### **19ENG09 BIOFMET**





# FINAL PUBLISHABLE REPORT

Grant Agreement number	19ENG09
Project short name	BIOFMET
Project full title	New metrological methods for biofuel materials analysis

Project start date and duration:		1 June 2020, 36 months			
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Project website address: http://www.bio	ofmet.eu				
Internal Funded Partners:	External Funded Par	tners:	Unfunded Partners:		
1. DTI, Denmark	8. AMU, France				
2. BRML, Romania	9. CTU, Czech Rep	ublic			
3. CETIAT, France	10. IST, Portugal				
4. CMI, Czech Republic	11. PROMETEC, Fir	land			
5. IMBiH, Bosnia and Herzegovina	12. VERDO, Denma	rk			
6. PTB, Germany					
7. TUBITAK, Türkiye					
RMG: -					

Report Status: PU Public

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Final Publishable Report



#### TABLE OF CONTENTS

1	Overview	3
2	Need	3
3	Objectives	3
4	Results	4
5	Impact	. 20
6	List of publications	. 22
7	Contact details	. 22



### 1 Overview

Biofuels, originating from biomass, can be defined as fuels produced by means of technologically advanced biological processes, and are a key factor in supporting EU aims of reducing greenhouse gas emissions while diversifying the energy fuel supplies. Reliable information on the nature and quality of the biomass or biofuel used is important to support the optimisation of their combustion with respect to higher efficiencies and lower emissions in energy production. In this project, more accurate measurement methods were developed and advanced traceable measurement standards for the determination of biofuel calorific value and for determining impurities and ash content were established. Three new reference materials were developed and produced to make end-users able to calibrate their equipment. Significant improvements to the method applied for bomb calorimetry has been made. In addition, two new moisture transfer standards have been developed and calibration facilities are in place and ready for end users. Automatic sampling for solid biofuels has been researched and a new device made commercially available for safe and representative sampling. These results will aid enable a fair trade between biofuel suppliers and users and with that a reduction of disputes.

# 2 Need

United Nations has included efficient utilisation of renewable energy sources such as biomass in its sustainable development goals for 'Affordable and Clean Energy', 'Industry, Innovation and Infrastructure', 'Sustainable Cities and Communities', 'Responsible Consumption and Production' and 'Climate Action'. The EU aims for biomass to increase to at least a 27% share of renewable energy consumption. Currently the total primary production of renewable energy based on biomass is about 130 kilotons of oil equivalent annually. One of the goals in the revision of the EU Energy Taxation Directive is that heat production taxes are calculated using estimated energy content of solid biofuels. This estimate is subject to errors caused by non-representable sampling and measurements based on non-ideal, slow, offline methods. This has major economic consequences.

The rapidly increasing use of biomass impacts international trade, whilst the need for optimal fuel utilisation, the minimisation of emissions and increased sustainability of the supply have increased the need for fast, reliable, traceable measurements and characterisation of biofuels as essential for this development and requires:

- Knowledge of the accurate content of water in solid biofuel materials such as wood pellets or wood chips is important for ensuring optimal combustion efficiency and fair payment of the fuel.
- Improved sampling methods for solid biofuels to reduce the significant associated errors in fuel quality control which can lead to error in moisture content measurement.
- Knowledge of the ash content for the accurate determination of its calorific value thereby establishing the energy content of the fuel.
- Knowledge and reliable methods for determination of the level of impurities in biofuels which are essential to limit precipitation of solids in the fuel transportation system and to establish the energy content of the fuel and regulate its combustion, as well as for fiscal billing of the fuel.

Achieving such measurements has been a significant challenge given the use of adapted and slow offline methods and documentary standards that originally was developed for measurement of coal and are unverified for biofuels and insufficient for understanding the contents of these fuels. Traceability will provide the legal and financial regulatory means for "trackability" and determining the calorific value, as well as the bio-origin of the samples/blends and their carbon dioxide contribution.

# 3 Objectives

The overall objective of the project was to research online metrological methods for the analysis of solid and liquid biofuels, such as wood pellets and fatty acids methyl esters (FAME, a form of liquid biofuel), with the purpose of establishing traceable calorific values and ensuring optimal combustion. The end goal was to provide metrologically sound data sets for a larger fraction of biofuels, hence providing background information for their more efficient use.

The specific objectives were:



- 1. To develop traceable online measurements for water content in solid biofuels for the measurement ranges 5-12 % (wood pellets) and 20-75 % (wood chips) with a target uncertainty of 5 %.
- 2. To develop improved methods for the sampling of biofuels (i.e., in cases where online methods cannot be used). This should include using data-science techniques such as machine learning to optimise calibration curves and uncertainties.
- 3. To traceably determine the calorific value of the solid biofuels by developing validated methods for the online measurement of ash content. In addition, to develop accurate methods < 0.1 % absolute repeatability and < 0.2 % absolute reproducibility (as per EN ISO 18122) for determining the amount and composition of ash content in the measurement range 0-1.2 %.
- 4. To develop validated methods to determine the amount and nature of impurities in liquid biofuels, including quantifying and qualifying inorganic (i.e., Na, Ca, K, Mg, P) for a measurement range of 0.5 mg/kg to 2.5 mg/kg with a relative target uncertainty of 5-10 % (*k*=1) and organic by-products (i.e., total glycerol) < 5 % relative repeatability for 0.1-0.3 % range as per EN ISO 14105. In addition, to develop a traceable method for the online determination of the calorific value of liquid biofuels.</p>
- 5. To disseminate and facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, standards developing organisations (CEN TC 335, CEN TC 19) and end users (e.g. the European Technology and Innovation Platform (ETIP) and European Biodiesel Board (EBB).

# 4 Results

In the following the results of the research and testing performed in project is summarised. Supplementary information in the form of reports, open access papers and videos demonstrating the developed methods are available via the project website www.biofmet.eu.

# Objective 1: " To develop traceable online measurements for water content in solid biofuels, for the measurement ranges 5 % - 12 % (wood pellets) and 20 % - 75 % (woodchips) with a target uncertainty of 5 %."

Knowing the content of water in solid biofuel materials such as wood pellets or wood chips is important for two main reasons, that is ensuring optimal combustion efficiency and fair payment of the fuel. If the combustion cannot be regulated according to the water content, the worst-case scenario is that the boiler will not be able to combust the material, leading to shut down of the Combined Heat and Power (CHP). Therefore, there exists a pronounced requirement for representative measurements of the water content of the biofuel material. It is very valuable to have online data available for both producers and consumers.

During the project, the consortium had a unique opportunity to demonstrate the applicability of the developments by implementing the "mise en pratique" of the entire traceability chain from the reference method to the end-user level. Thus, the project has covered all the steps needed for traceable online measurements of water content. Reference methods for water content have been developed at DTI and CETIAT and validated by inter-laboratory intercomparison an (ILC). Simultaneously, two transfer standards were developed by CMI, CTU and CETIAT and calibrated against these reference methods, and their consistency was demonstrated during the ILC. Subsequently, a measurement campaign involving CETIAT, DTI, VERDO and CMI was conducted on a power plant to showcase the applicability of the developed transfer standards and to address the traceability gap when dealing with in-line measurements under industrial conditions.





