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IMAROS 2

Deliverable D3.1

Summary report of WP3 Characterisation and Impacts

DELIVERABLE D3.1

SUMMARY REPORT OF WP3
CHARACTERISATION AND IMPACTS

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FINAL REPORT

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DISCLAIMER

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EXECUTIVE SUMMARY

In the frame of the IMAROS 2 project (2024 – 2025), the work package 3 (WP3) was dedicated to the characterisation of the Low Sulphur Fuel Oils collected by the project's partners, at the laboratory and pilot scales, in order to evaluate the oil behaviours when released in sea and fresh waters and get insights for operational guidelines. The second objective was to use the experimental data to develop weathering models. The results obtained from the modelling study are presented in a separate Deliverable, D3.3.

Nineteen samples were obtained, five of them were ULSFOs and fourteen VLSFOs. Fresh oils characterisation consisted in physical-chemical characterisation at 15°C (viscosity, density, pour point, flash point, waxes and asphaltenes contents, evaporation rate, full detailed chemical characterisation and fingerprinting). A comparison with the samples obtained in the frame of the first IMAROS project (2020-2022) allowed the analysis of the market evolution since 2020. Three of the samples (two VLSFOs and one ULSFO), selected by the project partners and acquired in higher volumes, were studied at the pilot scale at 5°C and 25°C in order to assess their behaviour and weathering in sea and fresh water. Oil spill responses were investigated (chemical dispersibility, adhesion on oleophilic plate for mechanical recovery, biodegradability rates).

Regarding the characterisation of the fresh oils, the main results are the following:

- No major changes were observed between 2020 and 2024 in terms of physical-chemical characterisation.
- Flash points of all the samples analysed were well above 60°C, excluding safety issues related to flammability.
- Evaporation was limited, below 10% for the majority of the samples, leading to persistent products.
- Viscosity highly varied between samples and could reach more than 300 000 mPa.s at 15°C. At ambient temperature (15 – 20°C), half of the oils had a semi solid aspect. ULSFOs are expected to exhibit higher viscosity than VLSFOs at sea water temperatures.
- Pour point and waxes' contents varied a lot, with the highest values observed for ULSFOs. For those products, a pour point higher than 30°C could be measured, with waxes content above 25%.

In terms of behaviour and weathering, based on the three oils tested at the pilot scale at 5°C, slicks of LSFOs are expected either to fragment in small diffuse granular tarballs or remain very compact with slow kinetics of emulsification. At 25°C, the three oils exhibit a much more traditional behaviour, with more fluid slicks emulsifying more rapidly.

- When weathered, viscosity could reach up to 350 000 mPa at 5°C and 50 000 mPa.s at 25°C. At 5°C, oil recovery and pumping operations could thus be challenging.
- Maximum density was 1.00. The effect of water salinity (seawater versus freshwater) on the oil behaviour was not clearly observed. In case of turbulences (created in those experiments by the corners of the flume tank), the slicks could be dragged down below the surface. This phenomenon seemed more pronounced during the experiments performed with freshwater. In

real environment, the increase in density is not expected to cause the oil slicks to submerge below the water surface for seawater conditions. However, the buoyancy of some oils could be questioned close to estuaries, characterised by a lower salinity and a high mineral load which could increase the emulsion density.

- Water content was limited at 5°C and could be as low as 30%. At the beginning of the emulsification process, water is more likely trapped in pocket than forming a real oil-in-water emulsion. At 25°C, emulsification processes were much more traditional and water content reached between 80 and 90% after 24 – 48 hours. Emulsions were all very stable.

In terms of response options, based on the three LSFOs tested, characterised as persistent and not flammable, biodegradation in the environment will be very limited. The use of chemical dispersants, which was limited in the frame of the first project with tests performed at 15°C, seems not recommended on the three products tested in this project. Tests performed at 5°C and 25°C, on the fresh and moderately weathered LSFOs, did not conclude to a dispersion of the oil slicks. Recovery seems thus the response option to be selected. Tests on oleophilic plates show a good oil adhesion at the beginning of the weathering trials, slowly reducing with time. Oleophilic skimmers could thus be selected according to the expected weathering of the oil, in relationship with the device technology. For some very highly viscous oils, the viscosity could be compatible with oil trawling as complementary technique for recovery. However, the rapid tests performed during the weathering experiments in the flume tank did not mimic real recovery tests. Several LSFOs samples present complex visco-elastic behaviour that challenges mechanical recovery with skimmers such as the so-called "short" oil behaviour. Test in laboratory and in flume tank suggest that short oil occurs when the elastic behaviour dominates the viscous behaviour. These characteristics can be quantified with specific rheometers. As a proxy, our tests suggests that, for the tested LSFOs, there exists a correlation between the visco-elastic properties of the oil sample, its wax content and its pour-point. Specific trials dedicated to mechanical recovery are the core of the WP4 of the project. Results are presented in the Deliverable D4.2. The three large samples used in this WP are the one also tested in the WP4 and also in the WP5 dedicated to shoreline responses.

As during the first project, those data highlight the fact that LSFOs will behave and weather differently in case of spillage at sea. Additionally, obtention of physical-chemical data may not be sufficient to understand the oil behaviour at sea. Pour point and viscosity often known at 50°C may not inform on the fluidity and the elasticity of the oil at ambient temperatures. Response options will subsequently have to be adapted, depending on the oil properties.

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1 CONTEXT AND OBJECTIVES

The objective of the Work Package 3 (WP3) of the IMAROS 2 project is to characterise the Low Sulphur Fuel Oils (LSFOs) collected by the project's partners, at the laboratory and pilot scales, and to use the dataset obtained to improve weathering models. Table 1 details the different tasks part of the work package.

Table 1 *Details of the tasks part of the WP3 of the project*

Tasks	Partner involved / Conditions	Objectives
3.1 - In-depth characterisation	Cedre, Laboratory scale, 15°C	• Characterise the samples • Study the market evolution since 2020 • Focus on wax contents determination
3.2 – Oil weathering	Cedre, Pilot scale, 5°C, seawater and freshwater	• Give insight into response options thanks to information obtained on the weathered oils
3.3 – Behaviour in Mediterranean areas	Cedre, Pilot scale, 25°C, seawater	• Study the oil weathering at 25°C as Mediterranean became a SECA area on 01/05/25
3.4 - Biodegradability	Cedre, ambient temperature	• Know if LSFOs are suitable for bioremediation operation in case of lightly oiled sediments
3.5 - Identification	RBINS	• Analyse the samples following the recommendations of the CEN Technical Report • Enter the data on the EU- COSIWEB database
3.6 - Modelling	RBINS, RWS	• Use the data obtained in the different tasks to compare the OSERIT and OILMAP models. • Import the data in ADIOS database • Develop a visco-elasto-plastic model *Note: Results from this task are presented in a separate Deliverable (D3.3)*

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2 MATERIALS AND METHODS

2.1 Samples

Nineteen samples were obtained in the frame of this project. Sixteen of them are considered as “small samples” as the volume requested was 5 litres. Ten cubic meters of the three “large samples” (IM-27, IM-28 and IM-29) were purchased. Samples were coming from nine different oil deliverers or refineries across Europe. Five samples were ULSFOs and fourteen VLSFOs. Samples were labelled from IM-20 to IM-40. All those samples were sent to Cedre for laboratory and pilot scale testing. Table 2 details the nature and the origin of the collected samples.

Table 2 *Labelling, nature and country of origin of the 19 LSFOs analysed*

Samples	Nature	Origin	Comments
IM-20	VLSFO	Stena, Sweden	Small sample, for Task 3.1
IM-21	ULSFO	Stena, Sweden	Small sample, for Task 3.1
IM-22	VLSFO	BunkerOne, Sweden	Small sample, for Task 3.1
IM-23	ULSFO	BunkerOne, Sweden	Small sample, for Task 3.1
IM-24	VLSFO	Preem AB, Finland	Small sample, for Task 3.1
IM-25	VLSFO	Neste, Finland	Small sample, for Task 3.1
IM-27	VLSFO	Bunker One, Denmark	Large sample, for WP3, WP4 and WP5
IM-28	VLSFO	ZETA Energy Limited, Malta	Large sample, for WP3, WP4 and WP5
IM-29	ULSFO	Stena Oil, Sweden	Large sample, for WP3, WP4 and WP5
IM-30	VLSFO	FincoEnergies, Netherlands	Small sample, for Task 3.1
IM-31	VLSFO	FincoEnergies, Netherlands	Small sample, for Task 3.1
IM-32	ULSFO	FincoEnergies, Netherlands	Small sample, for Task 3.1
IM-33	VLSFO	FincoEnergies, Netherlands	Small sample, for Task 3.1
IM-34	ULSFO	FincoEnergies, Netherlands	Small sample, for Task 3.1
IM-36	VLSFO	Bunker from vessel, Netherlands	Small sample, for Task 3.1
IM-37	VLSFO	Bureau Veritas, Malta	Small sample, for Task 3.1
IM-38	VLSFO	Bureau Veritas, Malta	Small sample, for Task 3.1
IM-39	VLSFO	Peninsula Antwerp, Belgium	Small sample, for Task 3.1
IM-40	VLSFO	ExxonMobil, AmSpec, Netherlands	Small sample, for Task 3.1

The nineteen samples were residual black oils. At ambient temperature, nine of them were fluid whereas ten were semi-solid.

2.2 Oil characterisation (Task 3.1) and biodegradability (Task 3.4)

The samples were put in the oven at 50°C overnight before characterisation, to allow the oils to flow out.

Fresh oils characterisation of the sixteen small samples consisted in viscosity and density measurement at 15°C, pour point and flash point measurements, waxes and asphaltenes contents determination, evaporation rate and full detailed chemical characterisation (OSCAR composition).

Additionally, a temperature sweep from 50°C to 0°C was performed in order to get an overview of the oils' viscosity evolution with temperature. Finally, an in-depth study of the determination of the waxes, by comparing different protocols, was carried out with a view of better understanding the relationship between waxes and pour points.

The three large samples were subjected to the same screening phase and additional analyses were performed at the laboratory scale. Viscosity and density were assessed at 5°C and 25°C, temperatures of the flume tank experiments. Biodegradability rates were also assessed on those samples at the laboratory scale.

- *Appendix 1* details the methods of measurements used in this study.
- Results from Task 3.1 and Task 3.4 are presented in sections 3.1 and in *Appendices 2, 3 and 4.*

2.3 Oil weathering at 5°C (Task 3.2) and 25°C (Tasks 3.3)

2.3.1 Experimental protocol and conditions of test

The tests were performed in Cedre's hydraulic canal (the *Polludrome®*), which was used with the water being continuously circulated to simulate dynamic conditions (Figure 1). For each experiment 20 liters of preheated oil at 50°C were poured at the water surface.

The three large samples were weathered following three conditions: 5°C and 25°C in seawater conditions and 5°C in freshwater conditions, leading to a total of nine flume tank experiments. For those nine tests, wave height of 20 cm, current speed of 0.4 m/s and water depth of 0.90 m were set up and volume of water of 7 m^3 was used. Tests were run assuming a moderate situation (around sea state 2-3 on the Douglas scale, which corresponds in real conditions to wave heights between 0.5 and 1.25 m) with a simulated wind speed of 10 knots (it must be noted that the device is equipped with an exhaust fan for safety reasons, and the corresponding wind speed was assessed by calibrations and comparisons with evolutions in real environmental conditions). The photo-oxidation process (solar energy) was recreated by the use of two UV lights.

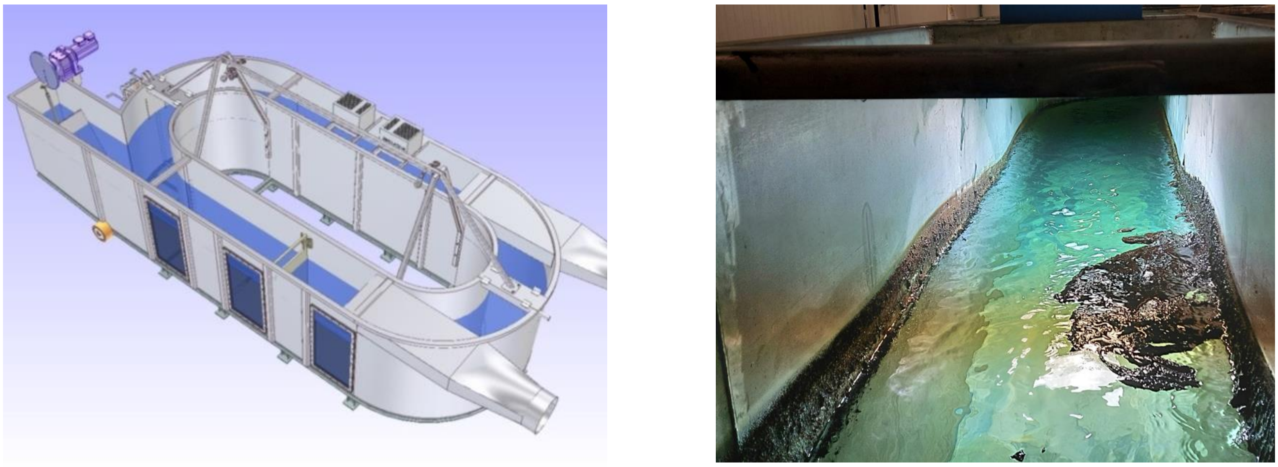


Figure 1 *View of the Polludrome® and of the IM-28 sample aspect after 72 hours of weathering at 5°C in seawater*

The weathering of the oils was monitored in the *Polludrome®* during seven days in order to evaluate their fate and behaviour during the first hours and first days after an oil spill. During the tests, surface oil samples were periodically taken to determine or assess the oil characteristics (see Table 3).

It must be noted that these experiments can simulate short-term weathering (up to 1 or 2 weeks) but cannot completely recreate long-term processes or incorporation of mineral fines, phenomena liable to stabilize emulsions.

***Table 3** Sampling times*

Sample reference	T 0	T 1	T 2	T 3	T 4	T 5	T 6	T 7	T 8	T 9	T 10	T 11	T 12	T 13	T 14	T 15	T 16	T 17
Time (hours)	0	1	2	4	6	8	14	24	26	30	34	48	53	72	77	96	101	168

In addition, the fresh and the weathered oils were subjected to dispersibility tests using the IFP (NF T 90-345 French Standard) and MNS laboratory protocols. The typical sampling points correspond to 6, 14, 24, 34, 48, 72 and 168 hours of weathering. However, tests can be stopped if the oils turn not dispersible. Model dispersant was used.

***Table 4** Characterisation of the oil samples collected during the weathering experiments*

Physical-Chemical parameters	
Viscosity	- Evolution of the oil and/or emulsion viscosity
Density	- Evolution of the surface oil and/or emulsion density
Emulsification	- Kinetics of emulsification and maximum water content - Changes in the stability of the emulsion
Evaporation (at 25°C)	- Modification of the oil composition - Kinetics of evaporation and maximum evaporation rate
Treatment possibilities	
Oil adhesion	- Adhesion of the oil to oleophilic surfaces; oil ability to be skimmed
Oil dispersibility	- Assessment of the efficiency of chemical dispersants application

- *Appendix 1* details the methods of measurements used in this study.
- Results from Task 3.2 and Task 3.3 are presented in section 3.2 and in *Appendices 5, 6, 7 and 8.*

2.4 Oil fingerprinting (Task 3.5)

The nineteen small and large samples were sent to RBINS for oil fingerprinting.

2.4.1 Equipment and Reagents

The analysis was performed using a Thermo GC-MS system, equipped with a DB-5 capillary column (30 m, 0.25 µm film thickness). Helium (grade 6.0) was used as the carrier gas. Other equipment includes a muffle furnace, analytical balance, ultrasonic bath, and Zymark TurboVap evaporation unit.
Key reagents include: Dichloromethane (solvent), Silica gel (for clean-up), Reference standards, such as Brent crude oil and a C_8–C_{40} alkanes mixture.

2.4.2 Sample Pretreatment

All oils were diluted to ± 8mg/mL DCM. A silica column clean-up was applied to remove asphaltenes and polar compounds.

2.4.3 Preparation of Columns and Reagents

Silica gel was activated by heating at 150 °C and stored in a desiccator. Clean-up columns were manually prepared using Pasteur pipettes, glass wool, and pre-treated silica gel.

2.4.4 GC-MS Parameters

Helium was used as the carrier gas under constant flow 1.1 ml/min

Oven Program

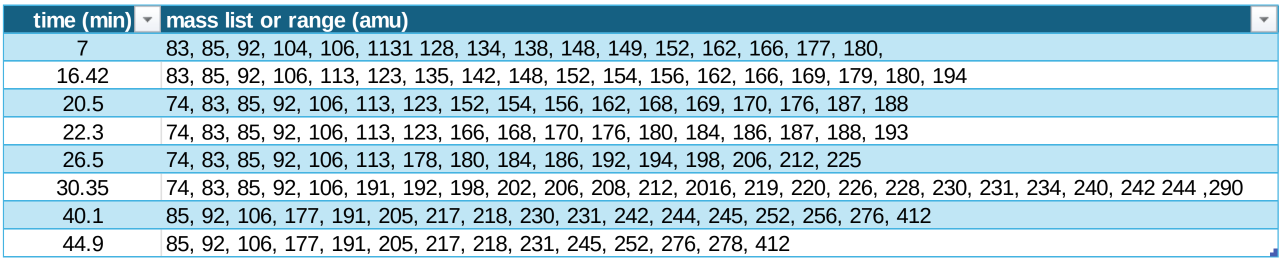
The initial temperature was set at 42 °C and held for 0.5 minute. The temperature was then increased at a rate of 5.5 °C per minute up to 180 °C, followed by a second ramp at 3 °C per minute until a final temperature of 330 °C was reached and held for 20 minutes.

Programmed Temperature Vaporizing (PTV) injection

The injector was initially held at 50 °C (0.1min), then heated at a rate of 8 °C per second to 350°C, which was maintained for 10 minutes.

TIC mode scans a mass range of 50–700 amu and was used for initial screening.
SIM mode was used to target specific ions associated with diagnostic biomarkers. The selection of masses and corresponding retention time windows was based on expected compound elution behaviour and is summarized in Table 5.

Table 5 *Masses measured in timeslot*



time (min)	mass list or range (amu)
7	83, 85, 92, 104, 106, 1131 128, 134, 138, 148, 149, 152, 162, 166, 177, 180,
16.42	83, 85, 92, 106, 113, 123, 135, 142, 148, 152, 154, 156, 162, 166, 169, 179, 180, 194
20.5	74, 83, 85, 92, 106, 113, 123, 152, 154, 156, 162, 168, 169, 170, 176, 187, 188
22.3	74, 83, 85, 92, 106, 113, 123, 166, 168, 170, 176, 180, 184, 186, 187, 188, 193
26.5	74, 83, 85, 92, 106, 113, 178, 180, 184, 186, 192, 194, 198, 206, 212, 225
30.35	74, 83, 85, 92, 106, 191, 192, 198, 202, 206, 208, 212, 2016, 219, 220, 226, 228, 230, 231, 234, 240, 242 244 ,290
40.1	85, 92, 106, 177, 191, 205, 217, 218, 230, 231, 242, 244, 245, 252, 256, 276, 412
44.9	85, 92, 106, 177, 191, 205, 217, 218, 231, 245, 252, 276, 278, 412

- *Appendix 9* presents the report from RBINS.
- Summarized results from Task 3.5 are presented in section 3.3.

3 RESULTS OF LABORATORY SCALE TESTS

3.1 Oil characterisation (Task 3.1) and biodegradability (Task 3.4)

3.1.1 Gas chromatography (GC-FID) characterisation

GC-FID analysis was performed to screen the samples. General shape of each oil and n-alkanes distribution can thus be compared. Figure 2 exhibits the example of four of the LSFO analysed: one ULSFO (in orange) and three VLSFOs (in blue). n-alkanes are represented as narrow regular and abundant peaks. Components with the lowest boiling points (i.e the lightest) appear with the lowest retention time (left part of the graphs). Heaviest compounds (> nC_{40}) cannot be analysed with this technique. “Unresolved Complex Mixture” (UCM) appears as a broad hump below the resolved peaks.

- The chromatograms of all the samples analysed are presented in Appendix 2.

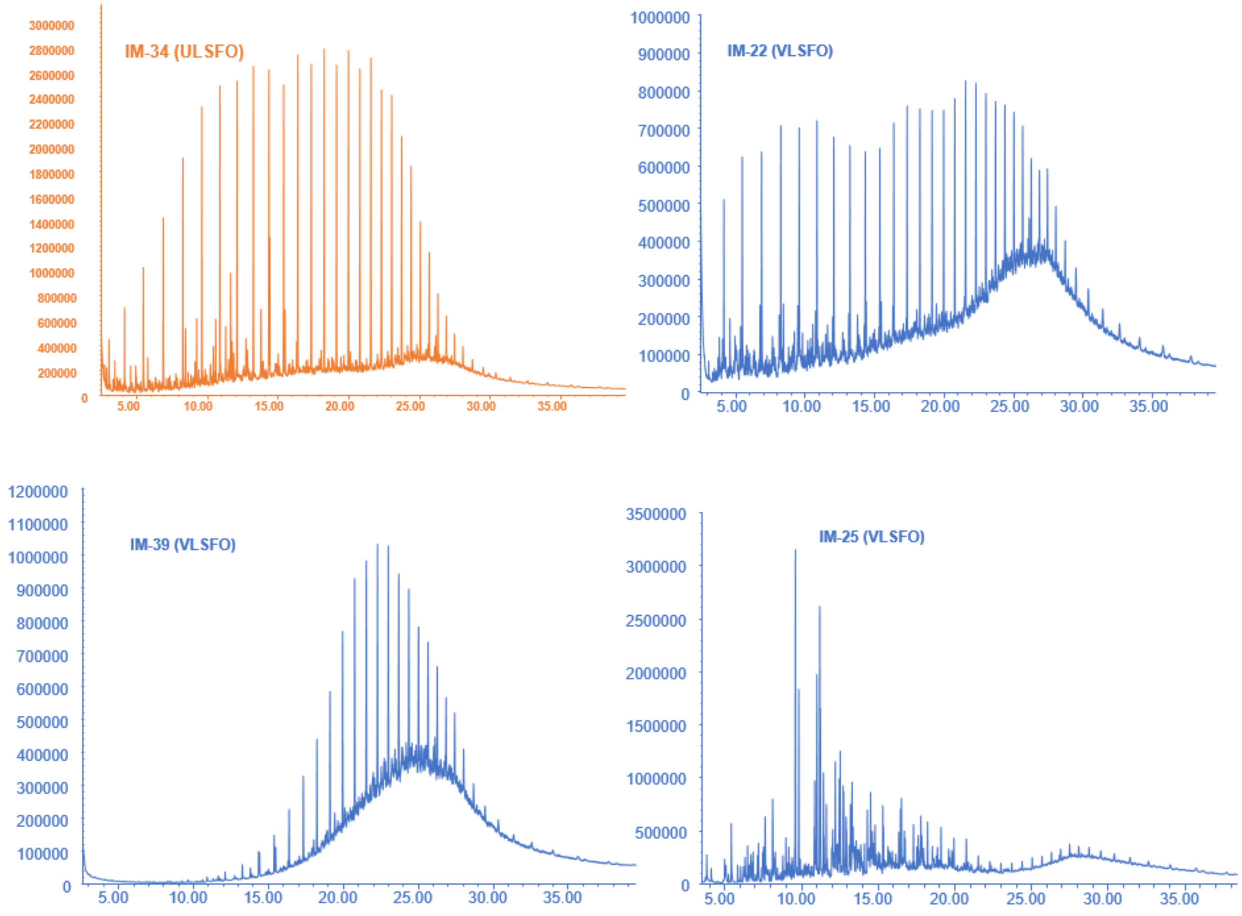


Figure 2 *GC-FID chromatograms (compounds abundance in function of retention time in minutes) of four of the fresh LSFOs. The graphs are represented with different “abundance” scale (y-axis). Before 14 minutes are represented n-alkanes < nC17. After 18 minutes are represented n-alkanes > nC20.*

Based upon the visual comparison of the chromatograms, LSFOs exhibit different hydrocarbons profiles, reflecting variability in terms of physical – chemical properties. This was already observed during the first IMAROS project. Based on the samples analysed, the variability seems less pronounced for the ULSFOs, often characterised by a broad range of n-alkanes (nC_{10} – nC_{36}). Some samples exhibit peaks of naphthalenes, represented on the graphs by the irregular peaks between 8 and 12 minutes (IM-21, IM-25, IM-31). Naphthalene contents can give insights on oils ecotoxicity. Abundance of n-alkanes eluting after 17 minutes (representing-alkanes > nC_{20}) reflects a high wax content (ULSFOs IM-21, IM-23, IM-32, IM-34).

Similarities between the samples IM-21 and IM-23 and between the samples IM-32 and IM-34, all of them ULSFOs, were observed. The two first samples come from Sweden (an oil refinery and a bunker delivery). The two other ones come from the same bunker delivery in the Netherlands. Those samples could come from the same source.

3.1.2 Physical-chemical properties

The physical-chemical characterisation of the initial fresh oils is presented in Table 6.

- OSCAR characterisations are presented in Appendix 3.
- PAH quantifications are presented in Appendix 4.

Table 6 *Physical-chemical characterisation of the nineteen fresh LSFOs*

Sample	**Nature**	Viscosity 15°C (mPa.s) (1)	Viscosity 50°C (mPa.s)	Density 15°C	Pour Point (°C)	Flash point (°C)	Asph. (%) (3)	Waxes (%)(3)	Evaporation (vol. %)	Max. distillation rate (%)(4)
IM-20*	VLSFO	29 521	283	0.97	9	>100 (2)	4.57	8.78	3.2	31
IM-21	ULSFO	327 173	61	0.90	33	>100 (2)	0.25	27.07	3.9	34
IM-22*	VLSFO	29 378	385	0.97	9	99.5	5.75	8.76	5.9	48
IM-23	ULSFO	262 086	63	0.90	30	>100 (2)	0.34	24.63	4.3	36
IM-24	VLSFO	54 107	189	0.94	27	>100 (2)	0.97	7.47	1.5	97
IM-25*	VLSFO	3 783	140	0.99	33	>100 (2)	4.87	6.15	8.8	60
IM-27	VLSFO	16 600	262	0.96	9	>100 (2)	2.1	7.7	3.0	98
IM-28	VLSFO	54 813	109	0.94	24	>100 (2)	1.5	14.1	1.2	26
IM-29*	ULSFO	10 559	11	0.89	18	>100 (2)	0.4	13.3	7.6	83
IM-30*	VLSFO	1 009	52	0.97	3	91	3.91	4.96	15.4	88

(1) Mean value of the ten first measuring points (over 60 points), shear rate of 10 s^{-1}

(2) Maximum value measured: 100°C

(3) Values recalculated for the fresh oils, from the 250°C residues, taking into account the evaporation rate at 250°C

(4) Maximum evaporation rate (%) obtained from simulated distillations (GC-FID) at 520°C

* sample fluid at room temperature

Table 6 (cont.) *Physical-chemical characterisation of the nineteen fresh LSFOs*

Sample	Nature	Viscosity 15°C (mPa.s) (1)	Viscosity 50°C (mPa.s)	Density 15°C	Pour Point (°C)	Flash point (°C)	Asph. (%) (3)	Waxes (%)(3)	Evaporation (vol. %)	Max. distillation rate (%)(4)
IM-31*	VLSFO	348	22	0.96	-12	92.5	ND(5)	ND(5)	ND(5)	ND
IM-32	ULSFO	116 276	43	0.89	30	82	0.36	24.43	6.4	51
IM-33*	VLSFO	10 154	209	0.98	-6	>100 (2)	5.02	8.14	4.8	42
IM-34	ULSFO	239 834	48	0.89	33	83	0.46	26.99	6.0	47
IM-36*	VLSFO	1 703	70	0.95	-6	96	1.90	10.16	11.7	56
IM-37	VLSFO	81 425	164	0.96	27	>100 (2)	2.30	11.80	2.5	50
IM-38	VLSFO	39 178	279	0.96	15	>100 (2)	3.46	8.03	6.7	63
IM-39	VLSFO	29 899	246	0.95	12	>100 (2)	1.96	9.84	2.2	85
IM-40*	VLSFO	3 535	154	0.98	ND	97.5	2.01	6.27	8.9	ND

(1) Mean value of the 10 first measuring points (over 60 points), shear rate of 10 s^{-1}
(2) Maximum value measured: 100°C
(3) Values recalculated for the fresh oils, from the 250°C residues, taking into account the evaporation rate at 250°C
(4) Maximum evaporation rate (%) obtained from simulated distillations (GC-FID) at 520°C
(5) Distillation vapor temperature 250°C impossible

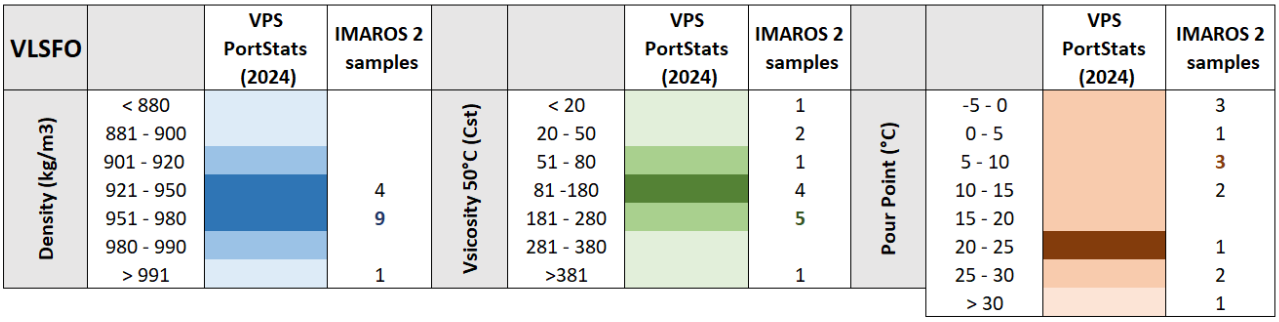
* sample fluid at room temperature

3.1.3 Market evolution

Characteristics of the samples collected in the frame of IMAROS 2 were compared to the statistics obtained from Veritas Petroleum Services (VPS) and already presented in the deliverable D2.2 (WP2 – Trends and Samples), in order to ensure their representativeness.

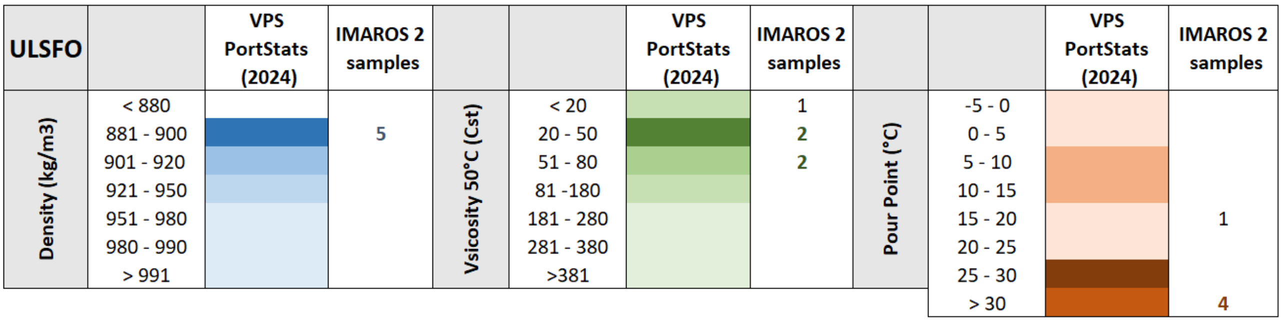
Table 7 and Table 8 compare the summarised data from VPS Portsats (focused on the year 2024) for density, viscosity and pour points with the fourteen VLSFOs and the five ULSFOs collected in the frame of the project.

Table 7 *Comparison of the density, viscosity and pour points between the VPS PortStats data from 2024 (dark colors represent high representation in the category) and the number of VLSFOs collected in the frame of the project in each category*



VLSFO		VPS PortStats (2024)	IMAROS 2 samples			VPS PortStats (2024)	IMAROS 2 samples			VPS PortStats (2024)	IMAROS 2 samples
Density (kg/m3)	< 880			Vsicosity 50°C (Cst)	< 20		1	Pour Point (°C)	-5 - 0		3
	881 - 900				20 - 50		2		0 - 5		1
	901 - 920				51 - 80		1		5 - 10		3
	921 - 950		4		81 -180		4		10 - 15		2
	951 - 980		9		181 - 280		5		15 - 20		
	980 - 990				281 - 380				20 - 25		1
	> 991		1		>381		1		25 - 30		2
									> 30		1

Table 8 *Comparison of the density, viscosity and pour points between the VPS PortStats data from 2024 (dark colors represent high representation in the category) and the number of ULSFOs collected in the frame of the project in each category*



ULSFO		VPS PortStats (2024)	IMAROS 2 samples			VPS PortStats (2024)	IMAROS 2 samples			VPS PortStats (2024)	IMAROS 2 samples
Density (kg/m3)	< 880			Vsicosity 50°C (Cst)	< 20		1	Pour Point (°C)	-5 - 0		
	881 - 900		5		20 - 50		2		0 - 5		
	901 - 920				51 - 80		2		5 - 10		
	921 - 950				81 -180				10 - 15		
	951 - 980				181 - 280				15 - 20		1
	980 - 990				281 - 380				20 - 25		
	> 991				>381				25 - 30		
									> 30		4

Regarding the two types of LSFOs, the samples collected are in the most represented ranges for density and viscosity. The pour point (measured in the project) of the VLSFOs collected in the frame of the project seems to be more heterogeneous and covers all the ranges. The pour point of the ULSFOs is high (as observed in the statistics) except for one sample (IM-29, 18°C). It can be noted that this sample is characterized by the presence of cardanol (a phenolic compound derived from cashew nutshell liquid) (see Task 3.5 Fingerprinting). This compound, which has not been quantified in the sample, is used as biocomponent for bio-fuels. When mixed with LSFOs, it can induce a decrease of the viscosity and the pour point of the fuel. However, the lack of quantification of this compound in the IM-29 sample prevent us classifying it as a bio-fuel.

The samples obtained in the frame of the project are thus mainly representative of the market in 2024. Only one ULSFO is characterized by a lower pour point than the statistics show.

3.1.4 Comparison with IMAROS

Physical-chemical parameters obtained in the frame of this project were compared to the ones obtained in the frame of the first IMAROS project (2020 – 2022). Summarised figures of viscosity, density, pour point, waxes/asphaltenes contents and evaporation rate are presented in Figures 3 - 7.

Flash point

As already observed during the first IMAROS project, flash point of the LSFOs collected are high, well above 60°C. According to the Globally Harmonized System of Classification and Labelling of Chemicals (GHS), several categories are defined for flammable products, but these only apply to flash points up to 60°C. Therefore, once the flash point exceeds 60°C, the risk of flammability in oil slicks can be considered negligible.
In the frame of the IMAROS 2 project, the lowest value is +82°C (IM-32). Twelve oils exhibit a flash point above 100°C.

Taking into account our set of samples, no safety issue related to flammability was raised.

Viscosity

Viscosity of fresh oils, which are often Newtonian fluids (i.e., the value of the viscosity is a constant over a wide range of shear rates) is measured at a shear rate of $100s^{-1}$. This allows a more stable measurement for the low viscosity oils. Most of the LSFOs collected in the frame of the IMAROS projects are characterised by high viscosities, leading to a semi-solid aspect at ambient temperature. Such oils are not Newtonian anymore but shear-thinning, with viscosity decreasing as the shear rate increases. Moreover, usual way to display viscosity measurements is to calculate the mean of sixty measurements (obtained over a total of one minute). Those sixty measures are quite constant over time. The sixty measures obtained with non-Newtonian LSFOs exhibit a decreasing exponential shape that may be explained by rearrangement of oil structuring and especially linear chains constituting waxes. In the project, the choice was made to display values representing the mean of the ten first values only (Table 6).
At 15°C, viscosities vary from 348 (IM-31) to more than 320 000 mPa.s (IM-21). ULSFOs stand out for their high viscosity, higher than 100 000 mPa.s at 15°C. Only the ULSFO IM-29 is distinguished by a lower value (10 559 mPa.s).
The temperature-sweeps for all the samples tested in the frame of the two projects are shown on Figure 3. For all the oils, viscosity vary significantly in the range of seawater temperature (~ 5°C-25°C). All the ULSFOs analysed are characterised by a huge increase of viscosity when the temperature decreases and reaches their pour point. On the other hand, at 50°C, the viscosity of ULSFOs is among the lowest. No major evolution is noticed since 2020.

In case of spill, the viscosity of the product involved is often communicated to the responders at a temperature of 50°C, because this is the one appearing on the Certificates Of Analysis (COA, following the ASTM D7042 norm).

By looking at the Figure 3, it appears that, if the product involved is a ULSFO, especially characterised by a high pour point, the viscosity of the oil at sea could extremely increase.

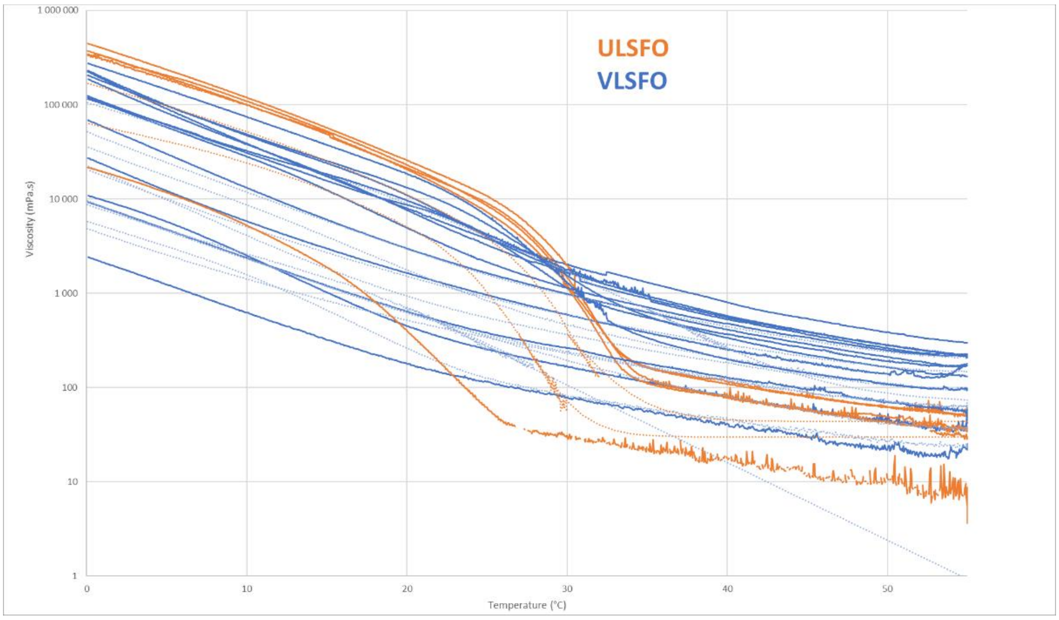


Figure 3 *Temperature sweep measurements (viscosities) of the LSFOs analysed during the 2 projects. IMAROS samples appear in dotted lines and IMAROS 2 samples in solid lines. USLFOs appear in orange and VLSFOs in blue.*

Pour point

Figure 4 presents the pour point of the samples collected in the frame of the two IMAROS projects. The same variability is observed between the two sets of data. IMAROS 2 values range from -12°C (IM-31) to +33°C (IM-21, IM-25 and IM-34). ULSFOs are characterised by a higher pour point than VLSFOs, as it was already observed during IMAROS. Only the ULSFO IM-29 exhibits a lower pour point (+18°C).
This variability induces different behaviors of the LSFOs if spilt at sea and implies the choice of different response options, especially different recovery techniques.

Depending on the water temperature, oil solidification will occur for oils characterised by the highest pour points.

It should be noted that pour point measurement seems to be subjected to uncertainties. For some products collected in the frame of the project, the pour point filled in the COA is different from the one measured by the project partners (the difference can reach 15°C). SINTEF performed an in-depth study on the three large samples and confirmed the variability of the data obtained according to the protocol followed. Those results are presented in Appendix 10. This could be due to a "thermal memory" of the samples, especially when their wax content is high, and to the different heatings/coolings experienced by the samples.

The pour point communicated to the people in charge of the response should therefore be questioned in terms of its representativeness of the oil's behaviour at sea.

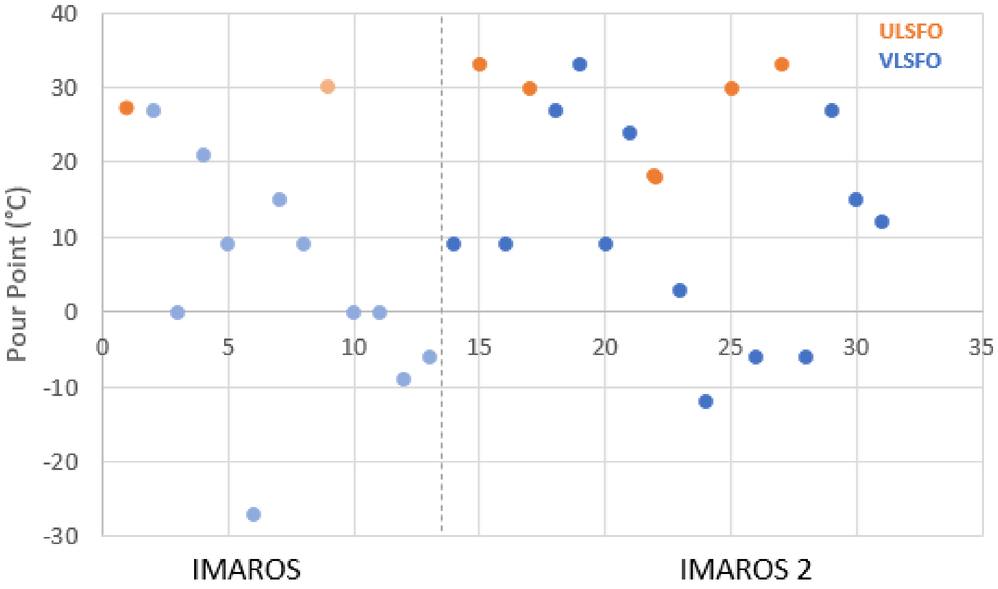


Figure 4 *Pour points measured for the small samples analysed in the frame of the 2 IMAROS projects (x-axis represents samples incrementation from IM-1 to IM-40)*

Waxes and Asphaltenes

Data are presented on Figure 5. Two clusters are observed: ULSFOs characterised by high wax contents and relatively low asphaltenes contents, and VLSFOs characterised by a more important variability but still identifiable by lower waxes content and potentially higher asphaltene contents. No major changes were observed between the two sets of data (IMAROS and IMAROS 2) except slightly higher waxes content for the ULSFOs collected in the frame of IMAROS 2.

Regarding IMAROS 2 data, in terms of chemical composition, asphaltenes content vary from 0.25% (IM-21) to 5.75% (IM-22) and are *a priori* in agreement with conditions required to form water-in-oil emulsions, the threshold limit of asphaltenes content being generally estimated around 0.2 to 0.3% when measured as insoluble in *n*-heptane. The wax content variation is high, from 4.96% (IM-30) to 27.07% (IM-21).

For most of the samples exhibiting high wax contents, the pour point is generally high, well above the minimum seawater temperature. This will induce a solidification of those oils when in contact with the seawater.

For comparison, this variability reflects the one observed for the crude oils. The average values calculated on the 98 crude oils tested at Cedre are 0.90% (values varying between 0.02 and 8.7%) for asphaltenes and 8.00% (values varying between 0.02 and 42.3%) for waxes content.

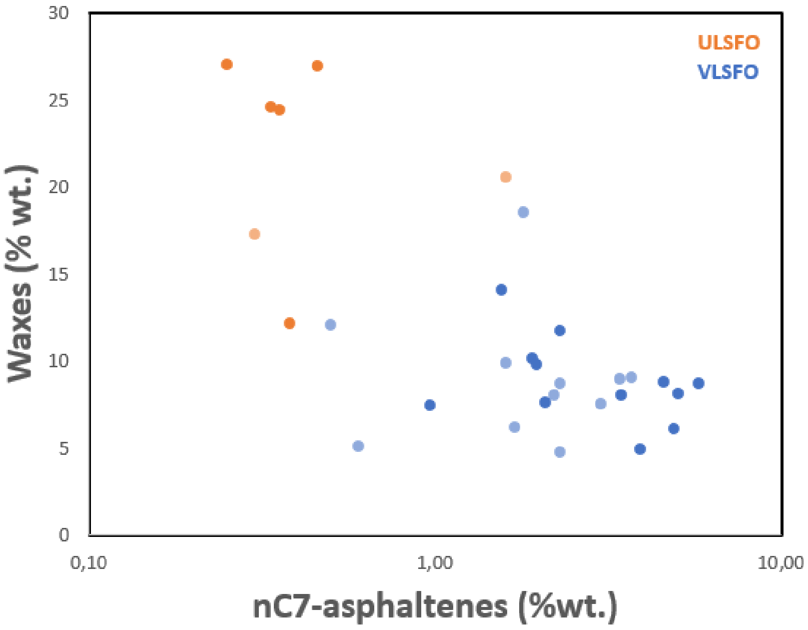


Figure 5 Distribution of the LSFOs tested at Cedre in the frame of the 2 IMAROS projects (IMAROS in light blue/orange and IMAROS 2 in dark blue/orange) based on asphaltenes and wax contents. ULSFOs are represented with orange dots, VLSFOs with blue dots.

Density

Data are presented on Figure 6. The main observation, already highlighted in the frame of the first project, is the lower values exhibited by the ULSFOs (around 0.90) whereas VLSFOs are characterized with higher values, mainly above 0.94.

