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Project memo

PAME – EPPR Methodology for screening properties of New Low Sulphur Marine Fuel

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Summary

This memo is revision of methodology followed in an inter-laboratory study by laboratories within the Arctic member states in the ongoing PAME/EPPR project "Low Sulphur fuels, fate and behavior in cold water conditions".

The memo describes the recommended screening test methodology in characterizing properties of "Very Low Sulphur Marine fuels" (VLSFO) and "Ultra Low Marine Fuels" (ULSFO). The purpose of a common methodology is to obtain comparable and consistent results between the different laboratories involved in analyses of oils. The recommended methodology is based on feed-back and suggestions from laboratories within the PAME /EPPR working group (WP-3) and the experience gained from the inter-laboratory comparison completed in 2022.

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1 Background

As a part of the WP3 - PAME/EPPR project "Low sulfur fuels, fate and behavior in cold water conditions", an interlaboratory comparison was performed and is reported in Faksness and Daling (2023). Three low sulfur fuel oils were characterized through this interlaboratory comparison study by laboratories from USA, Canada, China, Sweden, and Norway (Table 1.1). The oils analyzed were a WRG (<0.05% S), a MGO (<0.1% S) and a VLSFO (<0.5% S). A common guideline for the methodology was accepted by all laboratories that included characterization of oil properties relevant for fate, behavior, fingerprinting, and relative toxicity when spilled in cold or Arctic seawater (Daling et al., 2021).

The methodology applied in the interlaboratory comparison study was evaluated by the participants, and it was agreed that the methodology must be revised. The present memo is a revision of the guidelines described in Daling et al. (2021).

Table 1.1 Overview of the participating countries and the accompanying laboratories /institutions on the interlaboratory comparison study performed in 2021-2022.

Country	Laboratory	Contact person	Comments
Sweden	Chalmers University	Ida-Maja Hassellöv	Subcontractors: Saybolt, ALS Toxicon AB, and NFC
Canada	ECCC	Michael Goldthorp	Subcontractors: Intertek and Valor labs
USA	EPA	Mace Barron	
China	Tongji University (TJU)	Sijie Lin	Subcontractors: Ningbo Customs District Technology Centers, State Key Laboratory of Pollution Control and Resource Reuse, Shanghai Key Laboratory of Chemical Assessment and Sustainability
Norway	SINTEF	Faksness/Sørheim	Subcontractors: Intertek, Biotrix

2 Objectives and deliverables

The main objective has been to establish common methodology for characterizing properties that are relevant for screening the diversity of different marine LSFOs, both connected to fate and behavior, and the relative toxicity when spilled in cold or Arctic seawaters. The test methodology will be used by selected laboratories within the member countries of the LSFO-project.

The screening protocols include characterizing the physico-chemical properties of the parent ("fresh") fuel oil samples and their relative toxicity.

3 Pre-handling of the samples when arriving the laboratory

After the oil samples have entered the laboratory in containers (volume minimum 2-4 L) the following handling is important:

- Registration of the oil samples according to the laboratory's internal QA routines or protocols, including giving the oil unique internal sample number (to maintain "chain of custody")
- Visual description or photo documentation, and registration of enclosed paper etc.

Prior to transferring oil aliquots to smaller bottles, it is important to check the "homogeneity" e.g. presence of free water, w/o-emulsion, inorganic material on the bottom of the oil in the container. According to the ISO 8217 Fuel standard, the max. sediment content should be < 0.1 wt.% and water content < 0.5wt.%. These parameters are generally documented in *"Certificate of Analysis"* (CoA). For further details, see Daling et al. (2021).

4 Physico-chemical properties of the oil

Table 4.1 and Table 4.2 give an overview of the recommended test parameters. The ASTM methods are referred to as numbers in the table, but their entire titles are provided in the reference list (chapter 7).

Table 4.1 Analytical methods to determine the physical properties.

