

Initial work for the determination of Taste, Odour and Phenolic compounds in water using ITSP on the new Agilent GC-QQQ 7010

Anaïs Maury, Anatune Ltd. Girton, Cambridgeshire (UK).

Introduction

Taste, Odour and Phenol compounds in drinking water are one of the most widespread causes of customer complaints. Many of these analytes can be detected with the human nose and palate at extremely low levels. Therefore, some form of enrichment is required to detect these analytes by GC/MS.

With the current water quality criteria getting more and more exigent and the drive to get more results in shorter time, it becomes crucial to have a system that can reproducibly extract and detect analytes at very low concentrations to meet requirements. We see the automation as the way to increase your throughput and improve the reproducibility.

Within this application note, we show how SPE can be fully automated to enrich Taste, Odour and Phenolic compounds from water samples using ITSP (Instrument Top Sample Preparation) cartridges. ITSP is a consumable cartridge (manufactured by ITSP Solutions) designed to automate small scale SPE on the Gerstel MPS (MultiPurpose Sampler). In this application, an ITSP cartridge packed with 20 mg Biotage ENV+ sorbent is used.

The new Agilent Triple Quadrupole 7010 with its new High-Efficiency EI ion source combined with Multiple Reaction Monitoring detection (MRM) provides a highly selective and sensitive method for these compounds.

Figure 1 shows a photograph of the instrumentation which was used for this study.



Figure 1: Gerstel Dual Head MPS with Agilent GC-Triple Quadrupole

Instrumentation

Gerstel Multipurpose Sampler (MPS) 2 XL Dual head
Gerstel Cooled Injection System (CIS) 4
Instrument Top Sample Preparation (ITSP), ITSP Solutions
Agilent GC 7890B
Agilent 7010 Triple Quadrupole

Method

Compound list:

2-Isopropyl-3-methoxypyrazine	Phenol
2-chloroanisole	2-chlorophenol
3-chloroanisole	2,3-dichlorophenol
4-chloroanisole	2,4-dichlorophenol
2-Isobutyl-3-methoxypyrazine	2,4,6-trichlorophenol
MIB	
2,3,4-trichloroanisole	
2,4,6-trichloroanisole	
Geosmin	
2,4,6-tribromoanisole	

GC/MS conditions:

CIS4 inlet: 10 µl large volume injection
Column: DB-5 30 m x 0.25 mm x 0.25 µm
Thermal gradient performed up to 300 °C
MS: High-Efficiency EI source, MRM performed using two transitions for each analyte

Extraction procedure:

Using the left MPS fitted with a 2.5 ml headspace syringe (SPE needle), the ITSP cartridge (Biotage ENV+) was conditioned with dichloromethane, methanol and HPLC grade water. 10 ml of sample (spiked water) was then loaded and the cartridge was dried for 15 minutes with nitrogen. Drying is a critical step to get the best recovery of the compounds from the cartridge. After drying, the analytes were eluted with 400 µl dichloromethane into a standard GC vial.

Assuming 100% recovery, eluting in 400 µl DCM will give a 25 fold increase in concentration.

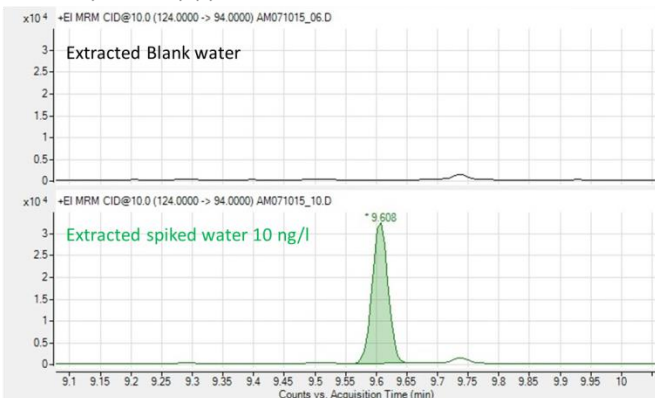
The right MPS head fitted with a 10 µl syringe was used to inject 10 µl of the extract into the Cooled Injection System (CIS 4).