The metrological basis for traceable methods for measurement of water content are reference methods. DTI and CETIAT have developed reference methods for water content, see Figure 1. In both cases the methods are based on the evolved water vapour (EWV) principle. In EWV, the water is extracted from the sample by applying heat in a flow of dry gas and depending on the method the desorbed water is measured by a  $P_2O_5$  detector (CETIAT) or using a chilled mirror hygrometer (DTI). Both detection methods ensures that only water and all the water from the sample is measured.

The reference method at DTI (see Figure 1) consists of three main parts: A calibrated flow measurement device, an oven and a calibrated dew point hygrometer. Using a sample chamber with an atmosphere of dry air inside the oven, water can be extracted from the sample by heating it up. The supplied dry air is passed over the sample, and the water vapour is transported to the dewpoint hygrometer. By continuously measuring the amount of dry air entering the system and the absolute water concentration of the air exiting the system, the total amount of water extracted from the sample can be determined.

CETIAT employed Evolved Water Vapor Coulometric Analysis as reference method. This technique involves the selective electrolysis of evaporated water from a solid matrix carried in a carrier gas stream. The process begins by heating the sample in an oven to evaporate the water. The water molecules then react with a  $P_2O_5$  coating, forming  $H_3PO_4$ . Within the electrolytic cell, the  $H_3PO_4$  molecules dissociate into hydrogen and oxygen. The electric charge consumed during electrolysis is measured and integrated over time to determine the total charge, which is directly proportional to the water content in the sample according to Faraday's law. This technique allows for selective electrolysis of water molecules, and the current passing through the cell indicates the amount of water present. See Figure 1.

The two reference methods developed thus involved two quite different traceability schemes. To validate the reference methods, they were compared through an inter-laboratory comparison (ILC) involving CETIAT, DTI and CMI (EURAMET project 1560). In parallel with the establishing of the reference methods, two transfer standards were developed and calibrated against these reference methods. Their consistency was also demonstrated during the ILC.

One of the two transfer standards developed by CETIAT within this project uses electromagnetic wave propagation to determine water content (Figure 2, Figure 3). The water content changes the electromagnetic properties of materials and consequently impacts the way electromagnetic waves propagate. CETIAT utilises a resonant cylindrical cavity as a transfer standard, in which the shift of the resonant frequency, measured with a Vector Network Analyser (VNA) is associated with the water content of the material being studied and placed inside the cavity. Alternatively, the dielectric permittivity can also be determined from the resonant cavity and correlated with the water content. In both cases, the calibration curve has been obtained by using reference methods for water content determination, thereby ensuring traceability to the International System of Units (SI). In the work with the prototype, finite element analysis (FEM) was applied to obtain knowledge of all influencing effects inside the cavity under its operation.



Figure 2 Scheme of the measurement system developed by CETIAT.





Figure 3. Photos of the measurement system developed by CETIAT.

Another transfer standard for measuring water content in wood pellets and wood chips was developed by CMI and CTU. It is based on measuring the power loss during the first longitudinal acoustic resonance mode in a cylindrical waveguide and relating it to the water content of a sample. The device employs a stainless-steel cylindrical cavity as a container for the samples. By comparing the sound power measured at the resonance frequency on a wet sample with a reference dry sample, the water content of a sample can be determined.

The device combines acoustic resonances, a cylindrical cavity design, and traceability is obtained using the loss-on-dry method and a calibrated balance. It estimates average moisture levels by analysing the acoustic signal produced when moist woodchips or wood pellets interact with the surrounding air, providing a relatively fast measurement. A principal scheme is shown in Figure 4. Two Brüel & Kjær 4939® ¼" microphones serve to excite and record an acoustic field in the cavity. The source microphone is connected through the GRAS RA0086® Transmitter Adapter and GRAS 14AA® Electrostatic Actuator Amplifier to the HP 33120A® function generator which also provides a reference signal for the lock-in amplifier. Receiving microphone with Brüel & Kjær 2669® microphonic preamplifier is connected through the Brüel & Kjær 2690-A NEXUS® Conditioning Amplifier to the measuring channel of the Stanford Research System (SRS) SR830® lock-in amplifier.



Figure 4. Scheme of the transfer standard based on an acoustic measurement system employing the lock-in amplifier.





Figure 5. Measurement setup for moisture content determination as sketched in **Error! Reference source not found.**, employing the lock-in amplifier.

The method is based on the influence of moisture content on the acoustic properties of wood. Wood chips and wood pellets are used as reference materials to establish the moisture-power loss function of the device. Moisture levels in the samples are achieved by introducing moist air using a gas mixing generator, and measurements are made using the Loss-On-Drying method with a calibrated balance. The measurement setup is shown in Figure 5. The acoustic transfer standard has been tested for wood pellets (Figure 6) and woodchips (Figure 7) provided by DTI.

An article was produced and submitted for publication to the "MDPI Sensors" peer reviewed journal (open access) by CMI. It



Figure 6. Test of the acoustic transfer standard using wood pellet. The graph shows characterization results with standard deviation.

is describing the experimental device for determination of moisture in wooden pellets and woodchips by measuring the power loss at the first longitudinal acoustic resonance mode that is excited in the cylindrical waveguide. Woodchips and wood pellets were used as reference materials. The stainless-steel cylindrical resonance cavity served as a container for measured samples. The ratio of the sound power measured using specific sample moisture to the sound power measured using the reference dry sample at the resonance frequency depends on the content of moisture in a sample. Construction, the principle of operation and uncertainty estimation are also discussed in this paper.

The proposed method has the potential to represent a transfer standard in the form of a non-destructive method of moisture measurement for woodchips and wood pellets. The presented prototype is an example of a financially effective solution of compact dimensions, to measure moisture in solid biofuels by sound. Since the metrological traceability is performed by the Loss-on-Dry method supported by calibrated balance and a set of reference materials, the device described here is a secondary system.





Figure 7. Results of measurement for wood chips with uncertainty estimations, reference material from the DTI.

The complete traceability scheme for obtaining calibration of an inline water-content measurement system was demonstrated at a Combined Heat and Power (CHP) plant in a collaboration between VERDO, DTI, CETIAT and CMI. The metrological traceability was achieved in two ways. Either directly towards a primary reference, or through a transfer standard, which can be transported and used on-site at the CHP. Thus, the use of transfer standard and reference methods was demonstrated to calibrate an inline MW-based sensor at the CHP plant of VERDO in Randers, Denmark. See Figure 8.



Figure 8. Calibration of the inline MW-based sensor for water content at VERDO's CHP plant. Left: measurements using MW sensor; Right: reference measurements using CETIAT's transfer standard.

The direct traceability route produces the smallest achievable uncertainty, however the use of a transfer standard also provided traceability to the hitherto un-traceable measurements and proved significantly less cumbersome for practical industrial applications.

A manuscript describing the reference methods, their validation and the industrial validation is in preparation.

In total, the outputs of this work have resulted in the submission or publication of 6 peer-reviewed articles and at least 4 four presentations during international congresses.

