CIRAM

ANALYTICAL REPORT
(INS-7.8-01_V2.3)

Report
N°1023-BS-1284-9a

DETERMINATION OF STABLE ISOTOPES AND BIO-BASED CARBON CONTENT USING EA-IRMS AND RADIOCARBON

EN 16640:2017 (Method B)

Customer :

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Martillac, March 8, 2024

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SAMPLE

Sampling was done by the customer. The results only apply to the sample as received.

CIRAM code	**CBIO-1922**
Name of the sample[1]	Biobased Cadmium Free Yellow Medium
Customer sample number[1]	**Sample 9**
Batch number[1]	nc
Date of reception item	10/12/2023
Non-conformity at arrival	none

ANALYSIS

CIRAM code	**CBIO-1922**
Customer sample number	**Sample 9**
Methods of analysis	$\delta^{13}C$ and $\delta^{15}N$ by EA-IRMS (MO 6.4-02) Carbon extraction by automated Graphitization before ^{14}C analysis (MO 6.4-03) pMC by radiocarbon (ASTM D6866-22 Method B - AMS)
Date of EA – IRMS – Graphitization	10/18/2023
Date of AMS	10/25/2023
Done by	SC / MG / OD / ZE
Special conditions	SO
$\delta^{15}N$ (‰)[2]	Undetected
$\delta^{13}C$ (‰)[2]	-25.68 ± 0.03
Percentage of Modern Carbon (pMC)[3]	48.60 ± 0.18
x_B^{TC} is the biobased carbon content expressed as a percentage of the total carbon content[4]	**49%**
x^{TC} is the total carbon content expressed as a fraction of the total sample mass (%)	18.25
x_B is the biobased carbon content by mass (%)	8.87

[1] Information supplied by the customer. CIRAM is not responsible about this information.
[2] Uncertainty at 2σ is 0.26‰ for $\delta^{13}C$ and 0.61‰ for $\delta^{15}N$. The value after "±" corresponds to the standard deviation of replicates. "undetected" indicates [N] < 5% et [C] < 1%.
[3] Radiocarbon analysis are partly carried out in joint-venture with a European accredited lab ISO 17025 (JSC «Barnas» LA.210-01).
[4] The standard EN 16640 :2017 indicates that the radiocarbon measure can suffer a deviation of ± 2 %.

SUMMARY

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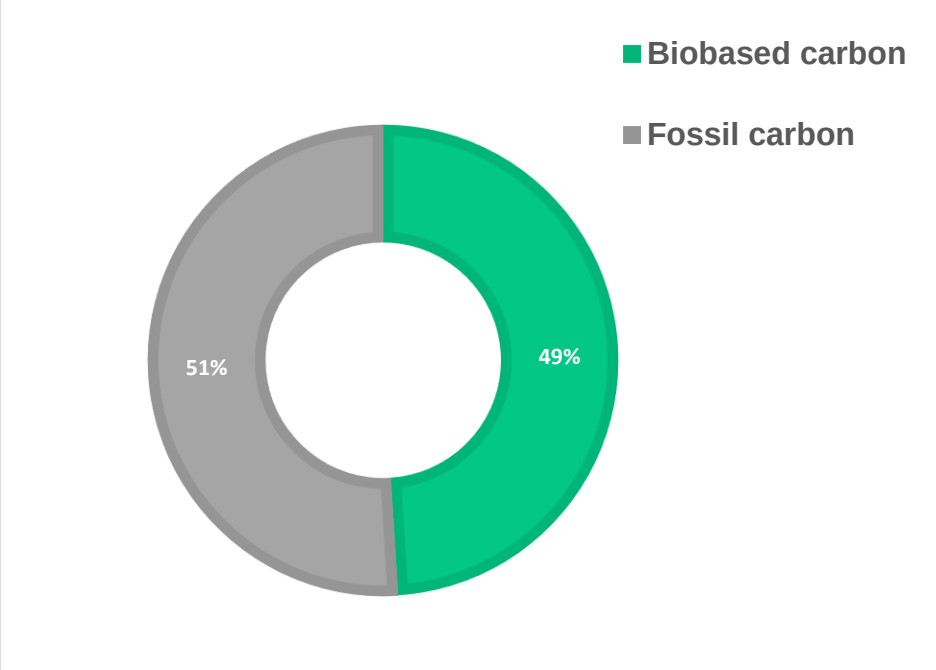
Customer sample number	**Sample 9**
$\delta^{15}N$ (‰)[2]	Undetected
$\delta^{13}C$ (‰)[2]	-25.68 ± 0.03
Percentage of Modern Carbon (pMC)[3]	48.60 ± 0.18
x_B^{TC} is the biobased carbon content expressed as a percentage of the total carbon content[4]	**49%**
x^{TC} is the total carbon content as a fraction of the sample mass	18.25
x_B is the biobased carbon content as a fraction of the sample mass	8.87