Based on the two sets of samples, LSFOs should float at the water surface. However, it should be noted that, depending on the salinity and on the suspended material concentration, some oils may be entrained into the water column.

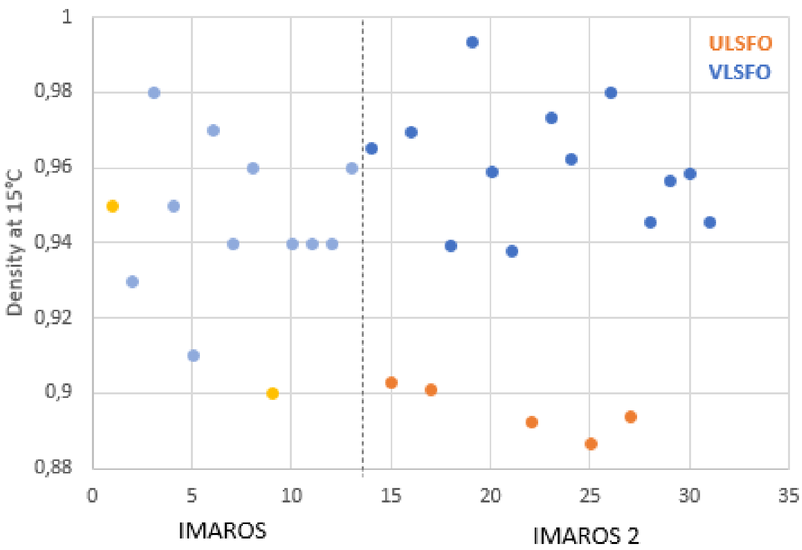


Figure 6 *Density of the samples collected in the frame of the IMAROS projects. Samples from IMAROS are located on the left part of the graph in light blue/orange and samples from IMAROS 2 on the right part of the graph in dark blue/orange. ULSFOs are represented with orange dots, VLSFOs with blue dots.*

Evaporation rate (vol. %)

Data are presented on Figure 7. Evaporation rate of the samples collected in the frame of IMAROS 2 exhibits even lower values than the samples collected in the frame of the first IMAROS project. In the frame of this project, values range between 1.1 % (IM-28) and 15.4 % (IM-30). No difference was noticed between VLSFO and ULSFO.

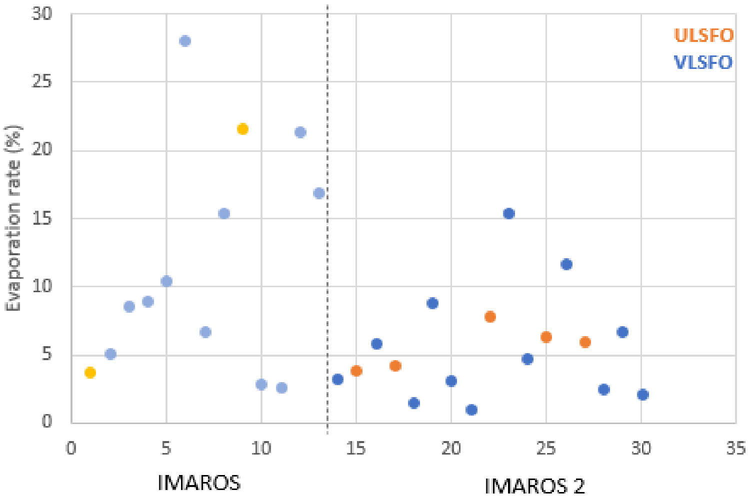


Figure 7 *Evaporation rate (%) of the samples collected in the frame of the IMAROS projects. Samples from IMAROS are located on the left part of the graph in light blue/orange and samples from IMAROS 2 on the right part of the graph in dark blue/orange. ULSFOs are represented with orange dots, VLSFOs with blue dots.*

Those results highlight the fact that LSFOs will be persistent in the aquatic environment.

3.1.5 In-depth wax determination study

The method historically used, required by certain behaviour modelling softwares and generally applied to crude oils, consists in quantifying waxes by gravimetry. The second method involves direct analysis of the samples after simple dilution, followed by gas chromatography analysis with flame ionisation detection (GC-FID). This second approach is much faster and less expensive than the first method because it does not require the prior removal of asphaltenes.

Results showed that the gravimetric method leads to systematic higher wax contents compared with the direct analysis of the diluted samples. This could be due to the precipitation of other compounds, such as resins, during waxes precipitation step performed in the gravimetric method.

Figure 8 presents the alkanes distributions of the samples between three cuts ($C_8 – C_{20}$, $C_{20} – C_{30}$ and $C_{30} – C_{40}$) obtained from the direct analysis of the samples. It appears that ULSFOs analysed in this study exhibit systematic higher proportion of alkanes in the range $C_{20} – C_{30}$ whereas VLSFOs exhibit a more regular wax distribution between the cuts $C_{20} – C_{30}$ and $C_{30} – C_{40}$. This result supports the addition of wax fractions to reduce reduction of sulphur content for ULSFOs.

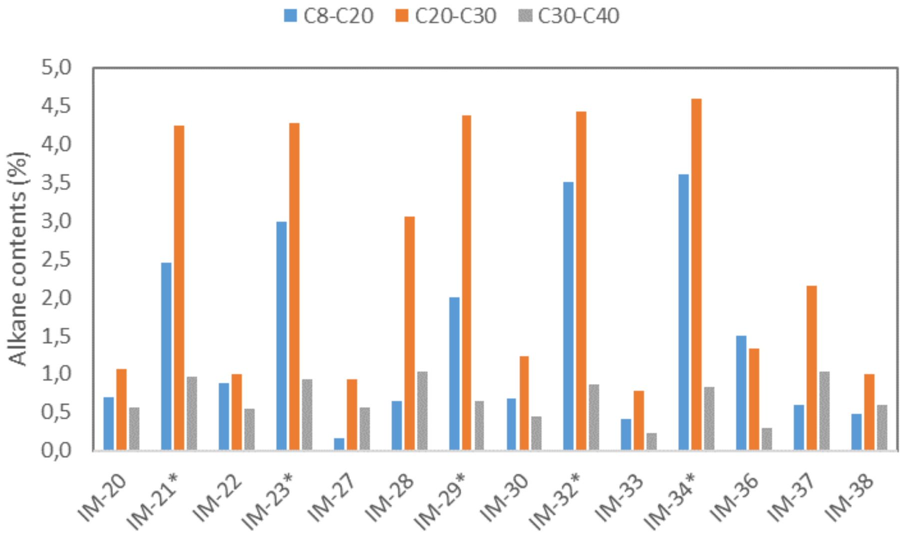


Figure 8 *Alkane distribution, determined from direct analysis of the oils. Waxes are part of the C_{20}-C_{30} and C_{30}-C_{40} ranges. ULSFOs are identified with a star.*

3.1.6 Biodegradability (Task 3.4)

The three large samples (IM-27, IM-28 and IM-29) were analysed for biodegradability. Figure 9 illustrates the relative extent of the weathering processes (evaporation and biodegradation). The contribution of the biodegradation process was calculated relatively to the fresh oil. Considering the evaporation and biodegradation processes separately, it appears that between 6 % in weight (IM-28) and 24 % (IM-29) of the LSFOs are biodegraded while between 1% (IM-28) and 7% (IM-29) are evaporated (Table 9). **Those results mean that, after natural degradation, between 73 % (IM-27) and 93 % (IM-28) of the LSFOs will persist in the environment (Table 9).** It must be noted that these rates were obtained over a period of 28 days in optimized conditions (low oil concentration, adapted bacteria and addition of nutrients).

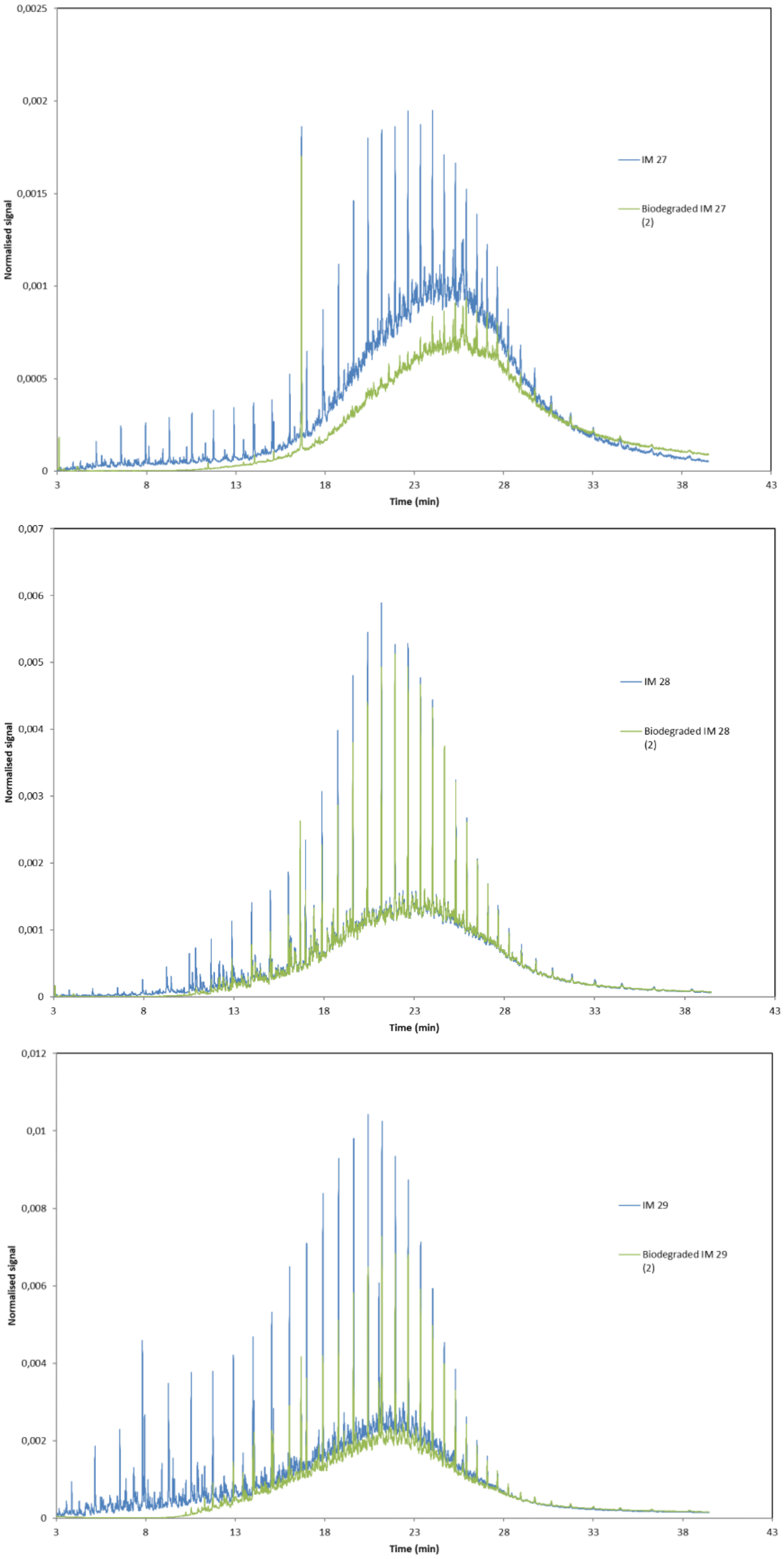


Figure 9 *GC/FID chromatograms of the fresh (in blue) and biodegraded (in green) LSFOs IM-27, IM-28 and IM-29*

Table 9 *Mass balance representing weathering processes affecting the 3 LSFOs IM-27, IM-28 and IM-29*

Mass balance	IM-27	IM-28	IM-29
Residue	0.73	0.93	0.76
Evaporated	0.03	0.01	0.07
Biodegraded	**0.24**	**0.06**	**0.16**

3.2 Oil weathering at 5°C (Task 3.2) and 25°C (Task 3.3)

3.2.1 Physical-chemical evolution of the oils

The three following sections describe the evolution of the physical-chemical properties of the three large samples IM-27, IM-28 and IM-29 under three different environmental conditions: 5°C in freshwater (FW), 5°C in seawater (SW) and 25°C in seawater (SW). Pictures of the three LSFOs at the beginning and at the end of each test are presented and the physical-chemical parameters were plotted against the weathering time.

Some common observations as regards the oils tested can be formulated and summarized as follows:

At 5°C, the oil slicks can either remain compact (IM-27) or fragment in small granular tarballs (IM-28 and IM-29). Weathering occurs then in two steps. During the first step, which can last up to 96 hours, the oils tend to stick to the flume tank walls, some very strongly (IM-27 and IM-28) with sometimes no free slicks remaining at the water surface. Oils have to be manually scraped off the walls in order to be sampled. IM-29 sticks less to the walls but still tends to cling to them as it moves forward. With time and emulsification, IM-27 and IM-29 turn less sticky and emulsified slicks freely float again at the water surface. IM-28 still exhibits this highly sticky behaviour until the end of the trials.
In those test conditions, the flume tank walls induce a bias on the natural behaviour and weathering of the slicks. **In real environment (open sea), slicks are expected to be either even more compact or fragment in diffuse smaller patches, with slower emulsification process.**

At 25°C, the three oils exhibit a much more traditional behaviour, with more fluid and less sticky slicks that slowly emulsify.

The effect of water salinity (seawater versus freshwater) on the oil behaviour is not clearly observed. In case of turbulences (created in those experiments by the corners of the flume tank), the slicks can be dragged down below the surface. This phenomenon seems more pronounced during the experiments performed with freshwater.

In real environment, the increase in density is not expected to cause the oil slicks to submerge below the water surface for seawater conditions. However, the buoyancy of some oils could be questioned close to estuaries, characterized by a lower salinity and a high mineral load which could increase the emulsion density.

3.2.1.1 Weathering of the VLSFO IM-27

Pictures illustrating the appearance of the fresh and weathered slicks are presented in Figure 10. Emulsification is clearly observed at the end of the trials. The greater fluidity of the fresh oil at 25°C is also clearly observed.
The main observations related to IM-27 are the following:

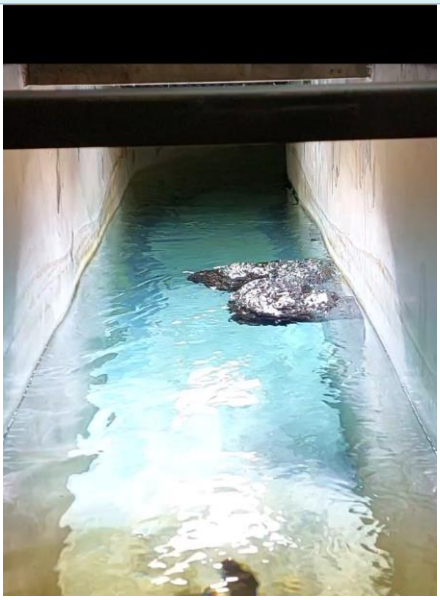
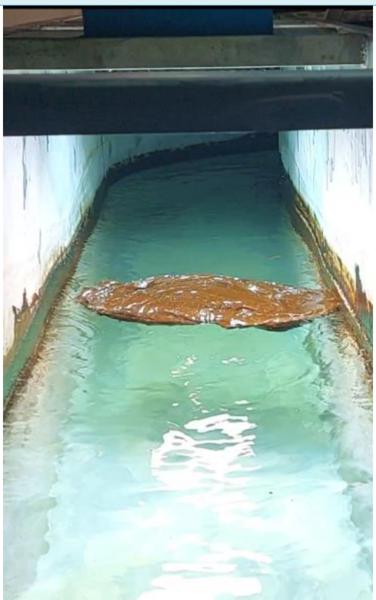
- Regarding the viscosity, stabilisation of the values is observed after 24 hours, reaching 80 000 – 120 000 mPa.s at 5°C and 2 000mPa.s at 25°C (Figure 11).
- Density reaches 0.99 at 5°C and 0.98 at 25°C (Figure 12).
- Water content represents respectively only 50% of the emulsions formed at 5°C and 80% at 25°C (Figure 13). The kinetics of emulsification is quite slow at 5°C as the plateau is reached only after 48 – 60 hours. The beginning of the water uptake is likely more related water pocket in the oil than real oil-in-water emulsions.
- All the emulsions formed are very stable (> 0.8) which means that nearly no water settled after 24 hours settling time.
- Evaporation at 25°C only reaches 3%.
- Oil adhesion stabilises at around 4 000 g/m^2 at 5°C and 600 g/m^2 at 25°C.
- Finally, evolution of the chemical composition (SARA) at 25°C between the fresh and the most weathered sample was as shown in Table 10.

Table 10 *Evolution of the chemical composition of the VLSFO IM-27*

Compounds	T_0	7 days / 25 °C
Saturates	48.2	40.6
Aromatics	31.3	23.7
Resins	16.1	29.5
Asphaltenes	4.4	6.3

Co-funded by the European Union

Figure 10 *Top views of the slicks of IM-27 at the beginning and the end of each flume tank experiment*

	T0	Tf
5°C / SW	 (+1h)	
5°C / FW		
25°C / SW		

Co-funded by the European Union

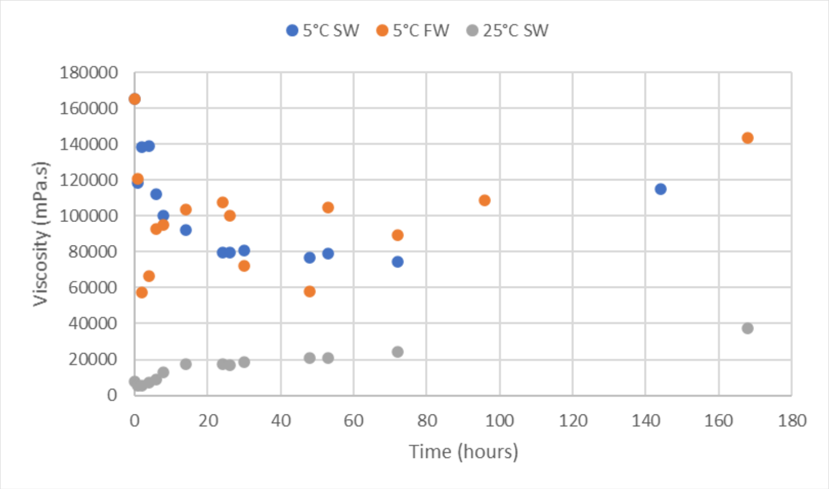


Figure 11 *Evolution of the viscosity of the VLSFO IM-27*

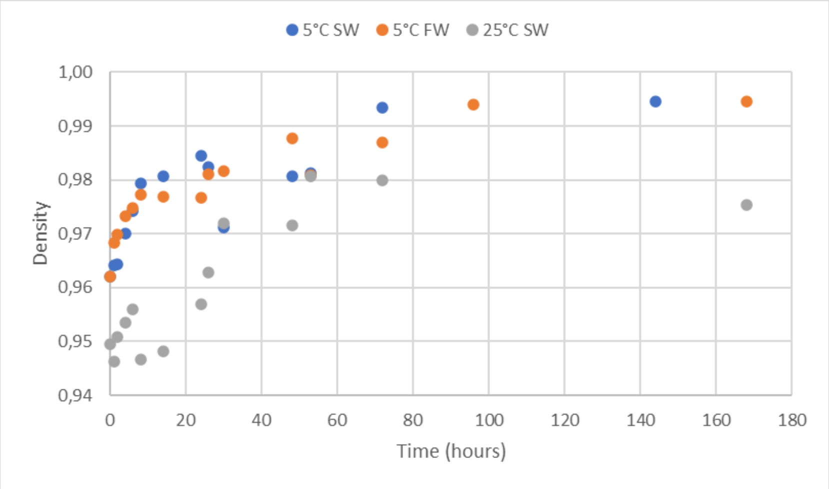


Figure 12 *Evolution of the density of the VLSFO IM-27*

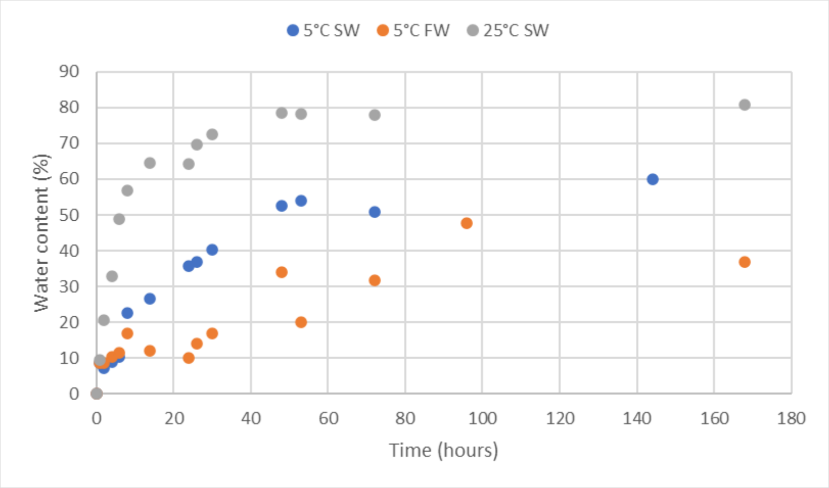


Figure 13 *Evolution of the water content of the VLSFO IM-27*

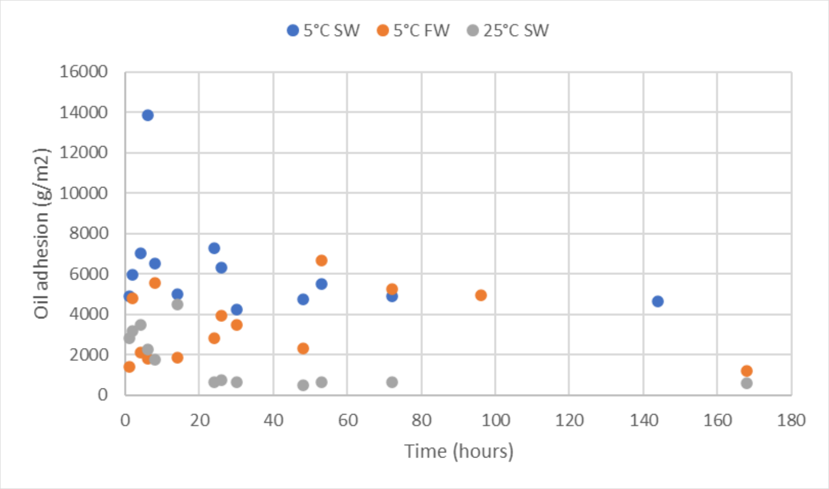


Figure 14 *Evolution of the oil adhesion of the VLSFO IM-27*

- Detailed results of the VLSFO IM-27 are presented in Appendix 5

3.2.1.2 Weathering of the VLSFO IM-28

Pictures illustrating the appearance of the fresh and weathered slicks are presented in Figure 15. The high viscosity and pour point (+24°C) of the oil is clearly observed since the beginning of the trials, with a fragmentation of the slicks in diffuse granular tarballs at 5°C. This phenomenon is less pronounced at 25°C.

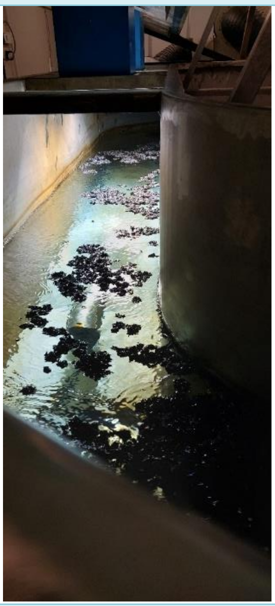
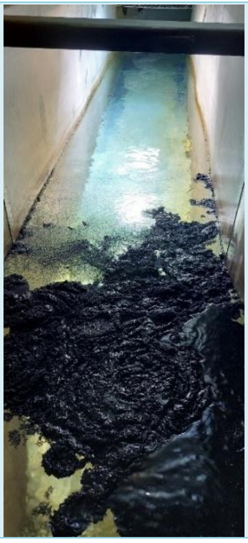
The main observations related to IM-28 are the following:

- Regarding the viscosity, values rapidly reach 300 000 – 350 000 mPa.s at 5°C and 60 000mPa.s at 25°C (Figure 16). Values obtained at 5°C could be challenging for oil recovery and pumping operations but could be compatible with oil trawling as complementary technique for recovery.
- Density reaches 0.96 at 5°C and 1.00 at 25°C (Figure 17).
- Water content represents only 30% of the emulsions formed at 5° (Figure 18) associated with kinetics of emulsification very slow as the plateau is reached only after 48 – 60 hours. Here again, beginning of the water uptake process is more likely due to water pocket in the oil than real oil-in-water emulsion. At 25°C, the water content reaches 80%.
- All the emulsions formed are very stable (> 0.9), which means that nearly no water settled after 24 hours settling time.
- Evaporation at 25°C only reaches ~3%.
- Oil adhesion data are very variable during the first 48 – 60 hours. After this time, values tend to reach a lower value of 2 000 g/m^2 at 5°C and 1 000 g/m^2 at 25°C.
- Finally, evolution of the chemical composition (SARA) at 25°C between the fresh and the most weathered sample was as shown in Table 11.

Table 11 *Evolution of the chemical composition of the VLSFO IM-28*

Compounds	T_0	7 days / 25 °C
Saturates	59.9	58.1
Aromatics	26.5	25.3
Resins	8.4	9.8
Asphaltenes	5.2	6.8

Figure 15 *Top views of the slicks of IM-28 at the beginning and the end of each flume tank experiment*

	T0	Tf
5°C / SW		
5°C / FW		
25°C / SW		

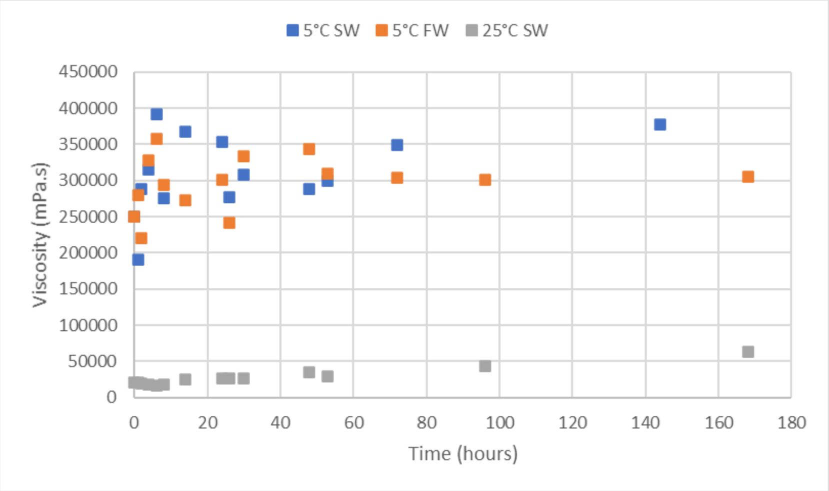


Figure 16 *Evolution of the viscosity of the VLSFO IM-28*

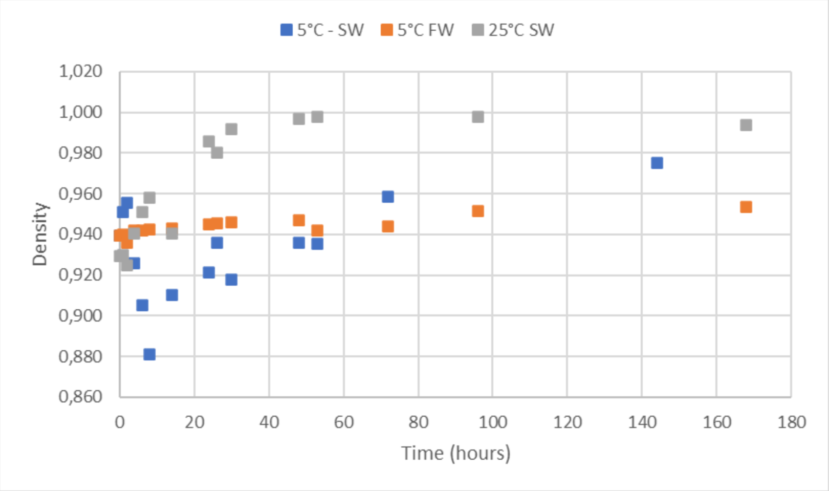


Figure 17 *Evolution of the density of the VLSFO IM-28*

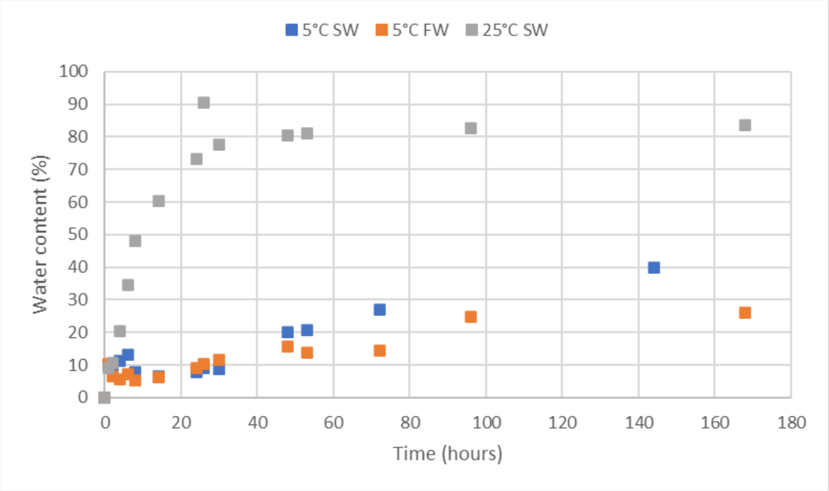


Figure 18 *Evolution of the water content of the VLSFO IM-28*

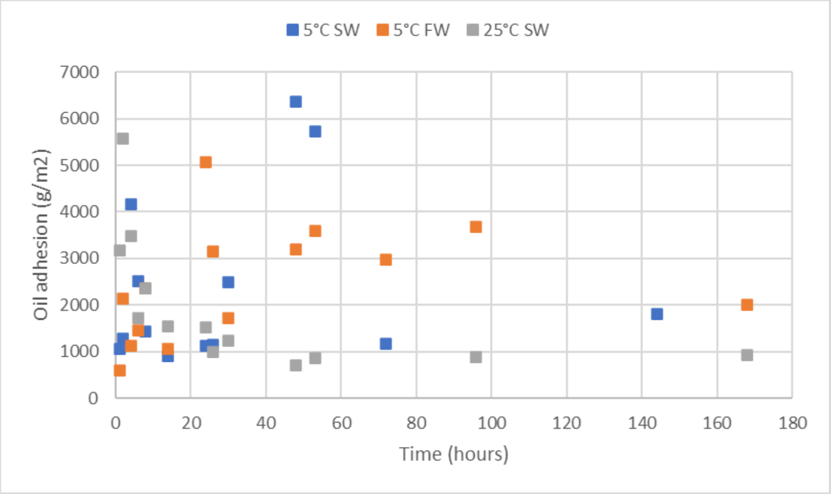


Figure 19 *Evolution of the oil adhesion of the VLSFO IM-28*

- Detailed results are presented in Appendix 6

3.2.1.3 Weathering of the ULSFO IM-29

Pictures illustrating the appearance of the fresh and weathered slicks are presented in Figure 20. At 5°C, a fragmentation of the slicks in smaller granular tarballs is observed in seawater. Emulsification is clearly observed at the end of the trials. The greater fluidity of the oil at 25°C is also clearly observed. Values obtained for the two experiments performed at 5°C differ significantly. This may be explained by an heterogeneity in the blend leading to a separation of phases between the two experiments (even if the batch was stirred before subsampling).

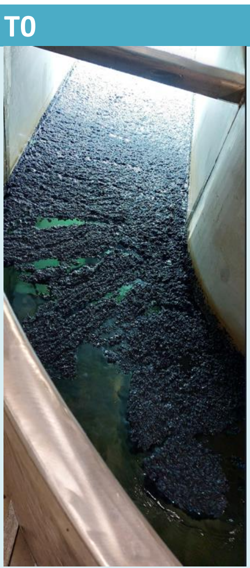
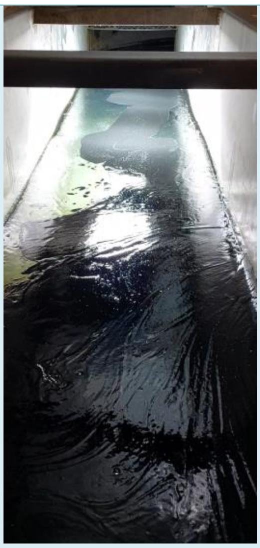
The main observations related to IM-29 are the following:

- Regarding the viscosity, values rapidly reach 40 000 – 60 000 mPa.s at 5°C and 8 000mPa.s at 25°C (Figure 21). Those values are quite low compared to usual values of viscosity observed when dealing with ULSFOs.
- Density reaches 0.98 at 5°C (in freshwater) and 1.00 at 25°C (Figure 22).
- Water content represents 50 – 70 % of the emulsions formed at 5°C, which is higher than the two other LSFO tested, and 90% at 25°C (Figure 23) and are associated with quick kinetics of emulsification.
- All the emulsions formed are very stable (1.0), which means that no water settled after 24 hours settling time.
- Evaporation at 25°C exhibits the highest values obtained for the three oils tested but does not exceeds 10%.
- Oil adhesion data are very variable during the first 48 – 60 hours. After this time, values tend to reach a lower value of 1 000 g/m^2 at 5°C and 2 000 g/m^2 at 25°C.
- Finally, evolution of the chemical composition (SARA) at 25°C between the fresh and the most weathered sample was as shown in Table 12.

Table 12 *Evolution of the chemical composition of the VLSFO IM-29*

Compounds	T_0	7 days / 25 °C
Saturates	77.3	73.0
Aromatics	13.1	16.0
Resins	7.5	6.9
Asphaltenes	2.1	4.1

Figure 20 *Top views of the slicks of IM-29 at the beginning and the end of each flume tank experiment*

	T0	Tf
5°C / SW		
5°C / FW	 (+1h)	
25°C / SW	 (+1h)	 (+168h)

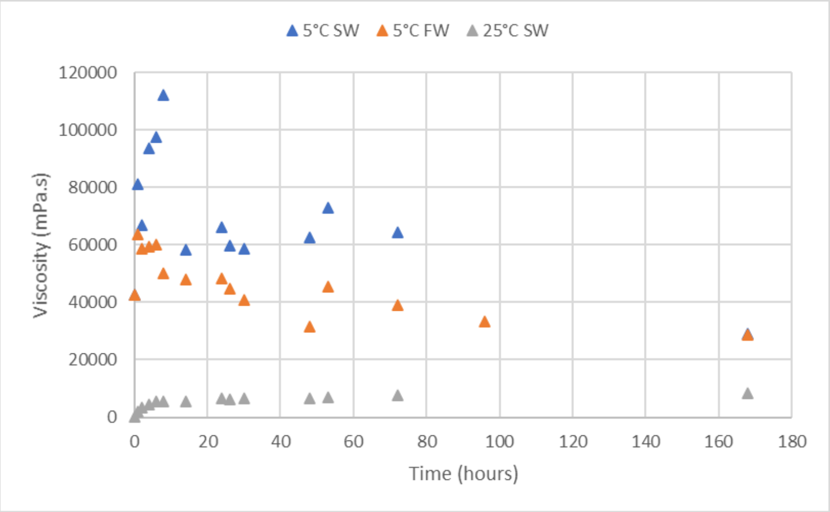


Figure 21 *Evolution of the viscosity of the VLSFO IM-29*

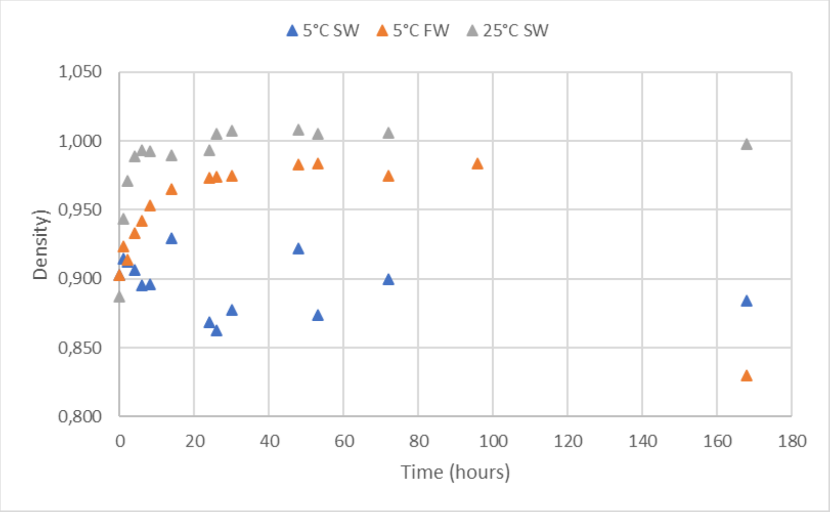


Figure 22 *Evolution of the density of the VLSFO IM-29*

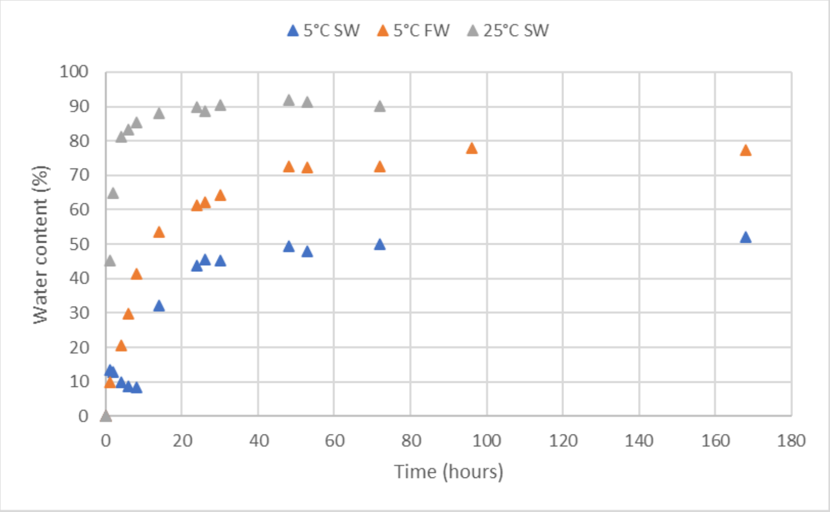


Figure 23 *Evolution of the water content of the VLSFO IM-29*

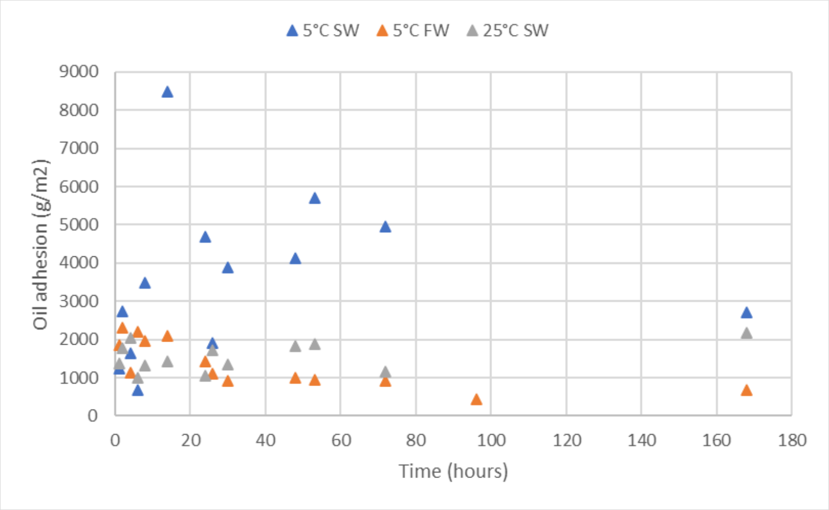


Figure 24 *Evolution of the oil adhesion of the VLSFO IM-29*

- Detailed results are presented in Appendix 7.

3.2.2 Dispersibility tests

The dispersibility of the three oils (IM-27, IM-28 and IM-29) was assessed at 5°C and 25°C, in seawater, according to the IFP and MNS protocols. Tests were performed on the fresh oils and also on a sample corresponding to 6 hours weathering time.

All those tests, performed with the Model dispersant, demonstrated that the three oils tested were not dispersible, neither fresh nor weathered. At the end of each test, the oil slicks remained intact at the tank surface. Figure 25 shows examples of the oil slick appearance at the end of the IFP and MNS tests. As no oil droplets were visible in the tank, no measurement/quantification was performed.

Those results are in agreement with values of viscosity measured for the oils. Usually, dispersibility becomes uncertain in the range of 5 000 – 10 000 mPa.s and oils are generally not dispersible for viscosity greater than 10 000 mPa.s. Only IM-29 at 25°C exhibits lower viscosity values that could theoretically be compatible with chemical dispersion.

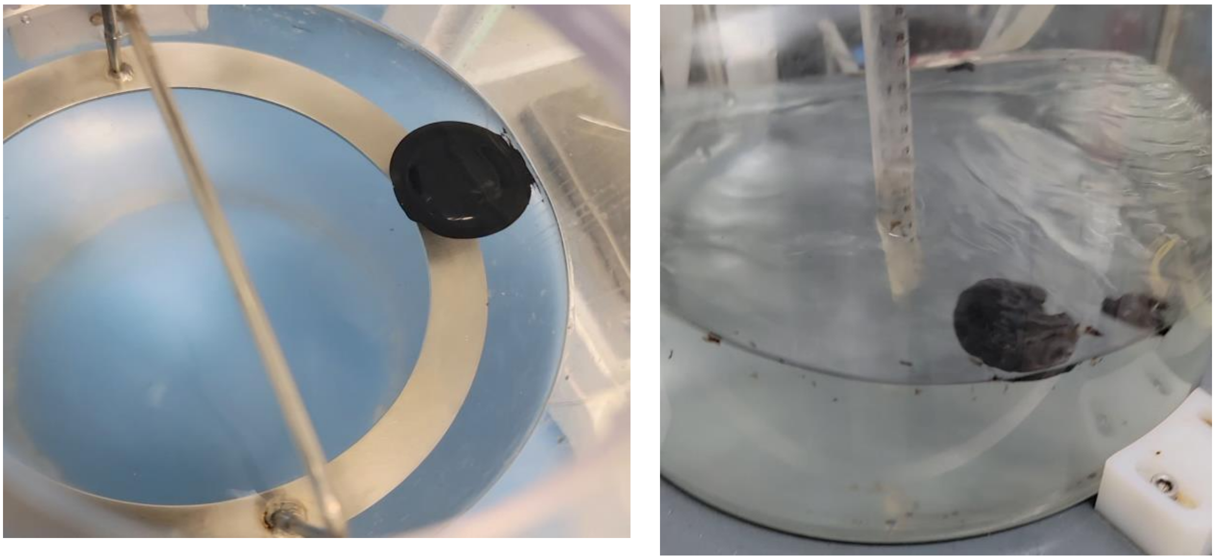


Figure 25 *Pictures taken at the end of the tests: left) Fresh IM-28, IFP test, 25°C; right) fresh IM-29, MNS test, 5°C*

Regarding the weathering experiments in the flume tank, given that the oils were not dispersible when fresh and after 6 hours of weathering, no additional tests were performed after this weathering stage.

During the first project, thirteen LSFOs were tested for dispersibility at 15°C. It appeared that some LSFOs were dispersible or possibly dispersible when fresh. When weathered at the laboratory scale, the dispersibility of the oils decreased and became uncertain or not dispersible. As a conclusion, it was thus stated that this response option was limited to some fresh oils.

- Detailed results are presented in Appendix 8

3.3 Oil fingerprinting (Task 3.5)

3.3.1 Sample Overview and Analytical Approach

A total of eighteen fuel oil samples were analysed under the IMAROS 2 project, comprising fifteen small and three large samples. The objective was to characterise and compare their chemical composition using oil fingerprinting methods. Analyses were performed by GC-MS in both Total Ion Count (TIC) and Selected Ion Monitoring (SIM) modes, following NBN EN 15522-2 (2023) standards. While all small samples underwent silica column clean-up, large samples were untreated to preserve potential FAME components. All data were interpreted using chromatographic patterns and diagnostic ratios calculated from selected molecular markers.

3.3.2 Total Ion Chromatogram (TIC) Observations

TIC plots provided an initial qualitative overview of the fuel compositions. The relative abundance of naphthalenes, lower alkanes (<C17), and waxes (>C20) varied across the dataset. However, TIC profiles were insufficient to reliably indicate sulphur content, as all combinations of these compound classes were present in both VLSFO and ULSFO samples.

Abnormal behaviour was observed in sample IM-29, which showed unusual peak patterns not consistent with other ULSFOs or VLSFOs. This prompted further investigation into possible presence of FAMEs. FAMEs were not found but the presence of cardanol could be confirmed (a phenolic compound derived from cashew nutshell liquid).

3.3.3 Biomarker Analysis

3.3.3.1 Hopanes and Oleanane/Lupane

Hopanes (m/z 191) are robust biomarkers indicating biodegradation resistance. These were prominent in most VLSFOs, while ULSFOs showed lower levels. Exceptionally, sample IM-25 showed negligible hopane content. Oleanane/lupane ratios were elevated in samples IM-24, IM-31, IM-33, IM-36, IM-37, and IM-38, suggesting these may be blended with sweet crudes, such as oils from Nigerian origin.

3.3.3.2 Aromatic Sulphur Compounds

Benzo(b)-naphto-(1,2-d) thiophene (BNT) and various methyl-dibenzothiophenes (C1–C3) are indicative of sulphur presence. These markers were consistently lower in ULSFO samples—specifically IM-21, IM-23, IM-29, IM-32, and IM-34. The BNT/hopane and dibenzothiophene/C2-phenanthrene ratios in these samples confirmed lower sulphur content. These ratios proved useful in distinguishing ULSFO from VLSFO and heavier fuel oils.