Physical property	**Analytical method**	**Comments**
Water content	ASTM E203	Volumetric Karl Fisher titration
Density	ASTM D4052 or ASTM 5002 At 15.5 °C (or 60 °F)	Digital density meter Oils with high pour point (viscosity, the density measurement if performed at higher temp. (e.g. 50 °C) and density is corrected to 15°C using volume correction factor tables (in ASTM D1250.
Pour point	ASTM D97 / IP 15 / ISO 3016 (In addition, Canada will apply ASTM D5949)	Standard Test Method for Pour Point of Petroleum Products
Flash point	ASTM D93 or ASTM D7094	Closed cup tester
Viscosity – temperature-sweep	Dynamic viscosity is measured over a temperature range from 50 down to 0 °C.	Shear rate 10 s^{-1} (Temperature rate: 1°C/min)
Dynamic viscosity	ASTM 7042 At 2 °C	Shear rates: 10, 100, and 1000 s^{-1}.
Interfacial tension	Pendant drop technique at air, water and saltwater interfaces at 0 and 15 °C.	ECCC in-house method
Distribution of hydrocarbons (nC_5-nC_{40})	US EPA method 8015). Gas chromatographic screening analysis (GC/FID).	30 m DB1 column. Suggested emp: program: 40°C (1 min) -6°/min.-330°C (10 min). 2.5 ml He /min.

Physical property	Analytical method	Comments
True Boiling point / Distillation curve	ASTM D 7169	Simulated Distillation Gas Chromatography (SimDist GC) System.

**Reflecting cold water /arctic seawater temperature.*

Table 4.2 Analytical methods used to determine the chemical properties.

Chemical property	Analytical method	Comments
"Hard" asphaltenes (SINTEF)* "Soft" asphaltenes (ECCC)	IP 143 ECCC-ESTS house method is using precipitation and gravimetric determination of "Soft" asphaltenes	• Precipitation in n-Heptane on "reflux for 1 hour • Asphaltenes are precipitated from the original oil by using excess n-pentane as the precipitating medium (30 mL n-pentane/g of oil).
Wax content	Bridiè et al, 1980, Modified. ECCC-ESTS house method is used for wax crystallization and gravimetric determination.	• Precipitation of the maltene fraction (de-asphalted) in a 1:1 2-MEK /DCM** solvent mixture at minus 10ºC *** for 4 hours. 6-fold wt. of solvent to wt. of oil. • Waxes are separated by crystallization in a polar solvent. After quantification of resin, the remaining diluted maltene is rotary-evaporated. Then, it is filtered after mixing with 50 mL of 1:1 (v:v) dichloromethane (DCM)/methyl ethyl ketone (MEK). The flask, funnel, filtering flask, and 1:1 DCM/MEK are placed in the -30 °C freezer to chill overnight before use to be able to crystallize the wax
Sulfur content (%w/w)	ASTM4294	
Elements/metals	ICPAES (Inductively Coupled Plasma Atomic Emission Spectrometry)	Iron Nickel Vanadium

**SINTEF will provide ECCC with the procedure for "Hard" asphaltenes.*

***MEK: Methyl-ethyl-ketone (2-Butanone); DCM: Dichloromethane*

**** SINTEF try to include the precipitation of wax at minus 30 °C, overnight to harmonize with ECCC`s procedure.*

Table 4.3 Additional parameters

Parameter	Analytical method	Comments
C, H, N (elemental analysis)	ASTM D5291	

5 Chemical characteristics and toxicity testing of Water Accommodated Fraction (WAF)

The standard method for preparation of water accommodated fraction is based on CROSERF Low Energy WAF (LE-WAF) (Aurand and Coelho, 2005). These guidelines were developed to standardize WAF preparation, laboratory exposures to aquatic organisms, and analytical chemistry measurements used to determine the acute toxicity of the water-soluble components in the oil. LE-WAF prepared in closed vessels with calm mixing (no vortex) of water below a surface layer of oil is regarded as a water solution of dissolved oil components.

