PUBLISHED DOCUMENT

PD CEN/TR 15591:2007

Solid recovered fuels — Determination of the biomass content based on the ^{14}C method

ICS 75.160.10

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

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This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 April 2007

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ISBN 978 0 580 50522 5

Amendments issued since publication

Amd. No.	Date	Comments

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TECHNICAL REPORT
RAPPORT TECHNIQUE
TECHNISCHER BERICHT

CEN/TR 15591

February 2007

ICS 75.160.10

English Version

Solid recovered fuels - Determination of the biomass content based on the ^{14}C method

Combustibles solides de récupération - Détermination de la teneur en biomasse, basée sur la méthode du C^{14}

Feste Sekundärbrennstoffe - Bestimmung des Gehaltes an Biomasse nach de ^{14}C-Methode

This Technical Report was approved by CEN on 1 January 2007. It has been drawn up by the Technical Committee CEN/TC 343.

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Foreword

This document (CEN/TR 15591:2007) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

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0 Introduction

0.1 General

This document has been prepared as a result of the CEN/TC 343/WG 3 meeting in Amsterdam in April 2005. It summarizes the state of the art in ^{14}C-based methods applied to determining the biomass content of SRF; as of yet no technical CEN standards for the application of ^{14}C-based methods to determine biomass content are available. The purpose of this Technical Report is to present the information available on this subject at this moment to assess if an extension of the available methods for determining the biomass content of SRF is required, wanted and technically possible.

Analytically proven standards exist for determining the biomass content of SRF by manual sorting and by selective dissolution (CEN/TS 15440 [1]). In the Netherlands these methods are available as NTA (National Technical Agreement) and have been in use for some years. Important advantages of these standards are their applicability using basic laboratory equipment and available personnel. However, they are not applicable to all kinds of solid recovered fuels. The manual sorting method fails if the constituents of the sample are shredded too finely, if they are strongly intertwined or compressed or if they cannot be recognized visually. The selective dissolution method fails if biomass constituents are present that do not dissolve, or fossil components that do. Both methods fall short if fossil and biomass carbon are mixed at the molecular level. ^{14}C based methods do not use chemical or morphological properties of the sample but physical properties of the carbon atoms themselves. Because ^{14}C based methods are based on these physical properties they avoid the problems of manual sorting and selective dissolution methods. On the other hand they need more instrumentation and skilled personnel. They are proposed here as an addition to the manual sorting and selective dissolution methods because they resolve analytical problems that are otherwise irresolvable.

The application of ^{14}C based methods for similar purposes are not new [2] [3]. In this document the information available in Europe and the USA concerning biomass carbon content determination in solid recovered fuels with ^{14}C based methods is presented to give the reader background information about possibilities and drawbacks of these methods.

0.2 Basis of the ^{14}C method

The ^{14}C method is a well-known method in global use, for determining the age of carbon containing matter. ^{14}C is a radioactive isotope; its presence in the air is a result of the interaction of cosmic radiation and the nitrogen in the atmosphere (see Figure 1). Fossil carbon contains no ^{14}C, however a trace amount of ^{14}C is present in living matter. The ^{14}C isotope is quickly converted to $^{14}CO_2$ after formation and enters living matter when atmospheric $^{14}CO_2$ is converted in the biosphere by photosynthesis to sugars and further converted to e.g. cellulose. The concentration of ^{14}C in air is considered constant all over the world. In living material the concentration of ^{14}C is stable and in equilibrium with the air concentration. In dead material the concentration of ^{14}C slowly diminishes to zero as the radioactive ^{14}C isotope decays. Measuring the amount of ^{14}C in solid recovered fuels is the basis for determining biomass content based on the ^{14}C method.

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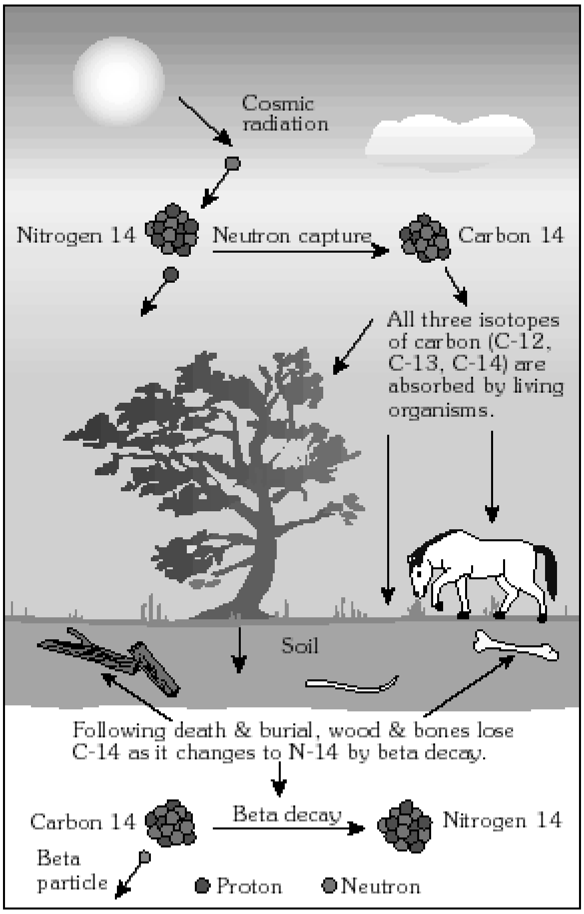

Figure 1 – Illustration of the basis of the ^{14}C method

Organic material is used for many purposes. One of the objectives is direct use as a fuel which is outside the scope of this report. However, after completing their primary use, many of these organic materials may ultimately be used in the form of solid recovered fuels.

Examples of organic materials in solid recovered fuels are:

- Packaging materials;
- Paper;
- Wood used in buildings;
- Kitchen waste;
- Waste (dung and offal) from the bio industry;
- Plastics;
- Car tires.

