



**California Air Resources Board (CARB)
Proposed Amendments to the Regulation on Methane Emissions from Municipal Solid Waste
Landfills | Public Consultation**

This comment recommends the use of the Carbon-14 testing method to determine the biogenic carbon content of landfill methane and subsequent fuels under the proposed Regulation on Methane Emissions from Municipal Solid Waste. Biogenic content measurements following methods such as ASTM D6866 Method B currently provide critical value to existing clean fuel standards and emissions reduction programs regulating landfill gas emissions, direct combustion and fuel production.

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Recommendations for the Regulation on Methane Emissions from MSW Landfills

Our recommendation is that the Regulation on Methane Emissions from MSW Landfills should require routine biogenic content testing requirements following the ASTM D6866 Method B standard (or similar equivalent standards) for any landfill methane emitted, directly combusted or upgraded to biomethane fuels. Routine direct biogenic testing requirements are the only reliable method of incentivizing the use of landfill gases and emissions reduction targets. Routine biogenic testing requirements currently play a critical role in prominent programs with similar goals.

In Section 95471 on Test Methods and Procedures, 95471(j) on Open Flares states that “the analysis shall identify any unusual changes (outside the range of each well typical historical month-to-month variation) in gas flow rate, gas composition and gauge pressure and determine the reasons for any changes.” Based on this requirement, routine biogenic content measurements should be required as a tool to reveal if any



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biogeochemical reactions are responsible for changes in gas composition as opposed to physical piping aspects of the landfill flares. This is important to consider because renewable emissions provide a net neutral impact, and are not penalized under the California Cap-and-Trade and the EPA Greenhouse Gas Reporting Program (GHGRP).

Our recommendation is to define the renewable portion of methane emissions from MSW landfills as the biogenic carbon content measured by ASTM D6866 test results. Routine direct testing is the only way to accurately determine the biogenic portion of waste and landfill gas and is an important tool for regulators to incentivize only the renewable portion of subsequent fuels and emissions. Testing requirements currently play an important role in prominent clean fuel standard and greenhouse gas reporting programs regulating landfill gas, MSW and subsequent fuels and emissions.

Two internationally prominent programs with quarterly biogenic testing requirements for biogas/RNG fuels produced from landfill gas, and for biofuels produced from MSW include (please see specific rules hyperlinked):

- The US RFS currently [requires](#) quarterly routine direct testing following ASTM D6866 for fuels produced from co-processing, municipal solid waste (MSW), [biogas and renewable natural gas \(RNG\)](#).¹
- The EU's RED [requires](#) quarterly routine direct testing for any fuels produced from co-processing or biogas and renewable natural gas (RNG).²

Biogenic testing also plays a critical role in emissions reporting regulations that address direct combustion of landfill gas and waste-to-energy facilities because direct measurement is the only way to quantify the renewable portion of these emissions, and therefore the reductions provided by the facility. For reference, current requirements of quarterly biogenic testing for MSW combustion under similar prominent programs include (please see specific rules hyperlinked):

- The US Greenhouse Gas Reporting Program (GHGRP) currently [requires](#) quarterly routine direct testing following ASTM D6866 for biogenic emissions from co-firing and municipal solid waste (MSW) combustion.³
- California's Cap-and-Trade Program [requires](#) quarterly routine direct testing following ASTM D6866 for biogenic emissions from co-firing and MSW combustion.⁴
- Canada's Greenhouse Gas Reporting Program (GHGRP) [requires](#) quarterly routine direct testing following ASTM D6866 for biogenic emissions from any renewable or biogenic fuels derived from

¹ 2023. "40 CFR Parts 80 and 1090—Renewable Fuel Standard (RFS) Program: Standards for 2023–2025 and Other Changes." EPA

² 2023. "Renewable energy- method for calculating the share of renewables in the case of co-processing." European Commission

³ 2016. "40 CFR Part 98 Subpart C— General Stationary Fuel Combustion Sources." National Archives Code of Federal Regulations

⁴ 2016. "40 CFR Part 98 Subpart C— General Stationary Fuel Combustion Sources." California Air Resources Board



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biomass including landfill gas and biogas, as well as for any fuels or fuel mixtures containing an unknown biogenic component.⁵

- The EU's Emissions Trading System (ETS) [requires](#) quarterly routine direct testing for biogenic portions of obligated materials, fuels and emissions.⁶