To benefit from the chemical characterization, a common WAF must be prepared for both chemical characteristics and toxicity testing. Hence, make sure that a sufficient volume of the WAF is prepared to cover both activities. For e.g. just chemistry and toxic unit, a 2 L WAF should be appropriate, if e.g. toxicity testing with an algae and a copepod are included, a 10 L WAF system is recommended (Figure 5.1).

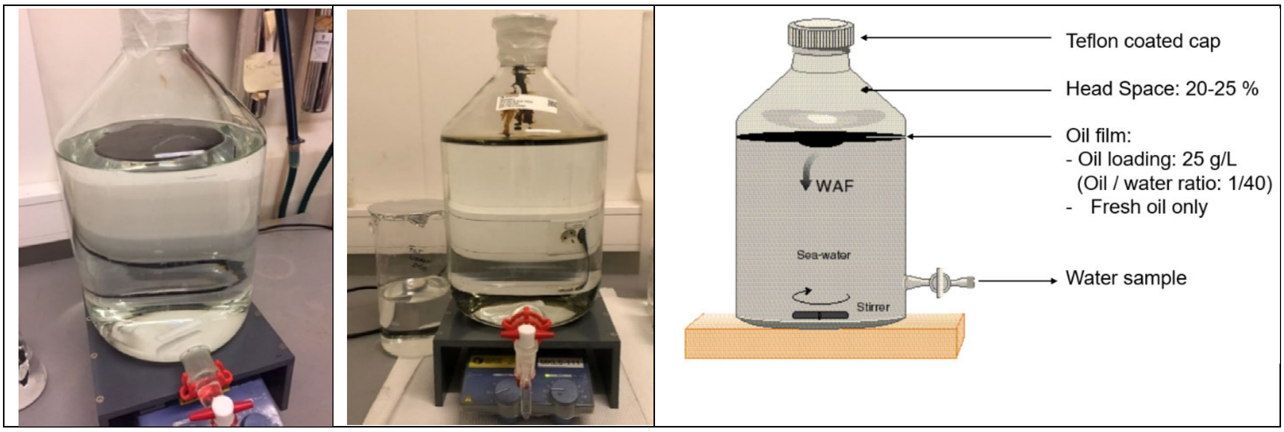

Figure 5.1 Set-up of the CROSERF systems used at SINTEF for preparation of Water Accommodated Fractions (WAF)

5.1 Preparation of water accommodated fraction (WAF)

- A volume (9.25 L) of sterile filtered (0.2 μm) natural seawater is added to 10 L bottles giving water to air headspace ratio of 4 to 1.
- A single WAF is prepared using one oil-to-water loading of 1:40 (i.e. 25 g oil/L seawater). The oil is carefully applied to the water surface.
    - Oil application:
        - Record the weight of a beaker and a glass Pasteur pipette before the oil is weighed into the beaker. Note the masses.
        - The oil is applied very gently to the WAF system by dripping/running it carefully along the Pasteur pipette from the beaker onto the water surface, avoid introducing oil droplets into the water.
        - Both the beaker and the pipette with the remaining oil are weighed after the oil is applied to the system and the exact amount of applied oil is calculated.
        - The WAF bottle must not be moved after the oil is added.
    - The bottle is closed immediately after oil application.
- The water is stirred gently with a magnetic stirrer (< 200 rpm) assuring that the oil film rest on the water surface without creating a vortex and without dispersing oil droplets into the water.

- The preparation should be carried out in darkness at low temperature (approximately 2 °C).
- Suggested mixing time is 72 hours (equilibrium time will depend on oil type).