3.3.3.3 Steranes and Other Diagnostic Markers

Steranes (m/z 217), diasteranes (m/z 218), and triaromatic steranes (m/z 231) are high-boiling biomarkers typically found in viscous, less processed oils. Their low abundance in samples IM-31, IM-32, IM-34, and IM-36 aligns with their classification as ULSFOs or light blends. These markers were absent or weak in chromatograms of lower-viscosity fuels, consistent with expectations based on refining intensity and weathering susceptibility.

3.3.4 Comparative Fingerprinting and COSIWeb2 Analysis

All samples were uploaded into the COSIWeb2 database[1] for digital fingerprinting. Several samples showed high correlation coefficients (>0.99), suggesting they may originate from the same batch or supply chain. Pairwise comparisons using chromatographic overlays and ratio calculations supported these matches. Notably, strong similarities were found between IM-20/IM-22, IM-21/IM-23, and IM-32/IM-34, reinforcing the reliability of the fingerprinting methodology.

3.3.5 Summary and Implications

The combination of TIC screening, ion chromatograms, and biomarker ratio analysis proved highly effective in differentiating marine fuel oil types. Although TIC profiles were insufficient to determine sulphur content, the inclusion of sulphur-containing aromatics and hopane markers provided clarity. ULSFOs were consistently characterized by low concentrations of BNT and C1–C3 dibenzothiophenes, as well as weak sterane signals. The integration of analytical chemistry with digital matching tools like COSIWeb2 enhanced source attribution and batch comparison.

In conclusion, this study demonstrates the strength of a multi-parameter fingerprinting approach in distinguishing low-sulphur marine fuels, assessing blending practices, and supporting source identification in environmental and regulatory contexts.

- Detailed report from Task 3.5 is presented in Appendix 9

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[1] https://www.bonnagreement.org/activities/osinet/cosiweb

4 ELEMENTS FOR OSR TECHNIQUES POTENTIALLY AVAILABLE

The nineteen LSFOs tested in the frame of this project are persistent in the environment (evaporation usually below 10 % and maximum biodegradation rate, measured on three samples, of 24%). The response option “leave alone”, sometimes possible, depending on the nature of the oils, their quantities and the localisation of the spill, cannot be recommended with such oils. Density measurements indicate that the slicks should float at the water surface. Response techniques can thus be focused on the water surface compartment. However, the buoyancy of some oils could be questioned close to estuaries, characterised by a lower salinity and a high mineral load which could increase the emulsion density. Pilot-scale experiments showed that, when poured in the water, LSFOs can either remain as compact slicks or fragment in smaller granular patches. This last behaviour could lead to a more diffuse pollution.

4.1 Safety: flammability

In terms of safety, considering the samples tested, recovery operations from ships can be conducted directly after a spill given the high flash points of the fresh oils (well above 60°C). However, if the flash point of the spilt LSFO is not known, at sea operations should be carried out provided that a specific safety procedure is implemented to check for flammable hydrocarbon vapours (using gas detectors for example). This risk of ignition of the volatile vapours emitted by the oil being governed by the release characteristics and environmental conditions. The need to maintain monitoring should be regularly reconsidered.

4.2 Chemical dispersibility

Dispersibility test performed at 5°C and 25°C on three fresh oils and on moderately weathered oils from the flume tank experiments showed that the use of dispersant was not efficient. The oil slicks were visually not modified by the dispersant treatment. During the first project, dispersibility tests performed on thirteen LSFOs at 15°C showed that some fresh oils could be dispersible but the efficiency of dispersant was highly reduced on samples weathered at the laboratory scale. Based on the samples tested in the frame of the two IMAROS projects, the use of chemical dispersants seems thus limited to some fresh oils. Oil dispersibility test using a field sample is thus recommended prior to dispersant application.

4.3 Recovery

Based on the samples collected in the frame of the two IMAROS projects, recovery seems to be the only short-term response option recommended to treat an LSFO spillage.

Tests on oleophilic plates showed a good oil adhesion at the beginning of the weathering trials, slowly reducing with time. Oleophilic skimmers could thus be selected taking into account this phenomenon. However, the rapid tests performed during the weathering experiments in the flume tank do not mimic real recovery tests. Several LSFOs samples present complex visco-elastic behaviour that challenges mechanical recovery with skimmers such as the so-called "short" oil behaviour. Test in laboratory and in flume tank suggest that short oil occurs when the elastic behaviour dominates the viscous behaviour. These characteristics can be quantified with specific rheometers. As a proxy, our tests suggests that, for the tested LSFOs, there exists a correlation between the visco-elastic properties of the oil sample, its wax content and its pour-point. Specific trials dedicated to mechanical recovery are the core of the WP4 of the project. Results are presented in the Deliverable D4.2.

Trawling of the LSFO slicks was not tested in the frame of this project but the viscosity of some weathered oils (> 300 000 mPa.s) could be compatible with this technique, in complement of the mechanical recovery.

5 CONCLUSION

This work package (WP3) has enriched the database on the LSFOs and allowed a better knowledge and understanding of the physical-chemical characteristics of those products since 2020.

In-depth weathering study has been possible on three samples, with pilot scale experiments conducted at 5°C and 25°C. Those trials allow simulating the oil behaviour and weathering with time in dynamic and realistic chosen conditions. They give insights for response strategies, as detailed in section 4.

The same three large samples were used for mechanical recovery trials and experiments dedicated to shoreline responses. Those experiments are part of the WP4 and WP5, respectively, and results are available in the Deliverable D4.2 (Summary report of the WP4) and Deliverable D5.1 (Summary report of the WP5).

APPENDIX

Appendix 1

Methods of measurements at the laboratory scale

1. Physical-chemical parameters

Viscosity

The viscosity of the oil samples was measured by using an Anton Paar viscosimeter (Rheolab QC) at dedicated shear rates and the mean of the 10 first values of the 10 s^{-1} shear rate was calculated. Viscosity of the emulsions formed in the flume tank experiments were measured in the same way. The equipment was calibrated by analysing reference oils.

Note : in the case of newtonian fluids such as most of crude oils, the value of the viscosity is a constant over a wide range of shear rates. Non emulsified oils were measured at 100 s^{-1}. As far as they get emulsified, these fluids follow a non-newtonian behaviour, generally shear thinning, which mean that the viscosity decreases as the shear rate increases. Therefore, viscosities of emulsions have to be expressed with the shear rate used to perform the measurement. Viscosity of emulsions is generally measured at 10 s^{-1}, which constitutes an implicit standard in the field of oil pollution studies.

The relationship between this dynamic viscosity η, expressed in mPa.s or cP, and the corresponding kinematic viscosity υ (cSt), is as follows:

$$\upsilon = \frac{\eta}{\rho}$$

ρ represents the fluid density, generally close to 1 for water-in-oil emulsions. Therefore, the viscosity measurement can be expressed, with the same figures, either in cP, mPa.s or cSt.

Temperature-sweep (viscosity) with a temperature range from 50°C to 0°C was also measured. As detailed in Rist Sørheim et al. (2020)[2] this method uses oscillated force to avoid wax lattice disturbance, hence the viscosity development can be followed over a wide temperature range. The temperature sweep is thus considered as a robust method when comparing viscosities of high viscous oils where the pour points are considerably higher than the test temperature.

Specific gravity

The density of the fresh oil and residues samples was determined according to the ASTM method D5002 “Standard Test Method for density and relative density of crude oils by digital density analyser” (ASTM, 2013). This measurement was performed using an Anton Paar D4500 analyzer.

Pour point

The pour point was measured according to the ISO 3016 norm (equivalent to ASTM D97), on the fresh oils and also on corresponding residues obtained by distillation at 250°C.

Flash point

The flash point was measured on the fresh oils according to the NF EN ISO 13736 norm (Abel method). This measurement was performed using an Anton Paar ABA 4 Abel flash point tester.

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[2] Kristin Rist Sørheim, P. Daling, D. Cooper, I. Buist, L.G. Faksness, D. Altin, T.A. Pettersen, O.M. Bakken, 2020, Characterization of low sulfur fuel oils (LSFO) – A new generation of marine fuel oils, SINTEF Report OC2020 A-050

Asphaltenes and waxes

Asphaltenes

Asphaltenes content was measured on the residues obtained by distillation at 250°C according to the IP-143/90 norm "Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products". This gravimetric method is more reliable when applied to non-volatile oils, such as 250°C+ residues. The result is then extrapolated to the fresh oils by taking into account the evaporation rate measured following the distillation.

Waxes

Traditional method

Waxes content was measured on the same residues after the precipitation of asphaltenes (the measurement performed directly on the residue is liable to conduct to co-precipitation of asphaltenes and waxes). The protocol, which was described by Bridié *et al*. in 1980, consists in a precipitation in a mixture of MEK (methylethylketone) and DCM (dichloromethane) 1:1 at -10°C.

Comparison with a direct method

An in-depth study was conducted in order to compare different protocols leading to wax content determination. The objective was to get a better understanding of the wax composition and of the link between this chemical family and the behaviour of LSFOs once released in the water. In comparison to the traditional method described above, the direct analysis of the oil (residue 250°C+) after dilution with dichloromethane was performed (Figure 1).

Regarding the GC-FID analysis, calibration curves were established for each paraffin (alkanes with 20 or more carbon atoms). The range of interest in this study is between C20 and C40. These calibration curves then enable precise quantification. For this purpose, a certified solution containing several even-numbered alkanes ranging from C8 to C40 is used.

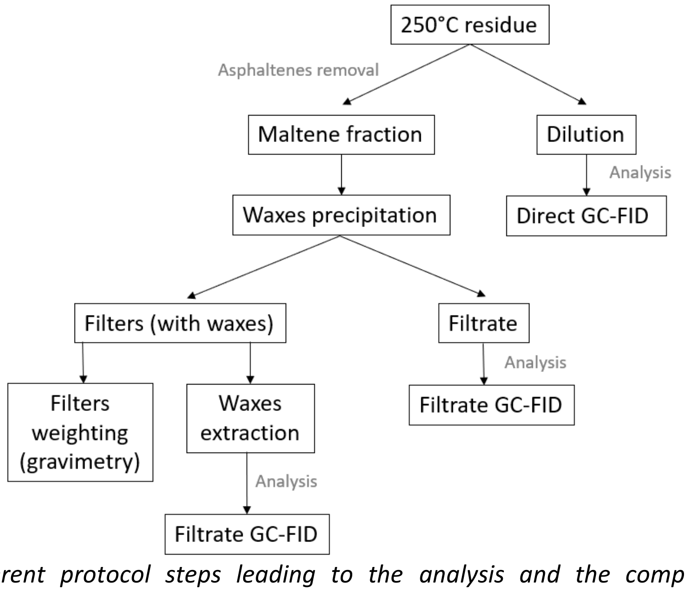

Figure 1 *Detail of the different protocol steps leading to the analysis and the comparison of different waxes determination*

Oil evaporation

To simulate the maximum evaporation at sea the fresh oils were topped at 250°C (vapour temperature). Evaporated samples are obtained by distillation according to the protocol developed at IKU/SINTEF (Norway). The distillation technique is described by Stiver and Mackay (1984) as a modified ASTM D86/82 method. The objective of this protocol is not to get the distillation curve of an oil, as intended by the ASTM method, but to obtain oil residues at given temperatures.

Detailed chemical analyses

A detailed chemical characterisation of the oils was performed:

- The boiling point curve was obtained up to 520°C using simulated distillation, in addition to the True Boiling Point (TBP) curve established during the sample preparation.
- A detailed chemical composition was obtained according to 3 different chemical analyses as presented in *Table 1*:
  - Volatiles Organic Compounds (VOCs) were quantified by Headspace-GC/MS (list of 31 molecules),
  - semi-volatiles compounds, mainly PAHs, were analysed by GC/MS (liquid injection) according to a list of 43 molecules (or groups of molecules),
  - the distribution of compounds not individually quantified was established according to their number of carbons up to C_{25} based on a GC/FID analysis.

***Table* 1** *Detailed list of the target molecules and definition of the dissolution potential*

Composition (% weight)	Individuals compounds	Analysis
C_1-C_4 (dissolved gas)	-	Crude
C_5-saturates (n-/iso-/cyclo)	*n*- pentane, isopentane, cyclopentane	HS-GC/MS
C_6- saturates (n-/iso-/cyclo)	*n*-hexane, methylpentane (2), methylcyclopropane, cyclohexane	
C_7- saturates (n-/iso-/cyclo)	*n*-heptane, dimethylpentane, methylhexane, methylcyclohexane	
C_8- saturates (n-/iso-/cyclo)	*n*-octane, methylheptane	HS-GC/MS
C_9- saturates (n-/iso-/cyclo)	*n*-nonane	HS-GC/MS
Benzene	benzene	HS-GC/MS
C_1-Benzene	Toluene	
C_2-Benzenes	o-, m-, p-xylène, ethylbenzene	
C_3-Benzenes	methylethylbenzenes (3), trimethylbenzenes (3), propylbenzene	
C_4 & C_5-Benzenes	n-butylbenzene, tetraméthylbenzene, n-pentylbenzene	
C_{10}- saturates (n-/iso-/cyclo)	No quantification of individual compounds	GC/FID
C_{11}-C_{12} (total saturates + aromatics)		
C_{13}-C_{14} (total saturates + aromatics)		
C_{15}-C_{16} (total saturates + aromatics)		
C_{17}-C_{18} (total saturates + aromatics)		
C_{19}-C_{20} (total saturates + aromatics)		
C_{21}-C_{25} (total saturates + aromatics)		
C_{25}+ (total)		
Naphthalenes 1 (C_0-C_1 alkylated)	N, N1	GC/MS
Naphthalenes 2 (C_2-C_3 alkylated)	N2, N3	GC/MS
PAHs 1 (medium solubility)	BT, BT1, BT2, BT3, BT4, N4, B, ANA, ANY, F, F1, P, A, P1, D, D1	
PAHs 2 (low solubility)	F_2, F_3, P_2, P_3, P_4, D_2, D_3, D_4, FL, PY, FL_1, FL_2, FL_3, BAA, C, C_1, C_2, C_3, BBF, BKF, BEP, BAP, PE, DBA, IN, BPE	GC/MS
Phenols (C_0-C_4)	Phenol, C_1 to C_4-Phenols	GC/MS

☐	High dissolution potential
🟦	Medium dissolution potential
🟧	Low dissolution potential

Co-funded by the European Union

Oil Composition – Quantification of Individual Components (PAHs, n-alkanes)

The PAHs and *n*-alkanes concentrations were measured on the crude oil in its initial state. 10 mg of the oil sample were spiked with internal standards (perdeutarated PAHs and eicosane). Compounds were then analyzed by Gas Chromatography coupled to Mass Spectrometry (GC-MS). The GC was an HP 7890 series II (Hewlett-Packard. Palo Alto. CA. USA) equipped with a Multi Mode Injector (MMI) used in the pulsed splitless mode (Pulse Splitless time: 1 min. Pulse Pressure: 15 psi). The injector temperature was maintained at 300 °C. The interface temperature was 300°C. The GC temperature gradient was: from 50°C (1 min) to 320°C (20 min) at 3°C/min. The carrier gas was Helium at a constant flow of 1 ml/min. The capillary column used was a HP 5-ms (Hewlett-Packard.Palo Alto. CA. USA): 50 m x 0.25 mm ID x 0.25 µm film thickness. The GC was coupled to a HP 7000 triple quadripole used in the Electronic Impact mode (Electronic Impact: 70 eV. voltage: 2000V). PAHs and *n*-alkanes quantifications were done using Single Ion Monitoring mode with respectively the molecular ion of each compound and a common fragment (generally m/z = 57) at a minimum of 2 cycles/s.

PAHs and *n*-alkanes were quantified relatively to the perdeuterated PAHs and eicosane introduced at the beginning of the sample preparation procedure.

Oil Composition – Quantification of Individual Components (COVs)

10 mg of the oil sample and 10 mL of reverse-osmosis purified water are added in a 20 mL flask. which is then spiked with 50 µL of the methanolic solution of internal standards (6 perdeuterated alkanes, from C_5 to C_{10}, and C_0 to C_4- perdeuterated benzenes). The flask is then closed with a cap.

Headspace (HS) parameters

Following an incubation time of 5 minutes at 60°C (stirring at 500 rpm with a 1 second break every 60 seconds), 1000 µL of the air is sampled and injected in the GC-MS system.

GC-MS analysis

The analysis was performed by Gas Chromatography coupled to Mass Spectrometry (GC/MS). The GC was an HP 7890N (Hewlett-Packard. Palo Alto. CA. USA) equipped with a Cooled Injection System CIS-4 (Gerstel. Switzerland). The injection was performed in splitless mode (CIS temperature: 225°C). The GC temperature program was: from 35°C (5 min) to 80°C (0 min) at 11°C/min, then 250°C (0 min) at 16°C/min. The carrier gas was helium at a constant flow of 1 mL/min. The capillary column used was a RXi-624 Sil MS (Restek. Bellefonte. PA. USA): 30 m x 0.25 mm ID x 1.4 µm film thickness. The GC was coupled to an HP 5975 Mass Selective Detector (MSD) (Electronic Impact: 70 eV. voltage: 1200 V). The interface temperature was 230°C. Alkanes and monoaromatics quantifications were done using Single Ion Monitoring mode at a minimum of 2 cycles/s.

Monoaromatics and and alkanes were quantified relatively to the perdeuterated linear alkanes, benzene and alkylated benzenes introduced at the beginning of the sample preparation procedure.

Oil Composition – Distribution according to the number of carbon atoms

10 mg of the oil were solubilized by 10 mL of CS_2. No purification was performed prior to the analysis. The analysis was performed by high temperature gas chromatography coupled to a flame ionization detector (HTGC-FID). The GC was an HP 7890N (Hewlett-Packard. Palo Alto. CA. USA) equipped with an “on-column” injector. The injector program was: from 50°C to 400°C at 6°C/s. The oven temperature program: from 50°C (1 min) to 420°C (10 min) at 15°C/min. The carrier gas was hydrogen at a constant flow (15 mL/min). The capillary column used was a DB-HT Simdist (100% diméthylsiloxanne): 5 m x 0.53 mm x 0.15 µm film thickness. The chromatograph was coupled to a FID detector maintained at 425°C. The volume injected was 1 µL.

Oil biodegradability / Simulation of the biodegradation process

A protocol adapted from De Mello et al. (2007) was used to simulate the biodegradation process. Around 10 mg of the fresh crude oil, dissolved in 50 µL of acetone, was biodegraded at the laboratory scale, in a 125 mL glass bottle containing 100 mL of seawater. Nutrients were added to the water, in agreement with the French norm NFT 90-347 « Essais des eaux – Produits dispersants - Evaluation en milieu aqueux de l'action inhibitrice sur la biodégradabilité du pétrole ». This method is applied within the French dispersant approval procedure, and aims at assessing the biodegradation of dispersed oil. The hydrocarbons-degrading microorganisms came from a chronically contaminated site. The bottles were loosely plugged with autoclaved cotton wool, placed onto a shaker table, in the dark and agitated for 28 days. The residual oil was extracted and purified prior to analysis.

2. Additional measurements from flume tank experiments

Oil Adhesion

The evolution of oil adhesion on oleophilic plates, such as PVC plates (10 x 10 cm), provides useful data for assessing the performance of oleophilic skimmers in oil recovery operations. These plates simulate the surface interaction between the oil and recovery equipment, allowing for the measurement of how much oil adheres to the material over time.

Density

The density of the weathered samples collected at 5°C was determined using a gas pycnometer Anton Paar Ultrapyc 5000. This instrument was used because of the lack of fluidity of the oils that made impossible the use of the densimeter.

Emulsion stability

The stability ratio is usually calculated by comparing the water content of the emulsion sample after 24 hour settling time to its initial water content. Emulsions with a stability ratio of 0.8 or higher are considered relatively stable, which corresponds to the settling of approximately half the water in an emulsion with an 80% water content. This method was used at 25°C.

At 5°C, due to the impossibility to fill the graduated cylinder with the semi-solid oils, a simplified protocol was used. A known mass of weathered oil was put in a small crystallizer and the quantity of free water after 24 hours was evaluated.

SARA determination (Saturates, Aromatics, Resins, Asphaltenes)

Typically, saturates and aromatics tend to volatilize, while resins and asphaltenes are considered non-volatile compounds, leading to an increase in their relative concentration as the oil weathers. However, the cut between polar compounds (resins/asphaltenes) and aromatics is very sensitive to weathering. The distinction between resins and asphaltenes is not always clear, as they form a continuum with no sharply defined boundary. Therefore, when evaluating various degrees of weathering, it is more relevant to consider the sum of resins and asphaltenes rather than treating them as separate groups.

The evolution of the oil composition was assessed by fractionation into 4 chemical families (saturates, aromatics, resins and asphaltenes). Asphaltenes were precipitated in *n*-pentane and were filtered on a glass fibre filter. The maltene fraction was separated on a silica-alumina column. Saturates were eluted with *n*-pentane, aromatics with a *n*-pentane/dichloromethane 80/20 mixture, and resins with a mixture methanol/ dichloromethane 50/50. The various fractions were weighed after evaporation of the solvent and stabilization of the masses. The mass balanced was calculated assuming that saturates and aromatics evaporates similarly whereas resins and asphaltenes do not evaporate.

Evaporation rate

Method 1

The saturate and aromatic fractions were analysed using gas chromatography. Chromatograms of these volatile compounds at different weathering times show the evolution of their distribution. The lighter molecules disappear progressively and the quantitative analysis of samples compared to the initial oil can give the evaporation rate. These analyses performed at various weathering times provide an assessment of the kinetics of evaporation. Considering the low rate of dissolution and natural dispersion compared to evaporation, water soluble fractions were not taken into account to assess the proportion of oil that evaporated.

To assess the evaporation rate of the whole oil, a calibration was established in the laboratory. Different samples of the initial oil were artificially evaporated and then analysed (*table 2*). This qualitative determination led to the calculation of a Weathering Index (WI), proposed by Wang and Fingas (1994), which was plotted versus the evaporation rate. The calibration curve established that way was then applied to real samples to get their evaporation rates.

The Weathering Index is defined in the general following equation:

$$WI = \frac{L_1 + L_2 + L_3 + L_4}{H_1 + H_2 + H_3 + H_4}$$

L_1, L_2, L_3 and L_4 represent components that volatilize while H_1, H_2, H_3 and H_4 represent the non-volatile compounds in the oil (the choice of these target compounds depends on the initial composition of the oil; in our case. the light compounds were the linear alkanes $n\mathrm{C}_{11}$ to $n\mathrm{C}_{14}$. while the heavy molecules were $n\mathrm{C}_{25}$ to $n\mathrm{C}_{28}$). Consequently, as the oil evaporates, the Weathering Index decreases and a calibration curve similar to the one presented in *figure 1* can be obtained:

Table 2 *Example of characterisation of distilled samples of one crude oil*

	Sample 1	**Sample 2**	**Sample 3**
Distillation temperature (°C)	150	200	250
Evaporation rate (%)	12.1	20.7	29.7
Density @ 1°C / 10°C	0.908 / 0.901	0.921 / 0.913	0.933 / 0.925

The calculation of the maximum evaporation rate can also be checked by comparing the oil density at its final weathering state with samples obtained in the laboratory and characterized by their evaporation rate (measured by gravimetry).

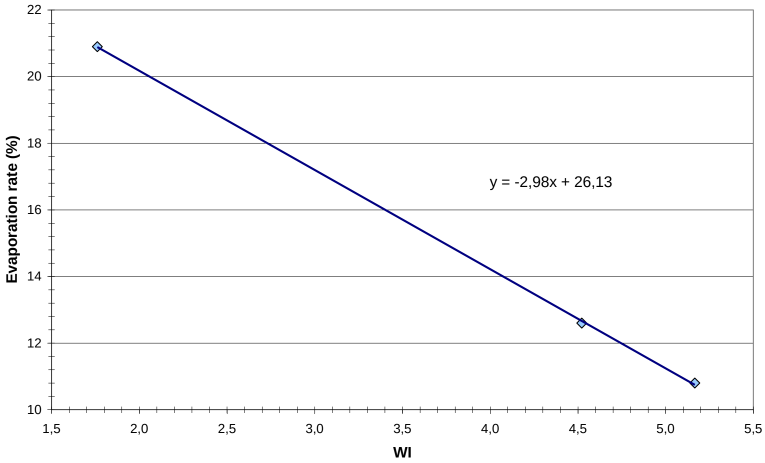

Figure 2 *Calibration curve established to measure the evaporation rate of weathered oils*

Method 2

The analysis of weathered samples collected from the flume was performed using high-temperature gas chromatography equipped with a flame ionization detector (HT-GC/FID). The total chromatographic area was calculated and compared to that of the initial oil, allowing for the assessment of evaporation losses. Results were further validated by analysing artificially evaporated samples with known evaporation rates. These calibration samples also facilitated the conversion of evaporation rates between volume and weight units.

Chemical dispersibility

Different dispersibility tests can be conducted, each protocol having its specificity. However, all of them were initially designed for the comparison of dispersants products (in particular the WSL and IFP tests). Some of them can be used for the assessment of an oil dispersibility in real conditions, particularly the IFP and MNS tests which were correlated to field or pilot scale experiments.

Except in case of obvious no dispersion, all the dispersibility tests are carried out twice (duplicates), and the relative differences of results between two similar tests have not to differ more than 14% (in that case, a third test has to be performed). However, this criterion cannot be strictly applied for efficiencies lower than 40%, and in that case, results have not to differ more than 3% compared to the mean value (10% ± 2% for example).

The chemical dispersibility of the oil samples was measured at 5 °C and 25°C and using the “model” dispersant. The IFP and MNS protocols were followed.

IFP Dispersibility

The chemical dispersibility was measured using the IFP test method (NF 90-345 French Standard). This protocol is characterized by a low energy and a dilution process from a main tank to a dilution tank was applied. The test equipment was checked with Cedre reference oil (FOREF: mixture of Heavy Fuel Oil and Arabian Light topped at 110°C, viscosity of 1 200 ± 100 mPa.s at 20°C) and with the “model” dispersant formulated at Cedre. Under these test conditions, the efficiency has to be 75 ± 3%. The oil, dispersed in the water column and entrained in the dilution beaker, was extracted by dichloromethane and the concentration measured by spectrophotometry at 390 nm.

The IFP test is representative of medium conditions, for a sea state around 2-3. Moreover, it provides information not only on the efficiency of the dispersant, but also on the quality of the dispersion due to the dilution process.

In order to calibrate the experimental design, additional dispersibility tests were carried out with the same dispersant using the French procedure for dispersant approval (test performed at 20°C; reference oil: FOREF, mixture of Heavy Fuel Oil and Arabian Light topped at 110°C, viscosity of 1000 mPa.s at 20°C). Under these conditions, the reference value is 76% ± 3 and the results of the calibration have to be within this range.
Oil was extracted using dichloromethane and the concentration was measured by UV spectrophotometry at 580 nm.

Table 3 *Composition of the model dispersant*

Denomination	% weight
Dipropylene-glycol-n-butyl-ether	18.5
Span-80	6.5
Tween-80	12.9
Tween-85	19.1
Aerosol-OT-75 (14/05/14)	27.8
Exxsol-D80	15.2
Total	100.0

MNS Dispersibility

It is a high-energy test, where air flow is controlled by measuring pressure drop. Various settings are possible, and a standard value of 25 mm was applied in this study. The MNS test simulates higher energy conditions, providing insights into the maximum viscosity of an emulsion that could be treated with dispersants. The 25 mm setting is representative of medium-strong conditions, similar to a sea state of around 4.

This methodology is described in the paper published by Mackay and Szeto (1981)[3].

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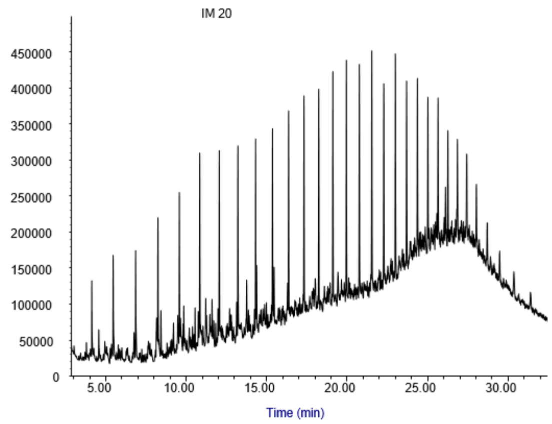
[3] Donald Mackay and Foon Szeto (1981) THE LABORATORY DETERMINATION OF DISPERSANT EFFECTIVENESS: METHOD DEVELOPMENT AND RESULTS. International Oil Spill Conference Proceedings: March 1981, Vol. 1981, No. 1, pp. 11-17.

Appendix 2

Chromatograms (from GC/FID analysis)

Co-funded by the European Union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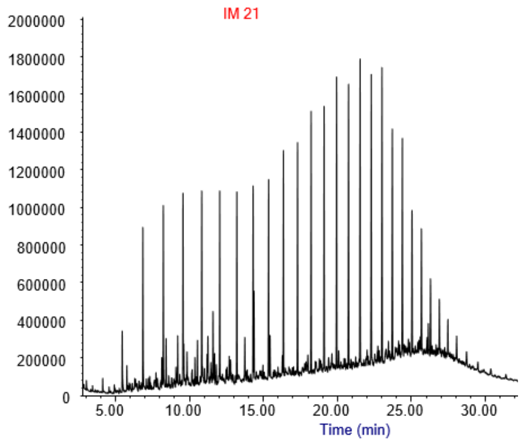
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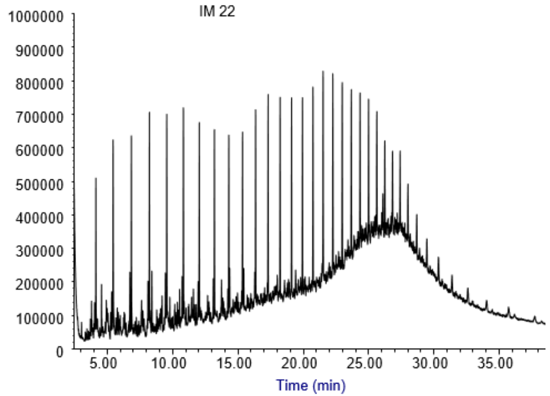
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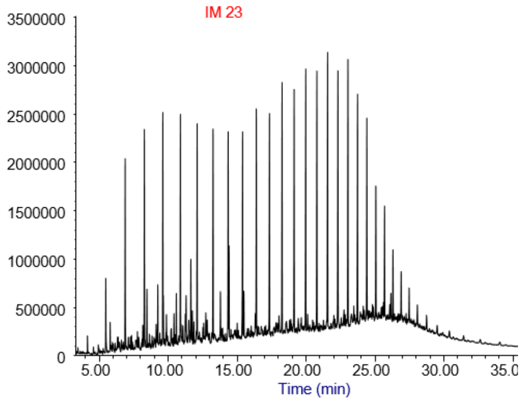
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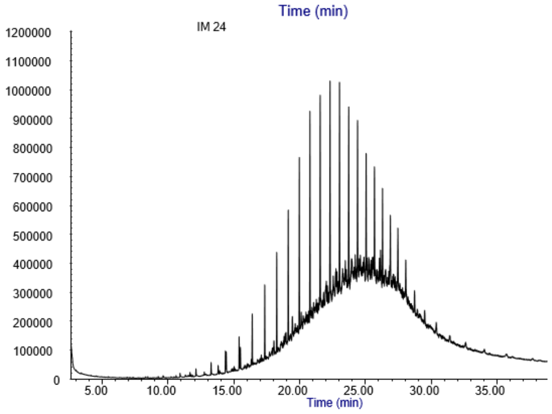
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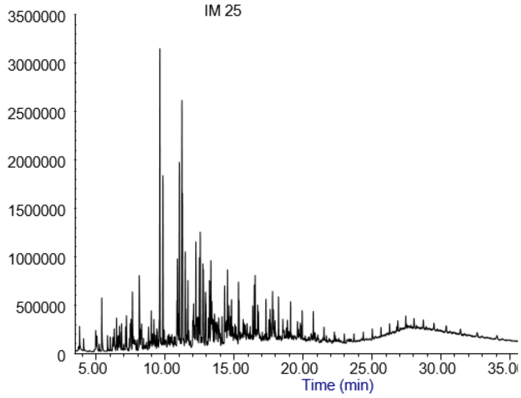
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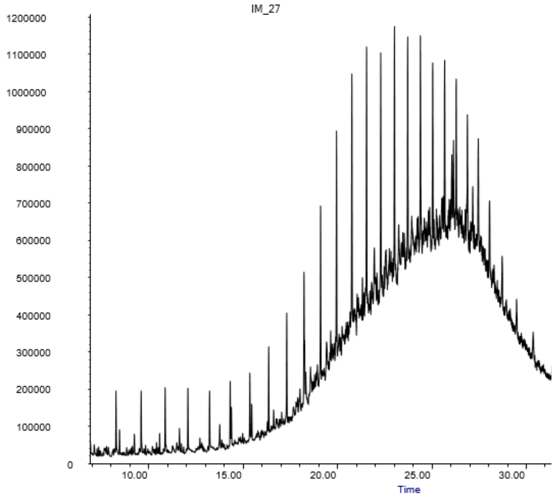
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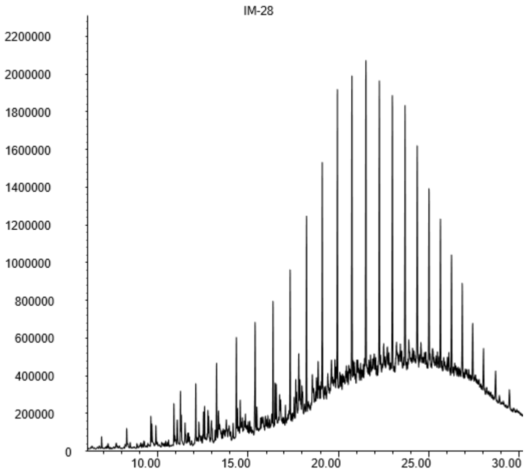
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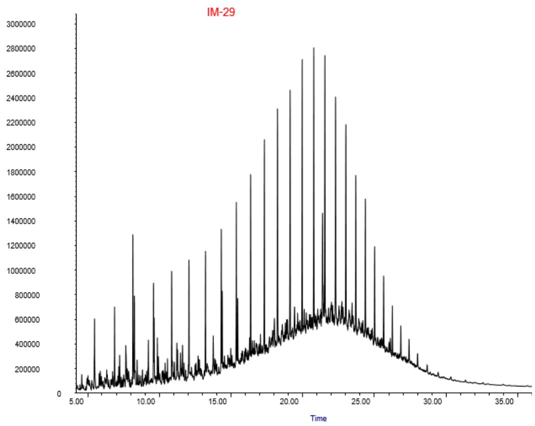
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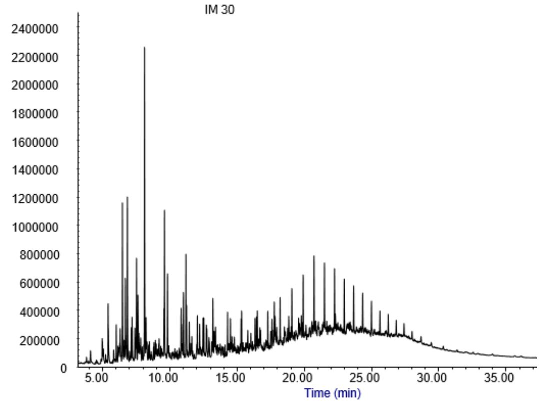
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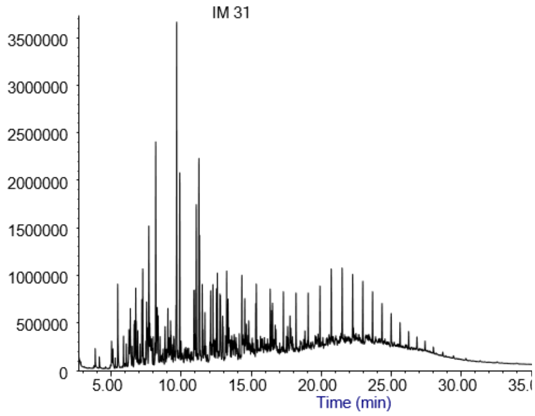
Time (min)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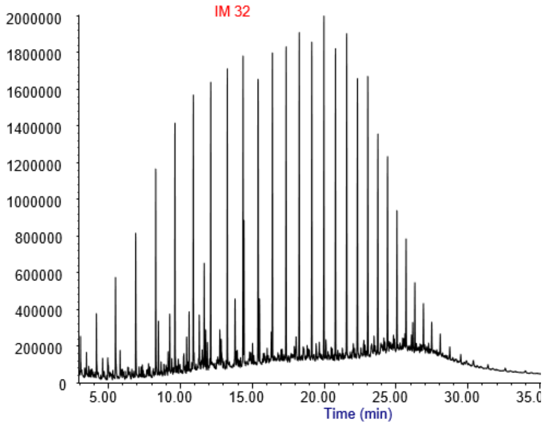
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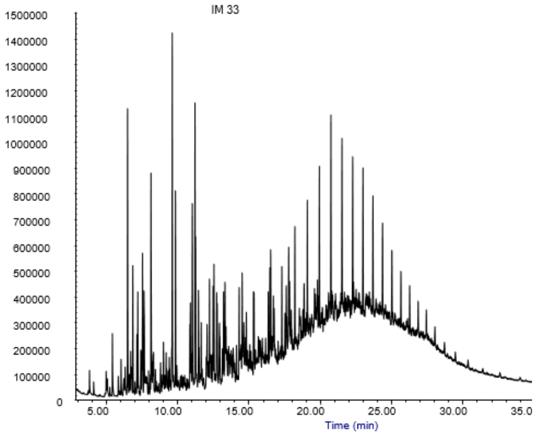
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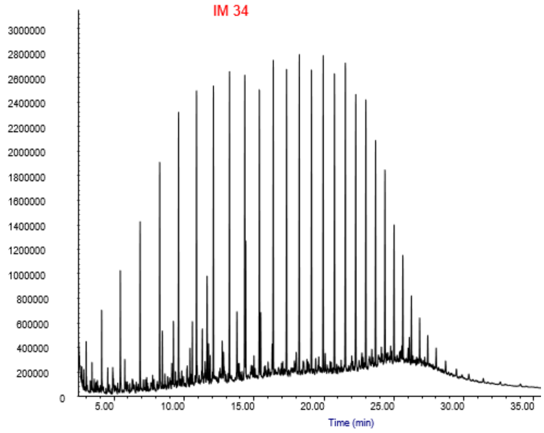
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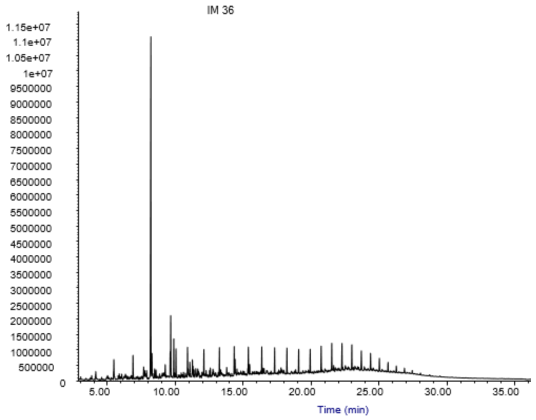
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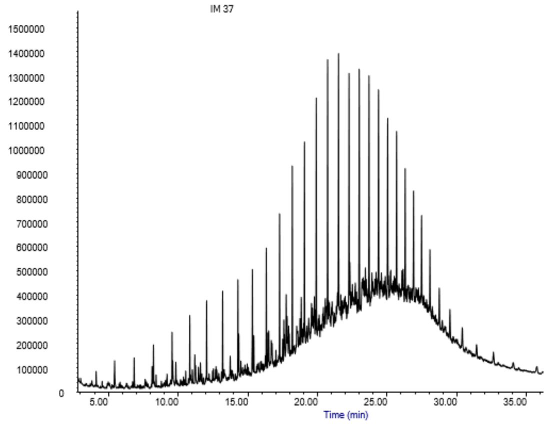
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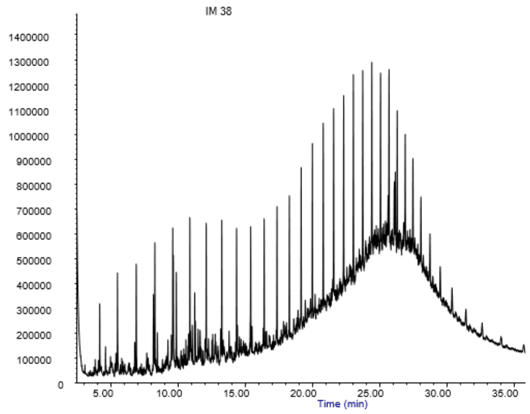
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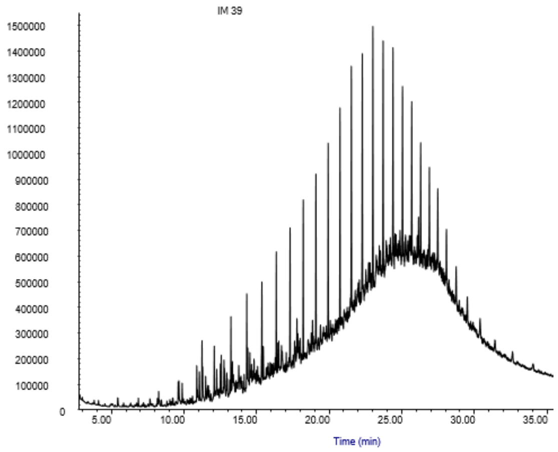
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Time (min)

Appendix 3

OSCAR characterisation of the oils

Table 1 *Results of the OSCAR characterisation of the fresh samples (Individual compounds)*

Composition (% weight)	IM-20	IM-21	IM-22	IM-23	IM-24	IM-25	IM-27
C_1-C_4 (dissolved gas)	0.03	0.00	0.04	0.00	0.01	0.00	0.00
C_5-saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_6- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_7- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_8- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_9- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Benzene	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_1-Benzene	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C_2-Benzenes	0.00	0.00	0.00	0.00	0.00	0.01	0.00
C_3-Benzenes	0.00	0.00	0.00	0.00	0.00	0.03	0.00
C_4 & C_5-Benzenes	0.00	0.00	0.00	0.00	0.00	0.01	0.00
C_{10}- saturates (n-/iso-/cyclo)	0.86	0.87	1.24	0.67	0.33	0.72	0.45
C_{11}-C_{12} (total saturates + aromatics)	0.83	2.10	1.44	2.25	0.05	0.82	0.50
C_{13}-C_{14} (total saturates + aromatics)	1.30	2.93	1.76	3.50	0.18	1.33	0.52
C_{15}-C_{16} (total saturates + aromatics)	1.69	3.42	1.79	4.03	0.32	4.10	0.51
C_{17}-C_{18} (total saturates + aromatics)	1.85	3.29	1.85	3.85	0.70	3.13	0.61
C_{19}-C_{20} (total saturates + aromatics)	2.19	3.67	2.19	4.11	1.53	2.76	1.17
C_{21}-C_{25} (total saturates + aromatics)	5.95	10.16	5.93	10.48	9.95	5.39	7.72
C_{25}+ (total)	84.35	72.55	83.05	70.00	86.38	76.35	88.05
Naphthalenes 1 (C_0-C_1 alkylated)	0.16	0.02	0.17	0.28	0.28	0.14	0.03
Naphthalenes 2 (C_2-C_3 alkylated)	0.14	0.26	0.07	0.23	0.01	1.86	0.02
PAHs 1 (medium solubility)	0.20	0.28	0.09	0.24	0.03	1.41	0.05
PAHs 2 (low solubility)	0.45	0.44	0.38	0.35	0.22	1.94	0.37
Phenols (C_0-C_4)	-	-	-	-	-	-	-

☐	High dissolution potential
🟦	Medium dissolution potential
🟧	Low dissolution potential

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Table 1 (cont.) *Results of the OSCAR characterisation of the fresh samples (Individual compounds)*

Composition (% weight)	IM-28	IM-29	IM-30	IM-31	IM-32	IM-33
C_1-C_4 (dissolved gas)	0.02	0.02	0.04	0.00	0.00	0.00
C_5-saturates (n-/iso-/cyclo)	0.02	0.00	0.00	0.00	0.00	0.00
C_6- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00	0.00
C_7- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.01	0.00
C_8- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.01	0.00
C_9- saturates (n-/iso-/cyclo)	0.00	0.01	0.00	0.00	0.01	0.00
Benzene	0.00	0.00	0.00	0.00	0.00	0.00
C_1-Benzene	0.00	0.00	0.00	0.00	0.00	0.00
C_2-Benzenes	0.00	0.01	0.00	0.01	0.00	0.00
C_3-Benzenes	0.00	0.02	0.04	0.06	0.00	0.00
C_4 & C_5-Benzenes	0.00	0.01	0.01	0.02	0.00	0.00
C_{10}- saturates (n-/iso-/cyclo)	0.49	1.79	1.03	1.19	1.63	0.36
C_{11}-C_{12} (total saturates + aromatics)	0.21	3.44	3.67	4.64	1.89	1.55
C_{13}-C_{14} (total saturates + aromatics)	0.33	3.73	2.34	3.94	3.08	1.15
C_{15}-C_{16} (total saturates + aromatics)	1.21	4.67	2.69	5.63	3.83	2.18
C_{17}-C_{18} (total saturates + aromatics)	1.84	5.94	2.15	4.56	3.84	2.33
C_{19}-C_{20} (total saturates + aromatics)	3.08	8.24	2.89	4.32	4.03	2.98
C_{21}-C_{25} (total saturates + aromatics)	14.65	27.42	11.12	11.25	9.44	11.09
C_{25}+ (total)	75.68	43.39	69.11	58.83	71.72	74.71
Naphthalenes 1 (C_0-C_1 alkylated)	0.19	0.00	0.77	0.01	0.06	0.36
Naphthalenes 2 (C_2-C_3 alkylated)	0.29	0.31	0.83	1.85	0.12	0.77
PAHs 1 (medium solubility)	0.47	0.39	0.91	1.44	0.10	0.80
PAHs 2 (low solubility)	1.52	0.61	2.40	2.25	0.23	1.72
Phenols (C_0-C_4)	-	-	-	-	-	-

