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Evaluation of the Performance of the Short-Listed Candidate Markers Regarding the Technical Requirements

Call for expression of interest to present products suitable for use as a marker in gas oils and kerosene

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Contents

Acronyms, abbreviations and explanations (not exhaustive)

CM	Candidate marker
CEI	Call for expression of interest to present products suitable for use as a marker in gas oils and kerosene
DAD	Diode Array Detector (normally in the UV)
JRC	Joint Research Centre
DG TAXUD	Directorate-General for Taxation and Customs Union
FID	Flame Ionisation Detector
GC	Gas Chromatography
HPLC	High Performance Liquid Chromatography
LC	Liquid Chromatography
M	mol / litre
m / m	mass / mass
MDGC	Multi-dimensional Gas Chromatography
MS	Mass Spectrometry
MSDS	Material Safety Data Sheet
n	Number of samples
RSD	Relative standard deviation
SCHEER	Scientific Committee on Health, Environmental and Emerging Risks
SD	Standard deviation
SY124	Solvent Yellow 124
UV	Ultra Violet (part of the electromagnetic spectrum)
v / v	volume / volume

Foreword

In 2001, the European Commission adopted the Euromarker (SY124) as a common fiscal marker to mark fuel to be sold with a lower tax in sectors such as agriculture, for marine use and for domestic heating. Unfortunately, SY124 is easy to remove or destroy which has made wide-spread fraud possible. To support DG TAXUDs policies to find a better fiscal marker, JRC has performed different kinds of tests to check the resilience of four candidate markers alongside with SY124. Based on these experiments one of the candidate markers outperforms the others and is resilient to most treatments.

Acknowledgements

Thanks are due to processing staff at JRC-Geel for performing most of the experiments. An external Customs Laboratory is gratefully acknowledged for helping with the analyses of two of the markers.

Abstract

The European Commission adopted the Euromarker (SY124) in 2001 as a common pan-European fiscal marker to label gas oil and kerosene. In parallel, several other marking systems based on other dyes and markers were already in use in different member states. Generally the use of a marker makes it possible to sell fuel with a lower tax for use in dedicated sectors like agriculture, marine use and for domestic heating. In contrast, non-marked fuels are normally fully taxed and intended for road transport. Unfortunately SY124 is easy to remove or destroy which has made wide-spread fraud possible. This has resulted in substantial losses of tax revenue and a number of member states have repeatedly notified the European Commission of this problem. Some member states currently invest considerable amounts of resources and effort in tracking illegal use of laundered fuel by performing >10,000 measurements per year of SY124 in gasoil and diesel. Other member states have recently adopted a new national marker to prevent this kind of fraud. To improve the situation on a European level DG TAXUD published an open call in order to find a new marker that could potentially replace SY124 as Euromarker. To support DG TAXUDs policies to find a better fiscal marker, JRC has performed different kinds of tests resulting in more than 1200 samples for checking resilience of four candidate markers alongside with SY124. Generally the tests involved laundering over different adsorbents, chemical break-down and different physical treatments. All new candidate markers are colourless but road-side detection of the candidate markers is possible and based on dedicated instrumentation. Based on these experiments one candidate marker outperforms the others and is resilient to most treatments. This candidate marker could potentially be used to replace SY124.

1 Introduction

In the European Union, gas oil and kerosene, which are exempt from excise duty or taxed at a rate other than the one applied to fuels used as propellants, need to be tagged with a fiscal marker (Council Directive 95/60/EC). Currently, Solvent Yellow 124, N-ethyl-N-[2-(1-isobutoxyethoxy)ethyl]-4-(phenylazo)aniline, (CAS number 34432-92-3), the so-called Euromarker, is used as a standard marker in all Member States on the basis of a Commission Implementing Decision ([1]). This Decision is subject to regular revision and the latest one needed to be carried out by the end of 2016 taking into account technical developments and fraudulent activity regarding the gas oil markers. Also, the suitability of Solvent Yellow 124, as the Euromarker, was to be evaluated.

At the beginning of the last revision period, the European Commission services were alerted of an increase in the fraudulent activities related to the removal of the Euromarker from marked gas oil and kerosene. Laundering of the Euromarker not only result in loss of revenue in the affected Member States but causes severe local environmental impact due to the illegal dumping of toxic waste generated by removal activities.

In September 2015, the Directorate General for Taxation and Customs Union, DG TAXUD, launched a Call for Expression of Interest to present new products suitable for use as a marker in gas oils and kerosene (CEI) (EU Official Journal No C299 of 11 September 2015) ([2]). The CEI was structured in two main stages: in Phase I, the new substances proposed by the applicants would be evaluated based on documentary evidence, and in Phase II, the short-listed candidate markers would be empirically evaluated using laboratory experiments. Six applicants came forward with eight candidate markers.

In Phase I, an administrative/legal, environmental and technical evaluation of the presented substances was performed by DG TAXUD, DG ENVIRONMENT and JRC based on the documentation provided by the applicants. Four candidate markers fulfilling all the criteria laid down in the CEI were short-listed for further evaluation. The short-listed candidate markers had been selected after an initial test phase where all the candidate markers were evaluated after an adsorption test on silica and checking of documentary evidence as concerns the validated analytical methods used for their determination in gas oil. The adsorption experiments were performed by the tenderers using the same

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([1]) The first Decision appointing SY124 as the common fiscal marker was adopted in 2001 (Commission Decision 2001/574/EC). The use of SY124 has been prolonged several times. It had to be prolonged in 2016 (Commission Implementing Decision 2017/74/EU) to avoid legal vacuum, before the current evaluation has been completed.

([2]) The text of the CEI is available on the website of DG TAXUD: http://ec.europa.eu/taxation_customs/sites/taxation/files/resources/documents/taxation/excise_duties/energy_products/aircraft_fuel/call_for_expression_of_interest.pdf silica adsorbent and following instructions provided by the JRC. Four markers were discarded for various reasons: e.g. incorrect performance of the experiments for removal of the markers with silica, or insufficient information about the analytical method for detecting the marker, etc.

In Phase II, the suitability of the short-listed candidate markers was evaluated against the technical criteria laid down in the CEI in a set of laboratory tests. The empirical results obtained in those tests were used to compare the performance of the candidate markers against the Euromarker, Solvent Yellow 124, which was subjected to the same series of tests. JRC performed these experiments and two external analytical laboratories measured the resulting 1260 samples.

This report focusses on the technical evaluation of the performance of the short-listed candidate markers in Phase II of the CEI. The planning and execution of the laboratory experiments and obtained results are described, and the evaluation of each candidate marker and its performance compared to that of the SY124 is presented. Based on these results, the conclusions of the study can be drawn and the best performing candidate marker system is proposed.

2 Evaluation criteria and planning

2.1 Evaluated candidate markers and fuel matrices

Four candidate markers fulfilling all the criteria listed in the CEI were consequently short-listed in Phase I for further technical evaluation. Those candidate markers were compared with the current Euromarker, SY124 in a series of tests. To establish an objective basis for comparison, SY124 was considered as an additional candidate marker (CM5) and subjected to the same performance tests as the four short-listed candidate markers. All new candidate markers are colourless. Hence without measurement it is not possible to say if a fuel is marked or not.

Marked kerosene has not been evaluated although this fuel is covered by the legislation. According to available information the quantities of kerosene that are being marked are much smaller compared to gas oil. In addition, jet fuel is not subject to the marking requirements in EU legislation, so there is limited risk of fiscal losses as concerns kerosene.

Hence the costs for testing both kerosene and gas oil would be disproportionate. For this reason only gas oil was selected as test matrix where substantial fraud already has been reported by some Member States.

2.2. Workflow Phase II

The timeline and workflow undertaken in Phase II of the CEI are summarised in Figure 1.

Phase I of the CEI was finalised in June 2016 with the selection of 4 candidate markers and an official notification to the applicants regarding the short-listed substances.

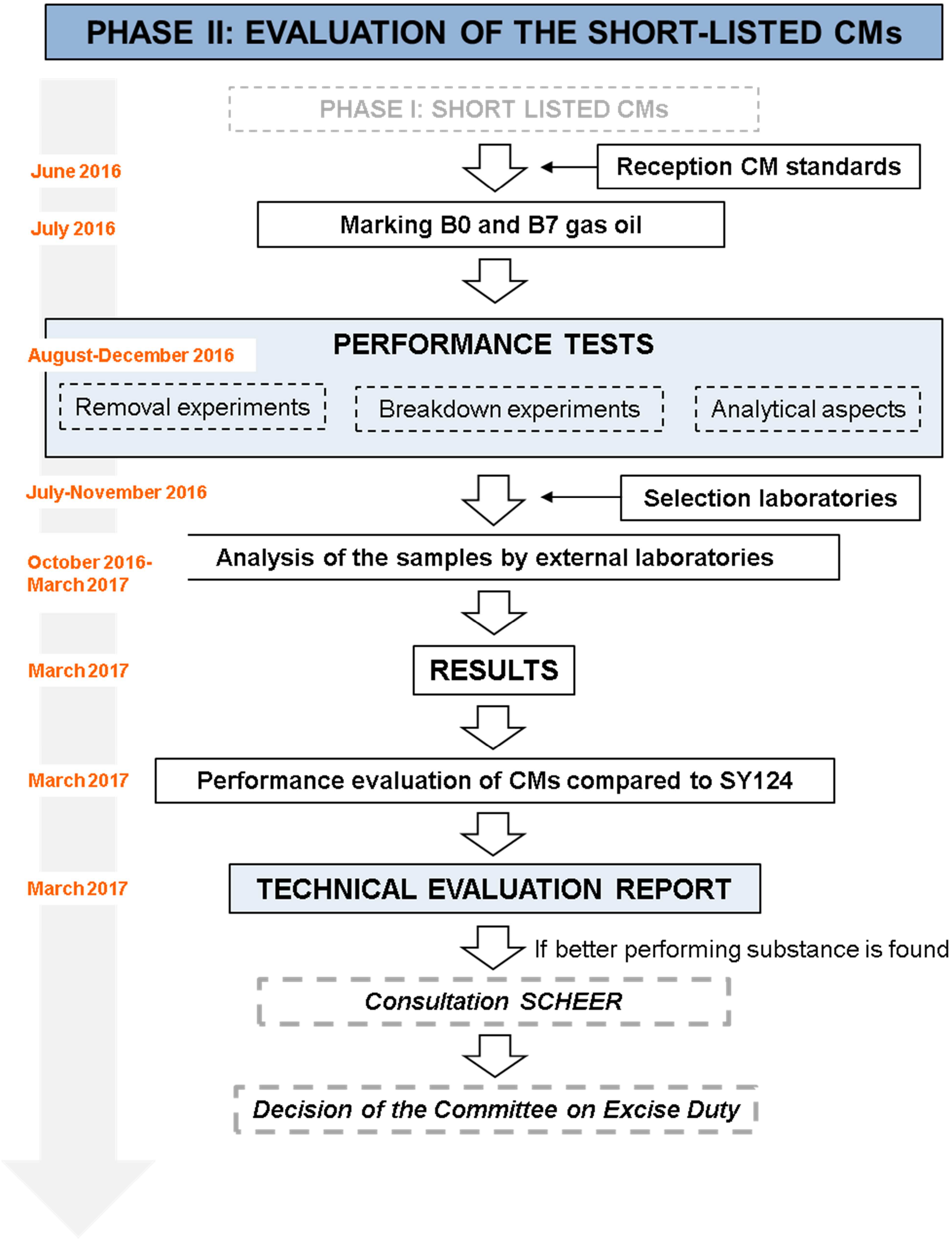
As foreseen in the CEI, DG TAXUD requested the successful applicants to send 10 g of the candidate markers by the end of June 2016 to JRC in order to conduct the performance tests. These candidate markers were used for two purposes:

- To mark B0 and B7 gas oil with the different candidate markers according to the marking levels proposed by the suppliers. B0 and B7 gas oil (according to EN 590 for the B0 and B7 and EN 14214 for the biodiesel part in B7) was selected as the test matrix in all the performance tests carried out by JRC.
- To be used as standards by the selected external laboratories during the implementation of the analytical methods and analysis of the samples resulting from the performance tests.

To this end, ten different solutions of marked gas oil were prepared, two solutions per candidate marker (five markers in two matrices i.e. B0 and B7 gas oil). In using these solutions, the performance tests were carried out between August and December 2016.

In parallel to the performance tests, laboratories for the analysis of the resulting samples were identified and selected. By the end of January 2017, all the samples from the performance tests had been shipped to the corresponding laboratories. The results were received at the end of March 2017. The performance of each candidate markers in the tests was compared to the one of the SY124, and their suitability as a new standard fiscal marker was evaluated. Depending on the results of the technical evaluation report and should a better performing substance be found, the Scientific Committee on Health, Environmental and Emerging Risks will be first consulted and in the last instance, the Committee on Excise Duty will vote on a Commission proposal for an implementing decision designating a new marker.

Figure 1. Outline of the timeline and workflow of the tasks carried out by DG-JRC in Phase II of the CEI.



2.3 Technical requirements for the Call for Expression of Interest

The objective of Phase II of the CEI was to test the performance of the short-listed candidate markers compared to the one of the current Euromarker, Solvent Yellow 124, against a pre-established set of technical criteria. These criteria evaluate the resilience of the substances to be removed from the marked gas oil and analytical aspects that could affect the detection of the new marker in use in the presence of already existing markers.

Originally, seven technical criteria, divided into three different categories were to be evaluated:

A. Removal tests: involving techniques that aim to entirely separate or partially separate the markers from the gas oil and/or kerosene matrices. To this end, laundering of the marked fuels using adsorbents and removal by a physical process was tested.

B. Breakdown tests: the marker is decomposed under extreme conditions with chemical or biological agents and a physical process, among others. The degradation products resulting from these reactions remain in the gas oil/kerosene, but they often have completely different structure than the original compound and, therefore, neither fulfil the same function nor are likely to be detected by analytical measurements targeting the original compound.

The different tests listed in the laundering and breakdown sections above are based on the methods reported by the Member States, which are commonly used in the illegal removal activities of the national markers and the Euromarker.

C. Analytical aspects: Several considerations related to the analysis of marked gas oil samples were also evaluated. Today, national markers or dyes, together with the Euromarker, are still an important part of the gas oil and kerosene tagging system in many Member States. Therefore, it would be desirable for a new standard fiscal marker to be measured in the presence of national markers that are still in use.

It is also necessary to be able to detect minimum amounts of the common marker in 'laundered' gas oil during routine roadside controls, as is now the case for SY124. For this reason, the proposed roadside tests should have the potential to detect the markers at 2 % of the initial concentration.

A few modifications to the initial technical requirements were deemed necessary. Some aspects, i.e. interferences with other dyes and markers were slightly modified while other tests, i.e. the bacterial breakdown tests, were completely removed from the technical requirement list because of the high technical complexity.

2.3.1 Modification from the Call for Expression of Interest

For logistical and technical reasons, some adaptations were made in the original list of performance tests in comparison with the call for expression of interest. These changes do not alter the overall set up dramatically; in fact, the only test that was abandoned was to test for possible effects on the markers from bacterial activity.

C1. Interferences with other dyes and markers

In the CEI, a list with ten national markers used together with the Euromarker was provided, amended by the corrigendum of 26 November 2015, where two extra dyes with CAS numbers 128-80-3 and 97862-23-2, used in Italy, were added.

During the acquisition of the dyes by JRC during the planning stage, the dye with CAS number 128-95-0, also known as Krisolamine, was found to be used for activities other than marking of gas oil and kerosene. Likewise, the dye with CAS number 71819-51-7 (Solvent Red 164), used to mark the gas oil in Poland, together with Solvent Red 19, was reported not to be in use anymore at the time of the consultation due to a shortage of supply in the market (information provided by the Ministry of Finance of the Republic of Poland to DG TAXUD, 25 February 2016). Consequently, after a consultation with DG TAXUD, it was decided to remove these two dyes from the list for the interference experiment.

2.3.2 Number of samples

JRC prepared a total of 1 260 samples as a result of the performance tests (Table 1). This figure was reached considering the following conditions for each test:

- Five markers were evaluated: four short-listed candidate markers plus SY124 (CM5) which was tested just as another candidate marker to realistically compare the performance of all the proposed marking systems.
- Five independent replicates per candidate marker were prepared in each experiment to achieve sufficient statistical significance to be potentially able to discriminate between two markers that perform similarly, provided that the method of measurement has sufficient precision.
- Two types of gas oil were evaluated: first purely petrogenic gas oil, B0 gas oil (straight run distillate, containing 0 % of biodiesel fulfilling EN 590) and B7 gas oil, (containing 7 % of rapeseed fatty acid methyl ester as biodiesel, fulfilling EN 14214).
- Five blanks per experiment and gas oil type were prepared. The blanks were used to check for accidental cross-contamination during the experiments.

- Five reference samples were prepared per type of experiment and candidate marker. The reference samples were marked, gas oil samples without treatment that allow a direct comparison with the treated samples. These samples were also used to assess the precision of the analytical methods. All reference samples were taken simultaneously when the experiment from the same marked gas oil solution was performed and immediately filled in discrete sample units. These samples provided additional information regarding the stability of the candidate markers during the testing period.

2.4 Modification of scoring of markers

In the CEI, it was described that a full score should be given to the best performing marker after which the results be ranked in descending order based on the recoveries obtained. In general, this is a sensible approach but with the available data, it was not an appropriate approach for two main reasons.

1. How to handle results that are well above 100 % recovery which are theoretically not possible although common in practice due to analytical variation?

2. How to deal with results from two different analytical methods with rather different precision (and measurement uncertainty) and comparing such results fairly?

During the phase of identifying external laboratories, it was requested by DG TAXUD **not** to change the analytical methods in any way, for example, with the objective to improve the precision and reduce measurement uncertainty. An alternative scoring approach not discriminating to any supplier has therefore been used and is more forgiving to recovery results, which are essentially high but have a relatively high variability because of relatively poor measurement precision. To understand the scoring approach, it is also worth mentioning that the expanded measurement uncertainty estimated by the external laboratories was ± 20 % for some of the markers. Hence, the approach was as follows: All recovery results above 80 % have been given a full score according to Table 2. Subsequently, all recovery results below 80 % have been given a score of zero points. The reason for choosing 80 % is that a 100 % recovery minus 20 % measurement uncertainty equals 80 % recovery. This is a simplified approach, but all candidate markers are treated in the same way. Very rarely results of just below 80 % recovery were obtained, which can be an issue for discussion. Fortunately, most of the results are clear *i.e.* either recovery is high and consistent, or very low, which is indicative of an effect the treatment have had on the marker. Some results require further discussion and considerations. More details about such results and particular considerations can be found in section 4.2 and under section 5.

