

DIN EN 15522-2:2025-07 (E)

Oil spill identification - Petroleum and petroleum related products - Part 2: Analytical method and interpretation of results based on GC-FID and GC-low resolution-MS analyses (includes Amendment A1:2025)

Contents		Page
European foreword		8
Introduction		10
1 Scope.....		12
2 Normative references.....		12
3 [A1] Terms and definitions [A1]		13
3.1 General.....		13
3.2 Sample comparison.....		15
3.3 Abbreviations.....		15
4 Strategy for the identification of oil spill sources		16
4.1 General.....		16
4.2 Basis for reliable conclusions – Numerical comparisons		17
5 General lab instructions		18
5.1 Sampling and sample preparation		18
5.2 GC-FID and GC-MS analysis		18
5.3 Conclusions and reporting		20
6 Sample preparation		20
6.1 General.....		20
6.2 Visual examination and description of samples.....		20
6.3 Preparation.....		21
6.3.1 Sample storage.....		21
6.3.2 Water samples		21
6.3.3 Oil samples from an Ethylene-tetrafluorethylene (ETFE) net.....		22
6.3.4 Thick oil and emulsified oil samples.....		22
6.3.5 Tar balls and emulsified lumps.....		22
6.3.6 Samples from oiled birds, fish and other animals and vegetation.....		23
6.3.7 Sediment		23
6.4 Sample clean-up		23
6.4.1 General.....		23
6.4.2 Particle removal.....		23
6.4.3 Asphaltenes precipitation		24
6.4.4 Alumina column clean-up of biogenic materials		24
6.4.5 Silica or Florisil® column clean-up.....		25
6.5 Recommended injection concentration		26
7 Characterization and evaluation of analytical data.....		27
7.1 General.....		27
7.2 Characterization by GC-FID – Level 1.....		28
7.2.1 General.....		28
7.2.2 Evaluation of the influence of weathering on sample comparison.....		28
7.2.3 Acyclic isoprenoids ratios – Level 1.2.....		31
7.2.4 Level 1 criteria		31
7.2.5 Level 1 conclusions		32
7.3 Characterization by GC-MS – Level 2.....		32
7.3.1 General.....		32

7.3.2	Visual inspection and overall characterization - Level 2.1.....	32
7.3.3	Treatment of the GC-MS results – Level 2.2.....	33
7.4	Treatment of the results using the MS-PW-plot- Level 2.2	33
7.4.1	General	33
7.4.2	PW-plot calculations.....	34
7.4.3	Evaluation of the variability of the analysis and peak integration.....	34
7.4.4	Evaluation of weathering.....	36
7.5	Treatment of the results using diagnostic ratios – Level 2.2	37
7.5.1	General	37
7.5.2	Diagnostic ratios calculation	37
7.5.3	Normative diagnostic ratios	38
7.5.4	Analytical error	42
7.5.5	Match-criterion for ratios.....	43
7.5.6	Criteria for selecting, eliminating and evaluating diagnostic ratios.....	44
7.6	Conclusions.....	48
8	Reporting.....	50
8.1	General	50
8.2	Internal documentation – technical report.....	50
8.3	Identification report – summary report.....	51
9	Quality assurance	52
Annex A	(normative) GC-FID analysis	53
A.1	General	53
A.2	Analytical standards for GC-FID analyses.....	53
A.2.1	N-alkanes	53
A.2.2	Injection concentration of the standard GC-FID.....	54
A.2.3	Storage of frequently used standard solutions.....	54
A.3	Suggested instrumental conditions	54
A.4	Measures to improve and verify the accuracy of the method – GC-FID.....	55
A.4.1	Mass discrimination	55
A.4.2	Column resolution.....	56
A.4.3	Linearity.....	58
A.4.4	Mid-level concentration	59
A.4.5	Variance	59
A.4.6	GC-FID sequence.....	59
Annex B	(normative) GC-MS analysis	60
B.1	General	60
B.2	Analytical standards for GC-MS analyses.....	60
B.2.1	General	60
B.2.2	Crude oil to be used around each sequence.....	61
B.2.3	Oil mixture	61
B.2.4	Analytical standards for PAH homologues.....	61
B.2.5	FAMEs.....	62
B.2.6	Storage of frequently used standard solutions.....	62

