HYDROCARBON OIL INDEX: TWO NOVEL APPROACHES TO SCALE DOWN AND AUTOMATE THE C10-C40 ANALYSIS

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INTRODUCTION

The TPH quantitation in environmental samples is regulated in many EU State members[1]. The work is focused on the EPH (C10-C40) fraction that requires extraction, cleanup and quite often sample concentration or large sample volumes to achieve the required LOQs.

SRA has developed and optimized two solutions that provide high throughput and almost unattended operations: the OIW GC platform and the "IN-TIP" Florisil® cleanup.

OIW GC – INTEGRATED EXTRACTION, CLEANUP AND INJECTION

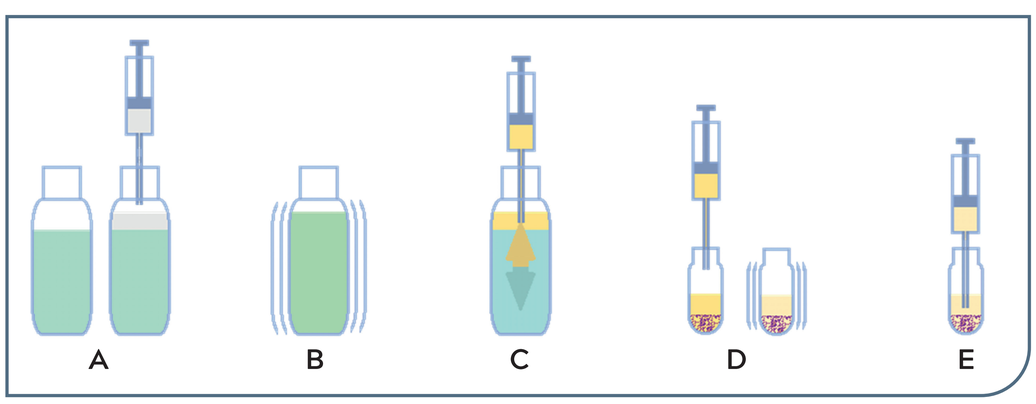
The OIW GC platform is based on a multipurpose robotic sampler (GERSTEL Robotic) and provides automated sample extraction, clean-up and GC injection.

The MPS is equipped with a decapper to prevent the siloxanes contamination of the extract, a centrifuge to force the phase separation and the GERSTEL QuickMix to allow efficient extraction in a very limited time.

EXPERIMENTAL

1.3 mL of n-hexane RTW mixture is added to 18 mL of water sample (**A**) and then extracted with using the GERSTEL QuickMix (**B**). After a 4000 rpm centrifugation step, the extract is transferred to a 4 mL vial pre-filled with Florisil®/Na_2SO_4 mix (**C**), which is shaken and centrifuged to prevent any suspended particle to be injected (**D**).

400 µl of the cleaned-up extract (**E**) are finally injected into a modified COC injector equipped with a solvent vapor exit device after the precolumn; this injection technique allows the system to reach detection limits below 25 ppb[2].

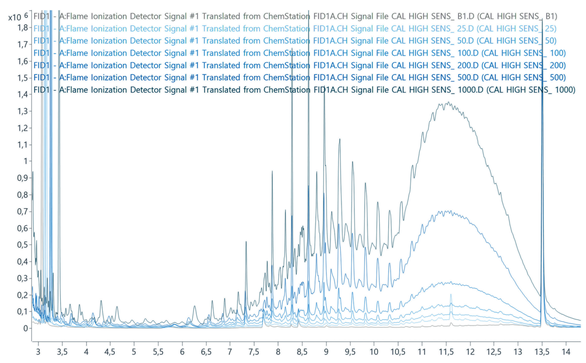


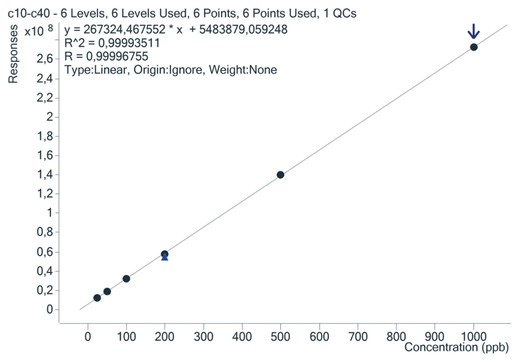
RESULTS

The system is calibrated in the range of 25 ppb to 1.000 ppb by directly spiking the blank water sample with a A+B mineral oil mixture properly dissolved in a water compatible solvent (Acetone).

This approach is applicable for underground, drinking and surface waters with limited amounts of surfactants. Different matrices might require variations of the extraction volumes ratio.

