Designation: D6866 – 24

Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis[1]

This standard is issued under the fixed designation D6866; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This standard is a test method that teaches how to experimentally measure biobased carbon content of solids, liquids, and gaseous samples using radiocarbon analysis. These test methods do not address environmental impact, product performance and functionality, determination of geographical origin, or assignment of required amounts of biobased carbon necessary for compliance with federal laws.

1.2 These test methods are applicable to any product containing carbon-based components that can be combusted in the presence of oxygen to produce carbon dioxide (CO_2) gas. The overall analytical method is also applicable to gaseous samples, including flue gases from electrical utility boilers and waste incinerators.

1.3 These test methods make no attempt to teach the basic principles of the instrumentation used although minimum requirements for instrument selection are referenced in the References section. However, the preparation of samples for the above test methods is described. No details of instrument operation are included here. These are best obtained from the manufacturer of the specific instrument in use.

1.4 *Limitation*—This standard is applicable to laboratories working without exposure to artificial carbon-14 (^{14}C). Artificial ^{14}C is routinely used in biomedical studies by both liquid scintillation counter (LSC) and accelerator mass spectrometry (AMS) laboratories and 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 door knobs, pens, desk tops, 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(s) 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. **(1, 2, and 3)**[2]

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—ISO 16620-2 is equivalent to this standard.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

***A Summary of Changes section appears at the end of this standard**

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D883 Terminology Relating to Plastics

2.2 *Other Standards:*[4]

CEN/TS 16640:2014 Biobased Products—Determination of the biobased carbon content of products using the radiocarbon method

CEN/TS 16137:2011 Plastics—Determination of biobased carbon content

ISO 16620-2:2015 Plastics—Biobased content—Part 2: Determination of biobased carbon content

EN 15440:2011 Solid recovered fuels—Methods for the determination of biomass content

ISO 13833:2013 Stationary source emissions—Determination of the ratio of biomass (biogenic) and fossil-derived carbon dioxide—Radiocarbon sampling and determination

3. Terminology

3.1 The definitions of terms used in these test methods are referenced in order that the practitioner may require further information regarding the practice of the art of isotope analysis and to facilitate performance of these test methods.

3.2 Terminology D883 should be referenced for terminology relating to plastics. Although an attempt to list terms in a logical manner (alphabetically) will be made as some terms require definition of other terms to make sense.

3.3 *Definitions:*

3.3.1 *AMS facility*—a facility performing Accelerator Mass Spectrometry.

3.3.2 *accelerator mass spectrometry (AMS)*—an ultra-sensitive technique that can be used for measuring naturally occurring radio nuclides, in which sample atoms are ionized, accelerated to high energies, separated on basis of momentum, charge, and mass, and individually counted in Faraday collectors. This high energy separation is extremely effective in filtering out isobaric interferences, such that AMS may be used to measure accurately the ^{14}C / ^{12}C abundance to a level of 1 in 10^{15}. At these levels, uncertainties are based on counting statistics through the Poisson distribution (**4**,**5**).

3.3.3 *automated efficiency control (AEC)*—a method used by scintillation counters to compensate for the effect of quenching on the sample spectrum (**6**).

3.3.4 *background radiation*—the radiation in the natural environment; including cosmic radiation and radionuclides present in the local environment, for example, materials of construction, metals, glass, concrete (**7**,**8**,**9**,**4**,**6-14**).

3.3.5 *biobased*—containing organic carbon of renewable origin like agricultural, plant, animal, fungi, microorganisms, marine, or forestry materials living in a natural environment in equilibrium with the atmosphere.

3.3.6 *biogenic*—containing carbon (organic and inorganic) of renewable origin like agricultural, plant, animal, fungi, microorganisms, macroorganisms, marine, or forestry materials.

3.3.7 *biobased carbon content*—the amount of biobased carbon in the material or product as a percent of the total organic carbon (TOC) in the product.

3.3.8 *biogenic carbon content*—the amount of biogenic carbon in the material or product as a percent of the total carbon (TC) in the product.

3.3.9 *biobased carbon content on mass basis*—amount of biobased carbon in the material or product as a percent of the total mass of product.

3.3.10 *biogenic carbon content on mass basis*—amount of biogenic carbon in the material or product as a percent of the total mass of product.