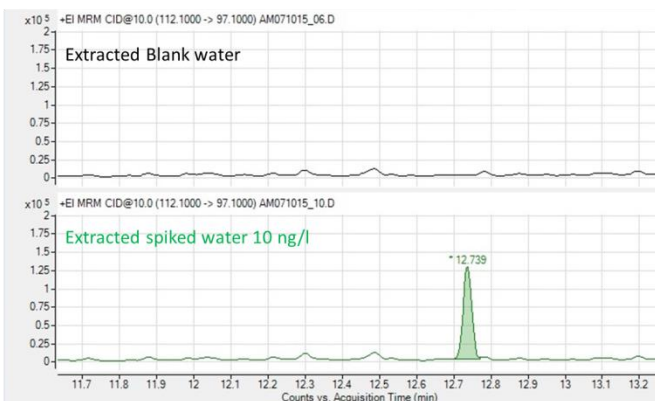
Results

Waters spiked at 10 ng/l with stock solutions have been extracted through ITSP. Below shows comparisons of blank water extracted and 10 ng/l spiked water extracted (MRM strongest transition).

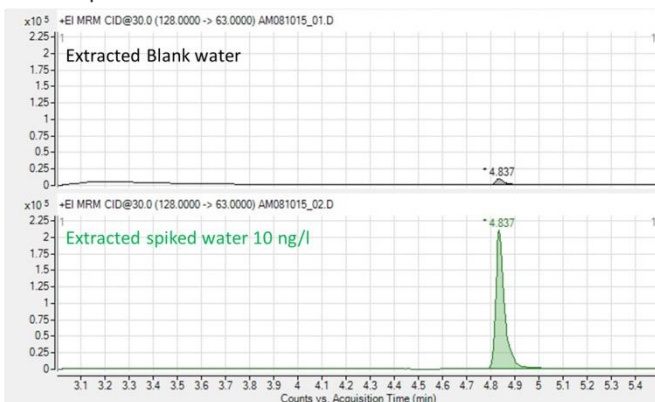
2-Isobutyl-3-methyl pyrazine



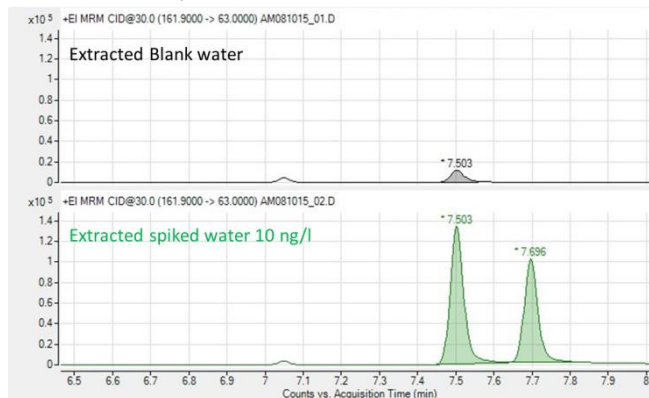
Geosmin



2-chlorophenol



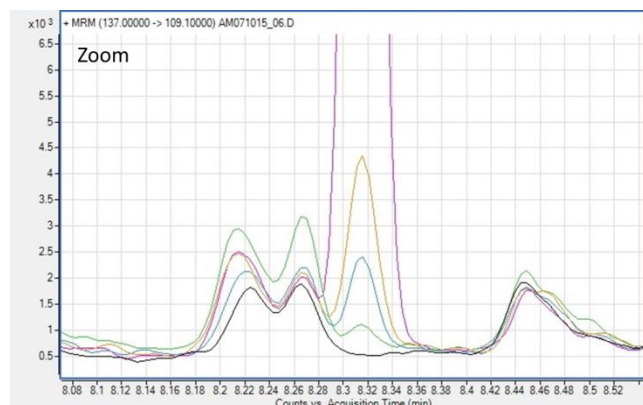
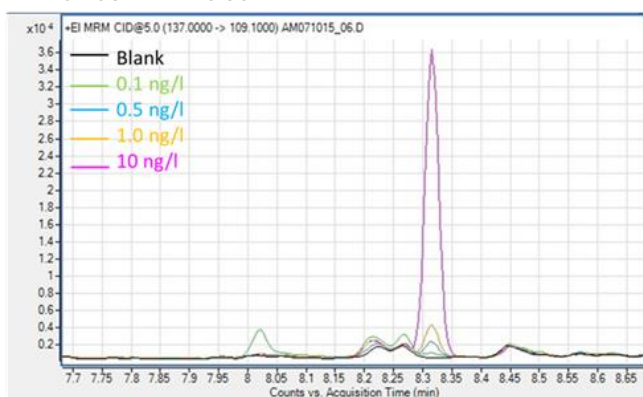
2,3 & 2,4-dichlorophenol



