

Beta Analytic Inc.

Please see our comments attached. The submission form is only showing the "individual" option, but this comment is intended on behalf of Beta Analytic Inc. We appreciate the opportunity to share our insight with the program very much, thank you!



Washington State Department of Ecology
Washington Clean Fuel Standard (CFS) Program | Stakeholder Feedback

This comment is intended to recommend the use of the carbon-14 testing method to determine the share of biogenic carbon content of feedstocks, fuels and emissions under Washington’s Clean Fuel Standard program. Biogenic content measurements following methods such as ASTM D6866 Method B currently provide critical value to existing clean fuel standard programs at the state and federal levels.

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Recommendations for Washington’s Clean Fuel Standard

Our recommendation is that Washington’s Clean Fuel Standard should include direct biogenic content testing requirements following the ASTM D6866 Method B standard for any fuels or feedstocks seeking recognition of renewable (biogenic) content. Routine direct biogenic testing requirements are the only reliable method of incentivizing the use of biomass derived content and guaranteeing compliance and currently play a critical role in prominent similar programs.

This comment is specifically meant to address two of Ecology’s key questions, “1) How might Ecology align with other clean fuel programs to streamline regulatory requirements and compliance?
2) What provisions of a third-party verification program will be most impactful for ensuring data accuracy and quality?” Implementing routine direct testing would incorporate best practices established by successful similar programs at the state, federal and international levels and would improve Washington’s program with respect to both of these key questions.



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Routine direct test results are currently used to verify biogenic content under the US EPA's [Renewable Fuel Standard \(RFS\)](#), California's [Low Carbon Fuel Standard \(LCFS\)](#), Oregon's [Clean Fuels Program](#), Canada's [Clean Fuel Regulations \(CFR\)](#) and the EU's [Renewable Energy Directive \(RED\)](#). All of these programs except the EU RED specifically require ASTM D6866, while the EU RED accepts ASTM D6866 or its European equivalents. ASTM D6866 is also required for prominent third-party verification programs, most notably the Roundtable on Sustainable Biomaterials (RSB).¹ Testing requirements allow clean fuel programs to exclusively incentivize the renewable portion of fuels. This is especially important given the recent history of attempted fraud in existing transportation fuel decarbonization programs.

Washington's Clean Fuel Standard program should specifically require direct biogenic testing for any fuels produced from co-processing, municipal solid waste (MSW) biogas & renewable natural gas (RNG) and any other fuels for which the final biogenic content is unknown. Current requirements of routine direct testing following ASTM D6866 under similar prominent programs includes:

- The [US RFS](#) currently requires routine direct testing following ASTM D6866 for fuels produced from co-processing, municipal solid waste (MSW) biogas & renewable natural gas (RNG).²
- [California's LCFS](#) requires routine direct testing for fuels produced from co-processing and recommends for fuels produced from MSW.³
- [Oregon's CFP](#) requires routine direct testing following the protocols of the US RFS third-party engineering reviews.⁴
- [Canada's CFR](#) requires routine direct testing for any fuels produced from co-processing and their co-products.⁵
- [The EU's RED](#) requires routine direct testing for any fuels produced from co-processing or biogas & renewable natural gas (RNG).⁶

It is critically important that this program require direct testing rather than allow calculation based approaches such as mass balance, which make claims based on material inputs in production. These calculations allow producers to assume that all of their biomass inputs end up in their facilities' outputs, despite it being well understood in the industry that the input of renewable feedstocks is not the same as the output because performance varies and renewable feedstocks don't produce the same quantity of material as their fossil counterparts.⁷ By basing their calculations solely on production inputs rather than outputs these methods systematically over-report the renewable share of fuels.

¹ 2023. "RSB Standard for Advanced Fuels." *Roundtable on Sustainable Biomaterials (RSB)*

² 2023. "40 CFR Parts 80 and 1090– Renewable Fuel Standard (RFS) Program: Standards for 2023–2025 and Other Changes." *EPA*

³ 2020. "Reporting Co-Processing and Renewable Gasoline Emissions Under MRR." *California Air Resources Board*

⁴ 2023. "Oregon Clean Fuels Program." *Oregon Department of Environmental Quality*

⁵ 2022. "Clean Fuel Regulations: Quantification Method for Co-Processing in Refineries." *Environment and Climate Change Canada*

⁶ 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." *European Commission*

⁷ 2006. "Determining the modern carbon content of biobased products using radiocarbon analysis." *Bioresource Technology*, 97(16), 2084-2090.



