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High-throughput mineral oil determination in water samples by automated in-vial extraction, clean-up, and on-line GC-FID analysis

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Keywords

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Goal

The aim of this work is to demonstrate the high-throughput and the cost-saving benefits of a validated automated sample preparation workflow for routine analysis of mineral oil in water samples through in-vial liquid-liquid extraction (LLE) and FLORISIL® clean-up, as implemented in a public testing laboratory for environmental services.

Introduction

For many decades, the worldwide economy has been based on crude oil and crude oil-derived products, such as fuels, lubricants, and bitumen. During the extraction, processing, transportation, and utilization of crude oil and its derivatives, spills into the environment inevitably occur, contaminating water and soils and posing serious risk to human health. Within the several hundred organic chemical compounds that originate from crude oil, ranging from light gas to heavy fuel oil, mineral oil represents a fraction of mainly saturated hydrocarbons in the range from C10 to C40. Since mineral oil is widely used in many industrial applications and contained in many consumer products, its environmental impact requires high attention to monitor and keep under control the level of hydrocarbon contamination. Various regulated methods are available as reference for its determination in environmental samples, including ISO 16703:2004[1] for soil samples, EN ISO 14039:2004[2] for solid waste, EN ISO 9377-2:2000[3] for water samples, and ASTM D7678-11[4] using an infrared technique.

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The quantification of mineral oil hydrocarbons (also referred to Hydrocarbon Index or H53) in water, soil, and sediment samples represents a typical task for environmental testing laboratories, which often face a demanding sample load and tight deadlines to provide results to clients. In case of water samples, which are the focus of this work, the sample preparation involves liquid-liquid extraction followed by a clean-up step with FLORISIL to remove the co-extracted more polar components such as lipids. The purified extract can then be concentrated further or can be directly injected for GC-FID analysis.

For testing laboratories, the sample preparation is the typical bottleneck of the entire workflow, in terms of labor time, costs of reagents, and logistics. Implementing an automated workflow is desirable to increase unattended operations and relieve sample load, but the benefits are manifold. This work describes the use of the Thermo Scientific™ TriPlus™ RSH robotic autosampler for a fully automated sample preparation workflow with in-vial liquid-liquid extraction, clean-up, and on-line GC injection for the analysis of total hydrocarbon index in water samples according to the method EN ISO 9377-2.[3]

Experimental

Instrument set up

A TriPlus RSH autosampler, configured and validated to automate the liquid-liquid extraction and clean-up[5] (Figure 1 and Table 1) of water samples (ground and wastewater), was mounted on a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph configured with a Thermo Scientific™ iConnect™ Programmable Temperature Vaporizer (PTV) injector and an iConnect FID detector.

Table 1. List of the tools included in the TriPlus RSH configuration

| TriPlus RSH tools	
1x	Automatic tool change
1x	Liquid syringe tool for 57 mm syringe needle (1 mL syringe volume) (LS3)
2x	Liquid syringe tool for 57 mm syringe needle (100 µL syringe volume) (LS1 and LS2)
1x	Standard washing station
1x	Fast washing station
1x	Vortex mixer
1x	Tray holder with 60 positions for 10 or 20 mL vials (R60)
1x	Tray holder with one 15 position tray for 10 or 20 mL vials (VT15) and two 54 position trays for 2 mL vials (VT54)
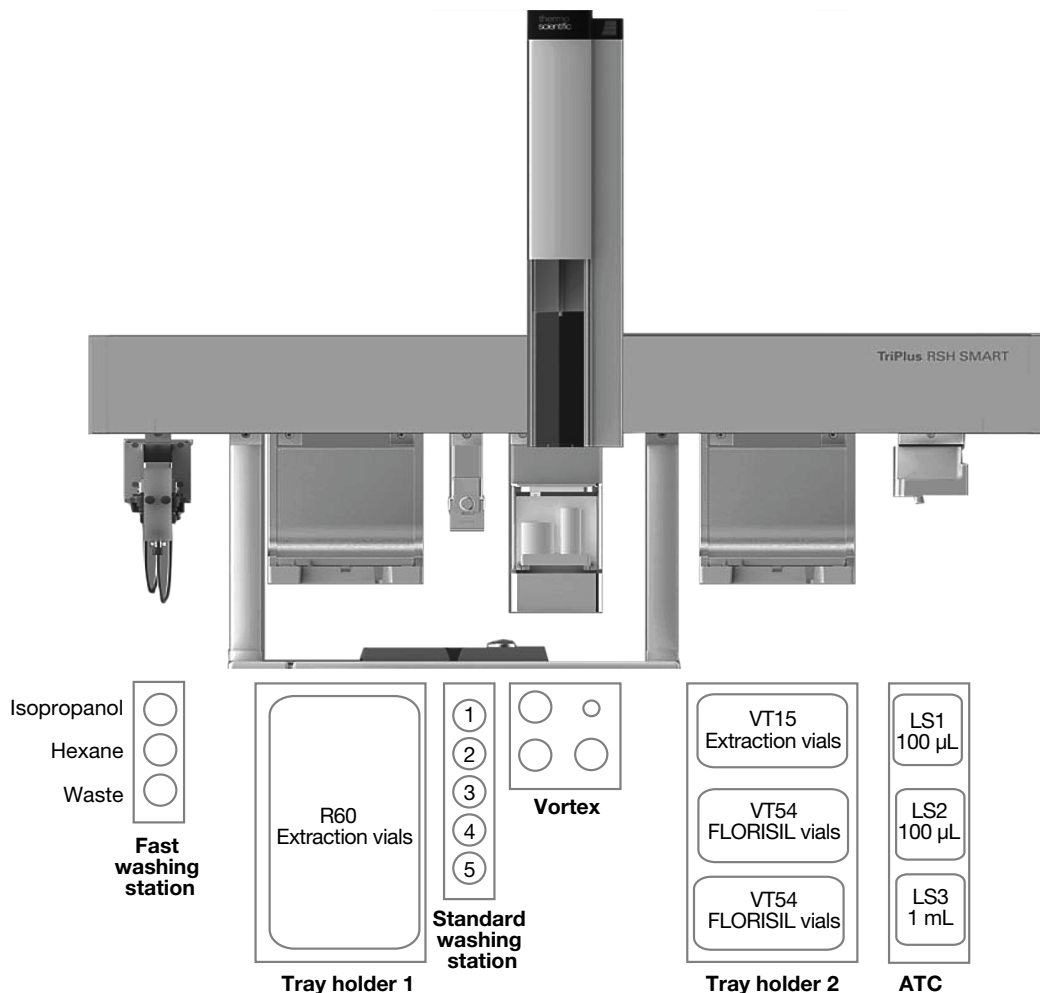
Figure 1. TriPlus RSH autosampler configuration for automated LLE of mineral oil in water samples, including clean-up on FLORISIL and on-line injection (also applicable to the TriPlus RSH SMART autosampler)

2

The use of the PTV injector allows for large volume injection to increase the method sensitivity to µg/L (ppb) levels with no need for a re-concentration step of the extract. The full instrument method parameters are listed in Tables 4 and 5.

