

**Workshop on oil fingerprinting and the
work of the Oil Spill Identification
Network (OSINet), 2 March 2016, Lisbon**

Workshop Report

Final version

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List of Abbreviations

OSINet	Oil Spill Identification Network of Experts within the Bonn Agreement
CTG MPPR	Consultative Technical Group for Marine Pollution Preparedness and Response
COSIweb	Central, browser-based database, which supports the work of OSINet
GC-FID	Gas Chromatography - Flame Ionization Detector
GC-MS	Gas Chromatography - Mass Spectrometry
CEN	European Committee for Standardisation

1. Background

One of the European Maritime Safety Agency's (EMSA) core tasks is to support Member States in their marine pollution preparedness, detection and response capabilities. The Agency's Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR), comprised of marine pollution response experts from EU Member States, EEA/EFTA coastal countries and coastal EU Candidate Countries facilitates the exchange of information and the identification of priority topics in the field of marine pollution response. Following discussions at the 9th CTG MPPR meeting in October 2014 regarding the importance of oil fingerprinting and the wish to make the work of the Oil Spill Identification Network of Experts within the Bonn Agreement (OSINet) wider known at EU level, EMSA hosted a workshop on 2 March 2016 to address oil spill identification and demonstrate the OSINet work and achievements.

The workshop '**Oil fingerprinting and the work of OSINet**' was organised by a group of OSINet experts from Germany, the Netherlands, Belgium, in close cooperation with EMSA, and was held on 2 March 2016 in Lisbon, as per workshop Agenda (attached as **Annex 1**). To facilitate the workshop's discussions, a document (attached as **Annex 2**) and a questionnaire (attached as **Annex 3**) were developed and sent to the participants in advance. The workshop's target participant group were experts from national (forensic) laboratories dealing with identification and fingerprinting of oil samples and experts from maritime emergency response organisations.

The workshop was co-chaired by Eric Donnay, Marine Environment Service, Belgium, and Walter Nordhausen, Pollution Response Services, EMSA, and was well attended by 29 experts from 19 countries representing either emergency response organisations or national laboratories dealing with oil samples (the list of participants is attached as **Annex 4**). All documents and presentations related to the workshop may be accessed online at: <http://emsa.europa.eu/workshops-a-events/188-workshops.html>.

2. Workshop opening

Leendert Bal, Head of Department Operations, EMSA, welcomed the participants to the workshop reiterating the significant work undertaken in oil fingerprinting by the OSINet group of experts, which currently includes experts from 14 EU countries and seven non-EU countries. He highlighted the importance of sharing best practices and expertise within Europe on oil spill identification and pointed out that this workshop provides the platform for such exchange of experiences at EU level.

Eric Donnay presented the scope and main workshop objectives, which were to:

- Achieve a better understanding on oil sampling and identification methods across Europe;
- Demonstrate the work of the Bonn Agreement Oil Spill Identification Network to all the EU Member States;
- Facilitate the exchange of knowledge and expertise in this area and widen cooperation among experts.

Walter Nordhausen presented a brief overview of EMSA's mandate, tasks and services in the field of maritime safety and pollution response, emphasising that the Agency provides information, technical and operational support and assistance to the European Commission and the EU Member States. The participants briefly introduced themselves, showing that experts from laboratories, MRCCs, pollution response authorities and legal experts attended this workshop.

3. Setting the scene (Session 1)

3.1 Questionnaire Outcome

Uta Kraus, Federal Maritime and Hydrographic Agency, Germany, presented a summary of the answers received from the Questionnaires (17 countries replied in total). The Questionnaire (template attached as **Annex 3**) aimed to provide an overview of how oil spill sampling is currently organised across Europe, in particular in countries currently not participating in OSINet. Based on the answers received, the following points were presented:

- Oil spill response is very differently organised in Europe.
- Several countries (but not all) make use of specialised governmental laboratories handling oil spill analysis and conduct their analysis based on national or international official guidelines (all OSINet members use the CEN/TR:15522 Guidelines).
- Almost half of the laboratories that analyse oil samples (six OSINET-members, two non-OSINET-members) are also used to analyse unknown or non-oil substances (e.g. paraffin, solvents, sea water quality).
- Countries already participating in OSINet seem to handle more court-related cases.
- The majority of countries do not have a centralised national database for oil sample analyses in place.

Uta Kraus concluded that, based on the information from the Questionnaires, OSINet seems to facilitate the practical procedures of sampling and analysis and simplifies co-operation between laboratories and countries.

During the discussions, Finland highlighted that the CEN methods (see 4.1), which were developed within OSINET, for oil spill sampling and oil identification can be a very useful tool in court, as they enable a robust and well prepared contribution from the lab experts, and it was noted that in Belgium the CEN methods have prevented cases even going to court, as the oil spill source had been rapidly identified. It was emphasised that depending on the country, and if there is not further evidence available, the oil spill analysis and identification may or may not be enough in court to identify the pollution source, but it is definitely a very important tool to be used. The costs of analysing an oil sample were discussed and they vary depending on the country where the sample is analysed, but the labs currently participating in OSINet are well equipped and with vast expertise and they can provide further information if needed regarding the costs and duration of an oil sample analysis.

3.2 *Flinterstar* Incident

Eric Donnay presented an overview of the oil sampling undertaken during the *M/V Flinterstar* incident which occurred in October 2015 in the Belgian territorial waters (while the oil removal operation was completed in November 2015, the incident is still ongoing, with the wreck removal phase). A total of 48 samples were taken (in triplicate), from several locations and sources (vessels' tanks; sea water; beach; drift wood), of which 21 were analysed in the lab. As per the Belgian standard, the maritime police registered all the samples taken as evidence, which is important should they be later used in court. It was underlined that a national sampling strategy and the OSINet procedures were key elements for establishing the causal link between the observed pollution and the source of the pollution and the OSINet and COSIweb database proved to be useful for sharing information about samples and results of analyses between the various countries involved (Belgium, the Netherlands and France).

During the discussions the best way to take samples from the water was addressed, with Finland noting the sampling net thrown from a helicopter as an example and Belgium mentioning sampling buoys or sampling from ships as possible options, noting that a challenge with the sampling buoys is their collection afterwards. The use of the Teflon net was mentioned as a practical and easy way to collect oil samples from the water.

4. The work of OSINet (Session 2)

4.1 Oil Spill Identification Network of Experts (OSINet)

Paul Kienhuis, RWS lab, the Netherlands, presented the timeline of establishing the OSINet within the Bonn-Agreement in 2005 and in parallel the progress made in establishing the oil identification analytical method, starting from the NORDTEST protocol in 1991 until the publication by the European Committee for Standardisation of the CEN/Tr 15522 method.

- Part 1 of this method was published in 2006 and it describes sampling techniques and the handling of oil samples prior to their arrival at the forensic laboratory (CEN/Tr 15522-1).
- Part 2 was published in 2012 and covers laboratory procedures of oil spill identification methodology, analytical techniques, data processing, data treatment, and interpretation and reporting of results (CEN/Tr 15522-2).