Another important methodology in the waste sector that we recommend the DCCEEW team review is the UNFCCC ACM0022 protocol on "Alternative waste treatment processes" (pages 41-42). This protocol requires biogenic testing following ASTM D6866 to determine the biogenic and fossil fractions of MSW for any cases where the biogenic content of waste cannot be classified or is unknown, which is critical to quantifying the sustainability of landfill gas combustion.⁷

Radiocarbon testing can also play a role in separating carbon sources within the system and, when combined with total carbon content, can help determine the rate of atmospheric carbon removal and establish a baseline scenario for GHG removals. Furthermore, a study by Kerfoot and all., Methane gas in landfills also gives insight into the Carbon-14 testing application to identify the source of hydrocarbons, especially in cases of carbon leakage.⁸ It is important to consider the risks of leakage during CO₂ transportation. The study aimed to evaluate the environmental tracers at a site where soil gas source and migration evaluation is complicated by the presence of multiple methane sources and a complex geological setting.

The study collected data in the studied areas using gas probes. Since the decomposition of organic matter produces methane and carbon dioxide, the relative concentrations and stable and radiogenic isotope composition can help track methane migration and detect changes in the gas composition and the source of methane. The study found dramatic differences in gas composition only in the transect. The study concluded that 37% of the methane measured was sourced from the landfill biogas, highlighting how dramatically the radiocarbon value of methane can vary at a single site for both concentration and the biogenic portion.⁹ This study highlights the need for constant monitoring, especially for projects close to oil/gas pipelines, and/or other sources of contamination, and during the transportation of CO₂ to detect potential leaks and to accurately determine the biogenic portion of carbon dioxide removed from the atmosphere.

In conclusion we recommend that CARB use direct biogenic testing to determine the eligible organic carbon content of landfill-generated methane under this regulation. Only direct measurements can

⁵ 2022. "Canada's Greenhouse Gas Quantification Requirements." *Environment and Climate Change Canada*

⁶ 2022. "Biomass issues in the EU ETS." *European Commission*

⁷ 2022. "Large-Scale Consolidated Methodology: Alternative Waste Treatment Version 3.0" *UNFCCC*

⁸ 2013. Kerfoot et al, "Evaluation of the age of landfill gas methane in landfill gas-natural gas mixtures using co-occurring constituents," *Environ. Sci Processes*, 15, 1153-1161

⁹ 2013. Kerfoot et al, "Evaluation of the age of landfill gas methane in landfill gas-natural gas mixtures using co-occurring constituents," *Environ. Sci Processes*



enable the agency to incentivize the renewable portion of landfill-generated methane, without incentivizing their fossil methane emissions. Including biogenic testing following ASTM D6866 as one of the test methods required to monitor potential changes in gas composition would enable CARB to more effectively regulate methane emissions from MSW landfills.

What is Biogenic Testing (Carbon-14)?

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

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

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

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

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



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

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

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

It is also important that the program should always require ASTM D6866 Method B, rather than allow Method C for any use. Where ASTM D6866 Method B uses the AMS Instrument to measure ¹⁴C, Method C uses Liquid Scintillation Counting (LSC). In Method B, the AMS Instrument directly measures the ¹⁴C isotopes. However, in Method C, scintillation molecules indirectly absorb the beta molecules that release 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.¹³ LSC measurements, like those used in Method C, are commonly used as an internal testing tool when samples are limited and accuracy does not need to be extremely high.

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

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



About Beta Analytic

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

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

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

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

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



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activity measurements including biobased analysis of consumer products and fuels, and for radiocarbon dating.

Required tracer-free facility for Carbon-14

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

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

References

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https://ww2.arb.ca.gov/sites/default/files/classic/cc/reporting/ghg-rep/regulation/subpart_c_rule_part98.pdf

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<https://cdm.unfccc.int/UserManagement/FileStorage/61X9DPNL7G4Y5JMB3CKFHUSREO2QV/>

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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

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<https://www.govinfo.gov/content/pkg/FR-2023-07-12/pdf/2023-13462.pdf>

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<https://www.iscc-system.org/news/press-release-27-july-2023/>

Demand a Tracer-Free Laboratory for Radiocarbon Dating

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

High Risk of Cross-Contamination

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

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

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

Avoid the Risks

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

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

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

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

Tracer-Free Lab Required

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

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

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

No Exposure to Artificial Carbon-14

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

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

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



Useful Reference

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

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

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

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

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



Beta Analytic

www.radiocarbon.com