Solvents and reagents

- Mineral oil standard mixture Type A and B for EN 14039 and ISO 16703

- Hexane (Honeywell, #1.07288.1000)

- Isopropanol (Honeywell, #1.00837.1000)

- FLORISIL

- $MgSO_4$

- HCl

- NH_4Cl

- *n*-decane (C10)

- *n*-tetracontane (C40)

- Florida Mix (C10-C40 all even)

Integration markers and calibration standards

The assessment of mineral oil content does not require the separation of each hydrocarbon, but quantitation is done by integrating a total peak area between the markers *n*-decane (C10) and *n*-tetracontane (C40), which are automatically added to each sample before the extraction and clean-up steps. A C10/C40 standard at a concentration of 50 mg/L in *n*-hexane is placed in position "standard wash 1" of the autosampler, and 60 µL are added to each sample.

Mineral oil calibration standards at six levels of concentration in the range of 0.1 to 10.0 mg/L (Table 2) were prepared to check the response linearity of the entire workflow during the method performance validation process. Two concentration levels at 0.5 mg/L and 5.0 mg/L were placed in tray holder 1 of the autosampler and used for a linearity check during daily operations.

To standardize the matrix between calibration standards and real samples, reduce emulsions, and promote the transit to the organic phase, 0.5 mL of a saturated salt solution ($MgSO_4$/NH_4Cl/HCl) was added to all samples and standards.

Mineral oil calibration standards in extraction solvent were prepared following the dilution scheme in Table 3, covering a total of six concentration levels. This calibration curve was used to check the recovery.

Table 2. Mineral oil calibration standards for linearity assessment in the range 0.1–10.0 mg/L

Level	Mineral oil in water (mg/L)
1	0.1
2	0.5
3	1.0
4	2.5
5	5.0
6	10.0

Table 3. Dilution scheme for six levels calibration curve of mineral oil in *n*-hexane

Mineral oil standard mixture (MR) Type A and B for EN14039 and ISO16703 (8,000 mg/L)			
Primary solutions			
Standard	µL (MR)	µL (hexane)	mg/L
std0	0	1,000	0
std1	10	990	80
std2	50	950	400
std3	100	900	800
std4	250	750	2,000
std5	500	500	4,000
std6	1,000	0	8,000
Working solutions			
Standard	µL (Primary solution)	mL (water)	mg/L
s0	12.5 (std0)	10	0.0
s1	12.5 (std1)	10	0.1
s2	12.5 (std2)	10	0.5
s3	12.5 (std3)	10	1.0
s4	12.5 (std4)	10	2.5
s5	12.5 (std5)	10	5.0
s6	12.5 (std6)	10	10.0

Automated workflow

The workflow, as reported in Figure 2, was optimized to run wastewater samples, routinely tested in the Veritas laboratory. An aliquot of 10 mL of a water sample was manually added to a 20 mL vial, and in-vial solvent extraction was performed with the automatic addition of the internal standard and 2 mL of *n*-hexane, followed by a clean-up step of the extract with FLORISIL to ensure the correct quantification of mineral oil content only. The robotic system worked together with the intelligent sequence capabilities of the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) to perform the clean-up step only to positive samples, accelerating the sample-throughput, according to the data structure shown in Figure 3. Both the extraction step and the clean-up step were programmed with on-line injection into the GC system.

The automatic tool change (ATC) station available on the autosampler was key to automatically selecting up to three dedicated syringes of different volumes for ISTD addition, dispensing reagents, and picking up the supernatant for injection into the analytical system. As shown in Figure 1, dedicated trays were used to accommodate the 20 mL vials with samples for the extraction step, while separate trays were used for hosting the same number of 2 mL vials in case of positive samples for the clean-up step.
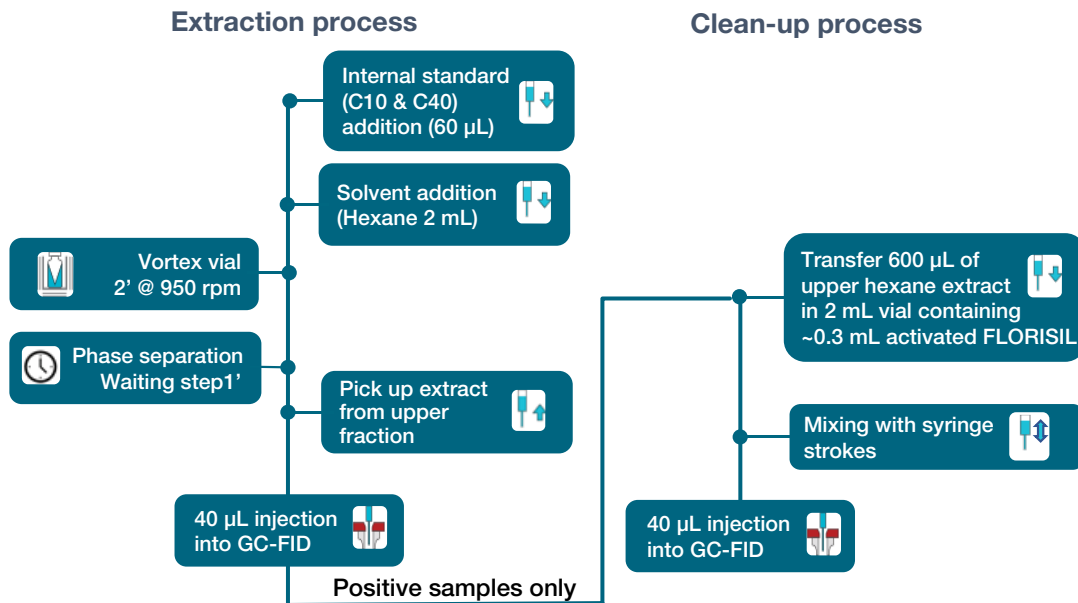
Extraction process · Clean-up process