To summarise: Two reference methods using different traceability routes to SI, were developed by DTI and CETIAT and validated by intercomparison. Two transfers standards were developed. One based on using electromagnetic wave propagation to determine water content, developed by CETIAT and one based on an acoustic measurement system developed by CMI and CTU. SI-Traceability of these transfer standards were obtained by comparison with the reference methods using wood pellets and wood chips. Uncertainties below the 5 % targeted was achieved. Finally, the calibration of an online measurement instrument was made at VERDO's CHP plant in Randers, Denmark demonstrating the applicability of the complete traceability chain.

Objective 1 has thereby been achieved.

Objective 2: "To develop improved methods for the sampling of biofuels (i.e. in cases where online methods cannot be used). This should include using data-science techniques such as machine learning to optimise calibration curves and uncertainties."

Typically, if laboratory analysis is required only a minor portion of the material is analysed and, in this case, sampling is critical. The sampling technique employed can have major impact on the total system uncertainty especially if the sample material is heterogenous, which often is the case for biofuel materials. Standards for sampling of biofuels exist (ISO 18135:2017) but are difficult to use in practice, e.g. in the case when truckloads





Figure 9. VTT professional consultant collecting manual samples.

of wood chips are to be investigated, the major contribution to the uncertainty of the measured water-content originates from sampling.

The development of automated sampling systems for biofuel materials in the project together with theoretically based sampling principles provides a solution to the sampling issue that ensures that proper sampling technique can be applied widespread. In the initial phases of the project the metrological foundation for sampling activities was researched. A survey conducted among producers, consumers, and laboratories provided information of stakeholders' requirements. Based on this, prior knowledge of the project partners, and a literature survey a report named "Sampling and Samples Handling Strategies" was produces by DTI, CETIAT, VERDO, and PROMETEC. The report covers the present sampling and sample handling methods. In the report the pros and cons of the methods are discussed, and suggestions made on how to solve

solid biofuel quality controlling issues.

Vast experimental research was made on the power-plant sites of two collaboration partners with two automated samplers (Q-Robots) during the project. The involvement of the power plants was essential since the Q-Robots was installed on both sites. In the project one of the objectives were to develop the sampler's sampling auger to suit better for the biomass material classified as P100 (i.e. the main fraction is less than 100 mm). Two comparison studies were made during the project between samples collected by Q-Robot and collected manually by truck drivers, professionals, and a professional consultant from VTT (Technical Research Center of Finland), see Figure 9. The tests with the Q-Robot were made at two different sites owned by Kuopion Energia and Kainuun Voima using identical Q-Robots. The goal was to find out the reliability of automated sampling with a new 250 mm auger which design was based on a biomass class P100 requirements (Figure 10).

A comparative study carried out by a consultant from VTT that routinely works with sampling according to ISO 18135:2017, was made in November 2020 at Kuopio Energy's power plant. For five days, samples from different types of biofuel loads were collected both using a Q-robot and manually by the VTT consultant. Loadspecific samples were taken daily from all loads delivered during the 10-hour test period. Samples for the comparison were collected from

a total of 59 loads, of which 26 loads were forest residue chips, 20



Figure 10. Prometec's fully automated sampling robot.

loads of whole wood and stem chips, 8 loads of peat and the remaining loads were sawdust and bark.

A second comparative study was made in April 2021 at Kainuun Voima power plant in Kajaani. Samples from different types of biofuel loads were collected in three ways, with a Q-robot, manually according to the documentary standard EN ISO 18135: 2017 and by the truck driver (which is a common way to take samples in Finland). Samples for the comparison were collected from a total of 24 loads, of which 2 loads were forest residue chips, 11 loads of stem wood chips, 11 loads of crushed stumps.

Moisture and particle size distribution were determined from all collected samples in Prometec's laboratory. Results from Kuopion Energia were analyzed by VTT and DTI. Results from Kainuun Voima were analyzed by DTI. The difference in average moisture of all fuel types over the test period between the tested sampling methods was 0.7 percentage points in Kuopio. In Kajaani the moisture difference was 0.3 percentage points between the standardised method and the Q-robot sampling. Results showed that there are no systematic errors made in moisture or in particle size determination when using a 250 mm wide auger compared to when applying the standardised manual sampling method. There is no overall significant difference between the particle sizes in samples obtained using the standardised method and samples obtained using the Q-robot. There are significant (i.e. detectable) differences between particle sizes, when comparing the Q-robot results to samples obtained by the truck driver.



The objective was achieved. With samples taken by Q-Robot it was demonstrated that the sampler collects representative samples according to the documentary standard ISO 18135:2017. The results were reproducible, and no systematic errors were observed i.e., this means that this sampling technique can be used widely.

The results have been communicated to European standardisation in the form of a summarising report with a proposal to include automatic sampling using robots in the next update of the standard. The input included information about how the samples should be collected by using an auger and how samples should be collected from randomly chosen places directly from the load before unloading. Collecting samples before unloading it enables quick moisture measurement for acceptance checking logistic optimising and billing.

DTI, with support from IST, CMI, CETIAT, and VERDO, have developed a method for the analysis of results of moisture content based on machine learning. The use of data-science techniques, in particular machine

learning, for optimising calibration curves for water content to improve uncertainties has been researched.

The technology of machine learning allows to extract the information from so-called big data, and it is possible to find and quantify the effect of correlations between different measurement parameters, which would be difficult or impossible to find otherwise. Thus, machine learning may make it feasible to combine all the readings to determine the water content of biofuel with improved precision compared to using traditional analytic techniques.

The data used to test the machine learning techniques were obtained at a CHP (combined heating and power) plant of VERDO in Randers, where an inline system for water-content measurements is



Figure 11. The use of machine learning for best possible water-content precision of woodchips by combining different sensors (MW + NIR). By splitting the data into several subsets to determine the uncertainty related to machine learning. Standard uncertainty of learning subset (fit): 1.2 %; Standard uncertainty of validation subset (validation): 1.6 %.

employed at the conveyor belt feeding the boilers. The signal of this system is essential for ensuring optimal combustion efficiency. An existing MW (microwave) system for water-content measurements was supplemented with a NIR (infrared) system and sensors for characterization of ambient conditions (relative humidity, sample surface temperature, air temperature). In addition, LoD (loss on drying) reference measurements were performed regularly. Since a large dataset is important for the use of machine-learning algorithms, data recorded during the project was supplemented by historic data (containing fewer parameters, but more measurements), and thus about 1000 reference measurements were made available. The machine-learning algorithms used for the research were extended linear regressions based on physical and virtual parameters.

A simple approach was developed to determine the uncertainty contribution related to a machine-learning. The available data were divided into 2 - 3 subsets: 1) a training set, 2) an optional optimisation set, and 3) a validation set. By using the training dataset to develop the machine-learning model, the validation dataset could be used to determine the uncertainty contribution.