Carbon present in material produced by living organisms, immobilized as fuel in present times is called biomass. Carbon present in material produced by living organisms immobilized as fuel in a past geological era is called fossil fuel. The difference between the two is that CO_2 from biomass or biomass origin does not

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contribute to a higher concentration of CO_2 in the atmosphere as its carbon has been recently extracted from the atmosphere.

In solid recovered fuels, the combustible carbon originates from fossil (mainly in the form of plastics), mixed sources like rubber tyres and packaging materials, and from biomass origin (e.g. wood, paper). Authorities require that emissions of CO_2 from fossil origin by companies is made known, thus, in order to determine these companies, knowledge about the biomass content by total carbon content of mixed fuels should be acquired. For this reason, methods such as the solid dissolution method and ^{14}C method were developed.

International acceptance of a ^{14}C based method can be expected, as can be illustrated by the recent publication of ASTM, ASTM D 6866-05, Standard Test Method for determining the Bio based Content of Natural Range Materials Using Radiocarbon and Isotope Ratio Mass Spectrometry Analysis [2].

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1 Scope

This Technical Report gives an overview of the suitability of ^{14}C-based methods for the determination of the fraction of biomass carbon in solid recovered fuels, using detection by scintillation, gas ionization and mass spectrometry.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
biodegradable carbon
mass fraction of the total carbon that is capable of undergoing biological anaerobic or aerobic decomposition under conditions naturally occurring in the biosphere

2.2
biogenic carbon
mass fraction of total carbon that was produced in natural processes by living organisms but not fossilized or derived from fossil resources

2.3
biomass carbon
equivalent to biogenic carbon

2.4
isotope abundance
fraction of atoms of a particular isotope of an element

2.5
repeatability
extent of the agreement between the results of subsequent measurements of the same quantity, performed under the same measuring conditions

2.6
reproducibility
extent of the agreement between the results of measurements of the same quantity, performed under variable measuring conditions.

3 Symbols and abbreviations

This Technical Report uses the following symbols and abbreviations:

^{14}C	Carbon isotope with an atomic mass of 14
AMS	Accelerator Mass Spectrometry
β	Beta particle, electron emitted during radioactive decay
BI	Beta Ionisation

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BP	Before Present (before 1950)
CPM	Counts per minute
DPM	Disintegrations per minute
ETS	Emissions Trading Scheme
GM	Geiger Müller
LSC	Liquid Scintillation Counter or Liquid Scintillation Counting
PSM	Proportional Scintillation-counter Method
PMT	Photo Multiplicator Tube
RSD	Relative Standard Deviation
SDM	Selective Dissolution Method
SRF	Solid Recovered Fuel
STP	Standard Temperature and Pressure (273,15 K (or 0 °C) and 101,325 Pa (or 760 mmHg))

4 Methods of measurement

4.1 Principle

The principle of the ^{14}C method is to determine the biomass content by total carbon by measuring the amount of ^{14}C present in the sample. This method utilizes the isotope abundance of ^{14}C similar to the way the age of objects is measured for archaeological purposes. In all organisms living ashore, ^{14}C has a known isotope abundance equal to its isotope abundance in atmospheric CO_2. As soon as an organism dies, the isotope abundance of ^{14}C in its organic material starts to decrease because ^{14}C is an unstable isotope with a half-life of 5 730 yr. The isotope abundance of ^{14}C may be considered zero after ten half lives or 60 000 yr. The biomass content by total carbon of a material is calculated as the proportion of the isotope abundance of ^{14}C in that material and the isotope abundance of ^{14}C in the atmosphere at the time when the biomass was laid down.

The method is especially useful for determining biomass carbon content, however, the relationship between biomass carbon content and biomass content should be determined for every type of waste; a limitation that is also valid for other existing methods. When information is available about how carbon atoms are chemically bound, the amount of bio energy can be calculated.

4.2 Sampling

For the ^{14}C based methods sampling procedures that are similar to those for determining major elements [4] are used. As carbon is one of the major components in solid recovered fuel, problems with homogeneity are not to be expected with laboratory samples. Typical particle size of the sample material should be $\leq$ 0,2 mm.

4.3 Transport and storage

For transport and storage of the samples, the same requirements are fulfilled as for normal lab samples. As part of the solid recovered fuel consists of organic material, dry and cool storage is applied to prevent conversion of the biomass part by microbiological activities.

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4.4 Preparation of the test portion from the laboratory sample

For the PSM and BI methods sample sizes of 1 g or more are used. However at the 1 g level problems still arise with homogeneity of the sample; the use of a lab scale combustion device (e.g. rotary kiln) is recommended, allowing sample amounts of 5 g to 20 g.

The AMS method only needs a few milligrams of sample. In this case combustion of samples at a scale of approximately 1g is necessary. After combustion the carbon is present in a gas phase as CO_2, and the next step is preparing a mg size sample from the gaseous combustion products.

4.5 Analysis by Proportional Scintillation-counter Method (PSM)

PSM (also called Liquid Scintillation Counter method, LSC) determines the isotope abundance of ^{14}C indirectly through its emission of β (beta, electron) particles. The β particles are detected through interacting with a solution of a scintillation molecule. This is possible only if the carbon is homogeneously distributed in the solution, as the β particles must be able to interact with the solution instead of being quenched in the solid fuel. Homogeneous distribution may be attained by four different methods:

- Conversion to CO_2, followed by absorption in an organic amine and mixing this absorbent with the scintillation fluid. The amine is produced using fossil carbon, in order not to cause a blank signal.
- Conversion to CO_2, followed by absorption in a $BaCl_2$ or $CaCl_2$ solution, and
    - after drying and grinding, transfer of $BaCO_3$ or $CaCO_3$ into the scintillation fluid forming a suspension; or
    - regeneration of CO_2 from the precipitate, which is absorbed in an organic amine, and mixing this absorbent with the scintillation fluid.
- Conversion to CO_2, followed by adsorption on a solid medium, regeneration of CO_2 which is absorbed in an organic amine, and mixing this absorbent with the scintillation fluid.
- Liquid fuels may be directly mixed with the scintillation fluid.

