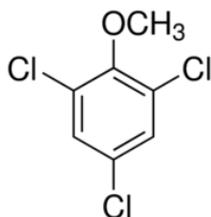


Jonathan Dunscombe, Anatune Ltd., Girton, Cambridgeshire, UK.

INTRODUCTION

Chlorinated and brominated phenols are used as fungicides to treat wood. For example, these compounds are used to treat corks destined for wine bottles and this is where the term 'corked wine' comes from. If the wine has a musty or mouldy taste then this is a result of the interaction of the chlorinated phenols used in the treatment of the wine corks with naturally occurring bacteria and fungi methylating the phenol hydroxyl group. Subsequently the produced chemicals – anisoles, cause of the malodour. Humans are able to detect these compounds in a few parts per billion (some even parts per trillion) and being able to analyse samples with this threshold is a must for environmental and food laboratories alike.



Recent work has been carried out at Anatune using this methodology and applied it to liquid-liquid extraction. This technique is heavily utilised in the environmental and water analysis sector. Whilst being tried and tested, current manual methods are very time and labour consuming. Application of automation in this field brings a lot of positives whilst remaining very sensitive.

METHODS

GERSTEL Robotic/Robotic Pro Dual Head
GERSTEL Cooled Injection System (CIS) 4 PTV
GERSTEL QuickMix
Maestro software integrated
Agilent 7010 High efficiency source triple quadrupole MSD
Agilent GC 7890B
Anatune CF-200 Centrifuge
MassHunter B.07.04

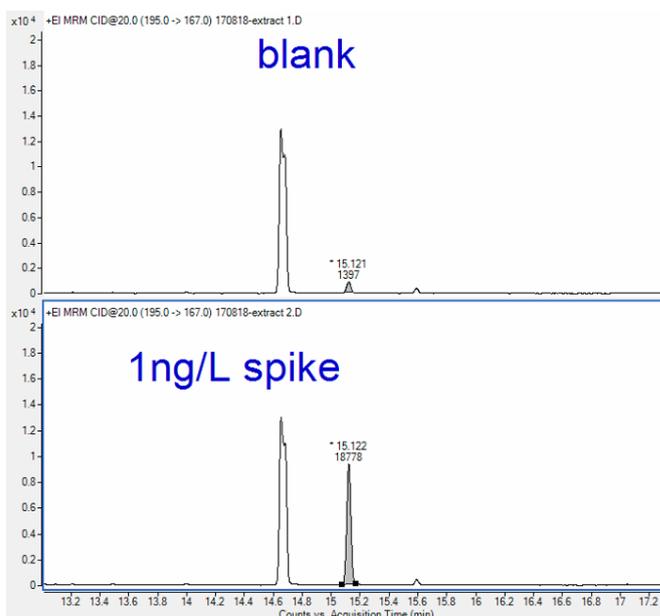
Optimised liquid-liquid extraction procedure:
-6mL sample pipetted into 10ml high recovery vial (manual step)
-Addition of standard with robotic pro equipped with 10µL syringe
-Addition of 200µL solvent with robotic pro equipped with 2.5ml syringe
-Shake in quickmix for 3 minutes
-Centrifuge for 5 minutes
-Inject 10ul of top layer of solvent into PTV inlet



The picture on the left depicts a 10mL high recovery vial which enables the recovery of small amounts of solvent.

RESULTS

A blank and 1ng/L spiked sample was analysed. The MRM chromatograms for transition m/z 195→167 can be seen below.



The selectivity of the triple quadrupole detector means that this compound is easily identifiable at a low level while retaining a good signal to noise. With chlorine being present, unique masses can also be used to reduce the background. The peak-to-peak signal to noise value at 1ng/L is 881.

CONCLUSION

This work demonstrates that sensitivity and selectivity can be achieved with combination of the right apparatus. Having a range of techniques available ensures that laboratories are able to meet customer needs. Automation and reduced sample size does not need to mean that sensitivity is lost.