An example of a test combining many physical input data is shown in Figure 11. This turned out to be a very effective approach, since in this case the machine-learning algorithm exploited the correlations between the different data to yield very small uncertainty contribution, 1.6 % abs. Another example, based on a much larger data set (of poorer quality, however) and illustrated in Figure 12**Error! Reference source not found.**, utilized virtual parameters (in the form of combinations of the physical parameters) to exploit possible correlations for enhanced precision. The result was a smaller scatter of the data in the training set, but the test on the validation

![](_page_10_Picture_1.jpeg)

set revealed that uncertainty contribution was not improved significantly. In addition, it was discovered that homogenous distribution of the training data over the measurement range (obtained by reducing the training

set, see Figure 12) was a prerequisite for establishing a good machinelearning model.

The results of these investigations will be reported by CMI in a paper ("A method for the analysis of results of wood moisture content in conveyor belt at power plant using machine learning and artificial intelligence") submitted to AMCTM conference the 2023 (Advanced Mathematical and Computational Tools in Metrology and Testing). In addition, the work has provided essential input to the "Good Practice Guidelines on uncertainty assessment of biofuel measurements" (deliverable D7) that is made available to the public on the BIOFMET web site.

To summarise: A method for improving the sampling of biofuels was developed and validated. The method is based on Prometec's Q-Robot equipped with a 250 mm auger allowing class P100 biomass material to be sampled. Extensive testing at two Finnish powerplants showed that representative

![](_page_10_Figure_6.jpeg)

Figure 12. The use of machine learning for best possible precision of the water-content of woodchips using a MW sensor: The machine learning is based on a combination of the physical parameters (phase, attenuation, and load) and virtual parameters (combinations of the physical parameters).

automatic sampling gave similar results to when manually sampling is performed according to ISO 18135:2017, but in a much more efficient and safe manner. Input has been provided to European standardisation to encourage inclusion of automatic sampling in the next version of the standard based on the research made here. The device has already been commercialised by Prometec. The use of data-science techniques, in particular machine learning, for optimising calibration curves for water content to improve uncertainties has been researched and the developed method tested on data obtained at a CHP (combined heating and power) plant of VERDO in Randers, where an inline system for water-content measurements is employed at the conveyor belt feeding the boilers. The tests showed that applying machine learning has potential in improving the precision of calibration curves for in-line moisture measurement equipment. The work has provided essential input to the "Good Practice Guidelines on uncertainty assessment of biofuel measurements" (deliverable D7) that is made available to the public on the BIOFMET web site (https://doi.org/10.5281/zenodo.8180289).

Objective 2 has thereby been achieved.

Objective 3: "To traceably determine the calorific value of the solid biofuels by developing validated methods for the online measurement of ash content. In addition, to develop accurate methods < 0.1 % absolute repeatability and < 0.2 % absolute reproducibility (as per EN ISO 18122) for determining the amount and composition of ash content in the measurement range 0 % – 1.2 %."

Knowing the calorific value of biofuels is important to establish the energy content and regulate its combustion, as well as for fiscal billing of the fuel. In combination with the calorific value the moisture content of solid biofuels determines the efficiency of the CHP and the biorefinery plants and are used to optimize the processes of such facilities. Therefore, with the increased shift towards a biobased economy, the biomass cost and its physical properties must be precisely determined.

Ash content quantification and its qualification are needed to gain insight in the possibility for slag formation, which may have significant economic consequences related to cleaning up after an incidence and it may lead to serious problems in the stability of the energy supply if a CHP is shut down. In addition, during the handling

#### **19ENG09 BIOFMET**

![](_page_11_Picture_1.jpeg)

of the waste and deposition as such, it is important to have measurement techniques that provide information about the total amount and composition of the ash.

Traceability of the online measured calorific value can be achieved through the reference material developed in the project (reported below) with known energy content. The energy content of the reference materials was determined by bomb calorimetry using benzoic acid as primary calibration standard. To guarantee most accurate measurement values and to create an enhanced technological methodology for the measurements of biofuels' calorific value, in a first stage an interlaboratory comparison on the calorific value of solid and liquid biofuels was realized with three different project partners, namely PTB, BRML and TUBITAK participating (with supplementary data by DTI). The calorific value of three solid biofuels, i.e., wood pellets, high-quality wood chips and low-quality (industrial grade) wood chips as well as of one liquid biofuel (biodiesel) was determined following strategies defined in the standards ISO 18125:2017 and ASTM D240-19, respectively. Based on the results an improved measurement strategy was developed addressing specific biofuel related problems usable for potentially updating the respective standards in the future (input has been provided to European standardisation). Using this improved measurement strategy, the measurements were repeated, and the final measurement results contributed to reduce the repeatability and the reproducibility of the measured data. See Figure 13.

![](_page_11_Figure_4.jpeg)

Figure 13: Graphical summary of the calorific value measurement results obtained for solid biofuels following ISO 18125:2017 (left; dark grey framed panels) and using the updated procedure (right; light grey framed

#### **19ENG09 BIOFMET**

![](_page_12_Picture_1.jpeg)

panels). Black point represents results on wet basis, red symbols show results on dry basis. It becomes obvious that with the improved strategy the spread between the three participating institutes became less.

Additionally, a thorough uncertainty budget (Figure 14) has been developed and incorporated for the calculation of the calorific value. Finally, a maximum expanded uncertainty of around  $\pm 1$  % (*k*=2, 95 % coverage) was achieved, indicating a major improvement compared to the 2-5 % uncertainty typically attained

![](_page_12_Figure_4.jpeg)

Figure 14. Uncertainty sources associated with the determination of the calorific value. The determined and by the project partners (PTB, BRML, TUBITAK) commonly agreed uncertainty sources lead to more realistic uncertainty budget.

in academic and industrial laboratories. "The results of the comparison were published as an peer-reviewed article in Energies (D6) accessible via the journal's website (<u>https://doi.org/10.3390/en15082771</u>) or via the project web site.

BRML-INM (Romania), TUBITAK UME (the Republic of Türkiye), IMBiH (Bosnia and Herzegovina), and BAM (Germany) assessed the content of impurities in biofuels by different, complementary approaches. Those partners have developed and validated various analytical methods and performed measurements of organic and inorganic impurities in solid and liquid biofuels.

These measurements were performed on three solid biofuels and one liquid biofuel. The solid biofuels under investigation were high quality wood chips (WC-HQ), industrial grade wood chips (WC-IQ) and wood pellets (WP). The samples were treated to reduce the moisture content to about 15 % and were delivered by DTI to the institutes in airtight inert bags, where each package containing about 1 kg of sample.

Regarding the liquid biofuels to be investigated, biodiesel was selected, and the samples were delivered by BRML-INM (Romania) in brown glass bottles of 1 liter. The gas chromatography - mass spectrometry (GC-MS) has been used as analytical technic for the determination of organic impurities and major components. In order to determine the inorganic impurities, the following methods were used: Inductively Coupled Plasma - Mass Spectrometry/Optical Emission Spectroscopy (ICP-MS/OES) and Microwave Plasma Atomic Emmission Spectroscopy (MWP-AES), Isotope dilution mass spectrometry (IDMS), including the classic wet chemical analysis and the Wavelength Dispersive/Energy dispersive x-ray fluorescence (WD/ED XRF) analysis.