With the Taste and Odour mix, a four point calibration was prepared in water at concentrations ranging from 0.1 ng/l to 10 ng/l. No internal standard has been used.

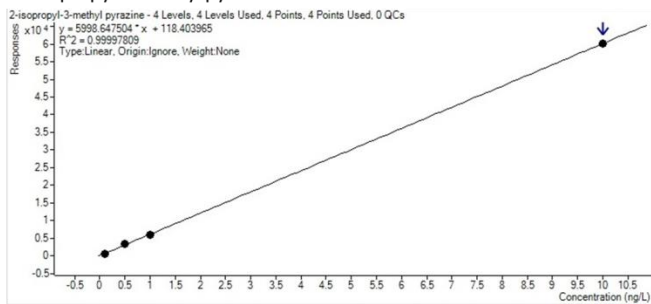
Below shows the overlaid MRM transitions and the linear calibration curves for 2-Isopropyl-3-methoxypyrazine, 2-chloroanisole & 4-chloroanisole and Geosmin. Correlation coefficients above 0.999 have been achieved. Sensitivity could be improved by increasing the injection volume.

2-Isopropyl-3-methyl pyrazine

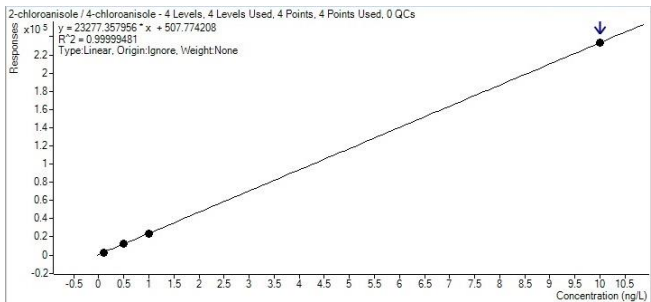
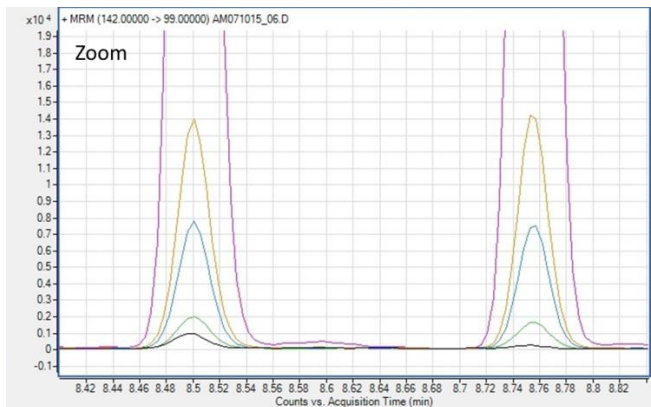
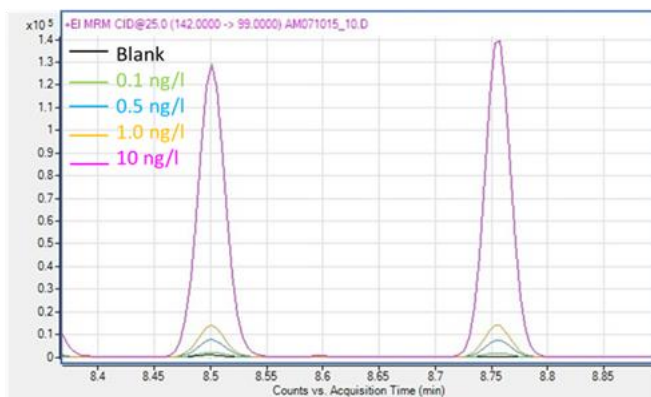