There is the intention to republish both parts one and two of the CEN method in 2018.

OSINet's main objective is to enhance cooperation and the transfer of knowledge between different countries through a network of expert chemists specialized in oil spill identification techniques. Information on the OSINet is available on the Bonn Agreement website: <http://www.bonnagreement.org/osinet>. To all OSINet participating members, a password-protected OSINet server is available to enable the transfer of confidential information and knowledge, as well as the exchange of data via the COSIweb database established in 2012. The annual intercalibration exercises (ring tests) conducted among the OSINet members deal with different scenarios each time, aiming to address different problems connected with oil identification by investigating different oil products. They also aim to facilitate quality assurance among the Network's lab work and to enhance the experts' experience in using COSIweb. For example the latest ring test, conducted in 2015 dealt with diesel mixed with biodiesel samples. The test results are then presented and discussed at the annual OSINet meetings.

Today, OSINet has expanded to become a worldwide group of experts with laboratories participating from 22 countries. Overall OSINet enhances cooperation in oil spill sampling and oil identification and fingerprinting and provides guidance and mutual assistance among its members with sampling, analysis and education/training activities. Finland commented the added value of cooperating with experts in this field, as expertise in this field is not readily available in every country.

4.2 Oil Fingerprinting (sampling and the analytical method)

Paul Kienhuis provided a brief overview of oil sampling procedures and materials, such as the Teflon net, and how to properly transport samples and described the processes, instruments and methods used in oil identification / oil fingerprinting, including:

- The Gas Chromatography-Flame Ionization Detector (GC-FID), a rapid analysis that provides an overview regarding the oil type of the sample, the degree of weathering and the similarity between spill and suspected sources as well as the needed injection volume for more detailed analysis (if necessary).
- The Gas Chromatography-Mass Spectrometry (GC-MS), a more detailed analysis that detects the presence of a particular array of compounds (like various PAHs and biomarkers) and the results of which are used to confirm the type of oil, based on the presence or absence of characteristic compounds in the samples.
- Visual and numerical comparison of samples with ratios and percentage weathering plots.

4.3 COSIweb

Uta Kraus demonstrated the use of the COSIweb database, which is the browser-based computerised oil spill identification database hosted by Germany and used by the OSINet members to share and compare oil sampling data. COSIweb currently includes data of about 2800 analysed oil samples (among them 300 crude oils from all over the world). COSIweb has two main functions:

- Characterisation of unknown samples (“mystery spills”) – based primarily on their comparison with the 2800 oil samples currently in the database.
- Comparison of two given samples by automatically producing the parameters needed for a conclusion according to the CEN guideline (this is the case for pollution incidents with one or more suspected polluters). The results can be shared and displayed through an automatically generated report, directly via the internet, which greatly facilitates cooperation among the various users.

These functions are achieved by an automated and rapid process based on the import and comparison of raw data (CDF-files) of oil samples from the COSIweb users. 20 registered laboratories currently have access to the COSIweb and are trained in its use.

Additional information on the work and tools of the OSINet, the oil fingerprinting methods and the COSIweb is included in the workshop Document in **Annex 2**.

5. Real cases (Session 3)

5.1 Oil Spill Identification in Spain and recent Oil Spill Cases

Following a brief overview of the main tasks and available resources of the Spanish Maritime Safety Agency (SASEMAR) by Laura de la Torre, SASEMAR, Spain, their role in oil sampling and the evolution of the sampling procedures in Spain since the *Prestige* incident in 2002, was presented. Every SASEMAR unit (maritime or aerial) and every Coordination Centre is equipped with sampling kits. The sampling efforts undertaken during the recent accidental spills (*Oleg Naydenov*, April 2015 and *Nele Maersk*, September 2015) were also presented, demonstrating the usefulness and importance of sampling for claims management and cost recovery.

When it comes to intentional (non-accidental) spills, real case examples were provided, highlighting the importance of sampling with regard to identifying the pollutant, the polluter and helping define if the discharge is legal or illicit, in accordance with international regulations. It was emphasised that having procedures in place to guarantee the quality of samples and having rapid access to a reliable laboratory with experience in this field are crucial for emergency responders.

Joan Albaigés, CSIC, Spain, presented the important role of a central laboratory in the overall response mechanism and through examples of real case, he highlighted the support a lab can provide to the response operations regarding the spill's source identification. This is achieved by analysing and comparing the various samples, and through this analysis identifying the pollutant (characterisation of oil) and the polluter (source of oil spill). He noted that having one central laboratory doing this analytical work has as advantage the historical memory and expertise of the lab, as it deals continuously with different oil spills.

5.2 *Flinterstar* case – Analytical results

Due to time limitations and considering that the sampling efforts and sample analysis conducted during the *Flinterstar* incident were already briefly addressed by Eric Donnay during the first session, this presentation by Paul Kienhuis was not provided during the workshop. The presentation is included in the workshop documentation and is available on EMSA's website.

6. Practical exercise (Session 4)

The workshop participants were separated in two break-out groups to discuss and go through a practical scenario introduced by Paul Kienhuis regarding a real harbour oil spill which occurred in the Netherlands in 2011, with more than one potential spill source. The break-out group discussions were facilitated by Paul Kienhuis and Uta Kraus and the main topics addressed in the two groups included:

- Initial actions to be taken after an oil spill.
- When, where and how to conduct oil sampling.
- How is sampling conducted in the various countries.
- How are sampling and oil identification used and accepted as evidence in court in the various countries.
- A live demonstration of the COSIweb tool was performed to demonstrate how samples are introduced and compared in the database and the type of oil identification results that can rapidly be provided to the user were shown.

The outcome of the practical break-out sessions was presented by both groups and discussed back in plenary, highlighting the following points:

- Sampling the oil spill and from all possible spill sources should be among the initial actions, following the spill mitigation and containment efforts.
- How the official registration and validation of the samples is done, by which (national) authority samples should be taken, and whether these samples would be accepted in court as sole evidence or as one type of evidence, varies among EU countries.
- Regular training in oil spill sampling (how, where, what to sample etc.) is only available in very few countries and was identified as a need by several participants. It is very important to sample correctly, in order to facilitate the lab to perform a proper analysis of the sample received.
- The COSIweb database is a very useful tool for supporting oil spill identification and the exchange of data. Its content keeps growing, with the import of more and different types of oil samples, an even though it was developed initially for crude and HFO oils, there is the will to expand it to also cover diesel, paraffin etc. Users (labs) must adapt their analytical procedures and must be trained to use COSIweb correctly.

