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ISO/IEC 17025:2017-Accredited Testing Laboratory

Australia Department of Climate Change, Energy, the Environment and Water (DCCEEW) Low Carbon Liquid Fuels (LCLF) Consultation | Stakeholder Input

This comment is intended to recommend that the sustainability criteria for any program created to promote Australia's low carbon liquid fuel industry require the use of the carbon-14 testing method to determine the biogenic content of any enrolled fuels. Biogenic content measurements following standards such as ASTM D6866 Method B currently provide critical value to prominent existing low carbon fuel programs and standards around the world.

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Recommendations for Australia's Low Carbon Liquid Fuels (LCLF) Consultation

Our recommendation is that the sustainability criteria for any program created to promote Australia's low carbon liquid fuel industry should require biogenic content testing results obtained using ASTM D6866 Method B or equivalent standards for any low carbon fuels seeking recognition of renewable content. This requirement is particularly essential for any fuels produced by co-processing, for which estimating biogenic content can be especially difficult.

This comment is specifically meant to address the consultation question, "What additional or alternative criteria would you want to see form part of the criteria?"

One scientific standard which should be required as evidence for any fuels claiming incentives for lower carbon based on their renewable content is the ASTM D6866 Method B standard. This standard for direct biogenic content testing is necessary for any such program because it provides an accurate, reproducible measurement of the renewable content following a scientifically rigorous methodology.



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This comment will discuss important best practices for implementing biogenic testing requirements based on the experiences of similar prominent low carbon fuel programs and standards.

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 the carbon-14 standard 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). 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.

Any program to promote low carbon liquid fuels in Australia 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 <u>US RFS</u> currently requires routine direct testing following ASTM D6866 for fuels produced from co-processing, municipal solid waste (MSW) biogas & renewable natural gas (RNG).²
- <u>California's LCFS</u> 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
- <u>Canada's CFR</u> requires routine direct testing for any fuels produced from co-processing and their co-products.⁵
- <u>The EU's RED</u> requires routine direct testing for any fuels produced from co-processing or biogas & renewable natural gas (RNG).⁶

Requiring routine direct testing is particularly important for any low carbon liquid fuels program because the alternatives likely to be proposed by industry proponents would rely on calculation-based methodologies which cannot provide the necessary consistency or transparency, specifically because they cannot offer direct measurement. Calculation-based approaches such as mass balance allow

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



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producers to assume that all of their biomass inputs end up in their facilities' outputs, despite 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. By basing their calculations solely on production inputs rather than outputs the method systematically over-reports the biobased share of products.

Calculation-based approaches also allow producers to 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 the case because biogenic content can go in different amounts to different products in the co-process. As a result, systems such as mass balance allow producers 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). This allows producers to intentionally claim unfounded renewable content in the products which can maximize their incentives without providing the decarbonization benefits those incentives are meant to promote. The free allocation system also exposes programs to the risk of producers double-counting their renewable content.

Routine biogenic testing requirements are particularly important for any fuels produced by co-processing. Co-processing is currently among the most commonly used production methods for many types of biofuels, including renewable diesel. Co-processing allows refineries to produce renewable fuels by mixing traditional fossil fuels with feedstocks such as animal fats and used cooking oils. It is critical that any co-processed fuels be required to submit biogenic content testing because the renewable content of these fuels are particularly difficult to estimate using calculation-based methods such as mass balance calculations.

The case of biogenic test requirements in the EU RED is a particularly relevant example to understand the best practices for quantification of renewable content under programs supporting the low carbon fuels industry. Initially the EU RED allowed regulated entities to choose whether to submit direct test results or calculations such as mass balance. However, in July 2023 the program faced challenges stemming from a case of mass balance fraud affecting biodiesel submissions from China certified by the ISCC. This case of fraud in the program, "caused a dramatic fall in biodiesel prices in European markets." The response from the ISCC pointed to the need for certification systems to continuously adapt to deliver on their credibility and stay in front of fraudulent practices.

As a result, the EU quickly updated the program's reporting requirements to require routine direct biogenic testing to verify any calculations used for fuels produced from co-processing or biogas.