The methods were validated by evaluating some parameters such as detection limit, precision, accuracy, uncertainty. The qualitative investigation involved the identification of 17 elements as inorganic impurities: Na, Cr, Ni, Cu, Cd, Pb, Ca, Mg, Al, K, Mn, Fe, Zn, S, Si, Ti, P and as organic impurities there were: mono-, di-, triglycerides, free glycerol, total glycerol, methanol. All impurities were evaluated quantitatively.

These inorganic impurities AI, Ca, Fe, Mg, P, K, Si, Na and Ti are major elements, which after combustion turns into a non-flammable solid residue in the form of ash.

![](_page_13_Picture_1.jpeg)

Ash content represents a very important quality characteristic of solid biofuels being an important parameter for fuel deliveries. Knowing the ash content can have economic consequences, because the presence of ash reduces the quality of fuels, especially the calorific value. Thus, BRML (Romania), TUBITAK (the Republic of Türkiye) and DTI (Denmark) determined the ash content by the same method according to the ISO 18022:2015 standard with different equipment on the same type of material.

The amount of ash was determined from the 3 types of solid biofuels that were agreed upon, namely highquality wood chips (WC-HQ), industrial-quality wood chips (WC-IQ) and wood pellets (WP) in the measurement range 0 % - 1.2 % with absolute repeatability less than 0.1 % and absolute reproducibility 0.2 % (according to EN ISO 18122). Obtained results are according to Table 1 for repeatability (Sr) and table 2 for reproducibility (SR).

Wood pellets (WP-HQ)			Wood chips high quality (WC-HQ)			Wood chips industrial quality (WC-IQ)			
Quantity %			Quantity %			Quantity %			
	Value	ue Sr		Value	Sr		Value	Sr	
	< 1%	< 0.1%		< 1%	< 0.1%		< 1.1 %	< 0.1%	
BRML	0.23	0.029	BRML	0.21	0.015	BRML	1.13	0.053	
TUBITAK	0.28	0.028	TUBITAK	0.24	0.006	TUBITAK	1.10	0.032	
DTI	0.28	0.009	DTI	0.22	0.039	DTI	1.14	0.104	

Table 1 repeatability (Sr) of ash measurement

#### Table 2 reproducibility (SR) of ash measurement

Wood pellets (WP-HQ)			Wood chips high quality			Wood chips industrial quality			
	Mean value ash (%) SR(%) C.2%		(WC-HQ) Mean value SR ash < 0 (%)		SR(%) < 0.2%	(WC-IQ) Mean value SR(%) ash (%) < 0.2%			
BRML TUBITAK DTI	0.26	0.041	BRML TUBITAK DTI	0.22	0.022	BRML TUBITAK DTI	1.12	0.029	

IMBiH has optimized the analytical method based on atomic emission spectrometry and performed measurements for major elements in ash of solid biofuels (K, Na, Mg, Ca and Fe). Ash sample materials produced by TÜBITAK UME (wood chips ash powder and straw ash powder - 4 bottles each), were analysed using the developed MWP-AES method in terms of homogeneity and characterization for elements of interest (Ca, Mg, K, Na and Fe), Analytical results were further subjected to adequate statistical evaluation (F test) to confirm that there was no statistically significant difference between bottles. In addition, ash samples acquired by DTI during the online industrial test at VERDO's CHP plant were analysed too. Assigned values were calculated for each element as an average value from four bottles of each sample. Assigned values were transferred to the wavelength dispersive X-ray fluorescence spectroscopy (WD-XRF) technique for the purpose of validating this method which can be easily transferred to portable XRF instruments used in industry. The results have been reported ("Development of new metrology methods for determining major elements in solid biofuels ash and establishment of results' traceability", submitted to the journal Accreditation and Quality Assurance) and there is a potential for preparing reference materials for calibration of XRF instruments used in the field for measurements of major elements in solid biofuel ash. Samples prepared for XRF methods are stable and can be used over longer periods of time (inert samples bonded with water repelling binder - wax) and are useful for on-site analysis for calibration of equipment used to estimate the ash composition.

![](_page_14_Picture_1.jpeg)

The objective was achieved by developed a metrological framework for online methods by first understanding the parameters needed to define the methodologies required for traceability. This includes the identification and quantification organic and inorganic impurities and the determination of ash content. The results were published as an peer-reviewed article in Energies (D6) accessible via the journal's website (https://doi.org/10.3390/en16135221)

These validated and improved methodologies were used for the certification (including homogeneity, stability and characterization tests of candidate reference materials produced within the scope of the BIOFMET project.

Certified reference materials were required for several project objectives. Therefore, new reference materials (Figure 15), i.e., wood pellet and biodiesel, for the calibration, quality control, validation/verification and the comparison of the developed methods for calorific value, ash content, impurities etc. were developed by TUBITAK in collaboration with other project partners and collaborators. Before the project, the availability of solid matrix reference materials certified for calorific value was very limited. For fossil fuels two coal materials and a furnace coke are available from EC-JRC (ERM®- EF411, EF412, EF413). As liquid matrix reference materials are available from EC-JRC (ERM®- EF001) and NIST (SRM® 2772), but these materials are not certified for determination of the calorific value.

In the BIOFMET project advanced traceable measurement standards for the determination of the calorific value and impurities were established. Two solid biofuel reference materials were produced: UME BIOFMET CRM 02, certified for calorific value, moisture, ash and mass fractions of Al, Cr, Cu, Mg, Mn, Ni, Pb, S, Zn elements and UME BIOFMET CRM 03, certified for calorific value and moisture. These materials were produced in accordance with requirements of ISO 17034 standard.

The raw material for the CRMs is wood pellet (property class labelled as A1 according to ISO 17225-2 by the manufacturer) which was produced in Poland. For UME BIOFMET CRM 02, the material was spiked with As, Cr, Ni, Pb, Hg and milled to obtain a powder material whereas UME BIOFMET CRM 03 was bottled as it is, in pellet form after homogenization without further processing except gamma irradiation which was applied to both materials. Homogeneity and stability of the material were assessed in accordance with ISO Guide 35. The material was characterized by an interlaboratory comparison among competent laboratories. Uncertainties of the certified values were calculated in accordance with GUM "Guide to the Expression of Uncertainty in Measurement" and includes characterization, homogeneity, stability components.

UME BIOFMET CRM 02-Wood Pellet Powder material is intended for method development and validation in determination of calorific value, moisture, ash and mass fractions of Al, Cr, Cu, K, Mg, Mn, Ni, Pb, S, Zn elements and for quality control purposes. The CRM is available in glass bottles containing approximately 50 g of powder material. UME BIOFMET CRM 03 material is intended for method development and validation in determination of calorific value and moisture and for quality control purposes. The CRM is available in glass bottles containing approximately 100 g of pellet material.