The scintillation fluid consists of a solvent and a dissolved fluorescent agent, the fluor. When a β is emitted, it rapidly transfers its energy to solvent molecules (< 5 ns) in the form of heat, ionisation and excitation. A part of the excited solvent molecules transfers energy to fluor molecules; the remaining energy is lost as heat by various quenching processes. A part of the excited fluor molecules release their energy in the form of photons in the blue part of the visible spectrum; again, the remaining energy is lost as heat by various quenching processes. A part of the photons are detected in the form of a light flash; the intensity is proportional to the β's initial energy. The remaining photons are lost by quenching, by reabsorption by the fluor, or by not being detected because of geometry. The standard addition technique can be used to determine quenching effects.

In practice, a pair of light detectors – classically, PMT's – are used for two reasons:

- It is necessary to compensate for geometry effects: an event close to a detector will produce a stronger signal because more photons will reach the detector. This is done by adding the signals of the detectors;
- Events caused by background radiation should be excluded, by admitting only those pulses that are seen by both detectors simultaneously (window size approximately 20 ns). A predefined intensity ratio threshold is set in order to exclude most events that occur outside the sample vial.

The remaining background counts occur mainly by decay of other naturally radioactive isotopes such as ^{40}K, ^{212}Bi, ^{212}Pb, ^{214}Bi, and ^{214}Pb and / or ^{137}Cs.

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The overall *a priori* efficiency of the detection is unknown. Therefore, the method has to be calibrated with samples of known ^{14}C isotope abundance.

4.6 Analysis by B-ionisation (proportional gas counting) (BI)

The B Ionisation method determines the isotope abundance of ^{14}C indirectly. This method employs the emission of β particles by ^{14}C, like PSM. It detects β particles by means of discharge current pulses between high-voltage electrodes in a proportional gas counter. Those pulses are initiated by the β particles. The detection principle resembles the way a Geiger-Mueller (GM) counter works, the difference being details of the electron avalanche in the counter.

To use this method, the sample has to be in the form of CO_2 or converted to CO_2, as is also the case for most applications of PSM.

The sensitivity of the gas counter is proportional to the contained gas quantity, and therefore to the specific mass/pressure of the gas and to the volume of the detector. In practice, 1,4 g carbon may be loaded (up to 10 l CO_2 at STP (Standard Temperature and Pressure)). Assuming a counting efficiency of 80 %, the performance based on counting statistics may be in the same order of magnitude as the performance of the liquid scintillation method. However, it is not necessary to absorb the CO_2 in a liquid, the method may be more manageable than PSM and use less consumables.

4.7 Analysis by Accelerator Mass Spectrometry (AMS)

The accelerator mass spectrometry method determines the presence of ^{14}C directly. The atoms in the sample are converted into a beam of ions. Accelerating them in an electric field, deflecting them in a magnetic field and detecting them in an ion detector determine the relative isotope abundances of these ions.

AMS is a form of mass spectrometry that uses a high potential electrostatic field, which serves not only to accelerate them but also to specifically form only C^{4+} ions that are allowed into the spectrometer, excluding all other ionic species. This greatly enhances sensitivity without compromising selectivity.

AMS uses only a few mg of sample, and the sample processing and counting time is considerably less time consuming compared with β counting. Logistic turnaround times of two weeks or less are possible. Due to the complex accelerator system cost is presently still higher but this issue may change in the near future due to the rather mature state of small accelerators. AMS may be even more attractive if reliable gas-ion-sources (now under development) may become routinely available, because the CO_2 can be fed directly into the accelerator.

5 Equipment and reagents

5.1 For the preparation of the test portion

5.1.1 Oxygen bomb

Commercially available oxygen bombs for determining the caloric values are used for the combusting the test portion.

In some cases intermediate storage of the combustion gases in a tedlar gas bag is used. The combustion gases from the oxygen bomb can then be released in a short time, and afterwards the combustion gases can be processed at the desired flow rate using a small gas pump. For longer storage of combustion gases gasbags with an aluminium layer are advised, as losses are observed in other types of materials.

If the carbon content in the sample is calculated from the amount of CO_2 present in the combustion gases, impingers filled with concentrated phosphoric acid and zinc pellets are used to remove water, and sulphur and halogen oxidation products, prior to the absorption of the CO_2.

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5.1.2 Rotary kiln oven

For very inhomogeneous samples a lab scale rotary kiln oven is used to combust several grams of sample. The sample is burned in the rotary kiln in an oxygen stream at ± 800 °C and the formed combustion gases are cleaned with impingers and the formed CO_2 is absorbed into the organic amine solution. The CO_2 concentration in the combustion gases is measured simultaneously with the CO and oxygen concentration to ensure the complete combustion of the sample.

5.2 For the analysis by PSM

- 1-methoxy-propane-3-amine absorption liquid;
- Sodium hydroxide solution (1 M);
- Scintillation cocktail, miscible with the used amine;
- Scintillation vials, glass, 20 ml;
- Scintillation counter.

The instruments [5] are able to use scintillation vials with a volume of at least 10 ml. Shielding from background radiation is applied as much as possible. The background level required is 12 DPM or less. To achieve this, the instruments are placed inside a heavy-walled room and shielded with a so-called lead castle, made from lead with a minimum of radioactive isotopes. Therefore lead preferably produced before 1600 AD is used. Well-known sources of low background lead are lead ballasts from sunken sail ships.

