\text{min}^{-1}$
CR bias	-21 V	-6.3 V
Q3 bias	-18 V	-12 V
Scan setting	0.1 s dwell time, 5 sweeps, 3 main runs	
Analysis time per sample	Total 3 min 33 s: including uptake (50 s) and wash out (25 s), Step Ahead (20 s), matrix stabilization time (14 s)	

The selection of the most appropriate isotopes per analyte, as well as the optimum analysis conditions (i.e., KED mode versus reactive gas, on mass mode versus mass shift mode) was automatically accomplished using the Reaction Finder Method Development Assistant available in the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. Measurement modes were optimized using the provided autotune procedures.

The iSC-65 Autosampler allowed for the use of Step Ahead functionality to shorten the overall measurement time per sample and increase the number of samples that can be analyzed per unit time.

Sample preparation

Pre-cleaned (72 hours in 2% HNO₃ (Optima™ grade, Fisher Chemical™)) polypropylene bottles were used for the preparation of all blanks, standards, and samples.

A silicon wafer was analyzed for this study as a proxy for photovoltaic grade silicon (Figure 1). To dissolve the sample,



Figure 1. Silicon wafer sample

10 g hydrofluoric acid (50% Optima™ grade HF, Fisher Chemical™) and 10 g nitric acid (68% Optima™ grade HNO₃, Fisher Chemical™) were added gently to approximately 1.45 g of silicon wafer in a PFA sample preparation vessel at room temperature. After the silicon wafer was fully dissolved, the sample was made up to a final volume of 100 mL using ultrapure water. The final analysis sample solution was then prepared by a manual 20-fold dilution.

To assess the potential for quantitative analysis of impurities in a sample matrix containing high levels of silicon, spike recovery tests were performed at a level of 10 µg·L⁻¹ in silicon solutions ranging from 10 mg·L⁻¹ to 5,000 mg·L⁻¹, prepared from the 10,000 mg·L⁻¹ silicon standard solution (SPEX™ CertiPrep™, Metuchen, NJ, USA).

To demonstrate the capability of the instrument for accurate and precise analysis despite the challenging sample matrix, the internal standards were closely monitored for matrix suppression.

A sample dilution, calibration blank, a series of standards and a quality control (QC) sample were prepared using 2% v/v HNO₃ (Optima™ grade, Fisher Chemical™). All samples and standards were spiked with an internal standard mix (10 µg·L⁻¹ Y, Rh, and Ir). The elements and final concentrations are shown in Table 2.

Results and discussion

Sensitivity and linearity

Table 3 summarizes the measurement modes, instrument detection limits (IDLs) obtained, and the coefficient of determination (R²) for all 42 elements analyzed in this study. The correlation coefficients (R²) obtained for all analytes were found to be greater than 0.9991, which suggests excellent linear response for the established concentration range for each analyte. The IDLs were calculated as three times the standard deviation of ten replicate measurements of the calibration blank. Note that these LODs include the dilution factor applied using AGD.

Table 2. Summary of the concentration details of the standard calibration, CCV (continuing calibration verification). All numbers are shown in µg·L⁻¹.

Standard group	STD-1	STD-2	STD-3	STD-4	STD-5	QC	Spike recovery
Al, As, Ag, Ba, Bi, Co, Cr, Ni, V, U, Pb, Mn, Ga, Tl, Se, Cd, In, Cu, Zn, Rb, Sr, Mg, Fe, Ge, Mo, Nb, Re, Ta, Ti, Zn, W, Sn, Sb, Hg	0.1	1	5	10	-	1	10
Na, P, K, Ca, S, B, Be	2.5	5	10	20	-	5	10
Ge (for silicon wafer sample)	10,000	100,000	500,000	-	-	1,000	10,000
Si	10	10,000	100,000	1,000,000	5,000,000	10,000	500

Table 3. Summary of analysis mode, and calibration results, R², LODs, and BECs for all analytes. All numbers are shown in µg·L⁻¹.