7. Enhanced cooperation in Europe (Session 5)

7.1 Cooperation between a national maritime response organisation and a national laboratory (The Netherlands experience)

Jan Kool, Rijkswaterstaat (RWS), the Netherlands, presented the national maritime response structure. He noted the role of the RWS, in charge of tactical and operational aspects of the response, and the role of the national laboratories which are responsible for analysing samples, providing sample boxes (on board vessels) and conducting training on sampling. Through real case examples, such as the salvage of the *Baltic Ace* and the unknown substances which appeared on the beach, the added value of the work of the laboratories was pointed out. Their work supports the cost recovery procedure, the analysis of unknown substances, which is a challenge for several countries, and provides input to the advice given by the National Environmental Water Pollution Coordination Commission (LCM) to the national response organisations.

7.2 Requirements for participation in OSINet

Uta Kraus explained the requirements for participating in the OSINet group of experts, noting that OSINet works on a self-financing base, meaning that participants pay their own travel costs to attend the annual meetings and there is no 'membership fee'. Intercalibration tests and annual meetings are organised and executed by every OSINet

member state in turn to share the associated costs; for example the next annual OSINet meeting will be hosted by Finland from 20 to 22 April 2016. The participation in the annual OSINet intercalibration tests, at least every second year, and the agreement to confidentiality regarding information shared within the network (e.g. oil spill cases etc.) are required by all participating members to the OSINet. A governmental lab or other lab can participate in the OSINet group.

7.3 Plenary discussion on how to enhance cooperation in oil spill identification across Europe

The high level of knowledge and expertise within the OSINet group and its importance in facilitating the exchange of best practice and data among experts facing the same challenges was emphasised by the countries participating in OSINet. The fact that all laboratories participating in OSINet work with a common analytical method, have common quality assurance in place and can easily exchange samples and analytical results was noted among the practical advantages of participating in OSINet.

EMSA clarified that oil spill sampling and oil spill identification do not fall under its pollution response mandate, but it can provide via dedicated workshops or under the framework of its Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR) the forum at EU level to further address this topic, if the need is identified at this workshop. EMSA's assistance in addressing this important topic was supported by the majority of the participants. Finland underlined that sampling is an integral part of the chain of response in addressing illegal discharges and that it is not to be seen in isolation. The work of OSINet could also bring together Europe's collective oil spill memory, by collecting and comparing old and new samples. The idea of developing a practical new guidance document on oil spill sampling and oil identification, similar to existing documents such as the "EU Operational Guidelines on Places of Refuge", the guidance document "Addressing illegal discharges in the marine environment" or the "EU States Claims Management Guidelines" was also discussed and strongly supported by the workshop participants.

8. Workshop conclusions

The significance of rapidly and correctly conducted oil sampling and oil fingerprinting during the response to an oil spill was recognised and the important work and high level of expertise of the OSINet group of experts were acknowledged.

The workshop concluded that:

- National practices in oil spill sampling, sample analysis and oil identification across Europe vary extensively; it is important to maintain a forum at EU level, with EMSA's support, to regularly address these important issues (e.g. via dedicated workshops) and share best practices.
- The need for the provision of training on oil spill sampling was identified;
- The usefulness of developing practical guidelines on oil spill sampling and oil identification based on the exchange of existing knowledge and expertise among Member States and in particular based on the experience of the OSINet group of experts was recognised.

The chairman thanked the participants and speakers for their high level of interaction and contribution to the workshop discussions and ended the workshop.

The outcome of this workshop will be presented to the 10th meeting of the CTG MPPR and this workshop report will be published on EMSA's website.

Appendix A List of Annexes

Annex 1	Workshop Agenda
Annex 2	Workshop Document
Annex 3	Questionnaire
Annex 4	List of Participants

Group Picture



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Workshop Agenda: Oil fingerprinting and the work of OSINet

EMSA offices, Lisbon, 02 March 2016 , Room 01/11

Wednesday, 02 March 2016

Time	Agenda Item	Speakers
09:00 – 09:30	Arrival & Registration of Participants	
09:30 – 09:40	Welcome	EMSA
09:40 – 10:40	Session 1 – Setting the scene Workshop objectives Brief roundtable introduction of participants Results of the Questionnaire <i>Flinterstar</i> : an actual case	Chairs: Eric Donnay & Walter Nordhausen Uta Kraus Eric Donnay
10:40 - 11:40	Session 2 - Oil spill identification and fingerprinting, oil sampling, OSINet OSINet network of experts Introduction to oil spill identification and sampling, and the basics of the analytical method COSIweb & oil cases within COSIweb	Paul Kienhuis Paul Kienhuis Uta Kraus
11:40 – 12:00	Group Photo & Coffee Break	
12:00 – 13:00	Session 3 – Real Cases Oil spill identification in Spain - SASEMAR and sampling - Oil spill cases in Spain <i>Flinterstar</i> case – Analytical results	Laura de la Torre Joan Albaiges Paul Kienhuis
13:00 – 14:00	Lunch Break	
14:00 - 15:30	Session 4 – Practical exercise Introduction to scenario: Oil spill in a harbour (10min) Break-out groups - 2 groups in smaller rooms to discuss the practical case (50 min) Plenary discussion of practical exercise outcome (30 min) - The two break-out groups to present the main points of their discussions in the groups, followed by discussion in plenary	Paul Kienhuis
15:30 – 15:50	Coffee Break	

Time	Agenda Item	Speakers
15:50 – 17:10	Session 5 – Enhanced cooperation in Europe Cooperation between national maritime response organisation and national laboratory (NL experience) Requirements for participation in OSINet Facilitated discussion in plenary – “ <i>How to enhance cooperation in oil spill identification across Europe</i> ”	Jan Kool Uta Kraus Eric Donnay & Uta Kraus
17:10 – 17:30	Conclusions & end of meeting	

Speakers / Facilitators:

- Eric Donnay, DG Environment, Marine Environment Service, Belgium
- Uta Kraus, Federal Maritime and Hydrographic Agency (BSH), Germany
- Paul Kienhuis, Rijkswaterstaat CIV, RWS Laboratory, The Netherlands
- Laura de la Torre, SASEMAR, Spain
- Joan Albaiges, CID-CSIC, Spain
- Jan Kool, Rijkswaterstaat, The Netherlands
- Walter Nordhausen, European Maritime Safety Agency (EMSA)
- Lito Xirotyri, European Maritime Safety Agency (EMSA)

International cooperation in oil spill identification: The work of OSINet

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1. Introduction and scope of the workshop

In recent years, intentional maritime oil pollution in Europe is declining due to international regulations and maritime surveillance. However, the probability of an accidental oil-spill incident in European seas is rising due to both an increase in seaborne traffic and the increase and expansion of offshore oil and gas activities resulting in a rising number of oil and gas installations (EMSA 2013; Bonn-Agreement 2014). While the average number of spills per year is declining, large quantities can be spilled in very few accidents. Accordingly, ITOPF (ITOPF Ltd 2015) reports that in the last six years (2010-2015), 86% of the spilled oil reported was originating from just 23% of the total number of incidents. Prominent European examples of significant spills during the last two decades are the accidents of the Tricolor (2002) and the Flinterstar (2015) in the North Sea and of the Prestige (2002) in the Atlantic Ocean off the Spanish coast. In case of large spills, international cooperation not only is important in direct response activities but is extremely helpful in regard of characterizing waterborne oils and the identification of possible sources. Since large-scale spills often reach across national borders, concerted analysis with comparable results is the most promising way to approach the issue of compensation claims. OSINet, the oil spill identification network of experts, has worked for this goal for more than ten

years, promoting exchange of best practice and expertise and developing a common oil identification method which nowadays is used worldwide.

The workshop hosted by EMSA on 2 March 2016 aims to demonstrate the work of OSINet to the EU Member States with the goal to achieve a better understanding on oil identification methods across Europe, and to widen cooperation between experts from national maritime emergency response organisations and/or national (forensic) laboratories for identification and fingerprinting of oil samples. This workshop document provides an overview and background of the work of OSINet for the experts participating at the workshop and aims to facilitate the discussions during the workshop.

2. Introduction to the Oil Identification Network (BONN-OSINet)

OSINet was initiated by the Bonn Agreement in 2005 after national laboratories had encountered difficulties in identifying oil spill sources from the *Tricolor* accident in 2002. On board there had been at least four different types of heavy fuel oil, in addition to marine diesel oil and lubricating oil. The complex, weathered oily mixtures that had escaped from the *Tricolor* wreck led to substantial difficulties during work on oil “fingerprinting”. This work was necessary for attempting to link the oil pollution to its source and to underpin governmental claims for reimbursement of costs by several countries. As a consequence, to enhance cooperation and the transfer of knowledge between different countries, the network of expert chemists specialized in oil spill identification techniques, now known as OSINet, was established (Bonn-Agreement 2014). Each Bonn Agreement country assigned at least one laboratory that is responsible for the analysis of the oil spill identification samples within OSINet. Today, OSINet has expanded beyond the Bonn Agreement region to become a worldwide group of experts with laboratories participating from Norway, Canada, USA, Brazil, China, Korea and Australia. Trustful, trans-boundary co-operation and the sharing of best practice and expertise builds professionalism in oil identification analysis, what provides a solid basis for a high level of preparedness in case of accidental spills by the possibility to defensibly identify polluters.

3. OSINet tools and achievements

While oil spill identification by chemical analysis is by no means the only tool available to law enforcement (Figure 1), it is an important, sometimes the most important instrument providing evidence in criminal proceedings. As such, it is crucial to carry it out in the highest and most reliable way possible.

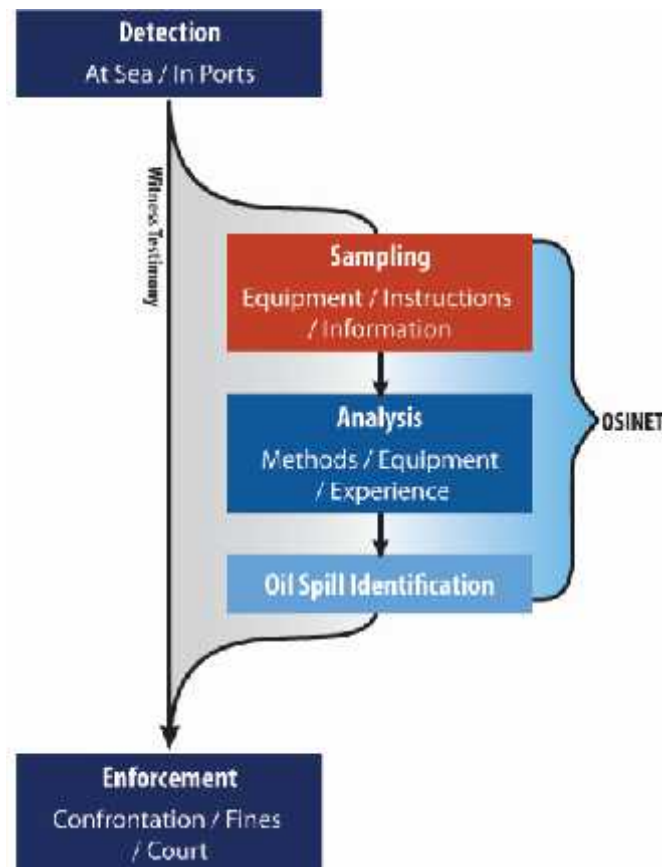


Figure 1: The contribution of OSINet in oil spill identification work for law enforcement (Bonn-Agreement 2014)

3.1 Certified oil identification methodology

Co-operation, mutual assistance and ultimately criminal prosecution are much easier to achieve when all participating laboratories analyse and compare oil samples using a **common method**. Furthermore, results achieved in this way are highly transparent, reproducible and objective (Dahlmann and Kienhuis 2015). Based on the NORDTEST method (NORDTEST 1991), the experts of OSINet developed a common methodology for oil identification work which was published as a CEN standard by the European Committee of Standardisation. Since sampling is a crucial part of reliable oil identification, sampling was also addressed in detail in the standard:

- Part 1 (European Committee for Standardisation 2006) describes sampling techniques and the handling of oil samples prior to their arrival at the forensic laboratory
- Part 2 (European Committee for Standardisation 2012) covers laboratory procedures of oil spill identification methodology, analytical techniques, data processing, data treatment, and interpretation and reporting of results.

