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ISO/IEC 17025:2017-Accredited Testing Laboratory

US Federal Aviation Administration (FAA)
Inflation Reduction Act (IRA) FAST-SAF Grant Program

This comment specifically regards the use of the carbon-14 testing method to determine the share of biogenic carbon content in sustainable aviation fuels (SAF) produced by co-processing. Biogenic content measurements under methods such as ASTM D6866 and BS EN 16640 are currently used to quantify the biogenic carbon content in a wide variety of biofuels.

Included here you will find:

About Beta Analytic

Beta Analytic was among the originators of the ASTM D6866 biobased / biogenic testing standard using carbon-14 to distinguish renewable carbon sources from petroleum sources in solids, liquids, and gasses. Renewable testing started in 2003 at the request of United States Department of Agriculture (USDA) representatives who were interested in Beta's Carbon-14 capabilities for their USDA BioPreferred® Program (www.biopreferred.gov). Carbon-14 third party testing is now standardized in a wide range of international standards including ASTM D6866, CEN 16137, EN 16640, ISO 16620, ISO 19984, BS EN ISO 21644:2021, ISO 13833 and EN 16785. Beta Analytic continues involvement in ASTM D6866 revisions with the current president, Ron Hatfield, serving as technical advisor and committee member to both the ASTM D20.96 and USDA BioPreferred Programs.

Carbon-14 standardized testing 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.

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Recommendations for the FAST-SAF Grant Program

Our recommendation is that the FAST-SAF grant program includes biogenic content testing requirements following the ASTM D6866 standard for any SAFs produced by co-processing.

Co-processing is a widely used method which is currently among the most common paths for producing many biofuels, including renewable diesel. While HEFA is currently the method used to produce most SAF available on the market today, many co-processing facilities designed for renewable diesel production are currently shifting to SAF. Co-processing facilities currently producing renewable diesel require very little modification to produce SAF.[1] Even further, the US EPA's recently published Renewable Fuel Standard volumes for 2023-2025 have been widely criticized by the US biofuels industry for planning for volumes far below the true production capacity for renewable diesel specifically.[2] That pressure, combined with the quick growing demand for SAFs led by new and proposed regulations around the world, will likely lead to even more renewable diesel co-processing facilities making the transition to SAF production.

It is critical that co-processed fuels be required to submit biogenic content testing because these fuels are particularly difficult to estimate using mass balance calculations. Biogenic content testing requirements would be in line with other similar regulations' treatment of co-processed fuels, including those which allow mass balance calculations for other fuels. For example co-processed SAF is required to submit biogenic content testing for the US RFS, California LCFS, Oregon CFS, Canada CFR and the EU RED II, though for most of these programs SAF is not yet a mandatory obligated product, but can apply for tax credits.[3] It is common best practice for programs such as those listed to mandate third-party testing for all analysis they require in order to guarantee accurate, properly obtained results.

International certification programs which specialize in SAF, such as the Roundtable on Sustainable Biomaterials (RSB)'s guide for compliance with the International Civil Aviation Organization (ICAO)'s Carbon Offsetting and Reduction Scheme for International Aviation (CORSIA) program, require carbon-14 testing following ASTM D6866 or an equivalent standard for biogenic content measurements as well.

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

[1] 2022. "The Role of Co-Processing in Aviation's Transition to a Low-Carbon Future" *Air bp*
[2] 2023. "Biofuels Groups Disappointed with EPA RFS Final Rule" *AgWeb*
[3] 2010. "40 CFR Part 80 Subpart M– Renewable Fuel Standard." *National Archives Code of Federal Regulations*
2018. "Directive 2018/2001 of the European Parliament and the Council." *Official Journal of the European Union*
2020. "Reporting Co-Processing and Renewable Gasoline Emissions Under MRR." *California Air Resources Board*
2022. "Quantification Method for Co-Processing in Refineries." *Government of Canada*

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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. This result is measured using an Accelerator Mass Spectrometer (AMS) instrument.