Analyte	Mode	Q3 analyte	Q1 resolution	Q3 resolution	Internal standard	LOD	BEC	R ²
⁷ Li	TQ-O ₂	⁷ Li	High	Normal	Y	0.013	0.002	0.9996
⁹ Be	TQ-O ₂	⁹ Be	High	Normal	Y	<0.001	<0.001	0.9999
¹¹ B	TQ-O ₂	¹¹ B	High	Normal	Y	0.17	0.21	0.9999
²³ Na	He KED	-	-	Normal	Y	1.2	12.5	0.9991
²⁴ Mg	He KED	-	-	Normal	Y	0.36	0.16	0.9997
²⁷ Al	TQ-O ₂	²⁷ Al	High	Normal	Y	0.10	0.18	<0.9999
²⁸ Si	KED	-	-	High	Y	33.5	259	0.9999
²⁸ Si	TQ-O ₂	²⁸ Si, ¹⁶ O	High	High	Y	0.9	2.9	0.9997
³¹ P	TQ-O ₂	³¹ P, ¹⁶ O	High	Normal	Y	0.12	0.14	0.9993
³¹ P	KED	-	-	High	Y	12.9	7.29	0.9101
³² S	TQ-O ₂	³² S, ¹⁶ O	High	Normal	Y	1.4	16.7	0.9994
³⁹ K	TQ-O ₂	³⁹ K	High	Normal	Y	0.25	3.1	<0.9999
⁴⁴ Ca	KED	-	-	Normal	Y	1.7	1.6	0.9994
⁴⁸ Ti	TQ-O ₂	⁴⁸ Ti, ¹⁶ O	High	Normal	Y	0.009	0.007	<0.9999
⁵¹ V	TQ-O ₂	⁵¹ V, ¹⁶ O	High	Normal	Y	0.002	0.000	0.9998
⁵² Cr	TQ-O ₂	⁵² Cr, ¹⁶ O	High	Normal	Y	0.067	0.114	0.9999
⁵⁵ Mn	TQ-O ₂	⁵⁵ Mn	iMS	Normal	Y	0.007	0.005	0.9999
⁵⁶ Fe	KED	-	-	Normal	Y	0.036	0.130	0.9998
⁵⁹ Co	TQ-O ₂	⁵⁹ Co	High	Normal	Y	0.009	0.005	0.9998
⁶⁰ Ni	KED	-	-	Normal	Y	0.040	0.078	0.9998
⁶³ Cu	TQ-O ₂	⁶³ Cu	High	Normal	Y	0.027	0.019	0.9999
⁶⁶ Zn	TQ-O ₂	⁶⁶ Zn	iMS	Normal	Y	0.024	0.036	0.9999
⁷¹ Ga	KED	-	-	Normal	Y	0.003	0.001	0.9999
⁷² Ge	TQ-O ₂	⁷² Ge	High	Normal	Y	0.011	0.006	0.9998
⁷⁵ As	TQ-O ₂	⁷⁵ As, ¹⁶ O	High	Normal	Y	0.021	0.006	0.9998
⁷⁸ Se	KED	-	-	Normal	Y	0.591	0.887	0.9965
⁸⁰ Se	TQ-O ₂	⁸⁰ Se, ¹⁶ O	High	Normal	Y	0.035	0.004	0.9996
⁸⁵ Rb	TQ-O ₂	⁸⁵ Rb	iMS	Normal	Y	0.002	0.002	0.9998
⁸⁸ Sr	TQ-O ₂	⁸⁸ Sr, ¹⁶ O	High	Normal	Y	0.003	0.001	0.9998
⁹⁰ Zr	TQ-O ₂	⁹⁰ Zr, ¹⁶ O	iMS	Normal	Y	0.033	0.126	0.9999
⁹³ Nb	KED	-	-	Normal	Y	<0.001	<0.001	0.9991
⁹⁵ Mo	KED	-	-	Normal	Y	0.003	0.001	0.9991
¹⁰⁷ Ag	TQ-O ₂	¹⁰⁷ Ag	iMS	Normal	Rh	0.006	0.010	0.9998
¹¹¹ Cd	TQ-O ₂	¹¹¹ Cd	iMS	Normal	Rh	0.003	0.001	0.9999
¹¹⁵ In	TQ-O ₂	¹¹⁵ In	iMS	Normal	Rh	<0.001	<0.001	0.9998
¹¹⁸ Sn	TQ-O ₂	¹¹⁸ Sn	iMS	Normal	Rh	0.003	0.002	<0.9999
¹²¹ Sb	TQ-O ₂	¹²¹ Sb	iMS	Normal	Rh	0.005	0.002	0.9993
¹³⁷ Ba	KED	-	-	Normal	Rh	0.001	0.000	0.9999
¹⁸¹ Ta	KED	-	-	Normal	Ir	<0.001	<0.001	0.9991
¹⁸² W	KED	-	-	Normal	Ir	0.001	<0.001	0.9998
¹⁸⁵ Re	KED	-	-	Normal	Ir	<0.001	<0.001	0.9999
²⁰² Hg	KED	-	-	Normal	Ir	0.010	0.014	0.9999
²⁰⁵ Tl	KED	-	-	Normal	Ir	0.001	<0.001	0.9999
²⁰⁸ Pb	KED	-	-	Normal	Ir	0.001	0.001	0.9998
²⁰⁹ Bi	KED	-	-	Normal	Ir	0.001	0.002	0.9998
²³⁸ U	KED	-	-	Normal	Ir	<0.001	<0.001	0.9999