⬜	High dissolution potential
🟦	Medium dissolution potential
🟧	Low dissolution potential

Co-funded by the
European Union

Table 1 (cont.) *Results of the OSCAR characterisation of the fresh samples (Individual compounds)*

Composition (% weight)	IM-34	IM-36	IM-37	IM-38	IM-39
C_1-C_4 (dissolved gas)	0.00	0.00	0.00	0.19	0.00
C_5-saturates (n-/iso-/cyclo)	0.01	0.01	0.01	0.00	0.01
C_6- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00
C_7- saturates (n-/iso-/cyclo)	0.01	0.00	0.00	0.00	0.00
C_8- saturates (n-/iso-/cyclo)	0.00	0.00	0.00	0.00	0.00
C_9- saturates (n-/iso-/cyclo)	0.01	0.00	0.00	0.00	0.00
Benzene	0.00	0.00	0.00	0.00	0.00
C_1-Benzene	0.00	0.00	0.00	0.00	0.00
C_2-Benzenes	0.00	0.00	0.00	0.00	0.00
C_3-Benzenes	0.00	0.01	0.00	0.00	0.00
C_4 & C_5-Benzenes	0.00	0.00	0.00	0.00	0.00
C_{10}- saturates (n-/iso-/cyclo)	0.86	0.43	0.57	0.49	0.31
C_{11}-C_{12} (total saturates + aromatics)	1.84	3.68	0.48	0.52	0.17
C_{13}-C_{14} (total saturates + aromatics)	3.72	3.31	0.60	1.46	0.18
C_{15}-C_{16} (total saturates + aromatics)	4.60	3.49	0.76	1.80	0.88
C_{17}-C_{18} (total saturates + aromatics)	4.60	3.45	0.82	1.64	1.43
C_{19}-C_{20} (total saturates + aromatics)	4.72	3.77	1.67	1.96	2.03
C_{21}-C_{25} (total saturates + aromatics)	10.83	8.85	9.41	7.51	7.64
C_{25}+ (total)	66.98	69.43	83.73	82.54	86.39
Naphthalenes 1 (C_0-C_1 alkylated)	1.36	1.77	0.02	0.89	0.01
Naphthalenes 2 (C_2-C_3 alkylated)	0.12	0.42	0.14	0.18	0.19
PAHs 1 (medium solubility)	0.11	0.48	0.28	0.24	0.25
PAHs 2 (low solubility)	0.23	0.90	1.51	0.58	0.51
Phenols (C_0-C_4)	-	-	-	-	-

☐	High dissolution potential
🟦	Medium dissolution potential
🟧	Low dissolution potential

Co-funded by the European Union

Appendix 4

PAH quantification

Co-funded by the European Union

Concentration (µg/g)	Abbrev.	IM-20	IM-21	IM-22
Benzo(b)thiophene	BT	5,3	3,0	0,10
C1-benzo(b)thiophenes	BT1	9,1	0,0	0,1
C2-benzo(b)thiophenes	BT2	12,1	0,0	0,3
C3-benzo(b)thiophenes	BT3	26,5	0,0	0,4
C4-benzo(b)thiophenes	BT4	16,7	0,0	0,2
Naphtalene	N	629,9	66,5	8,4
C1-Naphtalenes	N1	985,8	146,1	14,7
C2-Naphtalenes	N2	786,6	1386,7	4,2
C3-Naphtalenes	N3	647,5	1163,3	5,1
C4-Naphtalenes	N4	431,7	756,4	3,7
Biphenyl	B	147,3	285,1	0,0
Acenaphtylene	ANY	28,9	53,5	0,0
Acenaphtene	ANA	76,8	114,7	0,1
Fluorene	F	179,1	285,1	0,5
C1-Fluorenes	F1	377,7	418,5	2,2
C2-Fluorenes	F2	460,8	703,5	4,4
C3-Fluorenes	F3	217,4	387,7	2,7
Phenanthrene	P	227,1	359,8	0,9
Anthracene	A	22,7	18,2	0,1
C1-phenanthrenes/anthracenes	P1	377,4	490,3	3,0
C2-phenanthrenes/anthracenes	P2	472,4	647,6	3,9
C3-phenanthrenes/anthracenes	P3	293,9	473,9	3,0
C4-phenanthrenes/anthracenes	P4	240,4	236,5	1,9
Dibenzothiophene	D	11,8	4,4	0,1
C1-dibenzothiophenes	D1	60,2	58,7	0,7
C2-dibenzothiophenes	D2	87,6	33,5	0,9
C3-dibenzothiophenes	D3	66,2	27,3	0,8
C4-dibenzothiophenes	D4	47,6	14,6	0,5
Fluoranthene	FL	20,3	20,5	0,1
Pyrene	PY	109,3	88,7	1,0
C1-fluoranthenes/pyrenes	FL1	414,8	371,2	4,6
C2-fluoranthenes/pyrenes	FL2	469,7	415,3	6,5
C3-fluoranthenes/pyrenes	FL3	363,5	346,9	5,3
Benzo[a]anthracene	BA	97,2	62,9	1,1
Chrysene	C	206,9	103,4	2,7
C1-chrysenes	C1	368,5	188,3	5,1
C2-chrysenes	C2	333,7	201,6	4,3
C3-chrysenes	C3	189,6	115,2	2,2
Benzo[b+k]fluoranthene	BBF	21,8	7,5	0,3
Benzo[e]pyrene	BEP	98,5	24,2	1,5
Benzo[a]pyrene	BAP	55,8	13,0	0,8
Perylene	PE	18,0	0,0	0,2
Indeno(1,2,3-cd)pyrene	IN	25,9	15,4	0,4
Dibenz(a,h)anthracene	DBA	24,4	22,8	0,5
Benzo(g,h,i)perylene	BPE	53,9	10,4	0,7
TOTAL (µg/g)		**9818,2**	**10142,5**	**100,0**
N - N4		35%	35%	36%
BT - C3		62%	64%	60%
B(b+k)F - BPE		3%	1%	4%

Concentration (µg/g)	Abbrev.	IM-23	IM-24	IM-25
Benzo(b)thiophene	BT	9,6	0,0	0,0
C1-benzo(b)thiophenes	BT1	-0,1	0,0	183,9
C2-benzo(b)thiophenes	BT2	-13,7	0,0	32,8
C3-benzo(b)thiophenes	BT3	-9,9	0,0	22,7
C4-benzo(b)thiophenes	BT4	-20,3	0,0	0,0
Naphtalene	N	1038,5	1283,2	701,5
C1-Naphtalenes	N1	1765,1	1511,3	662,7
C2-Naphtalenes	N2	1206,0	63,3	10546,6
C3-Naphtalenes	N3	1102,4	86,3	8014,2
C4-Naphtalenes	N4	708,7	71,1	4425,4
Biphenyl	B	215,3	0,0	336,8
Acenaphtylene	ANY	34,8	0,0	0,0
Acenaphtene	ANA	90,2	0,0	468,7
Fluorene	F	225,0	13,5	852,0
C1-Fluorenes	F1	361,9	56,1	2282,4
C2-Fluorenes	F2	662,3	120,9	2954,4
C3-Fluorenes	F3	355,5	90,1	1416,5
Phenanthrene	P	306,6	39,3	1686,2
Anthracene	A	10,9	0,0	299,3
C1-phenanthrenes/anthracenes	P1	402,1	118,2	3059,0
C2-phenanthrenes/anthracenes	P2	496,9	198,4	2872,0
C3-phenanthrenes/anthracenes	P3	350,3	239,0	1699,8
C4-phenanthrenes/anthracenes	P4	209,5	196,3	681,0
Dibenzothiophene	D	5,3	8,5	90,9
C1-dibenzothiophenes	D1	46,4	29,1	314,8
C2-dibenzothiophenes	D2	39,4	42,1	260,6
C3-dibenzothiophenes	D3	22,2	35,9	133,0
C4-dibenzothiophenes	D4	-6,0	138,9	60,5
Fluoranthene	FL	16,5	0,0	54,8
Pyrene	PY	69,3	0,0	635,8
C1-fluoranthenes/pyrenes	FL1	234,0	110,2	2571,2
C2-fluoranthenes/pyrenes	FL2	312,3	253,1	2520,3
C3-fluoranthenes/pyrenes	FL3	231,3	334,5	1630,2
Benzo[a]anthracene	BA	43,2	37,6	120,1
Chrysene	C	96,9	49,9	204,9
C1-chrysenes	C1	125,0	104,1	571,3
C2-chrysenes	C2	135,6	119,7	620,0
C3-chrysenes	C3	87,8	112,5	387,2
Benzo[b+k]fluoranthene	BBF	8,7	1,0	18,8
Benzo[e]pyrene	BEP	29,1	7,9	88,0
Benzo[a]pyrene	BAP	14,1	9,4	65,8
Perylene	PE	9,2	14,9	32,2
Indeno(1,2,3-cd)pyrene	IN	13,5	0,0	64,8
Dibenz(a,h)anthracene	DBA	21,2	26,0	26,4
Benzo(g,h,i)perylene	BPE	6,8	4,7	277,6
TOTAL (µg/g)		**11069,4**	**5527,4**	**53947,1**
N - N4		53%	55%	45%
BT - C3		46%	44%	54%
B(b+k)F - BPE		1%	1%	1%

Concentration (µg/g)	Abbrev.	IM-27	IM-28	IM-29
Benzo(b)thiophene	BT	0,0	0,0	0,0
C1-benzo(b)thiophenes	BT1	0,0	7,5	27,8
C2-benzo(b)thiophenes	BT2	0,0	5,3	0,0
C3-benzo(b)thiophenes	BT3	0,0	14,4	0,0
C4-benzo(b)thiophenes	BT4	0,0	0,0	0,0
Naphtalene	N	86,4	812,5	0,0
C1-Naphtalenes	N1	175,1	1095,9	19,2
C2-Naphtalenes	N2	125,6	1373,4	1861,7
C3-Naphtalenes	N3	118,2	1563,1	1232,5
C4-Naphtalenes	N4	79,4	1083,6	758,6
Biphenyl	B	0,0	12,3	365,0
Acenaphtylene	ANY	0,0	0,0	17,6
Acenaphtene	ANA	0,0	50,7	261,6
Fluorene	F	26,8	181,0	392,8
C1-Fluorenes	F1	99,0	684,2	831,5
C2-Fluorenes	F2	251,8	1080,3	1193,7
C3-Fluorenes	F3	166,9	753,7	708,5
Phenanthrene	P	72,1	660,0	492,1
Anthracene	A	7,2	84,5	73,6
C1-phenanthrenes/anthracenes	P1	189,4	1464,6	639,1
C2-phenanthrenes/anthracenes	P2	291,7	1930,0	690,8
C3-phenanthrenes/anthracenes	P3	224,5	1360,0	454,1
C4-phenanthrenes/anthracenes	P4	233,9	830,8	311,7
Dibenzothiophene	D	6,5	109,1	12,1
C1-dibenzothiophenes	D1	28,2	376,8	71,1
C2-dibenzothiophenes	D2	35,8	473,6	72,4
C3-dibenzothiophenes	D3	40,2	335,3	46,8
C4-dibenzothiophenes	D4	26,7	186,8	56,3
Fluoranthene	FL	30,0	35,7	34,8
Pyrene	PY	77,9	426,8	98,2
C1-fluoranthenes/pyrenes	FL1	322,2	1659,5	507,2
C2-fluoranthenes/pyrenes	FL2	599,2	2324,7	557,0
C3-fluoranthenes/pyrenes	FL3	469,4	1637,9	520,8
Benzo[a]anthracene	BA	93,9	134,6	68,5
Chrysene	C	149,7	221,3	115,3
C1-chrysenes	C1	272,2	592,5	201,7
C2-chrysenes	C2	259,0	742,6	252,8
C3-chrysenes	C3	150,0	427,6	161,2
Benzo[b+k]fluoranthene	BBF	22,8	19,8	11,3
Benzo[e]pyrene	BEP	75,0	70,3	29,2
Benzo[a]pyrene	BAP	45,7	64,1	20,1
Perylene	PE	23,6	39,8	15,9
Indeno(1,2,3-cd)pyrene	IN	39,6	26,6	14,2
Dibenz(a,h)anthracene	DBA	24,3	33,8	24,5
Benzo(g,h,i)perylene	BPE	54,8	99,8	14,3
TOTAL (µg/g)		**4995,0**	**25086,5**	**13237,6**
N - N4		12%	24%	29%
BT - C3		83%	75%	70%
B(b+k)F - BPE		6%	1%	1%

Concentration (µg/g)	Abbrev.	IM-30	IM-31	IM-32
Benzo(b)thiophene	BT	37,0	0,0	0,0
C1-benzo(b)thiophenes	BT1	138,9	503,2	0,0
C2-benzo(b)thiophenes	BT2	74,2	347,8	0,0
C3-benzo(b)thiophenes	BT3	74,4	270,6	0,0
C4-benzo(b)thiophenes	BT4	29,9	108,9	0,0
Naphtalene	N	2236,1	38,9	130,5
C1-Naphtalenes	N1	5452,8	75,6	505,0
C2-Naphtalenes	N2	4927,6	10650,6	563,4
C3-Naphtalenes	N3	3346,1	7801,7	667,9
C4-Naphtalenes	N4	1879,1	4249,2	521,3
Biphenyl	B	497,9	336,3	0,0
Acenaphtylene	ANY	225,8	33,6	0,0
Acenaphtene	ANA	270,8	418,1	0,0
Fluorene	F	569,8	761,6	32,2
C1-Fluorenes	F1	1416,1	2032,2	149,8
C2-Fluorenes	F2	1796,3	3014,8	366,8
C3-Fluorenes	F3	1166,9	1370,2	268,9
Phenanthrene	P	1115,3	1574,9	86,0
Anthracene	A	182,4	250,2	0,0
C1-phenanthrenes/anthracenes	P1	2258,2	2700,0	167,9
C2-phenanthrenes/anthracenes	P2	2886,5	2518,4	344,5
C3-phenanthrenes/anthracenes	P3	1803,7	1516,8	278,8
C4-phenanthrenes/anthracenes	P4	1006,4	1190,2	189,3
Dibenzothiophene	D	63,2	240,3	2,4
C1-dibenzothiophenes	D1	248,3	599,5	40,7
C2-dibenzothiophenes	D2	330,9	503,8	23,2
C3-dibenzothiophenes	D3	242,0	278,3	16,7
C4-dibenzothiophenes	D4	185,1	146,9	0,0
Fluoranthene	FL	137,3	56,9	0,0
Pyrene	PY	606,9	348,2	7,3
C1-fluoranthenes/pyrenes	FL1	2711,7	1756,4	107,6
C2-fluoranthenes/pyrenes	FL2	3984,3	3436,0	191,7
C3-fluoranthenes/pyrenes	FL3	2934,3	2583,1	190,1
Benzo[a]anthracene	BA	324,4	244,0	25,4
Chrysene	C	504,5	363,1	62,8
C1-chrysenes	C1	1242,5	1255,2	85,1
C2-chrysenes	C2	1374,8	1141,2	95,3
C3-chrysenes	C3	789,2	766,4	64,5
Benzo[b+k]fluoranthene	BBF	69,2	59,6	4,2
Benzo[e]pyrene	BEP	180,0	110,0	18,1
Benzo[a]pyrene	BAP	137,4	73,1	6,9
Perylene	PE	69,4	32,7	6,4
Indeno(1,2,3-cd)pyrene	IN	33,2	22,5	12,7
Dibenz(a,h)anthracene	DBA	49,4	42,6	20,1
Benzo(g,h,i)perylene	BPE	79,6	42,2	5,5
TOTAL (µg/g)		**49690,1**	**55865,4**	**5258,8**
N - N4		36%	41%	45%
BT - C3		63%	58%	53%
B(b+k)F - BPE		1%	1%	1%

Concentration (µg/g)	Abbrev.	IM-33	IM-34	IM-36
Benzo(b)thiophene	BT	5,2	39,9	409,6
C1-benzo(b)thiophenes	BT1	115,2	0,0	67,0
C2-benzo(b)thiophenes	BT2	39,7	0,0	58,1
C3-benzo(b)thiophenes	BT3	34,1	0,0	55,4
C4-benzo(b)thiophenes	BT4	5,9	0,0	10,1
Naphtalene	N	2470,5	9000,4	7961,3
C1-Naphtalenes	N1	1125,3	4626,0	9724,9
C2-Naphtalenes	N2	4352,7	525,5	2402,8
C3-Naphtalenes	N3	3307,0	683,3	1840,1
C4-Naphtalenes	N4	1966,1	518,0	1155,4
Biphenyl	B	141,4	0,0	454,7
Acenaphtylene	ANY	0,0	0,0	5,9
Acenaphtene	ANA	211,7	0,0	112,9
Fluorene	F	426,2	32,4	239,4
C1-Fluorenes	F1	1351,3	162,0	667,2
C2-Fluorenes	F2	2355,9	472,0	1019,6
C3-Fluorenes	F3	1266,2	301,6	560,5
Phenanthrene	P	1001,7	80,4	443,2
Anthracene	A	174,6	0,0	64,7
C1-phenanthrenes/anthracenes	P1	2181,2	182,6	837,8
C2-phenanthrenes/anthracenes	P2	2465,1	322,0	959,6
C3-phenanthrenes/anthracenes	P3	1324,4	252,3	622,6
C4-phenanthrenes/anthracenes	P4	893,0	162,9	353,0
Dibenzothiophene	D	68,5	2,1	53,1
C1-dibenzothiophenes	D1	254,2	43,1	171,6
C2-dibenzothiophenes	D2	269,2	27,1	181,5
C3-dibenzothiophenes	D3	168,1	17,0	101,8
C4-dibenzothiophenes	D4	92,2	0,0	92,1
Fluoranthene	FL	61,0	0,5	18,9
Pyrene	PY	428,8	6,9	162,1
C1-fluoranthenes/pyrenes	FL1	1769,4	93,3	977,7
C2-fluoranthenes/pyrenes	FL2	2328,8	187,1	1327,1
C3-fluoranthenes/pyrenes	FL3	1604,9	166,0	1028,9
Benzo[a]anthracene	BA	192,7	22,9	108,1
Chrysene	C	293,0	52,8	152,3
C1-chrysenes	C1	687,3	81,0	452,5
C2-chrysenes	C2	628,9	94,6	514,9
C3-chrysenes	C3	338,9	66,0	326,9
Benzo[b+k]fluoranthene	BBF	33,8	2,8	15,0
Benzo[e]pyrene	BEP	93,6	15,7	62,8
Benzo[a]pyrene	BAP	63,4	6,3	60,8
Perylene	PE	33,5	0,0	31,0
Indeno(1,2,3-cd)pyrene	IN	16,4	0,0	24,8
Dibenz(a,h)anthracene	DBA	29,6	19,6	29,3
Benzo(g,h,i)perylene	BPE	66,6	4,5	103,1
TOTAL (µg/g)		**36737,3**	**18270,5**	**36022,0**
N - N4		36%	84%	64%
BT - C3		63%	16%	35%
B(b+k)F - BPE		1%	0%	1%

Concentration (µg/g)	Abbrev.	IM-37	IM-38	IM-39
Benzo(b)thiophene	BT	0,0	51,8	0,0
C1-benzo(b)thiophenes	BT1	1,6	0,0	12,6
C2-benzo(b)thiophenes	BT2	0,0	0,5	22,8
C3-benzo(b)thiophenes	BT3	3,2	6,2	38,7
C4-benzo(b)thiophenes	BT4	0,0	0,0	13,8
Naphtalene	N	65,6	4137,9	47,5
C1-Naphtalenes	N1	135,9	4803,6	101,0
C2-Naphtalenes	N2	761,0	1053,5	880,9
C3-Naphtalenes	N3	672,2	792,4	1043,8
C4-Naphtalenes	N4	487,1	462,6	745,9
Biphenyl	B	68,5	249,1	3,7
Acenaphtylene	ANY	7,5	39,5	0,0
Acenaphtene	ANA	42,1	119,2	37,5
Fluorene	F	129,2	247,2	92,3
C1-Fluorenes	F1	356,6	401,5	352,7
C2-Fluorenes	F2	753,4	482,2	581,4
C3-Fluorenes	F3	539,6	248,9	393,2
Phenanthrene	P	386,9	312,3	261,5
Anthracene	A	37,6	24,9	24,8
C1-phenanthrenes/anthracenes	P1	1049,6	385,7	597,6
C2-phenanthrenes/anthracenes	P2	1484,9	441,8	705,1
C3-phenanthrenes/anthracenes	P3	970,5	407,2	410,1
C4-phenanthrenes/anthracenes	P4	667,8	321,9	300,3
Dibenzothiophene	D	32,3	9,9	53,9
C1-dibenzothiophenes	D1	149,7	45,2	195,9
C2-dibenzothiophenes	D2	206,2	58,0	230,4
C3-dibenzothiophenes	D3	143,5	54,1	131,3
C4-dibenzothiophenes	D4	119,9	38,9	70,0
Fluoranthene	FL	46,5	25,6	6,4
Pyrene	PY	270,3	115,0	61,2
C1-fluoranthenes/pyrenes	FL1	1533,8	607,5	379,5
C2-fluoranthenes/pyrenes	FL2	2466,7	923,2	569,4
C3-fluoranthenes/pyrenes	FL3	1904,9	747,3	503,6
Benzo[a]anthracene	BA	255,2	120,3	46,8
Chrysene	C	471,1	170,0	67,6
C1-chrysenes	C1	1179,5	357,4	178,4
C2-chrysenes	C2	1306,3	441,8	240,5
C3-chrysenes	C3	733,9	245,5	217,9
Benzo[b+k]fluoranthene	BBF	51,9	16,7	4,8
Benzo[e]pyrene	BEP	189,4	79,5	17,8
Benzo[a]pyrene	BAP	112,4	55,2	12,0
Perylene	PE	46,8	31,3	6,9
Indeno(1,2,3-cd)pyrene	IN	20,9	29,3	9,5
Dibenz(a,h)anthracene	DBA	74,5	31,6	17,4
Benzo(g,h,i)perylene	BPE	68,5	40,5	9,1
TOTAL (µg/g)		**20005,2**	**19233,5**	**9697,8**
N - N4		11%	58%	29%
BT - C3		87%	40%	70%
B(b+k)F - BPE		3%	1%	1%
Concentration (µg/g)	Abbrev.	0,0	51,8	0,0

Appendix 5

Results of the IM-27 flume test experiments in 3 different conditions

(5°C/seawater, 5°C/freshwater and 25°C/seawater)

Co-funded by the European Union

Oil:	IM-27	Physical properties			Response techniques			
Temperature:	5°C / SW				Recovey		Chemical dispersion	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	165205	0,962	0	-	-	Not dispersible	Not dispersible
T1	1	118540	0,964	8,8	1,00	4870		
T2	2	138172	0,964	7,3	0,83	5970		
T3	4	138673	0,970	8,9	0,89	7045		
T4	6	111984	0,974	10,5	0,85	13860	Not dispersible	Not dispersible
T5	8	100241	0,979	22,7	0,92	6535		
T6	14	91955	0,981	26,5	0,84	5015		
T7	24	79775	0,984	35,8	0,91	7285	-	-
T8	26	79668	0,982	36,8	0,90	6300		
T9	30	81014	0,971	40,3	0,93	4240		
T10	48	76551	0,981	52,5	0,98	4725	-	-
T11	53	78869	0,981	54,0	0,95	5485		
T12	72	74710	0,994	50,9	0,98	4885	-	-
T13	144	114766	0,995	59,8	0,99	4645		

Oil:	**IM-27**	**Physical properties**			**Response techniques**			
Temperature:	**5°C / FW**				**Recovey**		**Chemical dispersion**	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	165205	0,962	0	-	-	Not dispersible	Not dispersible
T1	1	120962	0,968	8,6	1,00	1385		
T2	2	57161	0,970	8,8	1,00	4780		
T3	4	66408	0,973	10,5	1,00	2095		
T4	6	92628	0,975	11,5	1,00	1795	Not dispersible	Not dispersible
T5	8	95277	0,977	16,9	1,00	5545		
T6	14	103308	0,977	12,0	1,00	1830		
T7	24	107541	0,977	10,2	1,00	2820	-	-
T8	26	100122	0,981	14,0	1,00	3955		
T9	30	71988	0,982	16,9	1,00	3490		
T10	48	57783	0,988	34,1	1,00	2335	-	-
T11	53	104715	0,981	20,1	1,00	6665		
T12	72	89466	0,987	31,8	1,00	5245	-	-
T13	96	108506	0,994	47,6	1,00	4935		
T14	168	143659	0,995	37,0	0,99	1220		

Oil: **IM-27**
Temperature: **25°C / SW**

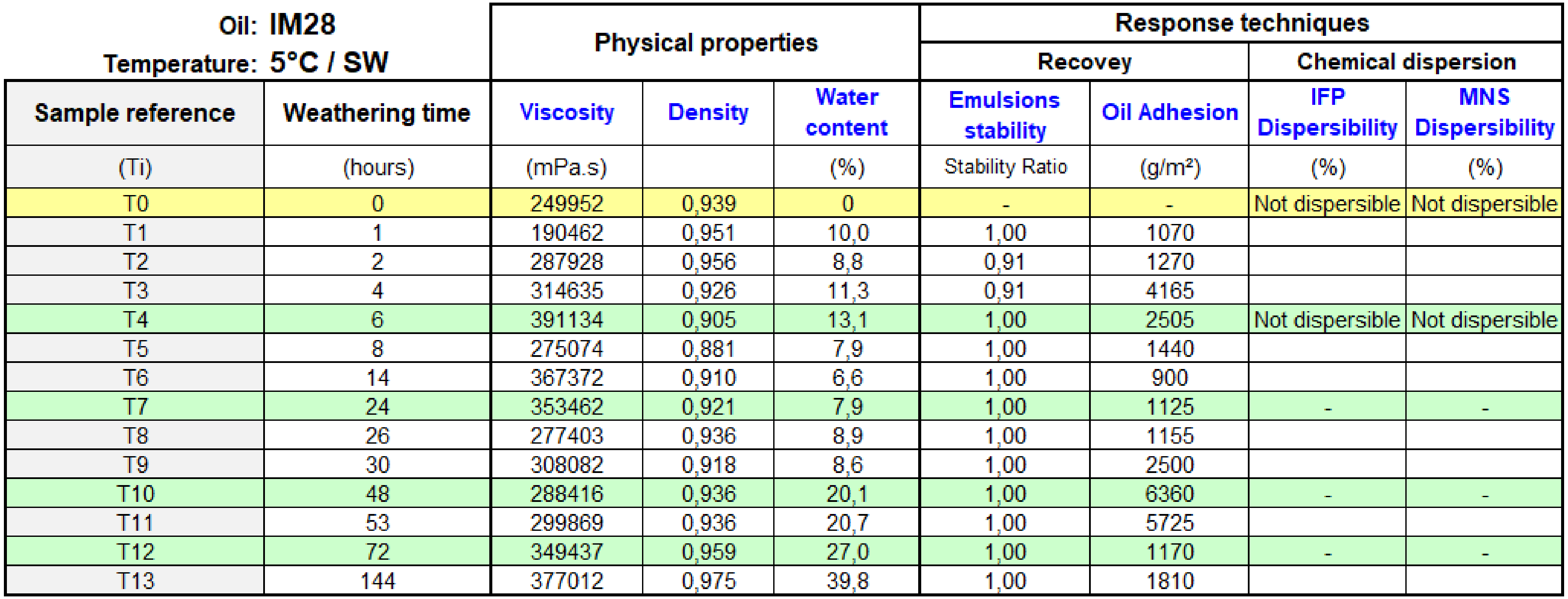
		Physical properties				Response techniques			
						Recovey		Chemical dispersion	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Evaporation rate**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	7592	0,949	0	0	-	-	Not dispersible	Not dispersible
T1	1	5407	0,946	9,5	-	1,00	2840		
T2	2	5623	0,951	20,5	0,2	1,00	3180		
T3	4	7056	0,953	33,0	3,2	1,00	3495		
T4	6	9020	0,956	49,0	3,1	1,00	2285	3,5	1,0
T5	8	13150	0,947	56,7	3,0	0,97	1745		
T6	14	17437	0,948	64,5	2,9	0,99	4470		
T7	24	17436	0,957	64,1	-	0,99	655	-	-
T8	26	16949	0,963	69,8	3,2	1,00	745		
T9	30	18462	0,972	72,6	2,9	1,00	630		
T10	48	20936	0,972	78,4	2,9	1,00	485	-	-
T11	53	20869	0,981	78,2	-	1,00	615		
T12	72	24430	0,980	78,0	2,9	1,00	660	-	-
T13	168	37161	0,975	80,7	-	-	600		

Appendix 6

Results of the IM-28 flume test experiments in 3 different conditions

(5°C/seawater, 5°C/freshwater and 25°C/seawater)

Co-funded by the European Union



Oil: **IM28**		Physical properties			Response techniques			
Temperature: **5°C / SW**					Recovey		Chemical dispersion	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	249952	0,939	0	-	-	Not dispersible	Not dispersible
T1	1	190462	0,951	10,0	1,00	1070		
T2	2	287928	0,956	8,8	0,91	1270		
T3	4	314635	0,926	11,3	0,91	4165		
T4	6	391134	0,905	13,1	1,00	2505	Not dispersible	Not dispersible
T5	8	275074	0,881	7,9	1,00	1440		
T6	14	367372	0,910	6,6	1,00	900		
T7	24	353462	0,921	7,9	1,00	1125	-	-
T8	26	277403	0,936	8,9	1,00	1155		
T9	30	308082	0,918	8,6	1,00	2500		
T10	48	288416	0,936	20,1	1,00	6360	-	-
T11	53	299869	0,936	20,7	1,00	5725		
T12	72	349437	0,959	27,0	1,00	1170	-	-
T13	144	377012	0,975	39,8	1,00	1810		

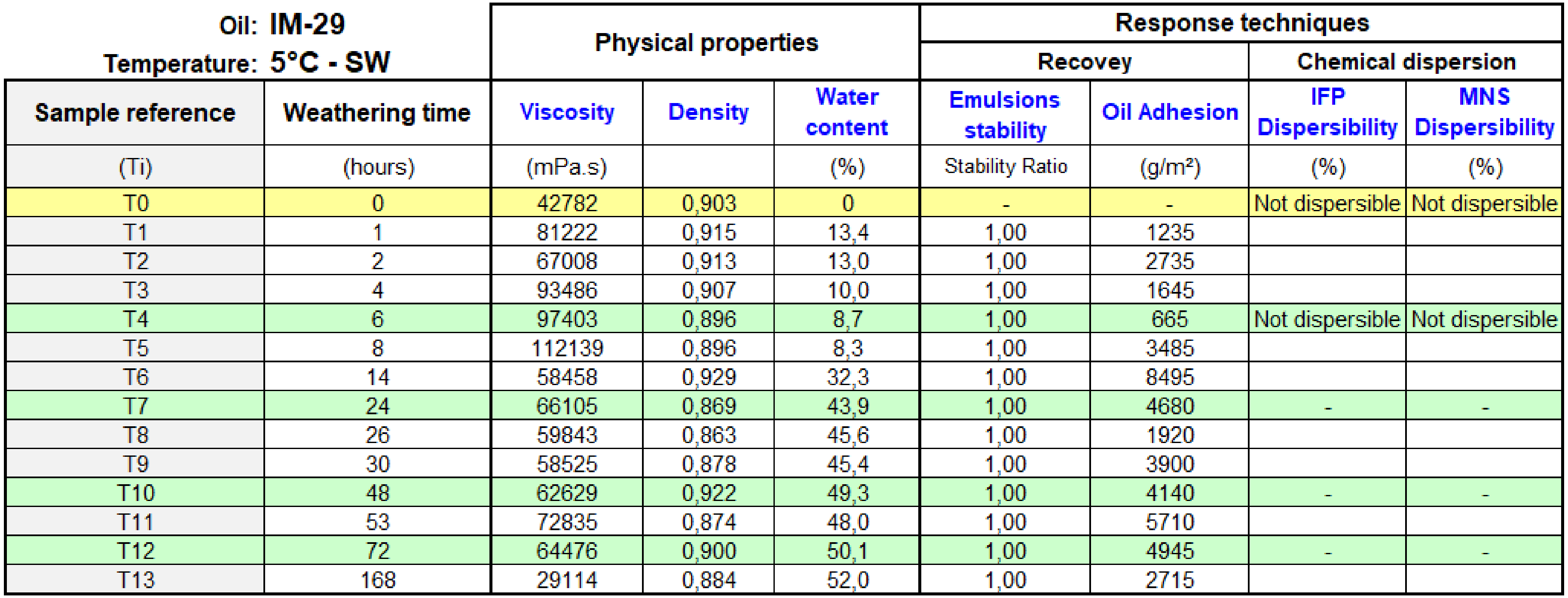
Oil: IM-28 Temperature: 5°C / FW		Physical properties			Response techniques			
					Recovey		Chemical dispersion	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	249952	0,962	0	-	-	Not dispersible	Not dispersible
T1	1	279376	0,940	10,5	1,00	600		
T2	2	220434	0,936	6,7	1,00	2150		
T3	4	346110	0,942	5,7	1,00	1135		
T4	6	357974	0,942	7,0	1,00	1460	Not dispersible	Not dispersible
T5	8	294711	0,943	5,4	1,00	2360		
T6	14	272325	0,943	6,4	1,00	1060		
T7	24	301701	0,945	9,1	1,00	5070	-	-
T8	26	241295	0,946	10,4	1,00	3160		
T9	30	333806	0,946	11,6	1,00	1720		
T10	48	342949	0,947	15,8	1,00	3185	-	-
T11	53	309127	0,942	13,9	1,00	3585		
T12	72	303417	0,944	14,5	1,00	2975	-	-
T13	96	300743	0,952	24,8	1,00	3675		
T14	168	305279	0,953	26,0	1,00	2010		

Oil: IM-28	**Temperature: 25°C / SW**	**Physical properties**				**Response techniques**			
						Recovey		Chemical dispersion	
Sample reference	**Weathering time**	Viscosity	Density	Water content	Evaporation rate	Emulsions stability	Oil Adhesion	IFP Dispersibility	MNS Dispersibility
(Ti)	(hours)	(mPa.s)		(%)	(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	21221	0,929	0	0	-	-	Not dispersible	Not dispersible
T1	1	21126	0,930	8,9	1,7	0,90	3175		
T2	2	20091	0,925	10,6	2,6	0,95	5580		
T3	4	18539	0,940	20,3	0,2	0,93	3485		
T4	6	16106	0,951	34,5	-	0,99	1730	Not dispersible	Not dispersible
T5	8	17502	0,958	48,2	3,7	0,98	2365		
T6	14	25088	0,940	60,4	2,5	0,99	1555		
T7	24	26935	0,986	73,4	2,3	0,99	1515	-	-
T8	26	26553	0,980	90,6	-	1,00	1000		
T9	30	26039	0,992	77,6	2,9	1,00	1235		
T10	48	35379	0,997	80,3	-	0,99	715	-	-
T11	53	28913	0,998	81,0	3,7	0,99	855		
T12	96	42913	0,998	82,6	1,4	0,99	875	-	-
T13	168	63861	0,994	83,7	1,6	-	925		

Appendix 7

Results of the IM-29 flume test experiments in 3 different conditions

(5°C/seawater, 5°C/freshwater and 25°C/seawater)



Oil: **IM-29**
Temperature: **5°C - SW**

		Physical properties			Response techniques			
					Recovey		Chemical dispersion	
Sample reference	Weathering time	Viscosity	Density	Water content	Emulsions stability	Oil Adhesion	IFP Dispersibility	MNS Dispersibility
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	42782	0,903	0	-	-	Not dispersible	Not dispersible
T1	1	81222	0,915	13,4	1,00	1235		
T2	2	67008	0,913	13,0	1,00	2735		
T3	4	93486	0,907	10,0	1,00	1645		
T4	6	97403	0,896	8,7	1,00	665	Not dispersible	Not dispersible
T5	8	112139	0,896	8,3	1,00	3485		
T6	14	58458	0,929	32,3	1,00	8495		
T7	24	66105	0,869	43,9	1,00	4680	-	-
T8	26	59843	0,863	45,6	1,00	1920		
T9	30	58525	0,878	45,4	1,00	3900		
T10	48	62629	0,922	49,3	1,00	4140	-	-
T11	53	72835	0,874	48,0	1,00	5710		
T12	72	64476	0,900	50,1	1,00	4945	-	-
T13	168	29114	0,884	52,0	1,00	2715		

Oil: IM-29
Temperature: 5°C / FW

		Physical properties			Response techniques			
					Recovey		Chemical dispersion	
Sample reference	**Weathering time**	**Viscosity**	**Density**	**Water content**	**Emulsions stability**	**Oil Adhesion**	**IFP Dispersibility**	**MNS Dispersibility**
(Ti)	(hours)	(mPa.s)		(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	42782	0,903	0	-	-	Not dispersible	Not dispersible
T1	1	63584	0,924	9,8	1,00	1845		
T2	2	58661	0,914	-	-	2315		
T3	4	59427	0,933	20,7	1,00	1135		
T4	6	60141	0,942	29,8	1,00	2190	Not dispersible	Not dispersible
T5	8	50143	0,953	41,4	1,00	1960		
T6	14	47893	0,965	53,6	1,00	2085		
T7	24	48513	0,974	61,4	1,00	1420	-	-
T8	26	44883	0,974	62,2	1,00	1100		
T9	30	40684	0,975	64,2	1,00	910		
T10	48	31701	0,983	72,5	1,00	1010	-	-
T11	53	45665	0,984	72,3	1,00	935		
T12	72	39184	0,975	72,7	0,93	910	-	-
T13	96	33388	0,984	77,9	0,92	425		
T14	168	28770	0,830	77,3	0,99	665		

Oil: IM-29
Temperature: 25°C

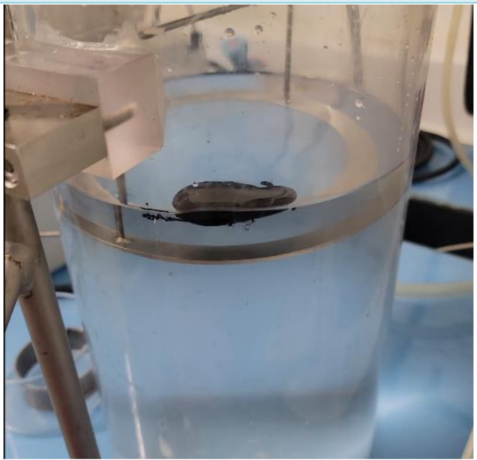
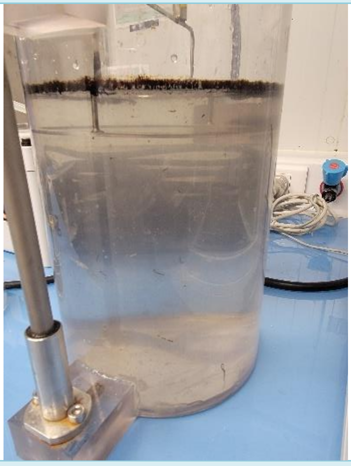
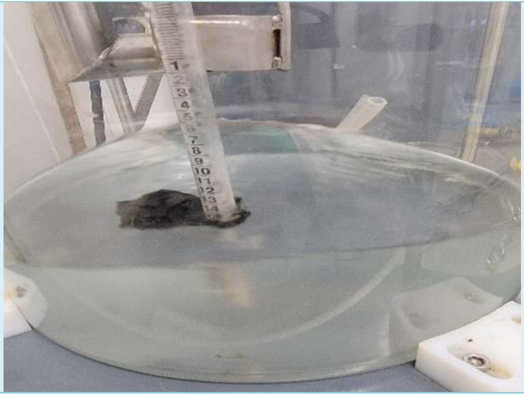
		Physical properties				Response techniques			
						Recovey		Chemical dispersion	
Sample reference	**Weathering time**	Viscosity	Density	Water content	Evaporation rate	Emulsions stability	Oil Adhesion	IFP Dispersibility	MNS Dispersibility
(Ti)	(hours)	(mPa.s)		(%)	(%)	Stability Ratio	(g/m²)	(%)	(%)
T0	0	331	0,887	0	-	-	-	Not dispersible	Not dispersible
T1	1	1976	0,943	45,3	3,8	1,00	1385		
T2	2	3356	0,972	65,0	-	1,00	1780		
T3	4	4618	0,989	81,3	-	0,99	2055		
T4	6	5654	0,993	83,4	-	0,99	1010	Not dispersible	Not dispersible
T5	8	5456	0,992	85,5	-	0,99	1310		
T6	14	5627	0,989	88,0	-	0,99	1435		
T7	24	6447	0,993	89,8	3,5	1,00	1065		
T8	26	6414	1,005	88,8	-	0,99	1730		
T9	30	6525	1,008	90,6	-	0,99	1340		
T10	48	6654	1,009	92,0	8,0	1,00	1825		
T11	53	6845	1,005	91,5	6,1	1,00	1890		
T12	72	7587	1,006	90,2	5,9	1,00	1160		
T13	168	8408	0,998	87,8	10,5	1,00	2165		

Appendix 8

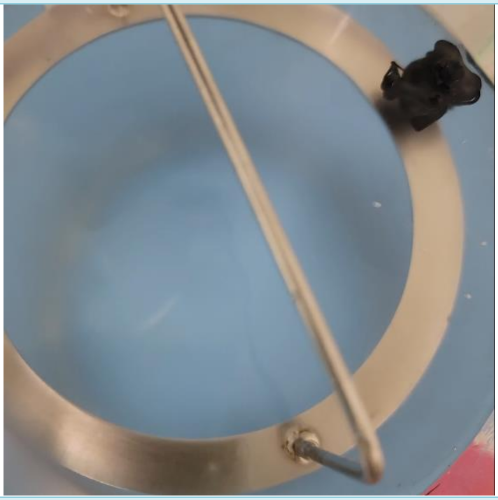
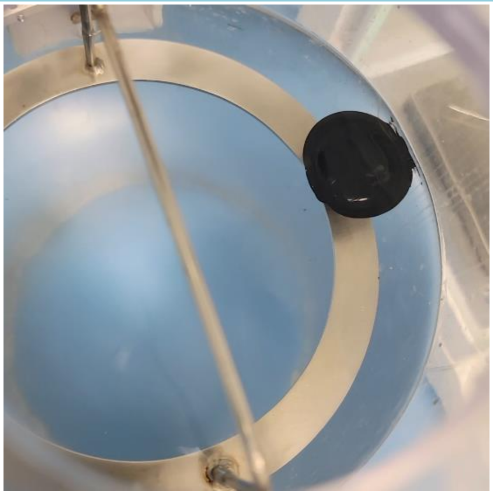
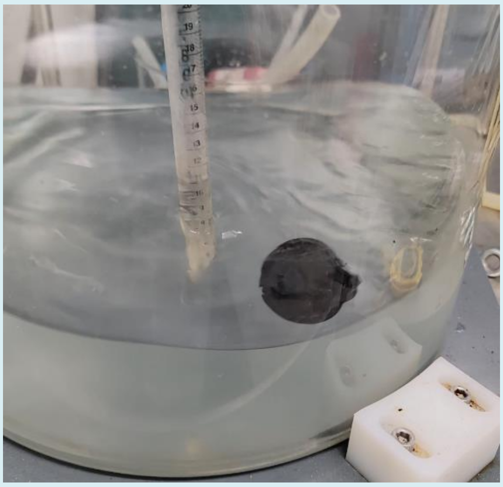
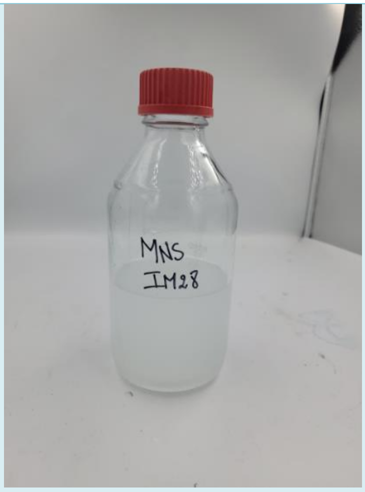
Pictures and data from dispersibility tests

Co-funded by the European Union

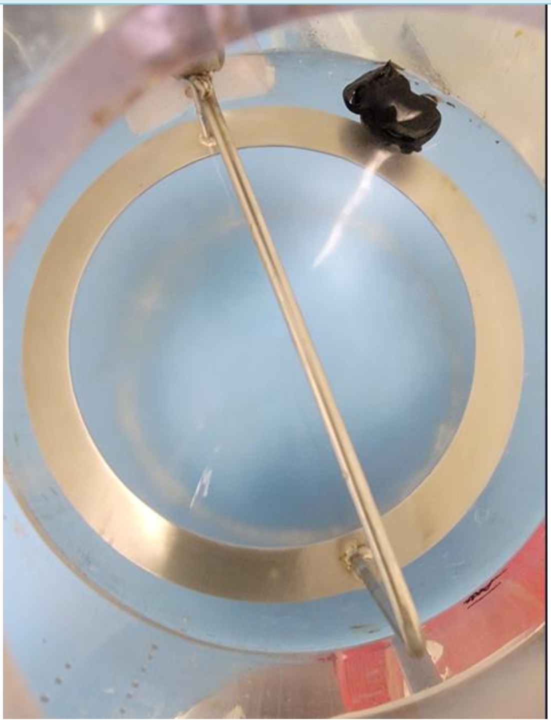
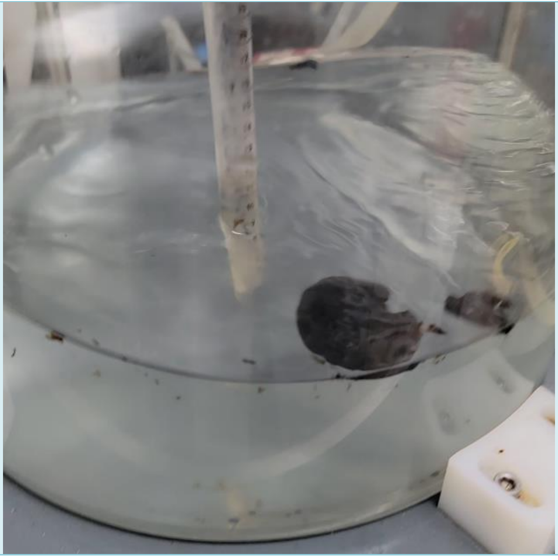
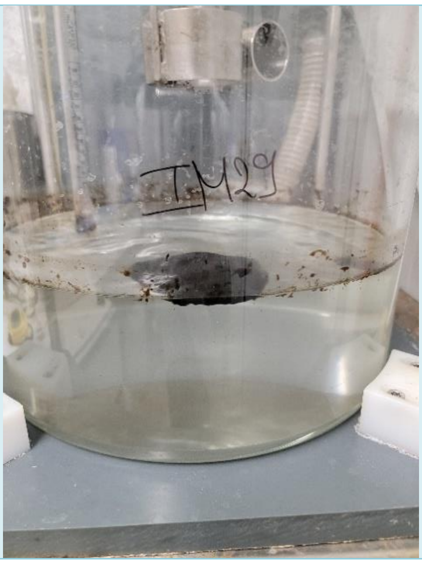
Fresh oil (T0)

	Fresh IM-27	
	5°C (SW)	25°C (SW)
IFP		
MNS		No photo available

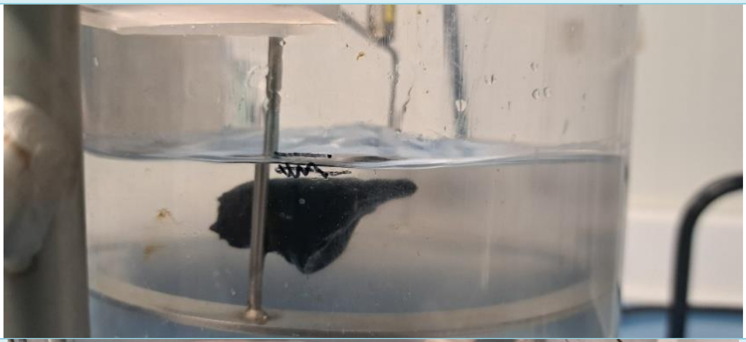
Fresh oil (T0)

	Fresh IM-28	
	5°C (SW)	25°C (SW)
IFP		
MNS		 (Water collected from the tank at the end of test)

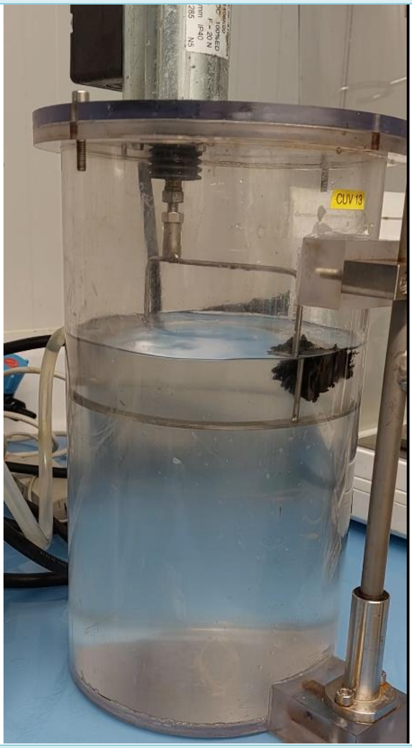
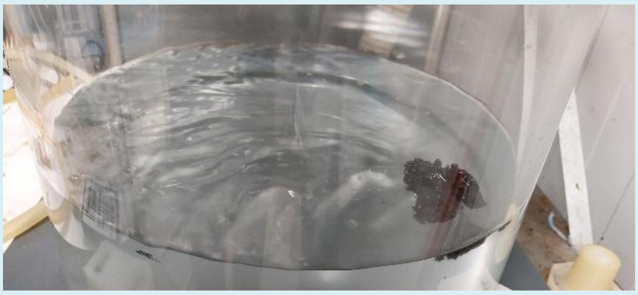
Fresh oil (T0)

	Fresh IM-29	
	5°C (SW)	25°C (SW)
IFP		No photo available
MNS		

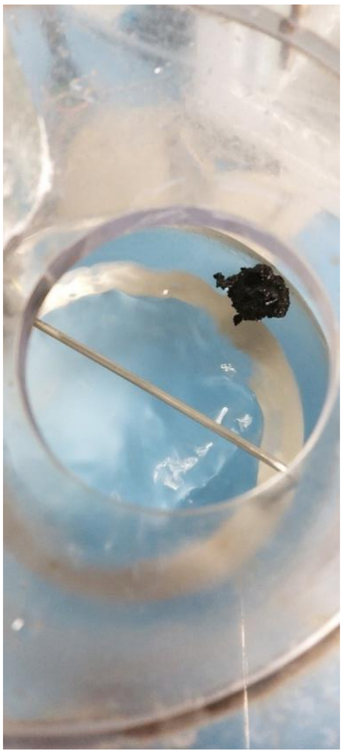
Weathered oil (+6h) / samples from the flume tank

Weathered IM-27 (+6h)		
	5°C (SW)	25°C (SW)
IFP		3,5%
MNS		1,0%

Weathered oil (+6h) / samples from the flume tank

<table>
<tr><th colspan="3">Weathered IM-28 (T+6h)</th></tr>
<tr><td></td><td>5°C (SW)</td><td>25°C (SW)</td></tr>
<tr><td>IFP</td><td></td><td></td></tr>
<tr><td>MNS</td><td></td><td></td></tr>
</table>

Weathered oil (+6h) / samples from the flume tank

Weathered IM-29 (T+6h)		
	5°C (SW)	25°C (SW)
IFP		
MNS		No photo available

Co-funded by the European Union

Appendix 9

Report from Oil fingerprinting analyses
(Task 3.5)

Co-funded by the
European Union

Royal Belgian Institute of Natural Sciences.