Certification measurements and their evaluation for homogeneity, short-term, long-term stability and characteriation studies have been completed. All target uncertainties for characterization have been achieved for the selected parameters as follows:

- Biodiesel material for Calorific value: 0.09 % (*k*=1)
- Wood pellet powder material Calorific value: 0.19 % (*k*=1)
- Wood Pellet material Calorific Value: 0.20 % (*k*=1)
- Wood pellet powder material for Ash: 6.4% (*k*=1)

Results of elemental impurities analysed by TUBITAK for the produced biodiesel reference material gave relative uncertainty values (k=1) as follows; Ca: 2.4% (ICP OES), K: 2.9% (ICP OES), P: 0.99% (ICP OES), 1.6% (ICP MS), Mg: 2.2% (ICP OES), 3.8% (ICP MS), Sodium: 3.9% (ICP OES), 4.0% (ICP MS), S: 2.2% (ICP OES), 3.7% (ICP MS), 0.32% (ID ICP MS). All uncertainties for the elements of interest are below target uncertainty of 10% and even below 5%. Total Glycerol analysed by BRML gave relative uncertainty value (k=1) of 4.5% which is also below target relative uncertainty of 5%.

![](_page_15_Picture_1.jpeg)

![](_page_15_Picture_2.jpeg)

Figure 15. Developed Certified Reference Materials (From Left to Right: UME BIOFMET CRM 02-Wood Pellet Powder, UME BIOFMET CRM 01-Biodiesel, UME BIOFMET CRM 03-Wood Pellet

#### The objective for target uncertainties was achieved.

To summarise: Improvements to the method applied for bomb calorimetry has been developed. The result has provided input to documentary standards. Several activities have been directed towards ash analysis, including traceable ash analysis developed in several laboratories, the development of new certified reference materials (CRM) and reference materials (RM) for industrial ash analysis using XRF for online analysis. The work has provided essential input to the "Report on certification of liquid and solid biofuel reference materials" (deliverable D5) that is made available to the public on the BIOFMET web site.

Objective 3 has thereby been achieved.

Objective 4: "To develop validated methods to determine the amount and nature of impurities in liquid biofuels, including quantifying and qualifying inorganic (i.e. Na, Ca, K, Mg, P) for a measurement range of 0.5 mg/kg to 2.5 mg/kg with a relative target uncertainty of 5 - 10 % (k=1) and organic by-products (i.e. total glycerol) < 5 % relative repeatability for 0.1 - 0.3 % range as per EN ISO 14105. In addition, to develop a traceable method for the online determination of the calorific value of liquid biofuels."

In bioliquids the presence of inorganics (residual catalyst, salts) and organic by-products (glycerol, partly converted fat, soap) can lead to undesirable precipitations of solids in the fuel transportation system/inlet to the combustion chamber. Furthermore, the presence of inorganics and thus possible formation of corrosive gases can lead to corrosion of the appliances. The amount of impurities, especially the inorganics, will also affect the caloric value and must be known to make an accurate determination of this.

In relation to quality control of the fuel, it is known that the remaining water after dehydration (or water taken up due to the hygroscopic nature of methyl esters), as well as remaining impurities after purification and filtering operations, have a negative impact on the energy performance after a long-term storage of fatty acid methyl esters (FAME) and other biodiesels. Moisture can result in microbial growth and can lead to undesirable diesel bugs, moulds, yeasts and bacteria spreading throughout the fuel. Storage of biofuels over extended periods of time induces degradation of their properties. In order to determine the calorific value, the impurities and

![](_page_16_Picture_1.jpeg)

water must then be measured simultaneously and accurately with validated methods with a fast, reproducible measuring technique. Accurate measurements of the calorific value of liquids biofuels are needed and the only reliable technique is bomb calorimetry which is generally adopted in metrology research and testing laboratories.

As a first step, PTB, BRML, DTI, IST, IMBiH, TUBITAK, CETIAT, VERDO, AMU, and PROMETEC made the "Report on required parameters and metrological methodologies for measuring calorific value of biofuels and qualifying impurities, moisture, and ash content", which was submitted to EURAMET as the deliverable D1. The report was based a survey among stakeholders and a literature review using peer-reviewed publications and documentary standards. This allowed to establish the metrological requirements for the certified reference material candidates and the impurity analysis.

In the BIOFMET project advanced traceable measurement standards for the determination of the calorific value and impurities were established. One liquid biofuel reference material was produced by TUBITAK in collaboration with other project and collaborators: UME BIOFMET CRM 01, certified for calorific value, density, viscosity and mass fractions of Ca, K, Mg, Na, P, S elements. The material was produced in accordance with requirements of ISO 17034 standard.

The raw material for the CRM is Biodiesel (B100 composed of 80% RME [rapeseed methyl ester] and 20% SME [soy methyl ester]) which was produced in Romania. The material was spiked with Ca, K, Mg, Na, P standards in mineral oil. Homogeneity and stability of the material were assessed in accordance with ISO Guide 35. The material was characterized by interlaboratory comparison studies among BAM, BRML, DTI, IMBIH, GUM, LGC, PTB, and TUBITAK. Uncertainties of the certified values were calculated in accordance with ISO Guide 35 and GUM "Guide to the Expression of Uncertainty in Measurement" (JCGM100:2008) and includes characterization, homogeneity, stability components.

The material is intended for method development and validation in determination of calorific value, density, viscosity and mass fractions of Ca, K, Mg, Na, P, S elements and for quality control purposes. The CRM is available in glass bottles containing approximately 500 mL of material.

Subsequently, BRML first determined in laboratory using the chromatographic method, the mass concentration of total glycerol, free glycerol and residual mono-, di- and triglycerides contained by fatty acid methyl esters (FAME) resulting from the transesterification of vegetable oils. The principle of the method consists in the transformation of free glycerol and mono-, di- and triglycerides into much more volatile and stable derivatives in the presence of pyridine and N-methyl-N-trimethylsilylfluoroacetamide (MSTFA). After the silanization reaction, the sample is analysed by gas chromatography on a short column with a small deposition of the stationary phase, injecting it directly into the chromatographic column (on-column) and detecting the compounds with a flame ionization detector (F.I.D.). After a calibration procedure, the quantification of free glycerol is performed in the presence of the internal standard 1,2,4-butanetriol. Mono-, di- and triglycerides are directly quantified in the presence of internal standards for each category of glycerides:

- monononadecanoin (Mono C19) for monoglycerides
- dinonadecanoin (Di C38) for diglycerides
- trinonadecanoin (Tri C57) for triglycerides

All the parameters (selectivity, linearity, trueness and accuracy) of the method for determining organic impurities were analysed and calculated.

The sources of uncertainty were established for both glycerides (Figure 16) and free glycerol

(Figure 17). In Figure 16 the following parameters are included:

- A<sub>Mono</sub>, A<sub>Di</sub>, A<sub>tri</sub> the sum of the areas corresponding to the peaks of mono-, di- and triglycerides in the sample
- A<sub>MonoC19</sub> the peak area corresponding to the internal standard Mono C19
- MMonoC19 the mass of the internal standard Mono C19, in mg;
- ADiC38 the peak area corresponding to the internal standard Di C38;
- M<sub>DiC38</sub> the mass of the internal standard Di C38, in mg;
- ATriC57 the peak area corresponding to the internal standard Tri C57;
- MTriC57 the mass of the internal standard Tri C57, in mg;
- *m* the mass of the biodiesel sample

![](_page_17_Picture_1.jpeg)

The measurement uncertainty was calculated, and the uncertainties budget was established.