Phosphorus and selenium were determined using both analysis modes to compare the performance, and the results showed that the use of TQ-O₂ mode delivered at least 10 times better LODs. In the case of selenium, the use of TQ technology also allows the analysis of ⁸⁰Se, the most abundant isotope of this element, which is normally inaccessible to analysis using KED mode due to abundant argon-based polyatomic interferences.

Accuracy and polyatomic interference removal for the analysis of silicon matrix samples

Although a small amount of silicon volatilizes following the reaction with HF during the sample dissolution process, the remaining amount of silicon in the solutions to be analyzed poses a significant challenge to the analysis of some analytes. Phosphorus is a key analyte in photovoltaic grade products; thus, it is very important to be determined both for research and development of new products but also for quality control during production. In general, kinetic energy discrimination (KED) mode is a powerful technique for suppressing the polyatomic interferences; however, the high silicon matrix generates both polyatomic and isobaric interferences (such as ³¹Si, ³⁰Si¹H) and leads to the reduction of sensitivity for low mass analytes.

In addition, phosphorus itself has a high first ionization potential energy (10.487 KJ·mol⁻¹), and, consequently, the sensitivity is lower than other analytes commonly analyzed using ICP-MS analysis.

As demonstrated from the data in Table 3, not only was the LOD significantly lower, but more importantly, the blank equivalent concentration (BEC) was almost 60 times lower. This reduction contributes to the accurate determination of phosphorus at ultra-trace levels in photovoltaic grade silicon.

The process of interference removal using oxygen is shown in Figure 2 using phosphorus as example. In short, whereas the analyte of interest reacts with oxygen and forms a molecular ion with a new mass-to-charge ratio (referred to as a mass-shift reaction), the previously isobaric (i.e., having the same nominal mass) interference does not react in a similar way and can therefore be eliminated in the third quadrupole of the system. The first quadrupole provides an additional mass filtration before the collision/reaction cell so that unwanted side reactions with other components present in the ion beam are effectively suppressed.