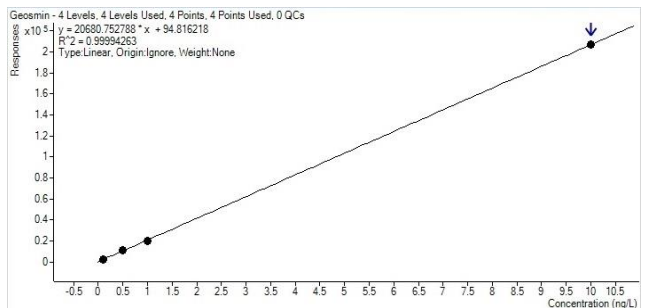
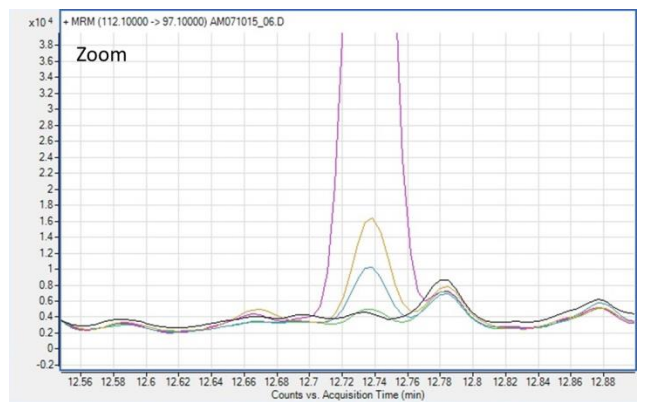
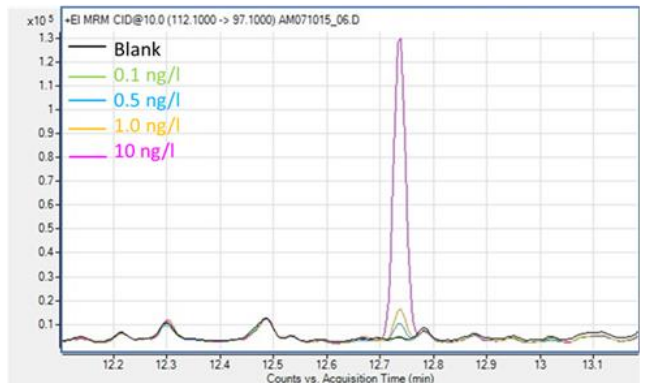
2-Isopropyl-3-methyl pyrazine



2 & 4-chloroanisole



Geosmin

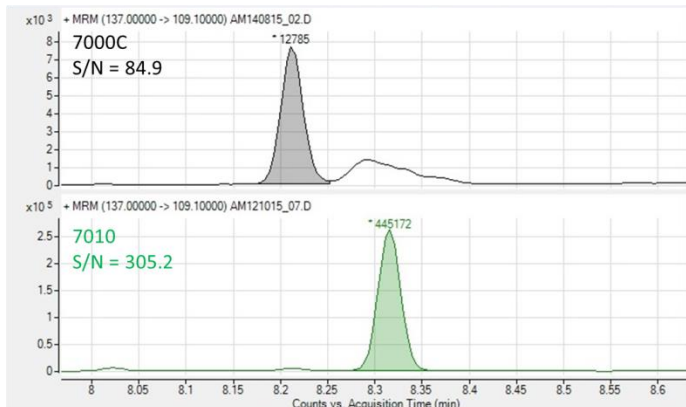


The sensitivity at 0.1 ng/l can be improved for compounds like Geosmin by increasing the injection volume (up to 50 µl for example).