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Even further, calculation based approaches leave low carbon fuel programs susceptible to high risk of greenwashing and fraud. For example this threat is highlighted by the recent mass balance fraud challenges faced by the ISCC regarding fraudulent biodiesel submissions from China which “caused a dramatic fall in biodiesel prices in European markets” in July 2023.⁸ In response to this situation the EU quickly updated the RED’s co-processing rules to uniformly require direct testing, including for producers choosing calculation based approaches to verify their calculations.⁹

Establishing this Clean Fuel Standard was a critical first step in the state of Washington’s decarbonization journey. By implementing best practices for verification established by similar state, federal and international fuel decarbonization programs the Department of Ecology can protect and strengthen its ability to successfully achieve and measure the goals of this program. Routine direct testing following ASTM D6866 Method B is the most effective way to incentivize and validate biogenic content under this program.

What is Biogenic Testing (Carbon-14)?

Carbon-14 analysis is a reliable method used to distinguish the percentage of biobased carbon content in a given material. The radioactive isotope carbon-14 is present in all living organisms and recently expired material, whereas any fossil-based material that is more than 50,000 years old does not contain any carbon-14 content. Since Carbon-14 is radioactive, the amount of carbon-14 present in a given sample begins to gradually decay after the death of an organism until there is no carbon-14 left. Therefore, a radiocarbon dating laboratory can use carbon-14 analysis to quantify the carbon-14 content present in a sample, determining whether the sample is biomass-based, fossil fuel-derived, or a combination.

The analysis is based on standards such as ASTM D6866 and its international equivalents developed for specific end uses, such as ISO 13833. ASTM D6866 is an international standard developed for measuring the biobased carbon content of solid, liquid, and gaseous samples using radiocarbon dating.¹⁰ There are also many international standards based on the specific use of direct Carbon-14 testing, such as ISO 13833, which is an international standard developed for measuring the biogenic carbon content of stationary sources emissions.¹¹

Carbon-14 analysis yields a result reported as % biobased carbon content. If the result is 100% biobased carbon, this indicates that the sample tested is completely sourced from biomass material such as plant or animal byproducts. A result of 0% biobased carbon means a sample is only fossil fuel-derived. A

⁸ 2023. “ISCC Press Release July 27, 2023.” *International Sustainability & Carbon Certification*

⁹ 2023. “Renewable energy- method for calculating the share of renewables in the case of co-processing.” *European Commission*

¹⁰ 2021. “Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis.” *ASTM International (D6866-21)*

¹¹ 2013. “ISO 13833:2013 Stationary source emissions: Determination of the ratio of biomass (biogenic) and fossil-derived carbon dioxide.” *International Organization for Standardization*



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sample that is a mix of both biomass sources and fossil fuel sources will yield a result that ranges between 0% and 100% biobased carbon content. Carbon-14 testing has been incorporated into several regulations as the recommended or required method to quantify the biobased content of a given material.

ASTM D6866 Method B - The Most Reliable Method

Carbon-14 is a very well-established method which has been in use by many industries (including the fossil fuel industry) and academic researchers for several decades.

Carbon-14 measurements done by commercial third party testing is robust, consistent, and with quantifiable accuracy/precision of the carbon-14 amount under **ASTM D6866 method B**. The EN 16785 is the only standard that allows a variant of the Mass Balance (MB) method of ‘carbon counting’ under EN 16785-2. The EN 16785-1 requires that the biocarbon fraction be determined by the carbon-14 method. However, when incorporating this EN 16785 method, certification schemes like the “Single European Bio-based Content Certification” **only** allow the use of EN 16785-1 due to its reliability and the value of a third-party certification. <http://www.biobasedcontent.eu/en/about-us/>

In ASTM D6866 method B, the carbon-14 result is provided as a single numerical result of carbon-14 activity, with graphical representation that is easily understood by regulators, policy makers, corporate officers, and more importantly, the public. The overwhelming advantage of carbon-14 is that it is an independent and standardized laboratory measurement of any carbon containing substance that produces highly accurate and precise values. In that regard, it can stand alone as a quantitative indicator of the presence of biobased vs. petroleum feedstocks. When carbon-14 test results are challenged, samples can be rapidly remeasured to verify the original reported values (unlike mass balance).