Figure 2. Schematic of the automated workflow including clean-up on FLORISIL for positive samples and on-line injection
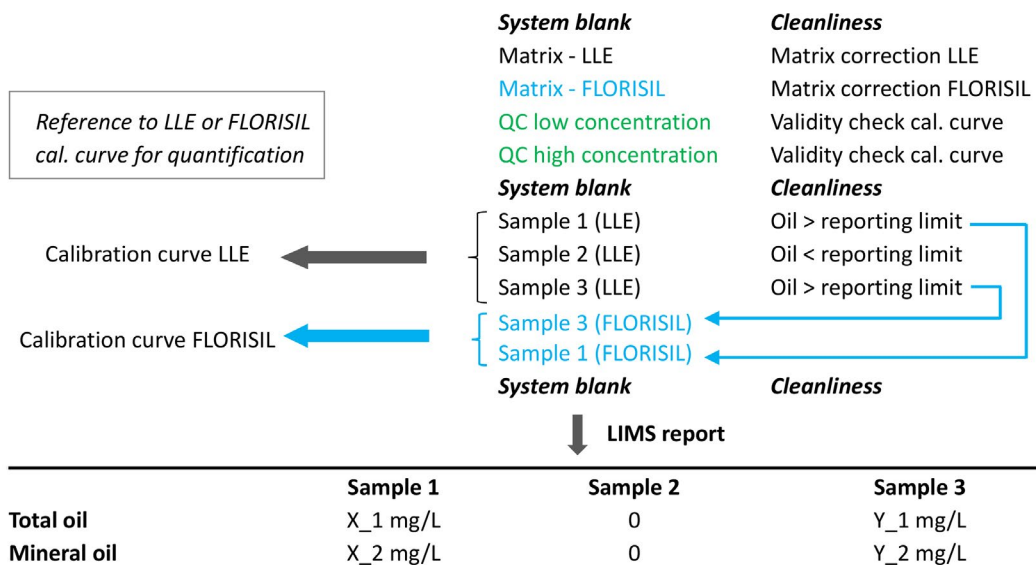

Figure 3. Schematic overview of the data structure and workflow, using the Chromeleon CDS Intelligent sequence. Samples highlighted in light blue are automatically generated in the sequence according to the System Suitability Test (SST) results.

Table 4. Autosampler method parameters

TriPlus RSH autosampler	
Syringes	100 μL gas tight syringe (P/N 365H2141) 1mL gas tight syringe (P/N 365K2811)
Sample volume	10 mL of water
Vial type for sample extraction	20 mL headspace vials (crimp top vial, P/N 6ACV20-1R, with magnetic caps and silicone/Teflon septum, P/N 6AMCC20ST3T)
Vial type for clean-up	2 mL screw cap clear glass vials with silicone/Teflon septum (P/N 6AK92W)
Extraction solvent and volume	Hexane, 2 mL
ISTD volume	60 μL (C10 and C40 mix)
Extract volume for clean-up	600 μL
Vortex mixing for extraction	950 rpm, 2 min
Syringe strokes for cleanup mixing	3
Syringe washing	10 washes (for total oil extract injection), 13 washes (for purified extract injection), with hexane and isopropanol (total volume < 0.5 mL/sample)
Injection volume	40 μL
Injection speed	12 μL/s

The vortex mixer was used to achieve an effective mixing of both the aqueous and the organic phase during the extraction step, while the sample mixing during the clean-up procedure was achieved through syringe strokes. This approach was preferred over the use of the vortex since it accelerates the workflow still providing the required mixing efficiency.

The cycle time for the automated LLE was 13 min. The Prep Ahead functionality of the TriPlus RSH autosampler allowed the preparation of a sample during the chromatographic run of the previous one, so that the preparation time was impacted only for the first sample of the sequence.

As noted in the workflow schematic (Figure 2), the automated in-vial extraction allowed the scaling down of the sample and solvent volumes to just 2 mL of organic solvent added to a 10 mL aliquot of a water sample, reducing waste and saving associated costs for extraction solvents.

The only manual steps required were pipetting 10 mL of water sample into 20 mL vials, pipetting the salt solution, preparing 2 mL vials with about 0.3 mL of FLORISIL, and placing the prepared vials on the TriPlus RSH autosampler vial trays.

Data acquisition, processing, and reporting

Chromeleon CDS was the heart of the automated workflow. In addition to handling the intelligent sequence workflow as reported in Figure 3, Chromeleon CDS was used for instrument

Table 5. Gas chromatograph method parameters

TRACE 1310 GC	
Injector type	iConnect PTV
Liner	Large Volume PTV Liner with glass wool (Interscience P/N 890571213)
Injector temperature	35 °C to 350 °C
Injection mode	Large Volume Solvent Split (18:1 Split ratio)
Column	Thermo Scientific™ TraceGOLD™ TG-5HT GC columns offer extended operation up to 400 °C, ideal for high temperature GC applications, 15 m, 0.25 mm i.d., 0.1 μm thickness (P/N 26095-0350)
Carrier gas	Helium @ 6 mL/min constant flow
Oven temperature	45 °C (4 min) to 330 at 60 °C/min
Run time	16 min
Detector	iConnect FID
FID hydrogen flow	35 mL/min
FID air flow	350 mL/min
FID make up (nitrogen) flow	40 mL/min

control, data visualization, data processing, and reporting the final data back to the LIMS system. An eWorkflow™ was used to create a sequence in a predefined form (Table 6). The data were automatically evaluated using four different system suitability tests (SST), and a final sequence was generated according to the number of samples. The first in-vial extracted matrix analysis (line two in the e-Workflow) generated a FLORISIL clean-up of the same extract as second analysis. Both QC analysis were evaluated using an SST, which stops the sequence if the value of the oil falls outside predefined boundaries. The last SST was used to evaluate samples. A FLORISIL clean-up analysis was added if the sample's oil content was higher than the reporting limit. After completion of the sequence, a LIMS report was created.