3.3.11 *break seal tube*—the sample tube within which the sample, copper oxide, and silver wire is placed.

3.3.12 *coincidence circuit*—a portion of the electronic analysis system of an LSC which acts to reject pulses which are not received from the two Photomultiplier Tubes (that count the photons) within a given period of time and are necessary to rule out background interference and required for any LSC used in these test methods (**9**, **6**, **12**).

3.3.13 *coincidence threshold*—the minimum decay energy required for an LSC to detect a radioactive event. The ability to set that threshold is a requirement of any LSC used in these test methods (**6**, **12**).

3.3.14 *contemporary carbon*—a direct indication of the relative contributions of fossil carbon and "living" biospheric carbon can be expressed as the fraction (or percentage) of contemporary carbon, symbol f_C. This is derived from "fraction of modern" (f_M) through the use of the observed input function for atmospheric ^{14}C over recent decades, representing the combined effects of fossil dilution of ^{14}C (minor) and nuclear testing enhancement (major). The relation between f_C and f_M is necessarily a function of time. By 1985, when the particulate sampling discussed in the cited reference was performed, the f_M ratio had decreased to approximately 1.2 (**4**, **5**).

3.3.15 *chemical quenching*—a reduction in the scintillation intensity (a significant interference with these test methods) seen by the Photomultiplier Tubes (PMT, pmt) due to the materials present in the scintillation solution that interfere with the processes leading to the production of light. The result is fewer photons counted and a lower efficiency (**8**, **9**, **12**).

3.3.16 *chi-square test*—a statistical tool used in radioactive counting in order to compare the observed variations in repeat counts of a radioactive sample with the variation predicted by statistical theory. This determines whether two different distributions of photon measurements originate from the same photonic events. LSC instruments used in this measurement should include this capability (**6**, **12**, **15**).

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.3.17 *cocktail*—the solution in which samples are placed for measurement in an LSC. Solvents and Scintillators—chemicals that absorb decay energy transferred from the solvent and emits light (photons) proportional in intensity to the deposited energy (**8**, **9**, **6**, **12**).

3.3.18 *decay (radioactive)*—the spontaneous transformation of one nuclide into a different nuclide or into a different energy state of the same nuclide. The process results in a decrease, with time, of the number of original radioactive atoms in a sample, according to the half-life of the radionuclide (**4**, **6**, **12**).

3.3.19 *discriminator*—an electronic circuit which distinguishes signal pulses according to their pulse height or energy; used to exclude extraneous radiation, background radiation, and extraneous noise from the desired signal (**6**, **12**, **13**, **16**).

3.3.20 *dpm*—disintegrations per minute. This is the quantity of radioactivity. The measure dpm is derived from cpm or counts per minute (dpm = cpm − bkgd / counting efficiency). There are 2.2×10^6 dpm / μCi (**6**, **12**).

3.3.21 *dps*—disintegrations per second (rather than minute as above) (**6**, **12**).

3.3.22 *efficiency*—the ratio of measured observations or counts compared to the number of decay events which occurred during the measurement time; expressed as a percentage (**6**, **12**).

3.3.23 *external standard*—a radioactive source placed adjacent to the liquid sample to produce scintillations in the sample for the purpose of monitoring the sample's level of quenching (**6**, **12**).

3.3.24 *figure of merit*—a term applied to a numerical value used to characterize the performance of a system. In liquid scintillation counting, specific formulas have been derived for quantitatively comparing certain aspects of instrument and cocktail performance and the term is frequently used to compare efficiency and background measures (**6**, **12**, **17**).

3.3.25 *flexible tube cracker*—the apparatus in which the sample tube (Break Seal Tube) is placed (**18**, **19**, **20**, **21**).

3.3.26 *fluorescence*—the emission of light resulting from the absorption of incident radiation and persisting only as long as the stimulation radiation is continued (**6**, **12**, **22**).

3.3.27 *fossil carbon*—carbon that contains essentially no radiocarbon because its age is very much greater than the 5,730 year half-life of ^{14}C (**4**, **5**).

3.3.28 *half-life*—the time in which one half the atoms of a particular radioactive substance disintegrate to another nuclear form. The half-life of ^{14}C is 5,730 years (**4**, **6**, **22**).