- Before water sampling: Loosen the top before water sampling and always discharge the first 50-100 mL from the WAF bottle before the samples are collected.
- Samples for chemical analysis and toxicity testing are collected in glass vials and bottles with Teflon lined caps. Samples for chemical analysis are acidified (hydrochloric acid to pH<2) immediately after sampling to avoid biodegradation during storage (in refrigerator).
    - SVOC/TPH (same extract): 500-800 mL (one backup sample is recommended, just in case something goes wrong e.g. during extraction)
    - VOC: 40 mL gas tight vials with no headspace (2-3 vials are collected)
    - Toxicity: Volume dependent on the requirements of the test systems. For testing with *Acartia tonsa* a volume of 1.5 L should be sufficient. It covers dilutions with 4 replicates in 100 mL ErlenMeyer flasks with 5 mL headspace.
- The toxicity tests are initiated the same day as sampling.
- Water samples for chemistry analysis must be stored refrigerated and must be extracted within 2 weeks.
- Target analytes are given in Table 5.1.

5.2 Chemical composition of the WAFs

5.2.1 Sample preparation

Surrogate internal standards (SIS, *o*-terphenyl, naphthalene-d_8, phenanthrene-d_{10}, chrysene-d_{12}, phenol-d_6, 4-methylphenol-d_8) are added to the water samples prior to processing, and recovery internal standards (RIS, 5α-androstane, fluorene-d_{10}, and acenaphthene-d_{10}) are added prior to analysis on GC/FID (gas chromatography/flame ionization detection) and GC/MS (gas chromatography/mass spectrometry).

For analyses of semi-volatile organic compounds (SVOC) and total petroleum hydrocarbons (TPH), the water samples are spiked with the appropriate surrogate internal standards and serially extracted with dichloromethane (DCM), thereby following a modification of EPA method 3510C (US EPA, 2015). The combined extracts are dried with sodium sulphate and concentrated to approximately 1 mL using a Zymark Turbovap® 500 Concentrator. The final extract is spiked with the appropriate recovery internal standards and analyzed on GC/FID and GC/MS.

5.2.2 Chemical analysis

The samples are analyzed for SVOCs using GC/MS, for TPH using GC/FID, and for volatile organic compounds (at least BTEX (benzene, toluene, ethylbenzene, and xylenes)), by use of P&T GC/MS (Purge and Trap Gas Chromatography Mass Spectrometry). A list of all target analytes is shown in Table 5.1. This list includes the recommended analytes given by Singer et al. (2000) and is a typical standard list for the target compounds used during post-oil spill damage assessments.

The GC/FID analyses are performed according to a modification of EPA Method 8015D (US EPA, 2015). TPH (resolved plus unresolved petroleum hydrocarbons) is quantified by the method of internal standards using the baseline corrected total area of the chromatogram and the average response factor for the individual C_{10} to C_{36} n-alkanes.

The SVOCs are quantified by modifications of EPA Method 8270E (US EPA, 2018). The mass spectrometer is operated in the selective ion monitoring mode to achieve optimum sensitivity and specificity. The quantification of target compounds is performed by the method of internal standards, using average response factors (RF) for the parent compounds. The PAH alkyl homologues are quantified using the straight baseline integration of each level of alkylation and the RF for the respective parent PAH compound. The response factors are generated for all targets and surrogates versus fluorene-d_{10}.

A total of 35 target volatile organic compounds (VOC) in the C_5 to C_{10} range may be determined by Purge and Trap (P&T) GC/MS using a modification of EPA method 8260D (US EPA, 2018). The samples are spiked with SIS (toluene-d_8 and ethylbenzene-d_8) and RIS (chlorobenzene-d_5). The quantification of individual compounds is performed by using the RFs of the individual compounds relative to the internal standards. All standards and samples are analyzed in a full scan mode. In the present project, at least BTEXs must be quantified.