Authorized by :

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EXPERIMENTAL METHOD

The study presented here takes place in the context of quantification of the bio-based carbon and the fossil carbon into industrial production. The purpose of the methodology described below is to quantify the percentage of biobased (derived from biomass) versus petrochemical (fossil carbon) carbon, based on the measure of the carbon and nitrogen.
The Modern Carbon is the contemporary carbon present today in the atmosphere and in the biomass. The radiocarbon measurement is expressed as part of Modern Carbon (pMC). This corresponds to the percentage of radiocarbon (^{14}C) measured in the sample. The percentage of bio-based carbon is calculated on pMC basis, the total carbon content and an atmospheric adjustment factor (REF).
The reference value used for the carbon year adjustment is 99.7 in 2024 (ASTM D6866-24). This means that a 100 % natural product manufactured in 2024 has a pMC of 99.7.
The percentage of bio-based carbon corresponds to the percentage of “natural” carbon (derived from biomass) versus “fossil” carbon (derived from petrochemistry). A 100 % bio-based carbon compound is made from 100 % plants and/or animal by-products. A 0 % bio-based carbon compound corresponds to a product entirely of fossil origin, which does not contain any carbon from plant and/or animal by-products. Therefore, a value between 0 and 100 % confirms a mixture of bio-based and fossil carbon, indicating the percentage of bio-based carbon in the total carbon.
The sample is combusted at a temperature of 920°C and is transformed into gas. During this first step, a measure of % C and N is performed using an elementar analyser (Elementar Vario ISOTOPE Select). The EA allows separation of combustion gases and also removal of water. Residual carbon dioxide (CO_2) from the EA outlet is absorbed in the zeolite trap of an AGE automated graphitization system (AGE 3, Ion Plus) and then released to the given reactor to be transformed into graphite by catalysis. Meanwhile $^{13}C/^{12}C$ ratio (expressed as $\delta^{13}C$) and $^{15}N/^{14}N$ (expressed as $\delta^{15}N$) were measured using a mass spectrometer dedicated to stable isotopic ratio (IRMS, Elementar Isoprime precisION). The different carbon isotopes were separated using a 250 kV accelerator mass spectrometer in joint venture with JSC Barnas (ISO 17025 and ISO 14001). ^{14}C content is determined by comparing the simultaneously collected ^{14}C, ^{13}C and ^{12}C beams with those of control products: Oxalic Acid, CO_2 standard, charcoal).
Conventional radiocarbon age is calculated according to the method described by Stuiver and Polach. It takes into account the $\delta^{13}C$ correction for isotopic fractionation, based on the comparison between the concentration measurements of $^{13}C/^{12}C$ and $^{14}C/^{12}C$. This factor enables the control of potential pollution and further evaluate the reliability of the measure, it is a good indicator of the quality of the sample. The precision on the analytical measure of pMC is 1σ (1 sigma relative standard deviation). International standards NIST 4990C, IAEA-C-7 et IAEA-C-9 were used. $\delta^{13}C$ is expressed per mile (‰) in conformity with international standard V-PDB (Vienna Pee Dee Belemnite). $\delta^{15}N$ is expressed per mile (‰) in relation to Air.

NOTE

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All traceability elements including measure uncertainty are available on request. For any subcontracted results supplied by accredited laboratories, measures are also available.

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