Application Brief

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Ultratrace Impurity Analysis of Ultrapure Water with Low Boron Background by ICP-MS/MS

Achieving extremely low detection limits with the Agilent 8900 ICP-QQQ

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Critical contamination control of process chemicals

The miniaturization of semiconductors is essential for achieving high integration of integrated circuits (ICs) on a chip. However, as devices become smaller, the effects of impurities become more problematic. Elemental impurities can lead to yield and performance issues, so even higher purity materials are needed for the fabrication of reliable and robust semiconductors. Ultrapure water (UPW) is one of the most critical semicon process chemicals as it is in direct contact with the wafer surface at many stages of manufacturing. Impurities must therefore be monitored in UPW at the ppt (1 x 10^{-9} g/kg) or sub-ppt level.[1, 2]

The Agilent 8900 Triple Quadrupole ICP-MS (ICP-QQQ) with s-lens and Octopole Reaction System (ORS^4) provides the sensitivity and interference removal capabilities for the ultratrace analysis of elemental impurities in UPW. The 8900 achieves Limits of Detection (LODs) and Background Equivalent Concentrations (BECs) at the ppq (1 x 10^{-12} g/kg) level for many elements in UPW using MS/MS methodology. However, measuring boron (B) below 1 ppt is challenging by ICP-MS due to its high abundance in the environment, supressed ionization efficiency, and memory effects. B backgrounds are highly dependent on water quality.

In this study, water produced by a Puric ω II UPW production system equipped with a filter for B removal (Organo, Japan) was analyzed using an 8900 Semiconductor Configuration ICP-QQQ. The instrument was fitted with a quartz nebulizer (Agilent part number G1820-65138) and Agilent I-AS autosampler.

Method and reagents

To optimize the LODs and BECs for all analytes, the 8900 ICP-QQQ was rinsed with Puric ω II UPW for more than 24 h, followed by a short rinse with 2% nitric acid (HNO_3). The UPW samples were either bottled or obtained directly from the fresh sampling port of the Puric ω II system (possible for Agilent ICP-MS only). Calibration standards were prepared using multi-element standards (SPEX) in 0.1% HNO_3.

Multi-element impurity analysis of UPW

The Agilent ICP-MS MassHunter software automatically calculated the LODs and BECs as 3-sigma of the standard deviation of 10 replicates of the bottled Puric ω II UPW blank (Table 1). The LODs and BECs of most elements—including B—ranged from 1 ppq to 0.63 ppt, confirming the exceptional sensitivity and interference removal capabilities of the ICP-MS/MS method. Low-level LODs and BECs were also achieved for silicon (Si) and phosphorus (P), which are difficult elements to ionize. Both elements are also prone to high backgrounds.

Table 1. LODs and BECs of analytes in UPW. Concentration units: ppt.

Q1/Q2, Analyte	LOD	BEC	Q1/Q2, Analyte	LOD	BEC
7>7 Li	0.002	<LOD	64>64 Zn	0.10	0.10
9>9 Be	0.005	<LOD	71>71 Ga	0.015	<LOD
11>11 B	0.12	0.63	75>91 As	0.031	<LOD
23>23 Na	0.042	<LOD	85>85 Rb	0.012	<LOD
24>24 Mg	0.011	0.012	88>88 Sr	0.005	<LOD
27>27 Al	0.018	<LOD	90>90 Zr	0.020	0.053
28>28 Si	2.5	63	95>95 Mo	0.011	<LOD
31>47 P	0.76	1.0	107>107 Ag	0.007	0.010
39>39 K	0.033	<LOD	114>114 Cd	0.014	<LOD
40>40 Ca	0.035	<LOD	118>118 Sn	0.020	0.022
48>64 Ti	0.007	<LOD	121>121 Sb	0.014	<LOD
51>67 V	0.021	<LOD	138>138 Ba	0.007	<LOD
52>52 Cr	0.025	0.028	182>182 W	0.014	0.014
55>55 Mn	0.010	<LOD	195>195 Pt	0.036	0.042
56>56 Fe	0.060	<LOD	197>197 Au	0.009	<LOD
59>59 Co	0.012	<LOD	205>205 Tl	0.006	<LOD
60>60 Ni	0.037	<LOD	208>208 Pb	0.018	<LOD
63>63 Cu	0.027	<LOD	238>238 U	0.001	<LOD

Comparing boron data from different UPW purification systems

The 8900 ICP-QQQ method was also used to acquire B data for UPW produced using an Organo Puric ω system (no boron filter). Comparing the LODs and BECs attained using both systems (Figure 1) shows an improvement of around 50% or better in both results when a boron filter was used. The filter of the Puric ω II reduced the LOD of B from 0.51 to 0.12 ppt and lowered the BEC from 1.2 to 0.63 ppt.

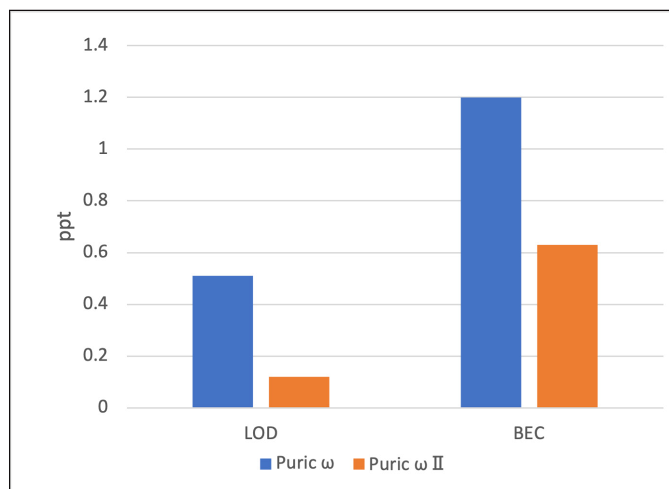

Figure 1. Boron LODs and BECs in UPW produced using two purification systems: no B filter (Puric ω) and with an effective B filter (Puric ω II).

Quality control of UPW by ICP-QQQ

The study demonstrates the high performance of the Agilent 8900 ICP-QQQ for quantifying boron and other critical ultratrace impurity elements in UPW. The data also shows that the level of boron can be reduced in UPW using the Puric ω II purification system fitted with an effective filter.

References

1. Measuring Inorganic Impurities in Semiconductor Manufacturing, Agilent publication, 5991-9495EN
2. Sakai, K., Shimamura, Y., Ultrapure Process Chemicals Analysis by ICP-QQQ with Hot Plasma Conditions, Agilent publication, 5994-4025EN

https://www.agilent.com/chem/8900icpqqq

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