Wellbrook Court I Girton Road I Cambridge I CB3 0NA I
I tel: +44 (0) 1223 279210 I fax: +44 (0) 1223 279253
I email: enquiries@anatune.co.uk I anatune.co.uk

# AUTOMATED EXTRACTION OF POLYCYCLIC AROMATIC HYDROCARBONS FROM SOILS

*Jonathan Dunscombe, Anatune Ltd, Cambridge, UK*

## INTRODUCTION

Soils are often tested for persistent organic pollutants such as pesticides, compounds from industrial waste and seepage and other compounds introduced to the environment from human activity.

Soil samples can be particularly tricky to extract and analyse with the analyst needing to ensure homogeneity of samples. Due to the heavy matrix, samples can require long extraction times to achieve good recoveries due to adsorbance of analytes onto sample particulates, which gives automated techniques the advantage of performing these extractions unsupervised.

Previous work using a methanolic extraction method for the analysis of BTEX compounds (benzene, toluene, ethyl benzene and xylenes) from soil.

In this work, dichloromethane is utilised as the extraction solvent to look for the sixteen Environmental Protection Agency Polycyclic Aromatic Hydrocarbons, PAHs, to expand soil extraction to less volatile compounds.

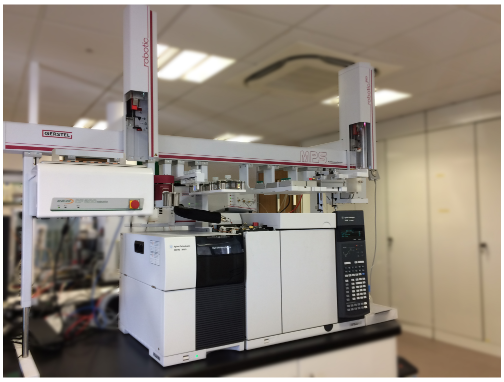

**Figure 1.** GERSTEL Dual Head Robotic/Robotic Pro MPS atop Agilent GC/MS.

## METHOD

Four aliquots of 2.5g of proficiency scheme reference material were weighed into four 10ml vials containing sodium sulphate and sodium chloride. The samples were then placed onto the MPS for extraction. An aliquot of surrogate solution containing labelled PAHs followed by dichloromethane. Samples were then mixed for 5 minutes followed by centrifugation. Extractions were then transferred to a 2mL autosampler vial with addition of internal standard. Extracts were then analysed by GC-MS at ALS Hawarden. Data was then assessed within a proficiency scheme to calculate a z score to statistically compare results against the mean values submitted by participating laboratories. A video showing the automated method can be seen here.

## RESULTS

Table 1 displays the data produced using the automated method for extraction. Z scores within ±2 are achieved for all analytes. This indicates that the data produced from the automated method closely match that of the target result.

| Analyte | Result mg/Kg | Interlab Average mg/Kg | Z score |
|---|---|---|---|
| Naphthalene | 4.58 | 4.65 | -0.07 |
| Acenaphthylene | 3.74 | 3.64 | 0.13 |
| Acenaphthene | 1.2 | 1.26 | -0.23 |
| Fluorene | 4.68 | 4.55 | 0.14 |
| Phenanthrene | 29.4 | 27.42 | 0.36 |
| Anthracene | 5.3 | 5.3 | 0.00 |
| Fluoranthene | 35.5 | 33.7 | 0.26 |
| Pyrene | 27.5 | 26.9 | 0.11 |
| Benz(a)anthracene | 14.1 | 13.64 | 0.17 |
| Chrysene | 10.7 | 12.6 | -0.74 |
| Benzo(b)fluoranthene | 17.5 | 15.1 | 0.78 |
| Benzo(k)fluoranthene | 6.57 | 6.59 | -0.02 |
| Benzo(a)pyrene | 13.4 | 13.1 | 0.11 |
| Indeno(123cd)pyrene | 7.02 | 8.25 | -0.73 |
| Dibenzo(ah)anthracene | 2.22 | 2.23 | -0.02 |
| Benzo(ghi)perylene | 7.68 | 7.6 | 0.05 |
| Benzo(b+k)fluoranthene | 24.07 | 22.8 | 0.28 |
| PAH 16 EPA Total | 191 | 190 | 0.03 |

**Table 1.** Analyte concentrations and respective z scores.

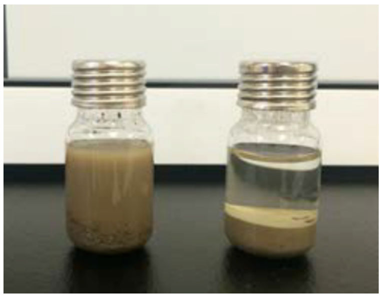

**Figure 2.** Samples before and after centrifugation.

## CONCLUSIONS

Use of automated sample preparation techniques can help to reduce manual steps whilst producing reliable data. Z scores for all analytes well within the regulatory tolerance of ±2, this gives good confidence of reliability of the method. Fewer manual steps results in fewer manual handling risks and smaller exposure of analysts to solvents and other chemicals used.

Centrifugation using Anatune CF200 makes this type of extraction appropriate for automation.

## ACKNOWLEDGEMENTS

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