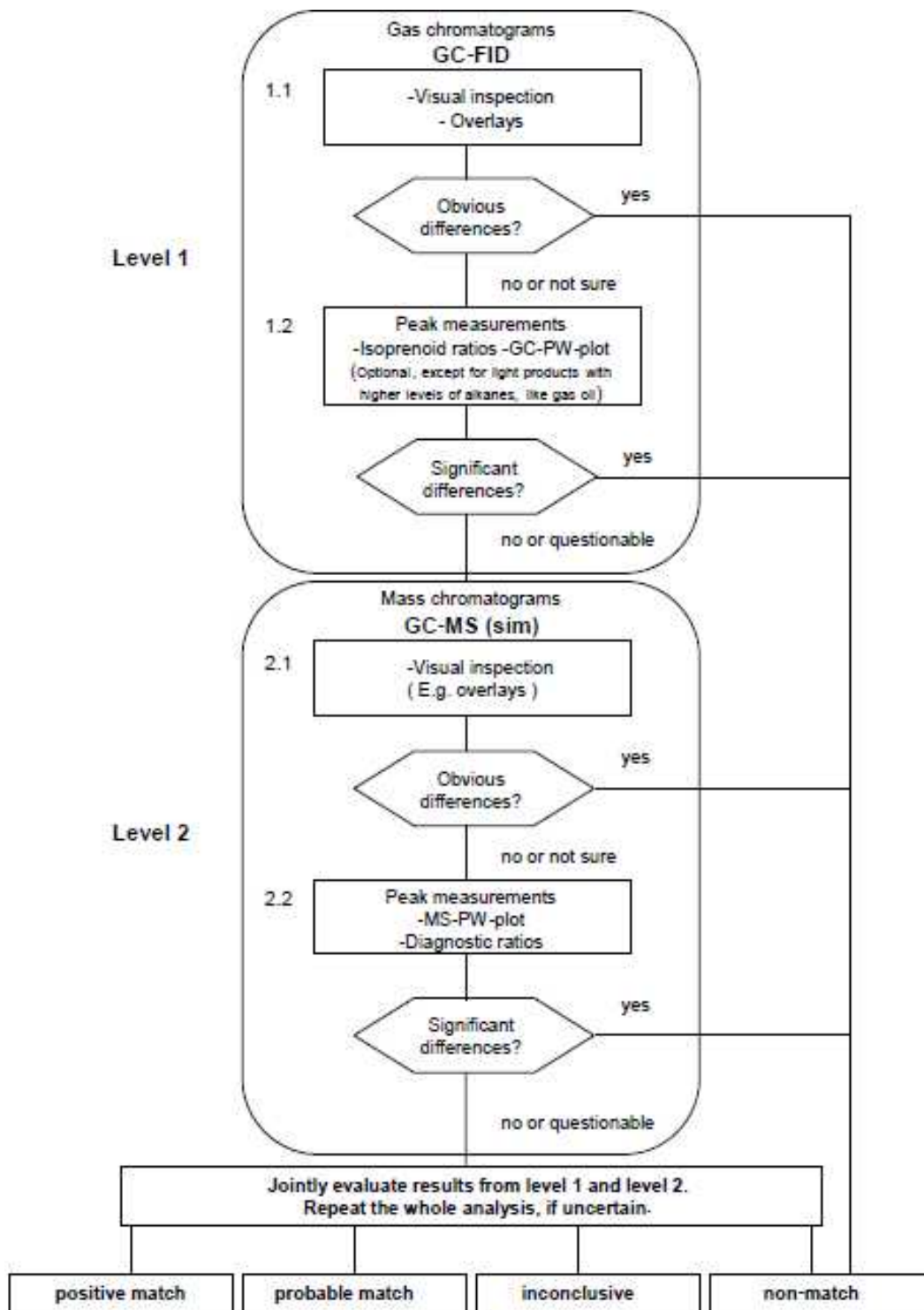


Figure 2: Flow chart of the steps of analysis (European Committee for Standardisation 2012)

3.1.1 Sampling

Collecting oil samples serves several different purposes (European Committee for Standardisation 2006). Always, it is important to initiate sampling before clean-up measures are started. Not only can analysis give important support information for the planning of response and clean-up work (Peperzak, Kienhuis et al. 2010), but for oil identification it is crucial to secure unbiased oil samples. All spills encountered and all potential sources of spills should be sampled. It is important to take samples from both spill and source even on occasions where it seems quite clear from where the spill originates. Reference locations, meaning sites without oil contamination that share environmental characteristics such as temperature and salinity with the contaminated site, have to be sampled as background samples. It is important to keep in mind that each oil spill incident is unique and actions have to be adapted to the current situation. Thus, CEN/TR 15522-1:2006 is a compilation of instructions and experiences from experts all over the world. It guides the user in sampling, storing and delivering oil samples for laboratory analysis.

3.1.2 Analysis

3.1.2.1 Gas Chromatography-Flame Ionization Detector (GC-FID)

The investigation of samples starts – after a clean-up step which depends on the sample and which is described in detail in CEN/TR 15522-2:2012 – with Gas Chromatography-Flame Ionization Detector (GC-FID) analysis. In a very short time, this fast analysis provides an overview regarding the oil type of the sample (Figure 3), the degree of weathering and the similarity between spill and suspected sources as well as the needed injection volume for more detailed analysis (if necessary). This quick analysis is also the basis for decisions regarding response actions (e.g. dispersant spraying) (Peperzak, Kienhuis et al. 2010).

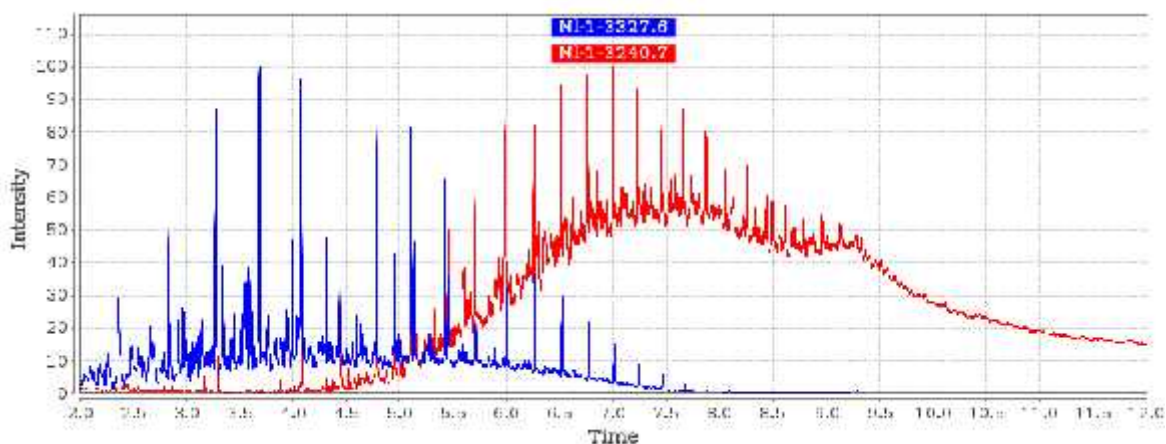


Figure 3: GC-FID chromatograms of a diesel (blue) and a heavy fuel oil sample (red) (screenshot from COSIweb, see 3.2 COSIweb database)

3.1.2.2 Gas Chromatography-Mass Spectrometry (GC-MS)

Especially when the spill sample must be compared with suspected source(s), often a more detailed analysis with Gas Chromatography-Mass Spectrometry (GC-MS) is needed. Based on the information of the GC-FID analysis (see 3.1.2.1 Gas Chromatography-Flame Ionization Detector (GC-FID)), samples are selected and, if necessary, adjusted in injection concentration. Samples are analysed in selected ion mode (SIM) to detect the presence of a particular array of compounds like various PAHs and biomarkers (Figure 4). The results of the GC-MS analysis are used to confirm the type of oil, based on the presence or absence of characteristic compounds in the samples. To reduce personal bias, CEN/TR 15522-2:2012 demands that the peak height or peak area of the compounds must be measured. From the peak areas, ratios must be calculated and compared between samples. The procedure assumes that the ratios can be analysed with a standard deviation of less than 5%. Rules to eliminate compounds influenced by weathering and small peaks with a higher standard deviation are given in CEN/TR 15522-2:2012. The relative difference between each ratio of a suspected source and the spill sample is based on the repeatability limit and must be equal or lower than 14%.