3.3.29 *intensity*—the amount of energy, the number of photons, or the numbers of particles of any radiation incident upon a unit area per unit time (**6**, **12**).

3.3.30 *internal standard*—a known amount of radioactivity which is added to a sample in order to determine the counting efficiency of that sample. The radionuclide used must be the same as that in the sample to be measured, the cocktail should be the same as the sample, and the Internal Standard must be of certified activity (**6**, **12**).

3.3.31 *modern carbon*—explicitly, 0.95 times the specific activity of SRM 4990B (the original oxalic acid radiocarbon standard), normalized to $\delta^{13}C = -19$ ‰ (Currie, et al., 1989). Functionally, the fraction of modern carbon equals 0.95 times the concentration of ^{14}C contemporaneous with 1950 wood (that is, pre-atmospheric nuclear testing). To correct for the post 1950 bomb ^{14}C injection into the atmosphere (**5**), the fraction of modern carbon is multiplied by a correction factor representative of the excess ^{14}C in the atmosphere at the time of measurements.

3.3.32 *noise pulse*—a spurious signal arising from the electronics and electrical supply of the instrument (**6**, **12**, **23**, **24**).

3.3.33 *phase contact*—the degree of contact between two phases of heterogeneous samples. In liquid scintillation counting, better phase contact usually means higher counting efficiency (**6**, **12**).

3.3.34 *photomultiplier tube (PMT, pmt)*—the device in the LSC that counts the photons of light simultaneously at two separate detectors (**24**, **16**).

3.3.35 *pulse*—the electrical signal resulting when photons are detected by the PMTs (**6**, **12**, **13**, **16**).

3.3.36 *pulse height analyzer (PHA)*—an electronic circuit which sorts and records pulses according to height or voltage (**6**, **12**, **13**, **16**).

3.3.37 *pulse index*—the number of after-pulses following a detected coincidence pulse (used in three dimensional or pulse height discrimination) to compensate for the background of an LSC performing (**6**, **13**, **24**, **16**).

3.3.38 *quenching*—any material that interferes with the accurate conversion of decay energy to photons captured by the PMT of the LSC (**7**, **8**, **9**, **6**, **10**, **12**, **17**).

3.3.39 *region*—regions of interest, also called window and/or channel in regard to LSC. Refers to an energy level or subset specific to a particular isotope (**8**, **6**, **13**, **23**, **24**).

3.3.40 *renewable*—being readily replaced and of non-fossil origin; specifically not of petroleum origin.

3.3.41 *scintillation*—the sum of all photons produced by a radioactive decay event. Counters used to measure this as described in these test methods are Liquid Scintillation Counters (LSC) (**6**, **12**).

3.3.42 *scintillation reagent*—chemicals that absorbs decay energy transferred from the solvent and emits light (photons) proportional in intensity to the decay energy (**8**, **6**, **24**).

3.3.43 *solvent-in scintillation reagent*—chemical(s) which act as both a vehicle for dissolving the sample and scintillator and the location of the initial kinetic energy transfer from the decay products to the scintillator; that is, into excitation energy that can be converted by the scintillator into photons (**8**, **6**, **12**, **24**).

3.3.44 *specific activity (SA)*—refers to the quantity of radioactivity per mass unit of product, that is, dpm per gram (**6**, **12**).

3.3.45 *standard count conditions (STDCT)*—LSC conditions under which reference standards and samples are counted.

3.3.46 *three dimensional spectrum analysis*—the analysis of the pulse energy distribution in function of energy, counts per energy, and pulse index. It allows for auto-optimization of a liquid scintillation analyzer allowing maximum performance. Although different manufacturers of LSC instruments call Three Dimensional Analysis by different names, the actual function is a necessary part of these test methods (**6, 12, 13**).

3.3.47 *true beta event*—an actual count which represents atomic decay rather than spurious interference (**20, 21**).

4. Significance and Use

4.1 This testing method provides accurate biobased/biogenic carbon content results to materials whose carbon source was directly in equilibrium with CO_2 in the atmosphere at the time of cessation of respiration or metabolism, such as the harvesting of a crop or grass living its natural life in a field. Special considerations are needed to apply the testing method to materials originating from within artificial environments. Application of these testing methods to materials derived from CO_2 uptake within artificial environments is beyond the present scope of this standard.

