The impact of low level organics on HPLC and LC-MS baselines of ultrapure water

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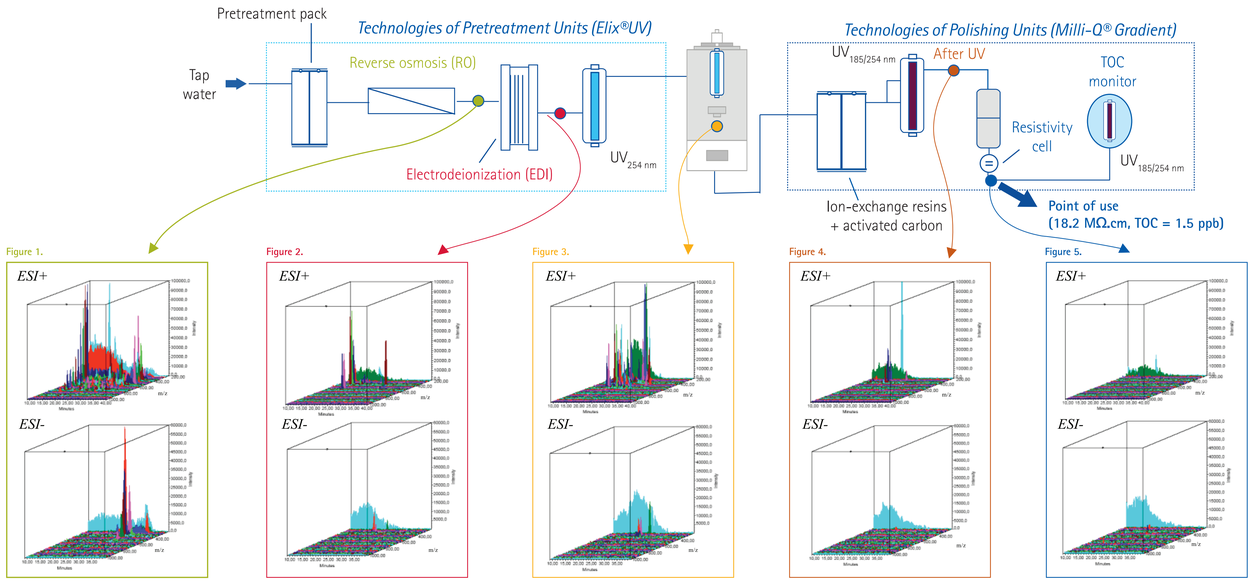
Fingerprints of Water at Various Stages of Purification

• Water is both the most widely used analytical laboratory reagent and the least-well characterized. While chromatographers take great care to assure the purity of salts, organic solvents, and other HPLC mobile phase components, they often take water quality for granted. High-purity water comprises by far the largest mobile phase component for most reversed phase HPLC and LC-hyphenated methods. Because of its wide utilization and because of the volumes used in sample preparation and liquid chromatography, extreme care must be taken with the water quality.

• In this study, reversed phase LC/MS analyses were performed on water at five different steps of a water purification chain that was chosen specifically because of its high potential for removing organic contaminants, thus for achieving the lowest HPLC or LCMS baselines. The samples of water were enriched on a pre-column prior to analyses. Water was analysed after the reverse osmosis step, after the electrodeionisation (EDI) step, after storage, after the UV photooxidation step, and finally after passing through a polishing cartridge made of activated carbon and ion-exchange resin (Jetpore). PDA and ESI-Simple Quadrupole (ESI positive and ESI negative modes) were the two modes of detection used to analyse the five types of water.



Analytical conditions

- Sampling : Waters to be analyzed were sampled in 1 L flasks that were rinced with MilliQ® Gradient in ultrasonic bath for 20 minutes
- Pre-concentration : 100 % of water was preconcentrated on preconcentration column for 1 h at 1 mL/min.

Flowrate was then lowered to 0.25 mL/min. Analytical column was adapted to preconcentration column.
System is stabilised at 100 % for 5 minutes before run

- Mass spectrometer parameters :

Scan from *m/z* 200 to 1000 in positive and negative mode
Scan time : 0.7 s - Inter-scan delay : 0.3 s
Capillary voltage ESI+ : 2.9 V - ESI- : 2.8 V
Cone voltage : 45 V
Extractor voltage : 5 V
RF lens : 0.2 V
Photomultiplier : 500 V
Source Temperature : 100 °C
Desolvatation Temperature : 250 °C
Cone Gaz : 100 L/h
Desolvatation Gaz : 300 L/h

- PDA parameters : 210 nm
- Eluants : Water (Milli-Q® Gradient, Merck Millipore), Acetonitrile (HPLC Grade, JTBaker)
- Injection volume : 0 µL
- Sample collection : A volume of at least 300 mL were collected and thoroughly mixed to ensure perfect homogeneity of the sample
- Equipment (Waters) :
- Column : Atlantis dC18 2.1 x 150 mm, 3 µm
- Pre-column : X-Terra MS C18 4.6 x 30 mm, 3.5 µm
- HPLC : Alliance 2995 Waters
- PDA : 2996 Waters
- Mass spectrometer : ZQ2000 Waters- Electrospray Ionization and Simple Quadripole Mass analyzer Software : Empower
- Gradient : 100 % water to 100 % ACN in 30 min then 100 % ACN for a further 10 min

Comparison of Various Purification Technologies

Fingerprints (HPLC and MS) were obtained for water purified using a variety of purification technologies. It is clear that Service Deionization and distillation are not appropriate to produce water suitable for LC-MS. EDI is used as a reliable pretreatment step before further purification on a polishing unit (Milli-Q®).

HPLC 210nm

ESI + from 200 to 600 *m/z*

Double distilled water

SDI water

EDI water

Milli-Q® Gradient water

References

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