5.3 For the analysis by B-ionisation (BI)

The instruments used for BI measurements are home made high tech devices developed at divers radiocarbon institutes. No commercial systems are available. Gas (in this case purified CO_2 derived from combustion gases) is loaded and counted in a copper counting tube (ultra pure copper) and the desired low background is obtained by applying heavy shielding with lead and anti-coincidence filtering of cosmic radiation. Usually BI devices are located below ground level, e.g. in cellars to obtain extra protection against cosmic radiation. Typical counting times are several days for low-level measurements.

5.4 For analysis by AMS (example from Utrecht University)

The AMS-facility in the Van de Graaff laboratory of Utrecht University is based on the 6 MV tandem Van de Graaff accelerator, which has been used originally for precision research of nuclear spectroscopy. The accelerator has been adapted for AMS with stable beam transmission and minimal beam loss. Precision measurements are made with fast switching between isotope beams in an automated scheme. Ions are selected on mass with a bending magnet and injected into a particle accelerator, where they become accelerated. Ions emerging the accelerator are selected on energy and mass and finally detected. The stable isotope is collected in a Faraday cup and its yield is determined from the accumulated charge. The rare isotope is identified and counted in a detector.

6 Procedure

6.1 For sampling

The procedures for sampling are similar to the procedures that are used for proximate-ultimate analysis of solid recovered fuels.

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6.2 For the preparation of the test portion

6.2.1 General

The procedures for the preparation of the test portion are similar to the procedures for the determination of the caloric value in the case of oxygen bomb combustion. When the lab scale rotary kiln oven is used, samples prepared for lab analysis are used. In some cases CO_2, directly collected in the flue gas of full-scale installations, is used to determine the biomass carbon content in fuels.

6.2.2 For PSM

After combustion of the sample, the formed CO_2 is absorbed in a sodium hydroxide solution, barium chloride solution or an organic amine, such as 1-methoxy-propane-3-amine, where CO_2 is absorbed by formation of a carbamate. Because this reaction is exothermic, the absorption flask is cooled. When the total carbon content is determined by the gravimetric determination of the precipitated barium carbonate or the determination of the density of the organic amine, scrubbers for the removal of water (concentrated phosphoric acid) or halogens (zinc pellets) are used. For the collection of carbon dioxide from the exhaust gas of the oxygen bomb in a number of cases a gasbag is used for intermediate storage.

When an absorption flask is loaded with a known volume of CO_2 absorbent, e.g. with 1-methoxy-propane-3-amine, the absorbing capacity of 1-methoxy-propane-3-amine of about 4,8 mmol/ml is to be taken into account; no more than 80 % of this capacity is used. The flask is cooled in ice during the absorption process. After absorption, the absorption fluid is mixed with a scintillation cocktail. Those cocktails are commercially available; their exact composition is not specified. In some cases the absorbent and scintillation cocktail is mixed in a 1:1 ratio before absorption, so that problems with evaporation, and crystallization of carbamates can be avoided.

Although not within the scope of this document, it is interesting to mention that PSM is also applied to liquid fuels, which consisted of mixtures of conventional fossil fuels and biomass fuels such as sunflower oil and palm kernel oil. Liquid fuels are just sampled and directly mixed with the scintillation cocktail. However, attention is paid to possible differences between the quenching properties and colour of the liquid analytes, standard addition followed by re-counting is used in order to determine the counting efficiency.

6.2.3 For BI

6.2.3.1 Gaseous samples

For gaseous samples, the CO_2 gas is absorbed as carbonate in NaOH. The carbonate is converted to CO_2 (100 mmol to 400 mmol) by adding HCl.

6.2.3.2 Solid samples

The sample (ca. 10 g) is combusted in a combustion bomb at an oxygen pressure of 10 bar. The CO_2 is absorbed in $CaCl_2$ for purification. CO_2 is then obtained by acidification with HCl

6.2.3.3 Gas purification

The CO_2 gas is purified using activated charcoal.

6.2.4 For AMS

The general procedure for AMS is:

- Combustion of the sample, formation of CO_2 that is frozen out.
- Convert the CO_2 to graphite by leading it over a hot Fe catalyst with H_2.
- Compress the graphite to a target of about 1 mg.

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6.3 Procedure for analysis

6.3.1 Procedure for analysis by PSM

After absorption of the CO_2, the absorbent is transferred to the measuring vial. An equal volume of scintillation cocktail is added and the mixture is homogenized. The vial is transferred to the scintillation counter and the counts are summed over the counting time interval.

Measurements are performed using a lw-level counter. For measuring, the predefined ^{14}C protocol of the instrument is used. Counting time is typically 1 500 min for each sample. The quench parameter is determined by an inbuilt external standard. The counting efficiency is determined by standard addition (e.g. 100 000 dpm ^{14}C, standard) and recounting for 10 min.

6.3.2 Procedure for analysis by BI

The CO_2 gas sample (2,5 l to 10 l STP) is transferred into a gas proportional counter. ^{14}C decays are counted for several days to obtain a statistical error of < = 0,3 % [6]. The ^{14}C activity (corrected for background and isotope fractionation) is calculated according to Stuiver & Pollach [7].

6.3.3 Procedure for analysis by AMS

Anaylsis has started once the graphite pellet is loaded in the sample holder. The sample holder can usually hold a number of samples enabling unattended measurements of samples for a long period of time.

Sequence for measuring:

- Hitting the target with Cs ions that release C^- ions, along with a small amount of CH^- ions.
- Breaking down molecular ions and converting all species and isotopes of carbon to C^{3+}, by colliding them with inert gas molecules in a electrostatic field of 0,5 MV to 6 MV. This avoids interference of the measurement of $^{14}C^-$ by isobaric compounds, such as $^{13}CH^-$ and $^{12}CH_2^-$, as would have been the case if $^{14}C^-$ had been detected directly. Also, interference by $^{14}N^-$ is avoided because nitrogen does not form positive ions.
- Fast alternating detection of $^{12}C^{4+}$ and / or $^{13}C^{4+}$, and $^{14}C^{4+}$.
- Computation of relative isotope abundances, almost always followed by a computation for radiocarbon dating of the sample.

