Application Note

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Analysis of High Purity Titanium Using an Agilent 9500 ICP-QQQ

Ensuring purity and performance of titanium materials used in high-tech industries

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Introduction

Importance of high-purity titanium and the role of ICP-QQQ

High-purity titanium (Ti) is a critical material in high-tech industries such as semiconductor manufacturing and aerospace engineering. In semiconductor applications, Ti is widely used as a sputtering target for thin-film deposition, where even trace levels of impurities can negatively affect film uniformity, electrical properties, and device reliability. In aerospace applications, Ti is valued for its high strength-to-weight ratio and corrosion resistance, making it an ideal material for engine components and structural parts. However, any impurities can compromise the mechanical integrity and diminish the long-term performance of components, especially when operating under extreme conditions.

Ensuring the reliability and functionality of Ti in these demanding environments requires precise quantification of trace impurities in the raw material. Triple quadrupole Inductively Coupled Plasma Mass Spectrometry (ICP-QQQ) provides robust suppression of spectral interferences and ultra-low detection limits (DLs), enabling accurate determination of impurities at or below the 1 mg/kg (ppm) level in the solid. This level of sensitivity is particularly important for quality control (QC) of high-purity Ti, where conventional techniques such as XRF, ICP-OES, and GD-MS lack the sensitivity and DLs.

For unrivalled interference reduction using reactive cell gases, Agilent ICP-QQQ instruments include two unit mass (1 u) quadrupole mass filters, Q1 and Q2.[1] Q1 is positioned before the collision/reaction cell (CRC) to select which ions enter the cell for reaction with the cell gas. Q2 filters ions that exit the cell before they pass to the detector. This tandem mass spectrometer (MS/MS) operation allows for unmatched control of reaction chemistry in the CRC, making the technique useful for handling intense spectral interferences, including polyatomic, doubly charged ion (M^{++}), isobaric, and peak overlap interferences.

Using the newly developed Agilent 9500 Triple Quadrupole ICP-MS featuring a Dual-Cell System (DCS) CRC and equipped with an optional m-lens, analysts can confidently analyze Ti matrix samples and verify material purity over extended measurement periods. The m-lens is designed with an optimized geometry that minimizes background signals from easily ionized elements that can deposit on interface components during long runs. This feature enables the instrument to maintain high-power, matrix-tolerant plasma conditions while achieving ultralow background equivalent concentrations (BECs), which is critical for ppt-level impurity analysis. Operating the DCS with a suitable cell gas effectively controls spectral interferences from Ti-based species (Ti^{2+} and TiO^+) that impact elements like sodium (Na), magnesium (Mg), copper (Cu), and zinc (Zn). Together with the m-lens, this approach ensures accurate measurement of trace elemental impurities, enabling compliance within stringent industry standards. The effectiveness of the m-lens in minimizing background signals and enabling ppt-level impurity analysis under robust plasma conditions has also been demonstrated in high-matrix applications using ICP-QQQ.[2, 3] In addition to interference control, the 9500 ICP-QQQ system's robustness and reproducibility make it well suited for routine analysis in both research and production settings. This capability supports the continued advancement of high-tech applications that rely on ultra-clean titanium.

In this study, a 200 ppm Ti solution prepared from a high-purity Ti powder was used to represent a typical sample encountered in advanced material analysis. The performance of the 9500 ICP-QQQ for quantifying trace impurities, including Na, Mg, Cu, and Zn, in a high Ti matrix was assessed through spike-recovery experiments and long-term stability tests.

Experimental

Instrumentation

The Agilent 9500 ICP-QQQ fitted with the optional m-lens and the Agilent I-AS autosampler were fully controlled using Agilent OpenLab ICP-MS software version 1.1. Designed for ultra-trace metal analysis, the sample introduction system comprised a MicroFlow PFA nebulizer with I-AS probe (operated in self-aspiration mode), a temperature-controlled quartz spray chamber, and a quartz torch with a 2.5 mm inner diameter (id) injector. A platinum-tipped sampler cone with copper base and a platinum-tipped skimmer cone with nickel base for m-lens were used.

To simplify the tuning process, only two cell conditions were applied: H_2 and NH_3 mixed with H_2. Introduction of H_2 and NH_3 gases into the cell enables the removal of argon (Ar)- and Ti-based interferences through selective ion–molecule reactions. The makeup gas was adjusted to achieve a CeO/Ce ratio of approximately 0.8% in no gas mode, and other plasma and lens parameters were optimized for low background levels and high stability (Table 1).