Table 1. The total number of samples in the technical evaluation experiments. M: number of candidate markers; Rep: number of replicates; Ref S.: reference samples; Total S.: total number of samples.

	Test	Test	M	Rep.	Fuel types	Samples	Blanks	Ref S.	Total S.
REMOVAL TESTS	A.1-5. Adsorption	A.1	5	5	2	50	10	50	350
		A.2	5	5	2	50	10		
		A.3	5	5	2	50	10		
		A.4	5	5	2	50	10		
		A.5	5	5	2	50	10		
	B.1. Physical treatment	B.1	5	5	2	50	10	50	110
BREAKDOWN TESTS	C.1-4	C.1	5	5	2	50	10	50	290
		C.2	5	5	2	50	10		
		C.3	5	5	2	50	10		
		C.4	5	5	2	50	10		
	B.2. Physical treatment	B.2	5	5	2	50	10	50	110
ANALYTICAL ASPECTS	D.1-5. Interferences with other dyes and markers	D.1	5	5	2	50	10	50	350
		D.2	5	5	2	50	10		
		D.3	5	5	2	50	10		
		D.4	5	5	2	50	10		
		D.5	5	5	2	50	10		
STABILITY ASPECTS	Stability of the samples	F.1	5	5	2	50		50	50

TOTAL SAMPLES 1260

Table 2. Scoring adapted to the actual tests performed. The maximum score is 475 points per fuel quality B0 / B7 and 950 points (2 x 475 points) as a grand total for both fuel qualities.

	Test	Test	Max points per experiment
REMOVAL TESTS MAX 200 p.	A.1-5, Adsorption	A.1	20
		A.2	20
		A.3	20
		A.4	20
		A.5	20
	B.1, Physical treatment	B.1	100
BREAKDOWN TESTS MAX 175 p.	C.1-4	C.1	25
		C.2	25
		C.3	25
		C.4	25
	B.2, Physical treatment	B.2	75
ANALYTICAL ASPECTS MAX 100 p	D.1-5, Interferences with other dyes and markers	D.1.	15
		D.2	15
		D.3	15
		D.4	15
		D.5	15
	E.1, Detectable at concentration of 2%	E.2	25

3 Performance tests

3.1 Preparation of marked gas oil solutions

The performance tests were carried out using B0 and B7 gas oil solutions tagged with the five candidate markers at the intended marking level specified by the applicants (Table 3). In total, ten solutions of about 10 L each were prepared in clear glass bottles covered with aluminium foil. This number corresponds to two independent solutions per candidate marker: one in B0 gas oil and the other, with the same marking level, in B7 gas oil.

The solutions were prepared gravimetrically, mixing the candidate marker standards in B0 and B7 gas oil. The densities used for the conversion of the units from mg/L to mg/kg were taken from the MSDSs of the gas oils:

- ρ_{B0} = 0.8369 kg / L
- ρ_{B7} = 0.8403 kg / L

For the standards and gas oil mass measurements, marker concentration units were converted from mg / L to mg / kg, resulting in different target candidate marker mass fractions for the B0 and B7 gas oil solutions (see Table 3) because of the different densities. The target concentrations and mass fractions are valid for a 100 % purity of the candidate markers. In practice, the purity of the candidate markers must be taken into account to compensate for systematic differences.

Table 3. Target mass fractions (mg / kg) and concentrations (mg / L) of the candidate markers in the gas oil solutions prepared for the performance tests.

Candidate marker	**Concentration given by supplier (mg / L)**	**Target mass fraction (mg / kg)**	
		B0 gas oil	**B7 gas oil**
CM1a ([3])	3	3.58	3.57
CM1b	9.5	11.35	11.31
CM2	9.5	11.35	11.31
CM3	2	2.37	2.38
CM4	2	2.39	2.38
CM5	8	9.56	9.52

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[3] The candidate marker CM1 contained two chemical substances marked here as CM1a and CM1b.

All the solutions were kept in the dark by wrapping the bottles completely in aluminium foil and maintained at room temperature during the experiments as previously stated.

3.2 Description of the performance tests and sample preparation

Performance tests were completed between August and December 2016 at JRC Geel premises.

The additional stability test (Table 1) carried out by JRC but not considered in the CEI was performed as a quality control indicator to ensure the stability of the marked gas oil solution for the duration of the experimental period.

3.2.1 (A) Removal tests

The removal tests assess the potential of the candidate markers to be completely or fully separated from the gas oil matrix without chemical transformation or degradation.

A.1 Laundering experiments

The potential removal of the candidate marker was tested using simple column experiments, where 50 - 100 mL of the marked gas oil solution was passed through a pre-packed column with the test adsorbent. The gas oil collected at the end of the column was directly poured into a sample vial and sealed for shipment to the laboratories. The concentration of the candidate marker was determined, and recovery (percentage of the initial candidate marker remaining in the gas oil after passing through the column) was calculated for each sample. The average and relative standard deviation of the five replicates in each series were used to assess and compare the performance of the different candidate markers.

B.1 Physical process

Further technical details of this experiment will not be disclosed in this report.

3.2.2 (B) Breakdown tests

In these experiments, the marker is removed or changed by degrading it through several mechanisms.

C.1 Chemical breakdown

It has been reported that national dyes and the current Euromarker can be transformed and / or degraded in the presence of different chemicals.

The efficiency of the removal process depends on many factors such as the strength of the chemical solution, contact time and proportion between the solution and marked gas oil and reactivity of each type of dye/marker to a certain type of chemical solution amongst others.

Further technical details of this experiment will not be disclosed in this report.

B.2 Physical process

Further technical details of this experiment will not be disclosed in this report.

3.2.3 (D) Analytical aspects

Some analytical issues that do not imply the active removal or degradation of the markers from the gas oil were also included as a requirement in the technical evaluation. The presence of existing markers could potentially impact the correct determination of the candidate markers in gas oil and kerosene directly.

A new standard fiscal marker should preferably be compatible meaning unmistakably identified and quantifiable, in the presence of national dyes in use in the different Member States.

Also, to identify cases where laundered gas oil and kerosene are used, especially as motor propellant, the potential fiscal marker should also be quantified at low concentrations in routine roadside controls (set to 2 % of the marking level).

The assessment of both technical requirements was tackled using different approaches. The compatibility with national dyes was based on empirical data obtained in laboratory tests while the detection at low concentration by roadside tests was based on documentary evidence provided by the applicants.

D.1 Interferences with other dyes and markers

During the preparation stage of the CEI, representatives from the Member States knowledgeable about the use of national dyes and the Euromarker and detection of fraudulent activities and analysis of related samples, were consulted to prepare a list of the most commonly used national dyes. The final list, after the corrigendum of 14 November 2015 and the modification described in section 2.3.1 of this report, is shown in Table 4.

Two types of dyes can be found in the list from the Call of Expression of Interest. The first one corresponds to statutory markers (SM in Table 4), which are the official markers given in national laws. These dyes are usually sold by chemical product suppliers in relatively pure form and are used as analytical standards by control laboratories.

The other type of dyes, commercial dyes (CoM in Table 4), are derivatives of the statutory markers which are mixed with different hydrocarbons to make them more soluble. Dye producers supply them, generally provided in bulk and are the products used to mark the gas oil. Their response is compared to one of the corresponding statutory markers, and the results are reported in terms of the statutory marker.

The final concentration of each of the dyes was selected using the information from the document 'Dyes used in Member States: information taken from Vade Mecum', provided by DG TAXUD. The concentrations are reported as found in the mentioned document. For this reason, the measurement units are not the same in all the cases.

For the dyes used in more than one country and with different marking concentration, the highest concentration was selected for this experiment. If there is no interference at the highest dye concentration, it is unlikely that there will be an effect at a lower concentration.

National dyes currently in use were obtained from Sigma-Aldrich, John Hogg Solutions (UK) and the Italian and Danish governments. Ten different gas oil solutions were prepared per candidate marker, five in B0 gas oil and five in B7 gas oil, each containing two national dyes and one candidate marker at the selected marking level. Those solutions were sent to the assigned laboratories without further treatment, and the determination of the candidate markers was carried out. If the presence of national dyes does not interfere with the analysis and detection of the candidate markers, a recovery of around 100 % would be expected (marking level). Significantly higher or lower recoveries of the candidate marker would suggest an interference of the national dyes present in the solution in the analytical determination of the candidate marker since no other treatments were applied to the samples for this experiment.

Table 4. General information about the national dyes used in the interference experiment describing mixtures 1-5 i.e. interference tests 1-5.

MIXTURE	CAS No	Name	Type	Supplier	Colour	Marking levels (1)	Country	Selected concentration
MIX 1	6368-72-5	CI Solvent Red 19	SM	Sigma	Red	4-6 mg/L	Several	6 mg/L
	17354-14-2	CI Solvent blue 35	SM	Sigma	Blue	5-15 mg/L	Several	15 mg/L
MIX 2	34432-92-3	SY124	SM	Sigma	Orange	6-9 mg/L	All	8 mg/L
	81-64-1	Quinizarin	SM	Sigma	Orange	1.75-3 mg/L	UK/GR	3 mg/L
MIX 3	85-83-6	CI Solvent Red 24	SM	Sigma	Red	4-10 mg/L	UK	10 mg/L
	128-80-3	Green alizarin G base	SM	Sigma	Green	50 mg/kg	IT	50 mg/kg
MIX 4	56358-09-9	Solvent Red 19 analogue	CoM	JHTS	Red	5.3 - 6.4 mg/L	DE	5.8 mg/L (2)
	57712-94-4	Penetrant red				6.1 - 7.3 mg/L		6.7 mg/L
	64553-79-3	CI Solvent blue 79	CoM	Denmark	Blue	5 mg/L	DE	5 mg/L
MIX 5	97862-23-2	Solvent Green 33	CoM	Italy	Green	50 mg/kg	IT	50 mg/kg
	85750-13-6	CI Solvent Red 161	CoM	JHTS	Red	40 mg/kg	IT	40 mg/kg

SM: statutory marker; CoM: commercial marker; JHTS: John Hogg Technical Solutions.

(1) Obtained from the information sent by TAXUD (‘Dyes used in Member States: Information taken from the Vade Mecum’).

(2) The concentrations for these dyes were obtained by direct communication with the German Customs Laboratories.

E.1 Detectable at concentration of 2 %

This parameter was the only technical requirement evaluated using the documentation provided by the applicants and not using empirical data obtained from the performances tests performed in the JRC laboratory.

The evaluation was based on the information regarding the proposed roadside methods and instrumentation to test the corresponding candidate markers, detectability capacities and price of the instruments and consumables.

3.2.4 (F) Stability

Documentary evidence of the stability of the candidate markers in solution at the intended marker level, at least for three months, was provided by the applicants in Phase I of the CEI; therefore, this parameter was not formally part of the technical requirement in Phase II. Nevertheless, the stability of the marked gas oil solutions during the performance tests was monitored as an extra quality parameter. Results from the REF samples (which are aliquots of marked gas oil solutions taken directly from the main solutions) and a set of non-treated samples was compared to evaluate the stability of the candidate markers in gas oil at room temperature in the dark during the completion of the performance tests.

Results from the reference samples and stability samples can also be used as quality control samples during the analytical measurements in each sample series.

3.3 Selection of laboratories

European customs and other national testing laboratories in the Member States are in charge of the road-control and testing of the current Euromarker. It is safe to assume that, should a new standard fiscal marker be introduced, these laboratories will perform the same activities of measuring the new substance. For these reasons, the Customs Laboratories European Network (CLEN) was contacted during Phase I of the CEI in requesting support for the analysis of the 1 260 samples derived from the performance tests. Amongst the customs laboratories that showed interest in analysing the samples, an invitation to tender was launched at the end of Phase I upon the availability of specific information regarding the short-listed candidate markers and their analytical methods. Unfortunately, the contacted laboratories did not have the technical capabilities or the time available to perform the necessary analyses for CM1, CM2 and CM3. Finally, one Customs Laboratory graciously performed the analysis of CM4 and CM5.

Commercial laboratories were thereafter contacted for the analysis of the remaining candidate markers (CM1, CM2 and CM3). After an exhaustive market research and following the JRC's public procurement procedure, the analysis of the mentioned candidate markers was granted to an external company.

3.4 Analytical techniques used for analysis

Table 5. Method description and instrumentation used for determination of CM1a to CM5.

Marker	Short method description and instrumentation
CM1a	**Method description:** method does not include an extraction or preparation method as fuel samples are assayed directly. **Analysis by GC-MS** **Measurement uncertainty is estimated as:** Relative combined uncertainty (ur): ur=√((RSD^2)+(uR^2)) Laboratory reproducibility: (RSD) Relative uncertainty of recovery uR (bias): uR=100%-Yc Relative expanded uncertainty (uc): uc=k × ur; k=2 uncertainty of determination was 20 %.
CM1b and CM2	**Method description:** method does not include an extraction or preparation method as fuel samples are assayed directly. **Analysis by GC-MS** **Measurement uncertainty was estimated as:** "Relative combined uncertainty (ur): ur=√((RSD^2)+(uR^2)) Laboratory reproducibility (RSD) Relative uncertainty of recovery uR (bias): uR=100%-Yc Relative expanded uncertainty (uc): uc=k × ur; k=2 Uncertainty of determination was 20 % relative.
CM3	**Method description, sample preparation:** Addition of internal standard. **Analysis by GC-MS** **Measurement uncertainty is estimated as:** Relative combined uncertainty (ur): ur=√((RSD^2)+(uR^2)) Laboratory reproducibility (RSD) Relative uncertainty of recovery uR (bias): uR=100%-Yc Relative expanded uncertainty (uc): uc=k × ur; k=2 uncertainty of determination was 20 %.
CM4	**Sample preparation:** none **Analysis by GC-MS** **Measurement uncertainty is estimated as:** The intra-laboratory reproducibility was determined by measurement of the CM4-B0 and CM4-B7 samples (originally sent for method implementation) after each recalibration (separate B0 and B7 control charts with 34 points each). The derived reproducibility standard deviation for the CM4-B0 reference is s = 0,3018 (mean = 2,4636 mg/l). The resulting intra-laboratory reproducibility is R(L) = 0,85 mg/l. For the CM4-B7 reference sample we found the following values: s = 0,2683 (mean = 2,5041 mg/l), R(L) = 0,76 mg/l. Sample storage/preservation dark, at room temperature.
CM5	ISO 17025 accredited method: yes **Sample preparation:** none **LC apparatus:** Dionex Ultimate 3000 Column: Phenomenex Luna 250 mm length, 4.6 mm i.d. particles size 5 µm 20 µl injection volume, column temperature: 40 °C **LC program**: Euromarker Reference Method, Isocratic Toluene / EtOAc = 96 / 4 % **Detector:** DAD, Wavelength 450 nm SY124 from Sigma-Aldrich, dissolved in Xylene, corrected concentration with ERM-EF 318. **Calibration range:** 0,02 – 10 mg/L Number of calibration points 2x3 per 10 samples. Bracketed Recalibration every 10 Samples **Measurement uncertainty is estimated as:** For Repeatability and Reproducibility see Euromarker Reference Method

3.5 Identification and labelling of the samples

A double identification system was used to accurately identify the samples and the aliquots sent to the laboratories:

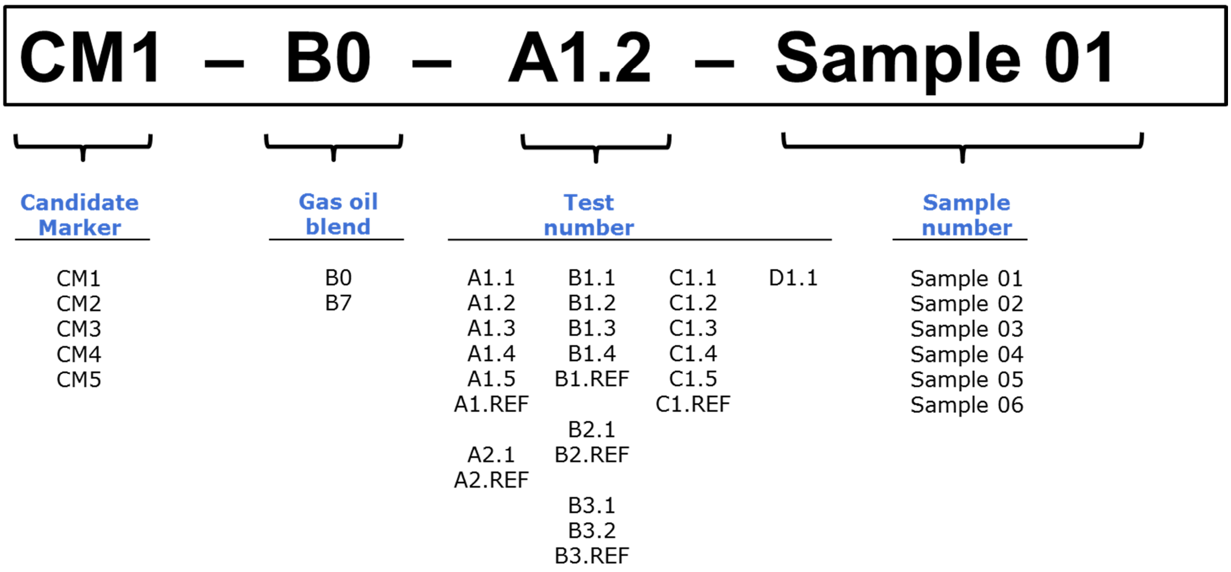
I. Sample ID name, with information regarding the candidate marker, performance test and gas oil type.

II. The sample number (in red): all samples had a unique number from 1 to 1 260, following the order of appearance in the general samples list.

The reason for the double identification system is that although the sample ID name provides all the necessary information regarding the sample, it could be misread. In fact, the difference between two entirely different samples may only be one letter of difference in the middle of a long ID name. Contrastingly, numbers are easy to differentiate to avoid mistakes during the laboratory analyses and manipulation of hundreds of vials.