In addition, to further evaluate the accuracy and precision of the analysis, a spike recovery test was performed (details are given in

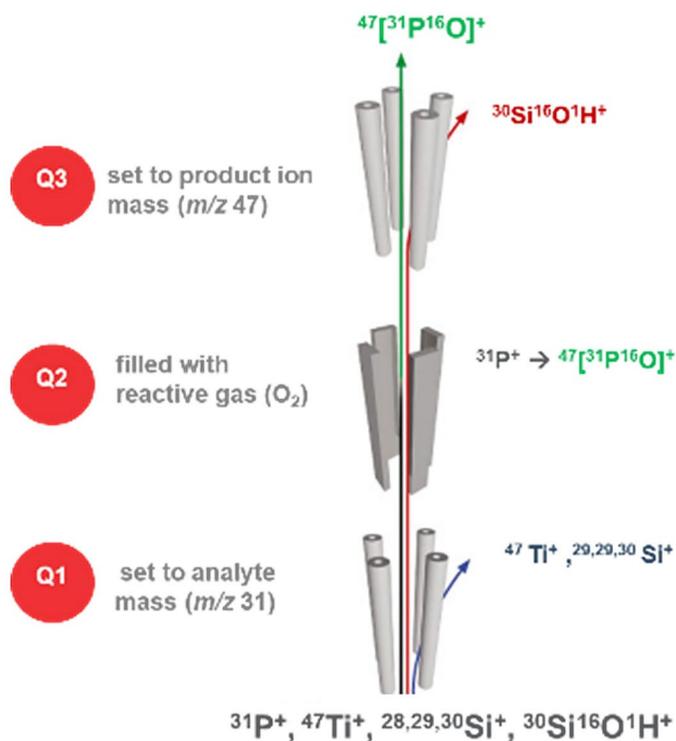


Figure 2. Schematic showing the use of TQ-O₂ mode and a mass shift reaction for interference free detection of phosphorus

Table 4). In general, the spike recovery was found to be between 85% and 108% and achieved reliable results. Although the concentration of silicon was not high in the testing solution, the 10 µg ·L⁻¹ spike recovery results showed the advantage of TQ-O₂ mode compare with He KED.

Evaluation of silicon matrix suppression

As discussed previously, silicon was lost during the reaction with HF (this is expected), and other sample preparation techniques may yield different silicon concentrations. Therefore, it is still necessary to obtain accurate results for the bulk analysis of silicon. While it is common to further dilute this sample type for ICP-MS analysis to improve analysis quality, to mitigate issues such as the internal standard recovery (due to sample matrix suppression) and damaging the detector and sample introduction parts (due to the high concentrations), AGD enables direct analysis. This further dilution process causes extra sample preparation operation and analysis time, thus the trace element and bulk analysis together on the same analysis method improves productivity. An iCAP MTX ICP-MS with AGD Level 5 was evaluated to obtain the fully quantitative result of 5,000 mg·L⁻¹ silicon.

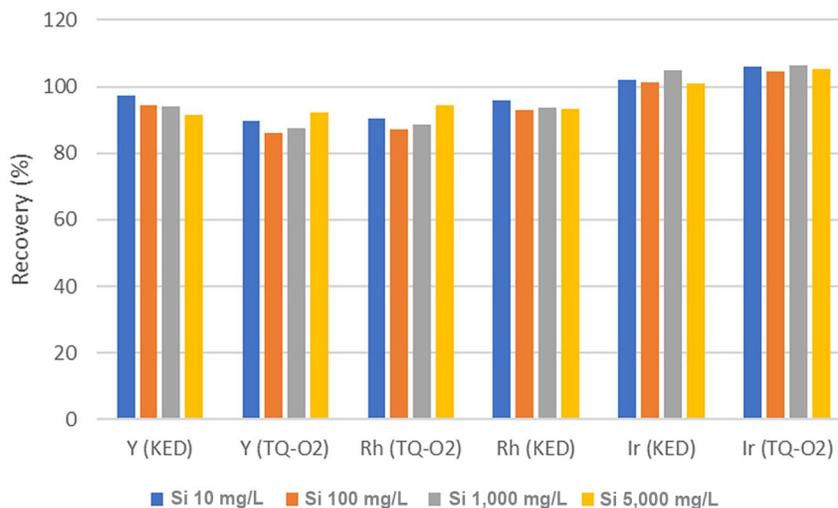


Figure 3. Internal recovery results with four different concentrations of silicon