Table 1. Agilent 9500 ICP-QQQ operating parameters.

Parameter	H_2	$NH_3 + H_2$
Scan Mode	MS/MS	
RF Power (W)	1600	
Nebulizer Gas (L/min)	0.7	
Makeup Gas (L/min)	0.5	
Extract 1 (V)	0	
Extract 2 (V)	−40	
Deflect (V)	1	−15
H_2 Gas Flow (mL/min)	5	2
NH_3 Gas Flow* (mL/min)	0	8 (80%)
Cell Bias (V)	−10	−20
KED (V)	−2	−7

**10% NH_3 balanced with 90% He.*

Reagents

The sample comprised high-purity titanium powder (99.99%, < 100 mesh), bought from FUJIFILM Wako Pure Chemical, Japan. Tamapure-AA-10 grade nitric acid (HNO_3), hydrochloric acid (HCl), and hydrofluoric acid (HF) from Tama Chemicals (Japan) were used for sample digestion and solution preparation.

Calibration standards and spike solutions were prepared using the following multi-element standards: XSTC-7, XSTC-8, and XSTC-331 (SPEX CertiPrep LLC, USA). Single-element standards for Mg, iron (Fe), and yttrium (Y) (Kanto Chemical, Japan) were also used. Indium (In) (Kanto Chemical, Japan) was used as the internal standard (ISTD).

Sample preparation

The Ti sample was digested in accordance with ASTM E2371-21a guidelines. Steps 2 to 5 were performed in a fume hood.

1. Ti powder (1 g) was weighed into a 100 mL PFA bottle.
2. 40 mL of 15% HCl was added.
3. All analytes except Mg and Fe were spiked at 1 µg each; 15 µg Mg and 25 µg Fe were added.
4. 2 mL of 38% HF and 3 mL of 30% HNO_3 were added, and the mixture was heated on a hot plate at 120°C for 15 minutes.
5. The solution was cooled to room temperature for 15 minutes.
6. The solution was diluted to 100 g with ultrapure water to prepare a 1% Ti digestion solution.
7. The 1% Ti solution was diluted 50-fold with 1% HNO_3 to prepare a 200 ppm Ti solution, and In was added to achieve a 2 µg/kg (ppb) concentration.
8. The 200 ppm Ti solution was introduced into the 9500 ICP-QQQ.

A procedural blank (no Ti matrix) was also prepared.

Calibration

The 9500 ICP-QQQ was calibrated using the method of standard addition (MSA), as is typical for the analysis of high-purity samples. Calibration curves were generated by spiking the procedural blank (no Ti matrix) and the Ti digestion solution (200 ppm Ti solution). For the procedural blank, all elements were spiked at concentrations of 0, 50, and 100 ng/kg (ppt). The blank was measured by performing five consecutive measurements of the same blank solution (*n* = 5) to evaluate the variability of the measurement. For the Ti digestion solution, all elements except Mg and Fe were spiked at 0, 200, and 400 ppt, while Mg and Fe were spiked at 0, 3, and 6 ppb.

Samples

Three types of Ti samples were prepared for this study:

- **Ti matrix sample**: A 200 ppm Ti solution without any spiked elements.
- **Sample A**: A 200 ppm Ti solution spiked with all target analytes except Mg and Fe. The analytes were spiked during Step 3 of the sample preparation procedure.
- **Sample B**: A 200 ppm Ti solution spiked exclusively with Mg and Fe. Mg and Fe were spiked in Step 3 of the sample preparation procedure.

The calibration curves were prepared by spiking the 200 ppm Ti digestion solution with standard solutions. Samples A and B were spiked with analytes before digestion. This approach allows spike recovery rates to be determined for Samples A and B, providing a clear assessment of quantification accuracy and any potential element losses during digestion. Samples A and B were measured ten times to evaluate spike recovery rates and reproducibility. A QC sample was prepared by spiking the Ti matrix sample at 200 ppt with the standard solution. This QC sample was measured once after every five measurements of Samples A and B.