The codification system for the sample ID name is as given in Figure 2.

Figure 2. Example of a sample ID name and summary of the codification system used in the labels.



Candidate Marker	Gas oil blend	Test number				Sample number
CM1	B0	A1.1	B1.1	C1.1	D1.1	Sample 01
CM2	B7	A1.2	B1.2	C1.2		Sample 02
CM3		A1.3	B1.3	C1.3		Sample 03
CM4		A1.4	B1.4	C1.4		Sample 04
CM5		A1.5	B1.REF	C1.5		Sample 05
		A1.REF		C1.REF		Sample 06
			B2.1			
		A2.1	B2.REF			
		A2.REF				
			B3.1			
			B3.2			
			B3.REF			

Candidate markers (CM): Five candidate markers were tested in total. The first four, from CM1 to CM4 corresponded to the short-listed candidate markers in Phase I of the CEI and the last one, CM5 to Solvent Yellow 124 (Table 1).

Gas oil/biodiesel blends: Two types of gas oil/biodiesel blends compliant with EN 590 were used in the experiments, following the specifications laid down in the CEI.

- B0 gas oil: a straight run distillate, without any biodiesel, identified as B0 (meaning the 0 % of biodiesel in the product).
- B7 gas oil: a blend of the same gas oil as above containing 7 % of pure rapeseed biodiesel (based on a fatty acid methylester). The biodiesel component was compliant with EN 14214.

Test numbers: For the code system, the nomenclature used in the Call for Interest was followed (see Table 1), from test A1.1. to D1.1.

Note that the coding in Table 1 is somewhat different from the codification described in Figure 2 because of deliberate omission and rearrangement of some critical technical details in this report.

REF indicates the five reference samples corresponding to each batch and type of test.

Replicate number: samples were numbered from 01 to 06 in each experiment. This corresponded to five replicates, (from Sample 01 to Sample 05) and a blank sample (Sample 06) containing only the adequate gas oil/biodiesel blend, but not the markers.

3.6 Packing and shipping of the samples

Each gas oil sample comprised of 5-10 mL of B0/B7 gas oil sealed in a 10 mL amber glass vial. Each vial was wrapped in an absorbing material and packed in a leak-proof plastic vial as a safety measure to protect from breakage during shipment. The tubes were wrapped in thermally-sealed plastic bags in groups of 4. Inside each box was placed a note with information about the sample identification, handling and storage, together with a detailed sample list and the necessary material safety data sheets.

Samples were sent to the designated laboratories by courier in special safety boxes designed for the transport of dangerous goods in exempted quantities (provided each sample unit contains less than 30 mL). All appropriate safety labelling, according to the UN globally harmonised system (GHS), was also placed before shipment.

4 Evaluation of the results

4.1 Processing of the results

Excel data sheets for reporting was prepared by JRC-Geel and sent to the two external laboratories to simplify the evaluation process. The analytical results were taken directly from the reporting sheets and subjected to calculations and evaluation. This approach simplified data handling and calculations since all analytical packages were the same with 252 samples per tested marker. First, the average and standard deviation (SD) of the five reference samples per test was calculated. The reference samples were taken from the stock solution **at the same time** as the experiment was performed. After that, the average and standard deviation of the five replicate samples (n = 5, where n is the number of samples) resulting from each type of test was calculated. All blank samples were also checked to verify that no accidental contamination had occurred either during the preparation of the test samples or at a later stage during analysis. All the blank samples measured during these series of tests were essentially free of analytes, thus confirming that no contamination had occurred and that the proposed markers cannot be found in non-marked fuel samples. Next, a recovery was calculated by dividing the amount of analyte found after the experiment with the **average of all reference samples** for that marker (Equation 1). The result is given as a percent recovery. Likewise, the relative standard deviation (RSD) was re-calculated in terms of % recovery, making it possible to plot error bars as ± one relative standard deviation for all the recoveries shown here in Figure 3a to Figure 18b. A high recovery (close to 100 %) should be taken as proof that the experiment or treatment **has had no or little effect** on the tested marker. Low recoveries or results with high variability should be interpreted as if the treatment **has had an effect on the marker**. As with all experimental data, one must here also consider the measurement uncertainty associated with the measurements. Five replicates of both reference samples and experimental samples were prepared independently to assess precision and measurement uncertainty to be expected from these measurements. It also allows a calculation of standard deviations and relative standard deviations to ascertain that a result is reliable rather than being a result of a random effect.

Equation 1 Calculation of the recovery.

$$Recovery\ (\%) = \frac{\text{Concentration of marker after experiment}}{\text{Average concentration of marker in all reference samples}} \times 100$$

A discussion about which quality criteria should be applied for a fair evaluation and scoring of the different markers can be found under section 2.4, which are applied in section 4.2 and 5.

Results obtained in the performance experiments have been displayed in graphs in the section from 4.1.1 to 4.1.5 (from Figure 3a to 18b) in order to compare and visualise the information easily. The data used to produce these graphs can be found in Table 8. The technical requirements regarding the detectability of the marker at 2 % marking level has been evaluated separately and was added for the final scoring in Table 9.

4.1.1 Adsorption experiments (A.1.)

Figure 3a. Adsorbent 1 in B0 gas oil. A low recovery is indicative of adsorption of the marker on the test adsorbent. Error bars: ± 1 SD, n = 5.

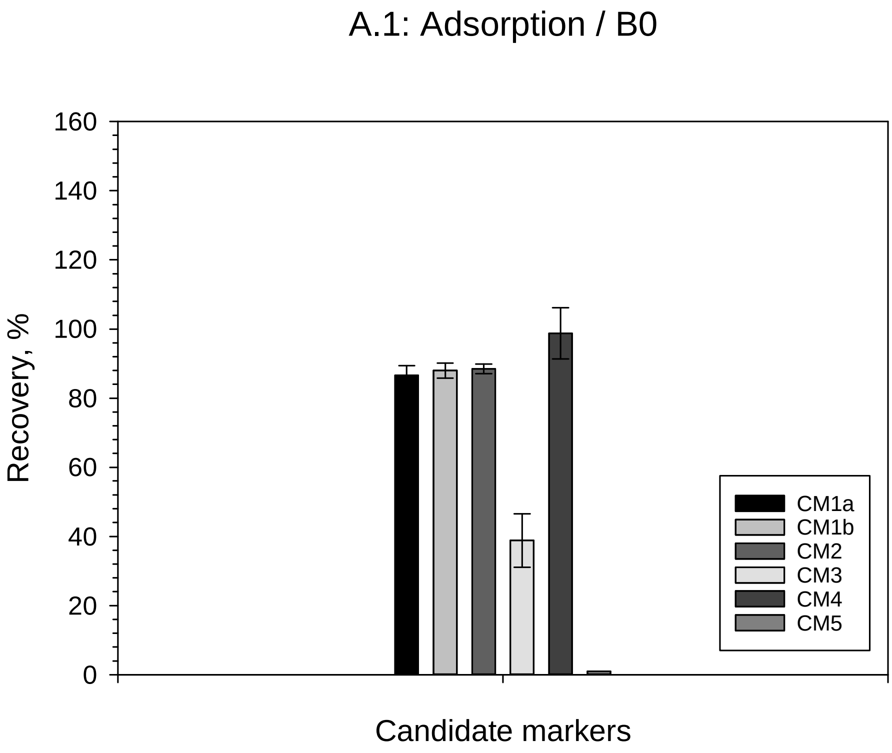


Figure 3b. Adsorbent 1 in B7 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.

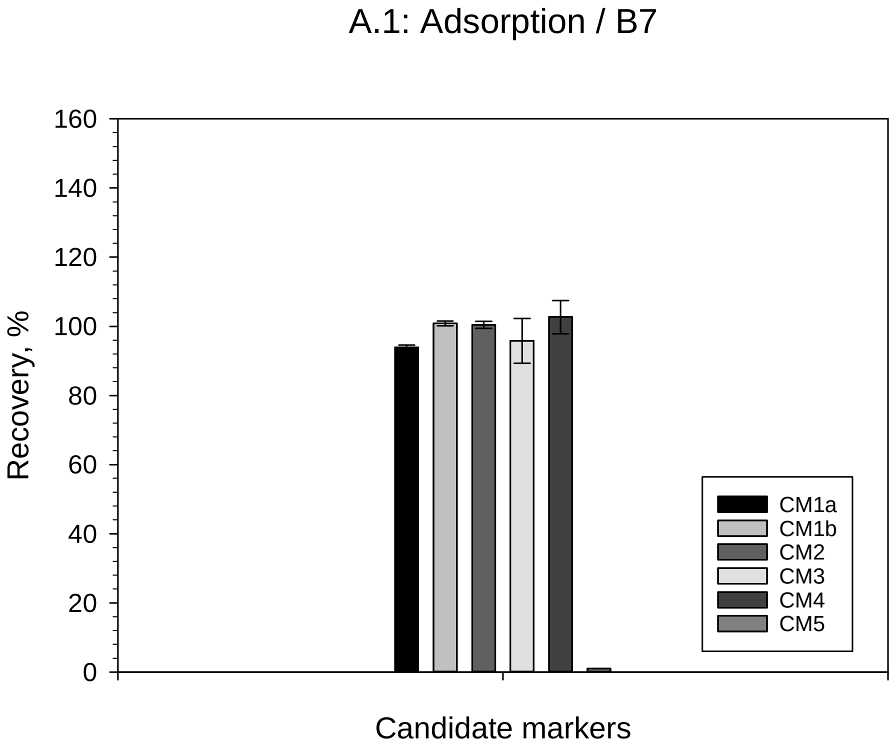


Figure 4a. Adsorbent 2 in B0 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars: ± 1 SD for n = 5.

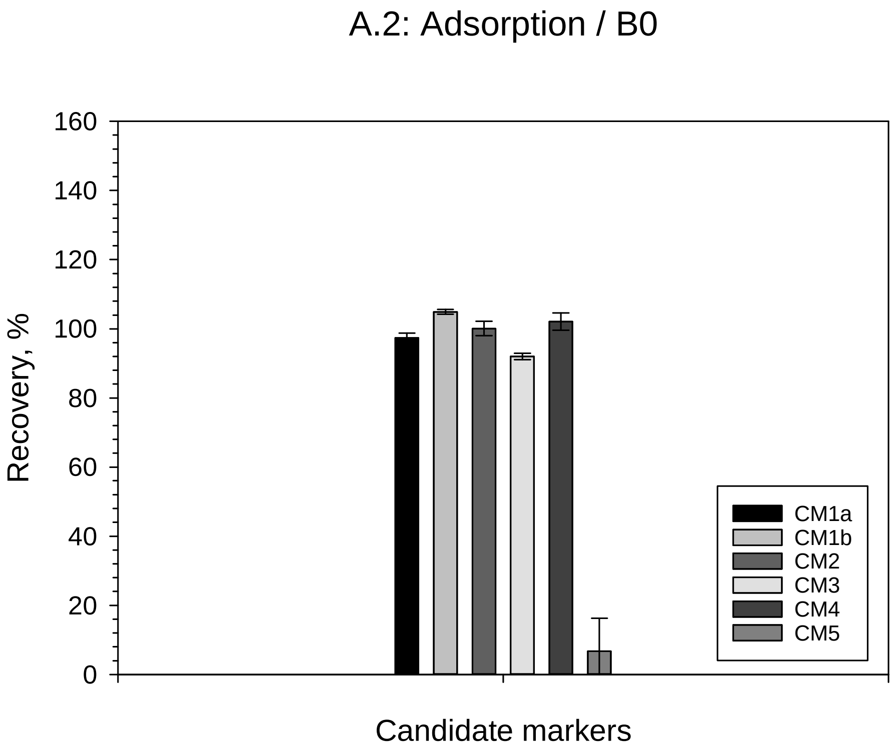


Figure 4b. Adsorbent 2 in B7 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.

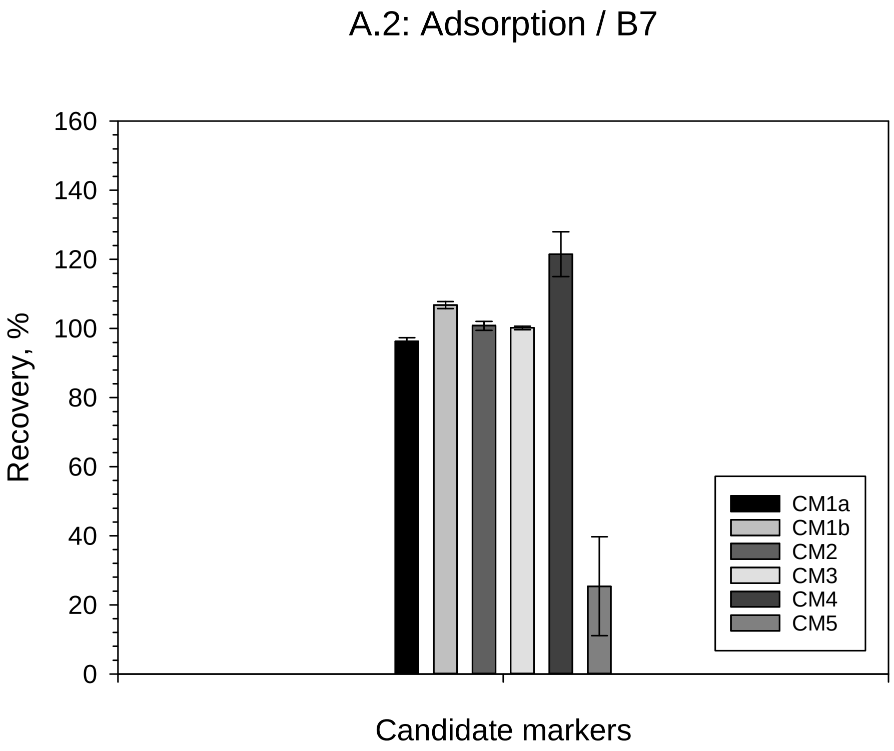


Figure 5a. Adsorbent 3 in B0. A low recovery is indicative of adsorption on the test adsorbent. Error bars: ± 1 SD for n = 5.

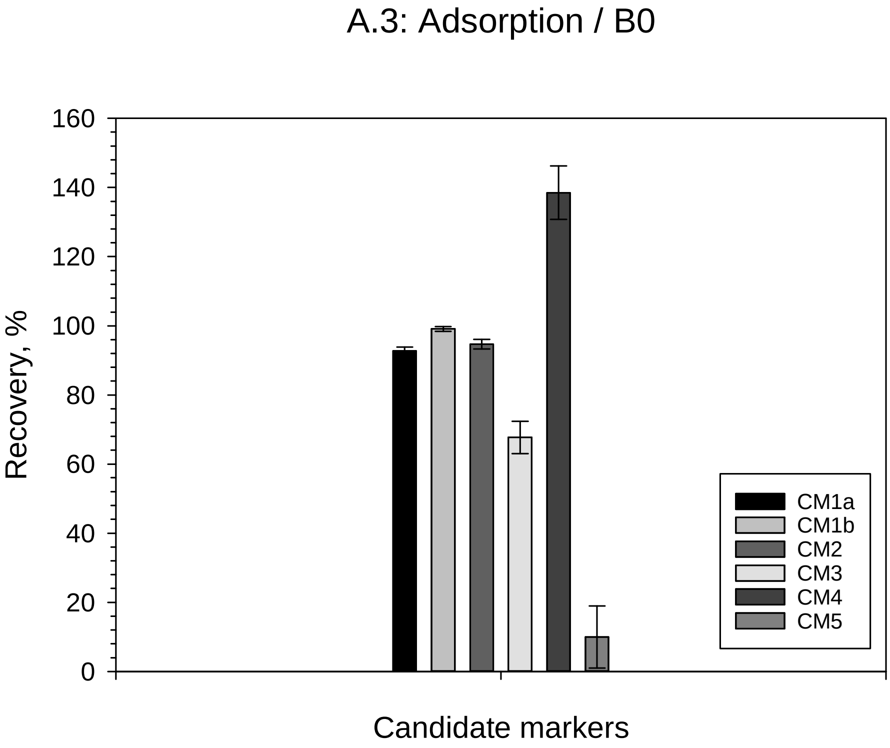


Figure 5b. Adsorbent 3 in B7 .Low recovery is indicative of adsorption on the test adsorbent. Error bars: ± 1 SD, n = 5.

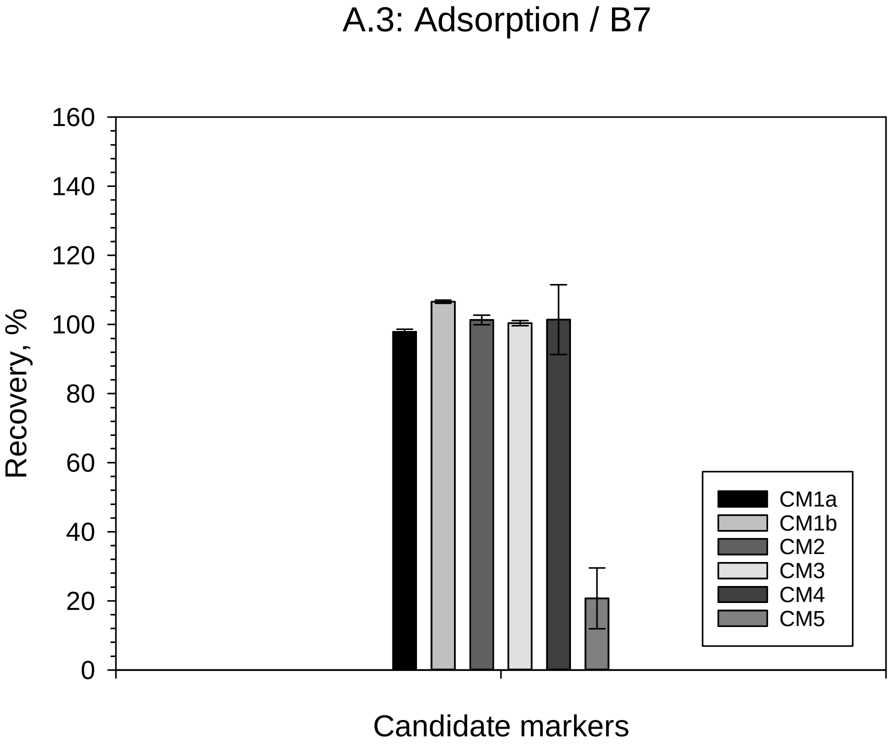


Figure 6a. Adsorbent 4 in B0 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.