Table 6. Sequence generated using the e-Workflow for n samples

Injection name	Instrument method	System Suitability Test (SST)
System blank	Matrix LLE	
Matrix	Matrix LLE	SST - FLORISIL clean-up matrix
QC low	LLE	SST - Peak amount limits QC 3
QC high	LLE	SST - Peak amount limits QC 6
System blank	Matrix LLE	
Sample 1	LLE	SST - FLORISIL Clean-up
Sample 2	LLE	SST - FLORISIL Clean-up
...	LLE	SST - FLORISIL Clean-up
Sample n	LLE	SST - FLORISIL Clean-up
System blank	Matrix LLE	

Results and discussion

System blank and matrix runs

The chromatogram of a blank run with no injection is indicative of the cleanliness of the system and is key for the correct integration of the samples chromatogram. Figure 4 reports a typical chromatogram of a system blank run.
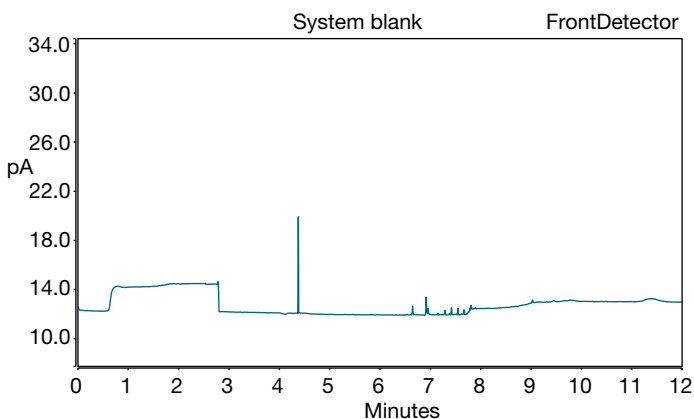

Figure 4. Typical chromatogram of a system blank run

To reproduce the matrix, a clean water sample was analyzed using the automated LLE with the exception that 60 µL hexane was added instead of 60 µL internal standard. Subsequently, a FLORISIL clean-up was performed. Both chromatograms were used to compensate for background contributions during LLE and FLORISIL clean-up quantification. Figures 5 and 6 show typical matrix chromatograms for the LLE and FLORISIL clean-up, respectively.

The contamination present in the clean water matrix was due to the hexane used. High purity hexane solvent (suitable for large volume injection) is recommended to minimize the contamination. The contamination has a negative effect on the limit of quantification (LOQ), but not on the quantitative results as the matrix chromatogram is subtracted from all sample analyses prior to quantification. The impact of this contamination was evaluated depending on the sample matrix and the required legal limit of detection.
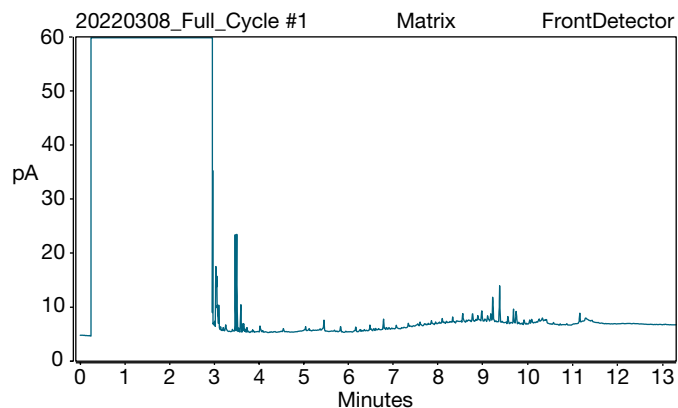

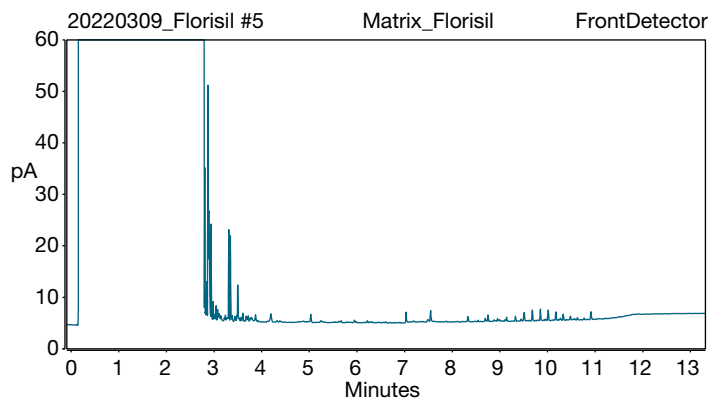Figure 5. Typical chromatogram of an LLE of a clean water extract

Figure 6. Typical chromatogram of a FLORISIL clean-up of a clean water extract

System performance

The performance of the system in terms of discrimination during the PTV injection was verified by injecting a Florida Mix standard (all even carbon numbers between C10 and C40) at 50 mg/L. To be compliant with the ISO standard, the ratios C10/C20 and C40/C20 must be greater than 80%. Figure 7 reports a typical Florida Mix chromatogram obtained with the PTV large volume injection, along with the ratio values to C20, all > 90%.

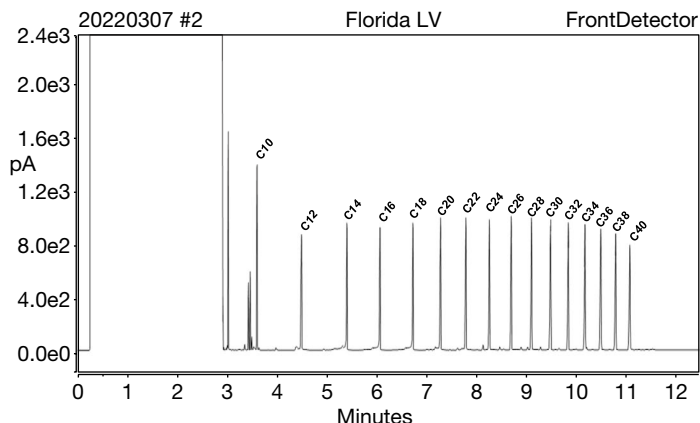|	Ratio to C20 (%)		Ratio to C20 (%)
C10	92	C26	102
C12	95	C28	104
C14	94	C30	102
C16	96	C32	100
C18	100	C34	101
C20	100	C36	99
C22	99	C38	97
C24	102	C40	100

Figure 7. Florida Mix chromatogram showing no injection discrimination in compliance with the ISO 9377-2 method

FLORISIL clean-up

FLORISIL clean-up is necessary to discriminate between mineral oil (apolar hydrocarbons) and non-mineral oil (polar fraction) contained in water samples, co-eluting with the sought hydrocarbons between C20 and C40, to ultimately avoid false positive results.

