
**Part 1 Publishable Final Report
(Synthesis Report)**

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Glossary

ANOVA	analysis of variance
CCD	charge-coupled device
CCP	critical control points
CEN	Comité Européen de Normalisation (European Committee for Standardisation)
CVAAS	cold vapour atomic absorption spectrometry; system for determination of Hg
FAAS	flame atomic absorption spectrometry; system for element determinations
FME	failure mode and effect
F-tests	statistical tool to test the homogeneity of two standard deviations
GC/MS	gas chromatography - mass spectrometry
GFAAS	graphite furnace atomic absorption spectrometry; system for trace element determinations
GHG	greenhouse gas
GROT	Swedish word for tops and branches of trees discarded during felling
IC	Ion chromatography
ICP-MS	inductively coupled plasma mass spectrometry; system for multi-element determinations of trace elements
ICP-OES	inductively coupled plasma optical emission spectrometry; non destructive system for multi-element determinations
MAF	Melt area fraction
MC	moisture content
NAS	Newly Associated States
NMS	New Member States
QA	Quality Assurance
QC	Quality Control
QM	Quality Management
SDTA	simultaneous difference thermo analysis
SEM-EDS	scanning electron microscope combined with energy dispersive X-ray spectrometry
TC	Technical Committees
TGA	thermo gravimetric analysis

t-tests	test for a difference between two means
WP	work package
XRF	X-ray fluorescence spectrometry; non destructive system for multi-element determinations

1 Introduction

In its White Paper for a Community Strategy and Action Plan the European Union has set the target to increase the share of renewable energy sources to 12 % of the gross energy consumption by 2010. Biomass is one of the renewable energy sources that has the potential to major contribute for reaching this goal. Today, biomass contributes about two-thirds to the total renewable energy production. If produced in a sustainable way (i.e. primarily from forestry and agriculture) biofuels support to greenhouse gas mitigation. Moreover, being a domestic energy carrier, they help Europe to become more independent of imported fossil fuels and thus increasing the security of energy supply [1].

To take benefit of these advantages, biomass has to be integrated much better into the energy system. Therefore, a dynamic and sustainable international market for biofuels is urgently needed. However, for managing and obtaining a smooth business in Europe firm and accomplishable “market rules” and standards for common understanding referring to terminology, sampling and testing as well as Quality Assurance on biofuels are required. Within the ongoing European standardisation process the Technical Committee CEN TC 335 "Solid Biofuels" currently develops such "market rules" or standards [1, 3]. From the elaborated comprehensive knowledge basis best practice guidelines are derived. Project results are introduced to the Technical Specifications drafted by CEN TC 335. These Technical Specifications are accounted as tool of importance to support the biofuels trade in Europe.

1.1 Objectives and strategic aspects

The main objective of BioNorm was to provide the scientific background for the European standardisation process of solid biofuels. The emphasis of the project was laid on a fuel Quality Assurance system for solid biofuels in order to support and extend the market. Such a system is based on extensive practical work on sampling and testing to improve the existing procedures and to allow for the provision of a high quality biofuel throughout the overall supply chain and thus improving the confidence of the consumer into the biofuel. Besides the work on terminology, specifications and classes, this includes the testing of physical-mechanical fuel characteristics (e.g. moisture content, bulk density and ash melting behaviour) as well as chemical fuel properties (among other elements particularly sulphur, chlorine and nitrogen content). The integration of different countries from the Newly Associated States (NAS) - meanwhile commonly New Member States (NMS) - ensured to take into account the standardisation requirements of these countries and to basically meet their specific needs. The work realised within the BioNorm project was closely linked to the work of CEN TC 335 “Solid Biofuels” guaranteeing an excellent exploitation of the results.

1.1.1 Scientific and technological objectives

Pre-normative investigations have revealed existing methods for sampling and testing as well as for Quality Assurance in use for solid fossil fuels (e.g. hard coal). However, shown

in several experiences these methods are only partly applicable to solid biofuels. Although CEN TC 335 "Solid Biofuels" will continue to develop standards on the basis of the best information currently available, it recognises the strong need for further research in some areas. This involve improvements of the reliability of selected sampling and testing methods, and the strong need for the development of an overall Quality Assurance system for solid biofuels taking practical needs into consideration.

Therefore, this project was aimed to carry out research on sampling and sample reduction procedures as well as physical-mechanical and chemical tests for a more successful practical implementation. Furthermore, the results were integrated into a fuel Quality Assurance system covering the overall supply chain. The research is aimed at comprehensive evaluation and identification of the best suitable methods for the respective determination, providing a knowledge basis for best practice guidelines. These guidelines and findings were also used for drafting of Technical Specifications within the standardisation process and thus contributing the work of CEN TC 335 "Solid Biofuels" to accelerate the development of improved standards with consideration of practical requirements. Coupled with this, the conveners of the different working groups of CEN TC 335 were involved deeply within this project.

1.1.2 Socio-economic aspects and contribution to the EU policies

The European Commission (EC) has given a mandate to CEN for the elaboration of standardisation in the field of solid biofuels. The implementation and application of European standards on sampling, testing and classification of solid biofuels as well as Quality Assurance is seen as an important element towards an increased utilisation and international trade of solid biofuels. Expanding markets are required to fulfil targets of the EC directives and to reach the political goals on national level.

An increased use of biomass for energy purposes is reflected in positive socio-economic effects on regional, national and global level. Regionally, it can create new market opportunities for farmers, contribute to the preservation of rural areas and improve the local infrastructure. On a national level, due to increasing biofuels application new jobs are created in the business sectors of engineering, manufacturing, energy production, energy distribution and consultancy. For instance, the broader implementation of small-scale biomass technology is labour intensive when compared to the larger-scale energy production using fossil fuel energy; thus creating employment especially in rural areas. It can be assumed that most of the new jobs will probably be related to fuel handling and plant operation. Being an domestic fuel, biomass also helps to reduce the significant dependence of the European on fossil fuel imports and thus, increasing the security of supply. Globally, the reduction of climate relevant greenhouse gas (GHG) emissions has a significant beneficial effect in long-term

Contributed by the close collaboration BioNorm work packages and CEN working groups (and thus ensuring the dissemination and exploitation of the pre-normative work), it was focused to develop highly sophisticated and therefore widely acceptable standards for the stimulation of the bioenergy markets throughout Europe. Users of these standards are mainly producers, traders and consumers of biofuels. Summing up all players involved into

the production, provision and use of solid biofuels throughout the overall value chain within Europe will benefit from this work realised within the BioNorm project.

Widely accepted standards resulting from the pre-normative work of BioNorm will also contribute to boost the market for devices for sampling and testing of solid biofuels. Additionally, the end use conversion technologies (e.g. combustion and gasification units) can be optimised with regard to the different fuel qualities. The same applies to the machinery and the devices required for the production and provision of solid biofuels, which in turn are needed to overcome the gap between biofuel provision and its utilisation. Finally, the results of the BioNorm project will help to contribute to the continued increasing biofuel market in Europe and therewith assist the European Commission to reach its environmental goals.

1.2 Structure of project and synthesis report

According to the objectives named above, the project work plan has consisted of interrelated work packages, as illustrated in Figure 1. Referring to this, the BioNorm project deals with issues of sampling and sample reduction, with tests determining the physical-mechanical as well as the chemical properties and with aspects of biofuel Quality Assurance. These different work packages were strongly linked to each other by means of collaboration and common meetings. As sampling and testing represent activities of high importance for Quality Assurance within the solid biofuel supply chain, these work packages should support the elaboration of such a Quality Assurance system. In order to ensure that effects of the BioNorm are of greater impact on the continuous increasing biofuel market BioNorm was supplemented by involving also NMS/NAS in the elaboration of suitable test methods and for research exchange.

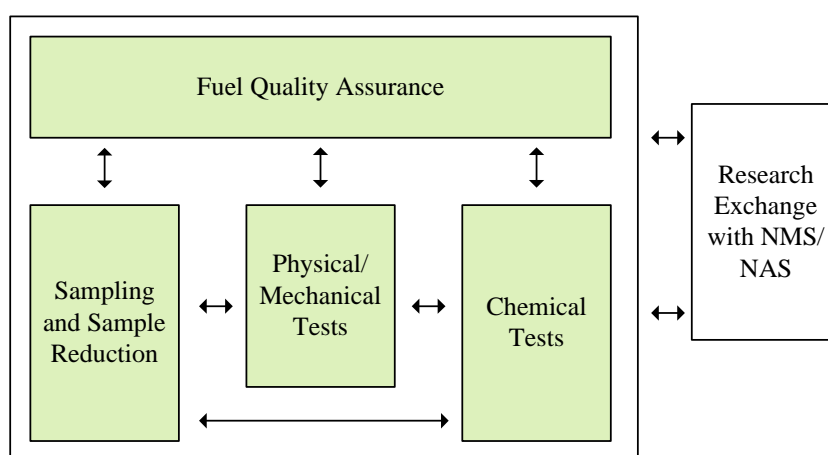


Figure 1: Structure of the BioNorm project

The biogenous materials investigated within the BioNorm project were predominantly chosen because of their importance on the European market of solid biofuels. Accordingly, relevant solid biofuels based on woody, herbaceous and fruit biomasses were analysed and tested in context of sampling and sample reduction as well as physical/mechanical fuel properties (e.g. moisture content, size distribution and durability) and chemical fuel

characteristics (e.g. sulphur, chlorine, major and minor elements). Thus, in the following the focus is set on sampling and sample reduction, physical-mechanical and chemical test as well as on Quality Assurance. Starting with a short introduction of respective objectives, the respective work packages are summarised in terms of their methodical approach and scientific conclusions as well as recommendations. More comprehensive information on the different work packages and the corresponding tasks can be gained from the detailed final reports given in Part 2 of this final technical report. This is also true for the results of the review in terms of respective country conditions given for the NMS/NAS.

This synthesis report (Part 1) is finished by a summary involving final conclusions and recommendations of the whole BioNorm project.

2 Sampling and sample reduction

In this work package (WP I) five partners dealt with methods for sampling and sample reduction that are of importance with regard to testing methods of fuel properties in order to ensure that the required fuel properties are met (e.g. methods for reducing samples for sample preparation for physical-mechanical and chemical tests). The potential impact of this issue on the biofuel markets is considerable.

2.1 Objectives

Often variabilities referring to sampling and sample reduction are much more significant than variabilities referring to testing methods. In average the possible error which can be made by an inappropriate sampling can be about ten times higher compared to testing. On this background, WP 1 is aimed on supporting information to Working Group 3 of the CEN/TC 335 'Solid Biofuels' in order to improve the reliability of the draft European standards for solid biofuels. Therefore, investigation of methods for sampling (Task I.1) and sample reduction (Task I.2) were carried out on a representative bulk sample such as straw bales and a range of woody biofuels (i.e. sawdust, wood-chips and wood pellets).

The main objectives of this work package are [5, 8]:

- to assess the bias which may be introduced when samples of solid biofuels are taken from containers or stockpiles instead of from moving streams
- to define the number and size of increments needed to provide a representative sample
- to assess the variations introduced when reducing the size of samples of solid biofuels to form suitable test portions
- to determine the usefulness of a range of practical sample reduction methods

2.2 Methodical approach

Determining the size and number of sample increments was the purpose of the experimental work on sampling. Comparing the efficacy of various methods was the purpose of the work on sample reduction [8].

For the woody materials (i.e. sawdust, GROT¹ and pellets), investigations on sampling methods were executed on moving streams (conveyor) as well as on stationary piles (heap). The sample increments received in three different sizes were used for determinations of moisture content, ash content and particle size distribution. Investigations on sample reduction methods involved using the methods of riffing, coning and quartering as well as the "long-pile" and the rotary divider.

¹ Swedish word for tops and branches of trees discarded during felling. GROT is a kind of wood chips.

Straw bales were sampled in two different ways: by using a coring device and a special hook. The resulting sample increments were tested for moisture content, chloride content, and ash content. The method of coning and quartering was also applied to reducing straw bale samples, with preliminary chopping of the straw in the increment. Further methods used were hand sampling with and without preliminary chopping of the straw as well as using a long pile, also with chopping of the straw [1, 4, 8].

In all uncertainties associated with testing within a single laboratory as well as associated with methods of sampling and sample reduction were measured. For this, statistical instruments (e.g. relative error, standard deviation) were applied, allowing the investigation of the variability between sampling increments (i.e. number of increments for a specified level of sampling variability), between samples, between determinations (i.e. number of determinations for a specified level of repeatability) as well as between the test results [5].

2.3 Conclusions and recommendations

The experimental work has allowed the relative bias of different sampling methods and the influence on sampling variability of different increment sizes to be assessed. This work has also allowed the number of sampling increments required to give a satisfactory level of sampling variability to be estimated, for the biofuels investigated (i.e. woody-fuels such as GROT, pellets and sawdust as well as straw bales). On the basis of these experiments it can be concluded that none of the methods used in the experiments gave disastrous results. Thus, no method can be ruled out from practical use and should be included in the CEN standard [8].

Below the main conclusions and recommended options of sampling and sample reduction trials are briefly summarised [5, 6, 8, 9].

2.3.1 Sampling

In sum it is revealed: with exception of particle size distribution, no evidence of a relative bias between the methods of sampling and testing can be revealed for testing moisture, ash and chlorine content of the fuels investigated. Preferred numbers and sizes for sampling are summarised in Table 1.

Sampling from tipped lorry-loads is not biased relative to sampling from a conveyor for the methods recommended for GROT and sawdust.

Based on the work on **GROT** it can be concluded that for moisture and ash, the results on the relative bias do not provide any reason for preferring sampling from the conveyor over sampling from the heap. Different from that, particle size distribution shows relative bias implying the necessity for the CEN standard to define one preferred method.

For moisture and ash of **sawdust** analysis the results concerning the relative bias of the two methods show the same as for GROT. For particle size distribution, the results imply a relative bias between the two methods in case of smaller increment sizes (0.2 litres or 1 litre). Thus, if sampling from the stopped conveyor will be the reference method, then sampling from the heap is acceptable provided that sufficiently large increments are taken.

The results obtained for **pellets** in the investigation of relative bias indicate that the moisture and ash content vary from lorry-load to lorry-load. However, both are not affected by the handling, what is true for the particle size distribution.

For **straw bales**, the experiment results have shown that sampling with the hook is not biased relative to sampling with the coring machine. Taking five increments per bale will result in a relative sampling error of about 10 %. Furthermore, doing one determination per sample will give a relative error of test results (i.e. repeatability) of about 5 %. Both relative errors might be acceptable for routine tests. Due to the effect of straw bale position, when using either the hook or the coring machine, for sampling straw bales should be turned on their side. In addition, increments should be taken from both sides, and from the full depth of the bale [5, 9].

Table 1: Recommended numbers and sizes for sampling

Biofuel	Test method	Sampling method	Increment size ^a	Number of increments	Number of determinations
GROT	moisture	heap / conveyor	1 litre	5	1
	ash	heap / conveyor	1 litre	10	4
	size distribution (passing 64 mm/2 mm)	heap / conveyor	20 litre	5	4
Sawdust	moisture	heap / conveyor	0.2 litre	2	1
	ash	heap / conveyor	0.2 litre	5	2
	size distribution (passing 5.6 mm/0.5 mm)	heap / conveyor	0.2 litre	5	2
Pellets	moisture	heap / bags	0.25 kg	5	1
	ash	heap / bags	0.25 kg	5	2
	size distribution (passing 5.6 mm/0.5 mm)	heap / bags	1 kg	1	2
Straw bales	moisture	core / hook	-	5	1
	ash	core / hook	-	5	1
	chlorine	core / hook	-	5	1

^a not analysed for straw bales

2.3.2 Sample reduction

Preferred methods (i.e. those that gave least variations between sub-samples) in each of the experiments are outlined in Table 2. Some general conclusions may be drawn tentatively from this. “Riffle” is the preferred method to use with the coarser materials (GROT and pellets), if a rotary divider is not available. If available, a rotary divider is the preferred method for determinations of moisture and particle size distribution on pellets. An explanation should be sought for the poor performance of the rotary divider in comparison with the riffle when determining ash. “Coning” is the preferred method with sawdust, but the riffle performed nearly as well. The special method used to sub-sample straw, handful sampling (on straw coarse cut prior to reduction), is the preferred method for determination

of liberated or partially liberated properties of straw, but not for determination of moisture [8, 10].

In general it was revealed, when a sample is taken for the purpose of determination of moisture-content, sample reduction should be avoided, since the materials dried noticeably during the reduction process and the result of the test will be affected.

Table 2: Recommended methods of sample reduction

Biofuel	Moisture content	Ash content	Chloride content	Particle size distribution
GROT	riffle	riffle	-	riffle
Sawdust	riffle / coning / long pile	coning	-	coning
Pellets	rotary*	riffle	-	rotary*
Straw	coning / long pile	hand 2	hand 2	-

* or “riffle” if no rotary divider is available

However, it is important that technicians should regularly and routinely check the achieved repeatability with whatever sample reduction methods applied for [5, 8].

Finally, it is suggested that there is further need to [5, 6]:

- extend the work on sampling other solid biofuels in order to cover the breadth of materials to be found throughout Europe (e.g. wood chips, bark, reed canary grass, olive waste and briquettes)
- develop a guideline on how users of CEN standards can ensure reliable results of sampling and sample reduction procedures as well as can decide the frequency of sampling

3 Physical / mechanical tests

Determining physical and mechanical fuel properties is matter of this extensive work package (WP II), in which 17 partners across Europe were involved. The physical and mechanical fuel properties are largely determining each other. Moisture is the parameter with the highest influence on other physical properties (cf. Figure 2). Testing of these properties is routine.

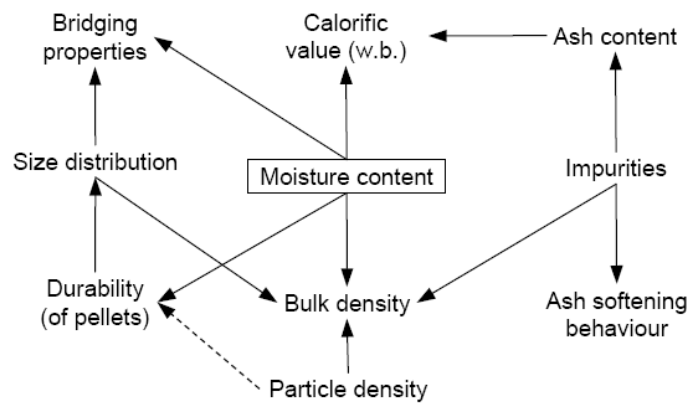


Figure 2: Interdependency among physical-mechanical fuel properties [19]

However, the precision and reproducibility of the results is often very scanty. In addition, rapid and reliable test methods are urgently required for e.g. on-site fuel acceptance or rejection. Therefore, within this work package existing procedures were improved concerning measuredness, and new procedures were developed respectively for moisture content and bulk density (Task II.1), the determination of ash melting behaviour (Task II.2), particle size distribution and dimension (Task II.3) as well as the determination of the durability and raw density of pellets and briquettes (Task II.4).

Except for ash melting behaviour all tests were conducted by international round robin trials by several participants. One important aim of the round robin trials is the comparability and transferability of the results by testing the quality of methods (i.e. repeatability) applied for physical-mechanical properties. The results provided a knowledge basis for best practice guidelines. These guidelines and findings were also used for drafting of Technical Specifications within the standardisation process.

3.1 Moisture content and bulk density determination

3.1.1 Objectives

Moisture content is a primary property for a successful utilisation of solid biofuels within the entire supply chain (e.g. for transportation costs, storage management, calorific value and conversion at end use). Today, several national standards for testing moisture content

are available. However, for common European standards or guidelines reliable tests have been identified. Thus, the research is aimed (i) to study different test methods for the determination of moisture content and non-water losses in comparison with the oven drying method (reference method) and (ii) testing of rapid on-sites applications using the oven drying method as reference (rapid methods). A wide scope of fuels from all over Europe was analysed to cover the broad range of biomass. This is also true for the applied technologies and measuring principles.

Strongly linked to the moisture content of solid biofuels is the bulk density, which is an important property with regard to space for storage and transportation and for volume based payment of biofuels. Furthermore, bulk density is influencing rapid moisture content measurements. The applied national and international standard methods (e.g. German, Swedish and American standard) for determining bulk density are highly inconsistent in practice and confusing in their size and the way of handling [14, 15, 18].

3.1.2 Methodical approach

The investigation was dominated by carrying out round robin trials to determine rapid moisture content (cf. Figure 3) and bulk density.

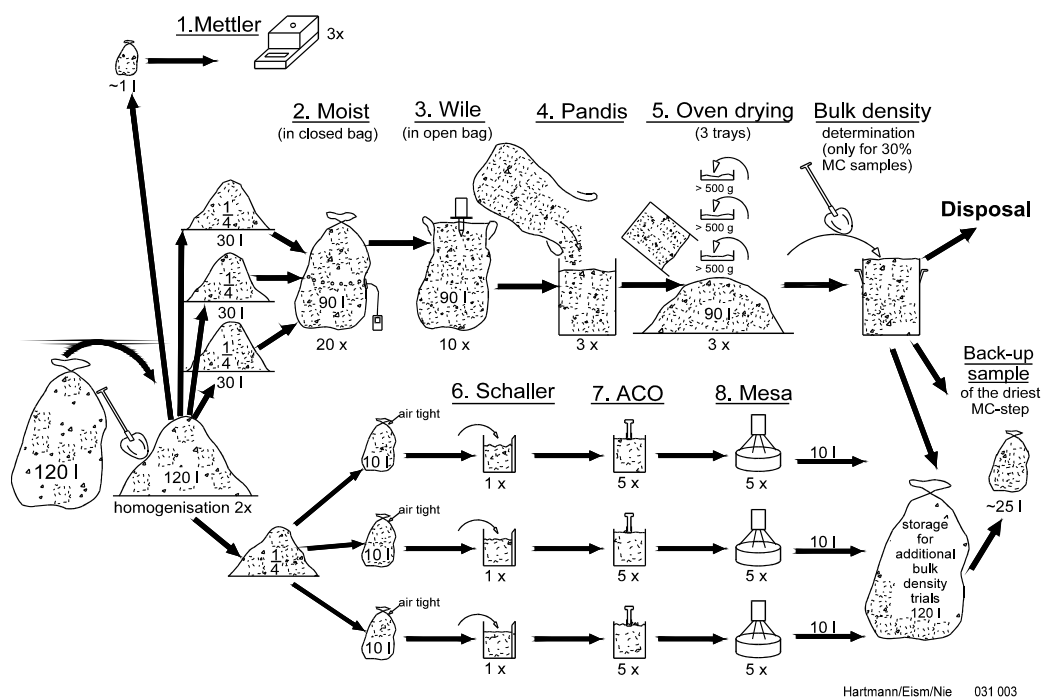


Figure 3: Procedure for moisture content round robin trials – moisture determination with rapid testers and sampling of reference material followed a standard sequence in order to obtain as uniform trial conditions as possible² [14]

² Please see the final report concerning moisture content for detailed information about deployed test equipment/devices like Mettler (thermogravimetric) or Moist (dielectric microwave).

Moisture content

The total moisture content and volatile components were determined by means of: (i) the oven drying method, (ii) the xylene distillation method and (iii) the freeze drying method. Furthermore, (iv) the new analytical GC-MS-method (“gas chromatography - mass spectrometry”) for measuring the non-moisture volatile matters (e.g. terpenes) released during the oven drying process was applied. About 21 biofuel types (e.g. wood chips, bark, pellets, sawdust, olive stones, miscanthus, cork) were analysed using these reference methods.

The rapid moisture test research was carried out using: (i) the thermogravimetric method by oven drying and halogen drying, (ii) the electrical method based on di-electrical properties of moisture subdivided in capacitive method and the microwave method as well as (iii) the optical measuring method based on infrared reflectometric determinations. About 11 laboratories participated in the round robin. All partners followed detailed sample preparation and measuring guidelines in order to reduce any effects from sample handling and preparation [14, 15]. The evaluation of results was done using ANOVA (i.e. ANalysis Of VAriance) for homogeneity as well as significance tests of variances and mean values (e.g. F-tests and t-tests).

Bulk density

For bulk density determination several test methods with research focus on container size and shape, shock impact on the measurement and varying moisture content were analysed by testing about 48 biofuels in total. Assuming that the volume remains constant (i.e. negligence of shrinkage) all original bulk density data were initially calculated to dry matter basis. These data were used for container and treatment evaluation. Furthermore, for the identification of any influence during measurements through fuels bulk density itself, all wood chips were allocated in high and low density fuels (at 180 kg/m³, dry basis).

For container comparison one cube (100 l of nominal volume) and three cylinders (100 l, 50 l and 15 l of nominal volume) were used. All measurements were performed applying two different treatments: (i) “without shock impact” (i.e. normal filling, levelling and weighting) and (ii) “with shock impact” (i.e. weighting after having dropped the container three times from 150 mm height and having it refilled). The influence of moisture content was tested by previous progressively drying of several fresh biofuels samples in order to provide identical sub-samples at varying moisture content levels. Measurements repeatability was tested by comparison of relative repeatability limits (coefficient of variation) [17, 18].

3.1.3 Conclusions and recommendations

Basic results of methods for testing moisture content and bulk density of solid biofuel, which are identified to be the most promising for standardisation in terms of e.g. suitable performance and high reproducibility, are briefly summarised below.

Moisture content – reference method

The applied moisture determination results gave overall comparable results. A statistical significant difference was found among the oven drying method between the standard

method at 105 °C compared to the temperatures 80 °C and 130 °C, respectively. According to this, a slightly increased amount of evaporated matter (volatiles and moisture) results for each temperature step.

The freeze drying method determined significantly lower moisture content values compared to the standard method, whereas the results from the xylene method were deviating only for some of the fuels. This difference was partly attributed by the small sample size used for xylene distillation.

The GS-MS-method has shown promising results as a standard reference method. So it was found that the amount of volatile matter was in order of 0.06 to 11.33 % calculated as percentage of moisture content. Furthermore, it was revealed that the mass loss differences found among the different methods were primarily due to α - and β -pinenes. Though, a more throughout optimisation of the method is required [14, 15].

Moisture content – rapid methods

Among the seven tested rapid test devices about four types were identified to be particularly applicable to measure the moisture content of solid biofuels. These are:

- as on-site types: the thermogravimetric Mettler-Toledo HB45 as well as the capacitive devices Pandis FMG3000 and the Schaller FS2002-H.
- as on-line type: the optical MESA MM710. Although MESA was only tested in a reduced moisture range of 10 to 40 % MC but the method is applicable to the full range of 0 to 100 % MC.

However, the small nominal size and high time need (both Mettler-Toledo) as well as the need of fuel specific calibrations (Pandis, Schaller and MESA) should be considered when selecting the device. For the electrical devices including the bulk density in the calibration function can significantly increase measuring exactness. Moreover, reducing the scope of fuels increases the power of the calibration functions.

The Moist100, Wile25 and ACO estimated the moisture content with a higher variation and therefore cannot be recommended for moisture content determination in solid biofuels [14, 15].

Generally, further work is required to optimise and evaluate e.g. blank values, recovery, adsorption and extraction efficiency as well as response factors [16].

Bulk density

The following was concluded for best practice in bulk density determination of solid biofuels [17, 18]:

- For all tested solid biofuels a measuring container size of 50 l is acceptable. A cylindrical container shape should be preferred for practical reasons due to higher stability and easier manageability.
- A standardised shock impact on the filled container significantly increases the measured bulk density (e.g. 6 % for wood pellets, 10 to 12 % for fuel chips and

18 % for chopped miscanthus), while there was only found a minor improvement for the relative repeatability limits.

- The fuels moisture content during the measurement is of high importance, and consequently has to be recorded since an obvious increase in bulk density (wet basis) was observed with increasing moisture content. Thus, the comparability of bulk density data is only given if any inconsistency in moisture content between the samples is accounted for by the use of a correction factor of 0.712 % for each 1 % moisture difference.

Moisture content effects are largely restricted to the moisture content range up to 25 %. Beyond this point possible effects can be neglected.

3.2 Ash melting behaviour

3.2.1 Objectives

The determination of biofuels ash melting behaviour is of high importance for all thermal conversion processes. The temperature range of sintering, softening and melting can vary broadly, depending primarily on ash composition. Nowadays, it is impossible to forecast ash melting behaviour by calculations based on standard elemental analysis. Standards for evaluating ash melting behaviour of coal and coke are also used for biofuels today. For biofuels known to be problematic in terms of their ash, the implementation of existing laboratory and recommended new methods were tested and assessed. The tested fuels were wood/bark (relatively unproblematic ash), olive stones (unknown ash behaviour), straw and Lucerne (sticky and very sticky ash) [20].

3.2.2 Methodical approach

Various methods for the characterisation of biomass ash melting behaviour were investigated. The production of biofuel ash has been carried out according to CEN TS 14775 at 550 °C. The methods used by the seven involved partners are characterised by computer-controlled analysis during thermal conversion of biofuels. Except for MAF (“melt area fraction”), they are established techniques adopted for this specific use.

The improved DIN³ and the MAF⁴ are methods based on analysis of images that are acquired from a high temperature light microscope by means of a CCD camera. Using these methods, ash melting behaviour (i.e. characteristic temperatures for sintering, deformation, sphere, hemisphere and flow) can be gathered from the specific contours of the analysed test pieces during the melting process. According to their melting behaviour, ashes can be classified into little, moderate and severe sintering/slagging, respectively.

Other applied methods are based on bed sintering methods that determine the onset of agglomeration by monitoring differential pressure and temperatures in the bed. It is

³ also called heating microscope method

⁴ new developed by the BioNorm partner FORCE Technology, Denmark

distinguished between batchwise CFBA (“controlled fluidised bed agglomeration”) and continuously fed types. Both methods are used for complementary as well as for comparison with the other methods reported.

Simultaneous TGA (“thermogravimetric analysis”) and SDTA (“simultaneous differential thermal analysis”) analyses imply continuous measurement of the sample weight and the sample temperature during heat-up. For a combustion ash, the melting will result in several peaks overlapping each other, corresponding to the melting of the different chemical compounds in the ash.

Tests were also performed by SEM/EDS (“scanning electron microscope” combined with a “energy dispersive x-ray analyser”). Based on that method the distribution of chemical species (e.g. Al, Ca, Cl, Fe) in individual particles from a sample. The crystalline phases present in an ash and the content of amorphous material (i.e. the crystalline) were determined by using XRD method (“X-ray diffraction”).

Besides named biofuels, synthetic ash samples (pyrolytic Orton Cones, typically used by potters) were tested using the laboratory methods DIN, MAF, TGA/ SDTA and XRD [20, 21, 22].

3.2.3 Conclusions and recommendations

Although biofuels ashes were tested by seven methods, only from improved DIN, MAF and CFBA temperature information can be derived that can be compared directly. For synthetic samples it was revealed that only the DIN and the MAF method are able to quantify melting temperatures. However, DIN and MAF are ash testing methods suitable for standardisation whereas CFBA may be used as reference for agglomeration/sintering. The recently developed MAF method shows large potential to become reproducible and repeatable. The MAF method has been proven to determine the temperature of 10 % and 50 % melt in the ash samples tested. Though, some modifications are needed for problematic ashes, e.g. straw ash, which tends to form “cakes” (agglomerates) during handling. Moreover, further improvement is required concerning melt viscosity and the possible sample shrinking.

The technologies of TGA/SDTA cannot be standardised at present, but they have the potential to confirm various phenomena associated with ash melting behaviour. SEM-EDS is predestined for providing valuable information on the ash compositions and species. Testing the XRD method revealed that the amount of amorphous phase is evaluable if the connection between amorphous phase and melt phase is clarified before.

Besides the suitability of test methods, issues such as really required information to forecast ash melting and the relation of acceptable analysis cost. It is generally accepted that testing of initial melt temperature as well as the rate of melt formation is essential. With regard to the costs, bed sintering methods and methods as TGA/SDTA that produce results that have to be evaluated by specialists can be eliminated. The improved DIN method is an attempt to get around with comparatively simple and low-cost manner in the standards. This is also true for the MAF method.

However, there certainly are some problems about the identification of the characteristic temperatures for biomass ashes. This is also true for variations (not studied within BioNorm) in the density of the test specimen that is produced by compression. Furthermore, the reproducibility has to be improved [20, 21, 22].

3.3 Particle size distribution and dimension

3.3.1 Objectives

For an unobstructed biofuel use (i.e. fuel handling, storage, transshipment and energetic conversion) particle size dimensions and their distributions in a bulk have often been identified as a key quality parameter. But, the determination of these parameters is still uncommon, since the provision of reproducible and comparable data on physical biofuel properties is a quite difficult issue. This is primarily due to high variability of measuring devices and principles and thus irregular international test standards. Coupled with test methods, there are several procedure-based sources of deviation, which are largely underestimated [24]. Particle sizes were investigated for 13 different conventional practice wood chip samples produced from debarked logs, whole trees, logging residues and wood thinning as well as two special prepared standard samples [23].

3.3.2 Methodical approach

For biofuel size classification several methods were tested in an international round robin that was conducted with two standard samples used as reference. For these standard samples the size parameters (i.e. maximum length, width and thickness) were determined by hand measurement using a digital calliper gauge. Furthermore, their weight was determined. Following a consistent length, standard samples were sorted into different size classes and coloured before sending to the round robin laboratories. In addition to the standard samples the conventional wood fuel samples were produced using four different chipper types, such as disc, drum, spiral chipper and shredder.

At the laboratories samples were analysed using four different methods represented by (i) horizontal screens, (ii) vertically vibrating screens as modification of the horizontal screen, (iii) rotary screens as well as (iv) image analysis system. The measures were carried out at different instrumental and procedure variations. For all screenings five progressively round hole diameters are applied.

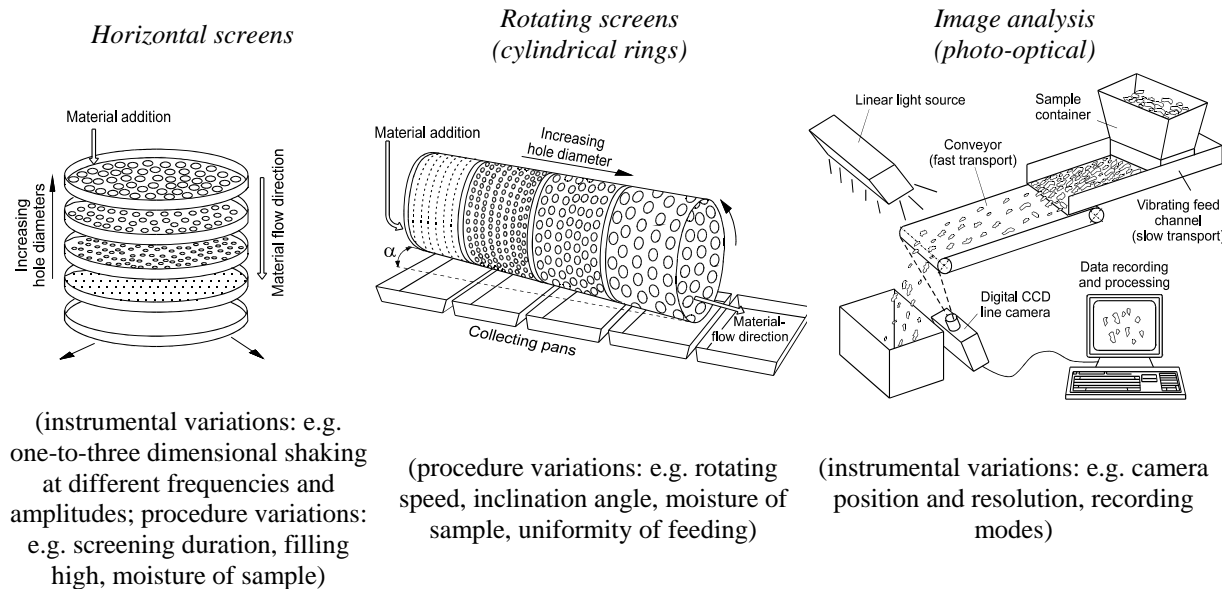


Figure 4: Functioning principles of screening and image analyses used in round robin trials [23, 24, 25]

In all trials about 8 litres of samples were passed. The usually used fractionating criterion is that of length distribution. Each sample was tested in three replications by means of every equipment, except for the image analysis system (five replications). The results of the test methods were compared on the basis of mean particle size and median value. Furthermore, the respective influences of various modes were investigated (i.e. moisture content, frequency, screening duration, feeding rate, rotation speed, inclination angle, mechanical wear as well as sample pre-treatment) [24, 25].

3.3.3 Conclusions and recommendations

The results revealed that the analysis of biofuels particle size is associated with high measuring uncertainties. This is basically due to the fact, that the tested major measuring principles (i.e. horizontal and rotary screening as well as image analysis) produced results which were largely incompatible. Results acquired from the image analysis system showed highest conformity to the reference values (standard samples). For all horizontal and vertical screening machines the median value of the size distribution (according to particle length) was only between one third to half of the reference median value. This is attributed to the high particle misplacement particularly found in larger fractions. For rotary screening the median particle length is between the results of image analysis and horizontal screening. Based on this, comparable measurements must consistently be made using only one of the three principles, while for the same principle modifications of the equipment type (e.g. different dimensional shaking operations) are usually acceptable.

With regard to influencing factors, for horizontal screening a critical shaking frequency (~ 190 rpm) was identified, while the chosen initial sample volume was found to be less important. For the screening duration a larger effect was observed; a fixed minimum time requirement of about 15 min was identified to be meaningful. For rotary screening the influencing factors are mainly the rotation speed and the inclination angle of the rotating drum. Also the feeding rate and the moisture content of the sample play an important role,

as reflected by the measured differences in the calculated median particle size. Generally, it should be attend to sample preparation due to the possible inhomogeneous moisture content within the sample. Coupled with this, the fixation of a tolerable moisture range is required if results from different fuels and test methods shall be compatible.

However, high reproducibility is only given, when all relevant measuring variables and the influencing factors are carefully considered and standardised accordingly [23, 24, 25].

3.4 Durability and raw density of pellets and briquettes

3.4.1 Objectives

The physical fuel quality of densified biofuels like pellets and briquettes is primarily characterised by durability (i.e. fuel resistance towards shocks and tensions) and particle density (i.e. ratio between mass and volume of a sample that is appropriate to estimate durability). Thus, durability is important with regard to handling, transportation and end-conversion processes where higher durability is required without falling into small pieces and dust [28]. The performance of existing methods have been tested and evaluated under technical and work-efficient aspects for different biofuels representative of the European market. Tested fuels are 15 pellets and 5 briquettes based on mixed wood, hard wood, soft wood as well as on agricultural residues [26, 27].

3.4.2 Methodical approach

Several test methods, devices and different procedures were investigated and evaluated by means of round robin tests involving five partners. Following the guidelines, each partner had to determine the moisture content of the appropriate fuels just before the tests.

Durability

Regarding durability determination, pellets were tested and compared: (i) using a tumbling device according to the American ASAE standard, and (ii) using a pneumatic tester based on the Austrian ÖNORM standard. The durability of briquettes was determined in a defined rotating drum in different test modes. The number of rotations is investigated in order to define the best appropriate rotating period. After treating in durability testers the samples (of pellets and briquettes) were screened with a sieve. The durability is calculated from the mass of sample remaining on the sieve and expressed as a percentage of the initial mass.

Particle density

Methods applied for particle density were (i) the stereometric method as well as methods based on liquid displacement such as (ii) the hydrostatic method and (iii) the buoyancy test method. Stereometric methods are based on measuring of sample dimensions (e.g. diameter, length, width, height) using e.g. a calliper rule or a palmer. The volume of the sample is estimated by calculating the volume of the nearest regular geometrical shape (e.g. cylinder, parallelepiped, cubic). Liquid displacement methods (see Figure 5) are based

on the Archimedes principle (i.e. an object is buoyed with a force equal to the weight of the liquid it displaces). The tests were performed for coated (by using paraffin) or non-coated samples in pure water as well as for non-coated samples in a mixture of water wetting agent. Accordingly, five methods were applied. Coupled with this, the increase of the volume, due to the increase of moisture content, was neglected.

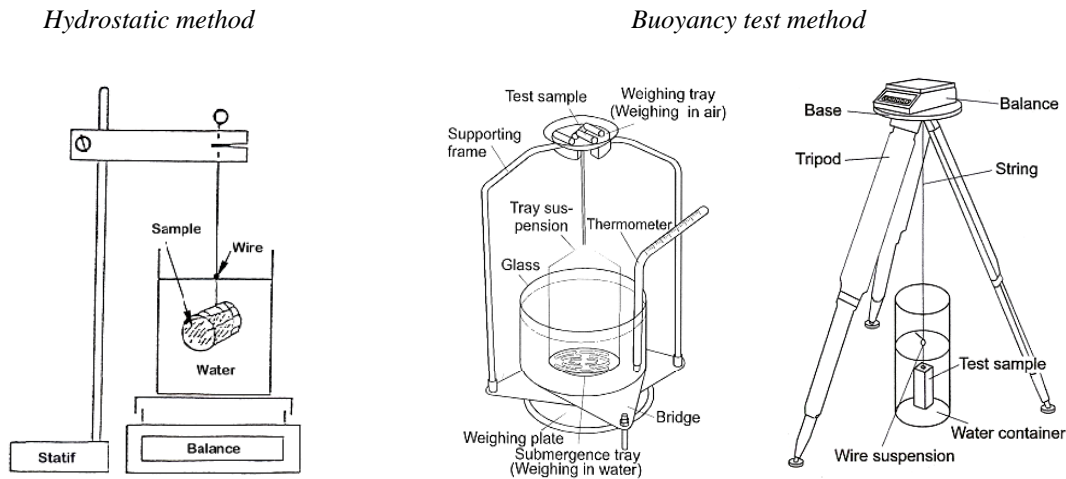


Figure 5: Liquid displacement methods applied for the determination of particle density [27]

The influence of tested methods and selected fuels on the particle density results was evaluated by calculating mean values and standard deviations (through 15 replications and participating laboratories). Furthermore, the repeatability and the reproducibility were calculated [26, 27, 28].

3.4.3 Conclusions and recommendations

Durability

For the estimation of the briquettes durability the most repeatable and reproducible method is to tumble the briquettes for 105 rotations corresponding to 5 min treatment. Nevertheless, the briquettes durability testing leads to highly variable results. But the variability of the method is influenced by the fuel itself and is smaller for briquettes of high durability. Furthermore, if all the tested briquettes are considered, it seems illusive to reach a higher accuracy than 10 %.

Also for determination of pellets durability, using the tumbling device (ASAE standard) shows better results compared to the pneumatic tester (ÖNORM standard). Though, there is no accurate relation between the results of pellets durability obtained by both devices. It clearly appears that the level of durability influences the variability of results: i.e. the lower the pellet durability, the higher the variability. Taking into account the number of replication needed, an accuracy level of 1 % could be reached in practice with the tumbling device.

Particle density

The different results revealed that the hydrostatic and buoyancy methods based on liquid displacement give lower repeatability and reproducibility than the stereometric methods and thus performed better. For all tested methods, it clearly appears that the fuels type influences the variability of the results.

For each analysed parameter the stereometric methods led to higher variability, higher bias between participated laboratories, higher values of repeatability and reproducibility and needed more replications to reach a given exactness.

The liquid displacement methods showed similar standard deviation. The values of repeatability and reproducibility were similar. This is particularly true for briquettes. Methods with addition of wetting agent give higher results than those obtained with paraffin coating methods. In the case of pellets, the buoyancy method using non coated samples and wetting agent mixed with water gives the lowest values in repeatability and reproducibility. However, the choice of the liquid displacement method used may be based on the material available at the laboratory. Thus, e.g. hydrostatic with paraffin coating needs to have a balance that can weight in a range of 10 to 15 kg with an accuracy of 0.1 g. Indeed applying buoyancy without paraffin coating decreases the time of sample preparation but the water has often to be changed because briquettes start to disintegrate very fast.

Generally, it is concluded that for given fuel types, the number of replications needed to reach a given accuracy level is by far smaller than for the others. It is suggested to further determine on which other parameters the number of replications (or the expected accuracy) may be based.

Comparing particle density and durability by using the most accurate methods for both parameters, no relation has been found between those two parameters. Thus, the particle density cannot be used to estimate the durability [26, 27, 28].

4 Chemical tests

For biofuel quality, in addition to physical-mechanical fuel properties also chemical properties are of high relevance. Appropriate test methods were evaluated within this work package (WP III) where about thirteen partners were involved.

Producers, traders and users of solid biofuels have to rely on reliable standardised methods for the determination of biofuels properties. This is particularly true with regard to fuel supply (i.e. handling and storage aspects) and to the utilisation of solid biofuels (i.e. predominantly thermo-chemical conversion in combustion systems), both associated with solid and gaseous emissions. Chemical biofuels characteristics are determined applying standards originally developed for the analysis of coal. Therefore, the exactness of existing procedures and the adaptation for solid biofuels had to be improved or new test methods had to be developed. Thus, this work package was concerned with the selection, evaluation and validation of suitable test methods for the determination of sulphur, chlorine and nitrogen content (Task III.1) and for major and minor elements (Task III.2). The results of these investigations provided the basis for preparing best practice guidelines and in turn were also destined for the respective CEN standards.

4.1 Sulphur, chlorine and nitrogen content

4.1.1 Objectives

To assess biofuel quality it is of high importance to know the concentration of sulphur, chlorine and nitrogen in biofuels. This is particularly true for biomass conversion processes. Contents of sulphur and chlorine in biofuels are of relevance for corrosion and fouling, for emissions of SO_x, HCl and PCDD/F as well as for aerosol formation. The concentration of nitrogen causes NO_x emissions. Standards defining analytical methods for the determination of these elements are not available for solid biofuels so far. Hence, different approaches and procedures are in use. The individual laboratory methods and system devices applied are originally designed for the analysis of coal samples, whereby certain deviations result. The analysed solid biofuels were selected with the intention to cover the most frequently traded biofuels in Europe and additionally cover a wide range of concentration of investigated elements (S, Cl, N). Testing methods were carried out for coniferous wood without bark, woodchips, bark, hardwood with glue, straw, rape straw, olive residues and herbaceous perennial cynara as well as hemp [36].

4.1.2 Methodical approach

Sample preparation and homogeneity tests

Depending on delivered biofuel size, sample preparation was performed stepwise using coarse cutting mills equipped with a 10 mm sieve and laboratory cutting mills equipped with tungsten carbide tools and sieves of 1 mm and 0.5 mm. The preparation is followed

by homogeneity tests by means of bomb combustion and determination of chlorine and sulphate using ion chromatography (IC). The results were evaluated with statistical methods. After distribution of the prepared homogeneous samples to the participating laboratories the elemental analysis for S, Cl and N was carried out [36].

Method investigation and improvement

For the determination of sulphur and chlorine existing procedures include methods based on (i) combustion (e.g. closed in a oxygen bomb or according to Wickbold, open in a high temperature tube furnace or automated) and (ii) digestion (e.g. high pressure acid in a closed vessel, in a open vessel according to Eschka) as well as (iii) AOX analyser, (iv) water soluble chlorine, (v) XRF (“X-ray fluorescence spectrometry”) and (vi) INAA (“irradiation neutron activation analysis”). Typically carried out subsequently, for S and Cl quantification methods such as IC (“ion chromatography”), ICP (“inductively coupled plasma”) and titration were used [35, 36]. Two standard methods applied for determining the nitrogen content in organic samples were used: (i) automated analysers and (ii) the Kjeldahl method. Initially, the analysis have taken into account all available methods for testing the prepared biofuel samples.

Based on the intermediate test results, not suitable methods for testing solid biofuels were excluded since they are not sensitive enough, difficult to handle or of poor reproducibility and repeatability. The most promising methods were selected to investigate in detail and to get improved. This involves [36]:

- sulphur: combustion in an oxygen bomb and quantification of sulphate by IC as well as acid digestion in closed vessels and quantification by ICP
- chlorine: combustion in an oxygen bomb and quantification of chloride by IC as well as the water soluble chlorine method
- nitrogen: automated analysers and the Kjeldahl method

Sensitivity analysis

For the identification of critical points in the analytical procedures, a sensitivity analysis was performed for selected parameter. This is applied for the influences of (i) moisture content, (ii) particle size, (iii) receiving solutions, (iv) oxygen pressure and (v) time for pressure release on the respective elemental determination. Moreover, for the separation of systematic deviations of independent analytical steps (i.e. combustion or digestion followed by quantification), both steps were investigated separately. Therefore, two approaches were performed:

- (a) A bomb digestion was prepared by one laboratory and send to all the other laboratories for the concentration analysis of sulphur and chlorine.
- (b) A bomb combustion was carried out by all laboratories and the content of sulphur and chlorine was measured by one laboratory.

The developed methods were finally tested using hemp as an additional sample [36].

4.1.3 Conclusions and recommendations

Concerning sample preparation, for S, Cl and N analysis a particle size of < 1 mm is sufficient in most cases. Smaller particles increase the repeatability but also increase the risk of contaminations with metals (i.e. from the inner materials of the used mills). The reproducibility of chemical analyses is usually improved when larger sample amounts are used (e.g. 1 g for the determination of S and Cl) [34]. Furthermore, in laboratory practise it should be especially considered that (i) the samples are always be within the calibration, and (ii) if low nitrogen concentrations are to be analysed, an increase in sample amount may improve the result.

Besides this, the method evaluation for element analysis led to the conclusions and recommendations given below. Basically, except the Kjeldahl method all of the identified most promising methods named above are recommended for standardisation. They are described in detail in the corresponding best practice guideline and draft standards [35, 36].

Chlorine and sulphur determination

Sample combustion in an oxygen bomb and the quantification of sulphate and chloride in the receiving solution is currently the best method. It enables the application of procedures presently standardised on European level.

For chlorine, the determination applying the method of water soluble chlorine led to similar results compared to the bomb combustion method (at least for all untreated biofuels investigated). For samples characterised by a high ash content even higher values were obtained.

In general, the required repeatability and reproducibility can only be obtained when strictly abided to the standard procedures. Solid biofuels that are characterised by low concentrations of sulphur and chlorine are difficult to analyse. Since currently the reproducibility is not satisfactorily, there is more need on research and method improvement. Coupled with this, the INAA method may be of interest for further scientific investigations, as for instance absolute values can be obtained. Furthermore, this method could contribute to improve and validate analytical methods.

Nitrogen determination

From the comparison of the obtained results for automated analysers (different and same brands and types) and for the Kjeldahl method the following was revealed.

With respect to certain types or brands of automated analysers available no systematic deviations were found. However, there were differences between the participating laboratories (persons operating the systems). Thus, for the obtained results the operation and especially the calibration is critical. Nevertheless, automated analysers are suitable for the nitrogen determination in solid biofuels and thus, involved in respective draft standardised. Since recognising comparable results for different designs of automated analysers, no specific system design is recommended in the draft standard. However, the applied apparatus should meet the functional requirements. No influence of moisture content on the nitrogen concentration could be found. Hence, it is not necessary to used dried samples which are difficult to handle since they are in general very hygroscopic.

Against the standardised methods developed so far, the work has generally revealed that there is further need on improvement with regard to the test methods, the lack of reference material and laboratory experiences as well as on own laboratory practice. Final recommendations involve to further investigate the chemical fuel concentrations of bromine and iodine (especially e.g. for recovered fuels, fruity biomass and seaweed) [35].

4.2 Determination of major and minor elements

4.2.1 Objectives

For appraising quality of solid biofuels important chemical parameters are the content of major (i.e. (Al, Ca, Fe, K, Mg, Na, P, Si, Ti) and minor (i.e. As, Ba, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Tl, V, Zn) elements. Major elements are of key relevance referring to ash melting, deposit and slag formation as well as to corrosion. Minor elements are of special importance for particulate emissions as well as the environmental impact of produced ashes and their subsequent utilisation [32]. Specific determination procedures for these elements in biofuels exist in few cases, but often are associated with considerable deviations between the results of testing laboratories. For the majority of investigations coniferous wood and bark mixtures and wheat straw are employed as test biofuels. Furthermore, olive residues and certified biomass reference materials were used [31].

4.2.2 Methodical approach

Sample preparation and homogeneity studies

To obtain satisfactory homogeneity spectrometry determinations (XRF and ICP-MS) were performed on three different particle sizes. For the actual tests about 10 kg wood and straw material were milled to homogeneous material of particle sizes < 0.25 mm and filled in bottles of 20 g each. Afterwards, prepared materials were analysed by spectrometry again and statistical F-tests were carried out to check within and between bottle homogeneity.

Method testing and improvement

Similar to the approach of Task III.1, in a first instance several advisable analytical methods were tested in order to choose the most suitable methods for further investigations. For this about 16 digestion and 8 determination (i.e. by means of several detection systems) methods were scrutinised. The digestion methods involved wet chemical digestion in closed vessels with different mixtures of HNO₃, H₂O₂, HF, HCl and HClO₄, heated conventionally or by microwaves. Furthermore, digestion tests were performed with or without neutralisation by H₃BO₃ as well as dry-ashing with subsequent dissolving in acids or dry-ashing with subsequent fusion in LiBO₂ or Li₂B₄O₇. The wet digestion procedures were followed by determination using (i) atomic absorption spectrometry (AAS) such as FAAS (“flame”), GFAAS (“graphite furnace”), CVAAS (“cold vapour”) and (ii) inductively coupled plasma spectrometry such as ICP-OES (“inductively coupled plasma optical emission spectrometry”) or ICP-MS (“inductively coupled plasma mass spectrometry”). These determinations showed good agreement between the different results for many elements investigated. Other determination methods

applied are hydride generation AAS (“atomic absorption spectrometry”), XRF (“X-ray fluorescence spectrometry”) as well as direct Hg determination.

Based on the results achieved with this procedure, in the second step the optimisation as well as improvement of a wet chemical digestion method was focused investigating the amount of HF required as well as different digestion temperatures (105 °C, 190 °C and 220 °C). For a broader applicability of the examined methods, olive residues as well as a certified biomass reference material were tested in addition to straw. Wood and bark mixture was excluded from these examinations due to restricted time and budget.

Method validation

Finally, based on the outcome of previous investigations different digestion methods for major and minor elements were validated. For that accuracy (i.e. precision and trueness) and application ranges as well as estimations of detection limits were examined [31, 32].

4.2.3 Conclusions and recommendations

In term of sample preparation, particle sizes < 1 mm or < 0.25 mm should provide satisfactory homogeneity to be used for the analyses of wood and bark mixtures or straw. Regarding this, it is noted to pay attention during size reduction in order to avoid contamination from the inner materials of the used mills. Thus, materials of that mill parts having contact with the sample should be chosen depending on the elements to be determined. If, for instance, minor elements such as Cr and Ni have to be determined, it is disadvised to use stainless steel materials, and recommended to use e.g. tungsten carbide or titanium instead. Generally, high-speed mills should not be used due to the higher abrasion rate.

In conclusion of the validation results, for solid biofuel analyses wet digestion with H₂O₂/HNO₃/HF/H₃BO₃ are proved to be the most suitable for the determination of major elements. For minor elements wet digestion using H₂O₂/HNO₃/HF are recommended.

Depending on the specific element to be determined, the application ranges revealed the suitability of tested methods for a wide concentration range, including potential concentrations in both natural and contaminated solid biofuels [32]. According to detection limits, the most suitable determination methods are summarised for the different elements in solid biofuels in Table 3.

Table 3: Recommended detection systems for major and minor elements [31, 32]

Detection system	Major elements	Minor elements
FAAS ^a	Ca, Fe, K, Mg, Na, Si	Mn, Zn
ICP-OES	Al, Ca, Fe, K, Mg, Na, P, Si, Ti	Mn, Zn, Ba, Cr, Cu, Mn, Ni, V, Zn
GFAAS ^b	-	Cd, Cr, Cu, Ni, Pb
ICP-MS	P, Ti	As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, V, Zn
Direct Hg determination	-	Hg
CVAAS	-	Hg

^a because of relatively poor detection limits, Al determination by FAAS is not recommended for solid biofuels containing low concentrations of this elements

^b because of relatively poor detection limits, As, Co, Mo, Sb and V determination by GFAAS is not recommended for solid biofuels containing low concentrations of these elements

Basically, applying XRF detection would be a suitable and fast alternative method for the determination of several major and minor elements. However, due to the requirement of reliable calibration standards, which are not available for solid biofuels so far, XRF systems for element determination presently not be recommended. Thus, the development of such standards is desirable [31].

5 Fuel Quality Assurance

Closely linked to the previous work packages, this work package (WP IV) dealt with Quality Assurance systems for the provision of solid biofuels. Six partners were involved in the elaboration of this topic.

5.1 Objectives

Standardisations of solid biofuel properties contribute to promote a more widespread use of biofuels by providing a base to facilitate the business of operators within the market. Besides this, a Quality Assurance system that takes the whole provision chain of solid biofuels into account is essential for an increased use and trade of biofuels. This system provides tools to demonstrate that the level of quality demanded by customers is achieved. Hence, activities on standardisation have to be accompanied by the development of a Quality Management (QM) system with the main focus on Quality Assurance (QA) and Quality Control (QC). However, currently no Quality Management system exists in practice in Europe, which fulfils these demands. Therefore, the aim of WP IV was to fill gaps of knowledge concerning Quality Assurance in the field of solid biofuels taking requirements from practice into consideration [11].

This has been achieved in three tasks taking into account that the resulting documents are applicable for operators dealing with solid biofuels:

- Objective of the first Task IV.1 was a review of existing relevant Quality Management systems.
- Emphasis of Task IV.2 was the development, implementation and improvement of a guideline for Quality Assurance by field trials to gain experience from practice. It was aimed to provide a methodology on how to develop and implement a Quality Assurance system (including Quality Control) within a company dealing with solid biofuels.
- The development of a proposal for a standard for Quality Assurance was object of Task IV.3 in order to directly support the Technical Specification on Quality Assurance for solid biofuels.

Considered biofuels are conform with the scope of Technical Specification “Fuel specifications and classes” [13].

5.2 Methodical approach

Following the objectives, initially there was an extensive review of existing, relevant QM systems (e.g. in accordance with the international standard ISO 9001:2000) already applied by producers, operators and users of solid biofuels. This review covers solid biofuels and excludes recovered fuels. Existing QM systems were analysed in ten different cases on the basis of a question list in order to identify (i) how QA and QC are currently performed, (ii) what are the main components as well as (iii) what are the needs and demands.

Furthermore, pros and cons of QM systems were discussed. Based on these results conclusions were drawn for the design of a guideline for QM with emphasis on QA and QC for solid biofuels.

The guideline sets out a step-by-step methodology in order to support each operator within a supply chain of solid biofuels to design a manual for Quality Assurance. Draft manuals were implemented and tested out in practical conditions. This was accomplished in “field-trials” at the industrial premises of a range of producers, traders and users of solid biofuels, referred to as host companies. The choice of host companies was well balanced covering main types of European biofuels and conversion processes and considered geographical distribution over Europe. This involves companies that produce refined solid biofuels and companies that buy such up-grade biofuels for end use. Furthermore, a wide range of possible circumstances has to be covered by including all cases between small-scale users (especially domestic, who require high-grade fuels in installations with narrow fuel specifications) and large-scale users (who can benefit from lower-cost fuels by using fuel flexible conversion units). The field trials were divided into three main steps [13]:

- (1) to develop a methodology on how to implement a QM system with emphasis on QA and QC in companies, and to write a first draft of a guideline for the development and implementation of such a system in companies
- (2) to investigate the implementation in different companies throughout Europe
- (3) to develop, evaluate and update the guideline for the development and implementation of QA and QC in companies

Finally, a proposal for a standard for Quality Assurance was elaborated.

5.3 Conclusions and recommendations

According to the different tasks, the results were briefly summarised as follows based on [11, 12, 13].

5.3.1 Review of quality systems

The review has clearly shown the need of the adoption of a QM system for solid biofuels with emphasis on QA and QC. Accordingly, aspects such as defining the product quality, traceability, documentation, statistical control, testing and sampling are of importance.

Because ISO 9001 is the most commonly applied Quality Management standard, it is considered meaningful to prepare the guideline for Quality Assurance and Quality Control using the terminology as used in ISO 9000.

Since biofuel product quality is defined in different manners, it is therefore concluded to define product specifications on a case-by-case basis by the market itself, not by prescribing normative fuel parameters by an external organisation. Being of value for biofuel end-users it is recommended to implement a system of tracing back the origin of the biomass raw material used, e.g. by means of delivery notes or batch identification numbers. Linked to this, a QM system for solid biofuels should demand that the different

owners within the entire provision chain are responsible for the documentation and filing of all relevant information on treatment, storing and transport of the biofuel under their specific ownership. In addition, the current owner should be responsible to provide all information back through the provision chain at all times via a batch number.

One important part of a QM system is the design of an appropriate sampling and testing procedure that provides confidence to customers that promised fuel specifications are always met. The costs of implementing such procedures within a biofuel production process depend on the selection of process parameters to be tested and the frequency of testing. In turn, the need for sampling certain process parameters depends on the type of production process, the variability in used raw materials and the allowable product variations. Therefore, the sampling and testing procedure is needed to be custom made.

5.3.2 Implementation of Quality Assurance in companies

The field trials have underlined the need for a guideline with a general methodology applicable by each operator throughout the supply chain of solid biofuels. The guideline is aimed to assist all operators to develop an appropriate manual for Quality Assurance according to their specific needs. It provides a better understanding of the issues, thereby enabling to design appropriate measures to control and assure quality.

Quality Assurance aims to provide confidence that a steady quality is continually achieved in accordance with customer requirements. However, the supply chains of solid biofuels can consist of different process chains and/or process steps, which can be distributed among different companies or organisational units. In this definition, the customer is the next operator (company or organisational unit) within the supply chain. The customer requirements, i.e. demanded quality, include not just the quality of product but also the quality of the company's performance. The quality of solid biofuels can be defined in terms of a number of key properties that relate to the suitability of the fuel for a specific use. Therefore, the quality of performance refers to documentation, timing and logistical issues.

The methodology, briefly described hereafter and illustrated in Figure 6, can operators apply to design a suitable QA system for a specific chain. Besides an efficient control of the processes considered, the methodology ensures also the control over the overall provision chain by integration of previous and subsequent process steps of other organisational units. It is recommended to document each implementation to these different steps of the methodology within a specific company in a (site-)specific manual, which can serve as an appropriate tool to illustrate to different parties that all the processes and their interaction are fully under control.

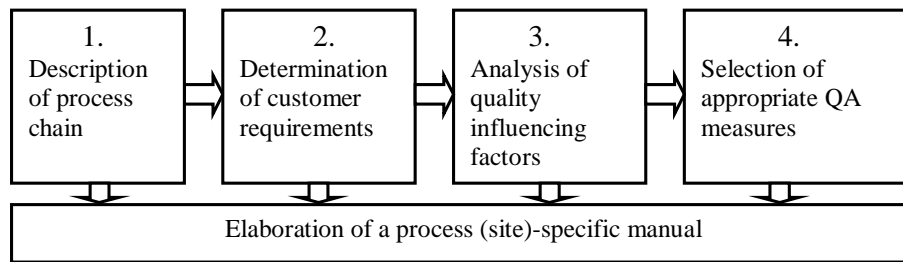


Figure 6: Methodology to develop and implement Quality Assurance (QA) [13]

Step 1: Description of process-chain. The first step is to describe the production process in the company including the different sub-processes at a very detailed level. Coupled with this, it should carefully be assessed which steps of the production process are significant for realising an appropriate QA. This is of vital importance for the following drafting of the QA manual.

Step 2: Determination of customer requirements. The position of the process unit(s) considered within the overall provision or process-chain is essential for QA because the customer requirements depend on previous and subsequent process steps. In principle the requirements of the next operator of the overall provision chain have to be fulfilled. Due to a lack of knowledge the fuel end user is unable to provide adequate Technical Specifications of the fuel. Hence, key properties of the final product should be determined either by a standard that specifies which fuels can be used, or by the fuel supplier that through knowledge of the user needs is able to define such specifications. Such an indirect demand is to be understood as requirement from a subsequent step.

Step 3: Analysis of quality influencing factors. After the description of the process unit(s) and the analysis of customers requirements the process should now be examined concerning factors considered to be most influential in terms of fuel quality. The following factors are involved in general in determining fuel quality and refer to the management of the company:

- effectiveness of preliminary inspections of fuel sources
- effectiveness of checking of incoming loads
- appropriateness of applied methods to handle, store and process materials
- quality-control measures adopted (frequency of testing)
- company management and responsibility
- qualification and knowledge of staff

Step 4: Selection of appropriate Quality Assurance measures. In accordance with the results of step 1 to step 3 appropriate QA measures (i.e. measures giving confidence) have to be identified and applied. Aspects to be taken into account are e.g.:

- allocation of responsibilities and elaboration of work instructions

- identification and documentation of Critical Control Points (CCP`s) and application of Quality Control (QC) measures
- training of staff and proper documentation of processes and test results
- failure mode and effect (FME) analysis

The field trials have clearly shown, that all parties who are active in the overall provision chain for solid biofuels (including suppliers of raw materials, producer of biofuels, transport and perhaps one or more retailers) should be integrated within an overall QA system. Based on the methodology described above an appropriate QA system can be implemented easily in a company dealing with solid biofuels, which fulfils those demands. The methodology ensures thereby a system that is simple to operate without undue bureaucracy, and which offers savings in costs.

5.3.3 Proposal for a draft standard for Quality Assurance

The proposal of a standard based upon the Technical Specification (TS) "Solid biofuels - Fuel Quality Assurance" under development in TC 335/WG 2 was aimed to guarantee solid biofuel quality through the whole supply chain and to provide adequate confidence that specified quality requirements are fulfilled. Main stage of this chain from the origin to the delivery of the solid biofuel can be gathered from Figure 7.

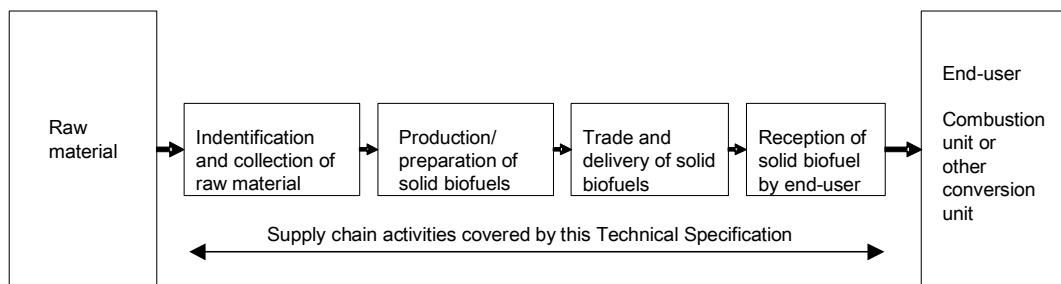


Figure 7: Supply chain of solid biofuels [12]

The objective of this document is to serve as a tool to enable efficient trading of biofuels. Thereby the end-user can receive a fuel, which corresponds to his needs, and the producer/supplier can produce a fuel with defined and consistent properties. Besides instructions how to fulfil the requirements of the standard, general information is given about QA and QC explaining the approach and benefits of implementing an adapted QM system. This also involves QA measures (e.g. production requirements, transportation, handling and storage as well as quality declaration) and QC (e.g. specifications of biomass origin, source and of traded from as well as determination properties).

6 Summary

Due to its advantages (like wide availability, well-known with regard to supply and use and contribution to green house gas mitigation) biomass represents the most important source to address European policies increasing the share of renewable energy sources for a more sustainable energy system. For an improved integration of solid biofuels into the energy system a dynamic European market has to be created. Thus, accomplishable biofuel properties as well as procedures to test and control these defined parameters are required. Therefore, besides the elaboration of an overall Quality Assurance system for solid biofuels based among other on sampling and testing methods, the pre-normative research project (BioNorm) was aimed to directly contribute to the ongoing development of European standards for solid biofuels within CEN TC 335 “Solid biofuels”. Within this project the emphasis was laid on the identification and evaluation of the best appropriate sample-, test- and reference methods for the determination of specific fuel properties. Based on the experiences made respective best practice guidelines were compiled. These guidelines and research findings were incorporated for drafting Technical Specifications within the standardisation process.