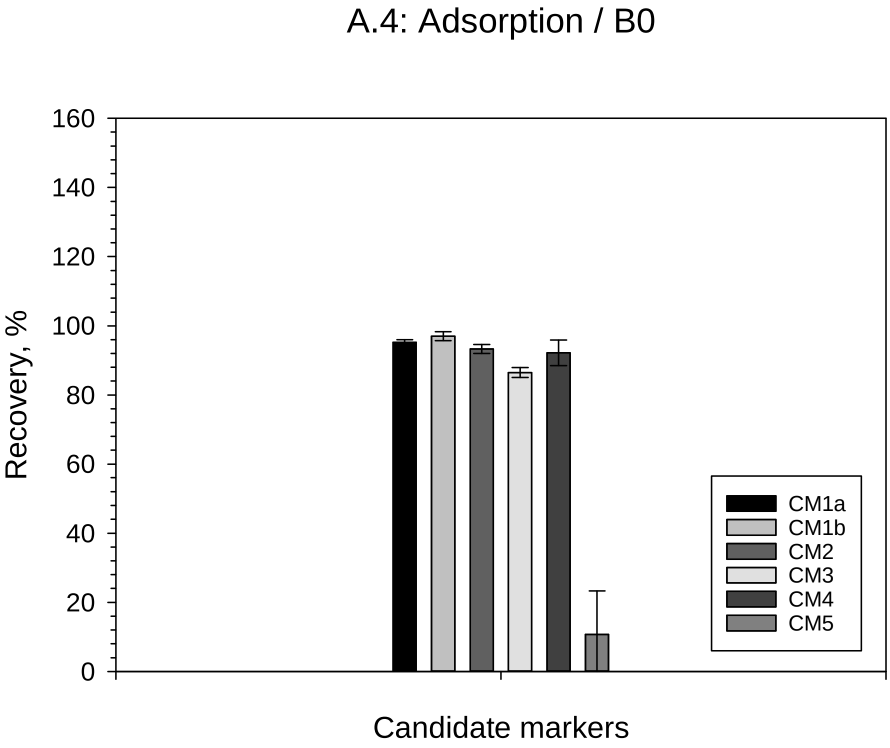


Figure 6b. Adsorbent 4 in B7 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.

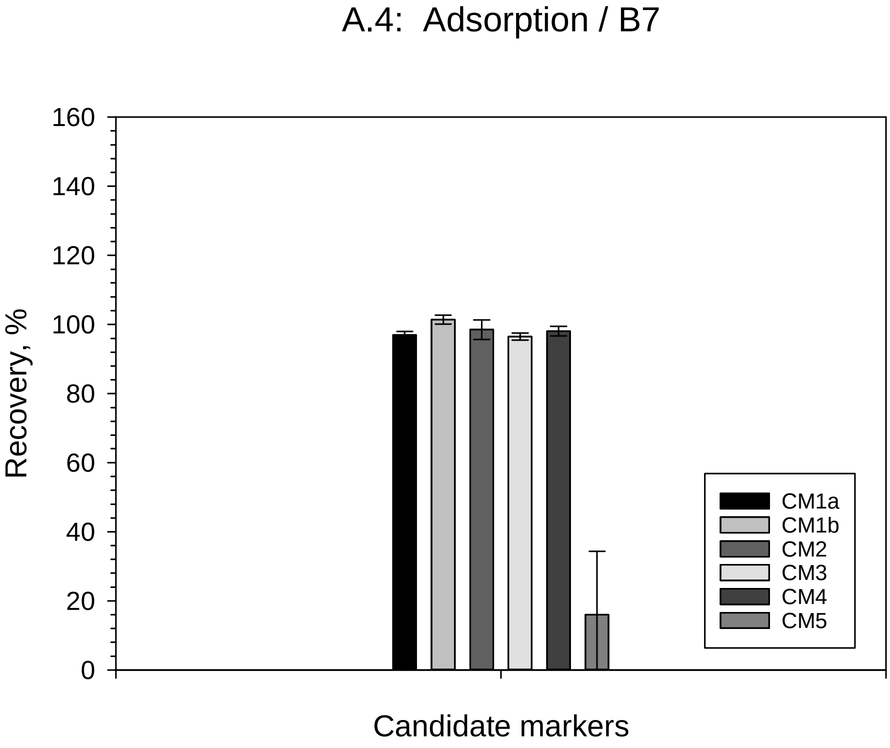


Figure 7a. Adsorbent 5 in B0 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.

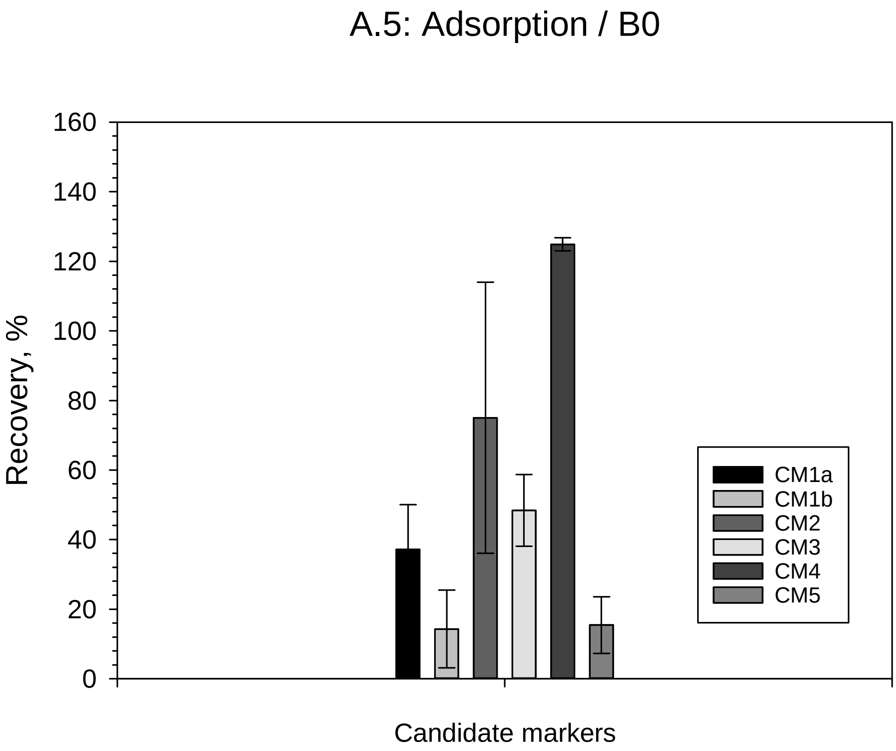
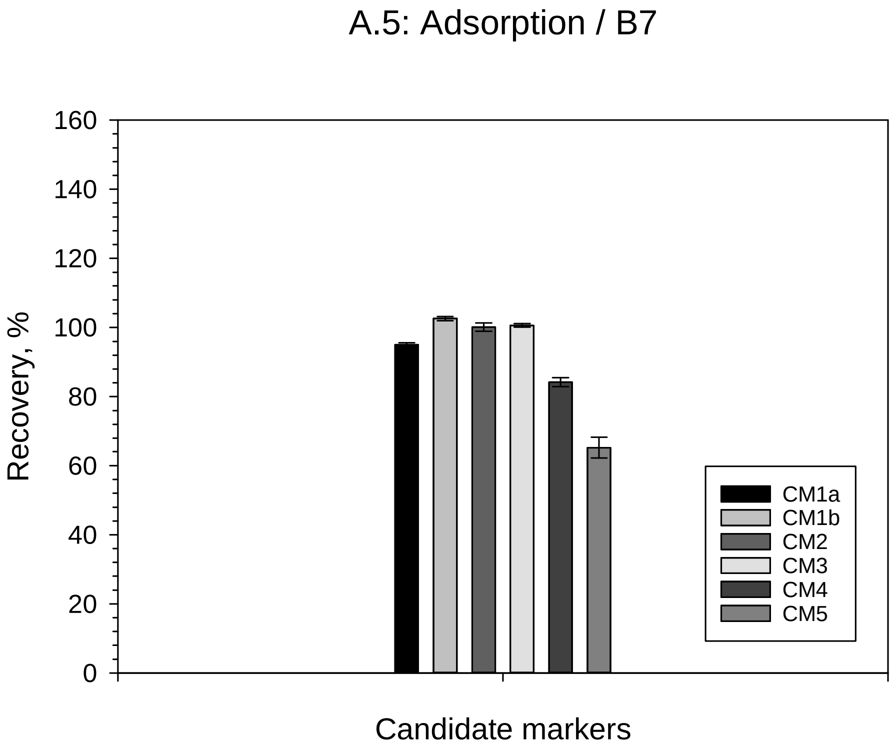


Figure 7b. Adsorbent 5 in B7 gas oil. A low recovery is indicative of adsorption on the test adsorbent. Error bars are ± 1 SD for n = 5.



4.1.2 Physical process (B.1.)

Figure 8a. A very low recovery demonstrates a loss as a result of the treatment. Error bars are ± 1 SD for n = 5.

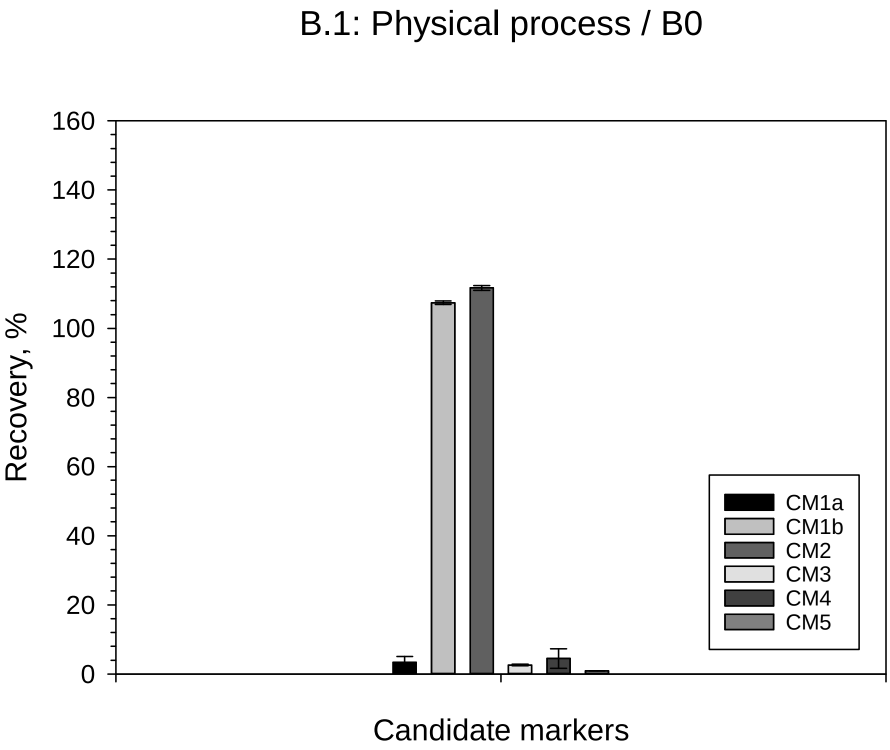
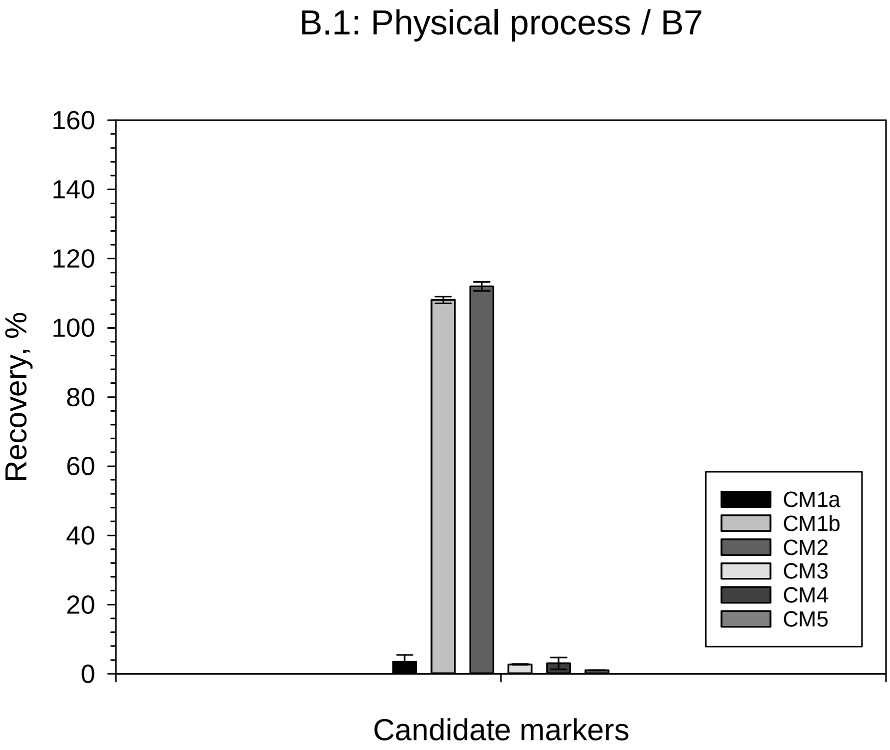


Figure 8b. A very low recovery demonstrates a loss as a result of the treatment. Error bars are ± 1 SD for n = 5.



4.1.3 Chemical treatment (C.1.)

Figure 9a. Treatment with chemical 1, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

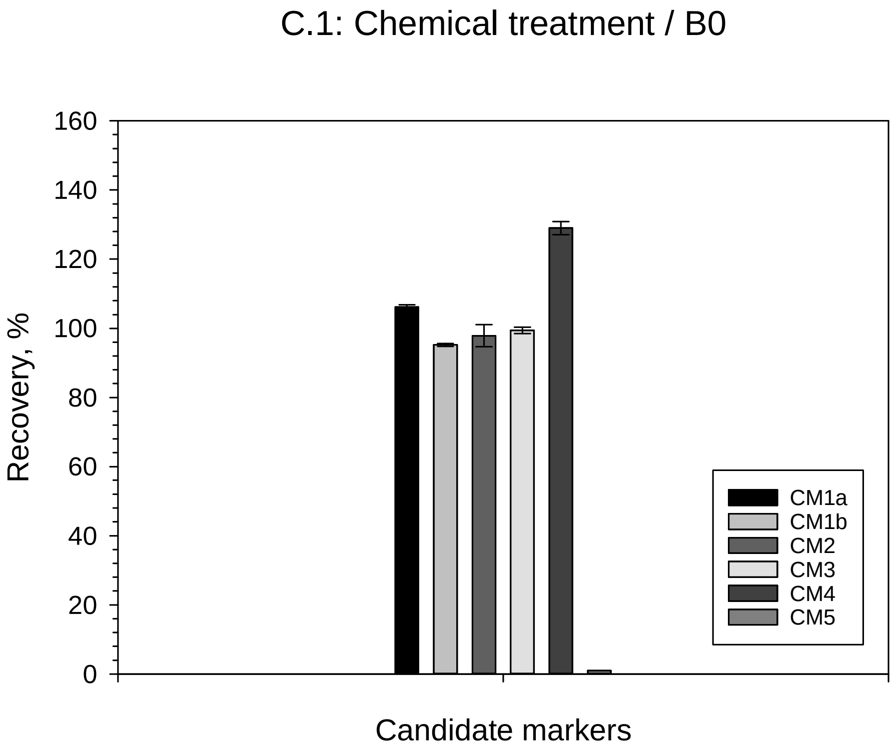


Figure 9b. Treatment with chemical 1, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

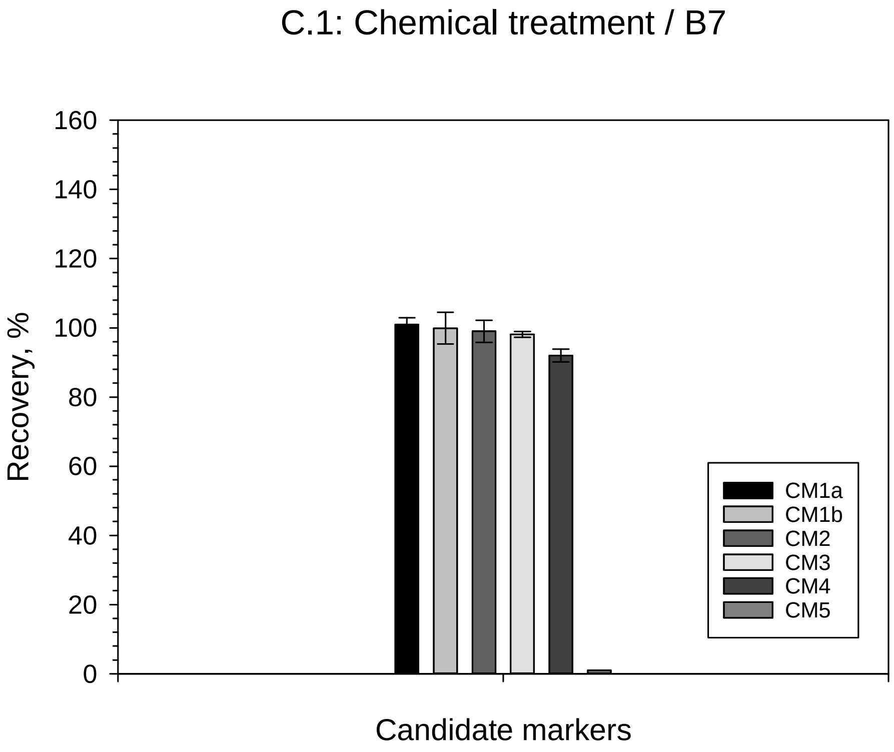


Figure 10a. Treatment with chemical 2, a very low recovery demonstrates a breakdown. Error bars are ± 1SD for n = 5.

