Application Note No. 094

TPHs in Wastewaters using the Focus Robotic Sample Processor

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Introduction

Traditional liquid-liquid extraction of wastewaters for TPH (Total Petroleum Hydrocarbons) generally requires large samples i.e.1L to be extracted with an appropriate solvent, the resulting extract concentrated to a suitable volume and a small i.e. 1-3µl volume injected into a GC-FID. This technique while effective is nonetheless labor intensive and requires large amounts of sample and solvent. As most of the glassware is used repetitively the possibility of cross contamination is present despite thorough cleaning.

The Focus robotic sample processor combined with Optic large volume injection provides the opportunity to overcome these issues while improving the quality of the results produced. Automated extraction using the Focus improves the reproducibility of the extractions, it minimizes any risk of carryover/cross contamination during extraction, it uses only a tiny volume of solvent/sample and it reduces the manual input dramatically.

These benefits are desirable for several reasons and impact on both analyst and client alike, as turnaround times can be dramatically improved by use of this technique. The Environment Agency in Leeds tested this instrumentation with some success and as a result it was decided that the technology be further investigated. It was decided that the method used by the EA would provide a sound starting point but that several of the steps employed by them were unnecessary for current requirements; as a result of this the drying and clean-up steps were removed and a standard extraction and injection method set up.

The extraction solvent volume was reduced and the sample size maintained, the aim of this was to increase the limit of detection. A further consideration was the salting of the vials, this increased the cost of the test and required weighing out to a high degree of accuracy as the impact on volume was critical. As a result of this acidification was tested. The acidification can also be carried out by the robotic sampler and thus the only manual inputs would be putting the sample in the vial and the data analysis.

The finalized method was tested by spiking blank water with diesel and oil at two levels and quantifying against an extracted 5 point calibration.

Instrumentation

- Focus Robotic Sample Processor
- Optic 2 Programmable Injector
- Agilent 6890 GC with FID
- Chromeleon data system

Analysis Method

- Accurately add 15 mL water sample to 20 mL headspace vial
- Add 2 mL 10% HCl with dispenser (can be automated)
- Crimp vial with magnetic cap with special septum
- Place in sample tray
- Focus automatically adds 1 mL extraction solvent
- Transports vial to Agitator and shakes sample for 10 mins
- Returns vial to tray and breaks any emulsion
- Allows sample to settle
- Injects 200 uL of the sample extract

Results

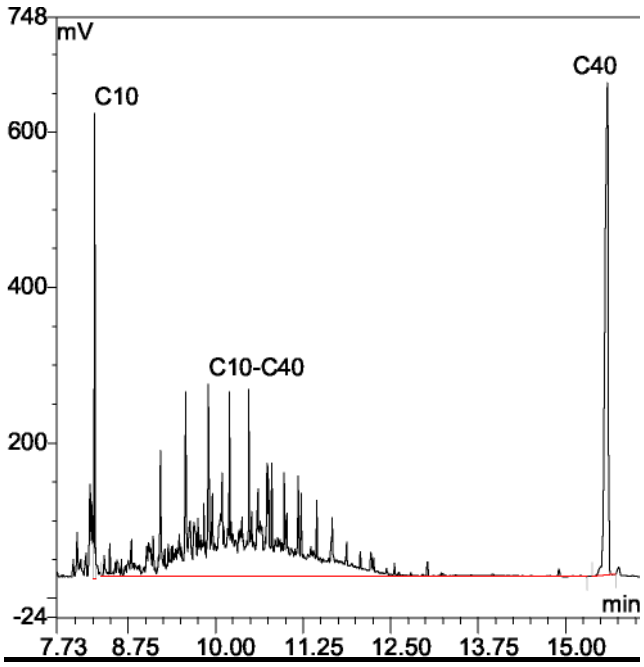

Figure 1: *An extracted 1 ppm diesel spike*

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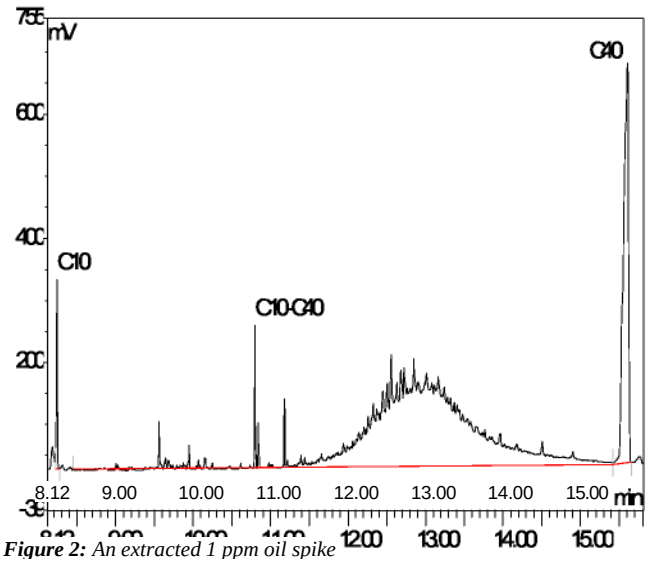

Figure 2: An extracted 1 ppm oil spike

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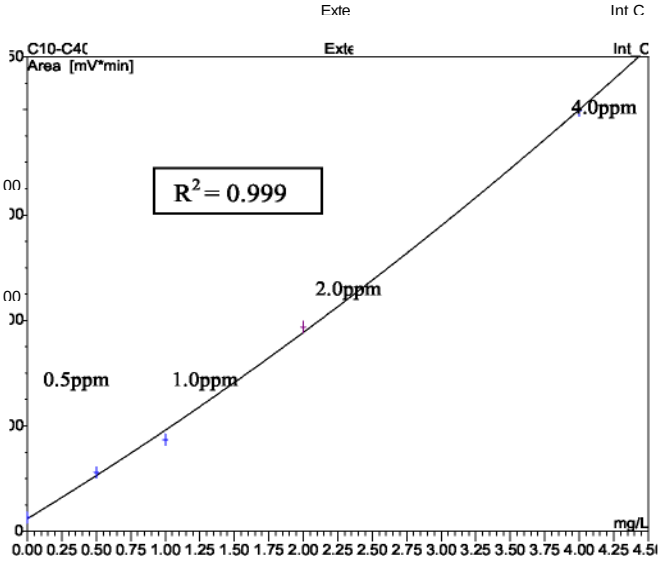

Figure 3: Calibration of diesel

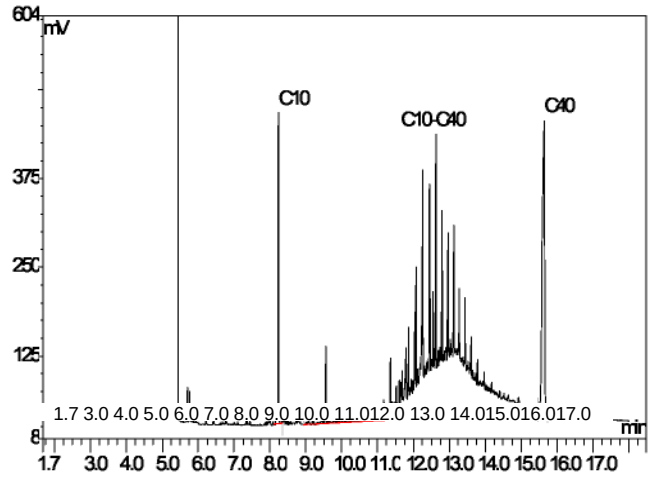

Figure 4: Real sample

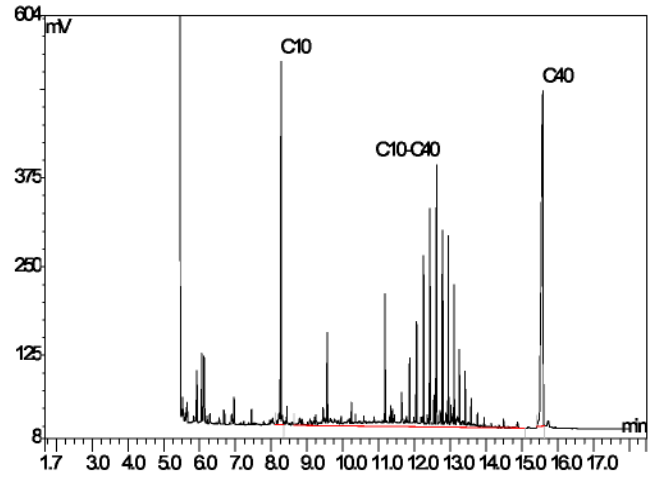

Figure 5: Real sample

Conclusions

Table 1: Performance data

Spike Level	0.0 ppm	1.0 ppm	2.0 ppm
Recovery	n/a	99%	105%
RSD	2.15%	11.2%	6.74%

The Focus combined with the Optic has provided a valuable tool for the rapid analysis of wastewaters, the advantages of such a tool are tremendous ranging from the ability to carry out a proper analyses on just 15ml of sample to the complete eradication of labor intensive and environmentally unfriendly manual liquid-liquid extractions. It must be noted that all this is achieved without a loss of detection limits which is rapidly becoming a key area of concern for many consultants and laboratories alike. This system should prove invaluable in the future as a tool for rapid TPH in water analysis.

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