Results and discussion

Titanium-based interferences

Ti has five stable isotopes, all with different natural abundances (%): ^{46}Ti (8.25%), ^{47}Ti (7.44%), ^{48}Ti (73.72%), ^{49}Ti (5.41%), and ^{50}Ti (5.81%). In a 200 ppm Ti sample, several elements will be affected by Ti-based interferences, as shown in Table 2.

Table 2. Examples of ion interferences in the 200 ppm Ti matrix samples.

Isotope	Ion Interference
^{23}Na	$^{46}Ti^{2+}$
^{24}Mg, ^{25}Mg	$^{48}Ti^{2+}$, $^{50}Ti^{2+}$
^{51}V	$^{48}TiHHH^{+}$, $^{49}TiHH^{+}$, $^{50}TiH^{+}$
^{63}Cu, ^{65}Cu	$^{46}Ti^{17}O^{+}$, $^{47}Ti^{16}O^{+}$, $^{47}Ti^{18}O^{+}$, $^{48}Ti^{17}O^{+}$, $^{49}Ti^{16}O^{+}$
^{64}Zn, ^{66}Zn, ^{68}Zn	$^{46}Ti^{18}O^{+}$, $^{47}Ti^{17}O^{+}$, $^{48}Ti^{16}O^{+}$, $^{48}Ti^{18}O^{+}$, $^{49}Ti^{17}O^{+}$, $^{50}Ti^{16}O^{+}$, $^{50}Ti^{18}O^{+}$

To reduce background concentrations arising from various TiO interferences on Cu and Zn, the 9500 ICP-QQQ was operated in reactive cell mode using $NH_3 + H_2$ gas. The same controlled reaction chemistry conditions were also used to remove the Ti^{2+} interferences on Na and Mg and Ti-hydride overlaps on vanadium (V).

A relatively high concentration of Mg (8 ppm) was detected in the Ti powder used in this study (Table 3). To verify that spectral interferences from $^{48}Ti^{2+}$ on ^{24}Mg and $^{50}Ti^{2+}$ on ^{25}Mg had been effectively removed, three Mg isotopes were measured. Because ^{26}Mg is free from Ti^{2+} interference, it served as a qualifier ion for data confirmation. The BECs of ^{24}Mg, ^{25}Mg, and ^{26}Mg in the 200 ppm Ti matrix samples were determined to be 1.60, 1.64, and 1.60 ppb, respectively. The close agreement among the three isotopes confirms that Mg was inherently present in the Ti powder and that Ti^{2+} interferences were successfully eliminated by the 9500 ICP-QQQ method.

As shown in the two standard addition calibration curves for Cu in Figure 1, the mass-shift method reduced the BEC by 20 ppt (from 125 to 104 ppt), compared to the on-mass measurement. This suggests that Cu^+ reacts with the cell gas more readily than TiO^+, enabling measurement as $Cu(NH_3)_2^+$ at *m/z* 97. The two calibration curves for Zn in Figure 1 show that ^{68}Zn yielded a sufficiently low BEC (14 ppt) even with on-mass measurement, since TiO at mass 68 is relatively less abundant than at masses 64 and 66.

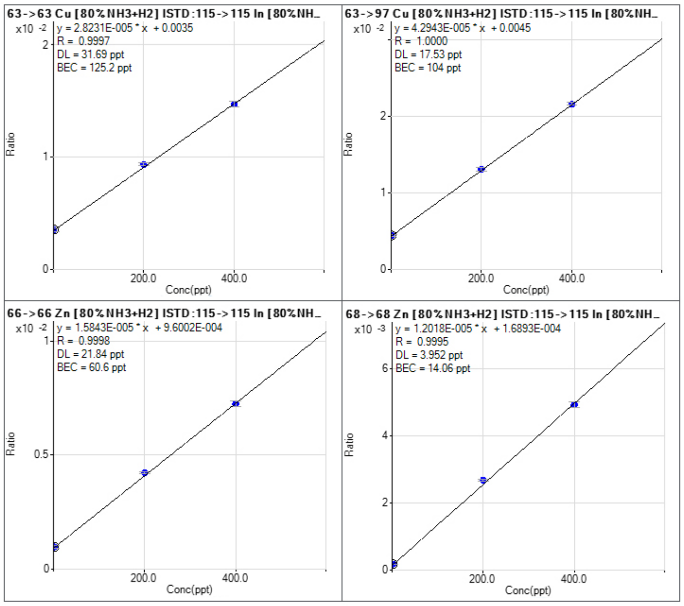

Figure 1. Calibration plots for Cu and Zn. Top: The BECs of Cu obtained from on-mass and mass-shift measurements were 125 and 104 ppt, respectively. Bottom: The BECs of ^{66}Zn and ^{68}Zn were 60.6 and 14.1 ppt, respectively.