7 Calculations

7.1 General

The atmospheric $^{14}CO_2$ level is accurately known for the relevant past and is uniform within the hemisphere. Contamination from local fossil or nuclear facilities is considered very minor. The uncertainty of determining the fossil fraction in organic waste is dominated by the growth interval of the modern component. It is largest for low fossil fraction. For a fossil fraction of around 70 %, scenarios bracketing potential sources, lead to an uncertainty of ± 5 %. For a fossil fraction of around 30 %, the uncertainty is strongly dependent on the composition of the organic waste fraction. Assuming domestic waste with biomass fractions of paper and packaging materials as the source for solid recovered fuels, an uncertainty of 5 % can also be expected. For solid recovered fuels containing biomass sources that were formed before or during the American bomb tests, the uncertainty will be higher.

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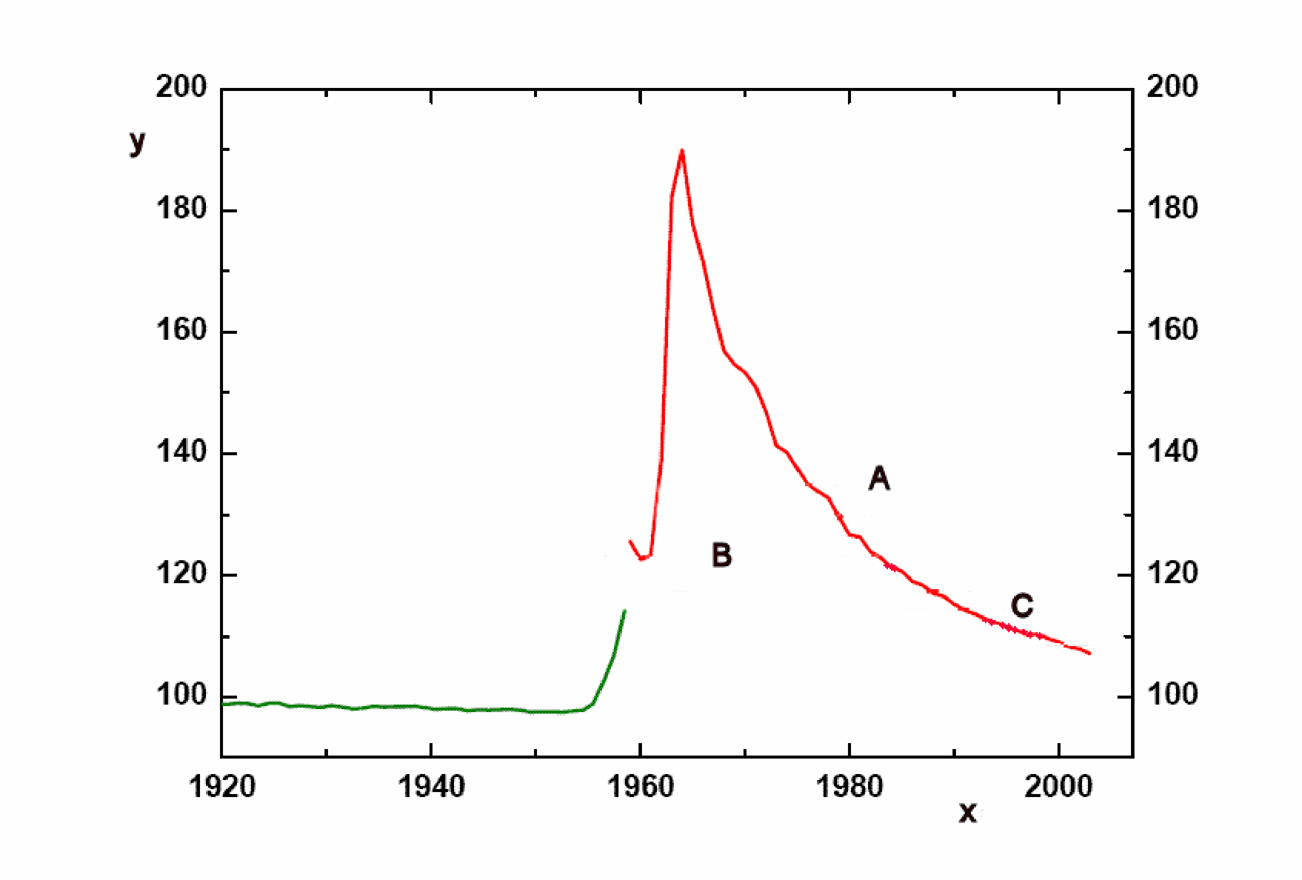

Key

🟥	**Atm. CO_2 (Levin & Kromer, 2004)**	X	Year AD	A	40 years, 131 pmC
🟩	**Tree-ring ^{14}C (Stuiver & Quay, 1981)**	Y	Percent modern Carbon (pmC)	B	70 years, 118pmC
				C	20 years, 114 pmC

Figure 2 — Illustration of accumulation of bomb ^{14}C in trees

For ^{14}C dating the reference year is 1950 (see Figure 2). This year is selected for historical reasons; nowadays we would have chosen 2000 to facilitate conversions. Even in 1950 the atmospheric ^{14}C level was affected by human activities, i.e. the Suess effect (a side effect of the industrial revolution was the increased use of fossil fuels lowering ^{14}C by a few percent), and by the early American bomb tests (raising the level by ca. 8 %). If one assumes constant ^{14}C production, the undisturbed level for the year 1950 can be calculated by measuring a tree-ring section of known dendro-age without these human interferences, about 1850, and extrapolate from this data the level of 1950, using the radioactive decay law. This is exactly how it was done in the 1960's by several labs to establish the ^{14}C standard level at 1950. Constant ^{14}C production is not exactly acurate, but is not an issue for dating purposes, as all ^{14}C ages are now calibrated (Libby ages) using the tree-ring calibration, where the same reference point of AD 1950 is used. Thus, the wrong half live (5 568 rather than the more realistic 5 730) and any shift of the 1950 reference level cancels during calibration.