4.2 Method B utilizes AMS along with Isotope Ratio Mass Spectrometry (IRMS) techniques to quantify the biobased content of a given product. 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 (see Section 22 on precision and bias).

4.3 Method C uses LSC techniques to quantify the biobased content of a product using sample carbon that has been converted to benzene. This test method determines the biobased content of a sample with a maximum total error of ±3 % (absolute), as does Method B.

4.4 The test methods described here directly discriminate between product carbon resulting from contemporary carbon input and that derived from fossil-based input. A measurement of a product's $^{14}C/^{12}C$ or $^{14}C/^{13}C$ content is determined relative to a carbon based modern reference material accepted by the radiocarbon dating community such as NIST Standard Reference Material (SRM) 4990C, (referred to as OXII or HOxII). It is compositionally related directly to the original oxalic acid radiocarbon standard SRM 4990B (referred to as OXI or HOxI), and is denoted in terms of f_M, that is, the sample's fraction of modern carbon. (See Terminology, Section 3.)

4.5 Reference standards, available to all laboratories practicing these test methods, must be used properly in order that traceability to the primary carbon isotope standards are established, and that stated uncertainties are valid. The primary standards are SRM 4990C (oxalic acid) for ^{14}C and RM 8544 (NBS 19 calcite) for ^{13}C. These materials are available for distribution in North America from the National Institute of Standards and Technology (NIST), and outside North America from the International Atomic Energy Agency (IAEA), Vienna, Austria.

4.6 Acceptable SI unit deviations (tolerance) for the practice of these test methods is ±5 % from the stated instructions unless otherwise noted.

5. Safety

5.1 The specific safety and regulatory requirements associated with radioactivity, sample preparation, and instrument operation are not addressed in these test methods. It is the responsibility of the user of these test methods to establish appropriate safety and health practices. It is also incumbent on the user to conform to all the federal and state regulatory requirements, especially those that relate to the use of open radioactive source, in the performance of these test methods. Although ^{14}C is one of the safest isotopes to work with, State and Federal regulations must be followed in the performance of these test methods.

5.2 The use of glass and metal, in particular with closed systems containing oxygen that are subjected to 700°C temperatures pose their own safety concerns and care should be taken to protect the operators from implosion/explosion of the glass tube. Safety Data Sheets should always be followed with special concern for eye, respiratory, and skin protection. Radioactive ^{14}C compounds should be handled and disposed of in accordance with State and Federal regulations.

NOTE 2—Prior to D6866 - 11, this standard contained a Method A, which utilized LSC and CO_2 absorption into a cocktail vial. Error was cited as ±15 % absolute due to technical challenges and low radiocarbon counts. Empirical evidence now indicates error may be ±20 % or higher in routine use. This method was removed in this revision due to the inapplicability of this low precision method to biobased analysis.

NOTE 3—Prior to D6866-16, this standard contained a CARBONATE OPTION A (CARBONATE SUBTRACTION) procedure, to exclude inorganic carbonate from the biobased result. Empirical evidence now indicates error may be unreasonably high in routine use, especially in products with very low in organic carbon and very high in inorganic carbonate. This method was removed in this revision due to potential low precision results which are not observed in CARBONATE OPTION B (ACID RESIDUE COMBUSTION).

5.3 In Method C, benzene is generated from the sample carbon. Benzene is highly toxic and is an EPA-listed carcinogen. It must be handled accordingly, using all appropriate eye, skin, and respiratory protection. Samples must be handled and disposed of in accordance with State and Federal regulations. Other hazardous chemicals are also used, and must be handled appropriately (see Safety Data Sheets for proper handling procedures).

METHOD B: AMS

6. Apparatus and Reagents

6.1 *AMS and IRMS Apparatus:*

6.1.1 A vacuum manifold system with capabilities for air and non-condensable gas evacuation, sample introduction, water distillation, cryogenic gas transfer, and temperature and pressure monitoring. The following equipment is required:

6.1.2 Manifold tubing that is composed of clean stainless steel and/or glass.

6.1.3 Vacuum pump(s) capable of achieving a vacuum of 101 Pa or less within the vacuum region.

6.1.4 Calibrated pressure transducers with coupled or integrated signal response controllers.

6.1.5 A calibrated sample collection volume with associated temperature readout.

6.1.6 Clean quartz tubing for sample combustion and subsequent gas transfer, quantification and storage.

6.1.7 A hydrogen/oxygen torch or other heating device and/or gas for sealing quartz tubing.

7. AMS and IRMS Reagents

7.1 A stoichiometric excess of oxygen for sample combustion; introduced into sample tube as either a pure gas or as solid copper (II) oxide.