![](_page_17_Figure_3.jpeg)

Figure 16. Sources of uncertainty in the determination of glycerides concentration in biodiesel.

![](_page_17_Figure_5.jpeg)

Figure 17. Sources of uncertainty in determining the mass concentration of free glycerol in biodiesel.

The accuracy component  $u_R$ , was estimated by means of the standard deviation of the repeatability of the differences between the experimentally determined values for the same sample.

The trueness of the method  $(u_{bias})$  was determined from the experiments used to validate the accuracy of the method as a combination of the difference  $RMS_{bias}$  between the concentrations of the analyzed standard solutions and the concentrations determined experimentally and the uncertainty of the preparation of the standard solutions,

$$u_{bias} = \sqrt{RMS_{bias}^2 + u_{CSS}^2}.$$

![](_page_18_Picture_1.jpeg)

Here  $RMS_{bias}$  is the difference between the glycerin concentration of the standard solutions and the glycerin concentration determined experimentally and  $u_{CSS}$  the standard uncertainty of preparation of standard glycerin solutions.

TUBITAK collaborated with BRML on measuring of the organic impurities and determined the methanol content according to standard ISO EN 14110. The results for organic impurities content from biodiesel are presented in Table 3.

![](_page_18_Picture_4.jpeg)

Figure 18. Test bench for biodiesel/ethanol mixtures.

Table 3 Organic Impurities - Biodiesel

Organic	monoglycerides		diglycerides		triglycerides		free glycerol		total glycerol		methanol	
impurities	Value	U	Value	U	Value	U	Value%	U	Value	U	Value	U
	%	( <i>k</i> =2)	%	( <i>k</i> =2)	%	( <i>k</i> =2)		( <i>k</i> =2)	%	( <i>k</i> =2)	%	( <i>k</i> =2)
BRML	0.565	1.76	0.171	1.18	0.098	1.84	0.0144	1.60	0.197	1,98		-
TUBITAK	-			-	-		-		-		0.172	0.08

The result of the analysis of the liquid biofuels has been reported in a joined publication by BRML, TUBITAK, IMBiH and DTI ("Development and validation improved metrological methods for the determination of inorganic impurities and ash content from biofuels" by Stratulat et al., Energies, doi.org/10.3390/en16135221).

The work has provided essential input to the "Report on certification of liquid and solid biofuel reference materials" (deliverable D5) that is made available to the public on the BIOFMET web site.

The work has provided essential input to the "Good Practice Guidelines on uncertainty assessment of biofuel measurements" (deliverable D7) that is made available to the public on the BIOFMET web site (https://doi.org/10.5281/zenodo.8180289).

At AMU's Institut Fresnel laboratory, a prototype has been developed (Figure 18) for on-line measurement of impurities, such as ethanol in biodiesel (Figure 19), or water content in bioliquids (Figure 20). The first step in this study was to determine the dielectric contrast between biodiesel or bioliquids with and without impurities or water. The main application of this project is the on-line evaluation of the ethanol content of biofuels using the prototype. An article has been published about this application (PIER Letters, Vol. 106, 1-6, doi:10.2528/PIERL22021104) The second application naturally arose for the detection of water adulteration in honey using the same prototype. A second article has

been published regarding this work (PIER Letters, Vol. 111, 1-7, doi:10.2528/PIERL23041205 These results will enable AMU Institute Fresnel us to envisage a second prototype for non-destructive fraud detection without chemical analysis or sampling.

![](_page_18_Figure_13.jpeg)

Figure 19. Permittivity of Ethanol/Biodiesel mixture; from 0% to 12% ethanol (step 2%).

![](_page_18_Figure_15.jpeg)

![](_page_18_Figure_16.jpeg)

![](_page_19_Picture_1.jpeg)

To summarise:

The metrological requirements for the certified reference material candidates and the impurity analysis were established and a liquid biofuel reference material was produced, certified for calorific value, density, viscosity and mass fractions of Ca. K. Mo. Na. P. S elements. The mass concentration was determined for total glycerol. free glycerol and residual mono-, di- and triglycerides contained by fatty acid methyl esters (FAME) resulting from the transesterification of vegetable oils. The targeted uncertainties were met and the work has provided essential input to the "Good Practice Guidelines on uncertainty assessment of biofuel measurements" public (deliverable D7) that is made available the on the BIOFMET to web site (https://doi.org/10.5281/zenodo.8180289). A prototype instrument for on-line measurement of impurities, such as ethanol in biodiesel or water content in bioliquids have been developed and tested. In the future this could be a promising tool for non-destructive fraud detection without chemical analysis or sampling.

Objective 4 has thereby been achieved.

# 5 Impact

Dissemination activities included:

- a) A project website (www.biofmet.eu) as well as a LinkedIn page (<u>https://www.linkedin.com/company/biofmet/</u>) to promote the project to a user community as wide as possible. The first five project newsletters are available at the website, and a last newsletter will be published after concluding the project. All important documents and open access papers produced during the project, including the guideline "Good Practice Guidelines on uncertainty assessment of biofuel measurements" developed in the project, are also available via the website.
- b) Formation of a formal stakeholders committee and collaborators joining the project. By the end of the project there were 18 stakeholders and collaborators from 11 countries. The stakeholder committee included representatives from national standardisation bodies, laboratories, regional metrology organisations, trade and industry associations, producers and traders of biofuels, operators of biofuel-fired powerplants, companies providing services and instrumentation for process automation and optimisation.
- c) Two workshops where the first took place at IST, Lisbon in June 2022. This event had 34 participants and included ten presentations and six posters from the project's partners with details on the BIOFMET's activities. One additional presentation was carried out by a Portuguese company, manufacturer of thermal energy equipment powered by biomass fuels. The second Workshop was held at PTB, Braunschweig, Germany, in March 2023. This Workshop had 24 participants and included twelve presentations and five posters including an oral presentation from a company from the Republic of Türkiye regarding calorimetry. More details on the workshops as well as the presentations can be found on the project's website.
- An online training course launched 2023 d) in May in the YouTube page: https://www.youtube.com/@biofmet. The training is divided into five modules, according to the different topics covered by the project. Each module is composed of a series of videos with presentations and laboratory equipment demonstrations.
- e) Fourteen presentations about the project and its aims were given at EURAMET EMN Energy Gases, TC-T and TC-MC, to the CIPM CCT and CCQM meetings and to COOMET TC1.12 and 1.10. These are the leading metrology committees in this field.
- f) The project was presented at meetings by ISO TC 193 WG 25, ISO TC 28 WG 24 and CEN-CLC/Eco-CG. Input in the form of a report with concrete suggestions for changes in EN-ISO 18135 and EN-ISO 18122 in connection with the next update has been made available for standardization groups (e.g. CEN/TC 19, CEN/TC 335, ISO/TC 238) via DS s358 (Biofuels).
- g) 10 conference presentations at the: 29th European Biomass Conference (EUBCE 2021), 24th Kalorimetrietage, Biomass PowerON Conference 2021, International Conference on biomass (IConBM2022), XVIIIth International Symposium PRIOCHEM "Priorities of Chemistry for a Sustainable Development" (PRIOCHEM 2022), 9th International Conference on Materials Science and Technologies – RoMat 2022, 20th and 21st International Metrology Congress (CIM 2021 and 2023) and 789. WE-Heraeus-Seminar: Sustainable Aviation Fuels - Design, Production and Climate Impact.

