ICS 13.020.40, 75.080 SLOVENSKÁ TECHNICKÁ NORMA August 2025

STN	**Identifikácia únikov oleja/ropy Ropné výrobky a príbuzné ropné výrobky Časť 2: Analytická metóda a interpretácia výsledkov založených na GC-FID a GC-MS analýze s nízkym rozlíšením**	**STN EN 15522-2+A1** 83 8290

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

Táto norma obsahuje anglickú verziu európskej normy.
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 07/25

Obsahuje: EN 15522-2:2023+A1:2025

Oznámením tejto normy sa ruší
STN EN 15522-2 (83 8290) z júla 2023

obsahuje
farebné
strany

140709

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15522-2:2023+A1

April 2025

ICS 13.020.40; 75.080

Supersedes EN 15522-2:2023

English Version

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

Identification des pollutions pétrolières - Pétrole et produits pétroliers - Partie 2 : Méthode d'analyse et interprétation des résultats sur la base des analyses par CPG-DIF et CPG-SM faible résolution

Identifizierung von Ölverschmutzungen - Mineralöl und verwandte Produkte - Teil 2: Analytische Methodik und Interpretation der Ergebnisse, basierend auf GC-FID- und GC-MS-Analysen bei niedriger Auflösung

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. EN 15522-2:2023+A1:2025 E

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European foreword

This document (EN 15522-2:2023+A1:2025) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2025, and conflicting national standards shall be withdrawn at the latest by October 2025.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document includes Amendment 1 approved by CEN on 2 March 2025.

This document supersedes A1⟩ EN 15522-2:2023 ⟨A1.

The start and finish of text introduced or altered by amendment is indicated in the text by tags A1⟩ ⟨A1.

In comparison with the previous edition CEN/TR 15522-2:2012, the following technical modifications have been made:

- adding compounds to be analysed in order to include light products in the diesel range;
- adding more information about biodegradation;
- adding a Reporting and a Quality assurance chapter;
- adding Annex C with precision data;
- adding Annex D with likelihood grade conclusions;
- introduction of characterization of FAME in Annex I;
- serious revision of Annexes H, I, J and K, adding new pictures and chromatograms.

EN 15522 is composed of two parts that describe the following:

- Part 1 on sampling, describing good sampling practice, detailing sampling equipment, sampling techniques and the handling of oil samples prior to their arrival at the forensic laboratory;
- Part 2 giving the analytical method, which covers the general concepts and laboratory procedures of oil spill identification, analytical techniques, data processing, data treatment, interpretation/evaluation and reporting of results.

This document has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

A list of all parts in a series can be found on the CEN website.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

Introduction

This document describes a forensic method for characterizing and identifying the source of oil spills in the environment resulting from accidents or intentional discharges. By following this method, data generated can be used as evidence in support of the legal process. This method is based on the experience gained from use of the earlier revisions.

This document is composed of two parts that describe the following:

- Part 1 on sampling, describing good sampling practice, detailing sampling equipment, sampling techniques and the handling of oil samples prior to their arrival at the forensic laboratory;
- Part 2 giving the analytical method, which covers the general concepts and laboratory procedures of oil spill identification, analytical techniques, data processing, data treatment, interpretation/evaluation and reporting of results.

Oil spill source identification is a complex process due to the large variation in sample types and oil spill situations that can be encountered. Part 1 is a compilation of instructions and experiences from experts all over the world which will guide the user in sampling, storing and delivering oil samples for laboratory analysis. Part 2 will guide the reader through the analytical process. It prescribes how to prepare and analyse oil samples using Gas Chromatography with Flame Ionization Detection (GC-FID) and GC with low-resolution Mass Spectrometry (GC-MS). Any compositional difference found between samples is only relevant if this difference is larger than the variability of the method itself. Good analytical performance and strict quality assurance are therefore essential. In the Annexes of Part 2, relevant information concerning different types of oil and oil comparison techniques are presented.

In a typical standard method for testing, instructions are given on performing an “analytical” procedure. However, oil spill identification comprises both analytical and assessment components. Sample preparation is described in Chapter 6. Analytical methodology for GC-FID is provided in Annex A, while GC-MS is covered in Annexes B, E and F. Other parts of the document describe how to assess the analytical data utilizing various tools to draw a conclusion. As every case can differ in situation, size, products and weathering, the evaluation part of the method is described as a toolbox. Annexes J and K provide example documents for an oil spill case and show how the assessment tools may be applied. Further examples of specific oil spill cases are available as summary reports of the annual round robins (RR-tests) organized by Bonn-OSINet [11] and in literature.

The purpose of this document is to assist the reader in defensibly identifying the source of an oil spill by comparing the chemical composition of spill samples against suspected source oils. The basis for this method is the widely variable chemical compositions of oils, which allows oils from different sources to be distinguished by analytical techniques. The method relies upon detailed chemical characterization and statistical comparison between samples (i.e. a spilled oil and a suspected source) diagnostic features in order to determine whether they “match”. To minimize the danger of “false positive matches”, good laboratory practices are to be maintained. A “positive match” between a spilled oil and suspected source sample may not be sufficient to identify the PRP (potential responsible party) on its own. However, this result can be critical evidence in proving a case within the legal process.