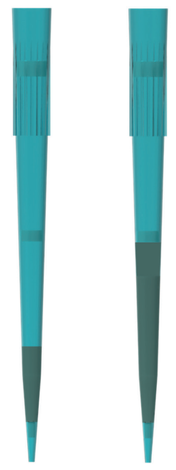
Unattended operation and the limited amount of solvents and reagents dramatically reduce the overall running costs of the analysis.





"IN-TIP" CLEANUP

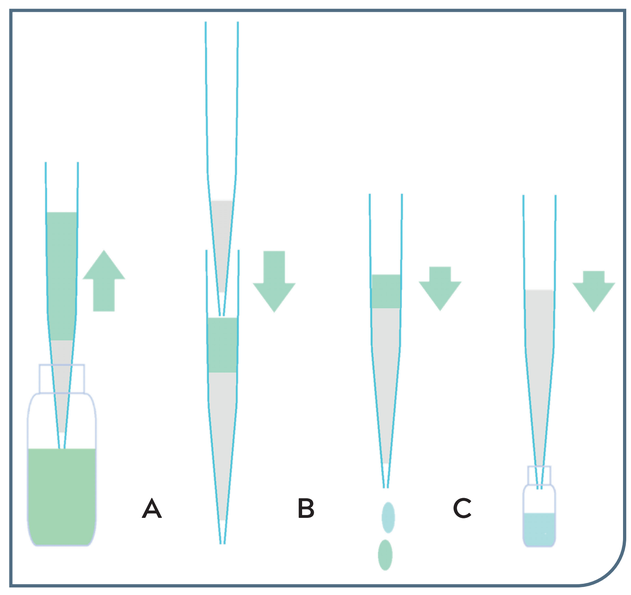
The "IN-TIP" Florisil® cleanup has been developed and optimized in terms of background and workflows consolidating the attractive possibility of scaling down the C10-C40 Florisil® clean-up.



EXPERIMENTAL

Common 1250 mL pipette tips filled with preconditioned Florisil® (230 mg or 280 mg) and Na_2SO_4 (200 mg) are provided by Chromline s.r.l.

The optimized operation is executed in just three simple steps:



(**A**) 1 mL of the extract is transferred with the Na_2SO_4 tip on top of the Florisl® tip.

(**B**) The Florisil® tip is eluted discarding the first 100 µL.

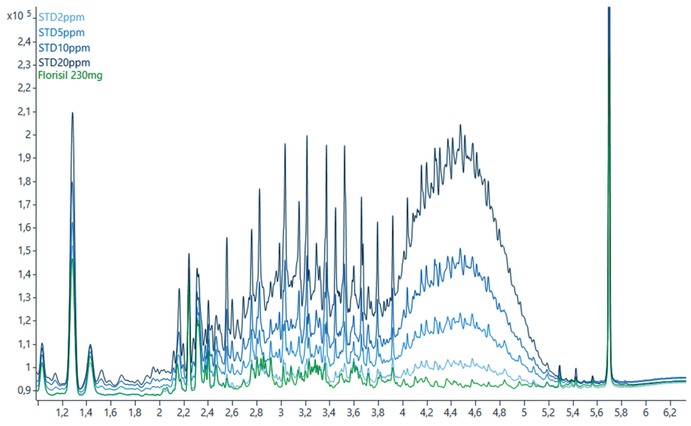
(**C**) The remaining extract is collected in the injection GC vial equipped with a 500 µl insert.

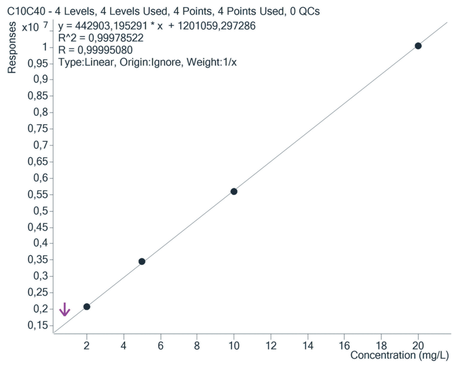
RESULTS

The background contribution to the C10-C40 concentration in the final extract is contained below 1 ppm, and the tips shelf life can be extended up to 6 months without cleanup efficiency losses.

The tips can be used with standard manual Pipettes saving the labor time related to the Florisil® conditioning, column preparation, glassware cleaning and eliminating the risk of cross contamination due to the consumable-based workflow;

The workflow is easily integrated on the CTC PAL/GERSTEL MPS systems allowing the automation of additional sample pre-treatments like dilution, spiking or combining the sample cleanup with the GC injection.





REFERENCES

(1) ISO 9377-2:2000. Water quality – Determination of hydrocarbon oil index – Part 2: Method using solvent extraction and gas chromatography.

(2) Grob, K. (1987). On-column injection in capillary gas chromatography: Basic technique, retention gaps, solvent effects. Hüthig Verlag Heidelberg. ISBN 3-7785- 1551-9.

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