7.2 Calibration

For the purpose of calibration, the measured number of disintegrations per min is converted to a "percent modern Carbon" value (pmC) using a default value for the specific activity of modern carbon. This value equals 13,65 $min^{-1}g^{-1}$. It is based on the pre-1900 value that was valid before human activity perturbed the atmospheric abundance of ^{14}C. This occurred by dilution with fossil CO_2 (the Suess effect [8]) and also in the early 1960s by enrichment of $^{14}CO_2$ by the explosions of nuclear fusion bombs.

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$$pmC_{sample} = \frac{\left(\frac{DPM_{sample}}{C \times \varepsilon} \times \frac{1}{M_{C,sample}} \right) \times 100\%}{pmC_{reference}} \tag{1}$$

where

pmC_{sample}	is the percentage of carbon in the sample that is of biomass origin, relative to the total mass of carbon in the sample;
DPM_{sample}	is the number of disintegrations per minute of the sample (min^{-1});
ε	is the total counting efficiency of the detection, including all quenching and geometry effects. This is determined with a reference sample of known ^{14}C abundance;
$M_{\text{C, sample}}$	is the mass of carbon in the sample (g);
C	is 13,65 $\text{min}^{-1}\text{g}^{-1;}$
$pmC_{\text{reference}}$	is 107 (at BP) or 114/118 (to be decided).

For the $pmC_{\text{reference}}$ value different options are possible:

a) 'Minimum' option: Adopt the lowest reasonable modern value, to avoid 'unjustified' payment. Suggestion: 20/70-year growth, ending in 2003: 114/118 pmC.

b) 'More exact' option: derive statistics about the activity ranges of the specific sample type, fraction of the total, and of the modern component. This option requires additional measurements.

c) In ASTM D-6866 [2] a correction of 0,93 × the specific activity of the sample is taken, giving a $pmC_{\text{reference}}$ value of 115. No further explanation for this value is given in this document.

7.3 Example for the calculation of a RDF sample analysed with PSM

Suppose 0,8 g of RDF sample was combusted in an oxygen bomb, the CO_2 collected from the combustion gases was counted in a liquid scintillation counter and a net value of 2 dpm was counted.

Using 16 dpm as the standard value for 1 g biogenic carbon the calculation would be:

The amount of biogenic carbon in the sample is 2/16 × 0,8 = 0,1 g or 12,5 %

Assuming wood (with 50 % C) as the biomass fraction there would be 0,2 g wood in the sample, equivalent to a 0,2/0,8 = 25 % biomass content.

8 Uncertainty of measurement (PMS and BI measurements) based in Poisson statistics

The instrumental precision for measuring radioactive decay mainly depends on the statistics of the disintegration process and on the overall counting efficiency, which in turn depends on quenching and geometry. Using Poisson statistics for the numbers of counts yields a tentative estimate for the precision of determining the percentage of carbon in a sample that is of biomass origin, relative to the total mass of carbon in the sample.

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Table 1 — Theoretical instrumental precision as a function of biomass content and background level. Sampled carbon mass in vial = 1 g

Background (dpm)	16 (standard equipment)	2 (low level equipment)
Counting time (h)	3	72
Biomass content (%)	RSD (%)	RSD (%)
100	3	0,4
50	4	0,6
25	8	0,9
10	20	2
5	40	3
2	90	7
1	180	13

In this table only the instrumental uncertainty is shown. The uncertainty of the whole method has other sources as well, the contribution of the other sources are strongly dependable of the method that is used to produce the material for the ^{14}C determination.

It can be concluded that there are no technical barriers to determine the biomass content in the ranges that are required. With counting times of 72 h even low biomass contents can be measured with acceptable precision.

9 Strengths and weaknesses

9.1 Comparison of ^{14}C based methods with SDM

SDM is a widely used and accepted method to determine the biodegradable matter content of compost. It was originally developed for that purpose [9] but was adapted when a need arose to determine the biomass content of SRF. (Pre-normative research on SRF [10] for ERFO (European Recovered Fuel Organization) and SenterNovem (Netherlands Organization for Energy and the Environment), 'Solid Recovered Fuels – Method for the determination of biomass content' (CEN/TS 15440)). By doing so, an implicit assumption was made: 'biomass' is equivalent to 'biodegradable'. In many cases, the assumption holds true, however, uncertainties arise if biodegradable material is present that is not biomass such as nylon or PUR foam. In such cases, the estimated biomass content is too high. Conversely, if biomass material is present that is not fully biodegradable such as wool, frying fat, or charcoal the estimated biomass content is too low. On the other hand, SDM can estimate the biomass content by calorific value whereas ^{14}C based methods cannot. This is because SDM physically produces a sample from which the biomass (actually: biodegradable) fraction was removed. In this sample the calorific value can be determined. ^{14}C methods do not physically separate the non-biomass part from the sample, so they cannot determine its calorific value. By similar causes, they cannot determine the biomass content by weight.

A special case is determining the biomass content in peat.

With the SDM method peat behaves as a biomass. With the ^{14}C method, however, the measured biomass content depends on the age of the peat. Peat with an age of 6,000 years will give a value of 50 % (the half-life of ^{14}C is about 6 000 years) peat with an age of 50 yr to 100 yr will give a value of 100 % and peat with an age of 100 000 years will give a value of less then 3 %.