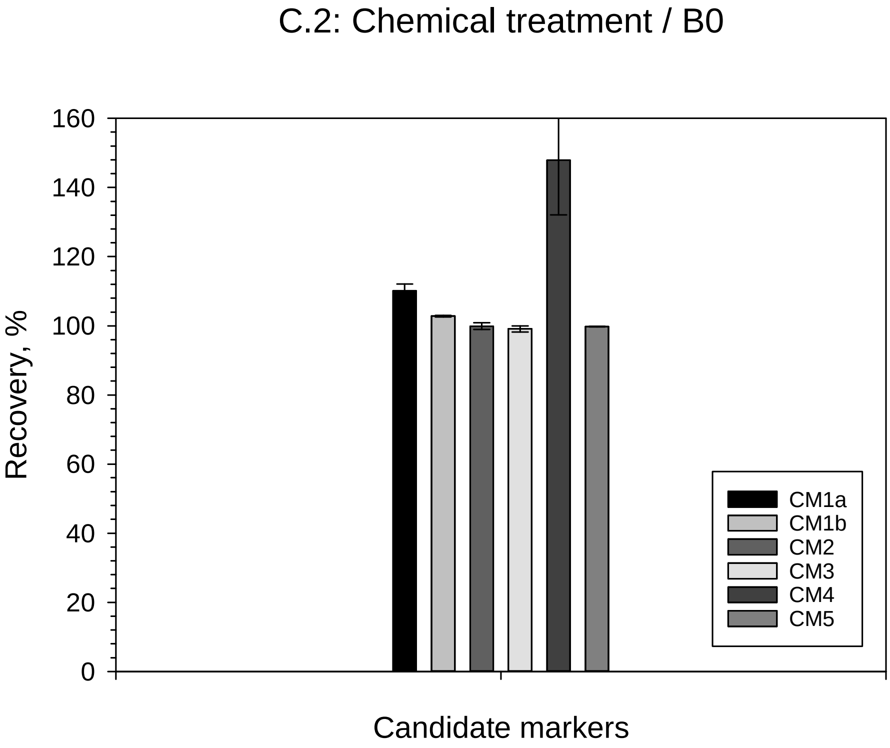


Figure 10b. Treatment with chemical 2, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

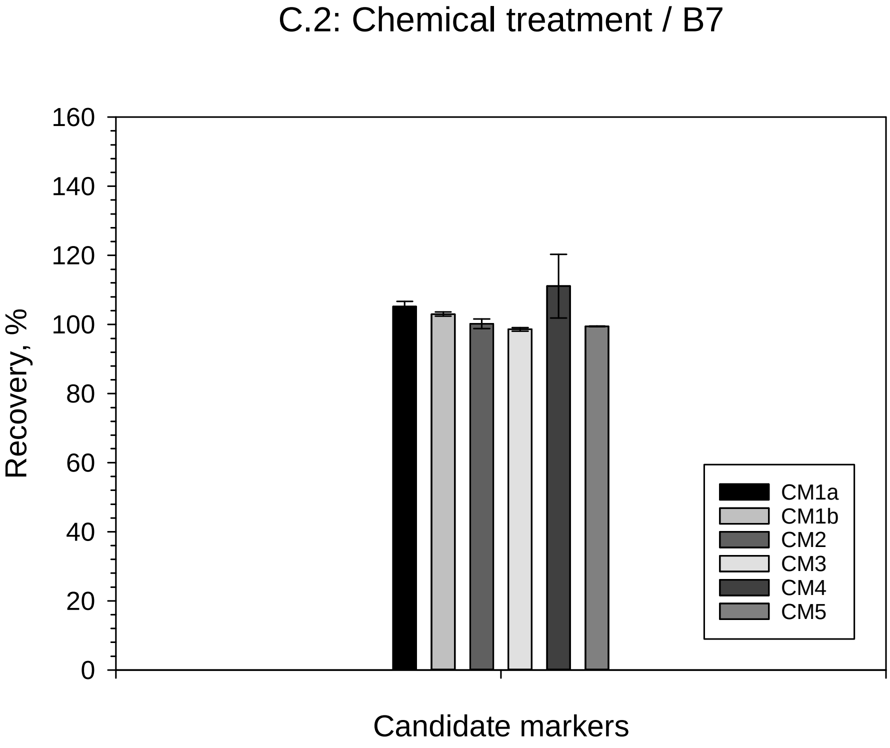


Figure 11a. Treatment with chemical 3, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

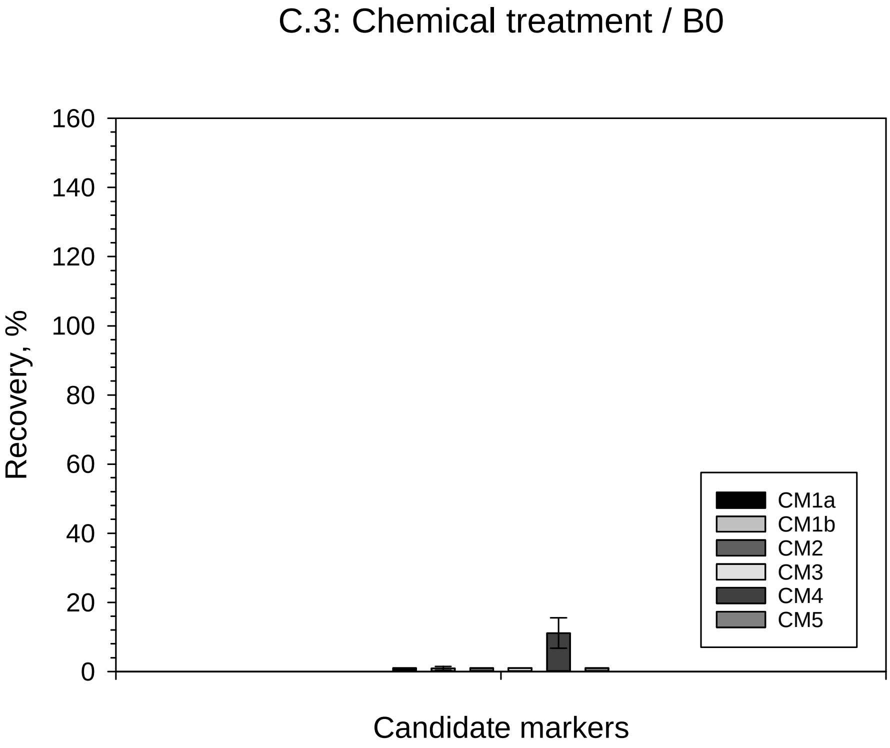


Figure 11b. Treatment with chemical 3, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

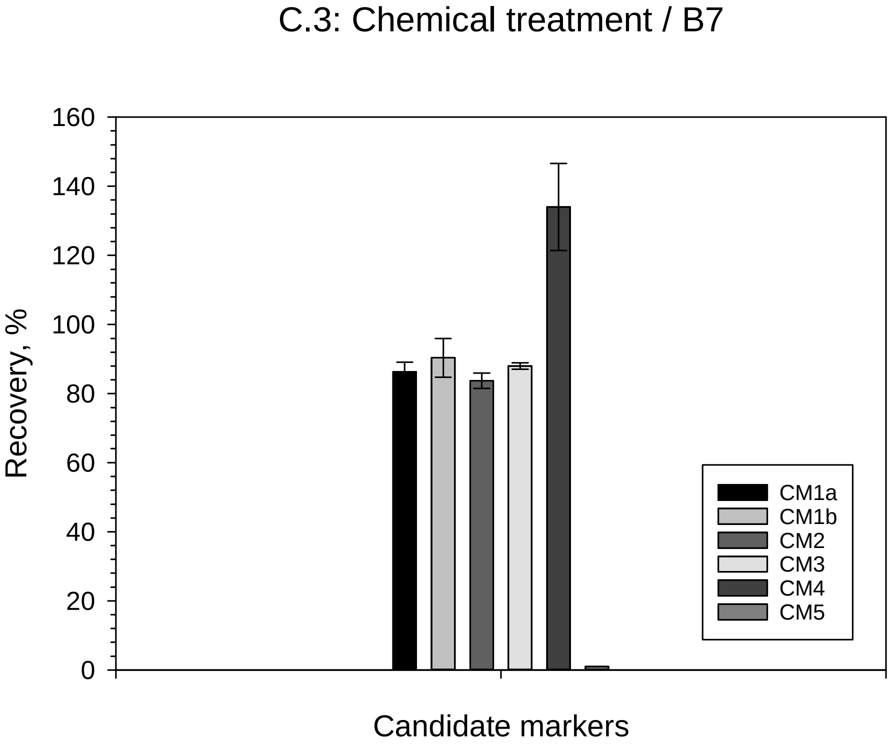


Figure 12a. Treatment with chemical 4, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.

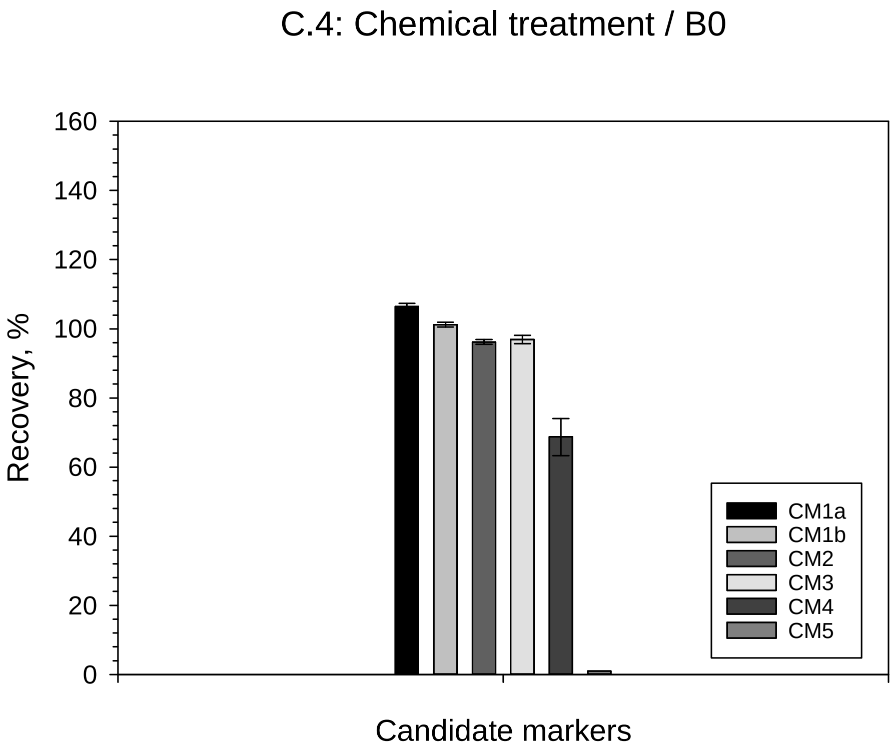
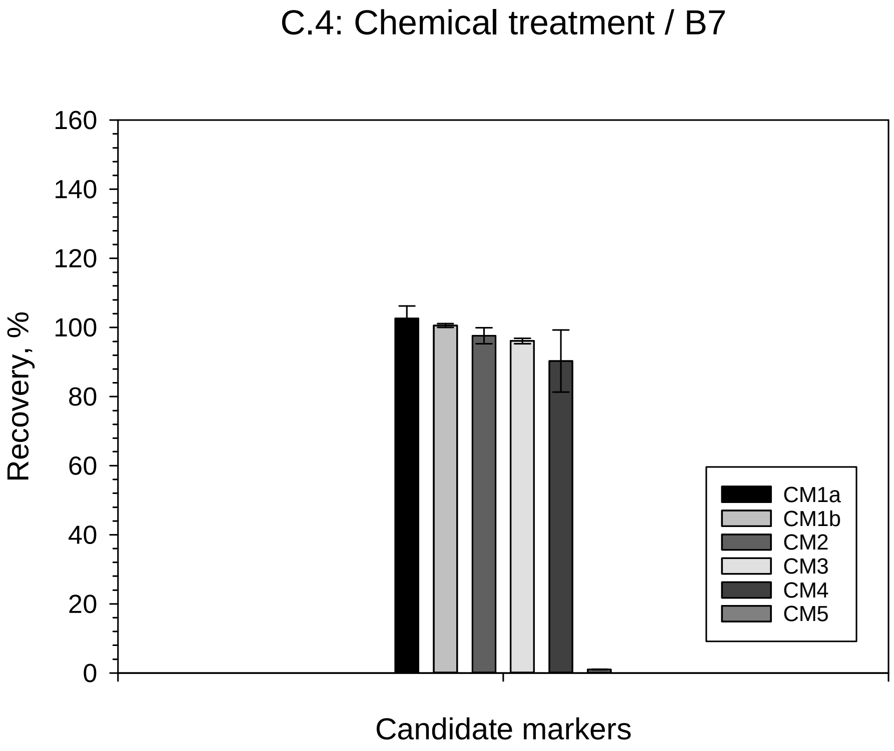


Figure 12b. Treatment with chemical 4, a very low recovery demonstrates a breakdown. Error bars are ± 1 SD for n = 5.



4.1.4 Physical process (B.2.)

Figure 13a. A very low recovery demonstrates a loss as a result of the treatment. Error bars are ± 1 SD for n = 5.

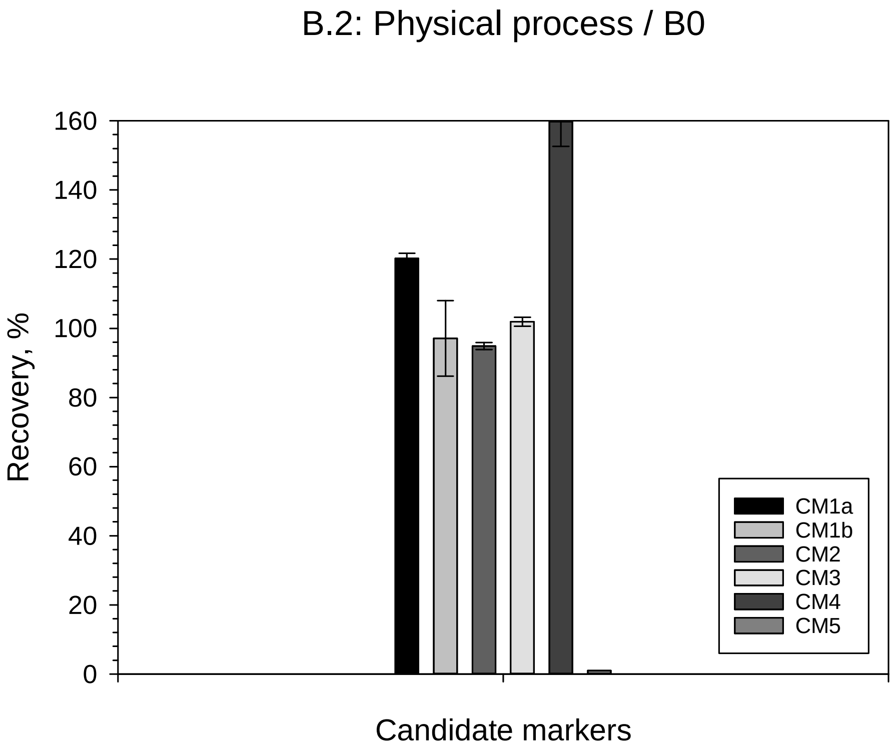
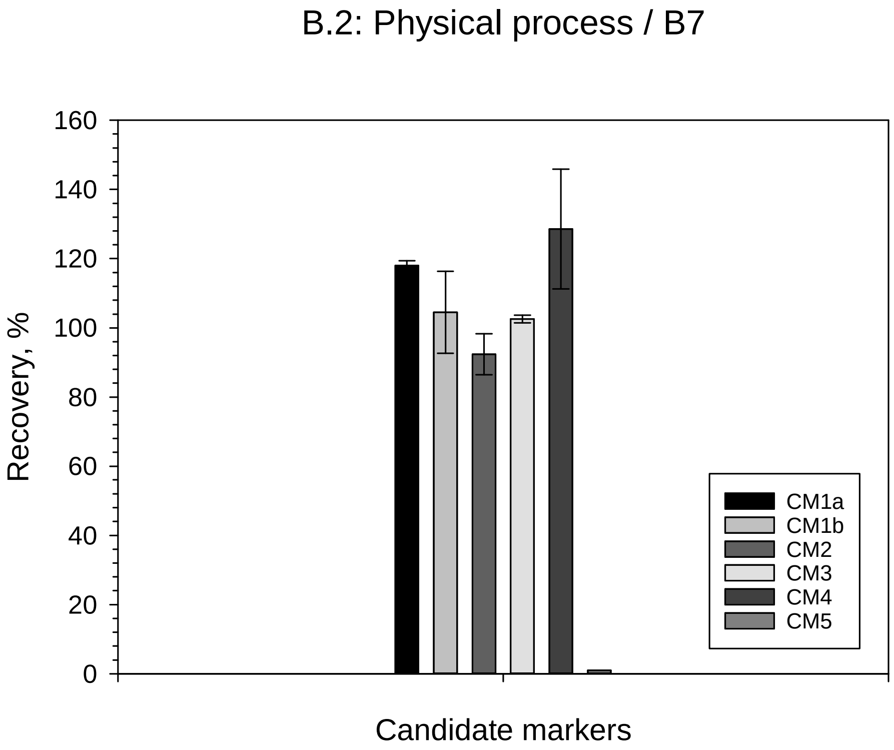


Figure 13b. A very low recovery demonstrates a loss as a result of the treatment. Error bars are ± 1 SD for n = 5.