Quantification of impurity metals in high-purity titanium

Table 3 presents the quantification results of 25 elements in the 200 ppm high-purity Ti solution. By subtracting the BECs of the procedural blank from the Ti matrix BECs, the total metal concentration was calculated as 31 ppm, based on the summed elemental contributions. The combined concentration of major metal impurities was verified to be below 0.01%, consistent with the material's specified purity (> 99.99%).

For simplicity, all elements were analyzed using either H_2 or $NH_3 + H_2$ mode. During data acquisition, the two measurement modes were switched automatically, enabling rapid, automated analysis. If higher sensitivity is required for the analysis of purer Ti samples, such as 99.999%, additional optimization of the cell gas flow rates may be needed. For example, reducing the NH_3 flow rate may improve sensitivity and lower DLs for some analytes; the conditions used in this study were optimized to reduce TiO interferences on Cu and Zn.

Spike recoveries in the digested titanium sample

To assess the method's accuracy, Ti powder was spiked before digestion. The spike amount of each analyte was 1 µg per 1 g (1 ppm) of the original Ti sample, except for Mg and Fe, which, due to their concentrations, were spiked at 15 µg (15 ppm) and 25 µg (25 ppm), respectively. Since a 200 ppm Ti digested solution diluted 5000-fold was introduced to the 9500 ICP-QQQ, the actual quantified concentrations were 200 ppt for all elements except Mg (3000 ppt) and Fe (5000 ppt). Table 4 shows the spike recovery results. All elements achieved recovery rates within ±10%, with relative standard deviation (RSD) values mostly between 1–3% and all less than 5%.

Long-term stability

Figure 2 shows the ISTD stability during approximately three hours of measurement of the 200 ppm Ti solution. The In signal consistently remained within the 90–120% range.

Figure 3 shows the recovery rates of the QC sample, which was measured six times during the analytical sequence. Most elements were recovered within ±10%, and all elements were recovered within ±20%.

The results confirm the stability, robustness, and matrix tolerance of the 9500 ICP-QQQ for the continual measurement of Ti matrix samples over several hours.

Table 3. Calibration curves were obtained by MSA for both the Ti digestion solution (200 ppm Ti matrix) and the procedural blank (no Ti matrix). For the blank samples (*n* = 5), the DL was defined as three times the standard deviation. The BEC was calculated by dividing the average signal intensity of blank samples by the slope of the calibration curve. The impurity metal concentration in the original Ti sample was calculated by subtracting the BECs of the procedural blank from the corresponding Ti matrix BECs and converting the result to the concentration in the original Ti powder.

Analyte	Q1	Q2	Tune	No Ti Matrix		Ti Matrix		Impurity Metal Conc in Original Ti Powder (ppb)
				DL (ppt)	BEC (ppt)	DL (ppt)	BEC (ppt)	
B	11	11	H_2	1.92	2.83	6.32	12.7	49.4
Na	23	23	$NH_3 + H_2$	2.03	3.25	7.52	91.4	441
Mg	24	24	$NH_3 + H_2$	0.66	< DL	42.8	1600	8000
Al	27	27	$NH_3 + H_2$	0.52	< DL	5.14	57.1	286
K	39	39	$NH_3 + H_2$	2.30	4.33	2.34	6.26	9.7
Ca	40	40	H_2	0.62	1.52	1.36	48.9	237
V	51	51	$NH_3 + H_2$	0.23	< DL	2.39	3.44	17.2
Cr	52	52	$NH_3 + H_2$	0.65	1.33	7.64	100	493
Mn	55	55	$NH_3 + H_2$	0.25	0.29	3.07	38.2	190
Fe	56	56	H_2	0.69	2.43	100	4050	20200
Co	59	59	$NH_3 + H_2$	ND	ND	0.67	0.84	4.2
Ni	60	60	$NH_3 + H_2$	0.84	< DL	18.6	90	450
Cu	63	97	$NH_3 + H_2$	2.35	2.4	8.22	110	538
Zn	68	68	$NH_3 + H_2$	1.48	5	8.94	18.3	66.5
Y	89	89	H_2	0.03	< DL	0.09	< DL	< DL
Zr	90	90	H_2	0.09	< DL	0.22	1.24	6.2
Nb	93	93	H_2	0.06	< DL	0.16	0.57	2.8
Mo	95	95	H_2	ND	ND	2.30	3.76	18.8
Ru	101	101	H_2	ND	ND	0.17	< DL	< DL
Pd	105	105	$NH_3 + H_2$	0.14	< DL	0.11	< DL	< DL
Sn	118	118	H_2	0.24	< DL	0.80	3.37	16.9
Hf	178	178	H_2	ND	ND	0.09	< DL	< DL
Ta	181	181	H_2	0.02	< DL	0.27	1.19	6.0
W	182	182	H_2	0.06	< DL	0.14	0.52	2.6
Bi	209	209	H_2	0.05	< DL	0.55	0.90	4.5