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9.2 Comparison of PSM, Gas Counting (BI) and AMS

Table 2 — Comparison of PSM, Gas Counting (BI) and AMS

	PSM	BI	AMS
Detection limit (% biomass)	1	0,2…	0,2
Detection limit (% fossil)	5	0,2…	0,4
Turn around time	6 h … 72 h	14 d	several days
Investment (2005)	k€ 100 … k€ 150	k€ 30 … k€ 80	M€ 1 … M€ 1,5
Laboratory requirements	Shielded	Extremely heavy shielding	General lab environment
Required operator skill	Skilled lab technician	Expert/specialist	Expert/specialist

The different techniques are all suitable for this specific application and the presently available laboratories in Europe (see Annex B) will enable rapid introduction of the biomass content determination based on the ^{14}C content.

10 Legislative aspects

10.1 General

At this moment a number of national legislations are effective, however, in the near future European directives will be introduced and national legislations will be replaced by European legislation.

10.2 Austria

On 16 February 2005 the Kyoto-Protocol was implemented. A greenhouse gas reduction of 13 % (2008 to 2012) is the Austrian target. It is not yet decided if the use of fuel derived from (domestic) waste will also be included in the allocation planning. One of the obstacles is the exact determination of the biomass carbon content. From 2004 landfills have been prohibited, resulting in a methane emission reduction of 24 %. By 2010 a reduction of 40 % is foreseen. Ökostromgesetz: In the EU Commission Decision ((2004/156/EC, page 24) [11] an extensive list of CO_2-neutral biomass materials is given that also will be applicable in Austria. At this moment political decisions regarding this directive are in preparation. Regulations for industrial waste and sewage sludge are in preparation. For determining the biomass fraction CEN methods should preferably be used. If not yet available ISO and Best Practice Guidelines should be used. Hand picking methods, selective dissolution methods and determination based on the ^{14}C content are mentioned.

10.3 The Netherlands

At the beginning of 2005 an important step was made in implementing the Kyoto protocol. Carbon dioxide credits were distributed to Electricity Companies and major industrial consumers of energy and energy producers were encouraged to use renewable sources, including bio fuels, by the governmental renewable energy policy support (MEP regeling). Determining the biomass fraction is described in the national technical specification NTA 8200[12]. Methods that can be used are the hand picking method, reductional calculation method and the selective dissolution method.

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10.4 Finland

Carbon dioxide emission allowances were allocated for all installations by the ministry of trade and industry. Site-specific allowances were then distributed for the accounts of all operators in one emission registry. Allocation is based mainly on calculated emission estimates for the first ETS (Emission Trading Scheme) period and utilising default factors used in the national greenhouse gas emission inventory. The Energy Market Authority (EMA) is the national emission trading authority in Finland. EMA is responsible for decisions related to the site level emission permits and associated monitoring methods on the detailed level. In most cases operators are allowed to use default emission factors and net calorific values for certain solid recovered fuels in their monitoring and reporting systems. These factors and associated fuel classification system are the same as those used in the national allocation plan and in the national greenhouse gas inventory. Defaults used in the greenhouse gas inventory are rather average in nature rather than overestimates regarding real emission figures. Thus there is no strong incentives to introduce more specific monitoring methods in all cases. However, in certain installations, use of activity-specific data and factors is permitted.

In Finland, an alternative monitoring methodology can be used if the operator can show the competent authority (EMA) that the specified monitoring procedure has the same permissible uncertainty as the required method in the ETS-Monitoring and Reporting Guideline (EU-MRG) or that the specified procedure is more accurate. Thus there is no known requirement to use ^{14}C-method, if the operator can demonstrate the overall uncertainty level and MRG-compatibility. E.g.: mass balance and energy approaches for determining activity data are approved for certain installations to estimate net calorific value and energy content (cumulative in nature) of heterogeneous fuels.

11 Conclusions

At this moment, a lot of experience on determining the ^{14}C content in organic materials is available in Europe. However, the applications where the ^{14}C content is used to determine the biomass content in fuels such as solid recovered fuels are rather new. Only a few laboratories have experience with this specific application. The techniques used to obtain samples for ^{14}C determination are almost standard laboratory procedures, therefore, developing a technical specification and implementing this method seems to be relatively easy to fulfil.

As a starting point, the existing ASTM documents D 6866-05 [2] and D 7026-04 [3] could be used. Those standards apply a default value for the specific activity of modern carbon, assuming a uniform contribution by nuclear explosions, irrespective of the age of its biomass constituents.

In general, combustion in a calorimetric oxygen bomb or in dedicated lab scale/bench scale combustion devices seems to be the preferred way to convert fuel carbon in carbon dioxide. Commercially available PSM instruments can be used, or the ^{14}C determination from the specialized laboratory using the dedicated gas counting and AMS techniques may be an alternative for samples with a low biomass contents.

Although not in the scope of this work field, determining carbon dioxide originating from biomass sampled in the stack of an installation would be a good solution for problems with homogeneity of fuel samples or long term monitoring of the biomass content.

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Annex A
(informative)

Origin of expertise present in the technical report

A.1 Introduction

To prepare this Technical Report five centres of competence were identified and visited. Those centres have long-term experience with the application of ^{14}C-based methods in archaeological dating or in determining biomass content. They were visited and expert opinions were obtained regarding the subject of this paper.

A.2 Leoben University, Institut fur nachhaltige Abfallwirtschaft und Entsorgungstechnik (IAE, Waste management and Technology)

Expertise: Several years of experience with the ^{14}C biomass method, mainly focused on solid recovered fuels.

Initiators Peter Kneissl and Dr. Georg Raber started experiments on this subject four years ago. At the beginning combustion of the sample in the calorimetric oxygen bomb was used to convert carbon into carbon dioxide from solid recovered fuels. Nowadays a dedicated bench scale rotary kiln oven is available equipped with on-line gas analysis equipment.