4.1.5 Interferences with other dyes and markers (D.1.)

Figure 14a. Effect of the presence of other fiscal markers in the sample in B0 gas oil (mix 1). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

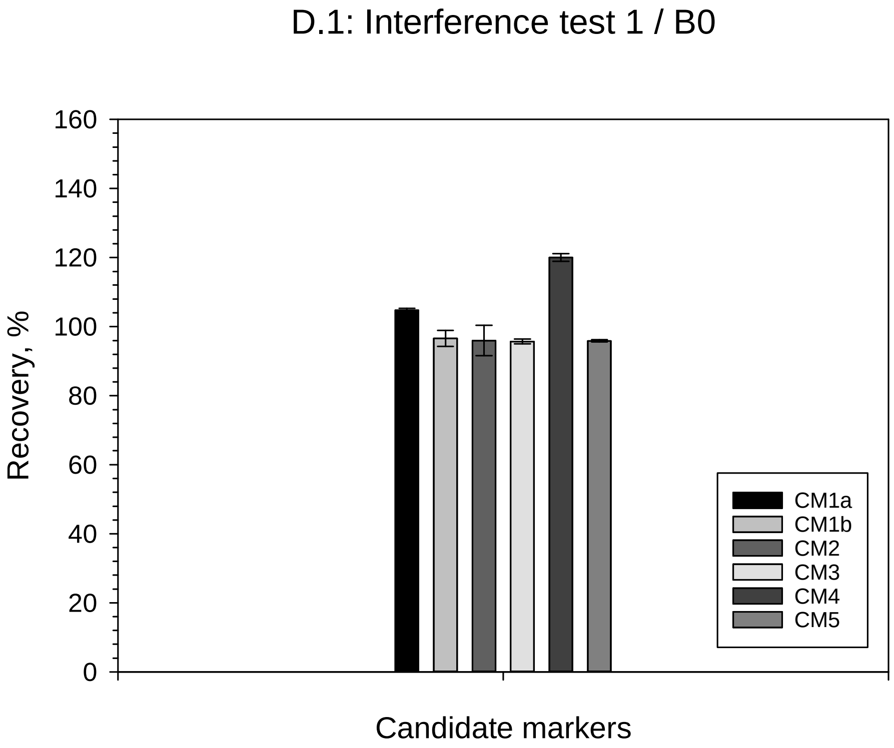


Figure 14b. Effect of the presence of other fiscal markers in the sample in B7 gas oil (mix 1). A 100 % recovery demonstrates little or no interference.

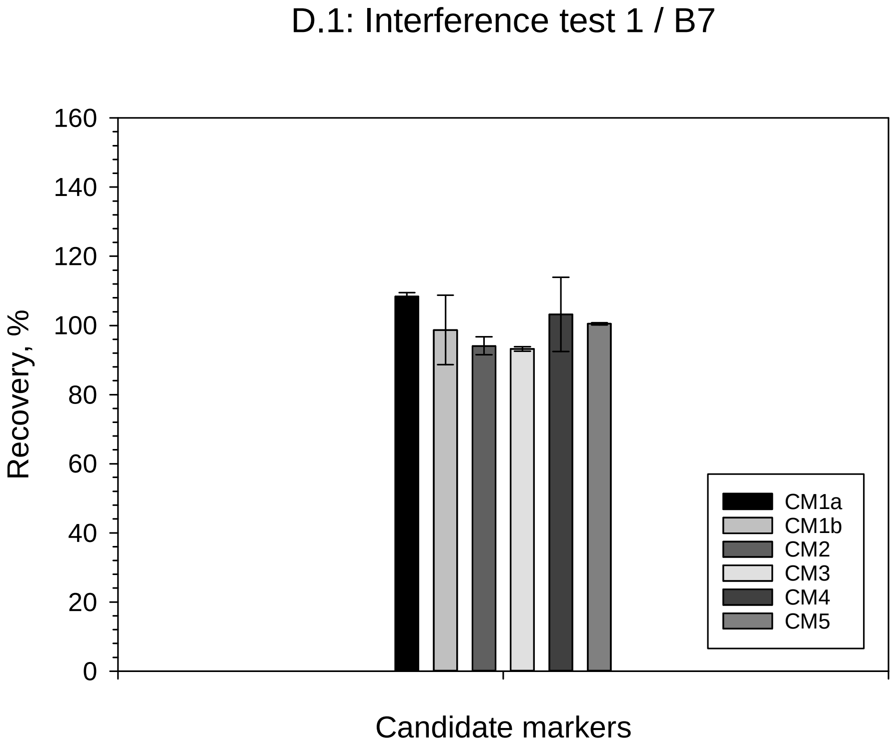


Figure 15a. Graphical results show the effect of the presence of other fiscal markers in the sample in B0 gas oil (mix 2). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

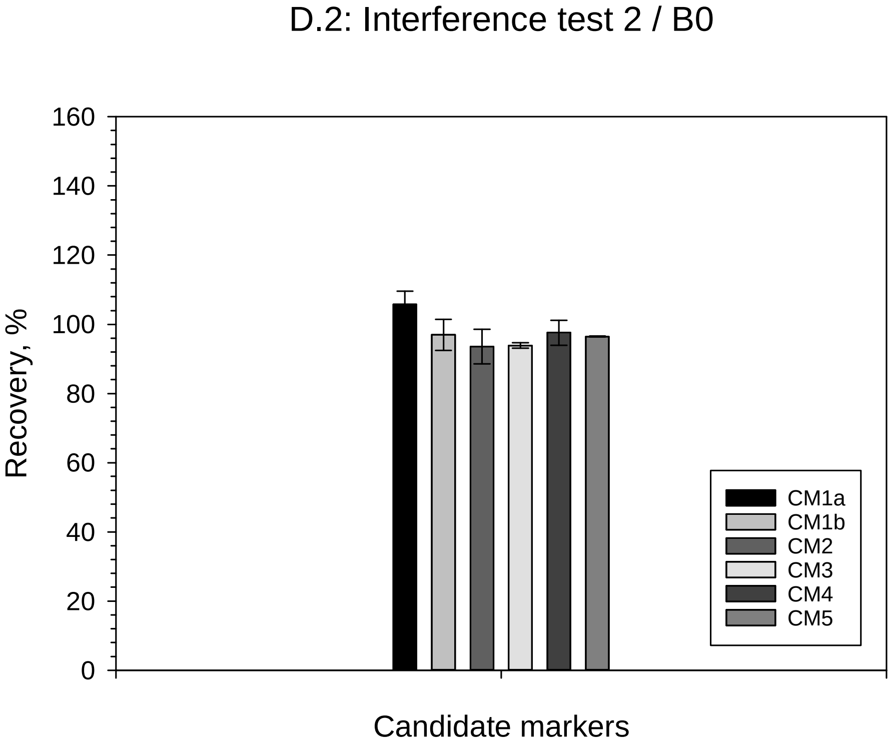


Figure 15b. Graphical results show the effect of the presence of other fiscal markers in the sample in B7 gas oil (mix 2). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

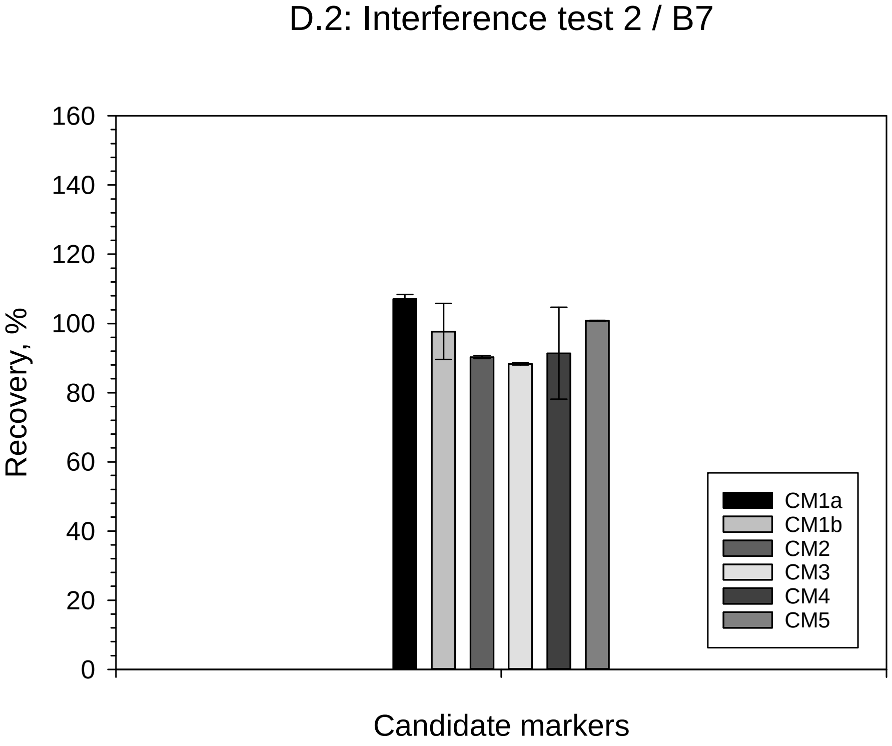


Figure 16a. Graphical results show the effect of the presence of other fiscal markers in the sample in B0 gas oil (mix 3). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

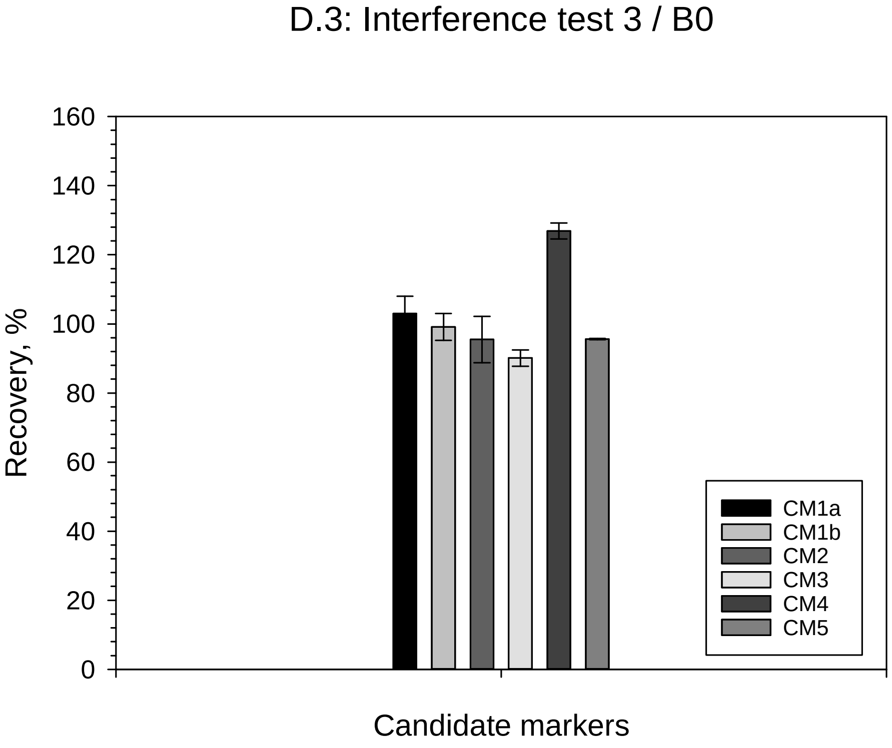


Figure 16b. Graphical results show the effect of the presence of other fiscal markers in the sample in B7 gas oil (mix 3). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

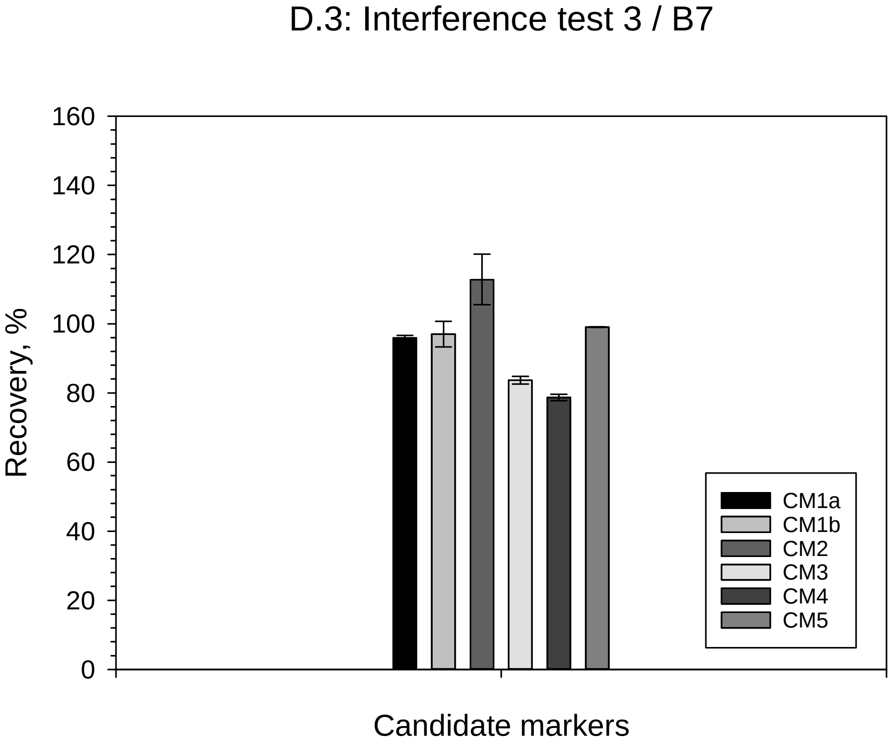


Figure 17a. Graphical results show the effect of the presence of other fiscal markers in the sample in B0 gas oil (mix 4). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

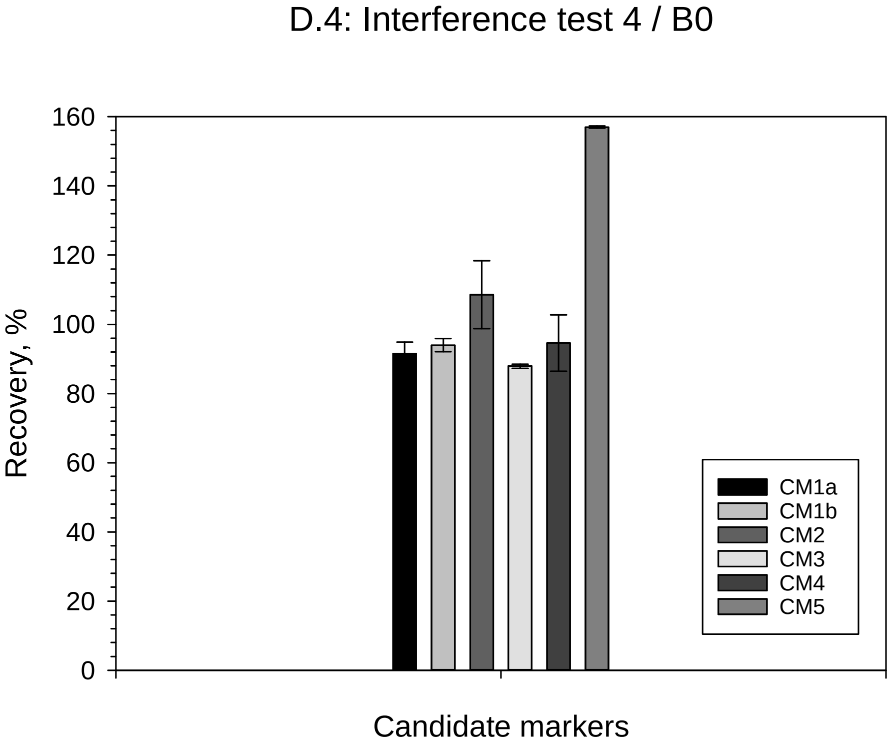


Figure 17b. Graphical results show the effect of the presence of other fiscal markers in the sample in B7 gas oil (mix 4). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

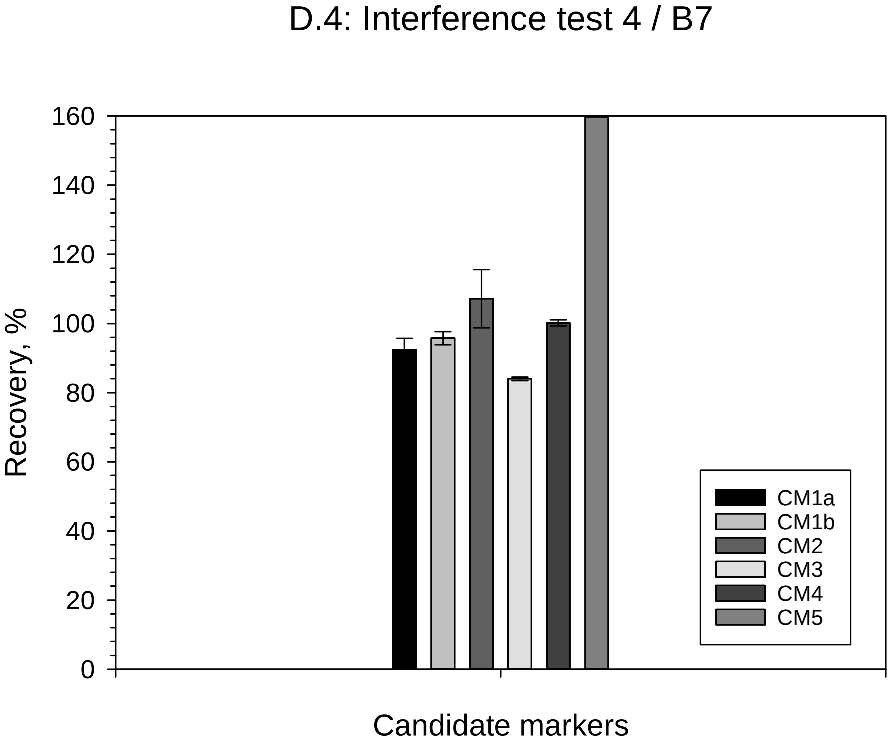


Figure 18a. Graphical results show the effect of the presence of other fiscal markers in the sample in B0 gas oil (mix 5). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.

