

# METHOD STATEMENT



## **Determinand:**

Screening of a list of selected target compounds and identification of 'unknown' compounds

## **Matrix:**

Sample Types: Raw and Potable waters.

## **Principle of Method:**

A direct aqueous large volume injection (LVI) on-line solid phase extraction procedure. Samples are analysed by high performance liquid chromatography with a high-resolution accurate mass spectrometer as a detector.

Samples are injected by large volume injection onto an enrichment column, the enrichment column is back flushed on to the analytical column. Target organic compounds are separated and then identified and quantified by using high-resolution accurate mass data. Quantitation is by either internal or external standard.

Unknown organic compounds are identified by library searching the 'all-ions' mass spectral data. No quantification is performed on any identified 'unknown' compound.

## **Sampling and Sample Preparation:**

Water samples should be collected in 40 mL amber glass vials with PTFE lined screw caps containing sodium thiosulfate (STL53).

Sampling, the vial is slowly and completely filled to exclude headspace. When the water just begins to overflow the vial, it is capped with the PTFE face of the septum in contact with the sample.

Storage - samples should be analysed as soon as possible after collection. When this is not possible, samples are stored under refrigeration at 1-5°C in the dark, until analysis can begin.

## **Interferences**

HPLC-HRMS is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical high resolution accurate mass,  $m/z$ , at the same retention time, this is extremely unlikely in potable water samples. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference. Samples containing high humic or fulvic loading have been demonstrated to not cause significant ion suppression for the compounds.

## **Performance of Method:**

### **Range of Application:**

Calculation of Concentration is performed using a two-point calibration curve and the Agilent MassHunter quantitation software.

### **Limit of Detection:**

50 ng L<sup>-1</sup>

### **Recoveries of Compounds, Bias and Uncertainty of measurement:**

N/A

## **References:**

Determination of Phenyl Urea and Triazine Herbicides in Potable and Groundwater by LC/MS Using API-ESI Selective Ion Monitoring and Direct Large Volume Aqueous Injection, Agilent Technologies Application Note.

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Transport of sulfonylureas from a barley field in Norway: Field and laboratory studies, Bioforsk Report Vol.3 Nr. 105 2008, ISBN-nr.:978-82-17-00409-7

Agilent 1200 Series, Reference Manuals

Agilent QQQ LC/MS Techniques and Operation, Agilent Technologies Course Number R1893A, Student Manuals Volumes 1 and 2.

Time-of-Flight Mass Spectrometry, 5990-9207EN.

Agilent 6500 Series Q-TOF LC/MS system, Maintenance Guide, G2581-90065.

Agilent MassHunter Workstation Software Data Acquisition for 6200 Series TOF and 6500 Series Q-TOF, Familiarization Guide, G3335-90066

Agilent 6200 Series TOF and 6500 Series Q-TOF LC/MS System, Concept Guide, G3335-90142.

Agilent 6500 Series Q-TOF LC/MS, Tuning Guide, D0006318, July 2021, Revision A.00.