To account for changes in samples due to weathering processes, partial weathering plots (PW-plots) are used. In these plots, the concentration of the measured specific compounds (e.g. PAHs, biomarkers etc.) in the spill sample are plotted against the corresponding concentrations in the suspected source sample. Corresponding ratios are given in percentages, and if not affected by weathering processes, all values must theoretically fall on a straight 100% line. However, practical results have to take into account the repeatability of the analysis (Figure 5).

In case of weathering, characteristic changes in the PW-plots are to be found, as is shown in Figure 6 for evaporation. Volatile substances (with earlier retention times in GC-MS analysis) are absent or highly reduced in the PW-plot, while more stable compounds (some aromatics and typically biomarkers) are less or not at all reduced in the spill sample.

The above mentioned techniques allow the exact characterisation of the spill and possible source samples and ultimately lead to a final conclusion by the analyst. CEN/TR 15522-2:2012 provides guidance on how results and conclusions of the GC-FID and GC-MS analyses should be described in a report that can be used for judicial procedures against offenders. Therefore, the following final conclusion definitions are used: Positive match, probable match, inconclusive, non-match (see Figure 2).

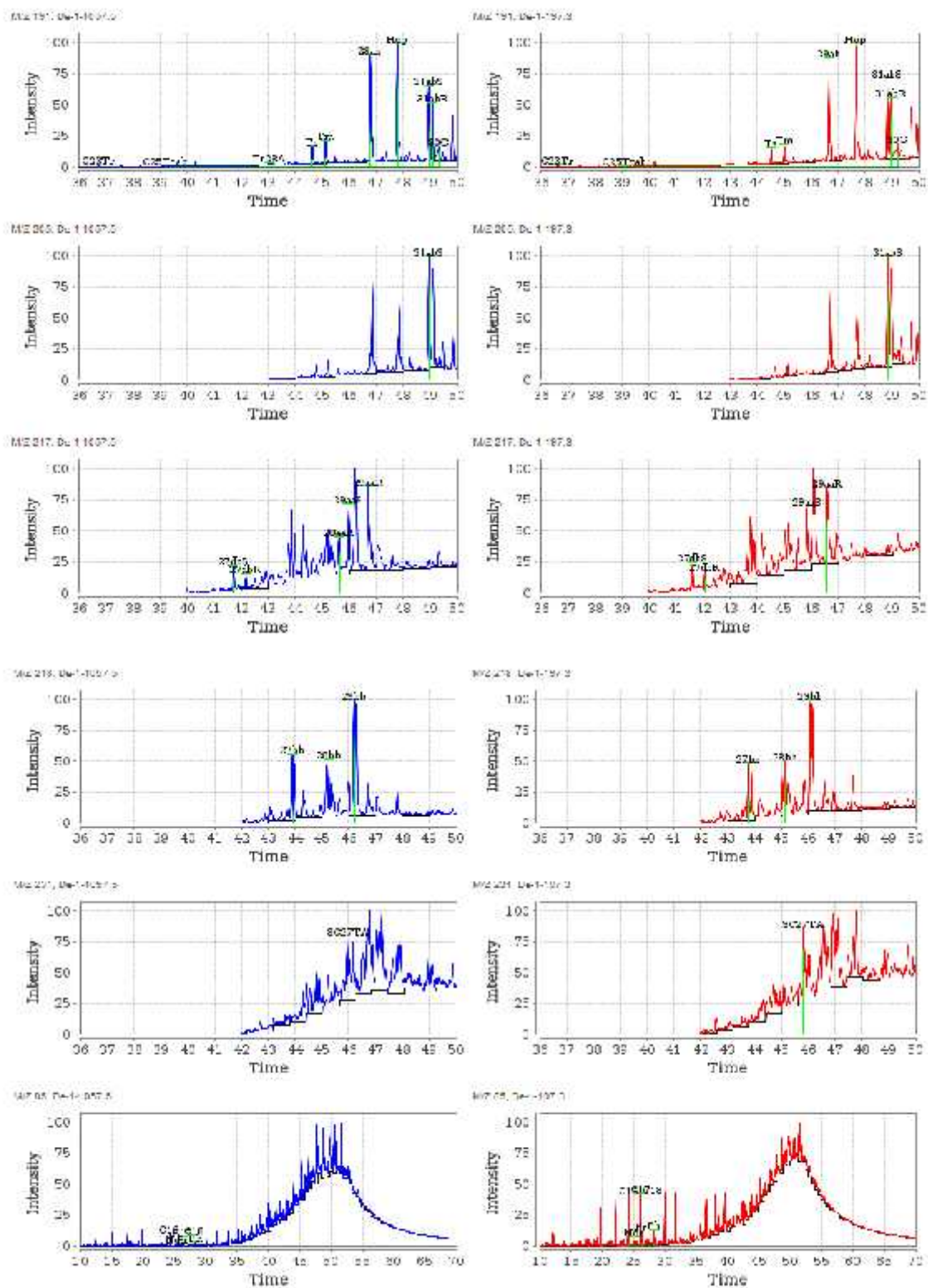


Figure 4: GC-MS chromatograms of characteristic ions for two samples (blue and red) containing both a mixture of heavy fuel oil and lubricating oil (screenshot from COSIweb, see 3.2 COSIweb database)