ND: not detected.

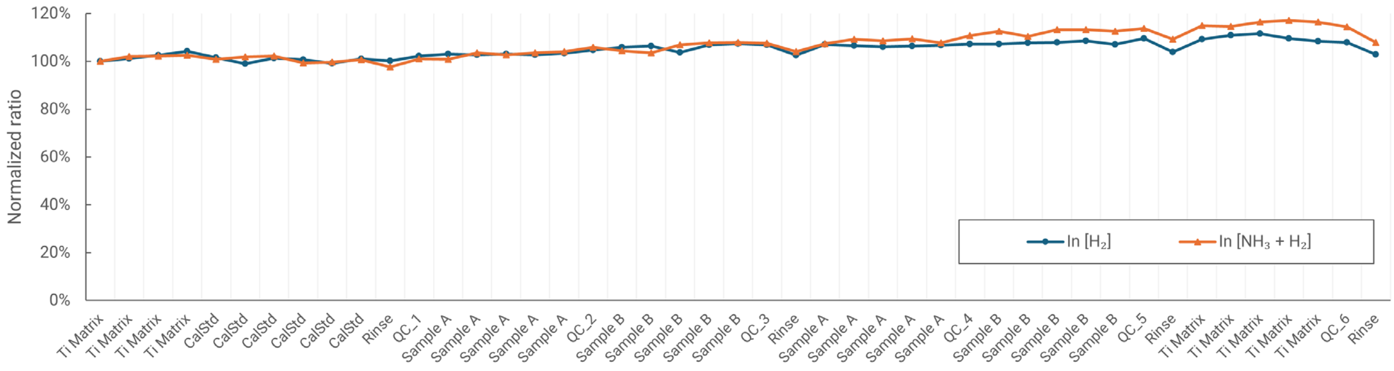

Figure 2. The normalized ratio of the In ISTD signal to the calibration blank sample of the Ti digestion solution. A total of 42 Ti matrix samples were measured over approximately three hours.

