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ISO/IEC 17025:2017-Accredited Testing Laboratory

Washington State Department of Ecology
Washington Clean Fuel Standard (CFS) Program │ Scoping Comment for Sustainable Aviation Fuel (SAF)

This comment is intended to recommend the use of the Carbon-14 testing method to determine the share of biogenic carbon content of sustainable aviation fuels, feedstocks 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 state, federal and international clean fuel standard programs.

Included here you will find:

Recommendations for Washington's Clean Fuel Standard

Our recommendation is that Washington's Clean Fuel Standard should include direct biogenic content testing (carbon-14) requirements following the ASTM D6866 Method B standard for any sustainable aviation fuels (SAF) seeking to generate credits under the program. 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 including Washington's Clean Fuel Standard.

This comment is specifically meant to recommend the use of Carbon-14 test results for the Programmatic Environmental Impact Statement for Sustainable Aviation Fuel. Biogenic content is a critical lens to use when studying SAF because these results measure the renewable portion of the SAF, which must be tracked in order to claim environmental benefits at the point of use. This comment will

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discuss the important applications for determining the biogenic content of SAF relevant for this scoping period.

One aspect of biogenic content determinations for SAF which should be considered in this scoping period is the role of accounting for biogenic content in a book and claim system. As SAF is implemented by more airports around the world, airlines, fuel providers and regulators are exploring the potential for an indirect accounting system to allocate SAF use to flights and end consumers. In this scoping period for the Programmatic Environmental Impact Statement for Sustainable Aviation Fuel, Ecology should study how direct biogenic measurements following ASTM D6866 can be used to “book” the renewable content of SAF prior to blending with Jet A, so that the exact amount of renewable content “claimed” at the end use can correspond directly with the content demonstrated through biogenic test results. This would enable a comprehensive book and claim system which provides flexibility to airlines, while providing clear evidence to regulators.

Another topic related to biogenic content determinations for SAF which should be considered in this scoping period is when to test fuels that will be co-mingled with fossil Jet A. When chemically identical biofuels and fossil fuels are commingled for storage, transportation or distribution, the only way to identify renewable content is through Carbon-14 testing. This is because Carbon-14 is an atomic-level measurement which can differentiate between renewable and fossil versions of the same molecule. This is critical to a program like Washington’s Clean Fuels Program because many biofuels rely on existing infrastructure for their storage and transportation. In order to use existing infrastructure, fuels must be drop-in replacements and can therefore only be identified by Carbon-14 analysis once commingled.

One other aspect of biogenic content determinations for SAF which should be considered in this scoping period is the accuracy of direct testing, as opposed to indirect accounting methods for co-processing facilities. Producers and industry lobbying groups advocate for calculation based approaches such as mass balance because they enable facilities to make claims solely 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.[1] By basing their calculations solely on production inputs rather than outputs these methods systematically over-report the renewable share of fuels.

Calculation based approaches also use a system of free allocation, meaning they do not have to guarantee that there is any renewable content in a given fuel. Producers prefer this because if 10% of their feedstocks are biogenic they can claim that 10% of their products are biogenic, even if that's not

[1] 2006. “Determining the modern carbon content of biobased products using radiocarbon analysis.” *Bioresource Technology*, 97(16), 2084-2090.

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the case because biobased can go in different amounts to different products in the co-process. Even further, book and claim also allows them to claim that 10% of their products are 100% biogenic and the rest are 0%, even if all of the products should be 10% biogenic based on calculations (and would likely C14 test below that).[2]

These calculations' reliance on free allocation creates the potential for double counting of renewable content, leaving 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.[3] In response to this situation, the EU quickly updated the RED's co-processing rules to uniformly require direct testing, including to verify the calculations of producers choosing to use calculation based approaches.[4]

As a result, the EU quickly introduced biogenic testing requirements for fuels produced from biogas in a June 2023 update to the EU Renewable Energy Directive (RED) titled, "Renewable energy- method for calculating the share of renewables in the case of co-processing."[5] The EU investigation into this issue is still ongoing, and the full extent of the damage is not yet known, but this was a significant setback for the program and quickly plummeted biodiesel prices in the EU. The advantage of the updated RED protocol is that producers can choose to use calculations internally for their convenience, while the program still ensures the information reported is accurate through direct Carbon-14 analysis. This is the only way to mitigate the risk to the program introduced by these calculations.

In addition to the issues related to mass balance under the EU RED, Beta also recommends that Ecology review the following studies on co-processing conducted by the ASTM D02 Committee on petroleum products, liquid fuels and lubricants. We specifically recommend reviewing RR:D02-2052, which compares the results of ^{14}C and mass balance in co-processing facilities.[6] The table below shows an example of that study's key findings.