Table 5.1 *Target organic analytes (SVOC: Semi volatile organic compounds, VOC: Volatile organic compounds, TPH: Total petroleum hydrocarbons, UCM. Unresolved organic materials).* *Optional compounds in grey fonts.*

Group	Compound	Abb	Group	Compound	Abb
Decalins	Decalin	DE	C0-C5 phenols	Phenol	PH
	C1-decalins	DE1		C1-phenols	PH1
	C2-decalins	DE2		C2-phenols	PH2
	C3-decalins	DE3		C3-phenols	PH3
	C4-decalins	DE4		C4-phenols	PH4
Naphthalenes	Naphthalene	N		C5-phenols	PH5
	C1-naphthalenes	N1	BTEX	Benzene	
	C2-naphthalenes	N2		Toluene	
	C3-naphthalenes	N3		Ethylbenzene	
	C4-naphthalenes	N4		*m*-xylene	
2-3 ring PAHs	Benzo(b)thiophene	BT		*p*-xylene	
	Biphenyl	B		*o*-xylene	
	Acenaphthylene	ANY	C3-benzenes	Propylbenzene	
	Acenaphthene	ANA		1-methyl-3-ethylbenzene	
	Dibenzofuran	DBF		1-methyl-4-ethylbenzene	
	Fluorene	F		1,3,5-Trimethylbenzene	
	C1-fluorenes	F1		1-methyl-2-ethylbenzene	
	C2-fluorenes	F2		1,2,4-trimethylbenzene	
	C3-fluorenes	F3		1,2,3-trimethylbenzene	
	Phenanthrene	P	Other VOC	Isopentane	
	Anthracene	A		n-C5 (Pentane)	
	C1-phenanthrenes/anthracenes	P1		Cyclopentane	
	C2-phenanthrenes/anthracenes	P2		2-methylpentane	
	C3-phenanthrenes/anthracenes	P3		3-methylpentane	
	C4-phenanthrenes/anthracenes	P4		n-C6 (Hexane)	
	Dibenzothiophene	D		Methylcyclopentane	
	C1-dibenzothiophenes	D1		Cyclohexane	
	C2-dibenzothiophenes	D2		2,3-dimethylpentane	
	C3-dibenzothiophenes	D3		3-methylhexane	

Group	Compound	Abb	Group	Compound	Abb
	C4-dibenzothiophenes	D4		n-C7 (Heptane)	
4-6 ring PAHs	Fluoranthene	FL		Methylcyclohexane	
	Pyrene	PY		2,4-dimethylhexane	
	C1-fluoranthrenes/pyrenes	FL1		2-methylheptane	
	C2-fluoranthenes/pyrenes	FL2		n-C8 (Octane)	
	C3-fluoranthenes/pyrenes	FL3		n-C9 (Nonane)	
	Benz[*a*]anthracene	BA		n-C10 (Decane)	
	Chrysene	C		n-Butylbenzene	
	C1-chrysenes	C1		1,2,4,5-tetramethylbenzene	
	C2-chrysenes	C2		n-pentylbenzene	
	C3-chrysenes	C3			
	C4-chrysenes	C4			
	Benzo[*b*]fluoranthene	BBF	TPH	C10-C36	
	Benzo[*k*]fluoranthene	BKF	WAF	Sum of VOC and TPH	
	Benzo[*e*]pyrene	BEP	UCM	TPH - SVOC	
	Benzo[*a*]pyrene	BAP			
	Perylene	PE			
	Indeno[*1,2,3-c,d]*pyrene	IN			
	Dibenz[*a,h]*anthracene	DBA			
	Benzo(g,h,i)perylene	BPE			

5.3 Toxicity testing

The copepod *Acartia tonsa* is the common test species for the laboratories. A second, more Arctic relevant specie could also be included as a part of the screening. Calculation of Toxic Unit should be mandatory for all laboratories preparing WAFs.

5.3.1 Acute toxicity to *Acartia tonsa*

Potential effects on primary consumers will be assessed with the marine pelagic copepod *Acartia tonsa*. The bioassays are performed as a modification of ISO 14669 (1999) with lethal immobilization (LC_{50} or LC_{10}) as the endpoint.