OD Natuur -Ecochem

Report 2025-49 Revision of 2025-15
WL 6119: 15 IMAROS Samples
Fingerprint analysis

279 - TEST
ISO/IEC
17025

The report was revised because the vessel names had to be removed.

Report on analytical results.

Project: IMAROS 2.

Samples received:
Fifteen (15) small samples were received on the 11th of October 2024. Three (3) large samples were received on 27th of February 2025.

Required analysis.
Oil fingerprinting on all 18 samples

Analytical procedure

The samples were analysed in conformity with the NBN EN 15522-2 (2023) Oil Spill Identification.

Interpretation of the analysis

Parameter	**Standard procedure BMM LAB/SV…**	**Version**	**Analytical characteristics BMM LAB/AK….**	**version**	**Analyst**
Oil comparative analysis	BMM LAB/SV023	11	BMM LAB/AK023	5	Marijke Neyts

Normative and Information Sampling and Sample treatment

Parameter	**Standard procedure BMM LAB/SV/…**	**Analyst**
Qualitative analysis of mineral oil with GC-MS	BMM LAB/SV065	MN

Data analysis process

PARAMETER

1st-line check: Marijke Neyts on November 7th 2024 (large samples April 22nd 2025)

Validation of the data: Marijke Neyts on November 7th 2024 (large samples April 22nd 2025)

Data approved by Dr. K. Parmentier (Lab Manager) on May 5th 2025.

Author: Marijke Neyts

Supervisor Organic Analysis and Research
Name: Marijke Neyts
Date:
Signature

Head of Ecochem
Name: Dr Koen Parmentier
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Table of contents

1. Sample information and tasks

Fifteen Small samples were collected by Cedre and sent to the Royal Belgian Institute of Natural Sciences to perform a chemical analysis by fingerprinting. Samples were imported in the comparative database Cosiweb 2.0.

Three large samples were given later.

2. Sample treatment

The samples received were diluted to 8 +-1 mg/ml.
Samples were analysed in worklist 6119 (table 1).

Table 1: Worklist 6119.

Worklist	**6119**
Number	Sample
1	Imaros 2 : IM-20 VLSFO Stena, Sweden
2	Imaros 2 : IM-21 ULSFO Stena, Sweden
3	Imaros 2 : IM-22 VLSFO BunkerOne, Sweden
4	Imaros 2 : IM-23 ULSFO BunkerOne, Sweden
5	Imaros 2 : IM-24 VLSFO Preem AB, Finland
6	Imaros 2 : IM-25 VLSFO Neste, Finland
7	Imaros 2 : IM-30 VLSFO FincoEnergies, Netherlands
8	Imaros 2 : IM-31 VLSFO FincoEnergies, Netherlands
9	Imaros 2 : IM-32 ULSFO FincoEnergies, Netherlands
10	Imaros 2 : IM-33 VLSFO FincoEnergies, Netherlands
11	Imaros 2 : IM-34 ULSFO FincoEnergies, Netherlands
12	Imaros 2 : IM-36 VLSFO Bunker from vessel, Netherlands
13	Imaros 2 : IM-37 VLSFO Bureau Veritas, Malta
14	Imaros 2 : IM-38 VLSFOBureau Veritas, Malta
15	Imaros 2 : IM-39 VSFO Peninsula Antwerp, Belgium
Worklist	**6119bis**
16	Imaros 2 : IM27 VLSFO Bunker One, Denmark
17	Imaros 2 : IM28 VLSFO ZETA Energy Limited, Malta
18	Imaros2 : IM29 ULSFO Stena Oil, Sweden

All small samples were cleaned up by elution over a silica gel column (Figure 1). The large samples did not undergo clean-up, as there were questions whether or not FAMEs were present in the oils.

Figure 1: Samples with the silica gel columns, before (left) and after (right) clean-up.

1 µL of the diluted samples was injected into the GC.
All analyses were performed on a DB5 column: 30 m x 0.25 mm ID x 0.25 µm film thickness.

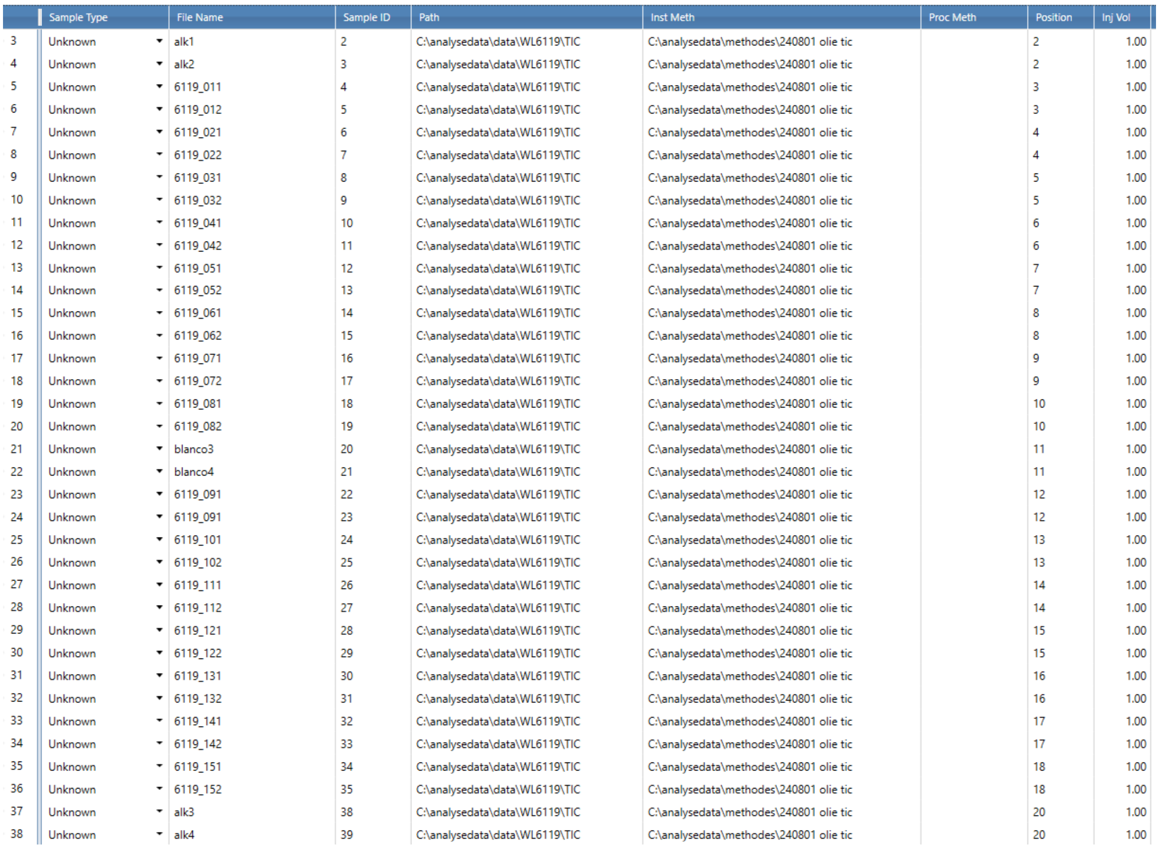
The oven was preheated to 42 °C, and the temperature maintained for 0.95 min, followed by a temperature increase at 5.5 °C/min to 330 °C (20 min hold) with a He carrier flow of 1.1 mL/min . The compounds were detected by a Thermofisher Trace MS in electron-impact ionization (EI) mode operating in TIC (Total Ion Count) for the first screening and selected ion monitoring (SIM) for the fingerprint analysis.

3. Sequences

3.1. Sequences GC-MS-TIC.

Table 2 lists the sequence of the TIC analysis.

Table 2: Sequence of the TIC analysis.



	Sample Type	File Name	Sample ID	Path	Inst Meth	Proc Meth	Position	Inj Vol
3	Unknown	alk1	2	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		2	1.00
4	Unknown	alk2	3	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		2	1.00
5	Unknown	6119_011	4	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		3	1.00
6	Unknown	6119_012	5	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		3	1.00
7	Unknown	6119_021	6	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		4	1.00
8	Unknown	6119_022	7	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		4	1.00
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16	Unknown	6119_062	15	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		8	1.00
17	Unknown	6119_071	16	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		9	1.00
18	Unknown	6119_072	17	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		9	1.00
19	Unknown	6119_081	18	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		10	1.00
20	Unknown	6119_082	19	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		10	1.00
21	Unknown	blanco3	20	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		11	1.00
22	Unknown	blanco4	21	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		11	1.00
23	Unknown	6119_091	22	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		12	1.00
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27	Unknown	6119_111	26	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		14	1.00
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29	Unknown	6119_121	28	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		15	1.00
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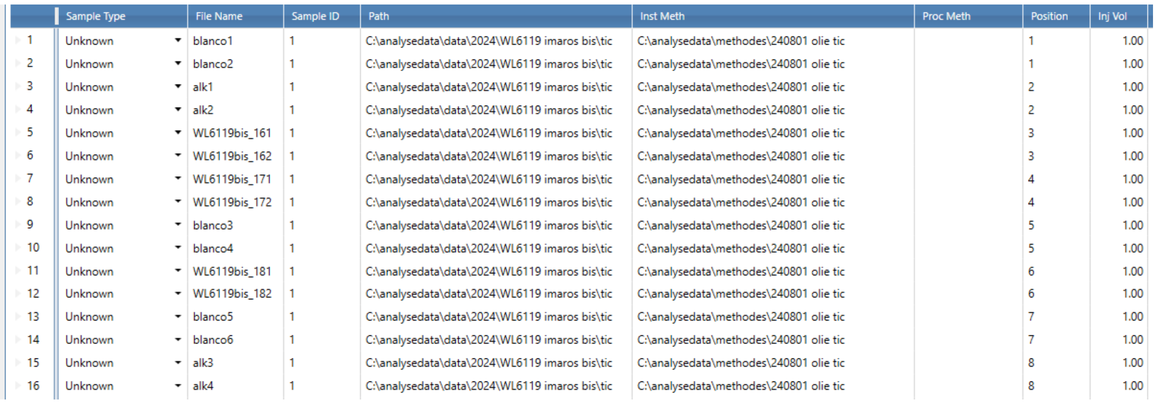
31	Unknown	6119_131	30	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		16	1.00
32	Unknown	6119_132	31	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		16	1.00
33	Unknown	6119_141	32	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		17	1.00
34	Unknown	6119_142	33	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		17	1.00
35	Unknown	6119_151	34	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		18	1.00
36	Unknown	6119_152	35	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		18	1.00
37	Unknown	alk3	38	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		20	1.00
38	Unknown	alk4	39	C:\analysedata\data\WL6119\TIC	C:\analysedata\methodes\240801 olie tic		20	1.00



	Sample Type	File Name	Sample ID	Path	Inst Meth	Proc Meth	Position	Inj Vol
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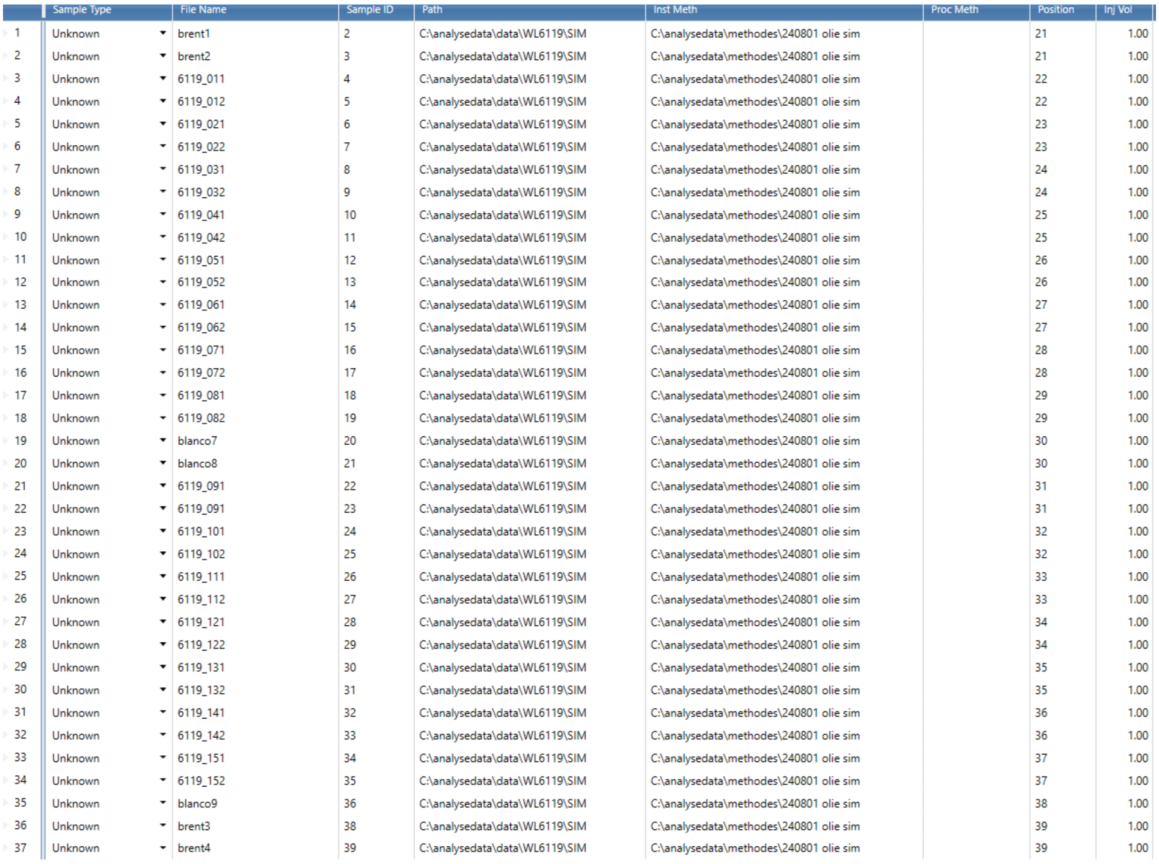
2	Unknown	blanco2	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		1	1.00
3	Unknown	alk1	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		2	1.00
4	Unknown	alk2	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		2	1.00
5	Unknown	WL6119bis_161	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		3	1.00
6	Unknown	WL6119bis_162	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		3	1.00
7	Unknown	WL6119bis_171	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		4	1.00
8	Unknown	WL6119bis_172	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		4	1.00
9	Unknown	blanco3	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		5	1.00
10	Unknown	blanco4	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		5	1.00
11	Unknown	WL6119bis_181	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		6	1.00
12	Unknown	WL6119bis_182	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		6	1.00
13	Unknown	blanco5	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		7	1.00
14	Unknown	blanco6	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		7	1.00
15	Unknown	alk3	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		8	1.00
16	Unknown	alk4	1	C:\analysedata\data\2024\WL6119 imaros bis\tic	C:\analysedata\methodes\240801 olie tic		8	1.00

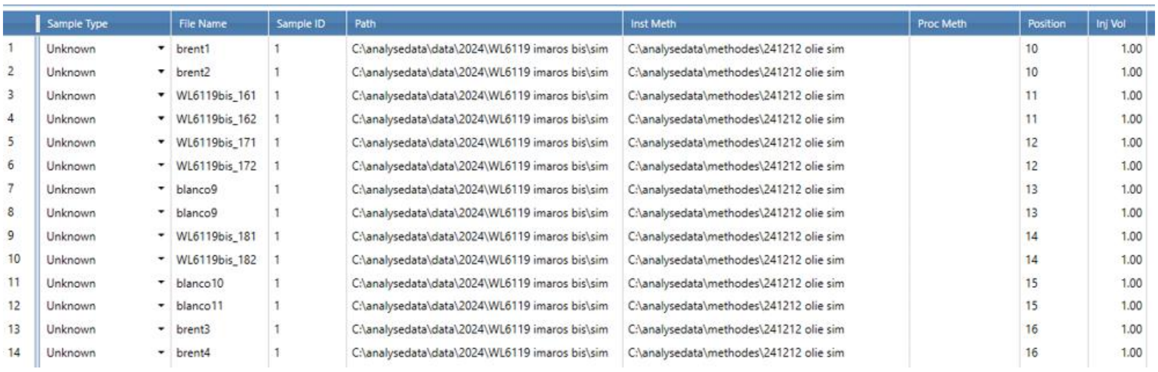
3.2. Sequences GC-MS-SIM.

Table 3 lists the sequence of the SIM analysis.

Table 3: Sequence of the SIM analysis.



	Sample Type	File Name	Sample ID	Path	Inst Meth	Proc Meth	Position	Inj Vol
1	Unknown	brent1	2	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		21	1.00
2	Unknown	brent2	3	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		21	1.00
3	Unknown	6119_011	4	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		22	1.00
4	Unknown	6119_012	5	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		22	1.00
5	Unknown	6119_021	6	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		23	1.00
6	Unknown	6119_022	7	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		23	1.00
7	Unknown	6119_031	8	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		24	1.00
8	Unknown	6119_032	9	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		24	1.00
9	Unknown	6119_041	10	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		25	1.00
10	Unknown	6119_042	11	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		25	1.00
11	Unknown	6119_051	12	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		26	1.00
12	Unknown	6119_052	13	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		26	1.00
13	Unknown	6119_061	14	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		27	1.00
14	Unknown	6119_062	15	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		27	1.00
15	Unknown	6119_071	16	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		28	1.00
16	Unknown	6119_072	17	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		28	1.00
17	Unknown	6119_081	18	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		29	1.00
18	Unknown	6119_082	19	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		29	1.00
19	Unknown	blanco7	20	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		30	1.00
20	Unknown	blanco8	21	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		30	1.00
21	Unknown	6119_091	22	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		31	1.00
22	Unknown	6119_091	23	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		31	1.00
23	Unknown	6119_101	24	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		32	1.00
24	Unknown	6119_102	25	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		32	1.00
25	Unknown	6119_111	26	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		33	1.00
26	Unknown	6119_112	27	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		33	1.00
27	Unknown	6119_121	28	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		34	1.00
28	Unknown	6119_122	29	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		34	1.00
29	Unknown	6119_131	30	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		35	1.00
30	Unknown	6119_132	31	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		35	1.00
31	Unknown	6119_141	32	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		36	1.00
32	Unknown	6119_142	33	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		36	1.00
33	Unknown	6119_151	34	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		37	1.00
34	Unknown	6119_152	35	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		37	1.00
35	Unknown	blanco9	36	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		38	1.00
36	Unknown	brent3	38	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		39	1.00
37	Unknown	brent4	39	C:\analysedata\data\WL6119\SIM	C:\analysedata\methodes\240801 olie sim		39	1.00



	Sample Type	File Name	Sample ID	Path	Inst Meth	Proc Meth	Position	Inj Vol
1	Unknown	brent1	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		10	1.00
2	Unknown	brent2	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		10	1.00
3	Unknown	WL6119bis_161	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		11	1.00
4	Unknown	WL6119bis_162	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		11	1.00
5	Unknown	WL6119bis_171	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		12	1.00
6	Unknown	WL6119bis_172	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		12	1.00
7	Unknown	blanco9	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		13	1.00
8	Unknown	blanco9	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		13	1.00
9	Unknown	WL6119bis_181	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		14	1.00
10	Unknown	WL6119bis_182	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		14	1.00
11	Unknown	blanco10	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		15	1.00
12	Unknown	blanco11	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		15	1.00
13	Unknown	brent3	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		16	1.00
14	Unknown	brent4	1	C:\analysedata\data\2024\WL6119 imaros bis\sim	C:\analysedata\methodes\241212 olie sim		16	1.00

4. GC-TIC Visual inspection

4.1. IMAROS TIC Analysis.

Figure 2 depicts the TIC chromatograms of all small samples. In Appendix 2, the chromatograms >C20 are illustrated with their intensities.

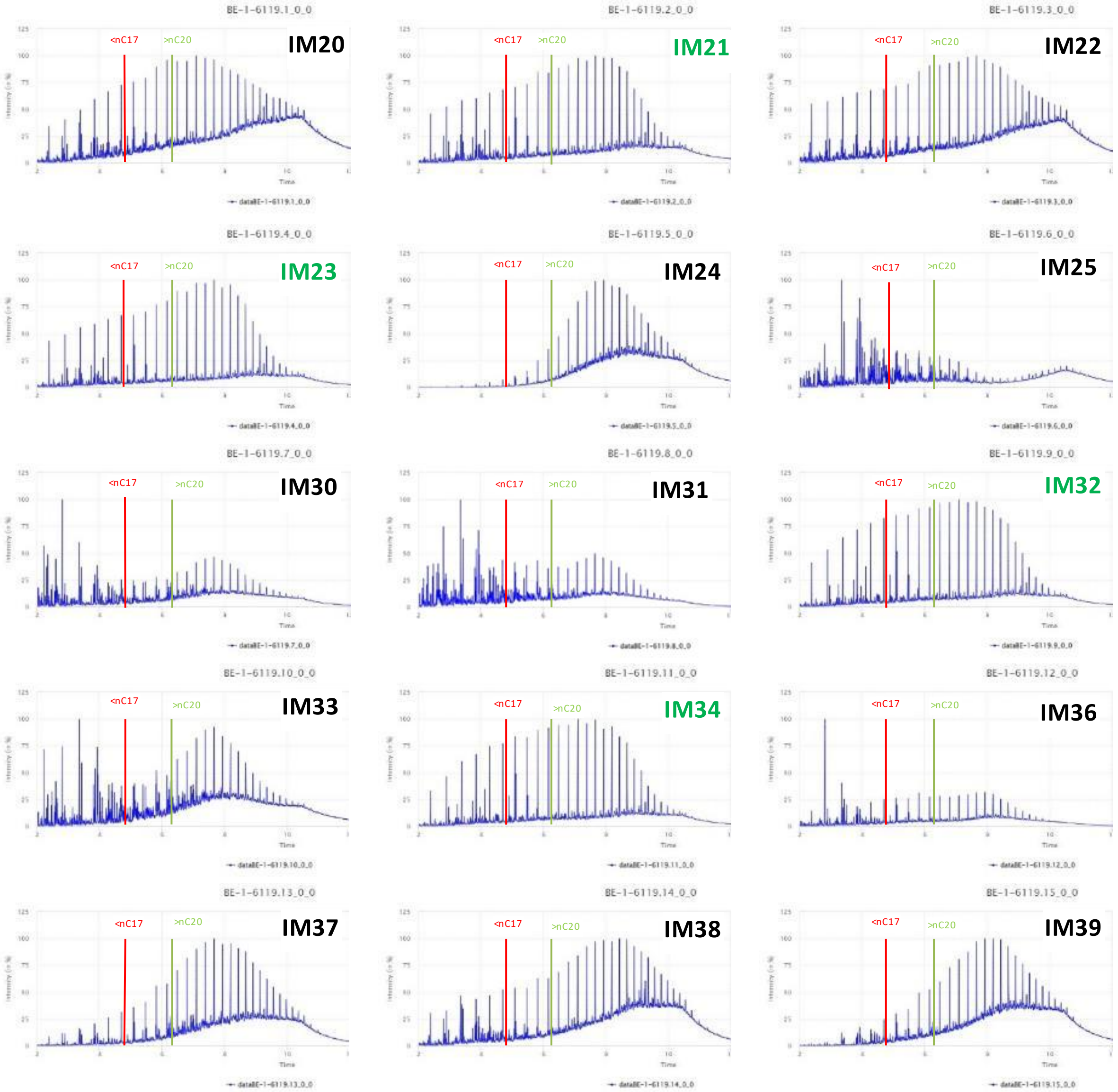


Figure 2: The chromatograms of samples IM20-IM25, IM30-IM34, IM36-IM39, respectively.

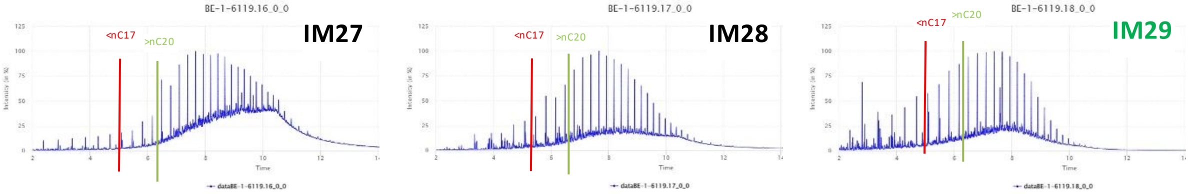


Figure 3 : The chromatograms of samples IM27, IM28 and IM29, respectively.

Samples with green label are ultra-low sulphur fuel oils (ULSFO), the others are very low sulphur fuel oils (VLSFO). Compounds before C17, between the alkanes, are Naphthalenes. Alkanes after C20 are considered waxes. Note the differences between the samples. Table 4 shows the intensity of the Naphthalenes, the lower alkanes and the waxes. Table 5 shows the resume of the different samples.

Table 4 : Intensity of the Naphthalenes, lower alkanes and waxes.

intensity x10e7	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
Naphthalenes m/z 142, 156, 170	4.14	7.16	1.02	3.9	0.238	25.9	13.4	26.7	1.99	13	1.8	14.2	2.87	5.07	2.19	0.485	4.91	11.18

intensity x10e7	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
lower alkanes <C17 m/z 85	5.51	9.24	3.47	7.14	0.59	2.4	1.95	4.07	10.8	1.19	10.1	3.54	2.32	2.28	1.15	1.51	5.36	8.89

intensity x10e7	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
Waxes >C20 m/Z 85	4.42	14.7	3.8	12.8	9.35	2.47	5.8	3.12	12.2	5.04	13.5	5.16	7.28	4.53	5.03	6.71	11.3	12.98

Table 5 : Resumé of the Naphthalenes, lower alkanes, and waxes.

	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
Naphthalenes			-		--	+	+	++		+	-	+				-		
lower alkanes		+		+	-				++	-	+				-	-		+
waxes	-	++	-	+		-		-	+		+							+

4.2. Conclusion GC TIC plots

The TIC analysis shows all kind of combinations of naphthalenes, lower alkanes and waxes are possible to make a fuel oil with a low sulphur content.

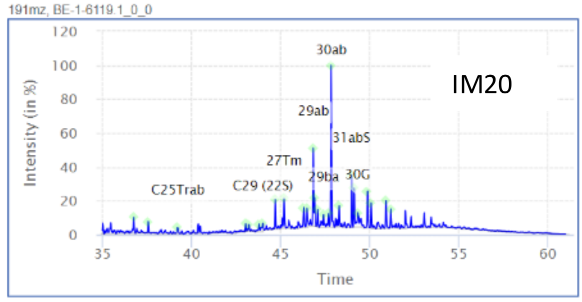
4.3. Strange behaviour of sample IM29

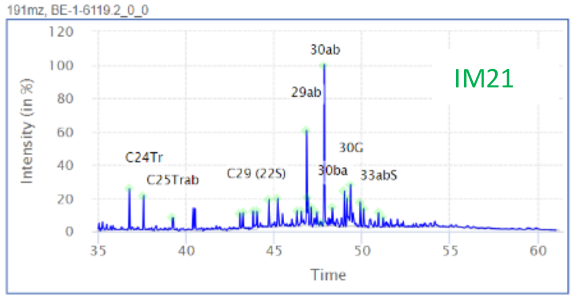
Because of the strange behaviour of sample IM29 the question rose whether FAME's were present. This is handled in Appendix 4.

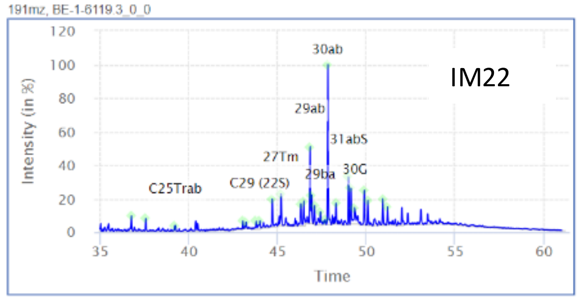
5. MS results

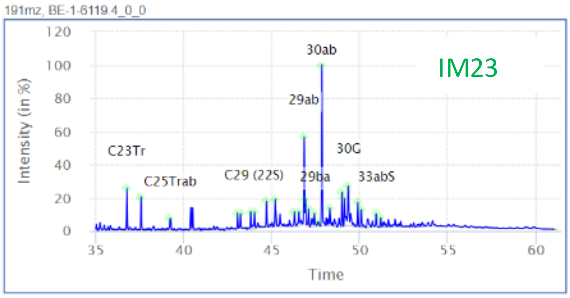
5.1. Hopanes (m/z 191)

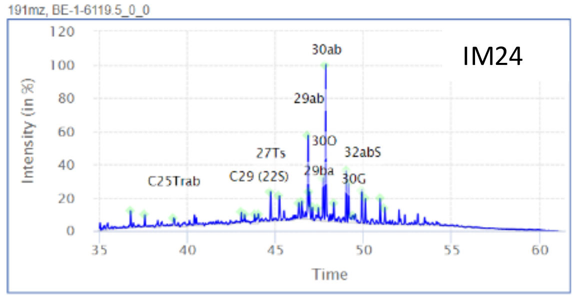
Hopanes are biomarkers that are much more resistant and are affected only by severe biodegradation making them the most important biomarkers in oil comparison. In light fuel there are hardly any hopanes present, but in heavy fuel these are prominent markers. Figure 4 shows the ion chromatograms of the hopanes.

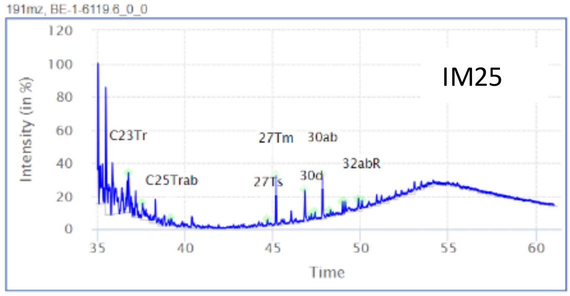


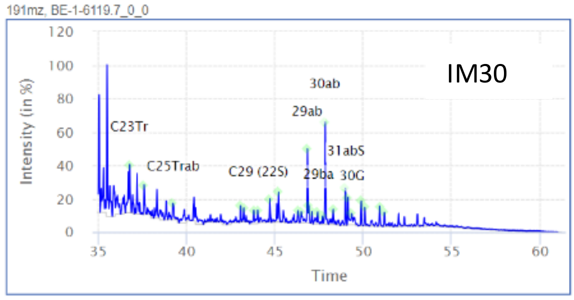


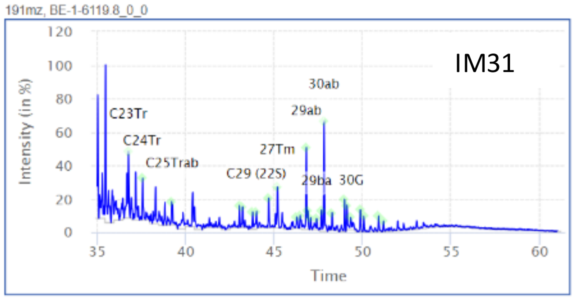












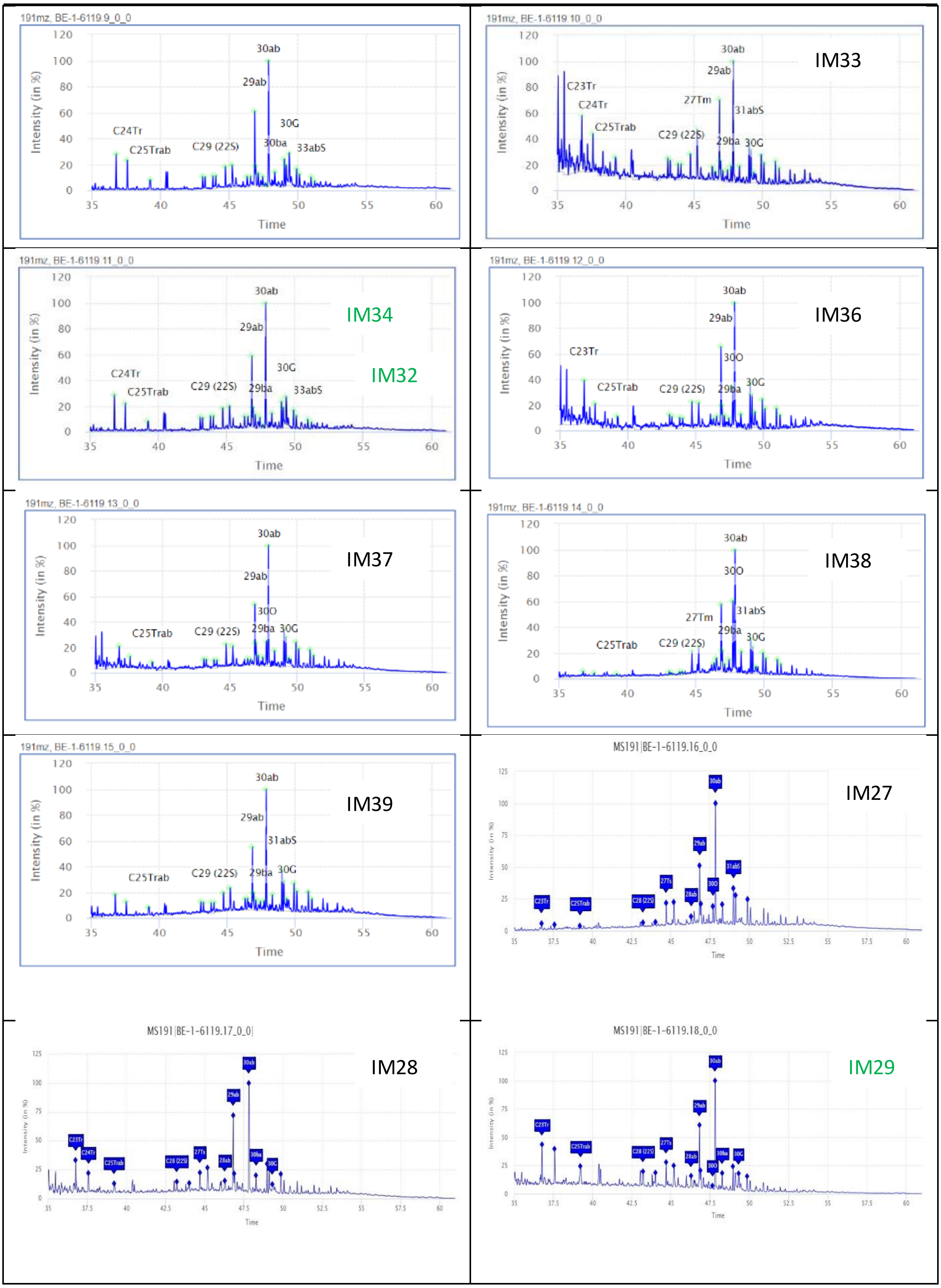


Figure 4 : Ion chromatograms of the hopanes.

Patterns look different but show a lot of similarity. The ion chromatograms of these ULSFO and VLSFO are comparable to other HFO. As an exception, sample IM25 does hardly have hopanes.

Oleanane/Lupane

Sweet crudes or crude oils that have naturally low sulphur concentrations, are reputed to have oleanane/lupane as a biomarker.

At retention time 47.70 the peak of oleanane/lupane is visible. Figure 5 presents the hopanes/oleanane (30O)/lupane of samples IM24, IM31, IM33, IM36, IM37 and IM38 with Oleanane/Lupane clearly visible. A study in 2006 shows that oleanane/lupane is present in Nigerian sweet crudes (Abrakasa, 2006). Lupane and oleanane co-elute on a non-polar GC column.

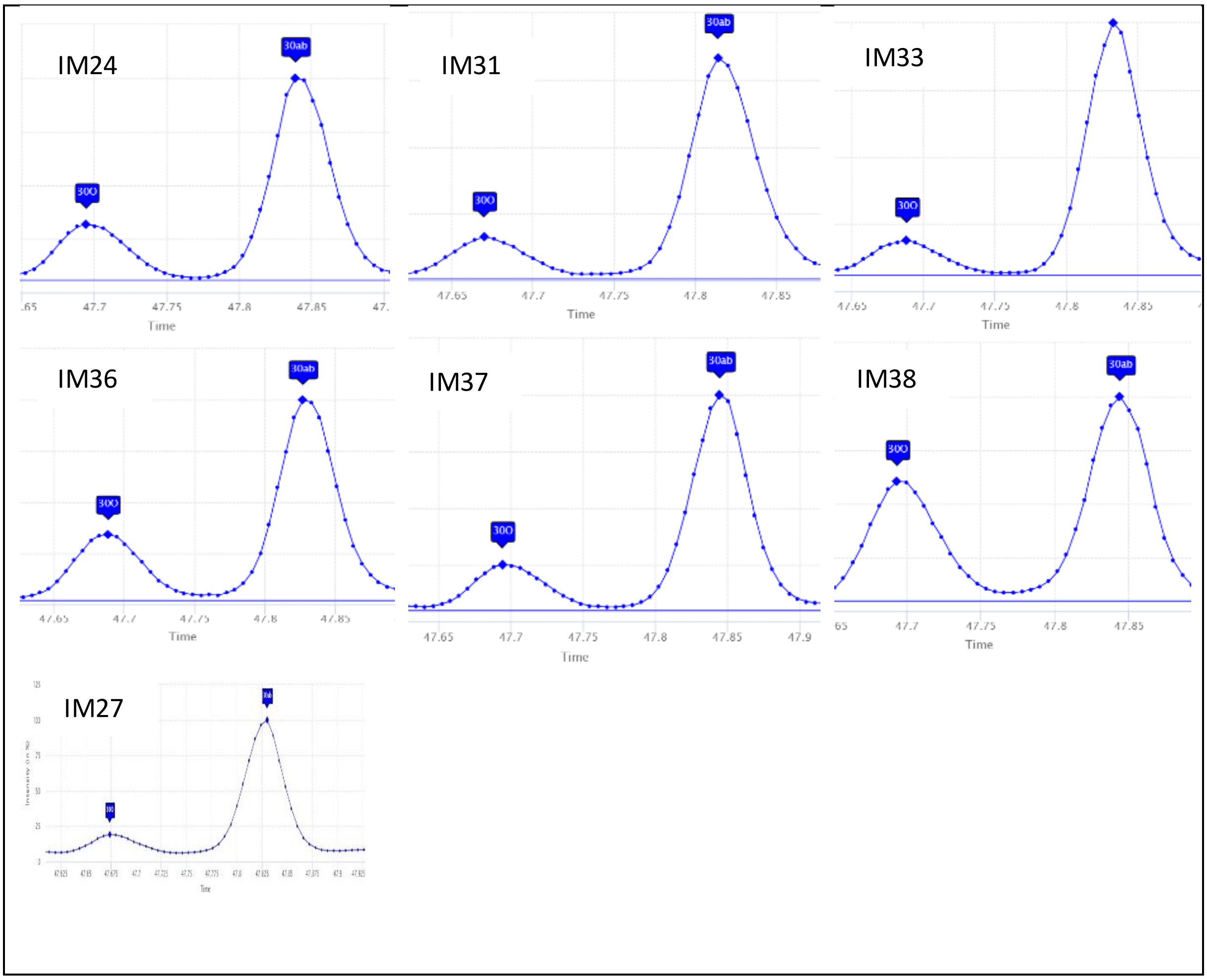


Figure 5 : Ion chromatogram of 30O/Hopane of samples IM24, IM31, IM33, IM36, IM37, IM38, IM27.

In table 6, calculations are made for hopane/concentration and the ratio 30O/hopane. Samples IM21, IM23, IM24, IM34, IM38 and IM27 show a large concentration of hopane. Samples IM24, IM31, IM33, IM37, IM37, IM38 and IM27 show a large concentration of 30O, which means that they might be blended with sweet-crude oil.

Table 6 : Calculation of hopane/conc and 30O/hopane.