![](_page_20_Picture_1.jpeg)

h) 12 scientific papers have been submitted to open-access peer reviewed journals. At the time of writing 7 papers have been published and are available from the BIOFMET web-page.

#### Impact on industrial and other user communities

This project will have an impact on both producers of liquid and solid biofuels and users of these products in the power industry to whom accurate determinations of the fuel's energy content supported by product quality documentation underpinned by rigorous and traceable testing are of key importance.

The new rigorous calibration methods, reference materials and procedures developed in this project will enable improvement of the quality control of liquid and solid biofuels. The two reference standards developed for the calibration of the transfer standards are ready to facilitate industry with calibrations of measuring instruments for water content in biofuels. The technologies developed for on-line measurement of water-content and impurities is expected in the future to be taken up by instrument manufacturers. Three new reference materials are ready for the market.

Automated and representative sampling of the fuels by means of robots, has been implemented and commercialised by an industrial partner which supports improved quality control and support fair trade as well as a safer working environment. The published guideline on uncertainty and the use of machine learning to optimise calibration curves will assist the efficiency and digitalisation of the industry using solid and liquid biofuels.

When the suggestions for improvement of normative standards have been implemented greater measurement harmonisation across the biofuel sector is enabled, creating increased operator confidence in supplied calorific content of the fuels. This is important to ensure that the energy production meets the output expectations.

#### Impact on the metrology and scientific communities

New advanced measurement guidelines for accurate determination of the calorific value of solid and liquid biofuels has been established, and new reference materials for the determination of the calorific value, ash and impurities have been made available. This will in the short term enable the intercomparison of calorimetric facilities at NMI level with an accuracy not obtainable today. In the longer term, measurement uncertainties within this field will be improved at European biofuel supplier and operator facilities, where current differences in measurements of moisture content and calorific value are pronounced.

The project has produced 12 scientific peer-reviewed publications regarding bomb calorimetry, the new methods for impurity determination, new techniques for determining water content in solid and liquid materials. The published results and designs are expected to be used by the scientific research community.

At NMI/DI level, 3 organisations outside the consortium joined the project as collaborators.

#### Impact on relevant standards

The earliest impact of the project on standards will be at the level of sampling, determination of moisture, ash, impurities and calorific value. The project was presented at meetings by ISO TC 193 WG 25, ISO TC 28 WG 24 and CEN-CLC/Eco-CG. Input in the form of a report with concrete suggestions for changes in EN-ISO 18135 and EN-ISO 18122 in connection with the next update has been made available for standardization groups (e.g. CEN/TC 19, CEN/TC 335, ISO/TC 238) via DS s358 (Biofuels).

The project has developed reference materials for solid biofuels which will supply a quality control material for verification of methods for determining the calorific value of solid biofuels according to the ISO 18125 standard.

#### Long-term economic, social, and environmental impacts

The development of the new methods, technology and knowledge within this project will support greater uptake of sustainable biofuels enabling greater diversification in the energy supply and a reduction of reliance on fossil-fuelled electricity production. This will have an impact on climate and environmental protection. The EU aims for a 40 % reduction of greenhouse gas emissions compared to 1990 levels. The indicative target for an improvement in energy efficiency at EU level is at least 27 % (compared to projections), to be reviewed by 2020 (with an EU level of 30 % in mind).

Within 5-10 years, the introduction of new broad range liquid fuels will enter the market aimed at the land and sea transport sector. It is anticipated that these will be in the form of methanol, ethanol, biodiesel, diesel made from plastic, ammonia or other energy carrying liquids that can be used in combustion engines. These new

![](_page_21_Picture_1.jpeg)

liquid fuels will come from multiple sources and origin of feedstock which affect the impurities in the fuel. Accurate real time methods for identification of impurities on an industrial scale will be needed. This project has provided a step towards this.

Energy storage technologies are essential when the production of  $CO_2$  neutral energy from e.g. wind power increases. Electro-fuels also known as Power-to-Liquid have a potential to replace fossil fuels in the future. Introducing new types of fuels for existing technologies (e.g. cars, ships, aviation) will require traceable measurements and documentation of the impurities in the fuel, not only after, but also during their production. The outcome of the project will thus support the transition to clean  $CO_2$  neutral energy and the independence from fossil fuels.

# 6 List of publications

- M. Shehab, C. Stratulat, K. Ozcan, A. Boztepe, A. Isleyen, E. Zondervan, K. Moshammer, A Comprehensive Analysis of the Risks Associated with the Determination of Biofuels' Calorific Value by Bomb Calorimetry, Energies. 15 (2022) 2771. <u>https://doi.org/10.3390/en15082771</u>
- M. Shehab, C. Stratulat, K. Ozcan, A. Boztepe, F. Coskun, F.S. Alper Isleyen, E. Zondervan, K. Moshammer, Improved Metrological Methodology to Address the Challenges Associated with the Determination of Biofuels Calorific Value by Bomb Calorimeter, Chemical Engineering Transactions. 92 (2022) 433-438. <u>https://doi.org/10.3303/CET2292073</u>
- F. Sparma, B. Tallawi, E. Georgen, and P. Sabouroux, Multi-Probe Sensor for Water Content Diagnosis of Liquid Biofuels, Progress In Electromagnetics Research Letters, Vol. 106, 1-6, 2022. https://doi.org/10.2528/PIERL22021104
- Floriane Sparma Sarah Sennoun Pierre Sabouroux, "Detection of Water Content in Honey by Electromagnetics Characterization Measurements," Progress In Electromagnetics Research Letters, Vol. 111, 1-7, 2023. <u>https://doi.org/10.2528/PIERL23041205</u>
- Bayan Tallawi Floriane Sparma Eric Georgin Pierre Sabouroux, "Towards Validating a Coaxial Transmission Cell for Dielectric Measurements on Liquids," Progress In Electromagnetics Research C, Vol. 134, 223-236, 2023. <u>https://doi.org/10.2528/PIERC23013104</u>
- Stratulat, C.; Ginghina, R.E.; Bratu, A.E.; Isleyen, A.; Tunc, M.; Hafner-Vuk, K.; Frey, A.M.; Kjeldsen, H.; Vogl, J. Development- and Validation-Improved Metrological Methods for the Determination of Inorganic Impurities and Ash Content from Biofuels. Energies 2023, 16, 5221.
  <a href="https://doi.org/10.3390/en16135221">https://doi.org/10.3390/en16135221</a>
- Shehab, M.; Moshammer, K.; Franke, M.; Zondervan, E. Analysis of the Potential of Meeting the EU's Sustainable Aviation Fuel Targets in 2030 and 2050. Sustainability 2023, 15, 9266. <u>https://doi.org/10.3390/su15129266</u>

This list is also available here: <u>https://www.euramet.org/repository/research-publications-repository-link/</u>

# 7 Contact details

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