The quantification of the biobased content of a given product can be as low as 0.1% to 0.5% (1 relative standard deviation – RSD) based on Instrumental error for Method B (AMS). This error is exclusive of indeterminate sources of error in the origin of the biobased content, and manufacturing processes. As such a total error of +/-3% (absolute) has been assigned to the reported Biobased Content to account for determinate and indeterminate factors.¹²

It is also important that the program should always require ASTM D6866 Method B, rather than allow Method C for any use. Where ASTM D6866 Method B uses the AMS Instrument to measure ¹⁴C, Method C uses Liquid Scintillation Counting (LSC). In Method B, the AMS Instrument directly measures the ¹⁴C isotopes. However, in Method C, scintillation molecules indirectly absorb the beta molecules that release

¹²2021. Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis. *ASTM International (D6866-21)*. pp 1-19. doi: 10.1520/D6866-21.



with the decay of ^{14}C and convert the energy into photons which are measured proportionally to the amount of ^{14}C in the sample. Since Method B directly measures the ^{14}C isotopes and Method C measures them indirectly, Method B is significantly more precise and should be prioritized in regulations.¹³ LSC measurements, like those used in Method C, are commonly used as an internal testing tool when samples are limited and accuracy does not need to be extremely high.

About Beta Analytic

Beta Analytic was among the originators of the use of Accelerator Mass Spectrometry (AMS) for the ASTM D6866 biobased / biogenic testing standard using Carbon-14 to distinguish renewable carbon sources from petroleum sources. Beta began testing renewable content in 2003 at the request of United States Department of Agriculture (USDA) representatives who were interested in Beta's Carbon-14 capabilities for their BioPreferred[®] Program (www.biopreferred.gov). At their request, Beta joined ASTM under subcommittee D20.96. Beta's previous president, Darden Hood, was positioned as a technical contact for the USDA and within 3 months completed the ASTM D6866-04 standard. The Carbon-14 technique is now standardized in a host of international standards including ASTM D6866, CEN 16137, EN 16640, ISO 16620, ISO 19984, BS EN ISO 21644:2021, ISO 13833 and EN 16785. Carbon-14 analysis can be used on various types of samples (gas, liquids and solids). Beta Analytic continues to be a technical contact for ASTM D6866 with current president Ron Hatfield and is involved with all their latest ASTM D6866 versions.

The Carbon-14 standardized method is also incorporated in a variety of regulatory programs including the California AB32 program, US EPA GHG Protocol, US EPA Renewable Fuels Standard, United Nations Carbon Development Mechanism, Western Climate Initiative, Climate Registry's Greenhouse Gas Reporting Protocol and EU Emissions Trading Scheme.

We are currently technical experts on Carbon-14 in the following committees:

- ASTM D6866 (D20.96) Plastics and Biobased Products (Technical Advisor)
- ASTM (D02.04) Petroleum Products, Liquid Fuels and Lubricants (Technical Advisor)
- ASTM (061) US TAG to ISO/TC 61 Plastics (Technical Expert)
- USDA BioPreferred Program TAC (Technical Advisor)
- ISO/TC 61/SC14/WG1 Terminology, classifications, and general guidance (Technical Expert)
- CEN/TC 411 Biobased Products
- CEN/TC 411/WG 3 Biobased content
- CEN/TC 61/SC 14/WG 1 Terminology, classifications, and general guidance (Technical Expert)

¹³2022. "Testing the methods for determination of radiocarbon content in liquid fuels in the Gliwice Radiocarbon and Mass Spectrometry Laboratory." *Radiocarbon*



ISO/IEC 17025:2017 Accredited Laboratory

To ensure the highest level of quality, laboratories performing ASTM D6866 testing should be ISO/IEC 17025:2017 accredited or higher. This accreditation is unbiased, third party awarded and supervised. It is unique to laboratories that not only have a quality management program conformant to the ISO 9001:2008 standard, but more importantly, have demonstrated to an outside third-party laboratory accreditation body that Beta Analytic has the technical competency necessary to consistently deliver technically valid test results. The ISO 17025 accreditation is specifically for natural level radiocarbon activity measurements including biobased analysis of consumer products and fuels, and for radiocarbon dating.