Figure 8 shows typical chromatograms of a sample containing both mineral and non-mineral oils after the LLE, before and after clean-up, with more polar compounds clearly removed.
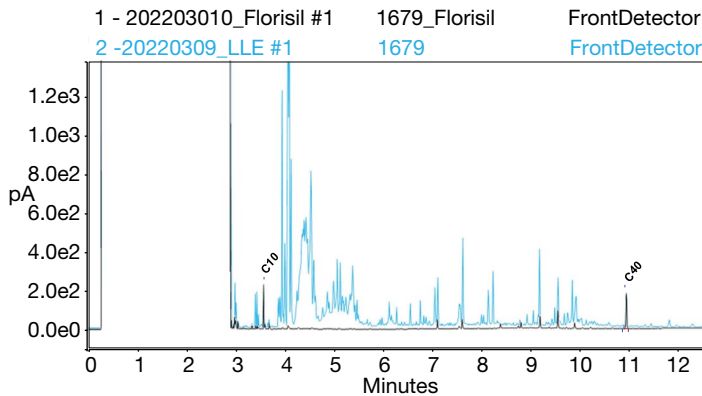

Figure 8. Typical chromatogram obtained after automated LLE of total hydrocarbons (blue trace), followed by a FLORISIL clean-up analysis (black trace) for the determination of the mineral oil content
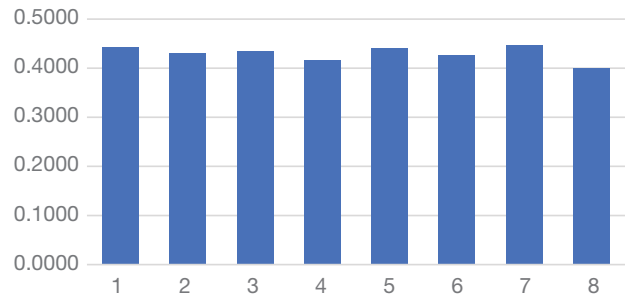
Repeatability and robustness

The retention time repeatability of the markers C10 and C40 is particularly critical to assess a reliable automated integration of the total peak area. A sequence consisting of 106 samples was analyzed throughout the automated extraction and clean-up workflow to assess the precision of the IS retention time. C10 shows a slightly higher RT variation then C40, since it is impacted by the solvent elution (Table 7).

The repeatability of the total area integrated between C10 and C40 was evaluated over a sequence of eight water samples spiked at low (0.4 mg/L) and high (5.0 mg/L) levels, and processed throughout the extraction and clean-up workflow, showing an RSD < 4% (Figure 9).

Table 7. Summary of IS retention time precision

Statistic	C10 Ret. time (min)	C40 Ret. time (min)
#	106	106
Minimum	3.588	9.050
Maximum	3.910	9.125
Mean	3.829	9.076
Std Dev	0.059	0.017
RSD (%)	1.6	0.2

Water + spike 0.4 mg/L	0.4423	mg/L
	0.4312	
	0.4354	
	0.4160	
	0.4419	
	0.4278	
	0.4469	
	0.3999	
Average	0.4302	mg/L
Std Dev	0.0157	
RSD (%)	3.6	

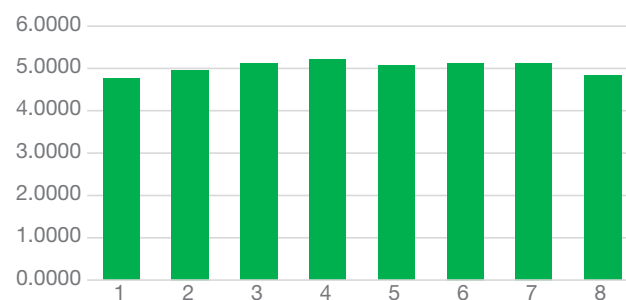| Water + spike 5.0 mg/L	4.7648	mg/L
	4.9613	
	5.1123	
	5.2093	
	5.0618	
	5.1257	
	5.1268	
	4.8342	
Average	5.0245	mg/L
Std Dev	0.1566	
RSD (%)	3.1	

Figure 9. Repeatability of spiked water samples at low (0.4 mg/L) and high (5.0 mg/L) concentration levels

Response linearity and recovery

A linear response for the mineral oil standards in *n*-hexane covering a concentration range of 0.1–10 mg/L and prepared as described above is shown in Figure 10, as external and IS calibration, reporting in both cases an $R^2 = 0.999$.