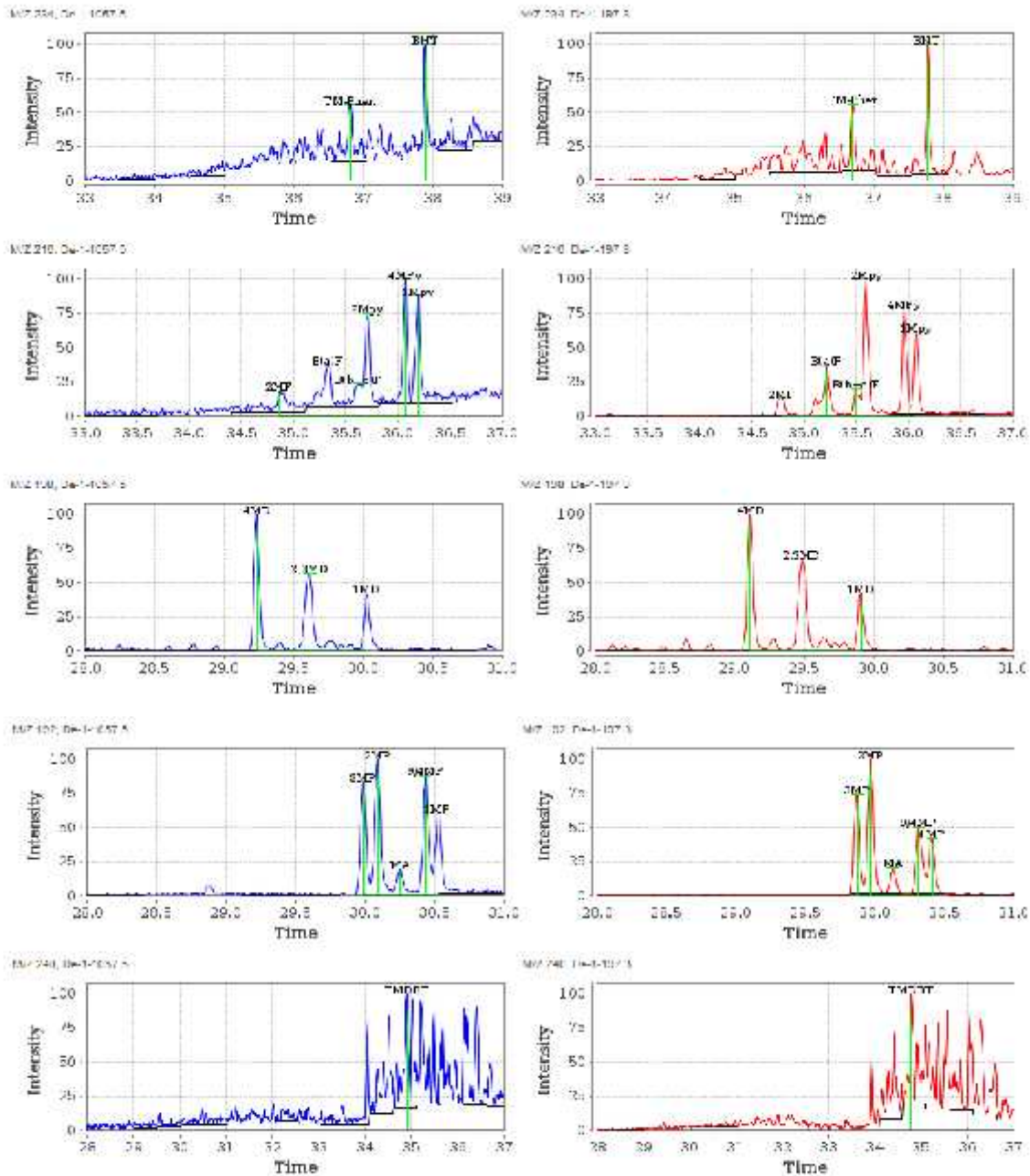


Figure 4 continued.

3.2 COSIweb database

OSINet’s work is supported by the **central, browser-based database** COSIweb which is hosted on a German server. COSIweb currently includes data of about 2800 analysed oil samples (among them 300 crude oils from all over the world) and provides information on the oil characteristics (oil ‘fingerprint’) used in CEN/TR 15522-2:2012. This allows users to rapidly compare data simultaneously with all the oils included in the database.

COSIweb has the following two main functions:

- a) searching for unknown samples in the database
- b) comparing two samples by automatically analysing the characteristic parameters for a statistical analysis of the sample and its comparison to the database within seconds.

When two samples are compared, correlation with all samples included in the database assures the uniqueness of the samples compared (Bonn-Agreement 2014).

Access to COSIweb is exclusively provided to members of the OSINet Network of Experts.

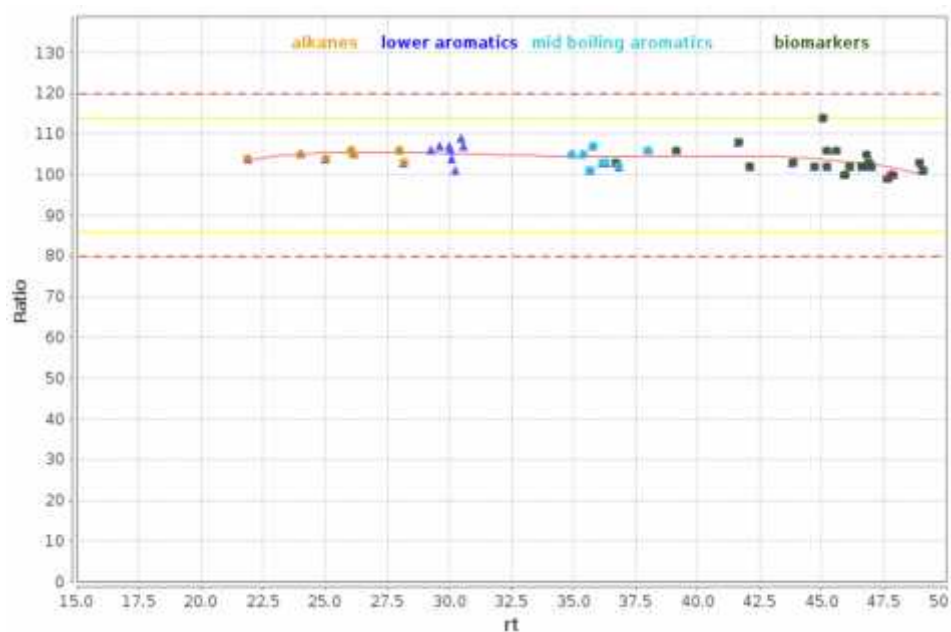


Figure 5: PW-plot of an un-weathered oil sample (screenshot from COSIweb, see 3.2 COSIweb database).

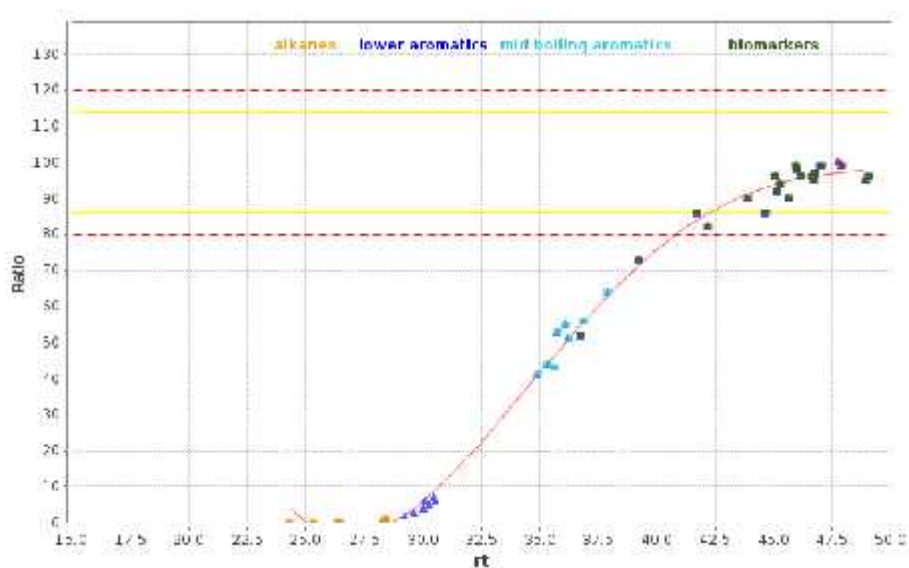


Figure 6: HFO evaporated/distilled at 400 degrees for 4 hours (comparison of sample 6 with sample 1 in Round Robin 2007, (Dahlmann and Kienhuis 2015)).