Required tracer-free facility for Carbon-14

For carbon-14 measurement to work, be accurate, and repeatable, the facility needs to be a tracer-free facility, which means artificial/labeled carbon-14 is not and has never been handled in that lab. Facilities that handle artificial carbon-14 use enormous levels relative to natural levels and it becomes ubiquitous in the facility and cross contamination within the facility, equipment and chemistry lines is unavoidable. Results from a facility that handles artificial carbon-14 would show elevated renewable contents (higher pMC, % Biobased / Biogenic values), making those results invalid. Because of this, Federal contracts and agency programs (such as the USDA BioPreferred Program) require that AMS laboratories must be 14C tracer-free facilities in order to be considered for participation in solicitations.

To learn more about the risks associated with testing natural levels Carbon-14 samples in a facility handling artificially enhanced isotopes please see the additional information provided after this comment.



References

2006. "Determining the modern carbon content of biobased products using radiocarbon analysis." *Bioresource Technology*, 97(16), 2084-2090.
2010. "40 CFR Part 80 Subpart M– Renewable Fuel Standard." *National Archives Code of Federal Regulations*
<https://www.ecfr.gov/current/title-40/chapter-I/subchapter-C/part-80/subpart-M>
2013. "ISO 13833:2013 Stationary source emissions: Determination of the ratio of biomass (biogenic) and fossil-derived carbon dioxide." *International Organization for Standardization*
2020. "Reporting Co-Processing and Renewable Gasoline Emissions Under MRR." *California Air Resources Board*
https://ww2.arb.ca.gov/sites/default/files/2020-09/MRR_coprocessing-slides_Sept_2020.pdf
2021. "Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis." *ASTM International (D6866-21)*. pp 1-19. doi: 10.1520/D6866-21.
2022. "Clean Fuel Regulations: Quantification Method for Co-Processing in Refineries." *Environment and Climate Change Canada*
<https://www.canada.ca/en/environment-climate-change/services/managing-pollution/energy-production/fuel-regulations/clean-fuel-regulations/compliance/quantification-methodco-processing-refineries.html>
2022. "Testing the methods for determination of radiocarbon content in liquid fuels in the Gliwice Radiocarbon and Mass Spectrometry Laboratory." *Radiocarbon*, 64(6), pp.1-10. DOI:10.1017/RDC.2022.35
2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." *European Commission*
https://ec.europa.eu/info/law/better-regulation/have-your-say/initiatives/12711-Renewable-energy-method-for-calculating-the-share-of-renewables-in-the-case-of-co-processing_en
2023. "40 CFR Parts 80 and 1090– Renewable Fuel Standard (RFS) Program: Standards for 2023–2025 and Other Changes." *Environmental Protection Agency*
<https://www.govinfo.gov/content/pkg/FR-2023-07-12/pdf/2023-13462.pdf>
2023. "Oregon Clean Fuels Program." *Oregon Department of Environmental Quality*
<https://secure.sos.state.or.us/oard/viewSingleRule.action?ruleVrsnRsn=293938>
2023. "RSB Standard for Advanced Fuels." *Roundtable on Sustainable Biomaterials (RSB)*
https://rsb.org/wp-content/uploads/2024/03/RSB-STD-01-010-RSB-Standard-for-advanced-fuels_v2.6-1.pdf

Demand a Tracer-Free Laboratory for Radiocarbon Dating

As part of its commitment to provide high-quality results to its clients, ISO/IEC 17025-accredited Beta Analytic does not accept pharmaceutical samples with “tracer Carbon-14” or any other material containing artificial Carbon-14 (14C) to eliminate the risk of cross-contamination. Moreover, the lab does not engage in “satellite dating” – the practice of preparing individual sample graphite in a remote chemistry lab and then subcontracting an AMS facility for the result.

High Risk of Cross-Contamination

Pharmaceutical companies evaluate drug metabolism by using a radiolabeled version of the drug under investigation. AMS biomedical laboratories use 14C as a tracer because it can easily substitute 12C atoms in the drug molecule, and it is relatively safe to handle. Tracer 14C is a well-known transmittable contaminant to radiocarbon samples, both within the AMS equipment and within the chemistry lab.

Since the artificial 14C used in these studies is phenomenally high (enormous) relative to natural levels, once used in an AMS laboratory it becomes ubiquitous. Cross-contamination within the AMS and the chemistry lines cannot be avoided. Although the levels of contamination are acceptable in a biomedical AMS facility, it is not acceptable in a radiocarbon dating facility.

Biomedical AMS facilities routinely measure tracer-level, labeled (Hot) 14C samples that are hundreds to tens of thousands of times above the natural 14C levels found in archaeological, geological, and hydrological samples. Because the 14C content from the biomedical samples is so high, even sharing personnel will pose a contamination risk; “Persons from hot labs should not enter the natural labs and vice versa” (Zermeño et al. 2004, pg. 294). These two operations should be absolutely separate. Sharing personnel, machines, or chemistry lines run the risk of contaminating natural level 14C archaeological, geological, and hydrological samples.