[2] 2024. "The Mass Balance Approach." *International Sustainability & Carbon Certification*
[3] 2023. "ISCC Press Release July 27, 2023." *International Sustainability & Carbon Certification*
[4] 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." *European Commission*
[5] 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." *European Commission*
[6] 2023. "RR:D02-2052." *ASTM International*

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Standard	**Report #**					
D1655	RRD02-2052					
Findings						
Pre-Fractionation Blend Analysis						
	Component, vol %	**Blend 1 Pre**	**Blend 2 Pre**	**Blend 3Pre**		
	Petrochemical Stream	**60**	**80**	**80**	***This Pre-Fractionation verification of biogenic content of the blends demonstrates the accuracy of ASTM D6866***	
	Feed 1	**40**				
	Feed 2		**20**			
	Feed 3			**20**		
	D6866 Method B	**40**	**20**	**20**		
Biogenic %, per ASTM D6866 Method B	**Blend 1 Pre**	**Blend 1 Post**	**Blend 2 Pre**	**Blend 2 Post**	**Blend 3 Pre**	**Blend 3 Post**
	40	**17**	**20**	**7**	**20**	**5**

Standard	**Report #**					
D1655	RRD02-1886					
Findings						
Biomass Input 5%, yield in final product by ASTM D6866 2.1%						

The black font in the table shows the expected values of biogenic content based on mass balance calculations, while the red shows the actual values reported by direct testing. The study shows that mass balance consistently over-estimated the biogenic content which ended up in co-processed fuels because biomass does not behave the same as fossil feedstocks, and not all of the biomass inputs necessarily end up in the same output.[7]

We recommend further reviewing RR:D02-2052, as well as the rest of this collection of technical reports which includes RR:D02-1886, RR:D02-1929, RR:D02-2052, RR:D02-1739, RR:D02-1810, RR:D02-1776, RR:D02-1884, RR:D02-1828, and RR:D02-2039. Several of these studies specifically compare the results of ^{14}C and mass balance in co-processing facilities in the context of sustainable aviation fuel production in particular. These studies found that mass balance calculations are consistently unable to estimate the renewable portion of co-processed fuels and should not be relied on as the sole method of verification for clean fuel programs. All of these technical reports are available from ASTM upon request.

Washington's Clean Fuel Standard program should require direct biogenic testing for any SAF 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 include (please see specific rules hyperlinked):

[7] 2023. "RR:D02-2052." *ASTM International*

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- 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).[8]
- California's LCFS requires routine direct testing for fuels produced from co-processing and recommends for fuels produced from MSW.[9]
- Oregon's CFP requires routine direct testing following the protocols of the US RFS third-party engineering reviews.[10]
- Canada's CFR requires routine direct testing for any fuels produced from co-processing and their co-products.[11]
- British Columbia's LCFS requires monthly testing for any fuels produced from co-processing and quarterly testing for their co-products, as well as to verify biogenic feedstocks.[12]
- The EU's RED requires routine direct testing for any fuels produced from co-processing or biogas & renewable natural gas (RNG).[13]

ASTM D6866 is also required for prominent third-party verification programs, most notably the Roundtable on Sustainable Biomaterials (RSB).[14] 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.

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.[15] There are

[8] 2023. "40 CFR Parts 80 and 1090– Renewable Fuel Standard (RFS) Program: Standards for 2023–2025 and Other Changes." *EPA*
[9] 2020. "Reporting Co-Processing and Renewable Gasoline Emissions Under MRR." *California Air Resources Board*
[10] 2023. "Oregon Clean Fuels Program." *Oregon Department of Environmental Quality*
[11] 2022. "Clean Fuel Regulations: Quantification Method for Co-Processing in Refineries." *Environment and Climate Change Canada*
[12] 2025. "Low Carbon Fuel Regulation: Co-Processing Methodology" *British Columbia Ministry of Energy and Climate Solutions*
[13] 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." *European Commission*
[14] 2023. "RSB Standard for Advanced Fuels." *Roundtable on Sustainable Biomaterials (RSB)*
[15] 2021. "Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis." *ASTM International (D6866-21)*

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.[16]

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 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 re-measured 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

[16] 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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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.[17]

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 ^{14}C, Method C uses Liquid Scintillation Counting (LSC). In Method B, the AMS Instrument directly measures the ^{14}C isotopes. However, in Method C, scintillation molecules indirectly absorb the beta molecules that release 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.[18] 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:

[17] 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.
[18] 2022. "Testing the methods for determination of radiocarbon content in liquid fuels in the Gliwice Radiocarbon and Mass Spectrometry Laboratory." *Radiocarbon*

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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)

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.

Areas where cross-contamination might occur include but are not limited to; biomedical or nuclear reactors, isotope enrichment / depletion columns, water, soil, plant, or air samples collected near or at biomedical / nuclear reactor sites, medical, industrial, or hazardous waste sites, samples specifically manipulated to study the uptake / fractionation of stable isotopes due to biological or metabolic processes. 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.

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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 14C 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 14C 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 14C. Accepted requirements are:

(1) disclosure to clients that the laboratory working with their products and materials also works with artificial 14C
(2) chemical laboratories in separate buildings for the handling of artificial 14C 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.

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"... 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 14C should not be allowed in a radiocarbon laboratory." "Despite vigorous recent efforts to clean up the room, the "blanks" we measured had 14C 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 14C 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 14C 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."