B.3	Suggested instrumental conditions.....	62
B.3.1	GC conditions for the exchange of analytical results	62
B.3.2	GC-MS conditions for full-scan analysis	65
B.3.3	MS preparation for selected ion monitoring (SIM) analysis	65
B.4	Measures to improve and verify the accuracy of the GC-MS method	66
B.4.1	Relative retention time.....	66
B.4.2	Mass discrimination.....	66
B.4.3	Peak symmetry and column resolution.....	66
B.4.4	Patterns.....	67
B.4.5	Linearity.....	67
B.4.6	Mid-level concentration	67
B.4.7	Variance.....	68
B.4.8	Sample analysis with GC-MS.....	68
Annex C (informative)	Precision statement.....	69
C.1	General.....	69
C.2	Precision of the MS-PW-plot	69
C.3	Precision of the ratio comparison	70
C.4	Reproducibility.....	71
C.5	The effect of the ratio type on the RSD.....	72
C.6	Example of a paired ratio calculation in Excel®	73
C.7	Calculation of the evaporation line for the MS-PW-plot in Excel®.....	74
Annex D (normative)	Evaluative reporting using match definitions or likelihood ratios	76
D.1	General.....	76
D.2	Match definitions	76
D.3	Likelihood ratios (LR)	77
Annex E (normative)	List of compounds and compound groups analysed by GC-MS-SIM.....	79
E.1	General.....	79
E.2	Compounds.....	80
E.2.1	General.....	80
E.2.2	Compound type.....	85
E.3	Normative ratios and informative ratios.	86
Annex F (informative)	Chromatograms and ratios of compounds and compound groups analysed by GC-MS-SIM.....	90
F.1	General.....	90
F.2	Alkanes	90
F.3	Cyclohexanes and polycyclic alkanes	92

F.4	Mono-aromatic and poly-aromatic compounds	97
F.4.1	Alkyl-benzenes and alkyl-toluenes	97
F.4.2	PAHs, alkyl-PAHs and S-PAHs	97
F.4.3	Tri-aromatic steranes	109
F.5	FAMES	109
Annex G	(informative) General composition of oils – chemical groups	113
G.1	General	113
G.2	Hydrocarbons	114
G.3	Aliphatic compounds	114
G.3.1	General	114
G.3.2	Paraffins	114
G.3.3	Naphthenes	115
G.4	Aromatic compounds	115
G.5	Heteroatomic organic compounds	115
G.5.1	General	115
G.5.2	Resins	115
G.5.3	Asphaltenes	116
Annex H	(informative) Weathering of oils spilled on water and land	117
H.1	General	117
H.2	Weathering processes	117
H.2.1	Weathering of oils spilled on water	117
H.2.2	Weathering of waterborne oils stranded on land or land based oil spills	119
H.2.3	Mixing and contamination	119
H.2.4	Dispersion	120
H.2.5	In-situ burning	122
H.3	Evaluation of weathering processes	123
H.3.1	Evaporation	123
H.3.2	Dissolution	126
H.3.3	Photo-oxidation	128
H.3.4	Biodegradation	134
H.3.5	Wax redistribution	137
H.3.6	Mixing	143
H.3.7	Contamination	145
H.3.8	In-situ burning	145
Annex I	(informative) Characteristic features of different oil types in oil spill identification	148
I.1	General	148

I.2	Crude oil	148
I.2.1	General	148
I.2.2	Analysis	149
I.3	Light fuel oil (gas oil, diesel, fuel No 2, biofuels, GTL)	155
I.3.1	General	155
I.3.2	Analysis, GC screening	156
I.3.3	GC-MS analysis	158
I.3.4	Biofuels	161
I.3.5	Gas to liquid products	162
I.4	Lubricating oil	163
I.4.1	General	163
I.4.2	Analysis	165
I.5	Heavy fuel oil (HFO, Bunker C, Fuel No 6) and low sulfur fuel oil	169
I.5.1	General	169
I.5.2	Analysis	170
I.6	Waste oil (bilge oil, sludge, slops)	178
I.6.1	General	178
I.6.2	Analysis	179
I.7	Conclusion	183
Annex J (informative) Example of external documentation – identification report of an oil spill case		
		185
J.1	General	185
J.2	Sample information	185
J.3	Analytical procedure	185
J.3.1	Method	185
J.3.2	Dilution/extraction	185
J.3.3	Analyses	185
J.4	Results	185
J.5	Interpretation	186
J.5.1	General	186
J.5.2	Positive match	186
J.5.3	Probable match	186
J.5.4	Inconclusive	186
J.5.5	Non-match	186
J.6	Conclusions	186
Annex K (informative) Example of internal documentation – technical report of an oil spill case		
		188

K.1	General	188
K.2	Sample information	188
K.2.1	Samples	188
K.2.2	Contact information.....	188
K.2.3	Request.....	188
K.2.4	Photo(s) of the samples.....	189
K.3	Sample preparation and analyses	189
K.4	Quality assurance	191
K.5	GC-FID results - Level 1	193
K.5.1	GC-FID chromatograms - Level 1.1	193
K.5.2	GC-FID numerical comparisons - Level 1.2	195
K.5.3	GC-FID conclusions.....	200
K.6	GC-MS results - Level 2	201
K.6.1	General	201
K.6.2	GC-MS chromatograms - Level 2.1	201
K.6.3	GC-MS numerical comparisons - Level 2.2	203
K.6.4	Visual inspection.....	210
K.6.5	Overall conclusions.....	211
	Bibliography	212