It should be noted that oil spill identification methodologies have limitations and may not necessarily lead to unequivocal conclusions. In certain cases, neither the oil spill nor suspected source(s) are unique or homogeneous in nature, e.g. due to the changing/variable nature of oil in bilge tanks or due to mixing of oils spilled from several sources. For such cases there is a risk that the chemical composition of the available spill and source samples may not match. Therefore, successfully identifying a spilled oil’s source depends upon the samples available for analysis. To minimize the danger for “false positive” and in particular “false non-matches”, good sampling practice is essential (as described in Part 1: Sampling).

When no source oil is available, the methodology within this document can be used to determine the spilled oil type and selected characteristics. The characterization of a spilled oil sample can still be useful for several reasons:

- Rapid identification of a spilled oil type can help authorities track down potential sources of a "mystery" spill before the culprit has left the area (e.g. ship) or removed additional evidence. The mere differentiation between unused refined petroleum products (e.g. diesel fuel, heavy fuel oil), crude oil or waste oil (e.g. bilge residues, sludge, slops) can provide potentially valuable information as the possible source(s);
- In some court trials, the differentiation between pure refined products and waste oil may be very important because it allows conclusions to be drawn regarding the cause of an oil discharge, e.g. technical failure, accidental discharge, intentional discharge;
- In some countries, aerial photography of a plume behind a ship, combined with evidence the plume contained mineral oil, has been enough for a conviction;
- Finally, characterisation of the spilled oil provides a baseline against which future impacts to the affected area/environment might be compared.

This document is the result of advancements in the field of oil spill identification [e.g. 22, 36, 66, 67, 78 and 85] that have been made since the Nordtest Method [54] was first introduced in 1991. These have included:

- advancements in analytical methodologies;
- improved understanding of the specific chemical compositions and diagnostic features of oils;
- improved understanding of how an oil's composition changes in the environment (e.g. due to weathering);
- improvements in the statistical and numerical analysis of chemical data.

These advancements have been made by researchers around the world and documented in a wide range of peer-reviewed literature. In addition, numerous Proficiency Testing Programs (PTPs. or round robin tests (RR)) have been conducted to evaluate and improve upon the methodology. Since 2004, in the framework of Bonn-OSINET (Bonn Agreement oil spill identification network of experts), annual interlaboratory studies have been organized jointly by RWS-lab (Rijkswaterstaat - Laboratory in the Netherlands) and BSH (Bundesamt für Seeschifffahrt und Hydrographie in Germany) in which laboratories from around the world participate. The studies have covered oil spill cases dealing with light fuel oil distillates (diesel oils), bilge water samples (a mixture of water, gas oils and lubricating oil), crude oils and heavy fuel oils. Findings from these studies have been discussed at annual meetings by the participating analysts and have been taken into account for refining the suggested methodology described herein. The final reports of the interlaboratory studies can be downloaded for free from the Bonn-OSINET part of the Bonn-Agreement website [11].

1 Scope

This document specifies a method to identify and compare the compositional characteristics of oil samples. Specifically, it describes the detailed analytical and data processing methods for identifying the characteristics of spill samples and establishing their correlation to suspected source oils. Even when samples or data from suspected sources are not available for comparison, establishing the specific nature (e.g. refined petroleum, crude oil, waste oil, etc.) of the spilled oil still helps to constrain the possible source(s).

This methodology is restricted to petroleum related products containing a significant proportion of hydrocarbon-components with a boiling point above 150 °C. Examples are: crude oils, higher boiling condensates, diesel oils, residual bunker or heavy fuel oils, lubricants, [A1⟩ sludge samples and mixtures of bilge, ⟨A1] as well as distillate fuels and blends. While the specific analytical methods are perhaps not appropriate for lower boiling oils (e.g. kerosene, jet fuel, or gasoline), the general concepts described in this methodology, i.e. statistical comparison of weathering-resistant diagnostic ratios, are applicable in spills involving these kinds of oils.

Paraffin products (e.g. waxes, etc.) are outside the scope of this method because too many compounds are removed during the production process [37] to correctly distinguish them from each other. However, the method can be used to identify the type of product involved.

Although not directly intended for identifying oil recovered from groundwater, vegetation, wildlife/tissues, soil, or sediment matrices, they are not precluded. However, caution is needed as extractable compounds can be present in these matrices that alter and/or contribute additional compounds compared to the source sample. If unrecognized, the contribution from the matrix can lead to false “non-matches”. It is therefore advisable to analyse background sample(s) of the matrix that appear unoiled.

When analysing “non-oil” matrices [A1⟩ (e.g. contaminated feathers, plants, rocks, sand) ⟨A1] additional sample preparation (e.g. clean-up) is often required prior to analysis and the extent to which the matrix affects the correlation achieved is to be considered. Whether the method is applicable for a specific matrix depends upon the oil concentration compared to the “matrix concentration”. In matrices containing high concentrations of oil, a positive match can still be concluded. In matrices containing lower concentrations of oil, a false “non-match” or an “inconclusive match” can result from matrix effects. Evaluation of possible matrix effects is beyond the scope of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15522-1, *Oil spill identification — Petroleum and petroleum products — Part 1: Sampling*

ISO 1998-1:1998, *Petroleum industry — Terminology — Part 1: Raw materials and products*

ISO 16703:2004, *Soil quality — Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography*