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## **BioNorm II**

**Pre-normative research on solid biofuels for improved European standards**



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## **Review for online measurement possibilities of chemical parameters for process control of industrial solid biofuel production**

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## Abstract

The development of European Standards (EN) is a precondition to expand the market for solid biofuels and a very important step to fulfil the political and environmental goals of the European Union.

The aim of the BioNorm II project is to carry out pre-normative research in the field of solid biofuels in close collaboration with the work of CEN TC 335 "Solid Biofuels". This includes the development of an overall quality assurance system for solid biofuels, their characterization and corpus of legislation.

In WP II Task II the work focuses on the development of chemical test methods. This includes the verification and limitations of existing methods, but also the development of rapid test methods and online measurements for fuel quality control.

High and constant product quality of industrially produced biofuels like pellets could benefit of more or less continuous quality control of the raw material and product. However, currently only the on-line measurements of the moisture content using infrared sensors is state of the art but even this technology is still not frequently used in solid biofuel production.

The future large scale production of industrial biofuels, aiming for the use of biomasses like olive stones, straw, grasses or other agricultural residues would benefit from an extended quality control including the chemical parameters chlorine, alkali metals and silicon. Such material classification may actually lead to constant and well characterized chemical properties of the fuels but also eventually to an increasing number of fuel qualities or even to fractions regarded as waste.

At present on-line systems are not commercially available for solid biofuels. A review of possible analytical principles and systems which are used in other fields and which may be applied for biofuel analysis seems a suitable step to induce a possible product development.

Analytical principles and technologies were evaluated on a theoretical basis in order to check whether they may be used as online measuring devices for production (for raw-materials and products) control. This study includes technological aspects as well as an assessment of the demands and requirements of these systems from solid biofuels industry.

### List of Abbreviations

dm	Decimetre
g	Grams
GHz	Gigahertz
h	Hour
IR	Infrared
keV	Kilo electron volt
kg	Kilogram
MeV	Mega electron volt
mg	Milligramm
MHz	Megahertz
min	Minute
mm	Millimetre
NIR	Near Infrared
PGNAA	Prompt gamma neutron activation analysis
t	Tonne
TDR	Time-Domain-Reflectometry
VOC	Volatile organic compound
XRF	X-ray fluorescence analysis

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## 1 Introduction

First on-line methods in fuel sector were developed for coal industry. The mainly monitored parameters of the fuels are moisture and ash content. These are important parameters both in fuel upgrading and use. Moisture content measurement is naturally relevant for solid biofuels and raw material while ash is not usually a great matter of interest. Although ash components contribute to slag formation, the content of ash itself does not indicate the tendency of slagging and fouling.

More or less similar requirements consist for on-line analysis systems for solid biofuels. Ash content of wooden biofuels is small compared to that of mineral fuels and it's limiting the use of these technologies, but for biofuels with high ash contents (e.g. olive cake) these systems are maybe adaptable. The same applies to sulphur and other contaminants which are in low concentration in some bio-fuels like wood.

For other chemical parameters also methods exist. E.g. some methods are capable to measure chemical constitution of fuels like prompt gamma neutron analysis (PGNAA) and X-ray Fluorescence. Methods measuring certain chemical components (like alkali metals or silica) are interesting but expensive to install.

In case of solid biofuels it is of course a matter of volume, too. Compared to coal industry, biofuel production site and plants are usually small or medium-size units with simply non-sophisticated technology (like X-ray or neutron activation) which usually sets limits for instrumentation as well.

All in all, the need of measuring chemical parameters of solid biofuels on-line in present situation is limited as biomass is presently used mostly in plants specially designed for biomass properties and these plants are operated without major problems using existing quality control measures. In some special cases like pellets production, where low level of ash or impurities is required, the situation may be different. Also in case of co-firing e.g. with coal, situation may be different.

First it should be evaluated which chemical properties should be beneficially to be measured continuously (on-line). Following parameters are discussed:

- Moisture (existing but not really widespread)
- Calorific value (only in large variation)
- Ash (total), not generally needed; the ash of pellets and raw material for pellets production is an exception
- Chemical parameters
- Alkali metals (K, Na) for slagging and fouling
- Silica also for slagging
- Chlorine (for herbaceous biofuels or olive cake and kernels)
- Heavy metals (Cd)?

For the moment there is not enough information on possible benefits gained through extended (but costly) measurement approach, especially if stricter chemical quality criteria than to-day are not set for bio-fuels or bio raw-materials.

The following review is mainly based on existing methods and on experiences in coal industry as on-line analysis systems have found widespread application in this sector. The continuous analysis and rapid response of these systems has led to improved control of mining, processing and blending operations with increased recovery of valuable minerals and coal combustibles. A fast, accurate,

real-time method of determining the elemental composition of coal is important to the coal industry for things such as control of fouling and slagging, fuel cost, boiler optimization, reduction of SO<sub>2</sub> emissions and so on. The analyzers can rapidly measure large volumes of coal without the need for preparing samples for laboratory analysis, even though in many case a side stream with further treatment of fuel is needed.

## 2 Methods for moisture determination

Moisture is one of the most important quality affecting parameter of solid biofuels. A lot of the used techniques in coal industry is already adopted and optimized for solid biofuels. Methods for the determination of moisture in coal are described in the ISO 15239:2005 and in annex A also on-line determination methods are included. [1]

Determination of the moisture content by measuring the loss of mass after drying in a drying oven is time-consuming for on-site quality control. However, it is the reference method, reliable and often used in biomass power plants, pulp- and paper industry etc. A need for a fast and reliable detection methods is evident. In recent years, some already established methods such as infrared reflectance were optimised for online use in solid biofuel production, whereas some other interesting new methods such as TDR have been developed.

The following methods are on-line methods but also some of them are for the stationary off line use. These methods are also described here because of the high importance of moisture monitoring and since they offer an alternative to the high costs of online measure equipment.

### 2.1 Methods overview

Illustration 1 gives a brief overview of the relevant moisture analysis methods for biofuels.

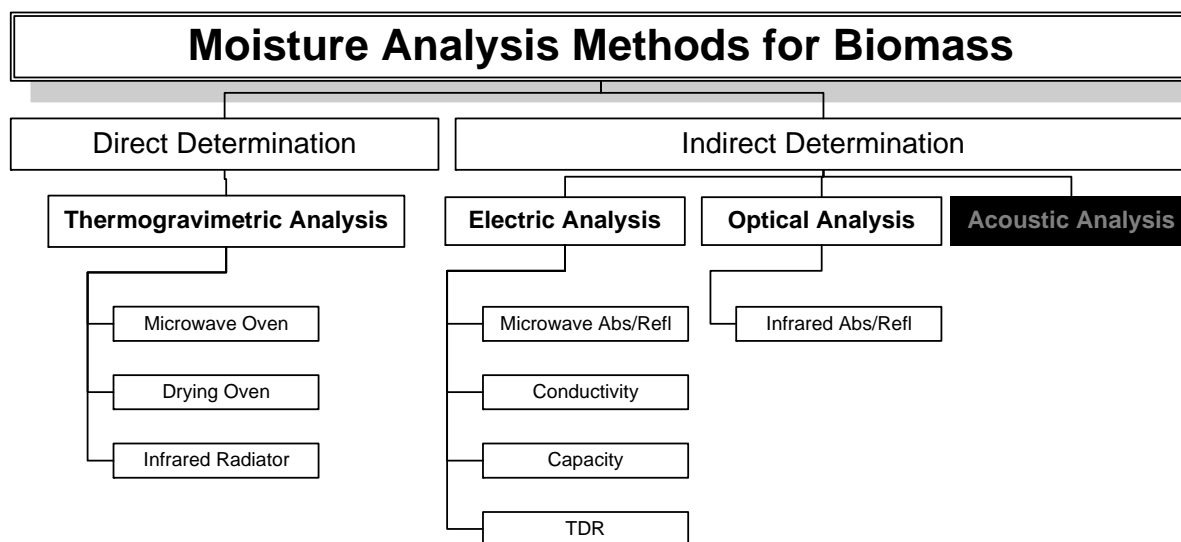


Illustration 1: Overview of moisture analysis methods for Biomass [2]

## 2.2 Direct determination

### 2.2.1 Thermogravimetric analysis

Thermogravimetric methods have a high measuring accuracy but they are usually time-consuming, stationary off line methods.

#### Drying oven

This technique is often used as a reference method (see prEN 14774). The sample is dried in a drying oven till constant mass is obtained. For biomass samples, VOC's are a problem since they adulterate moisture analysis. Significant reduction of the oven temperature solves this problem only partially because the duration of the measurement increases drastically (up to 1-2 days) and some moisture will remain in the sample. In general, acceptable results are obtained at 105°C (CEN/TS 14774). A measurement needs between 6 and 24 hours depending on particle size and material. This method is suitable for all solid biofuels. Drying ovens are in the market for 1.000.- € and more.

#### IR drying

Sample of less than 100g is placed on a balance under an IR radiator, which emits wavelengths that are absorbed by water, which is heated up and evaporates. VOC are hereby evaporated as well. Measurements are much faster than those performed with a drying oven, they take between 7 and 48 min, they are terminated automatically. This method is not suitable for larger pieces like log wood, but this is a good stationary off line method for biomass quality control during the production of pellets. Simple instruments are available from 1.000.- €, more sophisticated devices are available at around 5.000.- € and more.

#### Microwave oven

With microwaves, water can be evaporated specifically. Heat is generated by a homogenous microwave field, inducing the dipole of water molecules and exciting them. This leads to an increase in temperature. The heated water diffuses to the particle surface where it evaporates, causing a loss of mass to the particle. This loss of mass on drying is measured in 2-20 min, making the microwave oven method considerably faster than other thermogravimetric methods. This method is like all gravimetric methods also a stationary off line method.

Microwave ovens for home use cannot be used for moisture measurements, because of their inhomogenous microwave field. Professional microwave ovens are available at around 10.000.- €

## 2.3 Indirect Determination

Methods which determine the moisture content indirectly have the advantage of being faster and non-destructive, sometimes even non-invasive. On the other hand, calibration is required. Some methods can be automated very well, even on line analysis is possible.

### 2.3.1 Electric Analysis

#### Conductivity

The handling of this technique is rather simple (ram-in electrodes); the electrodes of the analyzer has to be recess into the wood log and out of the measured conductivity the moisture content can be calculated. This method is only applicable to wood logs, provided a characteristic curve for the type of wood that has to be analysed is available. Measurement of conductivity is not possible for high moisture contents. It has to be considered that the exact placement of the electrode has an influence on the accuracy and repeatability of the measurement, since wood logs are not completely homogenous. The illustration below shows the core principle of a conductivity moisture analyzer. These instruments cost around 500.- € extremely simple devices around 200.- €

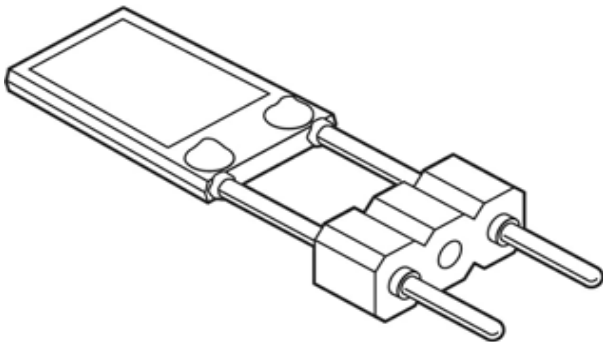


Illustration 2: Principle of a conductivity moisture analyzer [3]

#### Capacity

A few dm<sup>3</sup> of the biomass sample are used as dielectric of a capacitor. Since water has a much higher permittivity than dry wood, the moisture content can be determined by measuring the capacity. It has to be noted that factors like bulk density, frequency of measurement, layer thickness and ionic conductance have an influence on the result, which can be minimised, if they are held constant during the measurement.

Capacity measurements have the advantage of a large linear range of the calibration. To avoid perturbation by ionic substances and short circuits, measurements have to be made at high frequencies (>100MHz).

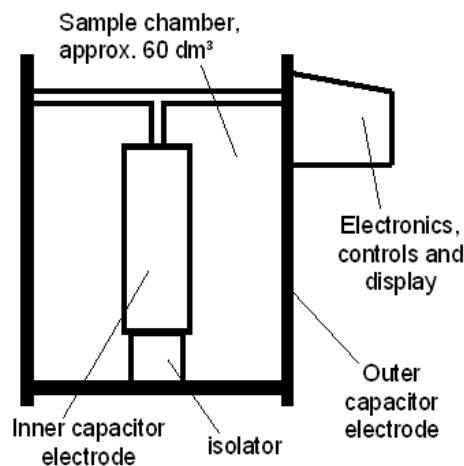


Illustration 3: Principle of a capacity moisture analyzer

### **Microwave absorbance/reflectance**

With this technique, microwave absorbance/reflectance are determined as a function of moisture content. Perturbation by ionic substances is minimised by the frequency of up to 20GHz. An advantage of this method is that results are available after a few seconds but the absorption behaviour becomes increasingly non-linear with higher moisture contents. Simple handheld instruments are available from 2.000.- €

### **TDR (Time-Domain-Reflectometry)**

Time-Domain-Reflectometry uses the reduction of the velocity of propagation of electromagnetic waves in media with high permittivity. A critical point in this method are control electronics: Times as short as a nanosecond have to be resolved. TDR devices are designed for stationary use and rather expensive.

## **2.3.2 Optical Analysis**

### **Infrared absorbance/reflectance**

Infrared absorbance or –reflectance techniques use IR-radiation of a wavelength that is absorbed by H<sub>2</sub>O. The intensity of the absorbed or reflected IR-beam is then compared to the intensity of an absorbed or reflected beam of a wavelength that is not affected by water. So, at least two different IR beams have to be used. A major disadvantage of this method is the low penetration of IR rays. In fact, IR reflectance determines the moisture content of the surface only. These devices are in the market for around 10.000.- €

## **2.3.3 Acoustic Analysis**

Acoustic moisture measuring techniques in wood are currently a research topic and not yet ready for the market. Thus, a manufacturer of ultrasound moisture meters for wood could not be found. Basically there are two processes of ultrasonic analysis: contact-free methods and such with contact. Both deliver results quickly without the need of complicated sample preparation.

Moisture content can be derived from the velocity of propagation of ultrasound waves but the type of wood has to be known for calibration because the velocity is influenced by type, temperature and moisture content. A contacting method uses an ultrasound sonotrode respectively an ultrasound plate which conducts ultrasonic vibrations to the wood. In the apparatus found on the Internet, the moisture content of wooden boards was determined. A contact-free method uses an Ultran iPass ultrasound chamber, which is often used in testing of building material. [3]

## **2.3.4 Neutron moderation**

This method is used for the determination of moisture content in coal up to 100mm top size and there are no experience with solid biofuels.

The degree to which high-energy neutrons are moderated (reduced to thermal energies by elastic scattering) by solid mineral fuel is a function of its moisture, providing that it also has low and con-

stant organic hydrogen. The intensity of the thermal neutrons flux arising from neutron-irradiated solid mineral fuel contained within a sub-stream configuration is measured. The technique is normally applied to coke up to 100mm top size.

## 2.4 Discussion of instrument types

The main advantages of instruments for direct determination is their accuracy and the fact that they can be moved from one place to another without expensive demounting from one and remounting in another production site. On the other hand, time-consuming direct determination needs much manpower.

There are two types of instruments for indirect determination: small, relatively cheap handheld devices and expensive stationary ones for on-line measurement.

Handheld devices are extremely easy to transport and easy to operate. They quickly deliver results and are a good option for moisture determination, e.g. at the delivery of raw material. However, since indirect methods rely on calibration, which is only valid for constant ambient conditions, handheld devices are relatively inaccurate. They also need manpower to be operated.

Stationary instruments for online measurements are extremely difficult to move: mounting, adjusting and calibration takes much time and a considerable amount of manpower. As soon as they are installed properly and calibrated, they deliver fast and reliable results, which can be processed automatically, so that no personnel is needed to operate these devices. The results can be displayed directly in a process control room and can be used for automated process control. The major disadvantage is their high price, starting at a few thousand euros.

## 2.5 A selection of instruments available

**Table 1: A selection of available instruments for moisture detection**

Principle	Technique	Instrument (example)	
Thermogravimetric analysis	Drying oven	Memmert UF series	
	Duration ca. 6-24h	Nabertherm TR series	
	IR drying	Duration up to 1h Working range 1-100% Sample Mass below 100 g	Sartorius MA series
			Kern ML series
			Mettler-Toledo HG63, MJ33
			A&P Ultra X series
	Microwave drying	Duration a few minutes Working range 8-100% Sample mass below 70 g	Sartorius LMA200
			Anton Paar Multiwave 3000
Electric analysis	Conductivity	Gann Hydromette Series	
		Testo 606	
	Capacity	Online measurement possible Working range 0-100%	Pandis FMG 3000
			Schaller Humimeter pellets analyzer
			Liebherr Litronic FMS II

	Microwave absorbance/reflectance	hf-Sensor MOIST series
	Online measurement possible Working range 0-100%	Sartorius PMD 300
	Time-Domain-Reflectometry	Imko Trime-GW or Trime-TDR
	Online measurement possible Working range 0-100%	
Optical analysis	IR reflectance	Sartorius LMA500
	Online measurement Working range 0-90%	Moist-Tech IR-3000
		Pier Reflexionsphotometer

### 3 Methods for ash content determination

Ash content is another important parameter of biofuels, but more for pellets monitoring than for e.g. wood chips. The following methods are used in coal industry and up to-day there is no use of this methods for solid biofuels known.

#### 3.1 X-ray backscatter

The intensity of low energy (normally less than 30 keV), X-radiation reflected (incoherently scattered) from the solid mineral fuel is a measure of the average atomic number which itself correlates responsibly with the ash. The analysis requires that the solid mineral fuel be of small particle size (less than 5 mm), well conditioned and carefully presented in a sub-stream configuration. The method is applicable to bulk solids and coal slurries. It is perturbed by iron content variations in the ash but may be combined with X-ray fluorescence measurements (see A.3) for iron to improve accuracy.

#### 3.2 Gamma ray backscatter

In principle, this test is similar to X-ray backscatter, but the use of higher-energy radiation (generally up to about 100keV) allows the interrogation of solid mineral fuel of maximum particle size up to 50mm. It may be applied to a sub-stream, but is mostly used in a mainstream configuration, directly on a conveyor belt. The method is applicable to bulk solids; some profiling of the bed of solid mineral fuel may be needed and a minimum bed depth of 100mm is required. It is perturbed by iron variations in the ash but some compensation can be effected by selective multi-channel signal measurements. A variant technique using 662 keV radiation and depending on multiple scattering to render it sensitive to ash variations enables the monitoring of bulk solid mineral fuel up to 300mm particle size in greater bed depths, directly on a belt or within a chute.

#### 3.3 Gamma ray backscatter

Measurement of forward (coherent) scattered gamma radiation, using incident radiation of less than 100keV, provides another technique for the on-belt, mainstream measurement of ash of bulk solids up to 25mm particle size, which is not sensitive to bed depth but is perturbed by iron variations.

#### 3.4 Dual energy gamma ray transmission

The intensity of un scattered gamma radiation (less than 100 keV) transmitted through a layer of solid mineral fuel is depend on its ash mass per unit area. Correction for the latter effect is available

by simultaneously measuring the transmitted intensity of higher energy gamma radiation that is insensitive to ash. This allows direct on-belt measurement of solid mineral fuel up to 200mm top size which is unaffected by bed depth to a minimum of about 50mm. It is less sensitive to ash than scattering techniques but perturbed by iron variations.

### **3.5 Pair production**

Interrogation by high-energy (greater than 1.02MeV) gamma radiation results in the emission of 0,511 MeV gamma radiation (via electron/positron pair production) which correlates strongly with average atomic number and hence ash. Less sensitive to iron variations, it is applied to coal up to 50mm top size, flowing at a rate of a few tons per hour, constrained within a vertical tube in a sub-stream configuration.

### **3.6 Natural gamma radiation emission**

The mineral matter in coal contains a small and reasonably constant proportion of naturally occurring radioisotopes that emit high-energy gamma radiation. The intensity of this emission provides a measure of the mineral matter in coal and hence the ash. The technique is applied directly to coal up to 300 mm top size on a belt. It is relatively unaffected by changes in mineral composition but may require compensation for belt mass loading variation.

### **3.7 Bulk density measurement**

For some coals, there is a correlation between ash content and bulk density. Density may be measured using an on-belt weighing system for a constant volume of coal or by the attenuation of transmitted high-energy gamma radiation. Accuracy is generally low but adequate for some control purposes. It is applicable to coal up to 300 mm top size.

### **3.8 Discussion of ash measurements methods**

Ash as parameter is limited important for the solid biofuel industry. The main problem in conjunction with ash content is slagging. Even though ash components contribute to slag formation, the content of ash itself does not indicate the tendency of slagging and fouling. Usually the ash content of solid biofuels is smaller compared to that of mineral fuels and is certainly a threshold of using these technologies. The only relevance where the methods could be used is the pellets production.

## **4 Methods for the determination of chemical components**

The following methods allow to measure few elements simultaneous

### **4.1 Prompt gamma neutron activation analysis (PGNAA)**

The parameters measured include carbon, hydrogen, nitrogen, sulphur, chlorine, oxygen, aluminium, silicon, iron, calcium, potassium, and titanium and derived values such as ash, heating content, sulphur dioxide emission rates and slagging and fouling factors. Irradiation of bulk coal by neutrons gives rise to the prompt emission of gamma rays with energies (1 MeV to 10 MeV) that are characteristic of most of the elements in the coal. Most instruments depend on thermal neutron capture interactions, but these are of low efficiency for carbon and negligible for oxygen. The two latter elements can be better determined from gamma rays arising from higher energy neutron inelastic scatter interactions. Coal up to 100 mm top size and at rates up to 400 t per hour is presented within the confines of a chute. The technique may also be adapted to determine sulphur content of coal in slurries. A similar use for biomass is theoretically possible, but PGNAA is extremely expensive (several 100.000€).

### **4.2 X-ray fluorescence analysis**

The parameters measured are potassium, iron, sulphur, calcium and silicon. The solid mineral fuel is interrogated by suitable low energy x radiation that excites X-rays characteristic of specific elements (X-ray fluorescence). The intensity of these emissions is measured and related to the element concentration. Particle size, sample homogeneity and sample presentation conditions are critical, especially for the lower atomic number elements and sub-stream configuration is necessary. For the stationary off-line measurements also the analysis of additionally major and minor elements is possible.

### **4.3 Discussion of chemical components measurements methods**

Methods measuring certain chemical components are interesting but very expensive to install. In case of biomass-fuels it is course a matter of production volume, too. Production sites and plants are usually small or medium-size units with simply non- sophisticated technology (like X-ray or neuron activation)which usually sets limits for instrumentation as well.

High costs is certainly a limitation factor for biomass. An installed cross-belt PGNAA (ash) analyzer may cost around \$300.000 inclusive of a moisture meter. A sample stream PGNAA analyzer, will cost from \$450.000 to \$750.000 installed. [5]

It seems obvious that PGNAA technologies are far too expensive for bio-fuel applications.

## 5 Image analysis for determination of the particle size distribution

The image analysis is a optical analysis for determination of particle size distribution, width-length ratio, maximum length and sphericity (shape factors). The data are monitored electronically (PC). The problem of this method is the high purchase price and the rarely available test equipment.



**Illustration 4: Image analysis [8]**

## 6 Methods for future bio-fuel market

### 6.1 Flue-gas measurements and modelling

As bio-fuel properties become better defined through real-life experiences and other sources of information like data bases, modelling may become an attractive tool for mass and energy balances.

The on-line calorific value sensor [6] is based on the assumptions that the waste comprises combustibles, water and inert materials and that the combustible part can be described by  $\text{CH}_y\text{O}_z$ . Extensive analysis has shown that the variation in  $y$  is negligible in the case of municipal solid waste combustion. Therefore this value can be taken as a constant. With this assumption and the assumption that the amount of converted carbon is known [mostly between 0.95 and 1, while the error in this assumption has only a slight (>1%) effect on the error in the result], the mass balances for  $\text{O}_2$ ,  $\text{CO}_2$  and  $\text{H}_2\text{O}$  can be solved. This method can not only be applied to MSWC but also to other combustion processes, for example fluidized bed combustion and wood fired grate systems. Together with the on-line measurement of the gas components  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{O}_2$  and the  $\text{H}_2\text{O}$  in the surrounding air, the model calculates the following quantities:

- The net calorific value of the combustible part of the waste – this is the net calorific value of the waste that burns at the grate at that moment, i.e. an average calorific value of the combustible fuel on the grate;
- The calorific value of the combustible part of the waste, i.e. without the inert fraction;
- The average moisture fraction of the waste, which burns at the moment.

If the inert fraction is known, the net calorific value of the ‘total’ (combustible, water and inert) waste can be calculated.

### 6.2 Sulphur detection in liquid bio-fuels

Liquid bio-fuels may become a treasure for on-line instruments developers as sampling and sample preparation conditions are not demanding and well-defined.

Sulphur is not normally considered critical for solid bio-fuels. However if these materials are used for e.g. biodiesel production and treated with chemicals, sulphur may be a problem. May on-line methods are available for liquid bio-fuel streams using e.g. UVF (ultraviolet fluorescence technology). [7]

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