3.3 Annual intercalibration exercises (Round Robins) and meetings

Quality assurance in oil spill identification among the members of the OSINet network is achieved by **annual meetings** and **annual intercalibration exercises** (“Round Robins”). These tests are organized by the network on a self-financing base. Each time, the intercalibration exercises address different problems connected with oil identification by investigating different oil products (heavy fuel oil, (bio)diesel, crude oils etc.) and/or the results of different cleaning procedures (e.g. burning of oil on water or the use of dispersants). Samples of oil from real or fictitious cases are sent to the network’s laboratories for analysis with a request to return their results for evaluation. Thus, a high standard in quality assurance is assured among the network’s laboratories. At the same time, the intercalibration tests provide a possibility of a practical learning experience for newly participating laboratories, since one of OSINet’s main achievements is a collaborative approach with open transfer of knowledge within the group to improve the standards of all participating laboratories. Thus, detailed results of these tests are treated confidentially and are used as the base of discussions among the OSINet members, (i.e. the experts of the participating laboratories) during the annual meetings. However, anonymised summary reports of the RRs 2004-2013 are publicly available and can be found at: <http://www.bonnagreement.org/osinet/oil-spill-identification>.

OSINET at a glance:

- International network of expert chemists specialized in oil spill identification techniques
- Promotes cooperation; shares information and data & exchange of knowledge
- Provides mutual assistance and co-operation in difficult cases in oil spill identification and fingerprinting
- promotes quality assurance in oil spill identification; in particular by developing and using a certified oil identification methodology
- Organises annual intercalibration exercises and provides education in oil spill sampling
- organises annual meetings to evaluate the quality assurance work on oil spill identification and evaluates the results of ring-tests, involving the experts of the participating laboratories
- Shares access to a central comprehensive online database with data on 2800 analysed oil samples with information on the oils’ characteristics (oil ‘fingerprint’)

4. Enhancing cooperation in oil spill identification across Europe

While better technology makes seaborne ship traffic safer, the increase in both traffic and offshore activities provides new challenges in risk management and oil spill response preparedness. This was e.g. acknowledged by the change of EMSA's mandate regarding oil and gas installations in 2013 (EMSA 2013). However, as the primary responsibility to react to an incident always remains with the Member State concerned, additional ways for coordinated actions become more important. The OSINet network provides an excellent platform for this purpose. The Network is active in oil spill sampling; analysis; mutual assistance; and education in oil spill sampling.

4.1 Requirements for participation in OSINet

Participation in OSINet requires no 'membership fee'. It is open to all laboratories analysing oil spill samples provided that they participate in the annual intercalibration tests. This requirement is the main condition for being part of the OSINet network. OSINet is a collaborative network for sharing information, expertise and assistance. Only the participants who accept to share their experience through the network are entitled to benefit of the expertise available in OSINet. Most of the participants are government-related, e.g. forensic institutes and military laboratories, however, some commercial laboratories carry out oil identification analysis under governmental contract as well. OSINet works on a self-financing base, meaning that participants pay their own travel costs. Intercalibration tests and annual meeting are organised and executed by every OSINET member state in turn to share the corresponding costs.

4.2 The way forward - how to enhance cooperation in oil spill identification across Europe

Since its creation more than ten years ago, OSINet has continuously worked to build an international community of experts in oil spill identification. Through exchange of best practice and expertise, OSINet has developed a reliable standard oil identification method to help authorities proving the link between an oil spill sample and the source of the pollution. Today, this method is a recognised guidance reference, which has proven its value in several oil spill cases such as *Prestige*, *Tricolor* and more recently *Flinterstar* and is used by countries across the world. However, not all European Member States are yet involved in OSINet. By joining, the laboratories of these EU countries could gain from the knowledge pooled within the network, while at the same time added expertise would further strengthen the network, resulting in coordinated, reliable efforts in oil spill identification across Europe.

With all EU Member States participating in OSINet

- All participating laboratories can share best practices within an international community of experts who are facing the same challenges for oil spill identification
- All participating laboratories work with a common method
- All participating laboratories have common quality assurance in place
- All participating laboratories can exchange samples and analytical results
- ➔ Reimbursement in cases of large (cross-border) oil spills is drastically facilitated

If you have any question about OSINet, please do not hesitate to get in contact with the network and write to paul.kienhuis@rws.nl or uta.kraus@bsh.de.

Current members of OSINET:

EU members

Belgium
Bulgaria
Germany
Estonia
España
Finland
France
Ireland

Italy
Latvia
Monaco
Netherlands
Norway
Sweden
United Kingdom

Non-EU members

Australia
Brasil
Canada
China
Korea
USA
Malaysia

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Annex 3 - OSINet workshop Questionnaire (Lisbon, 2 March 2016)

To be returned to uta.kraus@bsh.de by 19 February 2016

Please tick the appropriate answers and add short explanations when requested.

* The information provided in this questionnaire will only be used for the workshop on 2 March 2016; the results from the questionnaire will be summarised and presented during the workshop

Country:

Name / Institution of representative filling in the questionnaire:

A) Organisation / national responsibility

1) How is oil spill response currently organized in your country?

centralised

decentralised

Comments (e.g. name of the organisers, participating institutes and authorities):

2) Does a specialized laboratory exist that handles all oil cases in your country?

Yes

No

If yes, please indicate the name of the laboratory:

3) Does the national response organisation make use of the expertise of a (national) laboratory in case of other than oil and/or unknown substances as well?

Yes

No

If yes, please elaborate (e.g. specify substances):

4) Who is responsible for the sampling in case of an oil spill?

B) Procedures

5) Is the sampling following an official/common/national guideline?

Yes

No

If yes, please indicate the guideline:

6) Is the analysis done according to a certain guidance document?

Yes

No

If yes, please provide further details:

7) How do you deal with "shared" oil samples (e.g. oil spills affecting several countries)?

8) Is there a plan of action in place for such cases as mentioned in question 6?

Yes

No

If yes, please elaborate:

9) How are the results communicated and to whom?

10) Does a centralized national data base for the oil sample analyses exist?

Yes

No

If yes, please provide further details:

C) Experience and number of cases

11) How many oil cases did you encounter in your country in the last 3 years/per year?

2013:

2014:

2015:

12) In how many of the above mentioned cases was oil identification investigation carried out by the responsible laboratory/laboratories?

2013:

2014:

2015:

13) Are these oil spill identification analyses used in court/by arbitration?

Yes

No

If yes, please provide further details:

D) General comments

14) Is there any other information you would like to include with regard to the scope of this workshop and this questionnaire?

OSINet Workshop

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4	Belgium	Koen	Parmentier	RBINS-OD-Nature	kparmentier@naturalsciences.be
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