The recovery was verified by spiking clean water samples at the same concentration levels and analyzed throughout the automated extraction and clean-up workflow, obtaining a recovery between 96% and 104%, as reported in Table 8.
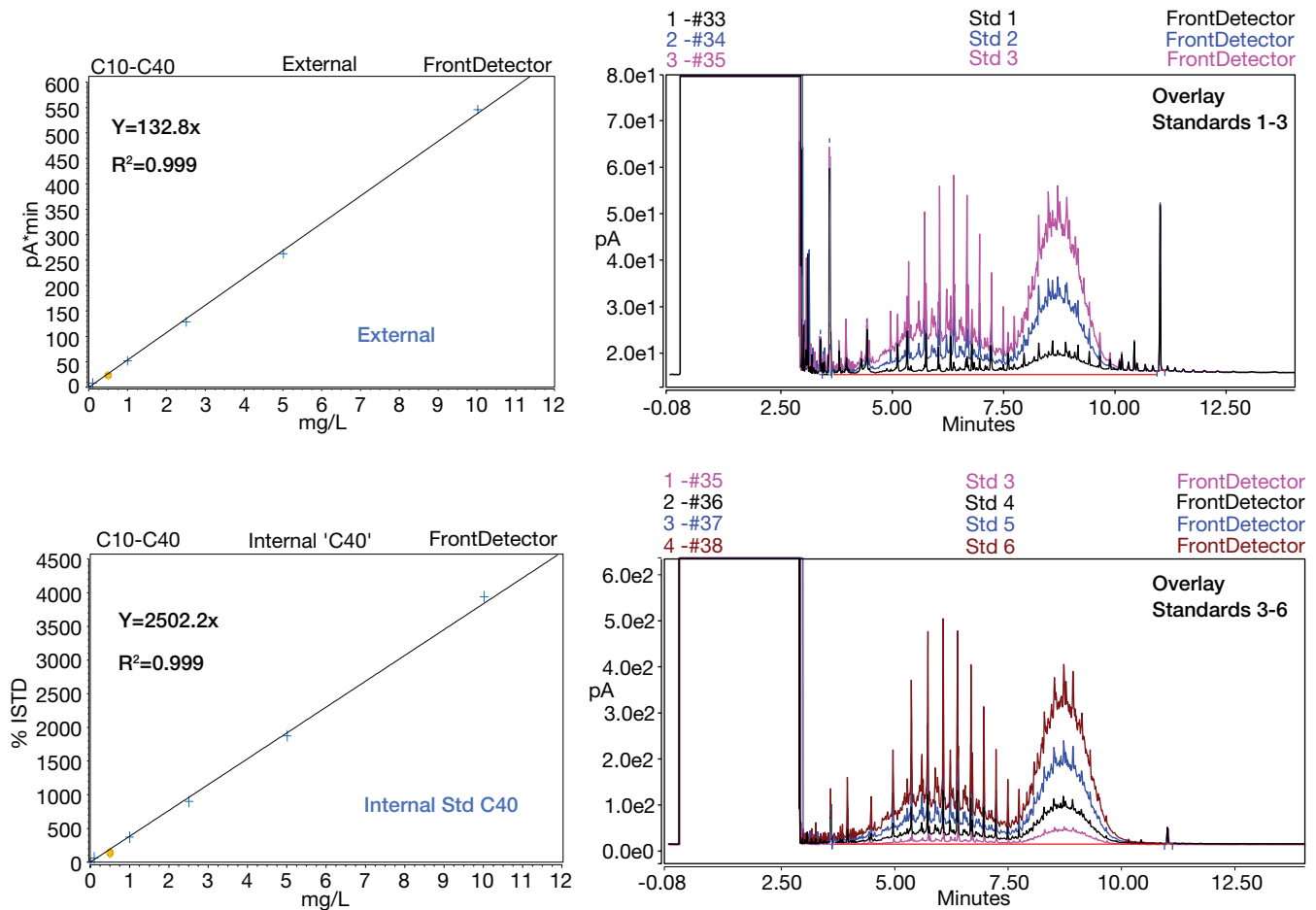
Figure 10. Linearity plot for mineral oil standards in n-hexane over six levels of concentration in the range 0.1–10 mg/L

Table 8. Recovery of mineral oil after the automated extraction and clean-up workflow

	Theoretical amount (mg/L)	Amount (mg/L)	Recovery (%)
Recovery standard 1	0.1	0.110	110
Recovery standard 2	0.5	0.502	100
Recovery standard 3	1.0	1.038	104
Recovery standard 4	2.5	2.402	96
Recovery standard 5	5.0	5.109	102
Recovery standard 6	10.0	9.552	96

The calibration curve for quantitative determination as used for wastewater samples was obtained by spiking clean water samples from 0.5 mg/L (standard #2) to 10.0 mg/L with five concentration levels. A calibration curve was prepared for the LLE step only and another one covering LLE and FLORISIL clean-up, as shown in Figure 11. A summary of the linear regression residuals is reported in Table 9.

LOD and LOQ
The required limits of detection and quantification depend on the sample matrix. Typically, groundwater samples require lower limits (<0.1 mg/L). For wastewater samples, to be tested at the inlet and outlet of the purification plant, the limits are higher (0.2–0.5 mg/L). To push the limit of detection as required for groundwater samples, the amount of extraction solvent can be reduced to 1 mL. For wastewater samples, more solvent helps to reduce emulsion effects, maintaining the required sensitivity.

Six clean water samples, spiked at 0.1 mg/L, were analyzed using the automated LLE procedure using 1 mL of extraction solvent. The limit of detection was calculated as 3 times the standard deviation, while the LOQ as 2 times the LOD (Figure 12).
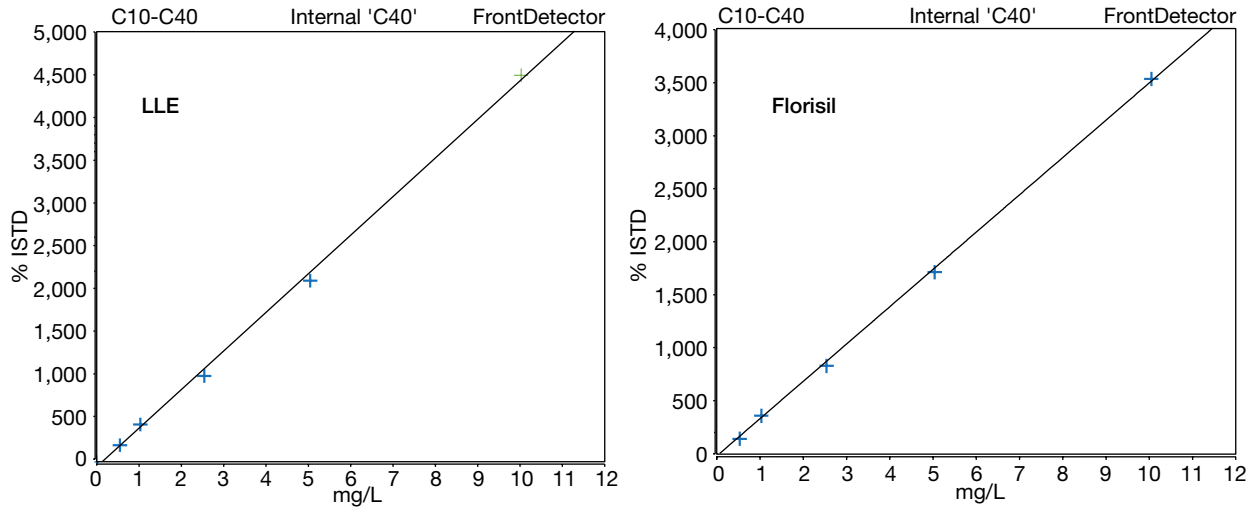
Figure 11. Calibration curve for the automated LLE (left) and FLORISIL clean-up (right) in the concentration rage 0.5–10.0 mg/L

Table 9. Calibration curve residuals %

Component	0.50 mg/L	1.00 mg/L	2.51 mg/L	5.01 mg/L	10.02 mg/L
Mineral oil (LLE)	2.36	6.30	-7.09	-5.01	1.63
Mineral oil (FLORISIL)	-2.90	2.40	-4.12	-2.19	0.79

