AUTOMATED LIQUID-LIQUID EXTRACTION OF GEOSMIN IN WATER BY GC-MS/MS

Jonathan Dunscombe, Anatune Ltd., Girton, Cambridgeshire, UK.

INTRODUCTION

Being able to achieve low limits of detection (LOD) is a must for environmental laboratories. The European Water Framework Directive is the driving force behind this. However, regulation is not the only factor. Environmental and water analysis laboratories also are affected by what humans can detect. This is the main criteria behind taste and odour analysis. For taste and odour compounds, there is no standardised limit for water treatment companies, with the DWI regulations for drinking water stating that the limit for taste and odour compounds is what is deemed acceptable by the customer. What this means that any compound that can be detected through a pleasant or unpleasant taste or odour is a problem and must be dealt with as such.This causes a problem for laboratory analysis as with many taste and odour compounds, the limit of detection for the human sense of taste or small can be far lower than what can be achieved by analytical instrumentation.

Recent work has been carried out at Anatune using liquid-liquid extraction. This technique is heavily utilised in the environmental and water analysis sector. Whilst being tried and tested, current manual methods are error-prone, time consuming and labour intensive. Application of automation of the required limits of detection is now a distinct possibility which will be explained below.

METHODS

INSTRUMENTATION

GERSTEL Robotic/Robotic Pro Dual Head
GERSTEL Cooled Injection System (*CIS) 4 PTV inlet*
GERSTEL QuickMix
Maestro software integrated
Agilent 7010A High Efficiency Source Triple Quadrupole MSD
Agilent GC 7890A
Anatune CF-200 Robotic Centrifuge
Masshunter B.07.04

A standard liquid-liquid extraction was employed with the MPS being used to spike standards, add solvent and shake samples automatically. Large Volume Injection of sample was carried out by the MPS from the solvent layer within the sample into a GERSTEL CIS PTV inlet.

Standards were spiked as follows:

Level (ng/L)	Spike (µL)	Standard concentration (µg/L)
1	1.2	5
5	6	5

Table 1: standard spiking procedure

RESULTS

Three samples were analysed, Blank 1ng/L and 5ng/L. The MRM chromatograms for transition m/z112→83 can be seen below.

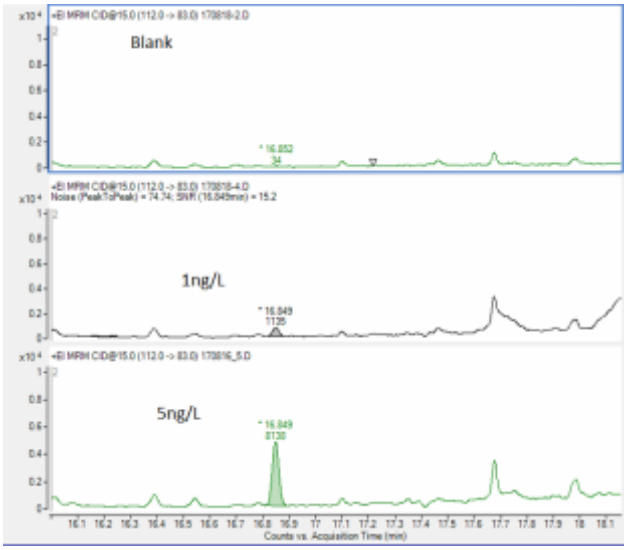

Figure 1 – comparison of spiked standards

The selectivity of the triple quadrupole detector shows geosmin is easily identifiable at a low level while retaining a good signal to noise value.

CONCLUSION

This work demonstrates that sensitivity and selectivity can be achieved by using the appropriate instrumentation (Agilent 7010 Triple Quadrupole). By combining this with fully automated sample preparation, this allows for a high throughput, efficient and reliable industry. This has the potential to save time within the laboratory but to also make a difference to the reactivity of the water quality teams when trigger level results are obtained. Further optimisation of this method with the right conditions could potentially provide LODs below 1ng/L.

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Anatune Ltd
Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223279210 Fax: +44 (0) 1223279253
Email: info@anatune.co.uk Internet: www.anatune.co.uk

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