The analysis is based on standards such as ASTM D6866, ISO 13833 and EN 16640. ASTM D6866 is an international standard developed for measuring the biobased carbon content of solid, liquid, and gaseous samples using radiocarbon dating. ISO 16620 is an international standard that measures the biobased carbon content of plastic products, polymers, and additives.

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/

It is very important that testing be required to follow ASTM D6866 Method B in particular. ASTM D6866 Method B uses Accelerated Mass Spectrometry (AMS), while Method C uses Liquid Scintillation Counting (LSC). In Method B, the AMS machine 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

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regulations.[4] LSC calculations, 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.

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 renewable vs. petroleum feedstocks. When carbon-14 test results are challenged, samples can be rapidly remeasured to verify the original reported values (unlike mass balance).

Also of significant importance is that carbon-14 measurements are strictly third party generated under ISO/IEC 17025:2017 Testing Accreditation with no contribution from the submitter, client, or manufacturer.

Most international standards do not cite error limitations, however, the ASTM-D6866 method B standard says that, “Instrumental error can be within 0.1-0.5 % (1 relative standard deviation (RSD), but controlled studies identify an inter-laboratory total uncertainty up to +/- 3 % (absolute). This error is exclusive of indeterminate sources of error in the origin of the biobased content.”[5] This has been applied across all industries and establishes a high degree of variability in indeterminate errors likely to exist between different manufacturing processes. This approximation is well understood as are any errors associated with the measurement.

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.

[4] 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.
[5] 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.

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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

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2018. “Directive 2018/2001 of the European Parliament and the Council.” *Official Journal of the European Union* https://eur-lex.europa.eu/legal-content/EN/TXT/HTML/?uri=CELEX:32018L2001

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.

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2022. “The Role of Co-Processing in Aviation’s Transition to a Low-Carbon Future” *Air bp* https://www.bp.com/en/global/air-bp/news-and-views/views/the-role-of-co-processing-in-aviation-s-transition-to-a-low-carb.html

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2023. “Biofuels Groups Disappointed with EPA RFS Final Rule” *AgWeb* https://www.agweb.com/news/policy/politics/biofuels-groups-disappointed-epas-rfs-final-rule-volumes-dont-match-current

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)

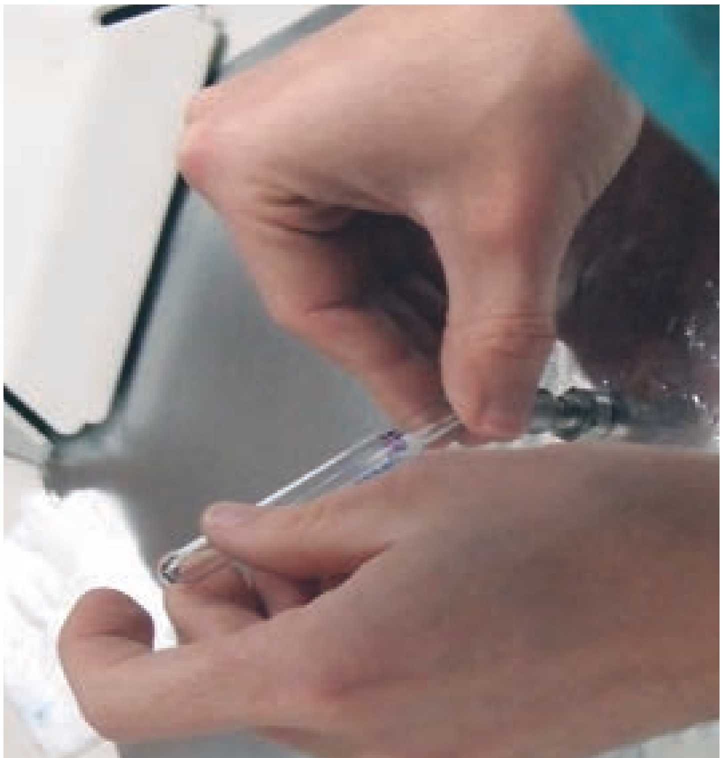
As a natural level radiocarbon laboratory, we highly recommend that researchers require the AMS lab processing their samples to be Tracer-free.

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."

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