



Measuring Ultratrace Levels of Metals in HF and H₂O₂ by ICP-MS

Role of HF and H₂O₂

Both hydrogen peroxide (H₂O₂) and hydrofluoric acid (HF) are widely used by the semiconductor industry. H₂O₂ is used in the formulation of the standard cleaning baths SC1 and SC2 to remove metallic, organic and particulate contaminants from silicon wafer surfaces. Solutions of HF are used for the wet chemical cleaning or etching of silicon dioxide on silicon wafers and integrated circuits (IC's). To avoid contamination of the wafer surface by the cleaning reagents themselves, the purity of the H₂O₂, HF and the other reagents must be extremely high. In response to this need, SEMI has recently proposed Tier D guidelines of a maximum contaminant level of 10 ppt for 21 elements in hydrogen peroxide.

Direct Analysis by ICP-MS

Direct analysis of concentrated H₂O₂ and HF using the Agilent 7500s ICP-MS combined with a trace metal free sample introduction system provides:

- Interference free measurements - a robust 27 MHz RF plasma completely ionizes the sample while Agilent's exclusive ShieldTorch System effectively removes argon and matrix based interferences
- Highest sensitivity of any ICP-QMS - ShieldTorch technology and optimized ion focusing deliver excellent performance for all critical elements including B, Zn, Ca, K and Fe
- Ease of use/Walk-away automation - determination of all elements in a single data acquisition and documented in a single report. No need for complex reaction cell technologies and potentially hazardous gases such as ammonia
- Clean sampling - Agilent's exclusive I-AS semiconductor auto-sampler is fully integrated and covered for ultra-trace analyses
- Clean sample introduction - O-ring free, PFA sample introduction system offers clean sample introduction, low backgrounds and excellent resistance to aggressive samples
- Cleanroom ready - the Agilent 7500s was designed specifically for cleanroom operation and undergoes a unique cleanroom preparation process prior to shipping.

*SEMI Semiconductor Equipment and Materials International

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Quantitative spike recoveries at the 5 ppt level, 50% of the proposed maximum level of 10 ppt as per SEMI methodology for H₂O₂ and HF indicate excellent measurement accuracy. This further highlights the suitability of the Agilent 7500s for the determination of all SEMI required elements in HF and H₂O₂ at both current and future required purity levels. Detection limit and spike recovery data for selected elements is given in Table 1.

Agilent Technologies: A Solutions Partner

Agilent Technologies has worked closely with leading semiconductor manufacturers and suppliers over many years developing products to meet the industry's dynamic needs. In addition to high performance instrumentation, Agilent offers the world-class applications and service support required to rapidly implement these measurement tools into your process. For more information, visit our semiconductor industry website at www.agilent.com/chem/semicon

Element	Detection Limit in HF (ppt)	Detection Limit in H ₂ O ₂ (ppt)	HF Spike Recovery 5ppt spike %	H ₂ O ₂ Spike Recovery 5ppt spike %
Li	0.05	0.02	99	99
Na	0.09	0.8	102	102
Mg	0.1	0.1	105	105
Al	0.2	0.3	117	117
K	0.1	0.2	108	108
Ca	2	0.6	98	98
Cr	1	0.7	104	104
Mn	0.2	0.2	99	99
Fe	0.3	0.6	111	111
Ni	0.1	0.3	102	102
Co	0.2	0.1	104	104
Cu	0.3	0.3	100	100
Zn	0.8	1	83	83
Zr	4	0.04	103	103
Sn	0.2	0.4	93	93
Ta	0.1	0.1	97	97
Au	0.2	0.2	96	96
Pb	0.08	0.08	98	98

Table 1. Detection limits (parts per trillion) and 5 ppt spike recoveries (%) in undiluted HF (38%) and H₂O₂ (35%).

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