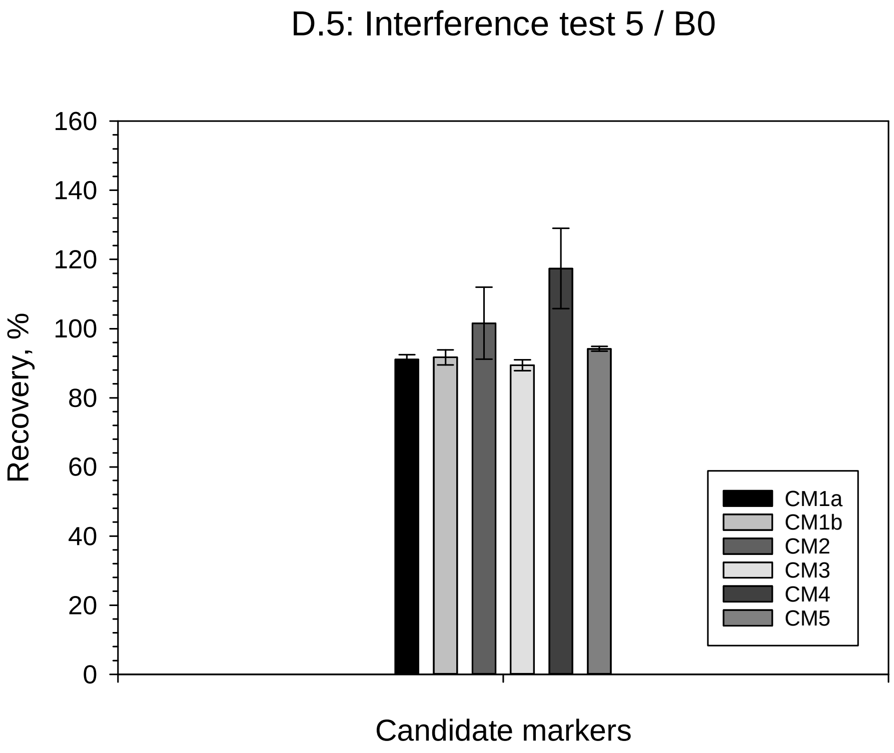
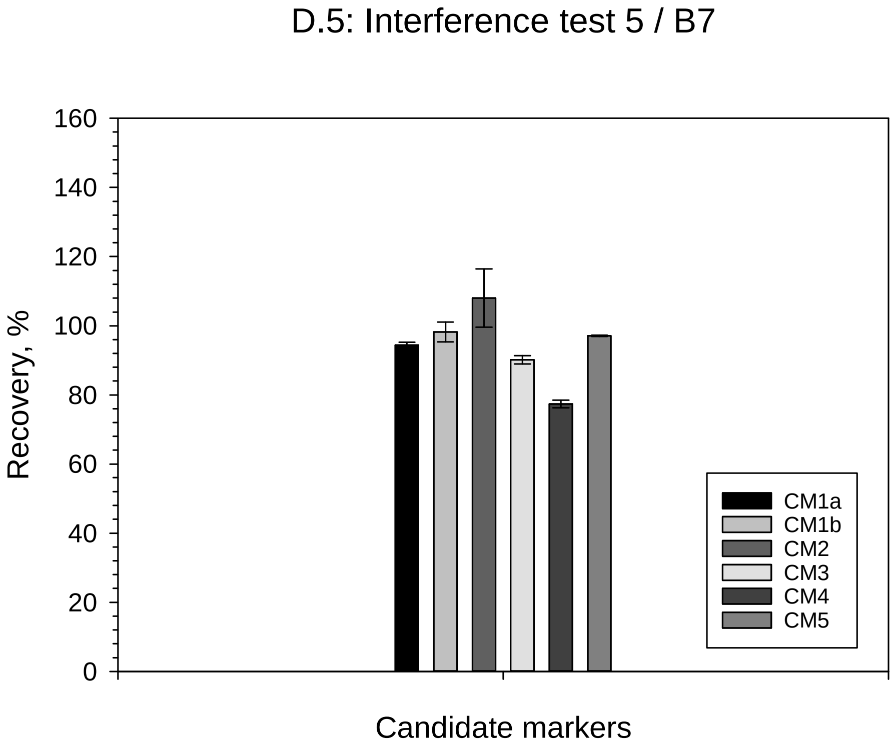


Figure 18b. Graphical results show the effect of the presence of other fiscal markers in the sample in B7 gas oil (mix 5). A 100 % recovery demonstrates little or no interference. Error bars are ± 1 SD for n = 5.



4.1.6 Detectable at a mass concentration of 2 % (E.1.) and roadside testing methods

An independent report was drafted for the evaluation of this technical requirement and was evaluated alongside the capability for roadside tests. The findings are summarised in Table 6.

Table 6. Evaluation of detectability at 2 % of the marking level. The roadside methods proposed for each marker fulfilling (+) and do not fulfil (-) this target are indicated. The ranking of the candidate markers regarding their roadside test cost is also given (1 being the least and 3 the most costly).

CANDIDATE MARKER		Detectability at 2% marking level	Price (ranking)
CM1	CM1b	+	3
	CM1a	-	
CM2		+	2
CM3		+	2
CM4		-	3
CM5		+	1

Solvent Yellow 124 has the quickest, easiest and most affordable roadside test amongst all the markers that fulfil the detectability of the candidate marker at the 2 % of the marking level. CM2 and CM3 require more sophisticated instruments for their detection, which result in higher initial investment in the instruments, and training of the officers that perform the tests.

CM1 and CM4 CMs could not reach the detectability at 2 % of the marking level at the time when the documentation was provided although there is the potential that the required limits can be reached with further method development.

A summary of the estimated costs, in euros, of the analysis of 30 samples per day during a year (365 days) for all the evaluated markers can be found in Table 7 together with the initial investment cost per instrument.

Table 7. Breakdown of the costs (*EUR*) associated with the analysis of samples using the roadside test equipment (as reported by the applicants).

	CM1	CM2	CM3	CM4	CM5
Estimated price of consumables per sample	1.75	0.88	0.56	0.77	0.24*/2.02**
Price of basic instrument	94 783	47 391	46 847	81 700	-
Accessories	41 364[1]	34 949[2]	14 037[3]	600	-

*Based on method SUDAN455-STRE, buying chemicals in bulk, **Based on the analysis of the samples using a commercially available kit. 1) Transport platform, gas generators and one year supply of spare parts. 2) Transport platform, gas generators and one year supply of spare parts. 3) Shipping case, hydrogen/air generator, air compressor and microshot injector.

4.1.7 Stability of markers in the marked fuels during the experiments (F.1.)

Without verified stability of the analyte concentration in the stock solutions, it would be difficult to draw appropriate conclusions from the rest of the experiments. A low recovery from an experiment could be a result of a changing concentration of the marker in the stock solutions in comparison with the initial levels.

The multitude of samples prepared during these experiments resulted in an experimental phase of more than four months. The reference samples were taken from the original preparations **at the same time** when a particular set of experiments was performed. In total, there were five different sets of reference samples collected at different time-points per tested marker and set aside at room temperature. (The five different sets of tests are listed in Table 1 with codes A1-5, B1-2 and C1-4 and D1-5.) The sixth time-point (F1) corresponds to the stability samples taken at the end of the experimental phase in January 2017. By plotting the measured concentrations of the candidate markers in the reference samples as a function of time, it can be established if the concentrations were the same or very similar to the theoretical values given in Table 3.

All tested reference samples had the same age originating from a preparation in July 2016. No special sample conservation or isochronous scheme for stability testing was applied. The laboratories used freshly prepared calibration solutions at the time of the analysis, which took place after the reception of the experimental samples in December 2016 to March 2017. Therefore, it should not be expected to detect any trend over time for the reference samples but rather just a confirmation of the theoretical concentration. Figures 19a to 20b indeed confirm the theoretical concentration levels of the markers, taking the variation of the measurements into account.

From these graphs, it also appears as if CM3 and CM5 can be measured with higher precision than CM1a, CM1b+CM2 and CM4. This can be explained by inherent properties of the measurement techniques and internal calibration approaches.

Figure 19a: Concentration of CM1a, CM3 and CM4 as a function of time in B0 gas oil. The error bars are ± 1 SD, (n = 5).

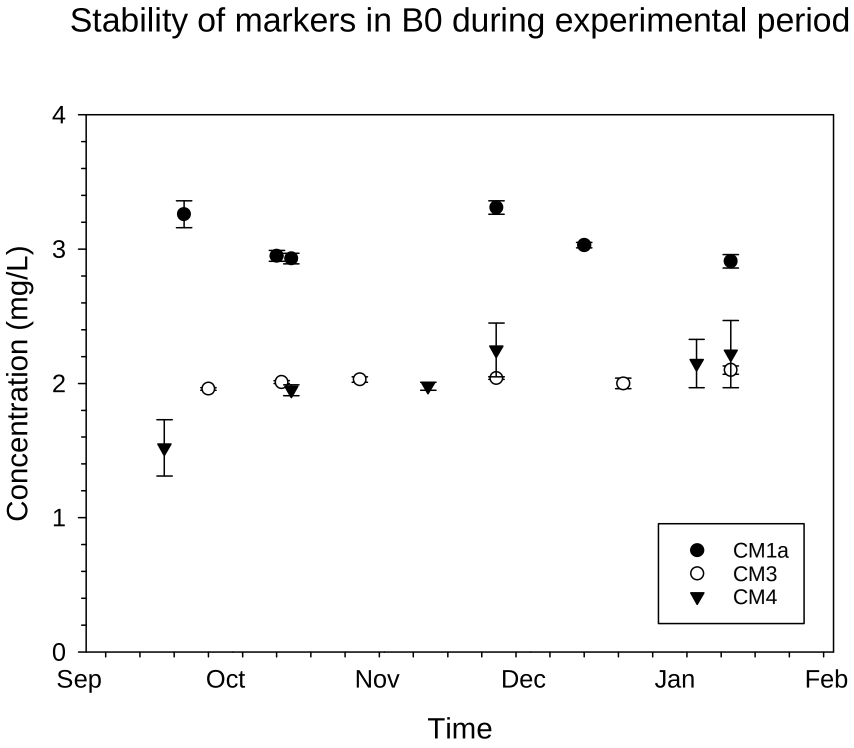


Figure 19b: Concentration of CM1b, CM2 and CM5 as a function of time in B0 gas oil. The error bars are ± 1 SD, (n = 5).

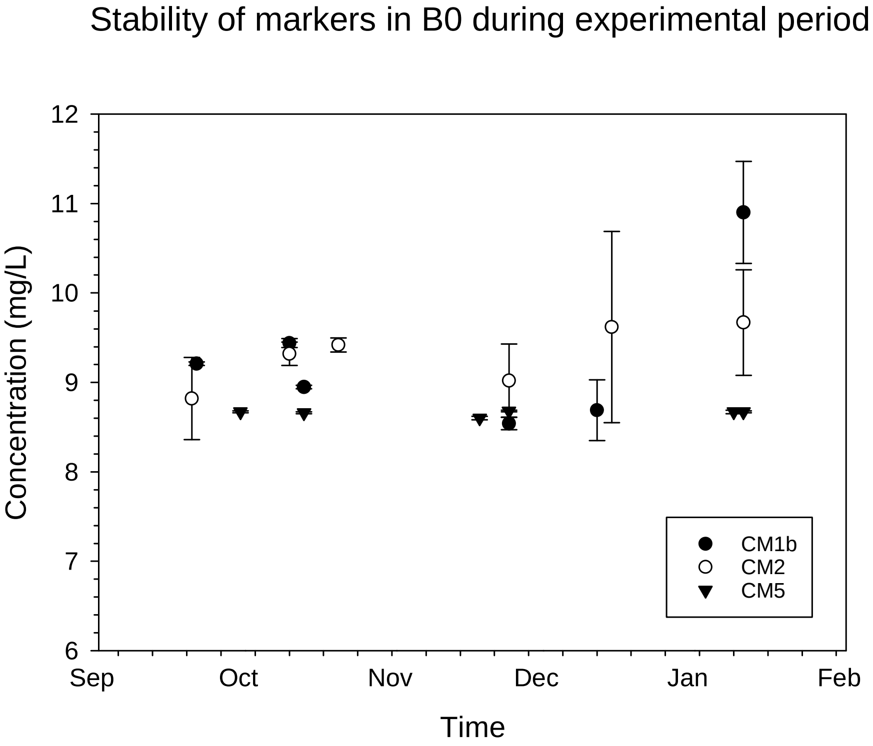


Figure 20a: Concentration of CM1a, CM3 and CM4 as a function of time in B7 gas oil. The error bars are ± 1 SD, (n = 5).

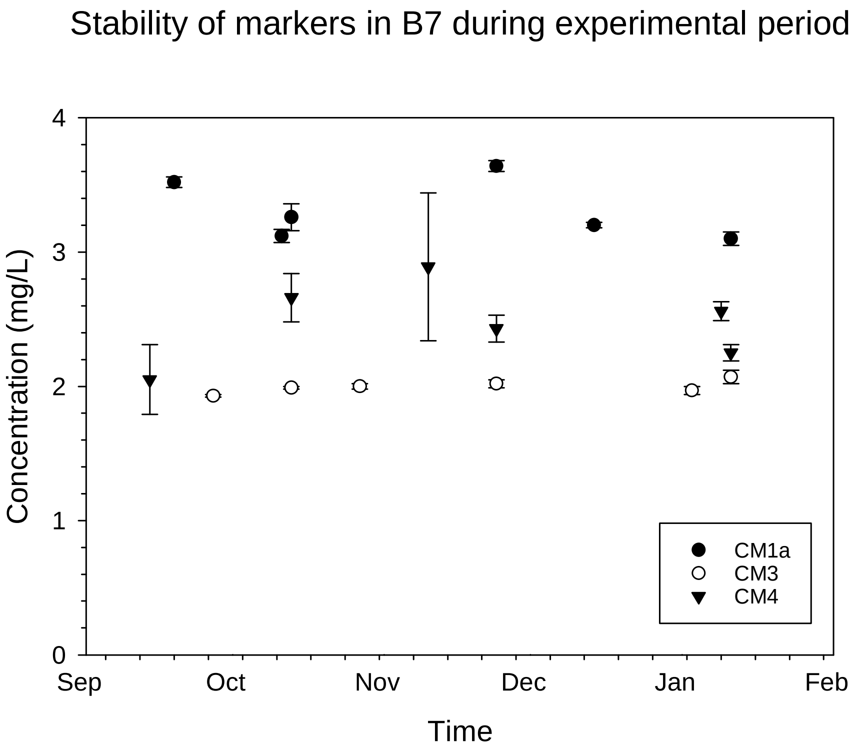
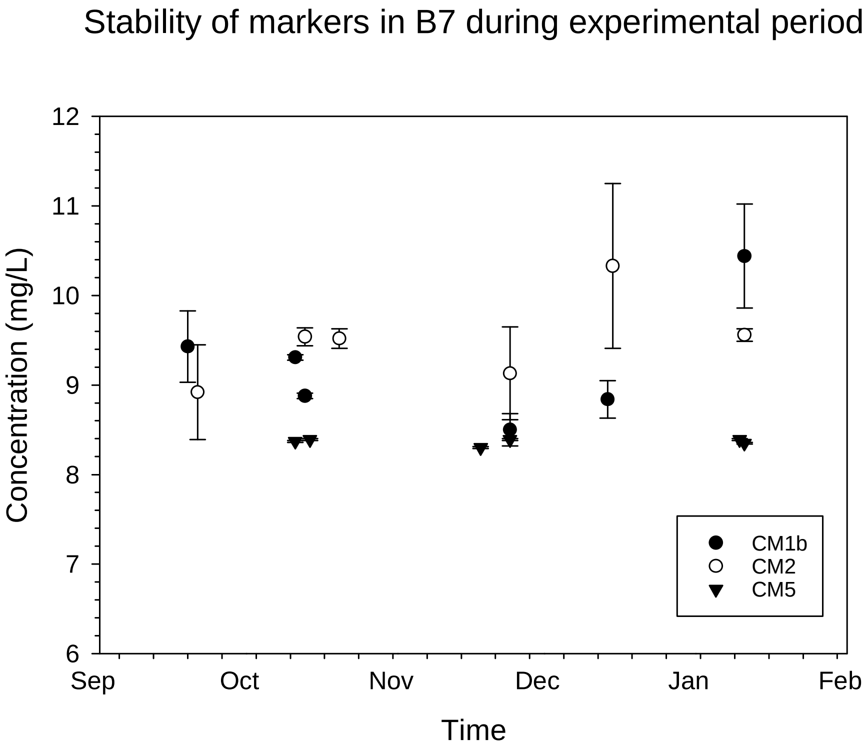


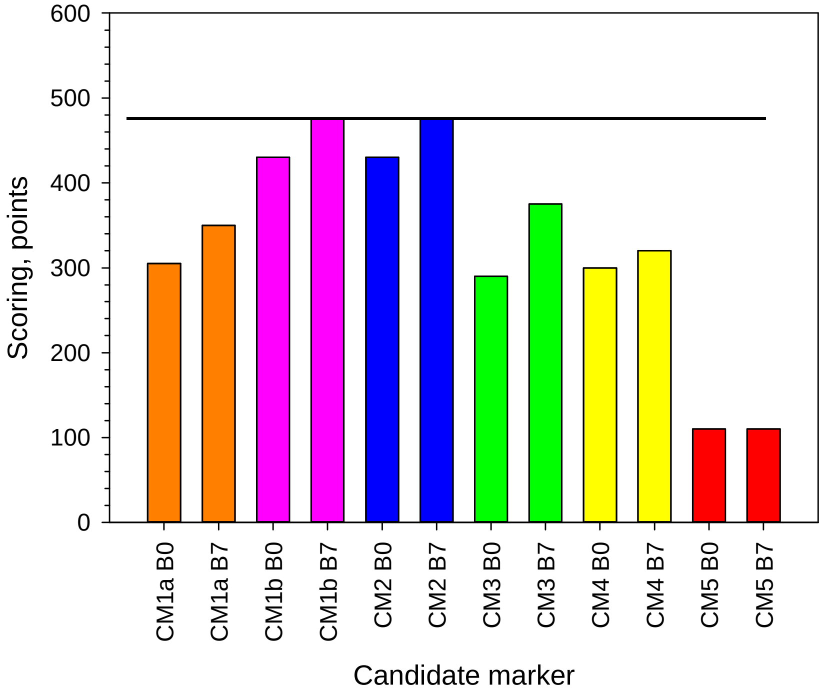
Figure 20b: Concentration of CM1b, CM2 and CM5 as a function of time in B7 gas oil. The error bars are ± 1 SD, (n = 5).



4.2 Performance of the candidate markers against SY124

A graphical representation of the scoring can be seen in Figure 21 with the full score of 475 points per fuel quality indicated as a horizontal line. Marker CM1b and CM2 (which is the same compound) performed flawlessly when added to the gas oil of B7-quality since none of the treatments had an effect on the marker. Irrespective of whether being added to B0 gas oil or B7 gas oil, this marker outperforms the other candidate markers since it is **the only marker** that is resistant towards a particular physical process (experiment B.1). However, since the CM1b / CM2 marking system shows some weaknesses in B0 gas oil (see below), an option **could be** a dual marking system comprising two compounds even if only CM1b / CM2 is un-affected by the physical treatment (i.e. the fuel remains marked after treatment). A dual marking system is nevertheless even more rugged with respect to the physical process applied in experiment B.1., mainly depending on which operation modus is applied.

Figure 21: Graphical representation of scoring, horizontal line depicts the maximum score of 475 points per fuel quality B0 / B7 gas oil.