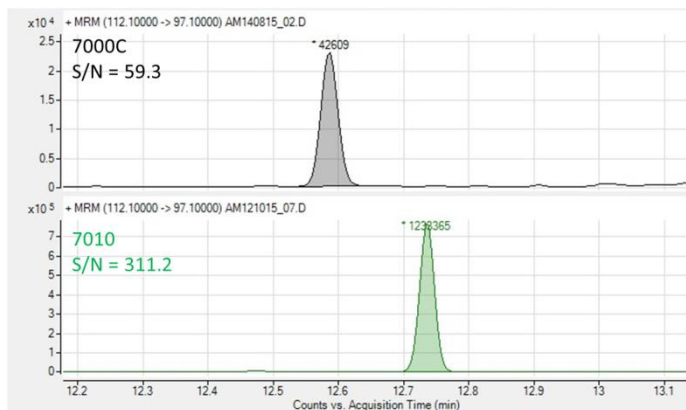


Finally, a comparison in performance has been done between the Agilent 7000C and the new 7010. The extract of a water spiked at 75 ng/l with the Taste and Odour mix has been run on the 7000C and the 7010. Below shows some compared chromatograms. The peak area is annotated on top of the peak.

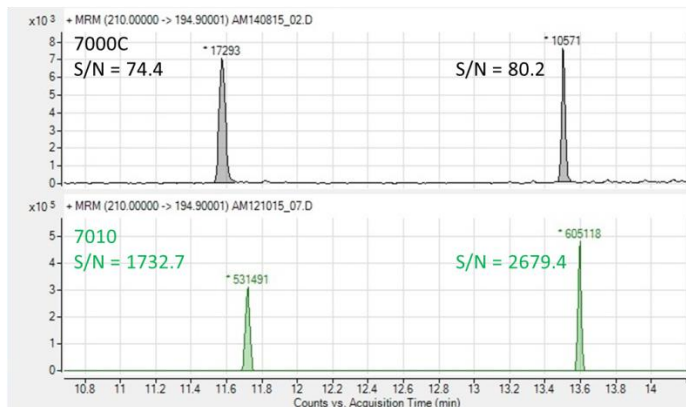
2-Isopropyl-3-methyl pyrazine - Extracted spiked water 75 ng/l



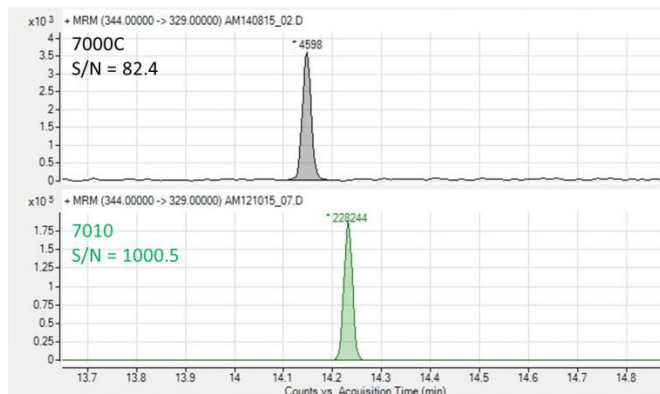
Geosmin - Extracted spiked water 75 ng/l



2,3,4 & 2,4,6-trichloroanisole - Extracted spiked water 75 ng/l



2,4,6-tribromoanisole- Extracted spiked water 75 ng/l



Discussion

This application note shows the excellent performance of the new Agilent 7010 triple quadrupole and how the extraction of Taste, Odour and Phenol compounds from water samples can be automated using ITSP.

ITSP allows scaling down the SPE steps; therefore, the amount of solvent and glassware used is significantly decreased.

Further work could be done using real waters (soft, medium and hard water). The calibration range can also be extended to a higher range, with more calibration levels.

If you would like to discuss this further, please do not hesitate to contact us, either by emailing enquiries@anatune.co.uk, or call us now on +44 (0)1223 279210.

Acknowledgement

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Dwr Cymru Welsh Water