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

⁸ 2024. "The Mass Balance Approach." International Sustainability & Carbon Certification

⁹ 2023. "ISCC Press Release July 27, 2023." International Sustainability & Carbon Certification



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Regulated entities producing low carbon fuels under the RED can still opt to use calculation-based methods such as mass balance, but now they are required to test at least quarterly to verify that those calculations align with reality. We recommend reviewing the RED's <u>updated method</u> for calculating the share of renewables in the case of co-processing which was released in June 2023 as an excellent example of a rule which allows operators to use a variety of calculation-based options by integrating routine testing requirements to ensure accuracy and combat fraudulent behaviors.¹⁰

The need to consistently require direct testing to verify any calculations is further emphasized by similar concerns raised about the transparency of ISCC mass balance certifications in comments made by the Nova Institute. Since the goal of incentivizing the low carbon liquid fuels industry is to achieve emissions reductions compared to fossil fuels, it is critical that biogenic content is accurately measured to ensure the program only incentivizes real decarbonization.

This consultation is an important first step to creating the proper regulations for Australia's low carbon fuels industry. Introducing regulations with the necessary verification protocols in place can help spur the growth of the low carbon liquid fuels industry in Australia. Even further, by implementing best practices for verification established by similar fuel decarbonization programs, Australia can prepare to successfully achieve and measure the carbon intensity reduction goals of this program. Routine direct testing following ASTM D6866 Method B is the most effective way to incentivize and validate biogenic content under any program created to promote this industry.

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 the European standard ISO 21644. ASTM D6866 is an international standard developed for measuring the biobased carbon content of solid, liquid, and gaseous samples using radiocarbon dating.¹³ There are also many specific international standards based on the use of direct

¹⁰ 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." European Commission

^{11 2014. &}quot;Can ISCC Plus Certification Be Misleading– If the Bio-based Share is Not Labeled Too?" Renewable Carbon News

¹² 2023. "ISCC Press Release July 27, 2023." International Sustainability & Carbon Certification

¹³ 2021. "Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis." ASTM International (D6866-21)



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Carbon-14 testing, such as ISO 21644, which is a European standard developed for measuring the biogenic carbon content of waste derived fuels as a fraction of total carbon content.¹⁴

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 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

^{14 2021. &}quot;ISO 21644:2021 Solid recovered fuels: Methods for the determination of biomass content." International Standardization Organization



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Content to account for determinate and indeterminate factors. 15

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 with the decay of ¹⁴C and convert the energy into photons which are measured proportionally to the amount of ¹⁴C in the sample. Since Method B directly measures the ¹⁴C isotopes and Method C measures them indirectly, Method B is significantly more precise and should be prioritized in regulations. ¹⁶ 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)

¹⁵2021. 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.

¹⁶2022. "Testing the methods for determination of radiocarbon content in liquid fuels in the Gliwice Radiocarbon and Mass Spectrometry Laboratory." *Radiocarbon*



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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.

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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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-reg ulations/clean-fuel-regulations/compliance/quantification-methodco-processing-refineries.html

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. "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. "ISCC Press Release July 27, 2023." *International Sustainability & Carbon Certification* https://www.iscc-system.org/news/press-release-27-july-2023/

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2024. "The Mass Balance Approach." *International Sustainability & Carbon Certification* https://www.iscc-system.org/certification/chain-of-custody/mass-balance/

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)

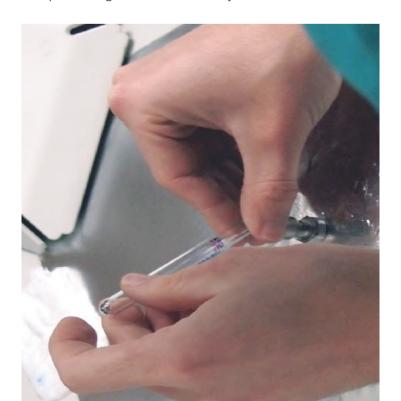
As a natural level radiocarbon laboratory, we highly recommend that researchers require the AMS lab processing their samples to be Tracer-free.

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.



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 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."