- The original ISO protocol is not designed for testing of solutions containing volatiles and has been adapted for WAFs from petrogenic oils.
    - The recommended test vessels (semi-open 50 mL flasks with 25 mL test solution) is replaced with Erlenmeyer flasks (100 mL) filled to approximately 5 mL headspace and the flasks are sealed with glass stoppers to avoid loss of volatiles.
- All bioassays are performed with a fixed number of vessels and a dilution series of WAF ranging from undiluted (100%) to 4% WAF in sea water with a spacing factor of 1.7 (i.e. 58.8%, 34.6%, 20.4%, 12.0%, 7.0% and 4.1%) and 4 parallel vessels for each dilution.
- To ease the distribution to the test vessels, each dilution can be prepared as a common dilution for the series and distributed to the 4 parallel vessels by filling the flasks to the lower level of the stoppers and removing 5 mL with a volumetric pipette to give the required headspace.
- Negative controls: Eight vessels filled with seawater, 5 mL headspace.
- Positive controls: Four vessels filled with 3.5-dichloro phenol at a nominal concentration of 1 mg/L, 5 mL headspace.
- Saturation of oxygen and pH are to be measured in the seawater used to dilute the exposure solutions and in the undiluted WAF.

- After preparation of the exposure solutions and control vessels, 10-15 copepodite V or adult *A. tonsa* are transferred to each vessel. The density of animals should not exceed 1 per 5 mL of solution.
- After all vessels are supplied with animals, the flasks are left in a temperature-controlled room at nominal 20±2°C under a light regime comparable to the culturing conditions.
- The vessels should be inspected daily for the next 2 days (48 hours) for immobilized animals and recording of temperature.
- At the end of exposure, the pH and saturation of oxygen are measured in one vessel in each of the exposure dilutions, in one of the positive controls, and in two of the negative control vessels.
- The calculated values are corrected for mortality in the control series and the effect is calculated within the span 0-100% by constraining the top and bottom of the concentration-effect curve to 100 and 0. In addition, the mortality in the controls is also reported.

- The acute toxicity, expressed as LC_{50}, should be calculated both in percent dilution of the WAF (relative toxicity, LC_{50} in percent (%)), as well as normalized to the total WAF concentration (specific toxicity, LC_{50} in mg/L or ppm). Due to low toxicity in WAFs of some LSFOs, LC_{10}s should be included. Total WAF concentration is sum of volatiles plus TPH.
- Low values of LC_{50} indicate a high toxicity, while a high value of LC_{50} corresponds to lower toxicity.
- Results should be presented both as relative and specific toxicity. The two approaches to express toxicity have different applications:
    - Specific toxicity expresses the toxicity of the WAF of a selected oil and test condition and is associated with the concentration of the WAF.
    - Relative toxicity expresses a given dilution to obtain a predefined effect (e.g. LC_{50}) and can be used for comparing the toxicity of WAFs from different oils with different WAF compositions and hence, solubility and chemical composition.

5.3.2 Acute toxicity of optional Arctic species

SINTEF has selected the copepod *Calanus finmarchicus* as a second organism as this copepod has been used for toxicity testing to WAFs of a large number of oils. The same ISO protocol is modified, but the toxicity testing is performed at 10 °C.

5.3.3 Calculation of toxic units (TU)

In the WAFs from petrogenic products, the compounds of concern for toxicity assessment are typically limited to the VOCs and SVOCs, which are structurally classified as Type I narcotics. The target lipid model of narcotic toxicity demonstrates that the acute toxicities of these chemicals vary and are correlated with the octanol-water partition coefficient (K_{ow}) such that LC_{50} decreases with increasing K_{ow} (DiToro et al. 2007).