	Amount (mg/L)
0.1_Rep1	0.098
0.1_Rep2	0.098
0.1_Rep3	0.108
0.1_Rep4	0.110
0.1_Rep5	0.111
0.1_Rep6	0.109
Average	0.106
Std Dev	0.006
RSD (%)	5.6
LOD	0.018
LOQ	0.036

Figure 12. Overlay of six clean water samples spiked at 0.1 mg/L used for LOD and LOQ calculation

Quality control

Quality control standards were analyzed to verify the validity of the calibration curve. If the returned amount for the QC fell well within predefined boundaries, the next line in the sequence was analyzed. When the QC failed, the whole sequence stopped and no sample was analyzed unnecessarily, resulting in less solvent and consumable waste. The "QC low" boundaries were set between 50% and 150% of the standard at 0.50 mg/L; the "QC high" boundaries were set between 70% and 130% of standard at 5.00 mg/L. Figures 13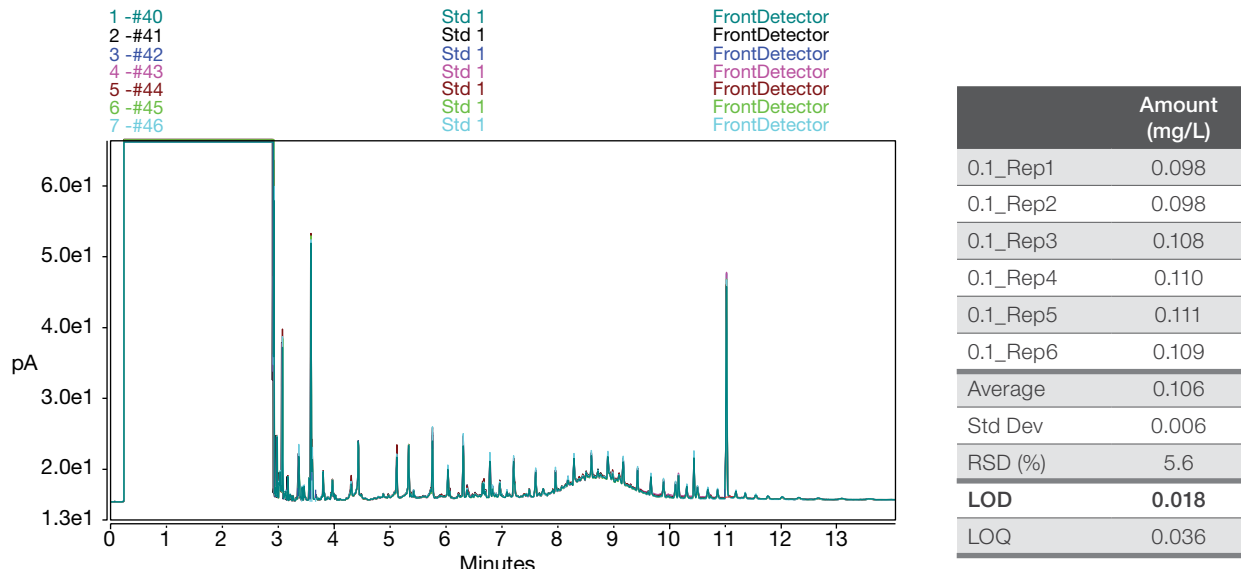 and 14 show typical chromatograms obtained for QC low and high, respectively. Note that the set boundaries are dynamic and linked to the values of the calibration solutions in the processing method.
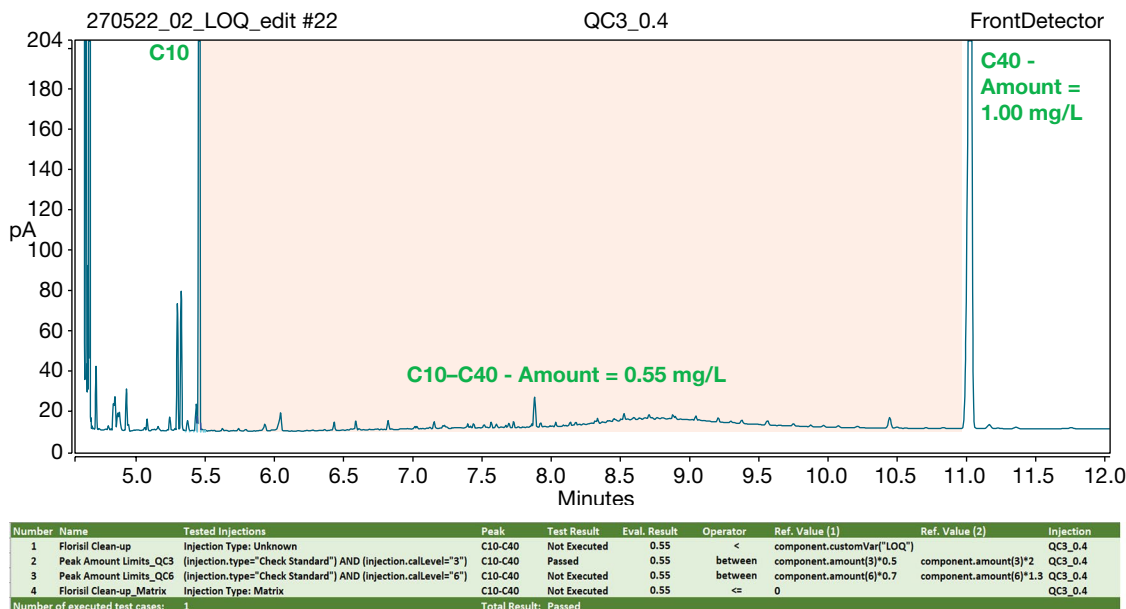

Number	Name	Tested Injections	Peak	Test Result	Eval. Result	Operator	Ref. Value (1)	Ref. Value (2)	Injection
1	Florisil Clean-up	Injection Type: Unknown	C10-C40	Not Executed	0.55	<	component.customVar("LOQ")		QC3_0.4
2	Peak Amount Limits_QC3	(injection.type="Check Standard") AND (injection.calLevel="3")	C10-C40	Passed	0.55	between	component.amount(3)*0.5	component.amount(3)*2	QC3_0.4
3	Peak Amount Limits_QC6	(injection.type="Check Standard") AND (injection.calLevel="6")	C10-C40	Not Executed	0.55	between	component.amount(6)*0.7	component.amount(6)*1.3	QC3_0.4
4	Florisil Clean-up_Matrix	Injection Type: Matrix	C10-C40	Not Executed	0.55	<=	0		QC3_0.4
Number of executed test cases:	1			Total Result: Passed					