Avoid the Risks

Find out from the lab that you are planning to use that they have never in the past and will never in the future:

- accept, handle, graphitize or AMS count samples containing Tracer or Labeled (Hot) 14C.

- share any laboratory space, equipment, or personnel with anyone preparing (pretreating, combusting, acidifying, or graphitizing) samples that contain Tracer or Labeled (Hot) 14C.

- use AMS Counting Systems (including any and all beam-line components) for the measurement of samples that contain Tracer or Labeled (Hot) 14C.

Tracer-Free Lab Required

Recently, federal contracts are beginning to specify that AMS laboratories must be 14C tracer-free facilities in order to be considered for participation in solicitations.

A solicitation for the National Oceanic and Atmospheric Administration (NOAA) has indicated that “the AMS Facility utilized by the Contractor for the analysis of the micro-samples specified must be a 14C tracer-level-free facility.” (Solicitation Number: WE-133F-14-RQ-0827 - Agency: Department of Commerce)

As a natural level radiocarbon laboratory, we highly recommend that researchers require the AMS lab processing their samples to be Tracer-free.

No Exposure to Artificial Carbon-14

According to ASTM International, the ASTM D6866 standard is applicable to laboratories working without exposure to artificial carbon-14 routinely used in biomedical studies. Artificial carbon-14 can exist within the laboratory at levels 1,000 times or more than 100 % biobased materials and 100,000 times more than 1% biobased materials. Once in the laboratory, artificial ^{14}C can become undetectably ubiquitous on materials and other surfaces but which may randomly contaminate an unknown sample producing inaccurately high biobased results. Despite vigorous attempts to clean up contaminating artificial ^{14}C from a laboratory, isolation has proven to be the only successful method of avoidance. Completely separate chemical laboratories and extreme measures for detection validation are required from laboratories exposed to artificial ^{14}C . Accepted requirements are:

- (1) disclosure to clients that the laboratory working with their products and materials also works with artificial ^{14}C
- (2) chemical laboratories in separate buildings for the handling of artificial ^{14}C and biobased samples
- (3) separate personnel who do not enter the buildings of the other
- (4) no sharing of common areas such as lunch rooms and offices
- (5) no sharing of supplies or chemicals between the two
- (6) quasi-simultaneous quality assurance measurements within the detector validating the absence of contamination within the detector itself.

ASTM D6866-22 – Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis.



Useful Reference

1. Memory effects in an AMS system: Catastrophe and Recovery. J. S. Vogel, J.R. Southon, D.E. Nelson. Radiocarbon, Vol 32, No. 1, 1990, p. 81-83 doi:10.2458/azu_js_rc.32.1252 (Open Access)

"... we certainly do not advocate processing both labeled and natural samples in the same chemical laboratory." "The long term consequences are likely to be disastrous."

2. Recovery from tracer contamination in AMS sample preparation. A. J. T. Jull, D. J. Donahue, L. J. Toolin. Radiocarbon, Vol. 32, No.1, 1990, p. 84-85 doi:10.2458/azu_js_rc.32.1253 (Open Access)

"... tracer ^{14}C should not be allowed in a radiocarbon laboratory." "Despite vigorous recent efforts to clean up the room, the "blanks" we measured had ^{14}C contents equivalent to modern or even post-bomb levels."

3. Prevention and removal of elevated radiocarbon contamination in the LLNL/CAMS natural radiocarbon sample preparation laboratory. Zermeño, et. al. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms Vol. 223-224, 2004, p. 293-297 doi: 10.1016/j.nimb.2004.04.058

"The presence of elevated ^{14}C contamination in a laboratory preparing samples for natural radiocarbon analysis is detrimental to the laboratory workspace as well as the research being conducted."

4. High level ^{14}C contamination and recovery at XI'AN AMS center. Zhou, et. al. Radiocarbon, Vol 54, No. 2, 2012, p. 187-193 doi:10.2458/azu_js_rc.54.16045

"Samples that contain high concentrations of radiocarbon ("hot" samples) are a catastrophe for low background AMS laboratories." "In our case the ion source system was seriously contaminated, as were the preparation lines."



Beta Analytic

www.radiocarbon.com