The Analysis and Stability of High Purity Tetra MethylAmmonium Hyrdoxide (TMAH) with the Agilent 8900 QQQ-ICPMS

Introduction

Tetramethylammonium hydroxide (TMAH) is used extensively throughout the semiconductor industry as a silicon etchant, and as a photo resist developer and stripper. The purity of TMAH is important because of this usage in the wafer manufacturing process and the desire for smaller and smaller chip dimensions. As technology gets smaller, the need for lower detection limits on all materials used in the process becomes more critical. The ability to analyze TMAH at low dilutions with long periods of stability, while still maintaining excellent detection limits, is addressed by the Agilent 8900 QQQ-ICPMS.



Fig 1. The Agilent 8900 ICP-MS/MS in Wilmington, DE

Experimental

The Agilent 8900 QQQ-ICPMS, with single amu resolution of both quadroples in MS/MS mode, along with UHMI technology, was used to analyze 25% TMAH at a 5:1 dilution. The five-fold diluted (w/v) TMAH was analyzed over 18 hours straight under the UHMI conditions set by the Agilent MassHunter Start Up functions. The 8900 was equipped with a standard Agilent quartz sample introduction system which included a quartz double pass spray chamber, a quartz concentric nebulizer, and a standard quartz torch with 2.5 mm injector.

Instrument Condition			
Scan Mode	MS/MS		
RF Power (W)	Set by Software for UHMI-25		
Sample Depth (mm)	Set by Software for UHMI-25		
Spray Chamber (C°)	Set by Software for UHMI-25		
Carrier Gas Flow (L/min)	Set by Software for UHMI-25		
KED (V)	Default for Tune Condition		
Cell Gas Flow (ml/min)	Default for Tune Condition		

Results and Discussion

The stability solution was prepared by diluting JT Baker Semiconductor grade TMAH 5:1 (50g into 250ml, w/v). The diluted TMAH was spiked with 10 ppb of 24 different elements. The figure below shows the normalized response of the spiked 5x diluted TMAH over an 18 hour period. Notice that all 24 elements are well within ± 20% over 18 hours.



Fig 3. Stability Plot of 5:1 Dilution of 25% TMAH solution over 18 hours

The elements analyzed in this experiment are listed below. These elements are crucial in the semiconductor industry as they are either common contaminants in the fabrication process or poisons to the wafers themselves. Additional elements are also possible based on analytical need.

Be	Na	Mg	AI	К	Ca
Cr	Fe	Со	Ni	Cu	Zn
Ga	As	Se	Rb	Sr	Ag
Cd	Cs	Br	ТІ	Pb	U

Fig 4. Elements Analyzed in the 5:1 Dilution Test



Conclusions

The robustness of the 8900 and the ability to analyze heavy matrix samples does not mean the analyst needs to sacrifice detection limits. The calibration plots below show the Detection Limits (DL) and Background Equivalent Concentration (BEC) for a subset of the elements analyzed in this study. These elements in particular are critical in the semiconductor industry.













Figs 5-14. Calibration Plots for Selected Elements