A regression model for the relationship between the acute toxicity and K_{ow} of target chemicals to estimate the threshold toxic concentration of each compound is used by several authors. It appears to be a linear negative relationship between the log of the median lethal concentration (LC_{50}) to the organisms and the log K_{ow} of compounds exhibiting toxicity by non-specific narcotic action (e.g. McCarty et al. (1992 and 1993) and Di Toro et al. (2007), Neff et al. (2005)). The relationship is described by a linear regression of log LC_{50} (mM/L) against log K_{ow} for each compound:

$$\log LC_{50} = m * \log (K_{ow}) + b \qquad (1)$$

where m is the slope, and b is the intercept of the equation. The slope is related to the partition behavior of the chemical and should therefore be constant from species to species. The y-intercept *b* can be interpreted as the lipid-normalized critical body burden corresponding to the observed endpoint, such as 50% mortality for the LC_{50} for the specific organism being considered. It is variable by species and likely life stage and condition (McGrath and DiToro, 2009).

There are several ways to predict toxicity, and here we have chosen the approach described in e.g. McCarty et al. (1992, 1993) and Neff et al. (2005). The acute toxicity of individual MAHs and PAHs were estimated by the regressions and are given in **Error! Reference source not found.**:

MAHs (McCarty et al.,1992): $\log LC_{50}\ (mM) = -0.9 * \log (K_{ow}) + 1.71 \quad (2)$

PAHs (Neff et al.,2005): $\log LC_{50}\ (mM) = -1.162 * \log (K_{ow}) + 2.496 \quad (3)$

The acute toxicity of phenols was estimated with the regression of McCarty et al. (1993) for polar narcosis:
Phenols: $\log LC_{50}\ (mM) = -0.55 * \log (K_{ow}) + 0.064 \quad (4)$

To calculate TU, LC_{50}s must be given in mg/L:
$LC_{50}\ (mg/L) = LC_{50}\ (mM) * M_w$ (M_w = molecular weight) (5)

The predicted toxicities of the individual compounds in the WAFs are given in *Table* ***5.2***.They are assumed to be additive and toxic unit (TU) for each compound was summed to produce a toxic unit, which is equivalent to an estimate of the acute toxicity of each WAF. The TU for each component (i) is calculated in the WAF system:

$$TU_i = C_i/LC_{50i} \qquad (6)$$

C_i is the concentration of component i in the WAF and LC_{50i} is the estimated acute toxicity for component i (*Table* ***5.2***) calculated from equations (2) to (4). The estimated toxicity of the total WAF is determined by the sum of the TUs of all component groups. A value of TU > 1 implies toxicity, i.e. the WAF is expected to cause 50% mortality in the test organisms.

The sum TU computed represents the TUs from all measured hydrocarbons in the exposure system. UCM (unresolved complex mixture), which could contribute to more than 90% of the total WAF concentration in e.g. WAFs of heavily weathered oils, is not included in TU calculations.

Table 5.2 *Predicted LC_{50} based on $logK_{OW}$. Equation for MAH is from McCarty (1992), PAHs from Neff et al. (2002), and phenols from McCarty (1993). Optional compounds in grey fonts.*