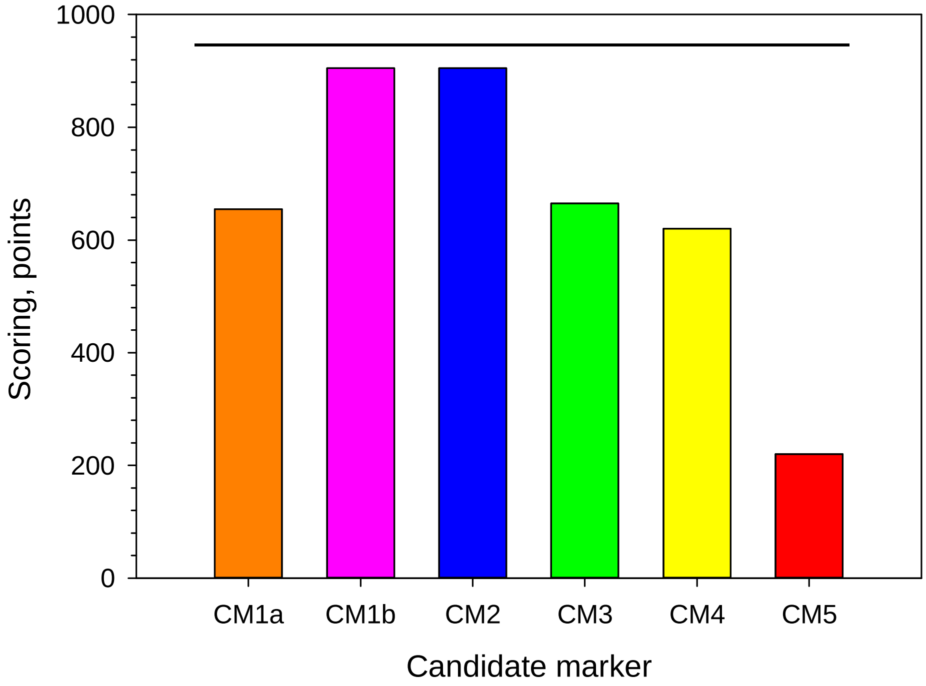
Consequently, when CM1b and CM2 are added to B0 gas oil, the results are somewhat worse since it appears as if adsorption on adsorbent 5 (test A.5) and resistance towards chemical 3 (C.3) could be a problem for this marker (CM1b and CM2 reaches 430 of 475 points in B0 gas oil). However, the results of adsorption on adsorbent 5 are associated with an enormous variability for the CM2 marker and a high variability and low recovery when used in the CM1 marking system (which contains the additional marker CM1a, which is also affected by adsorption on adsorbent 5. Moreover, it is likely that treatment with chemical 3 is unrealistic in fraudulent activity although it has had a clear impact on all markers. Hence, no marking system performs flawlessly when added to B0 gas oil. In B7 gas oil, the presence of 7 % biodiesel in the gas oil protects the markers to some extent because the fatty acid methyl ester is possibly reacting preferentially with chemical 3 on the one hand and changes adsorption properties on adsorbent 5 on the other hand.

As concerns the observed problem with adsorbent 5 in B0 gas oil, several options are possible. It is possible to perform more tests on different categories of adsorbent 5 and check break-through volumes for CM1a and CM1b and / or use another marker together with CM1b and CM2 that is not sensitive towards adsorption on adsorbent 5 in B0 gas oil. By finding out the break-through volumes, it would be possible to assess the amounts of adsorbent necessary for laundering of a particular volume of fuel in determining the economic viability of fraudulent activity.

It should, however, be realised that the effect of adsorption of CM1 / CM2 on adsorbent 5 is not nearly as severe as for the adsorption of SY124 in general (see scoring tests A.1 to A.5 in Table 9). Even when using adsorbent 5 on marked B0 fuels, it is likely that it would be difficult to efficiently remove the CM1/CM2 marking system below a level of 2 % (of the marking level) and to make this into a lucrative fraudulent activity. For this reason, the conclusion is that the observed weakness of CM1b and CM2 on adsorbent 5 is acceptable.

Figure 22 shows the grand total of points per marker by simply adding the points together for both fuel qualities. In this graph, the maximum score is 950 points with consequently no distinction made based on fuel quality. As can be seen, no marking system reaches the maximum score. CM1b and CM2 accumulate 905 points, which is more than 95 % of the available points.

Figure 22: Graphical representation of scoring. The horizontal line depicts the maximum score of 950 points per marking system for both fuel qualities.



The poor result of the the current Euromarker (CM5) is remarkable, and this essentially underscores the need for changing the pan-European marking system for gas oil and kerosene. With a total of 220 points, it accumulates less than 25 % of the available points. All new candidate markers outperform the current Euromarker. Perhaps, the most striking weakness of the current Euromarker is its high sensitivity towards a physical process B.2. whereas none of the other markers is affected.

For a marking system based on CM2, the costs for implementation are estimated at EUR 47 391 per roadside test instrument based on Portable MDGC with a heart-cutting event and two FID. For the CM1 system (which is a dual marker), the cost for implementation would be EUR 94 783 per roadside test instrument to measure both components at the roadside. For the latter system (for CM1), accessories for an additional EUR 41364 are necessary whereas the accessories for the first system (for CM2) cost EUR 34 949. These accessories (transport platform, gas generators and one year supply of spare parts) are seemingly essential for the proper functioning of the field equipment. Also, training would be necessary for the officers performing these tests in the field. Once the initial capital investment has been made, the cost in using the field-equipment would range from EUR 0.88 to 1.75 per test for the CM2 and CM1 system, respectively.

Should a decision be taken to replace the current Euromarker, a price reduction for the instrumentation and accessories for analytical testing in the field could be anticipated because of an expansion of business opportunity on a competitive EU-wide market. Currently, no instrumentation is needed for the road-side test for the Euromarker and is only based on a simple chemical test (EUR 0.24 to 2.02 per test). Obviously, more expensive equipment based on HPLC-UV is required for measuring this marker in the control laboratories. At the time of procuring the analyses for the candidate markers CM1 to CM3, it also became evident that the sophisticated GC-MS instrumentation with back-flush options and Dean's switch (or similar) was lacking in many official control laboratories. Access to this kind of equipment must also be considered in the event of replacing the current Euromarker. Such instruments are not limited in use to fuel-markers, but can also be used for measuring and identifying drugs, explosives and many other compounds of potential interest to the authorities. The costs associated with new marking systems are not negligible and must be weighed against the massive losses of tax revenues reported by some Member States. Apparently, the ruggedness and usefulness of SY124 as a marker is quite limited, and superior alternatives are available.

In Table 8 and Table 9, all recoveries and results of scoring are presented for the five different markers tested. Table 8 constitutes the basis for Figures 3a to 18b while Table 9 constitutes the basis for Figures 21 and 22.

Table 8. Recoveries calculated for CM1a, CM1b, CM2, CM3, CM4 and CM5 in B0 and B7 gas oil with five replicates per experiment. Values in bold are below 80 % recovery which is the threshold for a full score (see Table 9).

Test	CM1a		CM1b		CM2		CM3		CM4		CM5	
	B0	B7	B0	B7	B0	B7	B0	B7	B0	B7	B0	B7
A.1	86.7 ± 2.7	94.0 ± 0.6	88.0 ± 2.2	100.9 ± 0.7	88.5± 1.4	100.4 ± 1.0	**38.8 ± 7.7**	95.8 ± 6.5	98.8 ± 7.4	102.7 ± 4.8	**0**	**0**
A.2	97.4 ± 1.4	96.3 ± 1.0	104.9 ± 0.7	106.8 ± 1.0	100.1 ± 2.1	100.8 ± 1.3	92.0 ± 0.9	100.2 ± 0.5	102.1 ± 2.5	121.5 ± 6.5	**6.7 ± 9.6**	**25.4 ± 14.3**
A.3	92.8 ± 1.1	97.9 ± 0.7	99.1 ± 0.7	106.6 ± 0.5	94.7 ± 1.4	101.3 ± 1.4	**67.7 ± 4.7**	100.4 ± 0.7	138.5 ± 7.7	101.4 ± 10.1	**10.0 ± 9.0**	**20.7 ± 8.8**
A.4	95.2 ± 0.8	97.0 ± 1.0	97.0 ± 1.3	101.4 ± 1.3	93.3 ± 1.3	98.5 ± 2.8	86.5 ± 1.4	96.5 ± 1.0	92.2 ± 3.7	98.1 ± 1.4	**10.7 ± 12.6**	**16.0 ± 18.3**
A.5	**37.2 ± 12.8**	95.0 ± 0.6	**14.3 ± 11.2**	102.6 ± 0.6	**75.0 ± 39.0**[1]	100.1 ± 1.2	**48.4 ± 10.3**	100.6 ± 0.5	124.9 ± 1.9	84.2 ± 1.3	**15.4 ± 8.1**	**65.2 ± 3.0**
B.1	**3.4 ± 1.7**	**3.5 ± 2.0**	107.4 ± 0.5	108.1 ± 1.0	111.7 ± 0.7	112.0 ± 1.3	**2.6 ± 0.2**	**2.7 ± 0.1**	**4.5 ± 2.8**	**3.0 ± 1.7**	**0.9 ± 0.1**	**0.9 ± 0.1**
C.1	106.2 ± 0.6	101.0 ± 1.9	95.2 ± 0.4	99.9 ± 4.6	97.9 ± 3.2	99.0 ± 3.2	99.4 ± 0.9	98.1 ± 0.8	129.0 ± 1.9	92.0 ± 1.8	**0**	**0**
C.2	110.1 ± 2.0	105.2 ± 1.5	102.8 ± 0.2	103.0 ± 0.6	99.9 ± 1.0	100.2 ± 1.4	99.1 ± 0.9	98.6 ± 0.5	147.9 ± 15.8	111.1 ± 9.2	99.8 ± 0.1	99.5 ± 0.1
C.3	**0**	86.3 ± 2.8	**0.9 ± 0.5**	90.4 ± 5.6	**0**	83.7 ± 2.2	**0**	88.0 ± 0.9	**11.1 ± 4.4**	134.0 ± 12.6	**0**	**0**
C.4	106.4 ± 1.0	102.6 ± 3.6	101.2 ± 0.7	100.6 ± 0.6	96.2 ± 0.7	97.6 ± 2.3	96.9 ± 1.2	96.1 ± 0.8	**68.7 ± 5.4**	90.3 ± 9.0	**0**	**0.3 ± 0.1**
B.2	120.2 ± 1.5	118.0 ± 1.4	97.1 ± 10.9	104.5 ± 11.8	94.9 ± 1.0	92.4 ± 5.9	101.9 ± 1.3	102.6 ± 1.1	160.5 ± 7.9	128.6 ± 17.3	**0**	**0**
D.1	104.7 ± 0.6	108.4 ± 1.1	96.6 ± 2.3	98.7 ± 10.0	96.0 ± 4.4	94.1 ± 2.6	95.7 ± 0.7	93.2 ± 0.6	120.0 ± 1.1	103.2 ± 10.7	95.9 ± 0.3	100.5 ± 0.3
D.2	105.8 ± 3.8	107.1 ± 1.3	97.0 ± 4.5	97.7 ± 8.1	93.6 ± 5.0	90.3 ± 0.4	93.9 ± 0.8	88.3 ± 0.3	97.6 ± 3.6	91.4 ± 13.3	96.5 ± 0.1	100.8 ± 0.1
D.3	103.0 ± 5.0	95.9 ± 0.7	99.1 ± 3.9	97.0 ± 3.7	95.5 ± 6.7	112.8 ± 7.3	90.1 ± 2.4	83.7 ± 1.1	126.9 ± 2.3	**78.7 ± 0.9**[2]	95.6 ± 0.2	99.0 ± 0.1
D.4	91.5 ± 3.4	92.5 ± 3.2	94.0 ± 1.9	95.8 ± 1.9	108.6 ± 9.8	107.2 ± 8.4	87.9 ± 0.6	84.0 ± 0.5	94.6 ± 8.1	100.2 ± 0.9	**157.0 ± 0.3**[3]	**163.0 ± 0.4**[3]
D.5	91.1 ± 1.4	94.4 ± 0.8	91.7 ± 2.2	98.2 ± 2.9	101.6 ± 10.4	108.0 ± 8.4	89.4 ± 1.6	90.2 ± 1.2	117.4 ± 11.6	**77.4 ± 1.1**[2]	94.2 ± 0.7	97.1 ± 0.2

(1) The result is affected by an extreme variability, as one replicate was close to 140 % recovery while another was around 40 %.

(2) With CM4, the analytical variability was high as shown in Fig 19a and 20a. Due to these random fluctuations, relatively low results can occur, leading to recoveries below 80 %. An actual analytical interference is not imminent because of the high chromatographic resolution and the high specificity of the detection of this marker.

(3) The recoveries are well above 100 %, which in other cases have **not** resulted in a zero score. Despite several additional experiments, no explanation can be given for the high recoveries and also, given the high precision of the measurements of CM5 in general. Therefore and due to the non-specific detection of CM5, it was decided to give zero points because of these results.

Table 9. Scoring for markers CM1a, CM1b, CM2, CM3, CM4 and CM5 in B0 and B7 gas oil.

	Test	Test	Max points	CM1a B0 / B7	CM1b B0 / B7	CM2 B0 / B7	CM3 B0 / B7	CM4 B0 / B7	CM5 B0 / B7
A REMOVAL TESTS 200 p. per fuel quality	Adsorption	A.1	20	20 / 20	20 / 20	20 / 20	0 / 20	20 / 20	0 / 0
		A.2	20	20 / 20	20 / 20	20 / 20	20 / 20	20 / 20	0 / 0
		A.3	20	20 / 20	20 / 20	20 / 20	0 / 20	20 / 20	0 / 0
		A.4	20	20 / 20	20 / 20	20 / 20	20 / 20	20 / 20	0 / 0
		A.5	20	0 / 20	0 / 20	0 / 20	0 / 20	20 / 20	0 / 0
	Physical process	B.1	100	0 / 0	100 / 100	100 / 100	0 / 0	0 / 0	0 / 0
B BREAK DOWN TESTS 175 p. per fuel quality	Chemical treatment	C.1	25	25 / 25	25 / 25	25 / 25	25 / 25	25 / 25	0 / 0
		C.2	25	25 / 25	25 / 25	25 / 25	25 / 25	25 / 25	25 / 25
		C.3	25	0 / 25	0 / 25	0 / 25	0 / 25	0 / 25	0 / 0
		C.4	25	25 / 25	25 / 25	25 / 25	25 / 25	0 / 25	0 / 0
	Physical process	B.2	75	75 / 75	75 / 75	75 / 75	75 / 75	75 / 75	0 / 0
C ANALYTICAL ASPECTS MAX 100 p. per fuel quality	Interference test	D.1	15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 15
		D.2	15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 15
		D.3	15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 0	15 / 15
		D.4	15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 15	0 / 0
		D.5	15	15 / 15	15 / 15	15 / 15	15 / 15	15 / 0	15 / 15
	Detectable at concentration of 2% marking level	E.1	25	0 / 0	25 / 25	25 / 25	25 / 25	0 / 0	25 / 25
D STABILITY	Stability of the samples	F.1	Additional check, no scoring	OK	OK	OK	OK	OK	OK
SUM per fuel quality				305 (B0) 350 (B7)	430 (B0) 475 (B7)	430 (B0) 475 (B7)	290 (B0) 375 (B7)	300 (B0) 320 (B7)	110 (B0) 110 (B7)
Total SUM				655	905	905	665	620	220

5 Conclusions

- All tested candidate markers perform much better than the current Euromarker.

- One marker (CM1b and CM2) can resist all treatments when added to the B7 gas oil.

- When added to the B0 gas oil, CM1b and CM2 outperform all the other markers since it is the only that is un-affected by physical process B.1 since the fuel remains marked after treatment. However a dual marking system (CM1) is more rugged with respect to the physical process B.1., mainly depending on which operation modus is applied. Additional experiments could potentially resolve this issue.

- A dual marking system like CM1 is technically more complicated and more expensive to implement. In most cases, the CM2 marking system is very resistant to most treatments with a lower cost for implementation than for CM1.

- The CM1 / CM2 marking system shows some weaknesses related to adsorption on Adsorbent 5 and treatment with chemical 3 in B0 gas oil. In practice, it seems unlikely that an economically lucrative activity can be based on the use of adsorbent 5 for the new marker(s) because considerable amounts of marker still go through the adsorbent. To efficiently remove these markers, below 2 % of the marking level is therefore difficult. Provided that the detection power of the analytical systems used at the road side is high enough, traces of marker would most likely be detectable even after attempts of laundering fuel tagged with the CM1 / CM2 marking system using adsorbent 5.

- A full validation of the analytical techniques used for measurements of the markers in B0 and B7 gas oil is an absolute requirement. The method for CM4 has a high variability. Random fluctuations have probably led to relatively low results, with recoveries below 80 % which was the cut-off for a full score in two cases. It is likely that there is no real effect of interference in mixture 3 and mixture 5 in B7 gas oil for CM4. Consequently, this marker probably qualifies for another 30 points, but this will not change the overall conclusions of this study.

- The precision of the measurements could also be improved considerably e.g. by employing calibration based on internal standards for CM1, CM2 and CM4.

- A few additional experiments could shed light on a few remaining issues such as the susceptibility of marker breakdown in the presence of chemical 3 and adsorption behaviours on different types of adsorbent 5 for markers CM1a, CM1b and CM2. Alternative application modus of physical process B.1. will also be investigated.

- A certified reference material would greatly facilitate the implementation of a new marking system for gas oil and kerosene just like in the case of the current Euromarker.

- Following the results obtained in this study, it is recommended to replace the current Euromarker with CM2 provided that it can be shown that this compound is not present in non-marked fuels originating from multiple crude oil fields.

6 Final remarks

Full experimental and technical details have not been disclosed in this report because of the sensitive nature of the information gathered and because of confidentiality agreements with the tendering companies that have submitted candidate markers for further testing. A full report containing all information, experimental details and analytical results has already been provided to DG TAXUD to present the necessary scientific evidence. The current report has been assembled for the benefit of the tenderers and for the public to be informed about the process, results and conclusions as comprehensively as possible without disclosing sensitive information.

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