Figure 3 shows the results of the internal standard response with four different concentrations of silicon. As can be seen from the data, the response of the internal standard remains stable with recoveries between 87% to 106%, which demonstrates the excellent stability of the iCAP MSX ICP-MS for such sample types. This further complements the high data quality obtained from the spike recovery testing highlighted previously. Table 5 summarizes the $10 \mu\text{g}\cdot\text{L}^{-1}$ spike recovery test with four different concentrations of silicon matrix solution. As demonstrated previously for phosphorous, silicon-based polyatomic interferences were effectively removed. Therefore, excellent recovery within 86% to 118% was achieved at a spiked concentration of $10 \mu\text{g}\cdot\text{L}^{-1}$. The method developed for this study was validated for handling samples containing silicon concentrations of up to $5,000 \text{ mg}\cdot\text{L}^{-1}$ in solution.

Evaluation of long-term robustness

To simulate high-throughput analysis, a larger batch containing the sample solutions previously analyzed were scheduled for analysis.

Following calibration of the instrument, several blocks containing samples of both the digested wafer sample as well as the prepared solutions with varying content of silicon were scheduled for analysis, together with the required QC checks. The total number of analyses was 197 (including 120 silicon matrix samples, 15 calibrants, and 24 QC checks), requiring a total analysis time of approximately 9 hours (Figure 4).

Figure 5 shows the recovery for all analytes analyzed as part of the continuing calibration verification check (CCV, n=6) for all

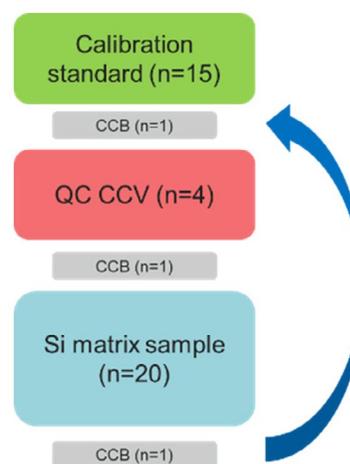


Figure 4. Schematic overview of the batch analyzed for testing the long-term performance of the proposed method. Six blocks, containing 20 silicon matrix samples each, were analyzed.

42 elements. The data indicates excellent recovery (within 88% to 124%). The iCAP MTX ICP-MS therefore allows for robust and reliable long-term analysis even for challenging samples containing high amounts of silicon as matrix.

The response of the internal standards was again observed to be consistent regardless of the matrix composition and well within the commonly accepted range between 80% and 120% of the response in the initial blank measurement for the calibration curve. All internal standards had predictable recovery (within approximately 87% to 110%) over the entire runtime of the batch, demonstrating robust analytical performance over 9 hours.

Table 4. Summary of the silicon wafer analysis and spike recovery results with 20-fold dilution samples

Analyte	MDL ($\mu\text{g}\cdot\text{L}^{-1}$)	Measured ($\mu\text{g}\cdot\text{L}^{-1}$)	Results ($\mu\text{g}\cdot\text{kg}^{-1}$)	Spike recovery (%)
^7Li	0.26	0.004	4.9	90.3
^9Be	<0.02	0.01	8.4	92.7
^{11}B	3.4	0.06	87	89.7
^{23}Na	24	2.1	2,891	95.8
^{24}Mg	7.2	<MDL	N.D.	88.9
^{27}Al	2	0.58	796.5	102.7
^{28}Si (He KED)	670	2,408	3,321,529	107.0
^{28}Si (TQ- O_2)	18	2,036	2,808,729	97.6
^{31}P (TQ- O_2)	2.4	<MDL	N.D.	88.6
^{31}P (He KED)	258	0.17	229	40.8
^{32}S	28	<MDL	N.D.	103.6
^{39}K	5	0.23	312.2	93.8
^{44}Ca	34	<MDL	N.D.	94.6
^{48}Ti	0.18	0.01	8	87.5
^{51}V	0.04	0.001	1.3	85.5
^{52}Cr	1.34	<MDL	N.D.	86.1
^{55}Mn	0.14	<MDL	N.D.	105.5
^{56}Fe	0.72	0.02	26.8	104.5
^{59}Co	0.18	0.002	3.4	104.1
^{60}Ni	0.8	0.02	29.9	100.8
^{63}Cu	0.54	0.02	26.6	107.2
^{66}Zn	0.48	0.02	24.4	89.1
^{71}Ga	0.06	7.1	9,789	91.5
^{72}Ge	0.22	617,603	851,866,157	98.0
^{75}As	0.42	0.98	1,354	109.3
^{78}Se (He KED)	11.82	< MDL	N.D.	93.0
^{80}Se (TQ- O_2)	0.7	0.01	13.8	106.6
^{85}Rb	0.04	< MDL	N.D.	102.5
^{88}Sr	0.06	0.02	30.3	98.0
^{90}Zr	0.66	0.07	100.7	97.3
^{93}Nb	<0.02	0.02	21.5	106.5
^{95}Mo	0.06	0.02	28.6	111.8
^{107}Ag	0.12	0.003	4.2	99.7
^{111}Cd	0.06	0.008	11.3	103.9
^{115}In	<0.02	0.001	1	106.1
^{118}Sn	0.06	< MDL	N.D.	107.2
^{121}Sb	0.1	< MDL	N.D.	108.2
^{137}Ba	0.02	0.001	1.1	107.8
^{181}Ta	<0.02	0.002	3.2	95.7
^{182}W	0.02	0.03	36.5	100.2
^{185}Re	<0.02	0.001	1.6	99.3
^{202}Hg	0.2	0.005	0.1	103.6
^{205}Tl	0.02	0.001	1.6	106.6
^{208}Pb	0.02	0.001	0.8	105.8
^{209}Bi	0.02	0.001	1.6	103.3
^{238}U	<0.02	0.001	1.3	104.0

Table 5. Summary of the 10 $\mu\text{g}\cdot\text{L}^{-1}$ spike recovery with four different concentrations of silicon

Analyte	Si 10 ($\text{mg}\cdot\text{L}^{-1}$)	Si 100 ($\text{mg}\cdot\text{L}^{-1}$)	Si 1,000 ($\text{mg}\cdot\text{L}^{-1}$)	Si 5,000 ($\text{mg}\cdot\text{L}^{-1}$)
^7Li	106	110	109	108
^9Be	103	105	96	83
^{11}B	98	99	91	80
^{23}Na	94	84	87	94
^{24}Mg	94	94	101	100
^{27}Al	97	102	109	92
^{31}P (TQ- O_2)	92	89	94	119
^{31}P (He KED)	36	43	45	100
^{32}S	87	95	87	103
^{39}K	86	87	96	93
^{44}Ca	88	101	89	118
^{48}Ti	89	89	97	105
^{51}V	88	88	89	89
^{52}Cr	90	89	93	96
^{55}Mn	93	94	100	106
^{56}Fe	99	103	101	85
^{59}Co	94	96	97	101
^{60}Ni	100	101	98	97
^{63}Cu	95	96	97	104
^{66}Zn	99	102	100	105
^{71}Ga	96	99	95	94
^{72}Ge (TQ- O_2)	102	95	94	110
^{72}Ge (He HED)	38	59	56	110
^{75}As	98	97	94	92
^{78}Se (He KED)	105	113	81	61
^{80}Se (TQ- O_2)	100	98	87	92
^{85}Rb	97	99	97	99
^{88}Sr	99	103	98	98
^{90}Zr	99	100	96	104
^{93}Nb	112	110	106	108
^{95}Mo	92	92	99	102
^{107}Ag	99	101	96	96
^{111}Cd	103	105	99	89
^{115}In	101	110	102	108
^{118}Sn	105	106	104	110
^{121}Sb	106	107	107	112
^{137}Ba	104	111	104	108
^{181}Ta	95	97	93	102
^{182}W	98	100	97	105
^{185}Re	99	103	95	100
^{202}Hg	108	101	101	109
^{205}Tl	101	106	102	111
^{208}Pb	100	106	102	115
^{209}Bi	99	106	100	109
^{238}U	103	106	104	114

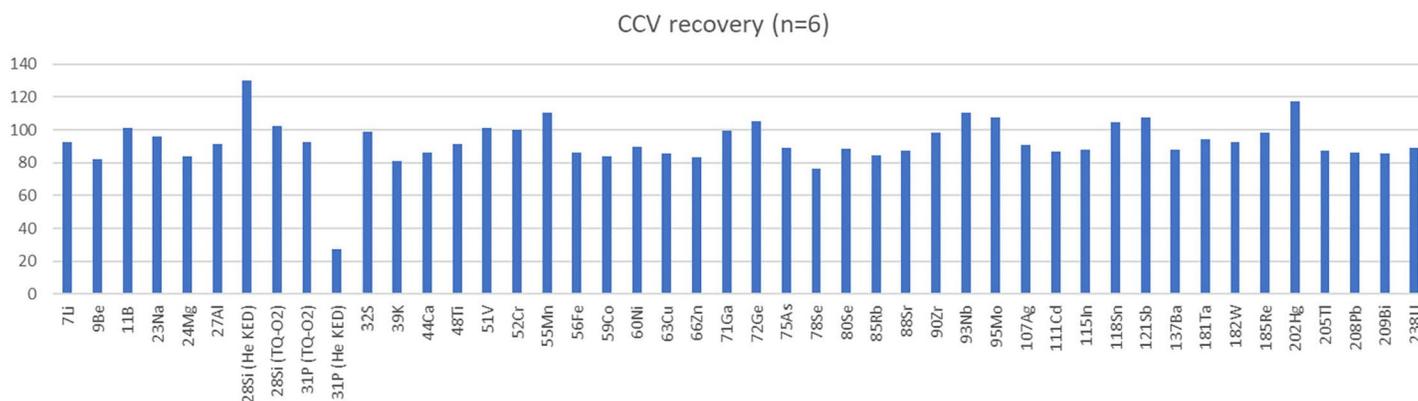


Figure 5. QC calibration verification results for all 42 elements measured

Conclusions

The iCAP MTX ICP-MS was employed to analyze 42 elements in a digested silicon wafer, similar to the materials used for photovoltaic grade silicon. In addition, several solutions with variable amounts of silicon were analyzed to further demonstrate reliable performance and accuracy of results. Among the analytes, several critical interferences can cause unexpected bias, which were investigated closely for effective and complete removal by means of selective collision / reaction cell reactions with oxygen. This analytical method was rigorously tested, and the results obtained clearly demonstrated the following analytical advantages:

- The combination of He KED and TQ-O₂ mode allows for highly sensitive analysis required for the accurate determination of the entire mass range (lithium to uranium), while effectively suppressing typical interferences.
- The developed method provides the required detection limits and a linear response for all analytes, which cover a dynamic analysis intensity range of silicon (10 to 5,000,000 µg·L⁻¹), this is enabled through the use of AGD.

- The iSC-65 Autosampler equipped with the Step Ahead feature allowed for a reduction of the total analysis time, and in combination with intelligent matrix handling, the analysis of the samples (photovoltaic, silicon wafer) could be carried out with improved productivity.
- The use of TQ-O₂ mode allowed for the removal of interferences of any kind, especially on key analytes such as phosphorus, and challenging elements such as arsenic or selenium in the high matrix silicon samples. This is proven by the excellent spike recovery results obtained for the analysis of silicon matrix samples.
- The total analysis time was 3 min 33 s per sample (including uptake and wash time) for 42 elements (covering bulk, major, and trace level analytes). The analysis time could be further shortened when using a discrete sampling valve.
- Robust and stable analytical performance was demonstrated over 9 hours, allowing the analysis of 197 samples with excellent CCV results.

Reference

1. Thermo Fisher Scientific, Product Spotlight 44485: Thermo Scientific iCAP Qnova Series ICP-MS PLUS Torch for Improved ICP-MS Analysis of Challenging Samples.

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