Sample	hopane/conc (x10^5)	30O/hopane (%)	30O/conc (x10^3)
IM20	6.2	7.8	48
IM21	12.6	2.3	29
IM22	4.9	2.9	14
IM23	10.1	2.2	22
IM24	8.6	26.4	228
IM25	0.3	0.0	0
IM30	2.4	7.3	17
IM31	1.0	17.8	17
IM32	8.9	0.0	0
IM33	2.3	13.0	30
IM34	11.0	2.0	22
IM36	1.4	31.9	43
IM37	5.3	19.9	105
IM38	10.4	58.0	601
IM39	5.4	7.3	40
IM27	12.0	14.0	168
IM28	4.0	0.0	0
IM29	4.0	3.3	13

5.2. Retene, Tetra-Methylphenanthrene, Benzo(b)naphto(1,2-d)-thiophene (BNT) and the C4-Phenanthrenes (m/z 234)

Retene, Tetra-Methylphenanthrene, Benzo(b)naphto(1,2-d)thiophene and the C4-Phenanthrenes (m/z 234) are characteristic clusters of aromatics from cracking residues these can be used for an unequivocal identification of HFOs if aromatics are not readily noticeable by GC screening. Here we are especially interested in Benzo(b)naphto(1,2-d)thiophene as it contains sulphur. The ion chromatograms are presented in Figure 6.

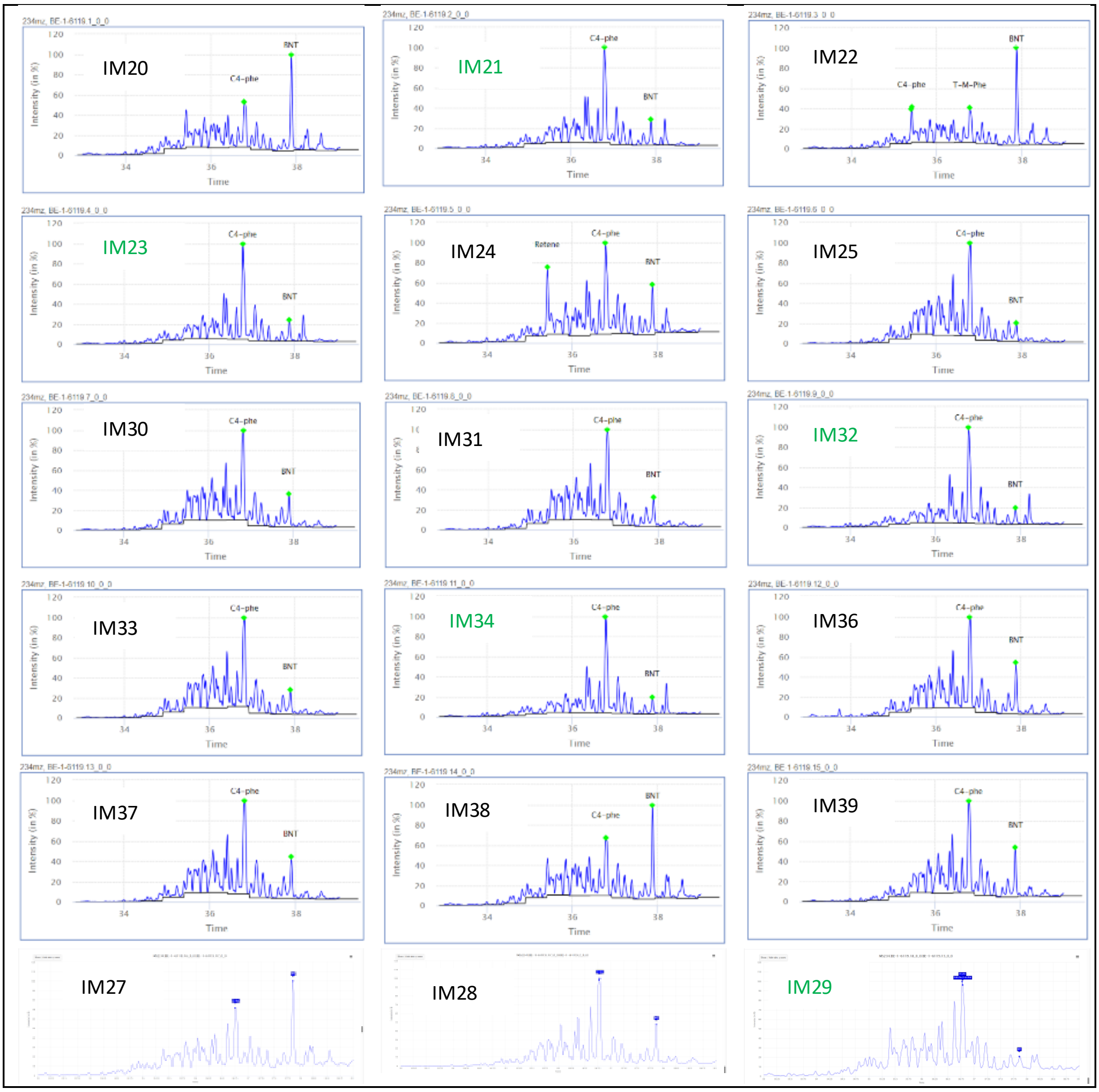


Figure 6 : Ion chromatogram of m/z 234 for all 18 samples.

In table 7, ratio calculations are made for BNT/concentration and BNT/hopane. A ratio calculation was also made between BNT/C2-Phenanthrenes as in the literature (Mössner, 1999).

The C2-dibenzothiophenes/C2-phenanthrenes ratio provides information about the sulphur content. Also, the ratio hopane/conc is added to the table.

Table 4 : Calculation of different Benzo-naphtothiophene ratios and hopane vs conc.

Sample	BNT/conc (x10^4)	BNT/hopane (%)	BNT/C2-phen (%)	hopane/conc (x10^5)
IM20	20.4	32.9	18.13	6.2
IM21	5.7	4.5	0.34	12.6
IM22	17.9	36.7	2.66	4.9
IM23	3.4	3.3	0.31	10.1
IM24	6.1	7.0	1.12	8.6
IM25	5.9	220.2	0.12	0.3
IM30	24.5	104.0	0.35	2.4
IM31	8.5	87.1	0.29	1.0
IM32	2.0	2.3	0.30	8.9
IM33	13.5	59.1	0.24	2.3
IM34	2.6	2.4	0.32	11.0
IM36	10.8	79.9	0.54	1.4
IM37	21.9	41.4	0.55	5.3
IM38	19.2	18.5	1.62	10.4
IM39	9.3	17.1	0.57	5.4
IM27	16.5	13.7	2.01	12.0
IM28	23.9	59.9	0.62	4.0
IM29	1.4	3.7	0.14	4.0

The ration BNT/conc demonstrates that ULSFO shows the lowest ratio. For BNT/C2 phenanthrenes, most of the ratios are low.

5.3. C1-fluoranthenes/pyrenes/benzofluorenes (m/z 216)

C1-fluoranthenes/pyrenes/benzofluorenes are examined in oil spill identification because they are easily affected by photo-oxidation, a phenomenon that is not observed in this study. These patterns are examined for differences (Figure 7).

Figure 7 : Ion chromatogram of m/z 216 of all 18 samples.

These compounds do not add to the sulphur content. There are no patterns to conclude that samples are enhanced in sulphur.

5.4. C1-Phenanthrenes and C1-Anthracene (m/z 192)

Cracking processes lead to distinct clusters of isomers. The cluster of isomeric M-phenanthrenes, for example, is typical of the high-temperature production of aromatics. It is generally found, e.g. in soot particles of diesel exhaust gases. Methyl-anthracene is never present in high concentration in crude oil. This is a product of the cracking process. Figure 8 shows the ion chromatograms.

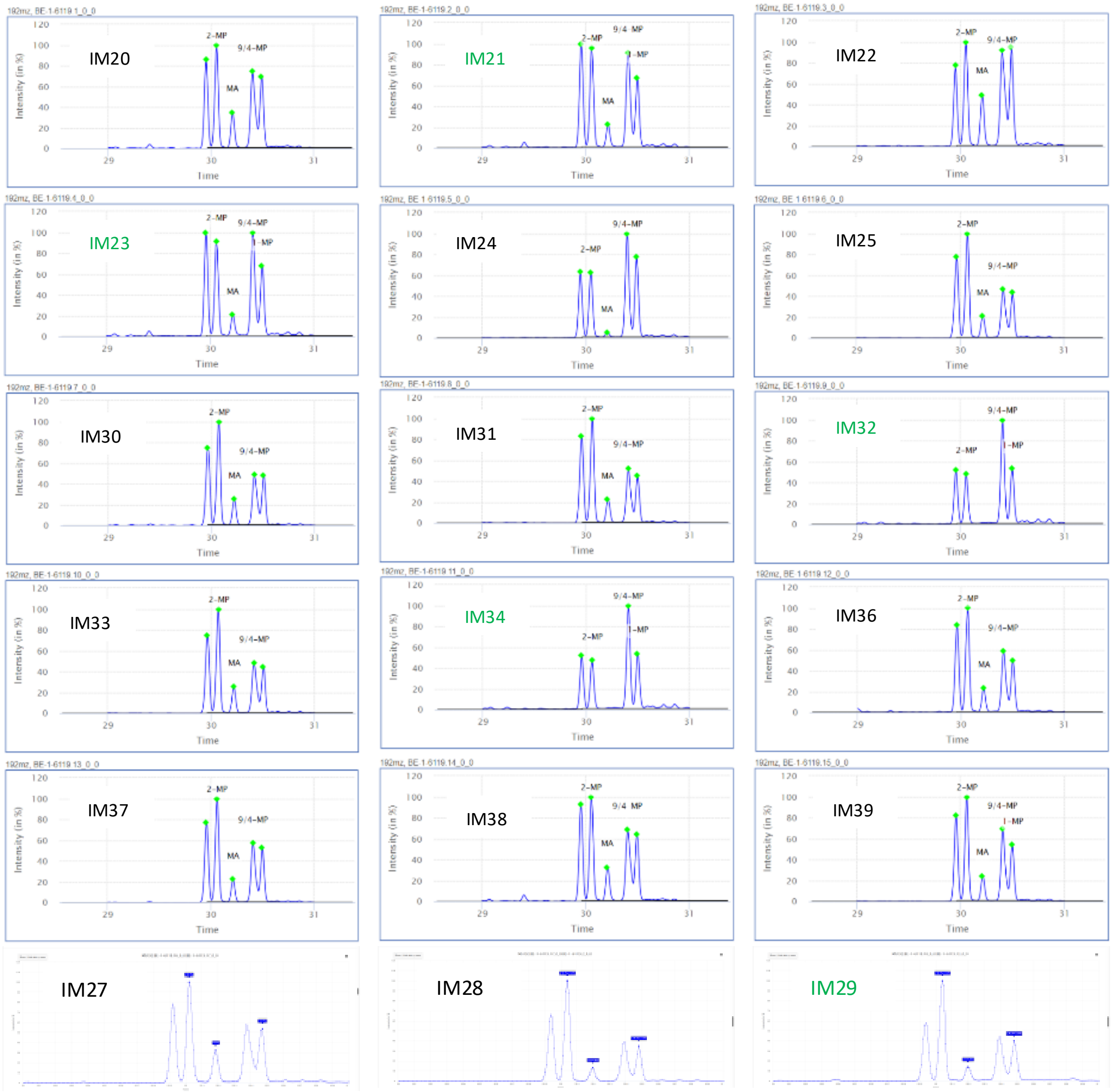


Figure 8 : Ion chromatogram of m/z 192 of all 18 samples.

These compounds do not add to the sulphur content. There are no patterns to conclude that samples are enhanced in sulphur.

5.5. C1-Dibenzothiophenes (m/z 198)

C1-Dibenzothiophenes (Figure 9) are very stable and contain sulphur. Calculations are made to compare the different samples.

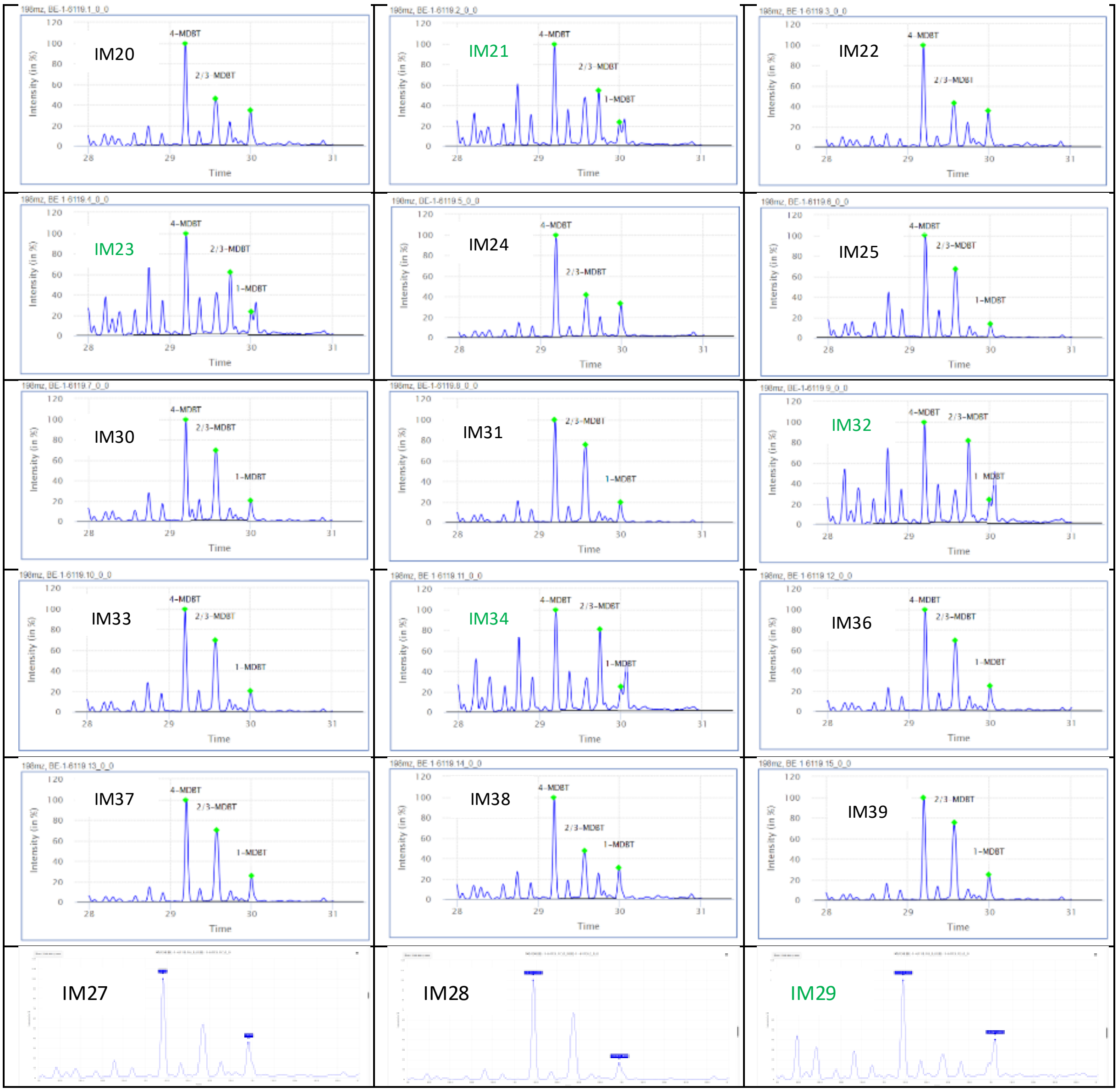


Figure 9 : Ion chromatograms of C1-Dibenzothiophenes of all 18 samples.

The peaks between 4-MDBT, 2/3-MDBT and 1-MDBT are visible in sample IM21, IM23, IM32 and IM34. According to the NIST library, these compounds are hetero compounds that do not contain sulphur, so relatively they are higher in ULSFO than in VLSFO.

A calculation was made between the methyl-dibenzothiophenes and the C2-phenantrenes (table 8). Sulphur compounds are often compared with C2-phenantrenes as it provides information about the sulphur content.

Table 5 : Calculations of some ratios of C1-Dibenzothiophenes vs. concentration and C2-phananthrenes.

Sample	4-M-Dbt/conc (x10^3)	1-M-Dbt/conc (x10^3)	4-M-Dbt/C2-phe (%)	1-M-Dbt/C2-phe (%)
IM20	152	52	13.6	4.60
IM21	119	25	0.7	0.15
IM22	117	40	1.7	0.60
IM23	78	17	0.7	0.15
IM24	107	34	2.0	0.63
IM25	548	73	1.1	0.15
IM30	626	125	0.9	0.18
IM31	722	140	2.5	0.48
IM32	58	13	0.9	0.19
IM33	589	118	1.1	0.21
IM34	70	15	0.9	0.18
IM36	336	82	1.7	0.41
IM37	372	93	0.9	0.24
IM38	122	37	1.0	0.31
IM39	410	100	2.5	0.61
IM27	96	32	1.2	0.39
IM28	841	128	2.2	0.33
IM29	122	16	1.2	0.15

The methyl-dibenzothiophenes / oil concentration ratio is markedly lower in samples IM21, IM23, IM29, IM32 and IM34 (ULSFOs), as is the ratio 1-and 4-methyl-dibenzothiophenes/C2-Phenantrenes.

5.6. C2-dibenzothiophenes (m/z 212)

These compounds contain sulphur. The ratio C2-dibenzothiophenes vs. C2-phenantrenes (C2-dbt_s/C2-phe_s) provides information about the sulphur content of the sample (Mössner, 1999). Figure 10 show the ion chromatograms of C2-dibenzothiophenes.

Figure 10 : Ion chromatogram of C2-dibenzothiophenes of all 18 samples.

When looking at the C2-dibenzothiophenes (group of peaks between the retention times 30.6 and 32.3 minutes), the intensity in ULSFOs are the lowest of all (here not visible because highest peak is set to 100 %). For calculations, the whole group is integrated. In table 9, the area of the group C2-

dibenzothiophenes/concentration and the ratio C2-dibenzeothiophenes/C2-phenantrenes are calculated.

Table 6 : Calculations of C2-dibenzothiophenes ratios.

Sample	C2-dbt/conc (x10^4)	C2-dbt/C2-phe (%)
IM20	184	163.7
IM21	131	7.9
IM22	145	21.6
IM23	86	7.7
IM24	119	22.0
IM25	415	8.4
IM30	727	10.4
IM31	531	18.2
IM32	66	9.8
IM33	564	10.1
IM34	81	9.9
IM36	346	17.4
IM37	483	12.1
IM38	155	13.1
IM39	460	28.1
IM27	126	15.4
IM28	773	20.0
IM29	129	12.2

Here samples IM21, IM23, IM29, IM32 and IM34 (ULSFO) show the lowest area/conc and one of the lowest ratio C2-dbt/C2-phe.

5.7. C2-Phenanthrenes (m/z 206)

C2-phenanthrenes are used to calculate the ratio C2-dbt/C2-phen. The peak intensity for this group is different, but no conclusions regarding sulphur content can be made from these patterns. Figure 11 shows the ion chromatograms of the C2-Phenanthrenes of all samples.

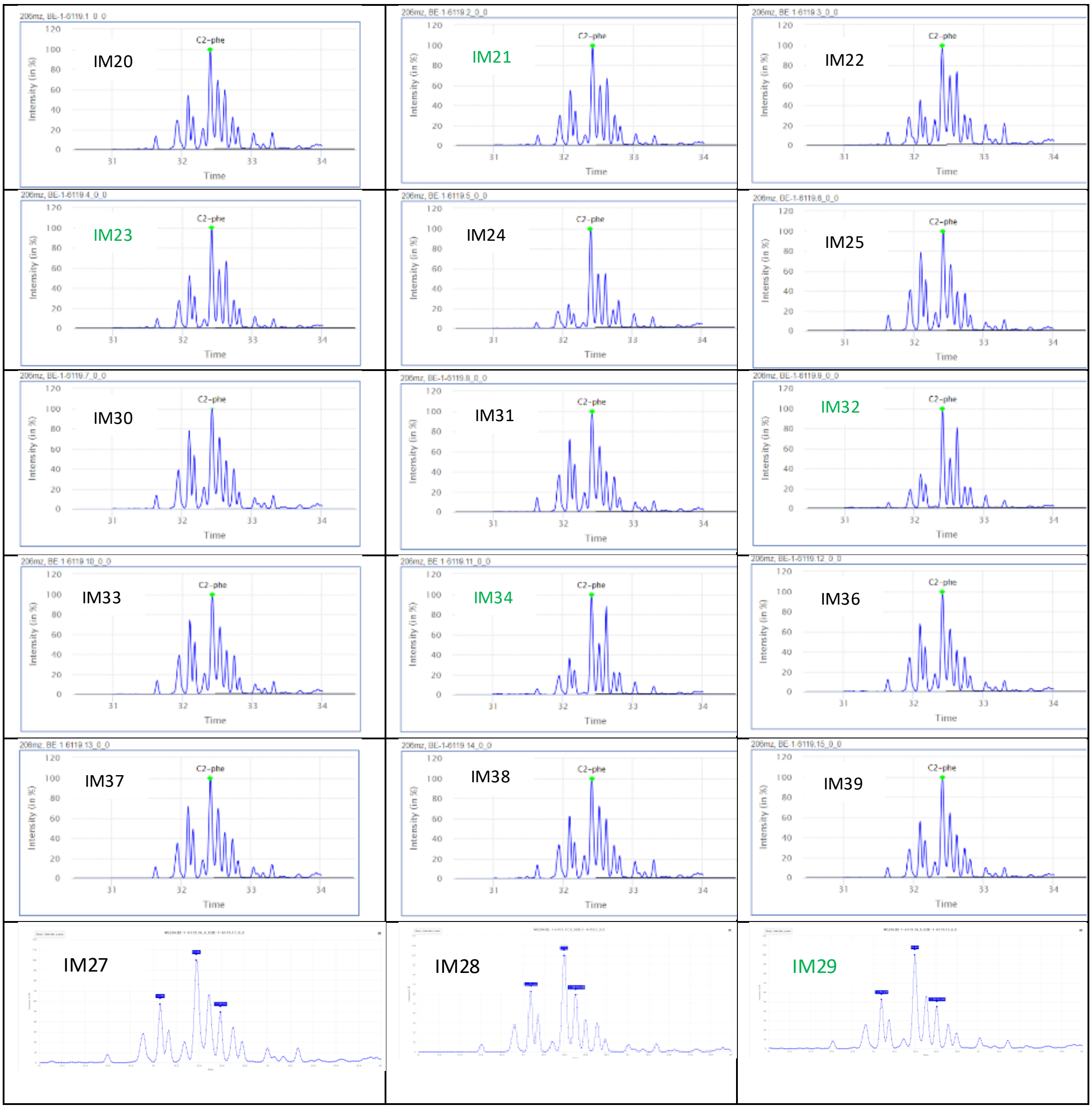


Figure 11 : Ion chromatograms of the C2-Phenantrenes of all 18 samples.

5.8. C2-benzothiophenes (m/z 162)

These compounds contain sulphur. The ratio C2-dibenzothiophenes/C2-phenantrenes provides information about the sulphur content of the sample.

Figure 12 : Ion chromatograms of all samples

When looking at the C2-benzothiophenes (Figure 12: group of peaks between the retention times 19.15 and 20.30) noting immediately stands out. For the calculations, the whole group is integrated.

Table 7 : Calculations of the C2-benzothiophenes.

Sample	C2-bt/conc (x10^4)	C2-bt/C2-phe (%)
IM20	54	48.2
IM21	20	1.2
IM22	48	7.1
IM23	14	1.2
IM24	2	0.4
IM25	68	1.4
IM30	159	2.3
IM31	300	10.3
IM32	8	1.2
IM33	93	1.7
IM34	10	1.2
IM36	112	5.6
IM37	29	0.7
IM38	40	3.4
IM39	56	3.5
IM27	22	2.7
IM28	53	1.4
IM29	17	1.6

Samples IM21, IM23, IM29, IM32 and IM34 are ULSFO. These compounds (C2-bt) are lighter and are present in ULSFO. The ULSFO do not show the lowest, but also not the highest ratios/concentrations.

5.9. C3-dibenzothiophenes (m/z 226)

These compounds contain sulphur. The ratio C2-dibenzothiophenes/C2-phenantrenes provides information about the sulphur content of the sample (Mössner, 1999). Calculations are made of C3-dibenzothiophenes/C2-Phenantrenes (table 11). Ionchromatograms can be seen in Figure 13.

Figure 13 : Ion chromatograms of C3-dibenzothiophenes of all 18 samples.

Looking at the C3-dibenzothiophenes (group of peaks between the retention times 32.30 and 34.20 minutes) patterns are similar but differ in intensity. The chromatograms with their intensity can be seen in Appendix 2 : figure 17.

Table 8 : Calculations of the C3-dibenzothiophenes.

Sample	C3-dbt/conc (x10^4)	C3-dbt/C2-phe (%)
IM20	194	173
IM21	114	7
IM22	150	22
IM23	75	7
IM24	125	23
IM25	294	6
IM30	719	10
IM31	361	12
IM32	56	8
IM33	489	9
IM34	69	8
IM36	291	15
IM37	499	13
IM38	184	16
IM39	392	24
IM27	150	18
IM28	683	18
IM29	121	11

Sample IM21, IM23, IM32 and IM34 (ULSFO) show the lowest ratio C3-dbt/conc and C3-dbt/C2-phe.

5.10. Steranes (m/z 217), Diasteranes (m/z 218) and triaromatic steranes (m/z 231)

The most common diagnostic biomarkers used in organic geochemistry and oil spill identification include terpenes, steranes and mono- and triaromatic steranes. Compared to n-paraffins or acyclic isoprenoids, these biomarkers are particularly useful because of their specificity, diversity and resistance to biodegradation and weathering.

In condensed samples and lighter fuels, these compounds are at a low level or absent (Peters, 2005) (Stout, 2008) (Wang, 1995) (Yang, 2016). The higher-boiling biomarkers are more pronounced in oils of higher viscosity. Ionchromatograms are presented in Figure 14, 15 and 16. The ionchromatograms with their intensities can be found in Appendix 2 : Figure 19, 20 and 21.

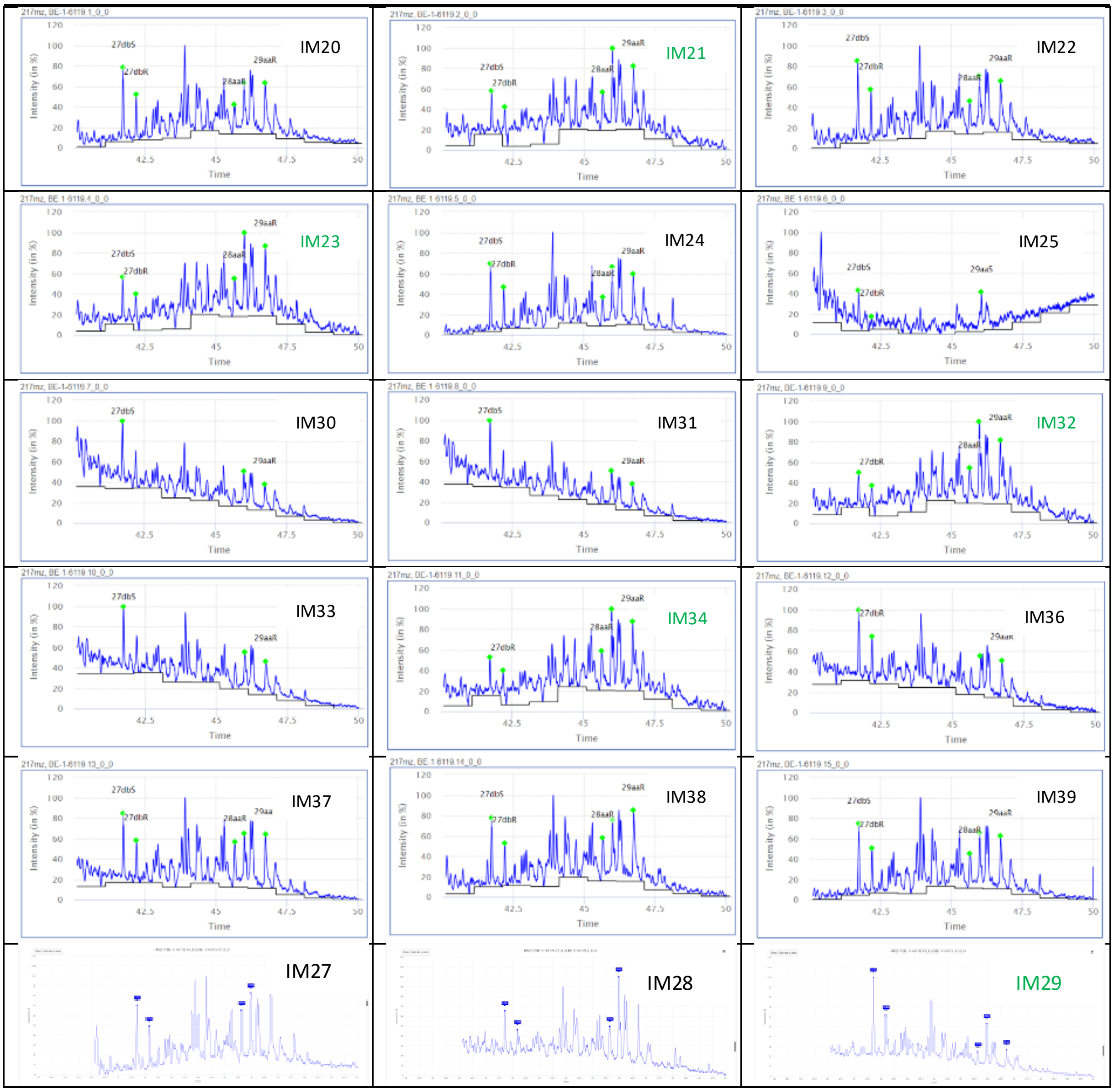


Figure 14 : ion chromatograms of steranes (m/z 217) of all 18 samples.

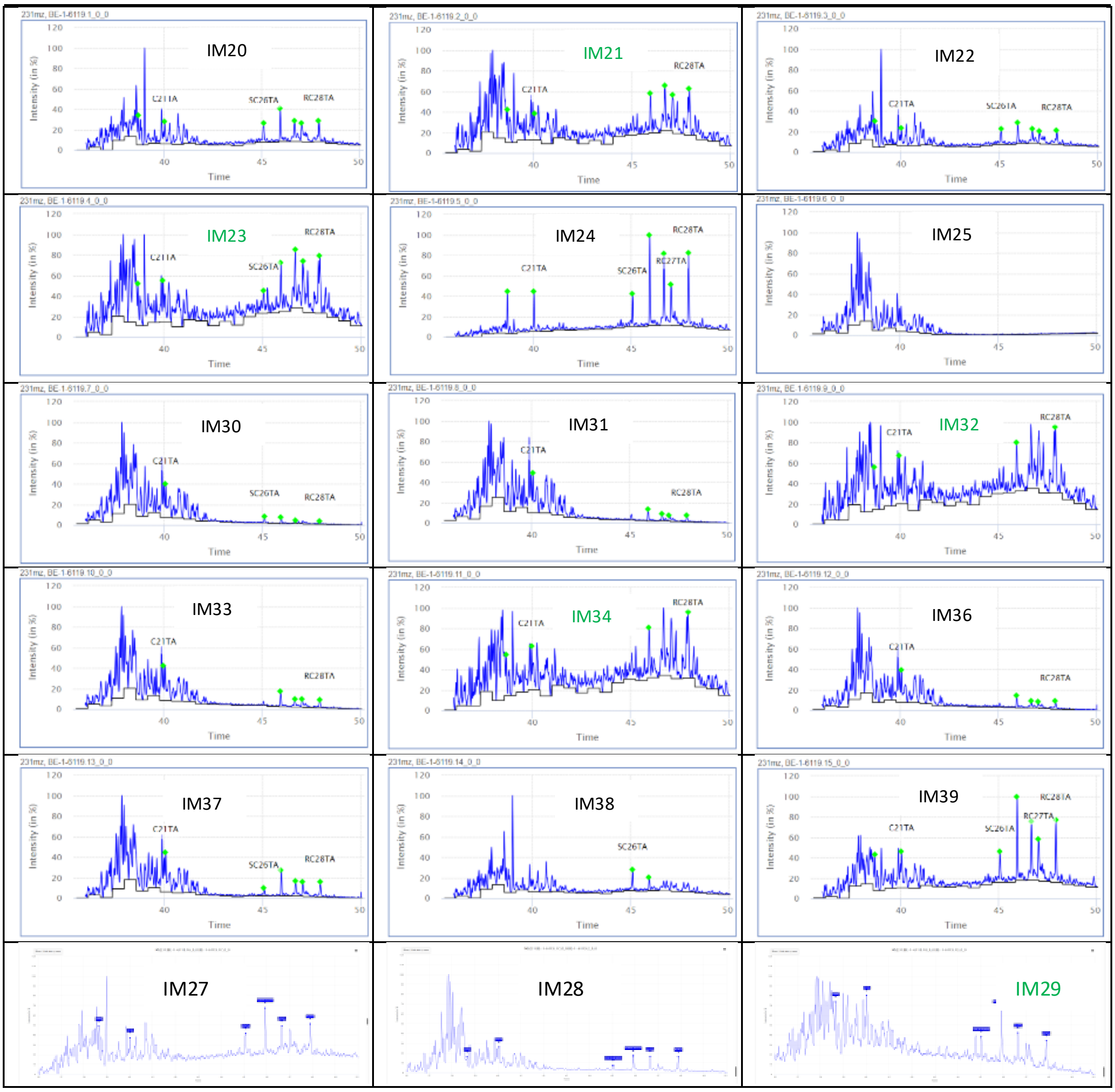


Figure 15 : Ion chromatograms of triaromatic steranes (M/z 231) of all 18 samples.

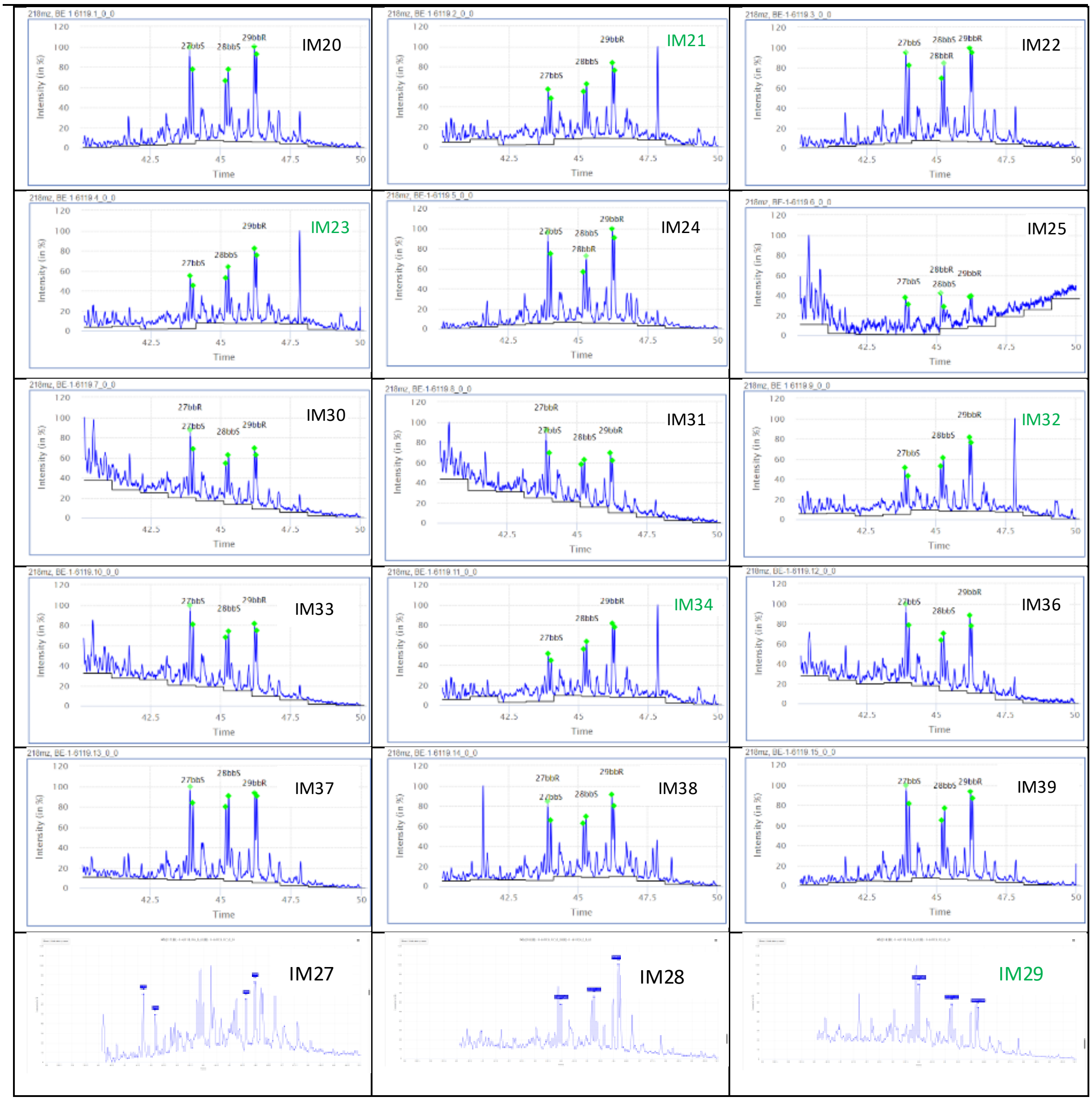


Figure 16 : Ion chromatograms of diasteranes (m/z 218) of all 18 samples.

6. Overall results

Analysis of the 18 samples led to the following conclusions :

Total ion chromatogram (TIC) analysis.

It is not visible from the TIC analysis whether the oil is low in sulphur. The concentration of naphthalenes, lower alkanes and waxes can vary. All combinations are possible.

Cosiweb2 comparison.

All samples were imported in CosiWeb2. Some samples show a correlation of >0.99. These samples are compared in Annex 3.

Inspection of the ion chromatograms

Hopanes : Sample IM25 shows a relatively low concentration of hopanes. Samples IM 21, IM23, IM27, IM34 and IM36 show a relatively high concentration of hopanes. Samples IM24, IM27, IM31, IM33, IM36, IM37 and IM38 show a relatively high concentration of Oleanane/Lupane which means they might be blended with sweet-crude oil.

Benzo(b)naphtho(1,2-d)- thiophene (BNT) : samples IM21, IM23, IM29, IM32 and IM34 show a relatively low concentration of BNT. These are the ULSFOs

C1-Dibenzothiophenes : samples IM21, IM23, IM29, IM32 and IM34 show a relatively low concentration of C1-dibenzothiophenes.

C2-Dibenzothiophenes : sample IM21, IM23, IM29, IM32 and IM34 show a relatively low concentration of C2-dibenzothiophenes.

C2-benzothiophenes : No conclusions could be drawn.

C3-Dibenzothiophenes : Samples IM21, IM23, IM29, IM32 and IM34 show a relatively low concentration of C3-dibenzothiophenes.

Steranes, Diasteranes and triaromatic steranes : relatively low in sample IM31, IM32, IM34 and IM36.

In table 12 a summary of the samples is given.

Table 9 : summary of the samples.

	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
Naphthalenes			-		--	+	+	++		+	-	+				-		
lower alkanes		+		+	-				++	-	+				-	-		+
waxes	-	++	-	+		-		-	+		+							+
Hopanes		+		+		-					+	+				+		
Oleanane					+			+		+		+	+	+		+	-	
BNT		-		-					-		-							-
C1-Dibenzothiophenes		-		-					-		-							-
C2-Dibenzothiophenes		-		-					-		-							-
C3-Dibenzothiophenes		-		-					-		-							-
Steranes	+				++	--		-	-			-	+	+	+	+		
Diasteranes	+				++	--		-	-			-	+	+	+	+		
Triaromatic steranes	+				++	--		-	-			-	+	+	+	+		

7. Overall conclusion

From the Total ion chromatogram, it is not possible to conclude now if the oil is a low sulphur oil.

The Ion chromatograms and the calculations of the C1-, C2-, C3-dibenzothiophens and BNT are indicative for less sulphur present in an oil.

The lack of Steranes, diasteranes and triaromatic steranes suggests there are no/few waxes present (Peters, 2005) (Stout, 2008) (Wang, 1995) (Yang, 2016). This was so with the HFOs. But for ULSFOs, this is not always the case (cfr IM32).

Appendix 1 : Calculation of ratios.

It is suggested that the oil samples are analysed in accordance with NBN EN 15522-2 (2023) and uploaded in COSI-2 to simplify the reporting of oil fingerprinting. Laboratories that do not have access to COSI-2 can use their inhouse method to calculate the ratios. Description of the components and recommended ratios are given in NBN EN 15522-2 (2023).

The following parameters are suggested (ratios in Table 13):

- Screening GC/FID
  - GC chromatograms
  - Calculation of the isoprenoid ratios (from COSI-2)
- GC/MS SIM-analysis (data from COSI-2)
  - Selected ion chromatograms (can be found in the report)
    - Hopanes (m/z 191) : fig 3 and 4
    - Triaromatic steranes (m/z 231) : fig 15
    - C4-phenanthrenes (m/z 234) : fig 6
    - C1-phenanthrenes (m/z 192) : fig 8
    - C1-dibenzothiophenes (m/z 198) : fig 9
    - C1-methylfluoranthenes/pyrenes (m/z 216) : fig 7

Table 10 : Calculations of the ratios of the 18 samples.