In each of the different work packages and tasks respectively various kinds of solid biofuels covering a wide scope of fuels were analysed and investigated by carrying out selected methods. The applied methods and principles were initially identified to be basically suitable for a reasonable determination of (i) the number of increments and tests as well as specific increment sizes in terms of sampling and sample reduction, (ii) the physical/mechanical fuel properties (i.e. moisture content and bulk density, ash melting behaviour, particle size distribution, durability and particle density) and (iii) the chemical fuel characteristics (i.e. sulphur, nitrogen and chlorine as well as major and minor elements). The research has two emphasises. It was focused on the investigation of existing methods and equipment (e.g. applied for solid fossil fuels) with regard to their accuracy for solid biofuels and thus their applicability followed by appropriate adaptation and improvements of these methods and laboratory equipment. In addition, it was also focused on the development of new methods. Referring to this, common statistical parameters such as accuracy, reproducibility and repeatability were used for the assessment of the different sampling and testing methods. Besides technical, also work-efficiency, economical and environmental aspects were considered.

Supported by these research results, new methods for the development and implementation of Quality Assurance systems for the entire biofuel supply chain were evaluated based on an initial review of existing systems as well as extensive field trials at several companies.

According to the work content of the BioNorm project, the results of the respective sampling and testing methods are summarised for the range of investigated and analysed solid biofuels in the following (see Table 4).

Table 4: Summary of preferred methods for investigated solid biofuels (to be continued next page)

		Origin and source of solid biofuels ^a				Recommended methods / determination devices / procedures	Best practice guidelines available	CEN TC 335 Draft Standards available
		Woody biomass	Herbaceous biomass	Fruit biomass	Blends and mixtures			
Sampling and sample reduction	Sampling	<ul style="list-style-type: none"> GROT^b sawdust pellets 	<ul style="list-style-type: none"> straw bales 	-	-	<ul style="list-style-type: none"> number and size of increments to take for a bulk sample given for methods determining moisture, ash content, size distribution and chlorine content * GROT and sawdust: heap / conveyor * pellets: heap / bags * straw bales: core / hook 		x
	Sample reduction					<ul style="list-style-type: none"> methods determining moisture, ash content, size distribution, chlorine content * GROT: riffle * sawdust: riffle / coning / long pile * pellets: rotary divider / riffle * straw bales: coning / ling pile / hand 		x
Physical-mechanical tests	Moisture content	various kinds of <ul style="list-style-type: none"> bark chips pellets residues needles 	various kinds of <ul style="list-style-type: none"> triticale grain pellets rape cakes 	various kinds of <ul style="list-style-type: none"> stones/ shells olive cake 	<ul style="list-style-type: none"> peat 	<ul style="list-style-type: none"> reference methods: <ul style="list-style-type: none"> oven drying, freeze drying, xylene distillation, GS-MS method rapid methods: <ul style="list-style-type: none"> on-site types: thermogravimetric Mettler, capacitive Pandis and Schaller on-line type: optical MESA 	x	x
	Bulk density					<ul style="list-style-type: none"> cylindrical container shape à 50 litres standardised shock impact for increasing bulk density for data comparability correction factor of 0.712 % per 1 % moisture difference for inconsistent moisture content between samples neglecting of effects based on moisture content above 25 % (MC) 	x	x
	Ash melting behaviour	various kinds of <ul style="list-style-type: none"> bark 	<ul style="list-style-type: none"> straw Lucerne 	<ul style="list-style-type: none"> olive stones 	-	<ul style="list-style-type: none"> ash sample preparation at 550 °C (CEN TS 14775) quantify melting temperatures: improved DIN and MAF reference for agglomeration /sintering: CFBA ash composition and species: SEM-EDS promising for further ash behaviour but not suited for standard: TGA/SDTA 		x
	Particle size distribution and dimension	various kinds of <ul style="list-style-type: none"> chips 	-	-	-	<ul style="list-style-type: none"> for comparability measurements have to consistently made using on of the three methods: <ul style="list-style-type: none"> horizontal screening (critical shaking frequency ~ 190 rpm, fixed minimum time 15 min) rotary screening image analysis 	x	x
	Durability	various kinds of <ul style="list-style-type: none"> pellets briquettes 	various kinds of <ul style="list-style-type: none"> pellets 	-	-	<ul style="list-style-type: none"> briquettes: tumbling drum for 105 rotations corresponding to 5 min treatment pellets: tumbling device (ASAE standard) depending on fuel properties: the lower the durability, the higher the variability 	x	x
	Particle density					<ul style="list-style-type: none"> methods based on liquid displacement: buoyancy and hydrostatic choice of method according to the material/devices available at the laboratory pellets: buoyancy method using non-coated samples and wetting agent mixed with water 	x	x

		Origin and source of solid biofuels ^a				Recommended methods / determination devices / procedures	Best practice guidelines available	CEN TC 335 Draft Standard available
		Woody biomass	Herbaceous biomass	Fruit biomass	Blends and mixtures			
Chemical tests	Sulphur, nitrogen and chlorine content	<ul style="list-style-type: none"> coniferous wood woodchips bark hardwood 	<ul style="list-style-type: none"> wheat straw rape straw cynara hemp 	<ul style="list-style-type: none"> olive residues 	-	<ul style="list-style-type: none"> sample preparation to particle sizes of 1 mm sample amounts of 1 g for sulphur and chlorine increase of sample amounts in case of low nitrogen concentration determination methods: <ul style="list-style-type: none"> combustion in an oxygen bomb and quantification of sulphate and chloride by IC acid digestion in closed vessels and quantification by ICP for sulphur water soluble chlorine automated analysers for nitrogen 	x	x
	Major elements	<ul style="list-style-type: none"> coniferous wood-bark mixture 	<ul style="list-style-type: none"> wheat straw 	<ul style="list-style-type: none"> olive residues 	-	<ul style="list-style-type: none"> sample preparation to particle sizes of < 1 mm or < 0.25 mm detection systems: <ul style="list-style-type: none"> FAAS for Ca, Fe, K, Mg, Na, Si ICP-OES for Al, Ca, Fe, K, Mg, Na, P, Si, Ti ICP-MS for P, Ti 	x	x
	Minor elements					<ul style="list-style-type: none"> sample preparation to particle sizes of < 1 mm or < 0.25 mm detection systems: <ul style="list-style-type: none"> FAAS for Mn, Zn ICP-OES for Mn, Zn, Ba, Cr, Cu, Mn, Ni, V, Zn GFAAS for Cd, Cr, Cu, Ni, Pb ICP-MS for As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, V, Zn Direct Hg determination & CVAAS for Hg 		
Fuel Quality Assurance		considered for all relevant solid biofuels through the entire supply chain				<ul style="list-style-type: none"> development and implementation of Quality Assurance (QA) systems in companies <ul style="list-style-type: none"> step 1: description of process chain step 2: determination of customer requirements step 3: analysis of quality influencing factors step 4: selection of appropriate QA measures 	x	x

^a For more detailed information on biofuels analysed and tested please refer to the respective final reports in Part 2 "Detailed Final Reports"

^b kind of wood chips

The outcome of the pre-normative work of the BioNorm project includes among methods for sampling and sample reduction, improved and new developed methods and procedures for the determination of physical-mechanical and chemical biofuel properties as well as the development and implementation of a company specific Quality Assurance system. Coupled with this, basic recommendations are with regard to apply both the proposal of a standard/TS and the guideline in practice.

In terms of the country conditions in the NMS/NAS recent trends have shown a continuous increasing international market of solid biofuels that also stimulates the domestic production of refined solid biofuels within these countries and thus increase the acceptance of renewable energy sources. Nowadays, these countries already use biomass (predominantly for domestic heat provision) and have very promising potentials concerning biomass and bioenergy utilisation. However, currently limited experience in utilisation of refined solid biofuels and missing R&D contributes to a lack of solid biofuels standards and Quality Assurance guidelines. In all of the NMS/NAS there are no specific biofuel standards and Quality Assurance implemented yet. Companies that produce refined solid biofuels for export currently apply national standards of the import countries. Referring to this, all NAS/NMS-partners clearly stated that common standards are urgently required for increasing the solid biofuel market. Hence, CEN standards currently being developed need to be quickly adopted.

Among the extensive and substantial outcomes during the pre-normative research and the progress of standardisation it has been acknowledged that there is still the demand on research in all purviews such as of fuel classification and Quality Assurance or sampling and testing methods and procedures. In this context, essential recommendations on further work are:

- to precise and revise data on sampling and testing methods
- to extent sampling and sample preparation to a broader range of solid biofuel materials, i.e. less common biomass with high variations in fuel properties such as olive and grape residues, shells and stones of fruit biomass as well as road side green
- to extent testing on further fuel parameters (e.g. particle size and shape factors, bridging behaviour, fuel impurities, fungi release)
- to improve and develop reference test methods and rapid test methods (e.g. for ash melting behaviour, chlorine potassium, nitrogen and sulphur as well as heavy metals and with regard to reference materials)
- to investigate aspects of Quality Planning and Quality Control on interactions of the various quality related activities in the quality policy of a company as well as to adapt measures of with regard to Quality Management systems

Moreover, further cooperation is recommended within the European countries, that already used biomass efficiently and their legislation and biofuels standards are harmonious developed.

Finally, the outcomes reveal that BioNorm has crucially contributed to clarify important issues and aspects associated with the ongoing European standardisation process for solid biofuels. This is in particular true referring to biofuel terminology and specification, appropriate procedure and methods for sampling and testing of fuel properties as well as the importance of company specific Quality Management systems. Furthermore, a basis for research exchange with the NMS/NAS has been established that will be further delved and extended in the future. However, BioNorm has also point out the urgent need on further investigations and method development. Therefore, continuing R&D support of the ongoing standardisation activities will be of high importance to close the gaps and to ensure that the European market for solid biofuels and thus the biomass industry will continue to grow rapidly in the future helped by acceptable standards.

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