# What's New at Balazs: Urea & Organic Acids

#### **TOC speciation for Ultra Pure Water monitoring**

The requirement for ultra-pure water in the semiconductor industry is well understood with parameters such as metals, anions, cations, silica, TOC, and bacteria being specified and analysis taking place at numerous locations throughout today's fabs. A recent article by Intel however highlights the fact that the requirements for DIW in the fab continue to evolve and require additional control and analysis to ensure a healthy manufacturing process.

In their article, "Identification of Critical Contaminants by Applying an Understanding of Different TOC Measuring Technologies" Ultrapure Water, February, 2002, Intel describes the common fertilizer and waste product urea as a major culprit in process problems. As shown in the simple cartoon below, urea can enter the groundwater and make its way through municipal water systems to the fab DIW systems. The eventual process problems that may arise from urea in DIW is that urea can break down to ammonia in the gas phase thereby affecting lithography processes from a shift in critical dimension or producing T-topping effects.

## <u>Urea Analysis</u>

Common online TOC systems were found to be inconclusive with respect to urea detection; some TOC monitors detected urea and some did not. This TOCinconsistency caused confusion as to the quality of DIW, the authenticity of the TOC excursions that were taking place as well as questions about the actual identity of the offending organic molecule. Eventually Intel put in place a "proprietary on-line urea-specific analysis" to confirm the presence of urea.



At Balazs we have developed a routine production method that provides urea-specific analysis that can easily be used off-line to cover as many locations as necessary. Shown in the mass spectrum below, urea is easily observed at the (M+H) mass/charge of 61 amu by using Ion Chromatography – Mass Spectrometry (IC-MS), a relatively new technique that is being pioneered in our laboratories.



Using this IC-MS technique Balazs has detected urea at concentrations down to 0.6 ppb, and can provide analyses on feed city water, process water used in the fab, as well as reclaim water for DIW and process engineers. DIW purities and concerns evolve, so does our analytical arsenal.



#### **Reclaim Water and Organic Acid Analysis**

For both cost reasons and water conservation, the use of reclaim water in semiconductor processes is on the way up. This trend is happening in the United States as well as in fabs around the world. With reclaim water being used in greater abundance comes the introduction of an "unknown" into DIW systems and the processes reclaim systems feed. The question begs, "What new contaminants could be introduced via the supply of reclaim water into the fab?"

Balazs labs routinely perform point-of-use and point-of-dispense analysis on DIW used in today's advanced fabs. Using ion chromatography, anions including chloride, fluoride, sulfate and cations such as ammonium are analyzed to process-driven specifications. A change in our chromatograms was observed in certain fabs where reclaim water was being used. Organic acid anions such as acetate, formate, and pyruvate were routinely being picked up. These findings were new, and were associated with the use of reclaim systems where organic acid compounds made their way into the final reclaim water.

With the development of a specific ion chromatography system to optimize separation, Balazs is now able to help in the analysis of organic acid compounds and troubleshoot reclaim water systems. Both inorganic and organic anions are separated and analyzed to concentrations down to 1 ppb, as shown in the chromatogram below. This setup allows fairly quick diagnosis of DIW reclaim systems to ensure new contamination is not being added. In combination with on-line TOC data, system owners are now able to quickly diagnose the source of an organic contamination.



1. Fluoride92. Glycolate13. Acetate14. Lactate15. Formate16. Propionate17. Chloride18. Nitrite

9. Tartrate
 10. Sulfate
 11. Oxalate
 12. Bromide

- 13. Nitrate
- Phosphate
  Citrate

## Presence of Urea in DIW can be a major culprit in process problems

Balazs is currently helping customers with this new organic method. The identification of organic anions is typically applied to reclaim and fresh DIW, as well as organic acid components in slurries. Another growing application involves leach tests on nitrile and vinyl gloves used within cleanrooms.

Ion chromatography may be an old technique but the applications are ever evolving.