A lot of validation experiments have been performed until now and in close cooperation with ARC Seibersdorf, the process to determine ^{14}C content has been optimised.

The developed sampling procedure is very robust and a clean 1-methoxy-propane-3-amine solution can be obtained due to the effective use of selective scrubbers.

Initially the formed carbon dioxide was collected by precipitation as barium carbonate. Simultaneously the total carbon content was gravimetrically determined by weighing the barium carbonate precipitate. Currently direct absorption of the carbonate in 1-methoxy-propane-3-amine solution is used. Still the total carbon content can be measured by determining the density of the 1-methoxy-propane-3-amine solution, as the density is linear proportional to the amount of bonded carbonate in the 1-methoxy-propane-3-amine.

Facilities: Normal laboratory equipment, bench scale rotary kiln oven with on line gas analysis equipment.

A.3 Austrian Research Centers, Seibersdorf research

Expertise: The Radiation Protection Unit of the Austrian Research Centre Seibersdorf (Health and Physics division) is the lab where the determination of the ^{14}C content for the Leoben University samples is performed. The Radiation Protection Group, directed by Dr. Erich Hrnecek, has a broad experience on low and ultra lowlevel radiation determination in a wide range of materials. In close cooperation with the Leoben University the measurement of the biomass content using the ^{14}C PSM determination has been optimised. Using the standard addition technique for the determination of the counting efficiency, a very robust and reliable PSM method has been developed. Ongoing investigations on this application of the PSM technique are focused on the further improvement of performance characteristics.

Facilities: Low background Liquid Scintillation Counter. A wide diversity of other dedicated low level radiation equipment. The lab is certificated for radiocarbon measurements.

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Performance characteristics of the Seibersdorf facilities:

Background:	1,22 cpm
Counting efficiency:	50 % to 62 % depending on quench (colour)
Detection limit:	0,27 dpm per sample for 1 500 min counting time.

A.4 Heidelberg Akademy der Wissenschaften, Institut fur Umweltphysik

Expertise: The radiocarbon lab, directed by Dr. Bernd Kromer, is one of the few labs in Europe that specialized in the use of the ^{14}C isotope for environmental purposes and archaeological applications. The equipment is very similar to the gas-counting facility of Groningen, as described below under A.6. The group participates in quality control tests organized by Glasgow University.

Main activities:

- Calibration of the ^{14}C time scale for archaeological/geosciences purposes.
- High precision ^{14}C analyses for key archaeological sites.

Their environmental expertise has a close relation to the ^{14}C-based biomass content determination. In Heidelberg the ^{14}C content of the carbon dioxide present in outdoor air is monitored continuously in a world-wide network, to determine the fossil fuel attribution to the carbon dioxide content. By monitoring the emission of fossil carbon dioxide over a long period of time the effect of implementing the Kyoto protocol can be effectively measured. Recent investigations on determining the biomass origin of vinegar and bio diesel fuel based on the ^{14}C content illustrates the suitability for this method for the SRF biomass content determination.

Facilities: Lab scale facilities for the conversion of carbon to the desired physical state for the available ^{14}C determination techniques. A number of ultra low ^{14}C detection instruments (Gas counting equipment). Devices for the collection of carbon dioxide from solid, liquid and gaseous matrices. High volume oxygen bomb enabling combustion of several grams of sample.

A.5 Utrecht University, Institute for Subatomic Physics

Facilities: The AMS-facility in the Van de Graaff laboratory of Utrecht University is based on the 6 MV tandem Van de Graaff accelerator, which has been originally used for precision research of nuclear spectroscopy. The accelerator has been adapted for AMS with stable beam transmission and minimal beam loss. Precision measurements are made with fast switching between isotope beams in an automated scheme. Participation in intercomparison studies confirms the quoted precision. Chemical preparation techniques have been set up to isolate the suited chemical fractions in applications.

Analyses are performed in a broad range of research disciplines. Most of these concern radiocarbon dating for applications in earth sciences and cultural sciences, and to a lesser extent with other radio nuclides. Analyses are performed as part of projects or are as a service at the corresponding tariff.

A.6 Groningen University, Center for Isotope Research

Person in charge: Dr. Ir. Hans van der Plicht. The research group is performing a lot of ^{14}C analyses, mainly for archaeology (Carbon-14 dating), but also for hydrology, geochemistry and sometimes for specific customers like Shell and Philips. Mainly the work can be divided into two categories:

a) Radiometry of ^{14}C, by using gas-counting equipment
   Samples are converted into CO_2, the gas is frozen, after which it is introduced into gas counting tubes. The gas counting device consists of a gas counting tube that is surrounded by GM tubes, which are anti-coincidentally connected with the gas counting tube, all the tubes are surrounded by a coffin of ‘old’ lead

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and the whole setting is placed in the basement of the building. Counting times may vary from 1 day up to 1 week. For a good determination grams of material are needed, per year some 1 000 samples are analysed.

b) ^{14}C determinations with Accelerator Mass Spectrometry (AMS)
For this technique the group needs some 1 mg of graphite. The starting point is mainly combustible material that is being burnt in a CHN analyser, after which the product CO_2 is frozen in a cold trap by using liquid nitrogen. CO_2 is converted on a hot Fe catalyst with H_2 into C (graphite) and H_2O. From the C a 1 mm target is being made, after which the target is placed in a sample holder of the AMS. It is bombarded with Cs ions from a sputter source after which the ions are accelerated in a 2,5 MV High Voltage field. After regulation of the beam it is passed through a configuration of magnets so that ^{12}C, ^{13}C and ^{14}C are determined simultaneously. Per year some 15 000 samples are analysed, some 60 samples per day are run. The group is involved in international ring tests (‘round robins’) and commissions.

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Annex B
(informative)

List of European lab's with radio carbon expertise

B.1 Conventional (radiometric) labs

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