Compound	Mw	log Kow	LC_{50} (mg/L)
Decalin	138	4.20	**0.57**
C1-decalins	152	4.61	**0.21**
C2-decalins	166	5.11	**0.06**
C3-decalins	180	5.60	**0.02**
C4-decalins	194	6.09	**0.01**
Benzo(b)thiophene	134	3.13	**9.68**
Naphthalene	128	3.30	**5.87**
C1-naphthalenes	142	3.79	**1.76**
C2-naphthalenes	156	4.24	**0.58**
C3-naphthalenes	170	4.73	**0.17**
C4-naphthalenes	184	5.22	**0.05**
Biphenyl	154	3.94	**1.27**
Acenaphthylene	152	3.44	**4.79**
Acenaphthene	154	3.88	**1.50**
Dibenzofuran	168	3.95	**1.35**
Fluorene	166	3.93	**1.41**
C1-fluorenes	180	4.37	**0.47**
C2-fluorenes	194	4.82	**0.15**
C3-fluorenes	208	5.32	**0.04**
Phenanthrene	178	4.58	**0.27**
Anthracene	178	4.55	**0.29**
C1-phenanthrenes	192	5.04	**0.08**
C2-phenanthrenes	206	5.45	**0.03**
C3-phenanthrenes	220	5.91	**0.01**
C4-phenanthrenes	234	6.36	**0.003**
Dibenzothiophene	184	4.37	**0.48**
C1-dibenzothiophenes	198	4.86	**0.14**
C2-dibenzothiophenes	212	5.33	**0.043**
C3-dibenzothiophenes	226	5.81	**0.013**
C4-dibenzothiophene	240	6.43	**0.0025**
Fluoranthene	202	5.19	**0.059**
Pyrene	202	5.13	**0.069**
C1-fluoranthenes/pyrene	216	5.26	**0.052**
C2-fluoranthenes/pyrene	230	5.56	**0.025**
C3-fluoranthenes/pyrene	244	6.38	**0.003**
Benz(a)anthracene	228	5.74	**0.015**
Chrysene	228	5.78	**0.014**
C1-chrysenes	242	6.19	**0.0049**
C2-chrysenes	256	6.59	**0.0018**
C3-chrysenes	270	5.97	**0,0098**
C4-chrysenes	284	7.42	**0.0002**
Benzo(b)fluoranthene	252	5.57	**0.027**
Benzo(k)fluoranthene	252	6.40	**0.0029**
Benzo(e)pyrene	252	6.45	**0.0025**
Benzo(a)pyrene	252	6.41	**0.0028**
Perylene	252	6,45	**0.0025**
Indeno(1,2,3-c,d)pyrene	276	6.16	**0.0060**
Dibenz(a,h)anthracene	278	7.13	**0.0005**
Benzo(g,h,i)perylene	276	7.47	**0.0002**
Phenol	94	1,50	**16.3**
C1-Phenols	108	1.98	**10.2**
C2-Phenols	122	2.35	**7.21**
C3-Phenols	136	2.70	**5,16**
C4-Phenols	150	3,31	**2.63**
C5-phenols	164	3.50	**2.26**
Benzene	78	1.94	**71.8**
Toluene	92	2.51	**26.0**
Ethylbenzene	106	3.01	**10.6**
m-xylene	106	3.04	**9.98**
p-xylene	106	3.04	**9.98**
o-xylene	106	2.95	**12.0**
C3-benzenes	120	3.58	**3.69**

6 Reporting and presentation of results

To obtain a common and standardized format for the presentation of results from the participating laboratories, customized spreadsheets (Excel templates) for standardizing the reporting of the results are provided, including:

- An overview table of the Physical properties
- Figures / graphs of e.g.:
  - Distillation curve
  - Viscosity Temperature sweep

- Tables with
  - TPH, SVOC and BTEX concentrations in the WAF systems
  - Toxic unit of the WAF systems (individual components and total TU
  - Results from acute toxicity tests, expressed as relative and specific toxicity (LC_{50} and LC_{10})

- GC-FID chromatograms must be included in the reported results from each laboratory:
  - Oils
  - WAFs

7 References

ASTM D93 (Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester)

ASTM D97 (Standard Test Method for Pour Point of Petroleum Products)

ASTM E203 (Standard Test Method for Water Using Volumetric Karl Fischer Titration)

ASTM D4052 (Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter)

ASTM D4294 (Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry)

ASTM D5002 (Standard Test Method for Density, Relative Density, and API Gravity of Crude Oils by Digital Density Analyzer)

ASTM D5291:2012 (Standard test method for instrumental determination of carbon, hydrogen, and nitrogen in petroleum products and lubricants).

ASTM D5949 (Standard Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method))

ASTM D7042 (Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity))

ASTM D7094 (Standard Test Method for Flash Point by Modified Continuously Closed Cup (MCCCFP) Tester)

ASTM D7169 (Standard Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High Temperature Gas Chromatography)

ASTM E203 (Standard Test Method For Water Using Volumetric Karl Fischer Titration)

IP 143 (Determination of asphaltenes (heptane insolubles) in crude petroleum and petroleum products)

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