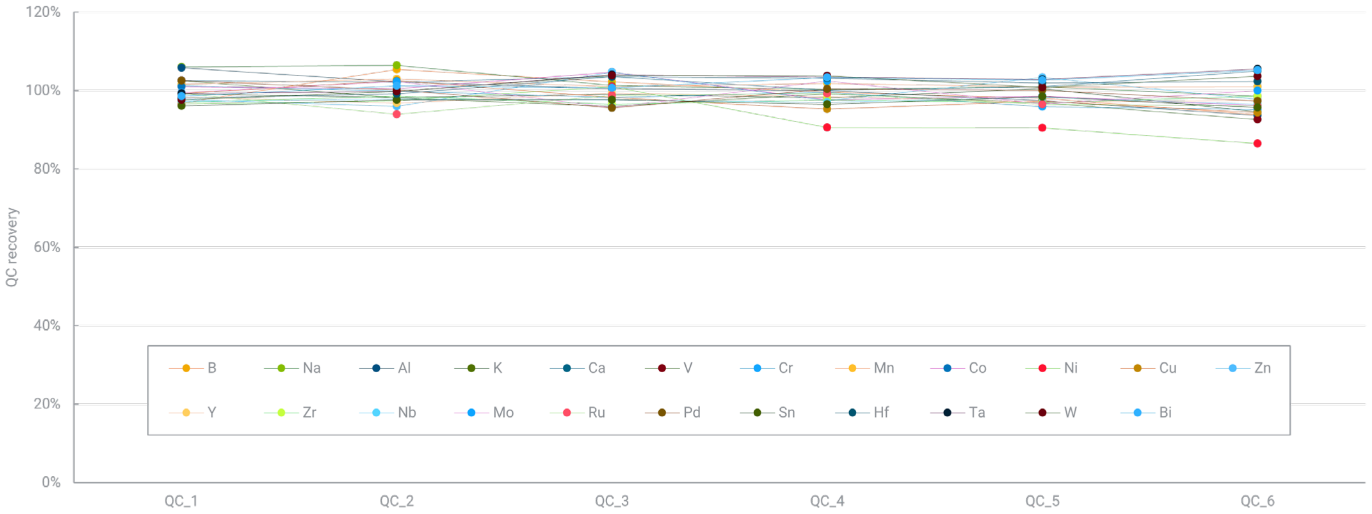

Figure 3. The recovery rates of the QC sample spiked at 200 ppt. The QC sample was introduced every five measurements of the spiked Ti digestion solution and was quantified six times. Recoveries were not calculated for Mg and Fe due to their high BECs.

Table 4. Spike recoveries of Samples A and B. The RSD was calculated from the variation in quantitative measurements of ten replicate analyses of the spiked samples.

Analyte	Spiked Concentration in 200 ppm Ti (ppt)	Recovery Rate (%)	RSD (%)
B	200	102	3.6
Na	200	95	2.0
Mg	3000	103	2.5
Al	200	100	2.1
K	200	99	0.9
Ca	200	99	2.1
V	200	96	1.9
Cr	200	92	2.2
Mn	200	103	1.4
Fe	5000	103	1.7
Co	200	98	2.0
Ni	200	105	1.5
Cu	200	97	2.9
Zn	200	95	4.0
Y	200	98	1.1
Zr	200	97	1.3
Nb	200	97	1.0
Mo	200	98	1.9
Ru	200	97	1.8
Pd	200	99	1.9
Sn	200	98	1.8
Hf	200	100	2.2
Ta	200	101	1.3
W	200	101	1.8
Bi	200	102	2.1

Conclusion

This study demonstrated the effectiveness of the Agilent 9500 ICP-QQQ with m-lens for the ultra-trace analysis of impurity metals in high-purity titanium. By applying optimized reaction gas conditions and mass-shift techniques, spectral interferences from Ti-based species such as Ti^{2+} and TiO^+ were successfully mitigated, enabling accurate quantification of elements like Mg, Cu, and Zn.

The method achieved limits of quantification in the sub-ppm range for the undiluted Ti digestion solution and maintained stable ISTD signals over extended measurement periods. Spike recovery tests confirmed high accuracy and reproducibility, with most elements showing recoveries within ±10% and RSDs below 3%.

These results confirm that the 9500 ICP-QQQ is a robust and reliable tool for quality control of high-purity Ti, supporting its critical applications in semiconductor and aerospace industries where impurity levels must be strictly controlled.

References

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2. Sugiyama, N. Analysis of Ultratrace Impurities in High Purity Copper using the Agilent 8900 ICP-QQQ, Agilent publication, 5994-0383EN
3. Ying, Y. Analysis of Ultratrace Impurities in High Silicon Matrix Samples by ICP-QQQ, Agilent publication, 5994-2890EN

Products used in this application

Agilent products

Product Type	Description	Part Number
Sample Introduction System	9500 ICP-MS quartz torch, 2.5 mm id for aqueous samples	M5150-67011
	9500 ICP-MS quartz connector tube, straight	M5150-67014
	9500 ICP-MS quartz spray chamber with straight exit port	M5150-67017
	MicroFlow PFA nebulizer with I-AS probe, self-aspirates at 200 µL/min	G3139-65102
Interface	ICP-MS sampler cone for 9500 ICP-MS, Pt tip with Cu base	M5150-67002
	Skimmer cone, Pt tip with Ni base for m-lens	G8400-67073
	Extraction-Omega lens assembly, m-lens, brass base	M5150-67023
Tubing Kits	Easy-fit peristaltic pump tubing, beige thermoplastic, yellow/blue, 1.52 mm id for drain	5005-0022
Bottle Kits	Waste container kit, includes a 10 L waste can, S60 StaySafe cap, fittings, and acid vapor filter	5005-0437

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