Water Analysis

Speciation of Organic Compounds in Ultrapure Water for Electronics Applications

Organic compounds present in ultrapure water (UPW) can pose serious problems for production processes within the electronics industry. UPW ITRS (International Technology Roadmap for Semiconductors) targets for advanced semiconductor applications are stringent, with TOC limits of 1 ppb for critical organics and 3 ppb for non-critical (volatile) organics. Organics speciation of the UPW system allows for detection of the critical organics and troubleshooting while serving as a method to support change control. It is particularly important when UPW recycling is considered. There are many methods for measurement of the organic compounds, but they are limited in either efficiency or specificity (ability to identify or detect significant portion of the organics.)

Organic Speciation using LC-OCD

Balazs™ Nanoanalysis offers organic speciation using liquid chromatography-organic carbon detection (LC-OCD) which provides an effective analysis of organic compounds with focus on non-volatile critical organics. This method allows for identification of biopolymers, humics, low molecular weight (LMW) acids, volatile organic compounds, and more, while characterizing nearly 100% of the organic composition. LC-OCD has an advantage over other methods when analyzing electronics UPW because it focuses on critical organics. Nevertheless, it is a non-target complementary method to existing target methods for detection of synthetic compounds. When accurate determination of specific compounds is required, more specific techniques may need to be used. LC-OCD analysis can help design treatment systems, monitor changes in water quality, and define solutions for a large array of problems without the need for expensive specialized tests.

Case Study

Analysis of UPW taken at different stages during the polishing steps is illustrated in Figure 1. Total TOC in the feed sample was around 1300 ppt. After passing through an additional reverse osmosis system, TOC was decreased to 77 ppt, where L2 was completely removed and urea was partially removed. The LC-OCD method allows for specific analysis of trace contaminants within UPW.

LC-OCD Method Overview and Definitions

The LC-OCD process begins with the injection of a small sample into a size exclusion chromatography column where high molecular weight (HMW) compounds are separated from LMW compounds. The sample is then fed to UV and organic nitrogen detectors, where all compounds containing nitrogen will be analyzed. A UV thin film reactor serves as the heart of the process where organic compounds are oxidized producing carbon dioxide, whereas the originally present CO_2 is removed through acidification and N_2 sparging. The resulting CO_2 is measured using a non-dispersive infrared detector (NDIR). The output of this analysis is a report containing the chromatogram (see Figure 2) and the spreadsheet providing organic speciation to the following content:

DOC – Dissolved Organic Carbon

DON – Dissolved Organic Nitrogen

HOC – Hydrophobic Organic Carbon (organic compounds that do not elute during the period of time of the test)

CDOC – Chromatographic (hydrophilic) Dissolved Organic Carbon (organic compounds that elute completely during the period of time of the test)

NOM – Natural Organic Matter

SOM – Synthetic Organic Matter > 10 ppb

Quantification and Characterization of:

- Humics
- Biopolymers
- Building Blocks
- LMW-acids

In addition to the above categories, specific organic compounds are identified based on the instrument library, characterized by chromatography retention time, nitrogen content, and presence of unsaturated bonds (identified by UV detector). Balazs™ also includes interpretations help for practical conclusions.
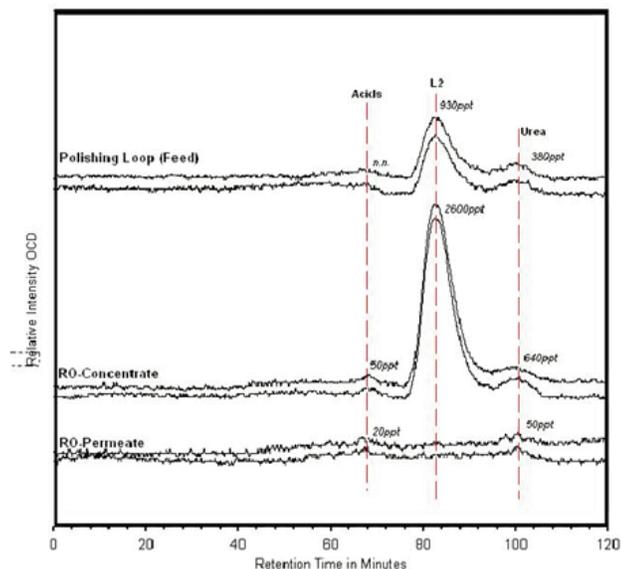

Figure 1. Analysis of water for injection in pharmaceutical applications
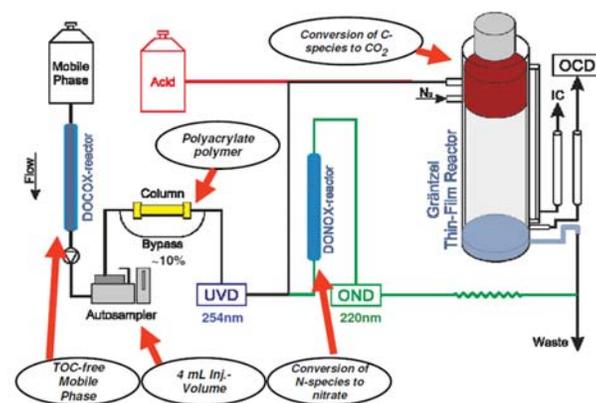

Figure 2. LC-OCD schematic diagram

References

- Huber, S.A.; Balz, A.; Abert, M., Pronk, W.: Characterisation of aquatic humic and non-humic matter with size-exclusion chromatography - Organic Carbon Detection - Organic Nitrogen Detection (LC-OCD-OND). Water Research 45 (2011), 879-885.
- Huber, S.A., Balz, A., Abert, M.: New method for urea analysis in surface and tap waters with LCOCD-OND (liquid chromatography-organic carbon detection-organic nitrogen detection). Aqua, 60.3, (2011), 159-165.
- Libman V. and Huber S. Part 1: An Overview of LC-OCD - Organic Speciation For Critical Analytical Tasks In the Semiconductor Industry ULTRAPURE WATER® , May/June 2014

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