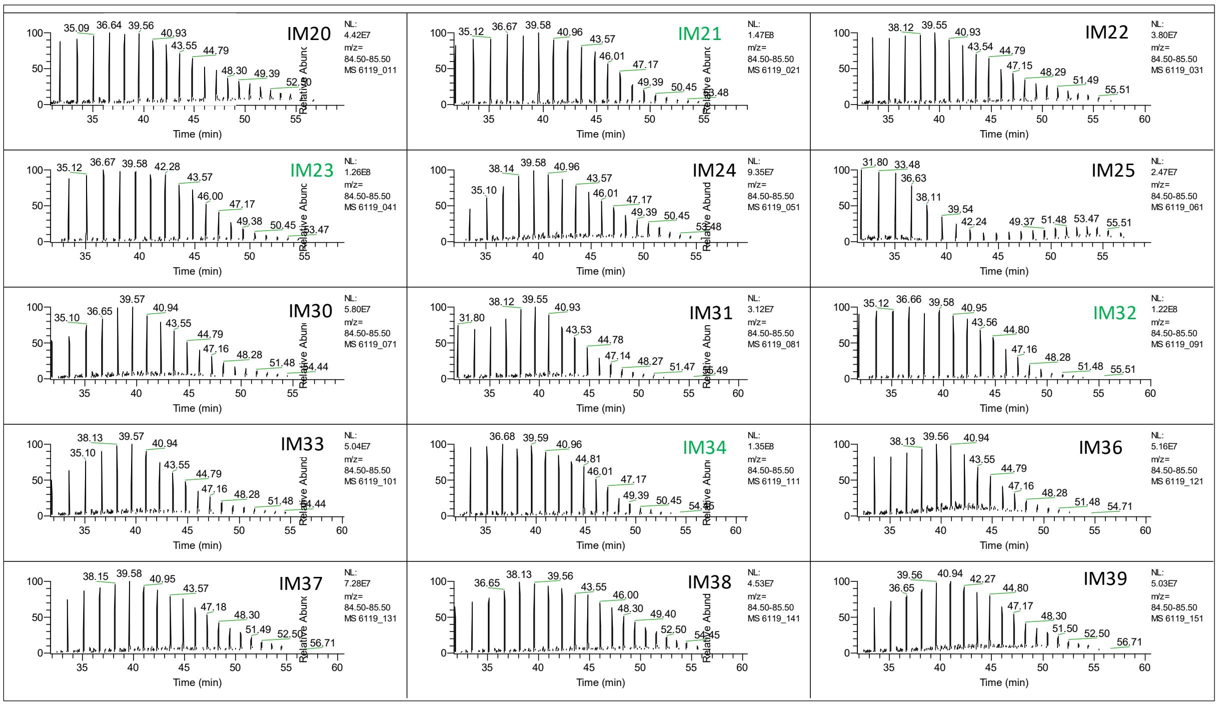
In colour: samples that could originate from the same batch.

	m/z	Ratios	Source	IM20	IM21	IM22	IM23	IM24	IM25	IM30	IM31	IM32	IM33	IM34	IM36	IM37	IM38	IM39	IM27	IM28	IM29
GC/FID		C17/pristane	COSI	2.56	1.72	3.36	1.75	1.28	0.94	1.26	1.31	1.70	0.93	1.76	1.34	1.67	2.62	1.43	1.67	1.62	1.48
		C18/phytane	COSI	3.57	3.29	3.85	3.72	1.77	1.23	3.59	3.50	3.16	2.94	3.17	2.23	2.73	4.20	2.44	2.07	3.14	1.20
		Pristane/phytane	COSI	0.80	1.49	0.76	1.47	0.74	0.95	0.99	1.11	1.54	0.93	1.52	1.17	0.86	0.86	0.80	0.78	4.99	2.06
GC/MS	191	Ts/30ab	COSI	0.18	0.17	0.17	0.16	0.19	0.15	0.23	0.27	0.17	0.21	0.16	0.21	0.19	0.19	0.15	0.22	0.22	4.48
	191	Tm/30ab	COSI	0.19	0.17	0.20	0.17	0.16	1.13	0.29	0.37	0.18	0.41	0.18	0.20	0.16	0.21	0.19	0.22	0.27	4.48
	191	28ab/30ab	COSI	0.13	0.10	0.14	0.09	0.10	0.00	0.12	0.10	0.09	0.11	0.10	0.06	0.06	0.07	0.09	0.11	0.15	2.78
	191	29ab/30ab	COSI	0.48	0.59	0.48	0.55	0.53	0.74	0.73	0.74	0.59	0.66	0.57	0.64	0.50	0.55	0.52	0.44	0.64	2.78
	191	30O/30ab	COSI	0.08	0.02	0.03	0.02	0.28	0.00	0.08	0.19	0.02	0.14	0.02	0.33	0.21	0.59	0.08	0.19	0.00	0.81
	191	31abS/30ab	COSI	0.33	0.22	0.30	0.22	0.33	0.30	0.36	0.29	0.22	0.36	0.21	0.36	0.32	0.27	0.34	0.33	0.30	0.81
	191	30G/30ab	COSI	0.09	0.26	0.10	0.25	0.04	0.00	0.11	0.11	0.26	0.09	0.25	0.13	0.07	0.06	0.10	0.23	0.12	3.95
	217	27dbR/27dbS	Manual integration	0.59	0.61	0.61	0.58	0.60	0.00	0.59	0.54	0.63	0.51	0.62	0.60	0.58	0.64	0.60	0.62	0.61	0.52
	218	27bb/29bb	Manual integration	1.02	0.72	0.97	0.70	0.97	1.02	1.12	1.14	0.62	1.09	0.65	1.01	1.04	0.95	1.08	1.18	0.75	1.82
	231	TASC26/RC26+SC27	Manual integration	0.52	0.40	0.65	0.39	0.35	0.00	0.95	0.45	0.00	0.30	0.42	0.32	0.27	1.53	0.32	0.45	0.29	0.46
	231	TASC28/RC26+SC27	Manual integration	0.56	1.14	0.57	1.18	0.77	0.00	0.46	0.53	1.29	0.50	1.33	0.54	0.53	0.61	0.68	0.52	0.96	0.58
	231	TARC27/RC26+SC27	Manual integration	0.61	1.13	0.61	1.17	0.78	0.00	0.51	0.52	1.40	0.52	1.27	0.62	0.56	0.57	0.72	0.62	1.00	0.52
	231	TARC28/RC26+SC27	Manual integration	1.48	1.47	1.09	1.39	0.82	2.37	2.10	2.26	0.92	2.30	0.90	2.05	1.94	1.61	1.89	1.89	2.90	2.58
	192	2MP/1MP	Manual integration	0.50	0.32	0.52	0.29	0.05	0.48	0.51	0.49	0.00	0.56	0.00	0.45	0.42	0.50	0.44	0.59	0.36	0.34
	192	MA/1MP	Manual integration	2.95	4.77	2.93	4.72	3.12	7.46	5.00	5.15	4.62	4.98	4.71	4.12	3.98	3.34	4.10	2.99	6.55	7.77
	198	4MD/1MD	Manual integration	0.15	0.14	0.15	0.14	0.16	0.11	0.18	0.19	0.08	0.16	0.08	0.10	0.16	0.15	0.14	0.19	0.10	0.18
	216	2MF/4MPy	Manual integration	0.61	0.58	0.65	0.63	1.23	0.21	0.39	0.44	0.59	0.32	0.60	0.28	0.36	0.61	0.43	0.44	0.20	0.45
	216	2MPy/4MPy	Manual integration	0.86	0.82	0.86	0.82	0.38	1.07	1.01	0.91	0.50	0.97	0.48	0.99	0.99	0.78	0.89	0.79	1.24	0.77
	216	1MPy/4MPy	Manual integration	0.72	0.81	0.67	0.79	0.63	0.93	0.94	0.84	0.58	0.92	0.57	0.94	0.95	0.76	0.88	0.69	1.05	0.66
	234	Retene/TMPhe	Manual integration	0.84	0.10	1.12	0.12	0.78	0.17	0.16	0.15	0.09	0.15	0.09	0.15	0.15	0.67	0.12	0.32	0.07	0.47
	234	BNT/TMPhe	Manual integration	2.14	0.25	2.88	0.20	0.51	0.18	0.34	0.30	0.15	0.25	0.16	0.53	0.40	1.64	0.51	1.47	0.44	0.13
		Sulphur content																			
GC/MS	198/191	1M-DBT/30ab	Manual integration	0.08	0.02	0.08	0.02	0.04	2.75	0.53	1.44	0.01	0.52	0.01	0.60	0.18	0.04	0.18	0.03	0.32	0.04
	198/191	4M-DBT/30ab	Manual integration	0.25	0.09	0.24	0.08	0.12	20.54	2.66	7.43	0.06	2.58	0.06	2.48	0.70	0.12	0.75	0.08	2.11	0.31
	234/191	BNT/30ab	Manual integration	0.33	0.05	0.37	0.03	0.07	2.20	1.04	0.87	0.02	0.59	0.02	0.80	0.41	0.18	0.17	0.14	0.60	0.04
		If 30ab is small or not present (lighter products)																			
	198/192	1M-DBT/1MP	Manual integration	0.09	0.03	0.11	0.03	0.12	0.03	0.04	0.09	0.05	0.05	0.05	0.09	0.06	0.06	0.13	0.09	0.07	0.02
	198/192	4M-DBT/1MP	Manual integration	0.26	0.16	0.33	0.16	0.37	0.20	0.20	0.46	0.22	0.23	0.22	0.37	0.25	0.21	0.54	0.27	0.49	0.19
	234/234	BNT/TMPhe	Manual integration	2.14	0.25	2.88	0.20	0.51	0.18	0.34	0.30	0.15	0.25	0.16	0.53	0.40	1.64	0.51	1.47	0.44	0.13
		Optional ratios sulphur content																			
	212/206	C2-DBT/C2-Phe	Manual integration	43.17	0.08	0.22	0.08	0.22	0.08	0.10	0.18	0.10	0.10	0.10	0.17	0.12	0.13	0.28	0.15	0.20	0.12
	212/206	C3-DBT/C2-Phe	Manual integration	45.53	0.07	0.22	0.07	0.23	0.06	0.10	0.12	0.08	0.09	0.08	0.15	0.13	0.16	0.24	0.18	0.18	0.11
	234/206	BNT/C2-Phe	Manual integration	4.78	0.00	0.03	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.02	0.01	0.02	0.01	0.00
	162/206	C2-BT/C2-Phe	Manual integration	12.72	0.01	0.07	0.01	0.00	0.01	0.02	0.10	0.01	0.02	0.01	0.06	0.01	0.03	0.03	0.03	0.01	0.02

Appendix 2 : ion chromatograms with their intensity

Alkanes (Figure 17).

Small samples



Large samples

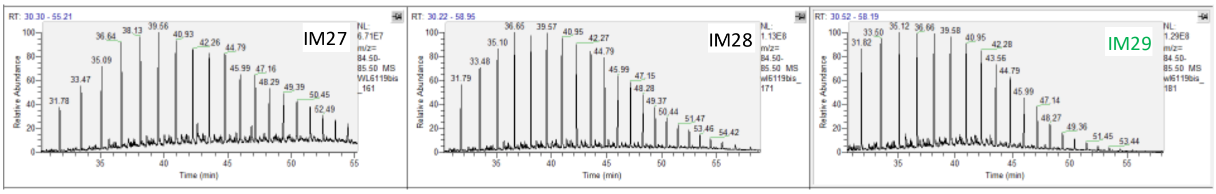
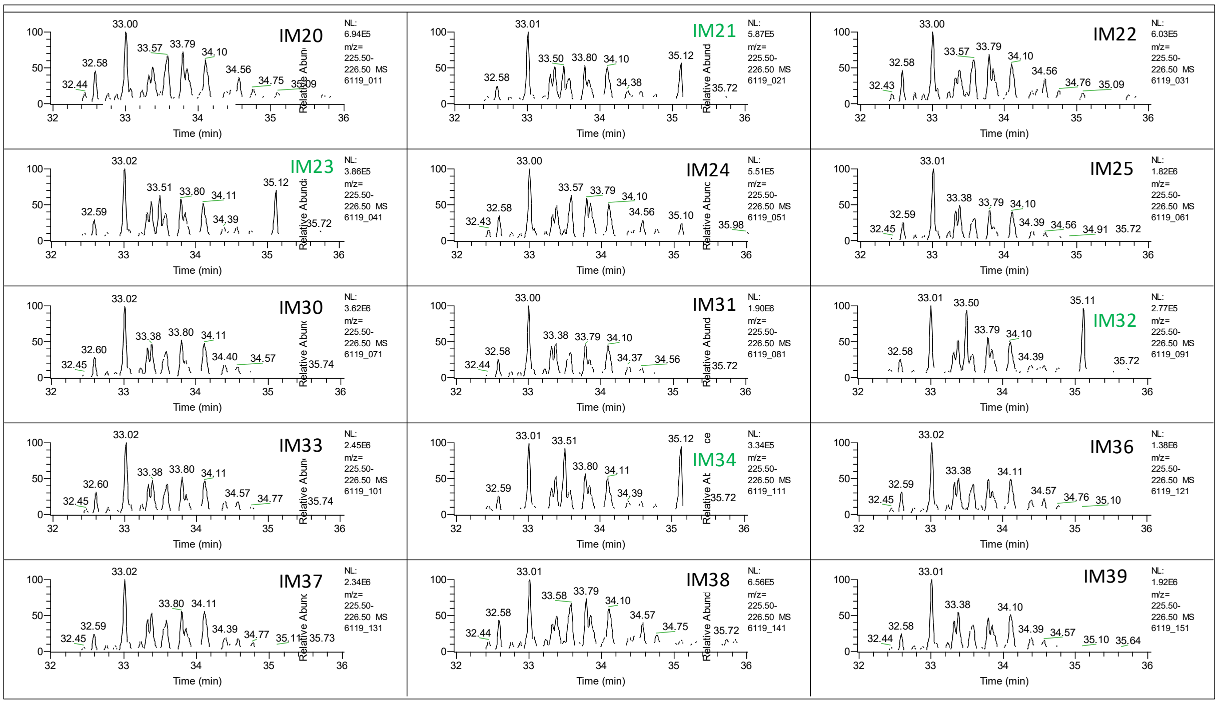


Figure 17 : ion chromatogram of alkanes (m/z 85) >C20 for all samples.

C3-dibenzothiophenes (Figure 18).

Small samples



Large samples

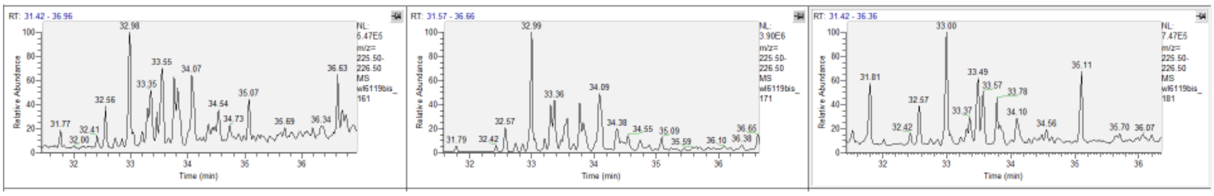
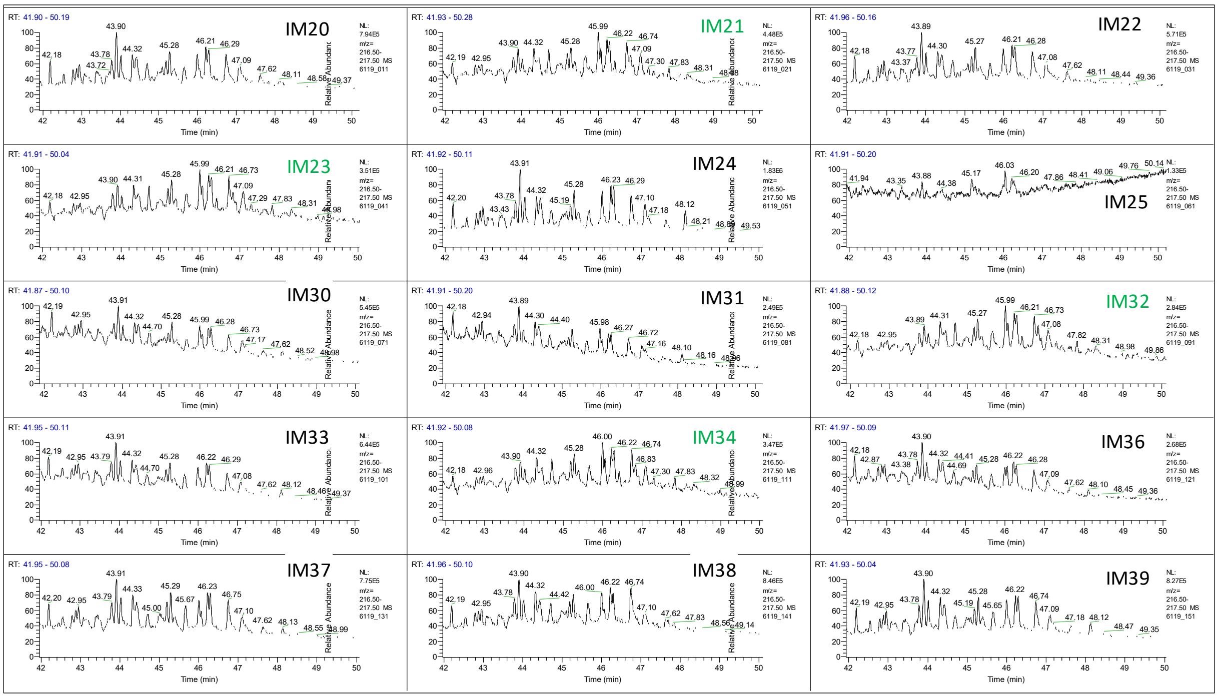


Figure 18 : Ion chromatograms of C3-dibenzothiophenes with their intensity for all samples.

Steranes (Figure 19).

Small samples



Large Samples

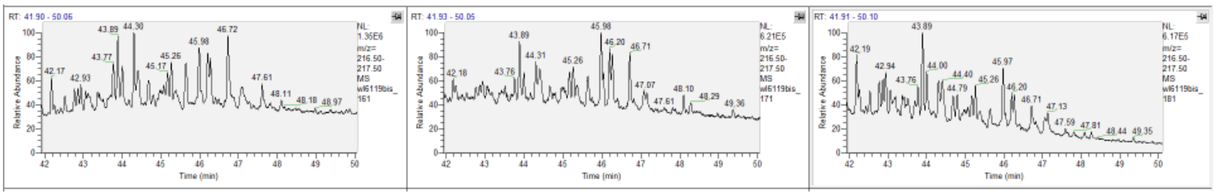
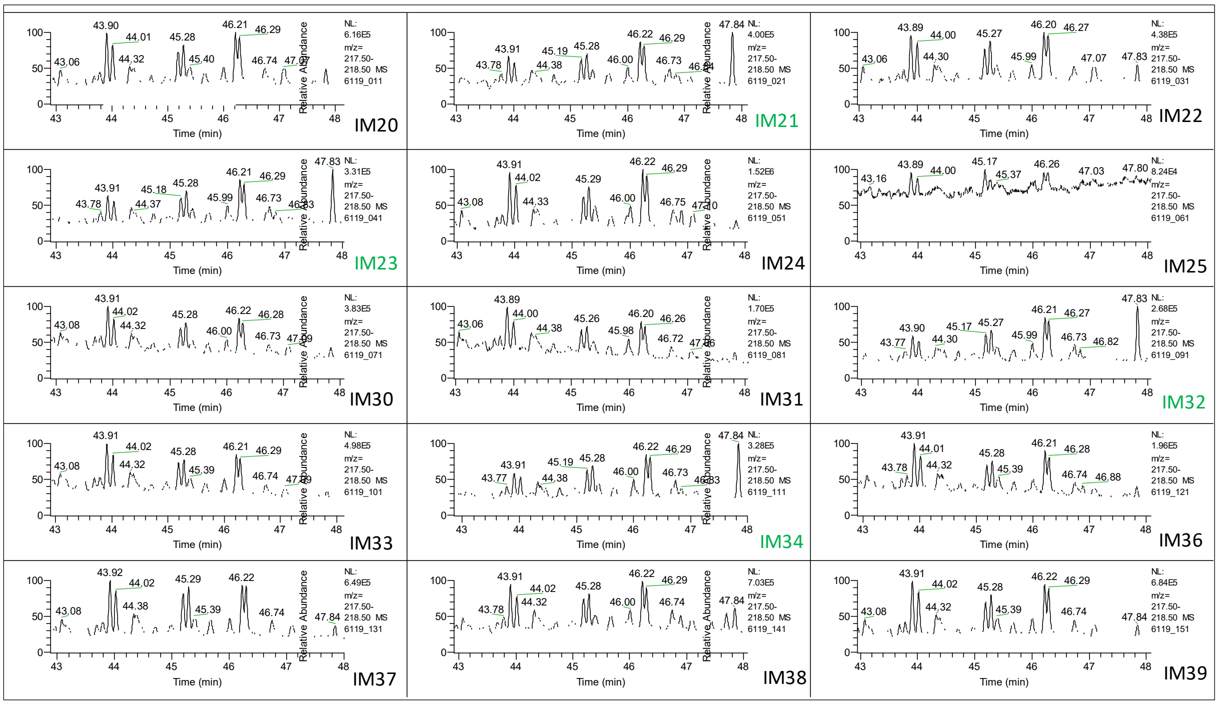


Figure 19 : ion chromatograms of steranes (m/z 217) of samples with their intensity for all samples.

Diasteranes (Figure 20).

Small Samples



Large samples

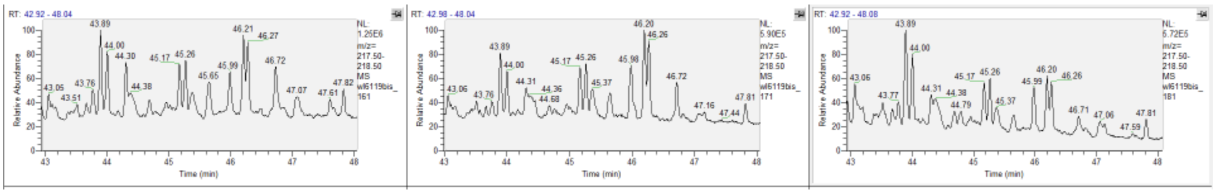
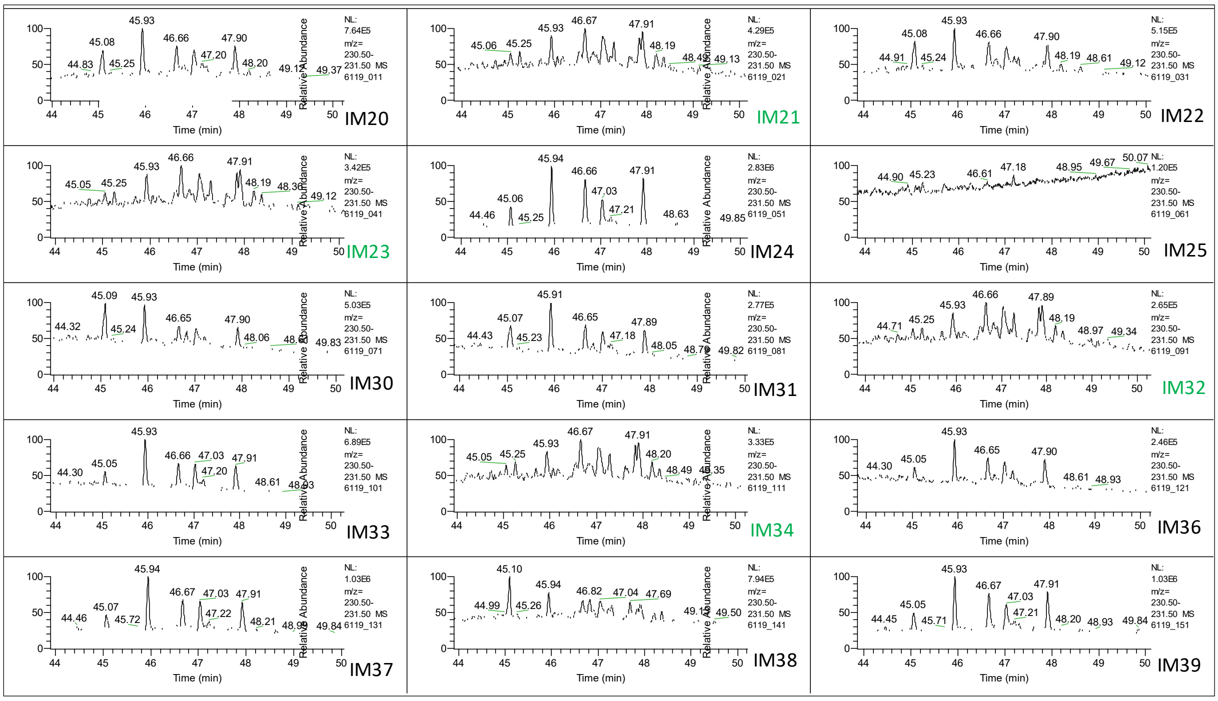


Figure 20 : Ion chromatograms of diasteranes (m/z 218) of all 18 samples with their intensity.

Steranes (Figure 21).

Small Samples



Large Samples

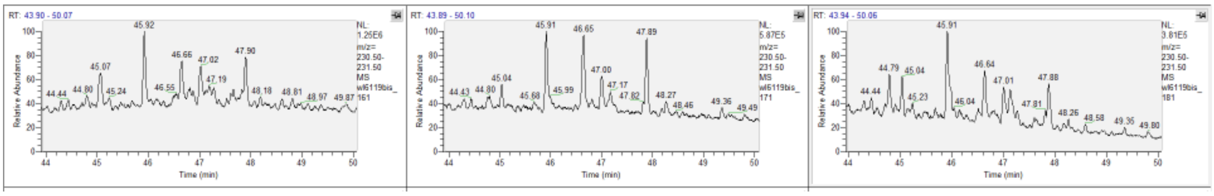
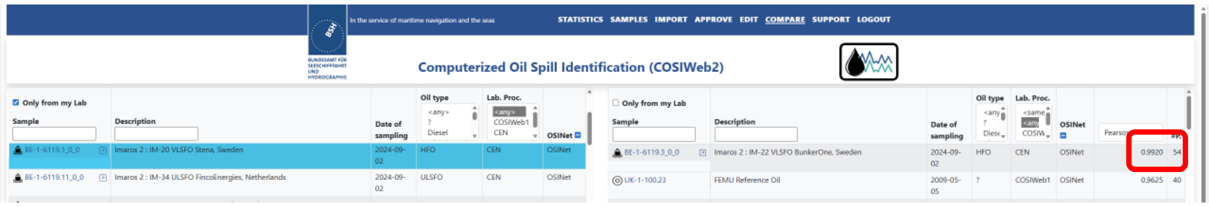


Figure 21 : Ion chromatograms of triaromatic steranes (m/z 231) of all 18 samples with their intensity.

Appendix 3 : Comparison of the samples

Samples were uploaded in Cosi 2. Samples that have a correlation higher than 0.99 are compared.

• IM20 -IM22



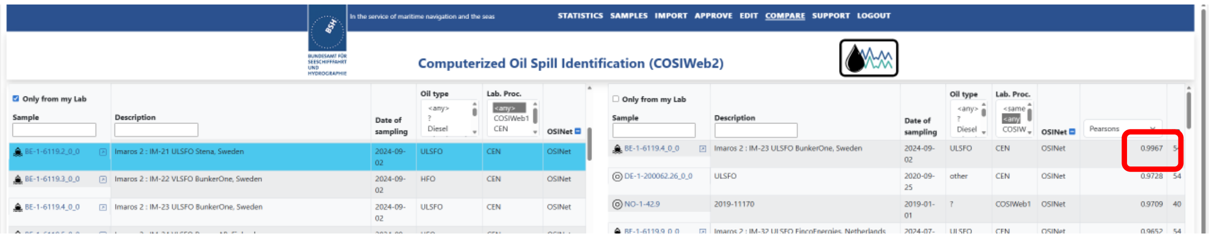
☑ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet
BE-1-6119.1_0_0	Imaros 2 : IM-20 VLSFO Stena, Sweden	2024-09-02	HFO	CEN	OSINet
BE-1-6119.11_0_0	Imaros 2 : IM-34 ULSFO FincoEnergies, Netherlands	2024-09-02	ULSFO	CEN	OSINet

☐ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet	Pearsons	#P.
BE-1-6119.3_0_0	Imaros 2 : IM-22 VLSFO BunkerOne, Sweden	2024-09-02	HFO	CEN	OSINet	0.9920	54
UK-1-100.23	FEMU Reference Oil	2009-05-05	?	COSIWeb1	OSINet	0.9625	40

• IM21-IM23



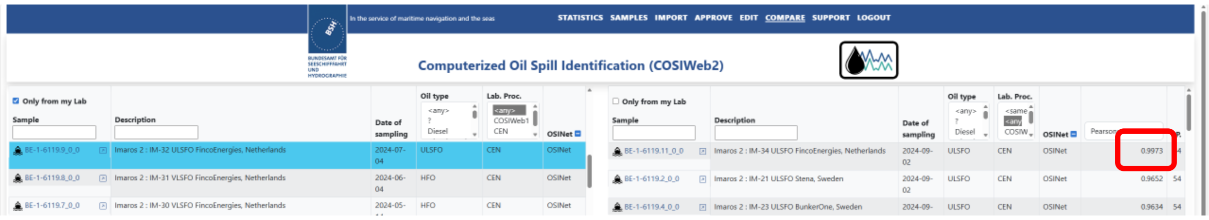
☑ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet
BE-1-6119.2_0_0	Imaros 2 : IM-21 ULSFO Stena, Sweden	2024-09-02	ULSFO	CEN	OSINet
BE-1-6119.3_0_0	Imaros 2 : IM-22 VLSFO BunkerOne, Sweden	2024-09-02	HFO	CEN	OSINet
BE-1-6119.4_0_0	Imaros 2 : IM-23 ULSFO BunkerOne, Sweden	2024-09-02	ULSFO	CEN	OSINet

☐ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet	Pearsons	
BE-1-6119.4_0_0	Imaros 2 : IM-23 ULSFO BunkerOne, Sweden	2024-09-02	ULSFO	CEN	OSINet	0.9967	54
DE-1-200062.26_0_0	ULSFO	2020-09-25	other	CEN	OSINet	0.9728	54
NO-1-42.9	2019-11170	2019-01-01	?	COSIWeb1	OSINet	0.9709	40
BE-1-6119.9_0_0	Imaros 2 : IM-32 ULSFO FincoEnergies, Netherlands	2024-07-	ULSFO	CEN	OSINet	0.9652	54

• IM32-IM34



☑ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet
BE-1-6119.9_0_0	Imaros 2 : IM-32 ULSFO FincoEnergies, Netherlands	2024-07-04	ULSFO	CEN	OSINet
BE-1-6119.8_0_0	Imaros 2 : IM-31 VLSFO FincoEnergies, Netherlands	2024-06-04	HFO	CEN	OSINet
BE-1-6119.7_0_0	Imaros 2 : IM-30 VLSFO FincoEnergies, Netherlands	2024-05-	HFO	CEN	OSINet

☐ Only from my Lab

Sample	Description	Date of sampling	Oil type	Lab. Proc.	OSINet	Pearsons	P.
BE-1-6119.11_0_0	Imaros 2 : IM-34 ULSFO FincoEnergies, Netherlands	2024-09-02	ULSFO	CEN	OSINet	0.9973	54
BE-1-6119.2_0_0	Imaros 2 : IM-21 ULSFO Stena, Sweden	2024-09-02	ULSFO	CEN	OSINet	0.9652	54
BE-1-6119.4_0_0	Imaros 2 : IM-23 ULSFO BunkerOne, Sweden	2024-09-	ULSFO	CEN	OSINet	0.9634	54

1. Comparison IM20 VLSFO with IM22 VLSFO.

Two VLSFOs from Sweden, one from an oil refinery (IM20), another from a bunker company (IM22).

1.1 Overlay of the chromatograms

Figure 22 shows the overlay of samples IM20 and IM22. The chromatograms look very similar. However in the area of the lower alkanes, differences in concentration are visible.

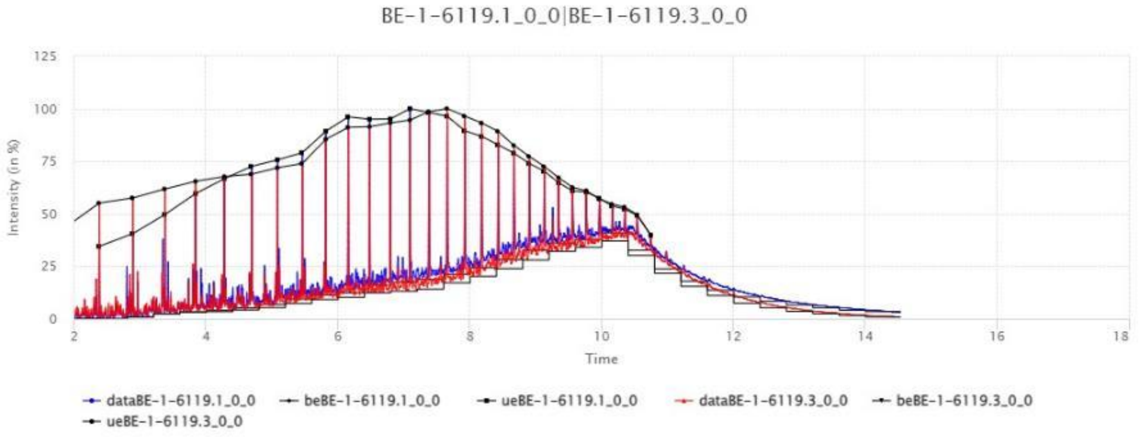


Figure 22 : Overlay of chromatograms of IM20 (bleu) and IM22 (red)

1.2 GC-PW plot

Figure 23 shows the PW plot (weathering curve) of samples IM20 and IM22, also showing a difference in the lower alkanes and a difference in the isoprenoid ratio (light blue dots). These are not in line with the other alkanes.

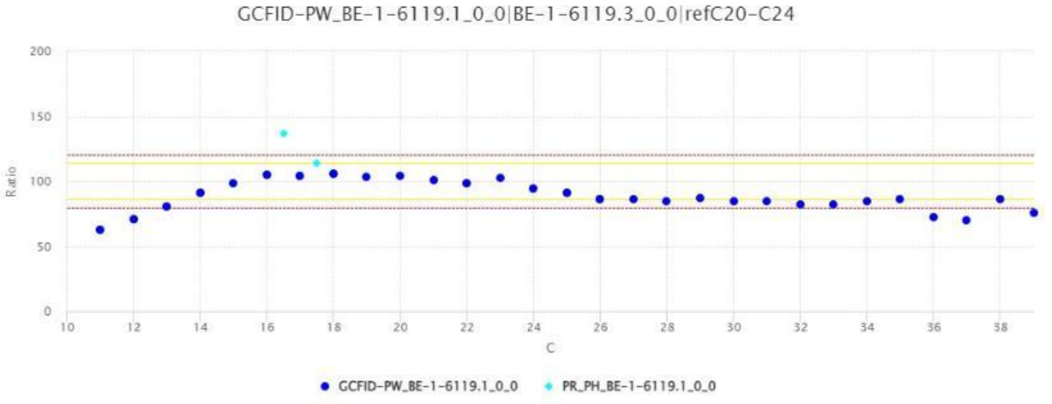


Figure 23 : GC-PW-plot of the comparison of sample IM20 with IM22.

1.3 Calculation of the isoprenoid ratios

Table 13 shows calculation of the isoprenoid ratios of sample IM20 vs. IM22 showing that 2 ratios are higher than the allowed difference of 14%.

Table 13 : Calculation of the diagnostic ratios of IM20 vs. IM22.

Param	Val 1	Val 2	D%
C17/Pris	2.56	3.36	26.89
Pris/Phyt	1.38	1.14	18.43
C1Phyt	3.57	3.85	7.41

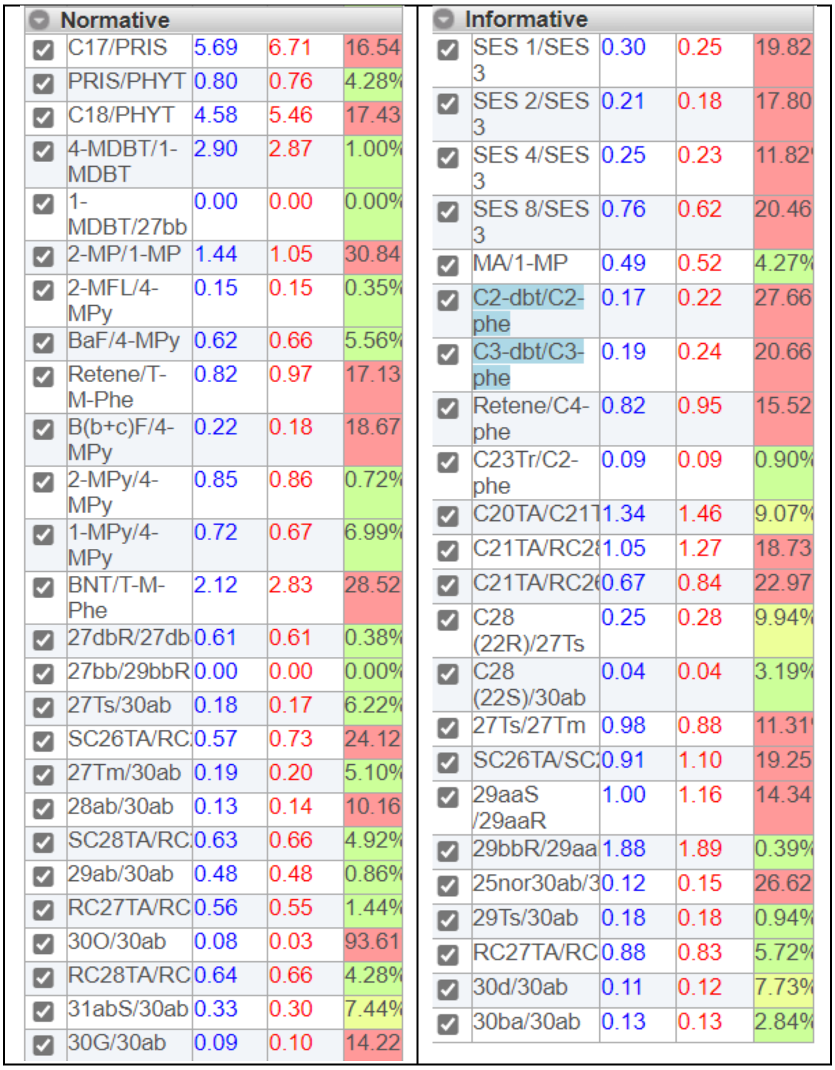
1.4 Conclusion of the total ion chromatograms

The oils do not match, though the different ion-chromatograms look very similar.

1.5 Ion ratios of the normative and informative ratios.

Table 14 shows the diagnostic ratios of the comparison of samples IM20 (blue) and IM22 (red). A lot of ratios have a relative difference of more than 14%, but most of them have a relative difference of <30% indicating a lot of similarity between the samples.

Table 14 : Normative and informative ratios of sample IM20 and IM22



	Normative			
☑	C17/PRIS	5.69	6.71	16.54
☑	PRIS/PHYT	0.80	0.76	4.28%
☑	C18/PHYT	4.58	5.46	17.43
☑	4-MDBT/1-MDBT	2.90	2.87	1.00%
☑	1-MDBT/27bb	0.00	0.00	0.00%
☑	2-MP/1-MP	1.44	1.05	30.84
☑	2-MFL/4-MPy	0.15	0.15	0.35%
☑	BaF/4-MPy	0.62	0.66	5.56%
☑	Retene/T-M-Phe	0.82	0.97	17.13
☑	B(b+c)F/4-MPy	0.22	0.18	18.67
☑	2-MPy/4-MPy	0.85	0.86	0.72%
☑	1-MPy/4-MPy	0.72	0.67	6.99%
☑	BNT/T-M-Phe	2.12	2.83	28.52
☑	27dbR/27db	0.61	0.61	0.38%
☑	27bb/29bbR	0.00	0.00	0.00%
☑	27Ts/30ab	0.18	0.17	6.22%
☑	SC26TA/RC	0.57	0.73	24.12
☑	27Tm/30ab	0.19	0.20	5.10%
☑	28ab/30ab	0.13	0.14	10.16
☑	SC28TA/RC	0.63	0.66	4.92%
☑	29ab/30ab	0.48	0.48	0.86%
☑	RC27TA/RC	0.56	0.55	1.44%
☑	30O/30ab	0.08	0.03	93.61
☑	RC28TA/RC	0.64	0.66	4.28%
☑	31abS/30ab	0.33	0.30	7.44%
☑	30G/30ab	0.09	0.10	14.22

	Informative			
☑	SES 1/SES 3	0.30	0.25	19.82
☑	SES 2/SES 3	0.21	0.18	17.80
☑	SES 4/SES 3	0.25	0.23	11.82
☑	SES 8/SES 3	0.76	0.62	20.46
☑	MA/1-MP	0.49	0.52	4.27%
☑	C2-dbt/C2-phe	0.17	0.22	27.66
☑	C3-dbt/C3-phe	0.19	0.24	20.66
☑	Retene/C4-phe	0.82	0.95	15.52
☑	C23Tr/C2-phe	0.09	0.09	0.90%
☑	C20TA/C21T	1.34	1.46	9.07%
☑	C21TA/RC2	1.05	1.27	18.73
☑	C21TA/RC2	0.67	0.84	22.97
☑	C28 (22R)/27Ts	0.25	0.28	9.94%
☑	C28 (22S)/30ab	0.04	0.04	3.19%
☑	27Ts/27Tm	0.98	0.88	11.31
☑	SC26TA/SC	0.91	1.10	19.25
☑	29aaS /29aaR	1.00	1.16	14.34
☑	29bbR/29aa	1.88	1.89	0.39%
☑	25nor30ab/3	0.12	0.15	26.62
☑	29Ts/30ab	0.18	0.18	0.94%
☑	RC27TA/RC	0.88	0.83	5.72%
☑	30d/30ab	0.11	0.12	7.73%
☑	30ba/30ab	0.13	0.13	2.84%

1.6 Conclusion of the comparison between IM20 and IM22

There are too many differences to conclude at match between IM20 and IM22. However, the samples might originate from the same batch but that something was added, or still present in the tank when the oil was sold from the refinery to the bunker company.

NO Match

2. Comparison IM21 ULSFO Stena Sweden with IM23 ULSFO BunkerOne Sweden.

2.1 Overlay of the chromatograms.

Figure 24 shows the overlay of IM21 (blue) and IM 23 (red) chromatograms. Both samples look very similar.

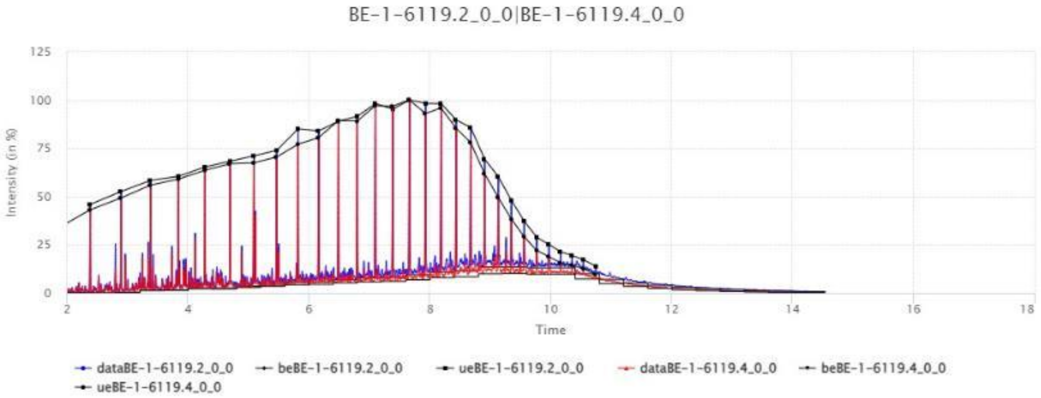


Figure 24 : Overlay of chromatograms IM21 (blue) and IM23 (red).

2.2 GC-PW plot

Figure 25 shows the PW plot (weathering curve) of samples IM21 and IM23. The higher alkanes show a difference. This can be caused by smaller peaks (that have a bigger fault) or by other compounds present.

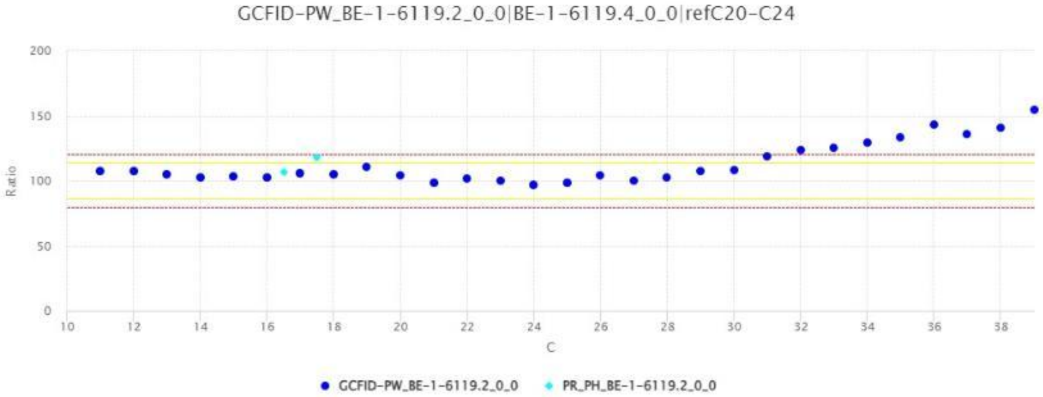


Figure 25 : PW Plot of samples IM21 and IM23.

2.3 Calculation of the isoprenoid ratios

Table 14 shows calculation of the isoprenoid ratios of sample IM21 vs. IM23: the allowed difference of 14% is not exceeded.

Table 14 : calculation of the isoprenoid ratios of sample IM21 with vs. IM23.

Param	Val 1	Val 2	D%
C17/Pris	1.72	1.75	1.41
Pris/Phyt	1.85	2.05	10.07
C1Phyt	3.29	3.72	12.13

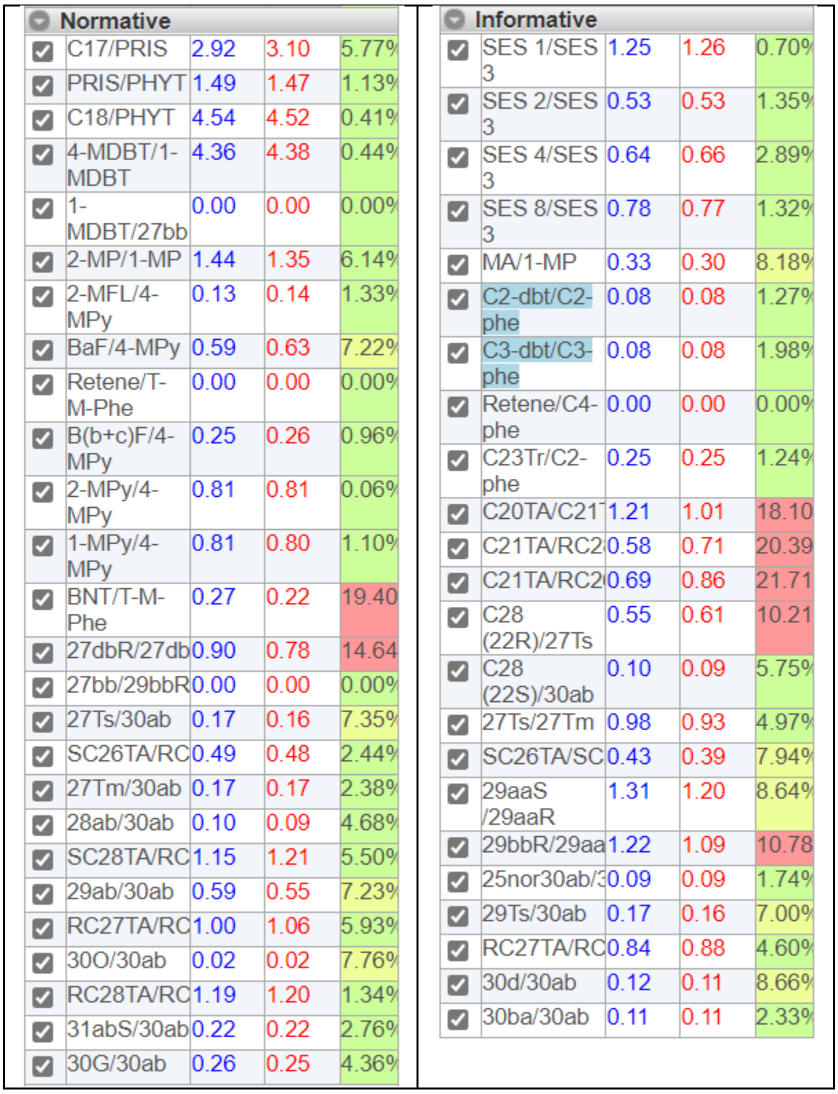
2.4 Conclusion of the total ion chromatograms

According to the first screening, there could be a match between sample IM21 and IM23.

2.5 Ion ratios of the normative and informative ratios.

Table 15 shows the diagnostic ratios of samples IM21 (blue) and IM23 (red) comparison. Some of the ratios have a relative difference of more than 14% (2 normative ratios and 3 informative ratios).

Table 15 : Diagnostic ratios of the comparison of samples IM21 (blue) and IM23 (red).