Figure 13. Example of chromatogram and SST result for QC low

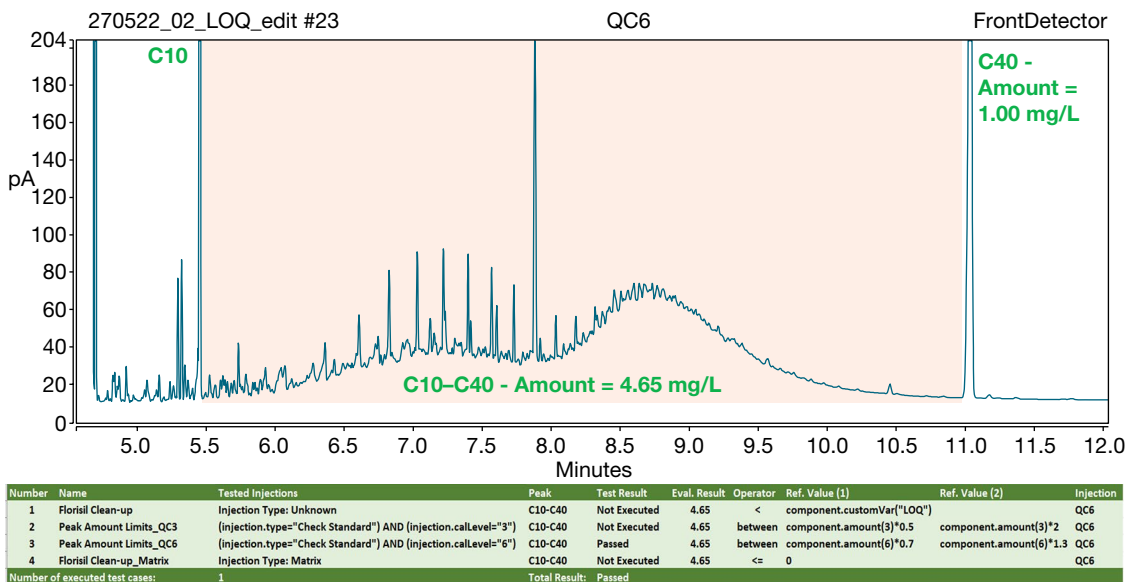| Number	Name	Tested Injections	Peak	Test Result	Eval. Result	Operator	Ref. Value (1)	Ref. Value (2)	Injection
1	Florisil Clean-up	Injection Type: Unknown	C10-C40	Not Executed	4.65	<	component.customVar("LOQ")		QC6
2	Peak Amount Limits_QC3	(injection.type="Check Standard") AND (injection.calLevel="3")	C10-C40	Not Executed	4.65	between	component.amount(3)*0.5	component.amount(3)*2	QC6
3	Peak Amount Limits_QC6	(injection.type="Check Standard") AND (injection.calLevel="6")	C10-C40	Passed	4.65	between	component.amount(6)*0.7	component.amount(6)*1.3	QC6
4	Florisil Clean-up_Matrix	Injection Type: Matrix	C10-C40	Not Executed	4.65	<=	0		QC6
Number of executed test cases:	1			Total Result: Passed					

Figure 14. Example of chromatogram and SST result for QC high

Table 10. Direct comparison between manual and automated workflow

	Manual procedure	Automated procedure
Sample volume	100 to 1,000 mL	10 mL
Solvent volume	100 to 250 mL hexane per sample (on average 150 mL/sample)	2 mL hexane per sample (plus 2 mL hexane and 0.5 mL 2-propanol for washing)
Solvent consumption	300 L hexane per year	8 L hexane + 1 L 2-propanol per year
Total cycle time	About 46 min/sample (ca. 30 min sample prep + 16 min GC run)	29 min for the first sample and 16 min/sample for the following ones (13 min sample prep overlapped + 16 min GC run)
Samples throughput	10 samples/day (one person)	40 to 75 samples/day, (24/7 operation)
Operational cost	3000 €	80 €

Note: Assuming the analysis of 2,000 samples/year, and a solvent price of 10 Euro/liter

Benefits of the automated workflow

Implementing an automated system for sample preparation and on-line injection is highly beneficial for environmental testing laboratories facing high sample loads. Thanks to the Prep Ahead function, the TriPlus RSH autosampler optimizes the overall cycle time, starting the sample prep operations during the chromatographic run of the previous sample. The number of samples per day can be increased more than six times thanks to unattended 24/7 operations, with much higher throughput and labor time saving.

Another important benefit is the reduction of sample and solvent volumes, leading to a significantly lower consumption of solvent per year. The reduction described in this application note helped to reduce the overall consumption from about 300 L/yr to only 9 L/yr, which means a significant reduction of cost, including sample storage and transportation costs.

Table 10 reports a direct comparison of key indicators for a classical manual sample preparation procedure and the automated workflow, showing an overall 97% solvent cost saving for the laboratory.

Conclusions

A fully automated sample preparation workflow with on-line large volume GC injection using the TriPlus RSH robotic autosampler is available for the analysis of the total hydrocarbon index in surface and wastewater samples. The workflow includes an in-vial extraction for the determination of the total oil followed by a FLORISIL clean-up only for positive samples for the quantitative determination of the mineral oil content. The system offers several benefits to relieve high sample workload:

- The method is fast (13 min LLE cycle time), fully optimized thanks to the prep ahead functionality of the TriPlus RSH autosampler.

- The system is fully controlled by Chromeleon CDS as single software. Thanks to the Intelligent Sequence and the eWorkflow functionalities, it offers a complete automated management of the samples, automated reporting, and transfer to LIMS.

- This system is currently operating 24/7 at a public environmental service provider, greatly increasing the achievable sample throughput, with significant labor and cost saving.

References
1. International Standard EN ISO 16703:11, Soil quality. Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography.
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4. ASTM D7678-11, Standard Test Method for Total Petroleum Hydrocarbons (TPH) in Water and Wastewater with Solvent Extraction using Mid-IR Laser Spectroscopy.
5. Automated environmental solutions | SampleQ
6. Thermo Scientific eb000396 - Guide to automated sample preparation for GC and GC-MS

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