Beta Analytic
TESTING LABORATORY

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These comments are submitted by Beta Analytic with regards to LCFS Application No. B0394. The application is for co-processing soybean oil in a "VGO" unit at Chevron's El Segundo refinery.

Beta believes it is critical for the LCFS program to require Carbon-14 testing following the ASTM D6866 Method B standard to ensure accurate measurements of biogenic content are reported by regulated entities. This application does not mention the standard ASTM D6866 anywhere throughout its discussion of compliance with Carbon-14 testing, despite the requirement for co-processed fuels to test to D6866 under the LCFS. Aside from the benefit of reliable results under the standard, it also ensures consistency across the board for anyone pursuing the Tier 2 Pathway.

While in the operating conditions section of the staff report the application recognizes that, "Third-party laboratory 14C analysis or ASTM-approved methodology is required," throughout the section they exclusively use the term "ASTM-approved methodology," without naming D6866. Even further, they repeatedly say "an independent lab or ASTM-approved methodology," suggesting that they see the two as separate, and potentially see independent testing as an alternative to following the D6866 standard.

Similarly in section 8.1 of the LCA report (pg 15) the application states that, "C-14 testing will be used to measure the renewable content of finished, co-processed products." However, testing to the D6866 standard is not mentioned, while it is named in this section in other applications for co-processing.

Further, this section concludes that, "to capture renewable fuel volumes, the approach used will be consistent with the protocol established to generate RIN volumes for EPA's Renewable Fuel Standard." While the EPA's RFS requires ASTM D6866 as well, the program allows producers of co-processed fuels with renewable contents higher than 10% to use ASTM D6866 Method C, which uses a Liquid Scintillation Counting (LSC) technique instead of the standard Accelerated Mass Spectrometry (AMS) machine and is recognized as far less accurate and precise.

In Method B, the AMS machine directly measures the ^{14}C isotopes (see below section ASTM Method B- The Most Reliable Method for more details). 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.[1] LSC calculations, like those used in Method C, are commonly used as an internal testing tool when samples are limited and accuracy / precision does not need to be extremely high.

[1]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

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In these comments Beta raises concerns that this application provides no indication that this pathway intends to follow the required ASTM D6866 standard, which is already in use by many other companies which have opted to follow this pathway. Therefore we recommend CARB ask this application to provide further information regarding its specific plans for conducting biogenic content testing using C-14.

Given the importance of third-party laboratories for proper C-14 testing we also recommend that CARB require evidence that laboratories involved in the program are tracer-free facilities and ISO/IEC 17025:2017 Testing Accredited for regulatory purposes. Please find information below expanding on the recommendations discussed, namely ASTM D6866 Method B and tracer-free facilities.

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 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."[2] 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.

[2]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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Tracer-Free Facilities for Accurate Testing

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.

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.

Beta Analytic serves as 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)

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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