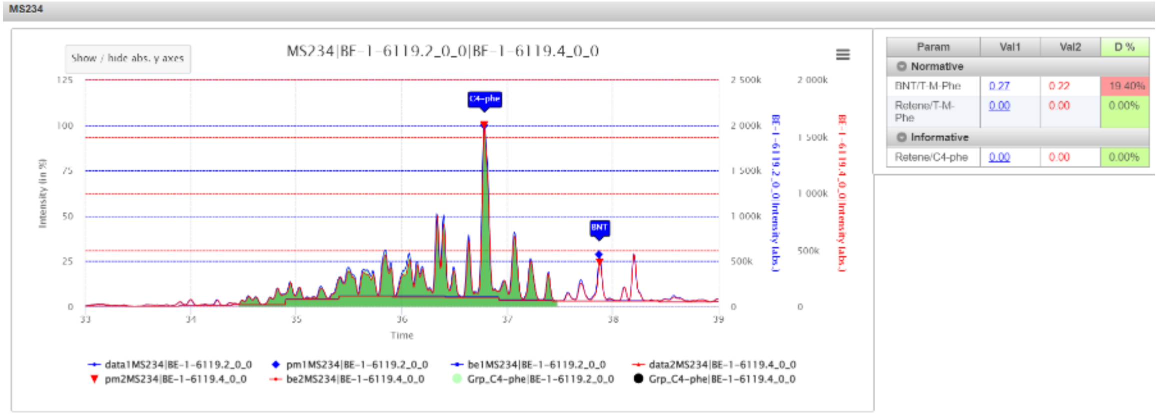


	Normative			
☑	C17/PRIS	2.92	3.10	5.77%
☑	PRIS/PHYT	1.49	1.47	1.13%
☑	C18/PHYT	4.54	4.52	0.41%
☑	4-MDBT/1-MDBT	4.36	4.38	0.44%
☑	1-MDBT/27bb	0.00	0.00	0.00%
☑	2-MP/1-MP	1.44	1.35	6.14%
☑	2-MFL/4-MPy	0.13	0.14	1.33%
☑	BaF/4-MPy	0.59	0.63	7.22%
☑	Retene/T-M-Phe	0.00	0.00	0.00%
☑	B(b+c)F/4-MPy	0.25	0.26	0.96%
☑	2-MPy/4-MPy	0.81	0.81	0.06%
☑	1-MPy/4-MPy	0.81	0.80	1.10%
☑	BNT/T-M-Phe	0.27	0.22	19.40
☑	27dbR/27db	0.90	0.78	14.64
☑	27bb/29bbR	0.00	0.00	0.00%
☑	27Ts/30ab	0.17	0.16	7.35%
☑	SC26TA/RC	0.49	0.48	2.44%
☑	27Tm/30ab	0.17	0.17	2.38%
☑	28ab/30ab	0.10	0.09	4.68%
☑	SC28TA/RC	1.15	1.21	5.50%
☑	29ab/30ab	0.59	0.55	7.23%
☑	RC27TA/RC	1.00	1.06	5.93%
☑	30O/30ab	0.02	0.02	7.76%
☑	RC28TA/RC	1.19	1.20	1.34%
☑	31abS/30ab	0.22	0.22	2.76%
☑	30G/30ab	0.26	0.25	4.36%

	Informative			
☑	SES 1/SES 3	1.25	1.26	0.70%
☑	SES 2/SES 3	0.53	0.53	1.35%
☑	SES 4/SES 3	0.64	0.66	2.89%
☑	SES 8/SES 3	0.78	0.77	1.32%
☑	MA/1-MP	0.33	0.30	8.18%
☑	C2-dbt/C2-phe	0.08	0.08	1.27%
☑	C3-dbt/C3-phe	0.08	0.08	1.98%
☑	Retene/C4-phe	0.00	0.00	0.00%
☑	C23Tr/C2-phe	0.25	0.25	1.24%
☑	C20TA/C21	1.21	1.01	18.10
☑	C21TA/RC2	0.58	0.71	20.39
☑	C21TA/RC2	0.69	0.86	21.71
☑	C28 (22R)/27Ts	0.55	0.61	10.21
☑	C28 (22S)/30ab	0.10	0.09	5.75%
☑	27Ts/27Tm	0.98	0.93	4.97%
☑	SC26TA/SC	0.43	0.39	7.94%
☑	29aaS /29aaR	1.31	1.20	8.64%
☑	29bbR/29aa	1.22	1.09	10.78
☑	25nor30ab/3	0.09	0.09	1.74%
☑	29Ts/30ab	0.17	0.16	7.00%
☑	RC27TA/RC	0.84	0.88	4.60%
☑	30d/30ab	0.12	0.11	8.66%
☑	30ba/30ab	0.11	0.11	2.33%

2.6 Differences explained.

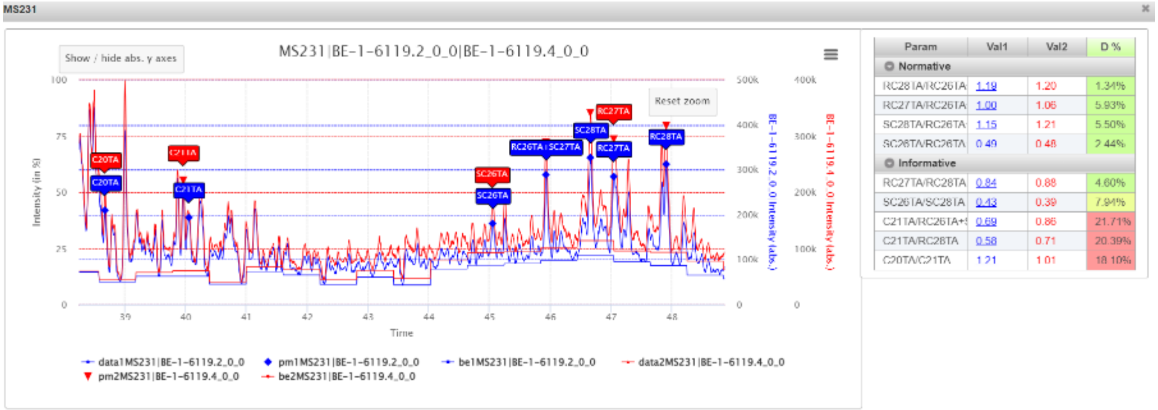
Figure 26 shows the ion chromatogram of Retene, Tetra-Methylphenanthrene, Benzo(b)naphtho(1,2-d)- thiophene (BNT) and the C4-Phenanthrenes (m/z 234). The relative difference of BNT/T-M-Phen is >14%. The pattern is the same. The peak of BNT is small, this can explain the relative big difference in ratio.



Param	Val1	Val2	D %
Normative			
BNT/T-M-Phe	0.27	0.22	19.40%
Retene/T-M-Phe	0.00	0.00	0.00%
Informative			
Retene/C4-phe	0.00	0.00	0.00%

Figure 26 : Ion chromatogram of Retene, Tetra-Methylphenanthrene, Benzo(b)naphtho(1,2-d)- thiophene (BNT) and the C4-Phenanthrenes (m/z 234).

Figure 27 shows the ion chromatograms of the triaromatic steranes. These peaks are also very small, which can explain the bigger difference.



Param	Val1	Val2	D %
Normative			
RC28TA/RC26TA	1.19	1.20	1.34%
RC27TA/RC26TA	1.00	1.06	5.93%
SC28TA/RC26TA	1.15	1.21	5.50%
SC26TA/RC26TA	0.49	0.48	2.44%
Informative			
RC27TA/RC28TA	0.84	0.88	4.60%
SC26TA/SC28TA	0.43	0.39	7.94%
C21TA/RC26TA+	0.69	0.86	21.71%
C21TA/RC28TA	0.58	0.71	20.39%
C20TA/C21TA	1.21	1.01	18.10%

Figure 27 : Ionchromatogram of triaromatic steranes (m/z 231).

2.7 PW plot of all ratios

Figure 28 shows the PW-plot of all ratios: not all ratios are between 80 and 120%, probably something else is mixed into the original batch.

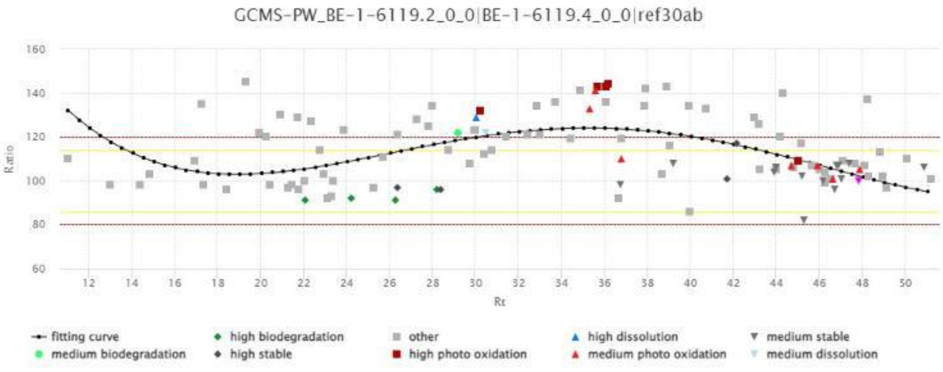


Figure 28 : PW plot of all ratios of IM21 vs. IM23.

2.8 Conclusion of the comparison of samples IM21 and IM23.

Although the diagnostic ratios show only 5 ratios that do not meet the <14% difference, the PW-plot shows a lack of correlation. Therefore a non match is concluded. However, both samples probably originate from the same batch.

3. Comparison IM32 ULSFO FincoEnergies, Nederlands with IM34 ULSFO FincoEnergies, Nederlands.

3.1 Overlay of the chromatograms.

Figure 29 shows the overlay of the chromatograms of IM32 and IM34. These look similar.

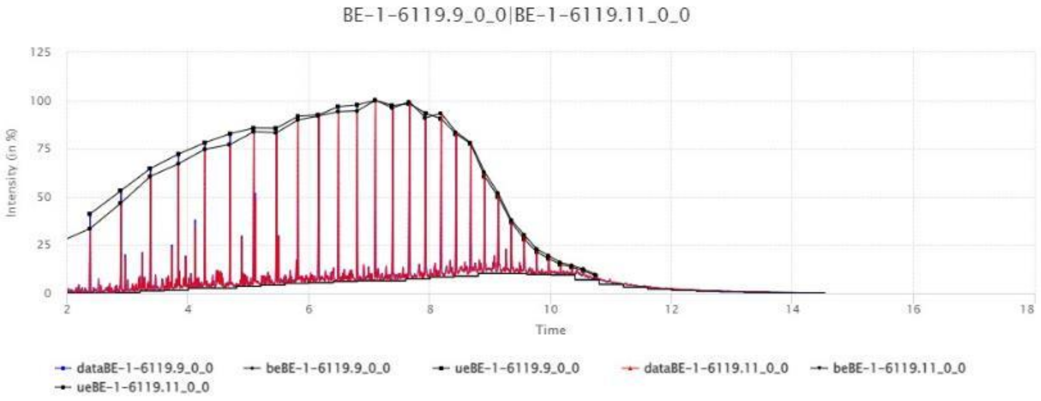


Figure 29 : Overlay of the chromatograms of IM32 (blue) and IM34 (red).

3.2 GC-PW plot

Figure 30 shows the PW plot (weathering curve) of samples IM32 and IM34. All ratios are between 80 and 120%

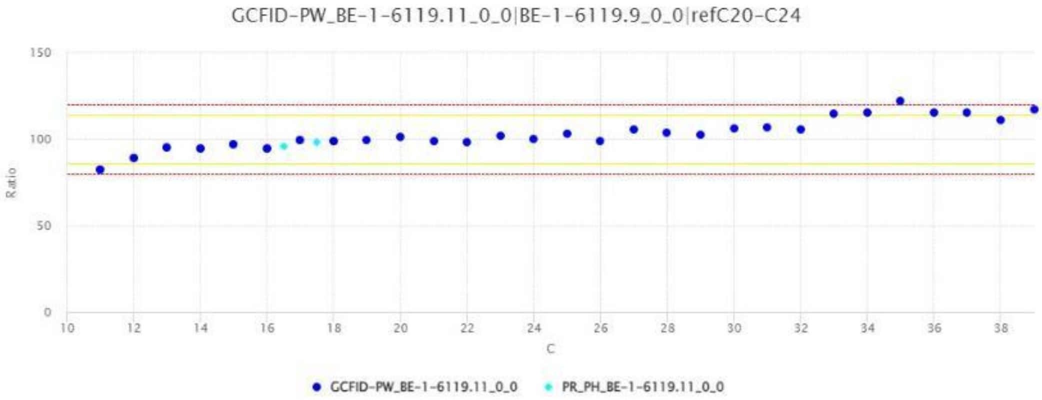


Figure 30 : PW-plot of IM32 compared to IM34.

3.3 Calculation of the isoprenoid ratios

Table 16 shows calculation of the isoprenoid ratios of sample IM32 and IM34: the allowable difference of 14% is not exceeded.

Table 16: Isoprenoid ratios of sample IM32 and IM34.

Param	Val 1	Val 2	D%
C17/Pris	**1.7**	**1.76**	**3.51**
Pris/Phyt	**1.87**	**1.82**	**2.61**
C1Phyt	**3.16**	**3.17**	**0.43**

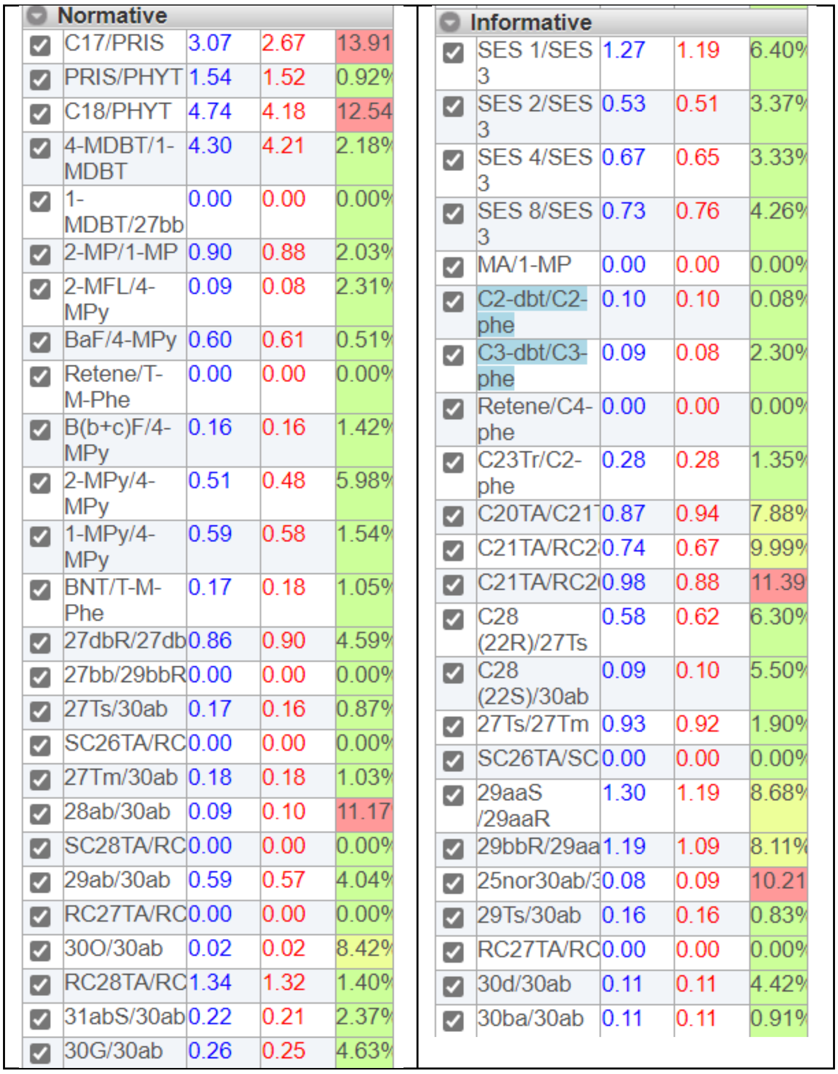
3.4 Conclusion of the total ion chromatograms

According to the first screening, there could be a match between sample IM32 and IM34.

3.5 Ion ratios of the normative and informative ratios.

Table 17 shows the diagnostic ratios of the comparison of samples IM32 (blue) and IM34 (red). All diagnostic ratios show a difference of <14%.

Table 17 : Normative and informative ratios of IM32 vs. IM34.



	Normative			
☑	C17/PRIS	3.07	2.67	13.91
☑	PRIS/PHYT	1.54	1.52	0.92%
☑	C18/PHYT	4.74	4.18	12.54
☑	4-MDBT/1-MDBT	4.30	4.21	2.18%
☑	1-MDBT/27bb	0.00	0.00	0.00%
☑	2-MP/1-MP	0.90	0.88	2.03%
☑	2-MFL/4-MPy	0.09	0.08	2.31%
☑	BaF/4-MPy	0.60	0.61	0.51%
☑	Retene/T-M-Phe	0.00	0.00	0.00%
☑	B(b+c)F/4-MPy	0.16	0.16	1.42%
☑	2-MPy/4-MPy	0.51	0.48	5.98%
☑	1-MPy/4-MPy	0.59	0.58	1.54%
☑	BNT/T-M-Phe	0.17	0.18	1.05%
☑	27dbR/27db	0.86	0.90	4.59%
☑	27bb/29bbR	0.00	0.00	0.00%
☑	27Ts/30ab	0.17	0.16	0.87%
☑	SC26TA/RC	0.00	0.00	0.00%
☑	27Tm/30ab	0.18	0.18	1.03%
☑	28ab/30ab	0.09	0.10	11.17
☑	SC28TA/RC	0.00	0.00	0.00%
☑	29ab/30ab	0.59	0.57	4.04%
☑	RC27TA/RC	0.00	0.00	0.00%
☑	30O/30ab	0.02	0.02	8.42%
☑	RC28TA/RC	1.34	1.32	1.40%
☑	31abS/30ab	0.22	0.21	2.37%
☑	30G/30ab	0.26	0.25	4.63%

	Informative			
☑	SES 1/SES 3	1.27	1.19	6.40%
☑	SES 2/SES 3	0.53	0.51	3.37%
☑	SES 4/SES 3	0.67	0.65	3.33%
☑	SES 8/SES 3	0.73	0.76	4.26%
☑	MA/1-MP	0.00	0.00	0.00%
☑	C2-dbt/C2-phe	0.10	0.10	0.08%
☑	C3-dbt/C3-phe	0.09	0.08	2.30%
☑	Retene/C4-phe	0.00	0.00	0.00%
☑	C23Tr/C2-phe	0.28	0.28	1.35%
☑	C20TA/C21	0.87	0.94	7.88%
☑	C21TA/RC2	0.74	0.67	9.99%
☑	C21TA/RC2	0.98	0.88	11.39
☑	C28 (22R)/27Ts	0.58	0.62	6.30%
☑	C28 (22S)/30ab	0.09	0.10	5.50%
☑	27Ts/27Tm	0.93	0.92	1.90%
☑	SC26TA/SC	0.00	0.00	0.00%
☑	29aaS /29aaR	1.30	1.19	8.68%
☑	29bbR/29aa	1.19	1.09	8.11%
☑	25nor30ab/3	0.08	0.09	10.21
☑	29Ts/30ab	0.16	0.16	0.83%
☑	RC27TA/RC	0.00	0.00	0.00%
☑	30d/30ab	0.11	0.11	4.42%
☑	30ba/30ab	0.11	0.11	0.91%

3.6 PW plot of all ratios

Figure 31 shows the PW plot of all the ratios between samples IM32 and IM34: almost all ratios are between 80 and 120%.

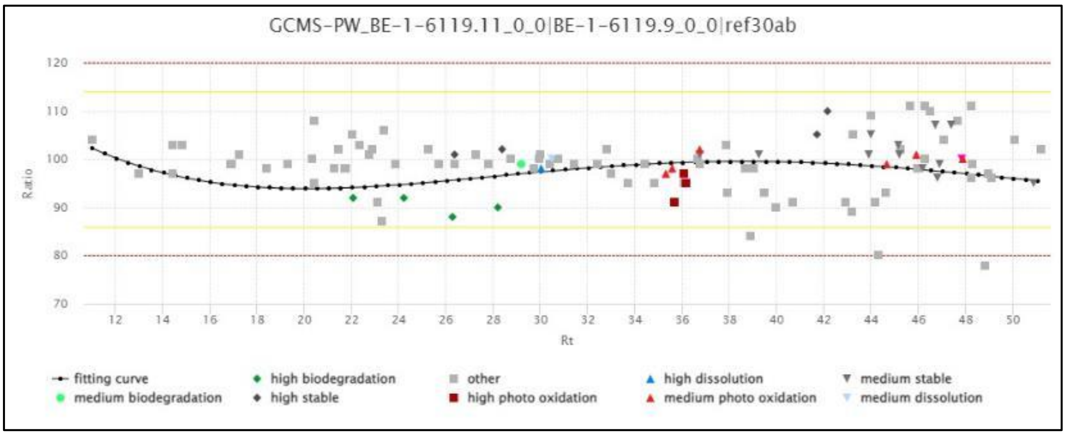


Figure 31 : PW plot of all ratios between IM32 and IM34.

3.7 Conclusion of the comparison between samples IM32 and IM34.

With all calculations made and after all ion chromatogram comparisons, a MATCH can be concluded between IM32 and IM34.

Appendix 4 : Strange behaviour of sample IM29

Because of the strange behaviour of sample IM29, the question was to have a closer look. In Figure 32, the total ion chromatogram shows at retention time 39.17 min a peak that could be a FAME. But when scanning on m/z 74 (mass of the FAMEs), no peak is visible on retention time 39.17 min. When looking in the library a certainty of 87% is given for the compound "Phenol,3-(8Z)-8-pentadecen-1-yl" (Figure 33) commonly known as Cardanol, a phenolic compound derived from cashew nutshell liquid. It is used in various industrial applications, including as a precursor for antioxidant additives in lubricating oils.

Cardanol and its derivatives are promoted as a green alternative to traditional petroleum-based additives due to: low toxicity, renewable sourcing, lower environmental impact in disposal and biodegradation.

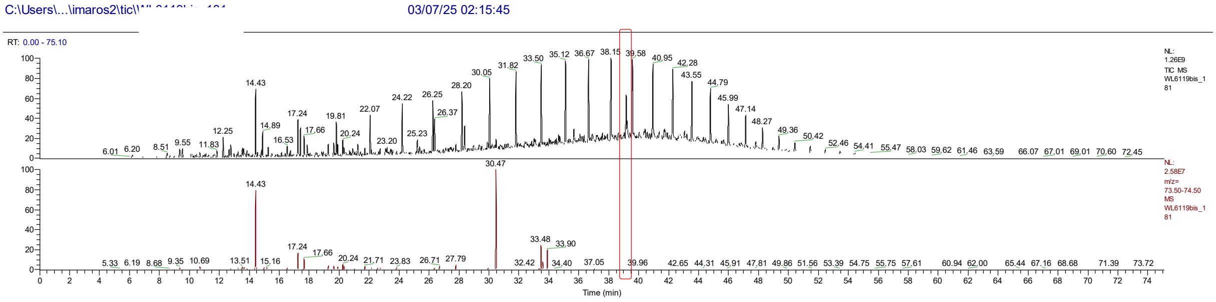
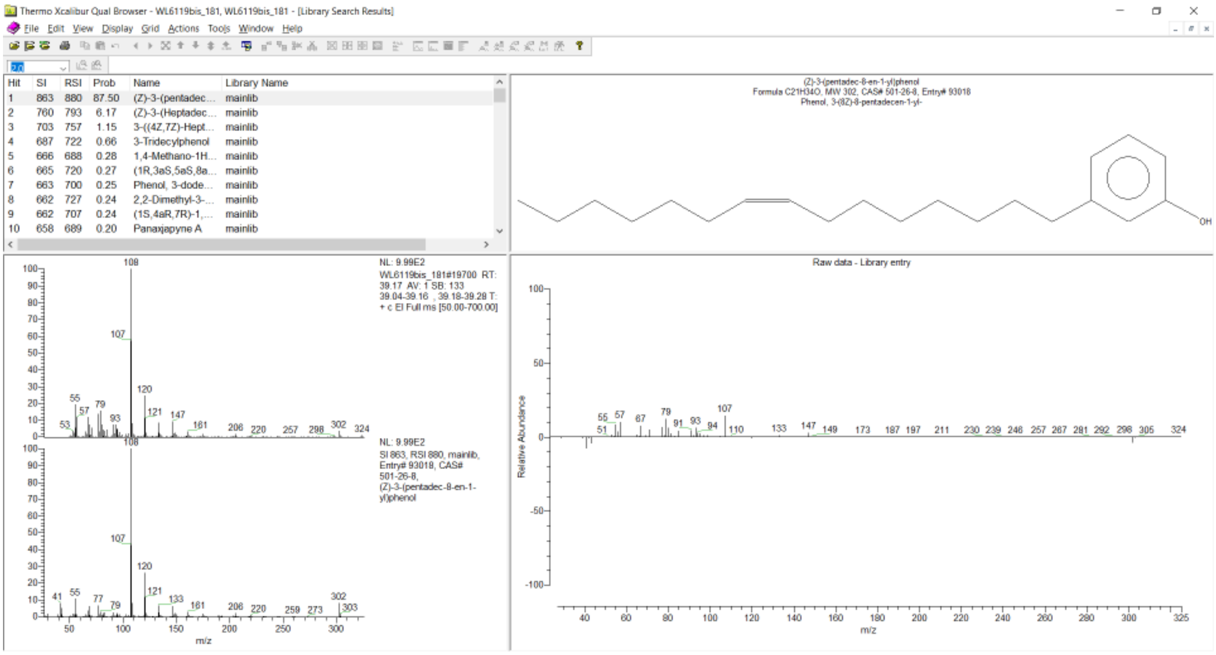


Figure 32 : Total ion chromatogram of sample IM29.



Hit	SI	RSI	Prob	Name	Library Name
1	863	880	87.50	(Z)-3-(pentadec...	mainlib
2	760	793	6.17	(Z)-3-(Heptadec...	mainlib
3	703	757	1.15	3-((4Z,7Z)-Hept...	mainlib
4	687	722	0.66	3-Tridecylphenol	mainlib
5	666	688	0.28	1,4-Methano-1H...	mainlib
6	665	720	0.27	(1R,3aS,5aS,8a...	mainlib
7	663	700	0.25	Phenol, 3-dode...	mainlib
8	662	727	0.24	2,2-Dimethyl-3-...	mainlib
9	662	707	0.24	(1S,4aR,7R)-1,...	mainlib
10	658	689	0.20	Panaxjapyne A	mainlib

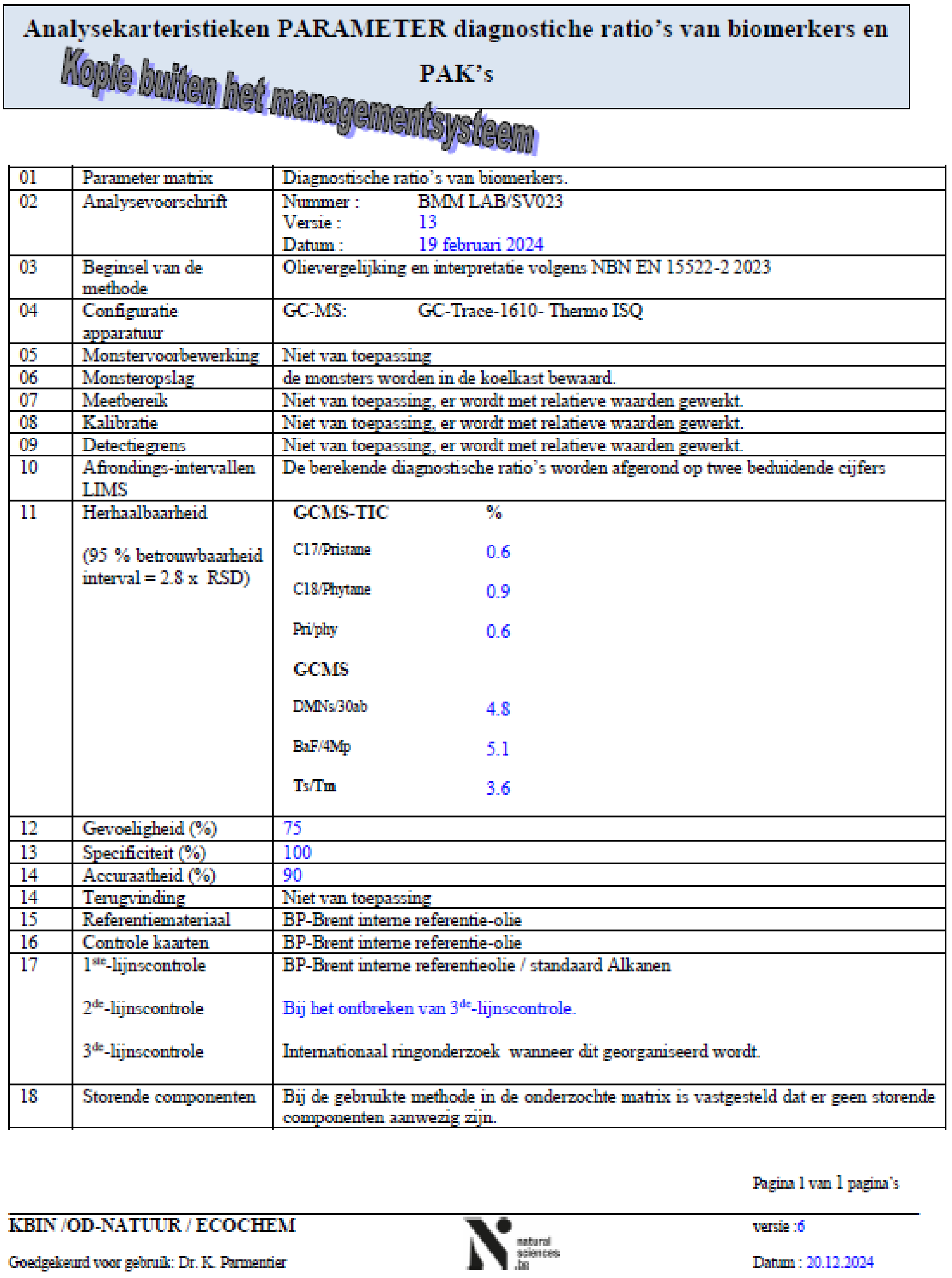
(Z)-3-(pentadec-8-en-1-yl)phenol
Formula C21H34O, MW 302, CAS# 501-26-8, Entry# 93018
Phenol, 3-(8Z)-8-pentadecen-1-yl-

NL: 9.99E2
WL6119bis_181#19700 RT: 39.17 AV: 1 SB: 133 39.04-39.16 , 39.18-39.28 T: + c EI Full ms [50.00-700.00]

NL: 9.99E2
SI 863, RSI 880, mainlib, Entry# 93018, CAS# 501-26-8, (Z)-3-(pentadec-8-en-1-yl)phenol

Figure 33 : Result of the NIST library.

Appendix 5: AK



Analysekarteristieken PARAMETER diagnostiche ratio's van biomerkers en PAK's

01	Parameter matrix	Diagnostische ratio's van biomerkers.
02	Analysevoorschrift	Nummer : BMM LAB/SV023 Versie : 13 Datum : 19 februari 2024
03	Beginsel van de methode	Olievergelijking en interpretatie volgens NBN EN 15522-2 2023
04	Configuratie apparatuur	GC-MS: GC-Trace-1610- Thermo ISQ
05	Monstervoorbewerking	Niet van toepassing
06	Monsteropslag	de monsters worden in de koelkast bewaard.
07	Meetbereik	Niet van toepassing, er wordt met relatieve waarden gewerkt.
08	Kalibratie	Niet van toepassing, er wordt met relatieve waarden gewerkt.
09	Detectiegrens	Niet van toepassing, er wordt met relatieve waarden gewerkt.
10	Afrondings-intervallen LIMS	De berekende diagnostische ratio's worden afgerond op twee beduidende cijfers
11	Herhaalbaarheid (95 % betrouwbaarheid interval = 2.8 x RSD)	**GCMS-TIC** % C17/Pristane 0.6 C18/Phytane 0.9 Pri/phy 0.6 **GCMS** DMNs/30ab 4.8 BaF/4Mp 5.1 Ts/Tm 3.6
12	Gevoeligheid (%)	75
13	Specificiteit (%)	100
14	Accuraatheid (%)	90
14	Terugvinding	Niet van toepassing
15	Referentiemateriaal	BP-Brent interne referentie-olie
16	Controle kaarten	BP-Brent interne referentie-olie
17	1ste-lijnscontrole 2de-lijnscontrole 3de-lijnscontrole	BP-Brent interne referentieolie / standaard Alkanen Bij het ontbreken van 3de-lijnscontrole. Internationaal ringonderzoek wanneer dit georganiseerd wordt.
18	Storende componenten	Bij de gebruikte methode in de onderzochte matrix is vastgesteld dat er geen storende componenten aanwezig zijn.

Pagina 1 van 1 pagina's

KBIN /OD-NATUUR / ECOCHEM

Goedgekeurd voor gebruik: Dr. K. Parmentier

versie :6

Datum : 20.12.2024

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Appendix 10

Memo from SINTEF

Characterisation of the 3 large samples

Co-funded by the European Union

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Oil analyses IMAROS-2

SAKSBEHANDLER / FORFATTER
Liv-Guri Faksness

	Behandling	Uttalelse	Orientering	Etter avtale

GÅR TIL
Silje Berge, Kystverket
Ingvild Frogner, Kystverket

PROSJEKTNUMMER / REFERANSE	**DATO**	**GRADERING**
302008620	2024-11-05	Fortrolig

SINTEF received three oil samples on September 12, 2024 (Figure 1), 400-500 mL of each oil. The oils were registered in the SINTEF LIMS system and are described in Table 1.

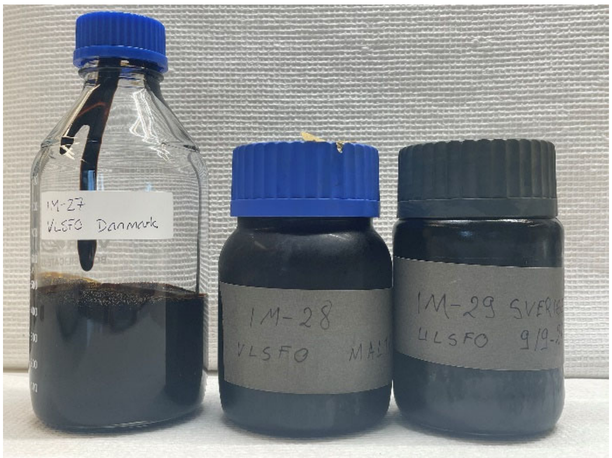

Figure 1 The oils: IM-27 (transferred to a new bottle), IM-28, and IM-29.

Table 1 *Description of the oils.*

SINTEF ID	IMAROS ID	Labeling	Comment
2024-5377	IM-27	VLSFO Danmark	Bottle leaked during transport to SINTEF
2024-5378	IM-28	VLSFO Malta	
2024-5379	IM-29	ULSFO Sverige	

GC chromatograms of the oils are shown in Figure 2 to Figure 4.

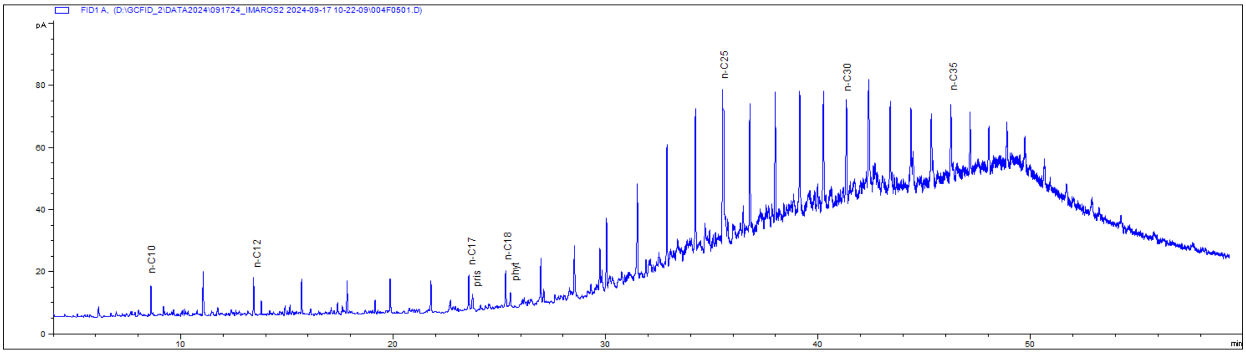

Figure 2 *GC chromatogram of IM-27 (SINTEF ID 2024-5377)*

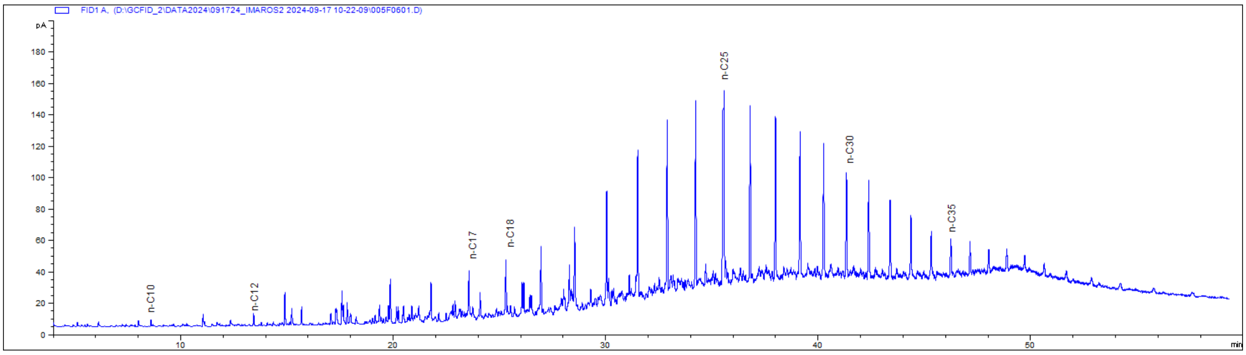

Figure 3 *GC chromatogram of IM-28 (SINTEF ID 2024-5378)*

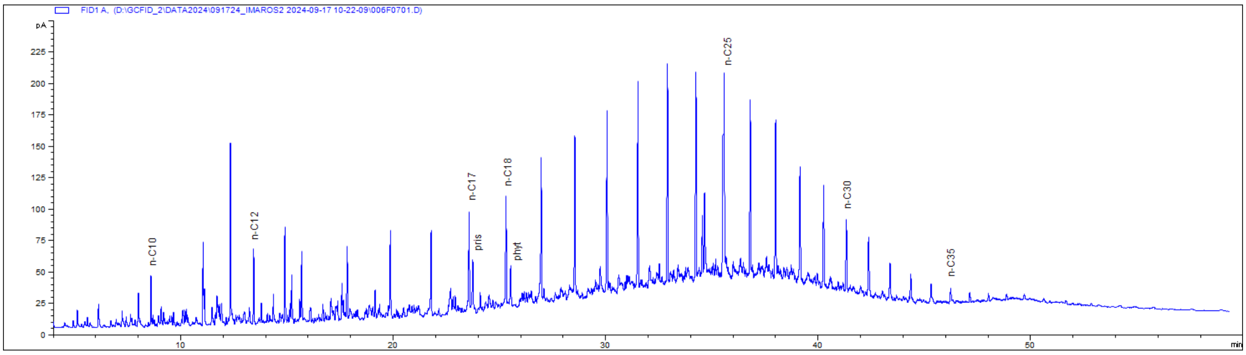

Figure 4 *GC chromatogram of IM-29 (SINTEF ID 2024-5379)*

The oils physical properties are reported in Table 2:

- Water content measured by Karl Fisher.
- Density was measured at 50 °C and re-calculated to 15 °C (ASTM D1250-80).
- Viscosity measured using temperature sweep from 52 °C down to -2 °C (1 °C/min) at shear rate $10s^{-1}$ (Figure 5). Viscosities at 10 and 50 °C from the temperature sweep are given in the table.
- Pour point was measured by an external laboratory following ASTM D97. The method has an uncertainty of ±3 °C.

Table 2 The oils physical properties. Viscosity measurements are from the temperature sweep.

SINTEF ID	IM no.	Viscosity, temp sweep (cP) 10 °C	Viscosity, temp sweep (cP) 50 °C	Density (g/mL) 50 °C (measured)	Density (g/mL) 15 °C (calculated)	Water content (%)	Pour point (°C)
2024-5377	IM-27	23104	282	0.931	0.954	0.1	21
2024-5378	IM-28	36277	110	0.909	0.932	0.1	30
2024-5379	IM-29	932	9.6	0.866	0.890	0.2	21

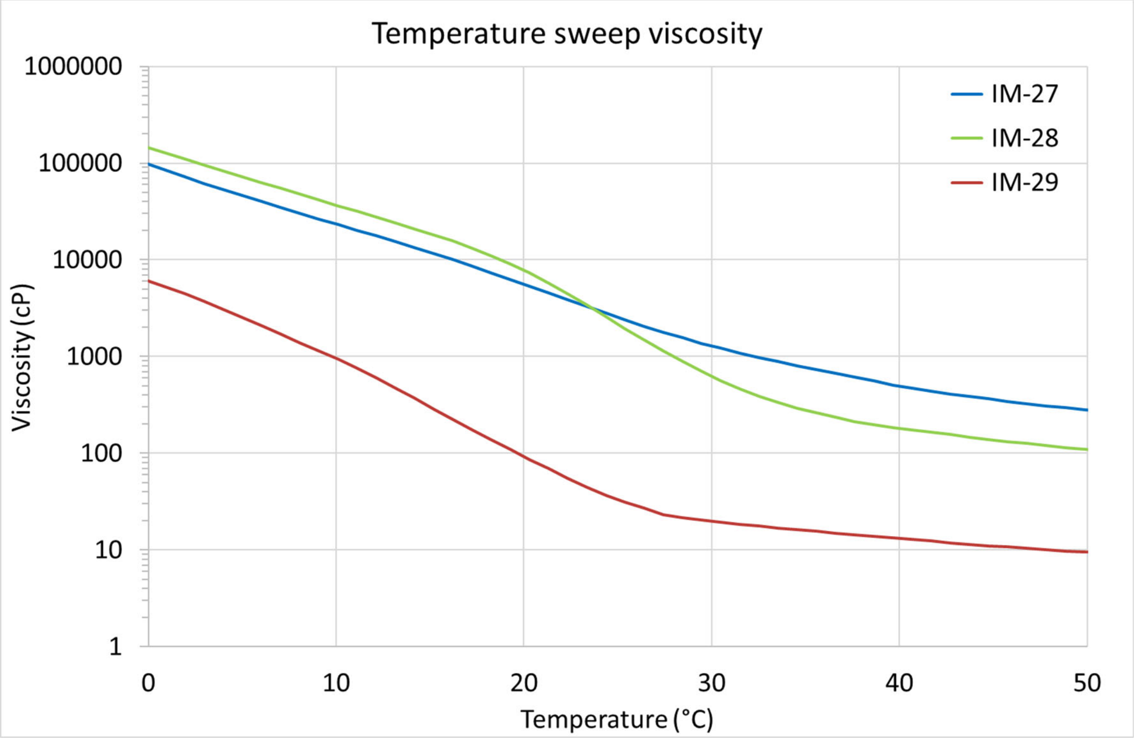

Figure 5 Temperature sweep for viscosity (cP) from 50 to 0 °C (1 °C/min) at shear rate $10s^{-1}$.

Additional measurements for pour point

The initial measurements of pour point were somewhat different than the fuels Certificate of Analysis (CoA). After a meeting with Intertek, it was decided that both minimum and maximum pour point should be measured (ASTM D-5853). Results are given in Table 3.

The analytical method is based on ASTM D-5853 Standard Test Method for Pour Point of Crude Oils. The maximum and minimum pour point temperatures provide a temperature window where a crude oil, depending on its thermal history, might appear in the liquid as well as the solid state. The test method can be used to supplement other measurements of cold flow behaviour. It is especially useful for the screening of the effect of wax interaction modifiers on the flow behaviour of crude oils.

Comments to the measurements from Intertek.

- Initial pour point measurements (ASTM D-97 Standard Test Method for Pour Point of Petroleum Products): The oil sample is heated to 60 °C for 2 hours, then stored over night at room temperature. Next day, the oil is heated in water bath to 48 °C, and cooled down to 30 °C. From 30 °C, the oil is observed every 3rd degree (tilted for 5 sec) until pour point is reached. Pour point is reported 3 °C higher than observed flowability (notes from meeting with Intertek on Oct 17, 2024).
- Maximum (upper) pour point: The oil is heated to 60 °C and cooled down to room temperature for 24 hours prior to analysis.
- Minimum (lower) pour point: The oil (50 mL) is transferred to a glass beaker that is placed in a pressure cell and flushed with nitrogen for 5 minutes. Then the pressure is set to 1 bar. The sample is heated to 80 °C. After 45 min at 80 °C, the sample is cooled down to 50 °C ± 2 °C within 1 hour and 40 min. When 50 °C is reached, the sample is stirred carefully before it is transferred to a pre-heated pour point beaker prior to measurements of minimum pour point (observed every 3rd degree until pour point is reached).
- After measurement of minimum pour point, the maximum pour point was measured in the same sample the next day.

Table 3 *Additional measurements of pour points: Minimum (min.) and maximum (max) pour point (ASTM D-5853). Max pour point (next day) is the same sample as min pour point.*

SINTEF ID	IM no.	Pour point (init. Measurement, ASTM D-97) (°C)	Min. Pour point (°C)	Max. Pour point (°C)	Max. Pour point (next day) (°C)	Pour point from CoA (°C)
2024-5377	IM-27	21	9	24	21	12
2024-5378	IM-28	30	21	30	27	27
2024-5379	IM-29	21	15	21	24	27

Visual observations in the SINTEF lab at different temperatures indicated that the IM-29 had flowability at lower temperature than IM-27, and that both oils had the ability to flow at room temperature (approx. 20 °C).