7.2 A stoichiometric excess of silver, nominally 30 mg, introduced into sample tube for the removal of halogenated species.

7.3 A −76°C slurry mixture of dry ice (frozen CO_2) and alcohol distillation and removal of sample water.

7.4 Liquid nitrogen.

8. Sample Preparation

8.1 Method B is a commonly used procedure to quantitatively combust the carbon fraction within product matrices of varying degrees of complexity. The procedure described here for Method B is recommended based on its affordability and extensive worldwide use. Nevertheless, laboratories with alternative instrumentation such as continuous flow interfaces and associated CO_2 trapping capabilities are equally suitable provided that the recovery of CO_2 is quantitative, 100 ± 5 %.

8.2 Based on the stoichiometry of the product material, sufficient sample mass shall be weighed such that 1-10 mg of carbon is quantitatively recovered as CO_2. Weighed sample material shall be contained within a pre-cleaned quartz sample container, furnace-baked at 900°C for ≥2 h, and torch sealed at one end. Typically 2 mm OD/1 mm ID quartz tubing is sufficient, however any tubing configuration needed to accommodate large sample volumes is acceptable.

8.3 The weighed sample shall then be transferred into an appropriately sized quartz tube, typically 6 mm OD/4 mm ID.

8.4 The sample, thus configured shall then be adapted to a vacuum manifold for evacuation of ambient air to a pressure 101 Pa or less.

8.5 If the material is known to be volatile or contains volatile components, the sample material within the tube shall be frozen with liquid nitrogen to –196°C prior to evacuation. The evacuated tube shall be torch sealed then combusted in a temperature controlled furnace at 900°C for 2 to 4 h.

8.6 After combustion, the quartz sample tube shall be scored to facilitate a clean break within a flexible hose portion of a "tube cracker" assembly adapted to the manifold. One example configuration of a tube cracker is shown in Fig. X1.2. The materials are composed of stainless steel. Compression fittings with appropriate welds are used to assemble the individual parts. This and alternative assemblies are given in the References section (**18**, **19**, **20**, **21**).

8.7 With the manifold closed to the vacuum pump, the quartz tubing is cracked, the sample CO_2 is liberated and immediately cryogenically (with liquid nitrogen) transferred to a sample collection bulb attached to a separate port on the manifold.

8.8 The contents of the sample collection bulb shall be distilled to remove residual water using a dry ice/alcohol slurry maintained at approximately −76°C. Simultaneously the sample CO_2 gas is released and immediately condensed in a calibrated volume.

8.9 The calibrated volume is then closed and the CO_2 shall equilibrate to room temperature.

8.10 Recovery shall be determined using the ideal gas law relationship.

8.11 The sample shall be transferred to a borosilicate break seal tube for storage and delivery to an AMS facility for analysis of $^{14}C/^{12}C$ and $^{13}C/^{12}$ C isotopic ratios.

9. Analysis, Interpretation, and Reporting

9.1 $^{14}C/^{12}C$ and $^{13}C/^{12}C$ isotopic ratios are measured using AMS. The isotopic ratios of $^{14}C/^{12}C$ or $^{13}C/^{12}C$ are determined relative to a standard traceable to the NIST SRM 4990C (oxalic acid) modern reference standard. The calculated "fraction of modern" (f_M) represents the amount of ^{14}C in the product or material relative to the modern standard. This is most commonly referred to as percent modern carbon (pMC), the percent equivalent to f_M (for example, f_M 1 = 100 pMC).

9.2 All pMC values obtained from the radiocarbon analyses must be corrected for isotopic fractionation using stable isotope data (**25**). Correction shall be made using $^{13}C/^{12}C$ values determined directly within the AMS where possible. In the absence of this capability (and citable absence of fractionation within the AMS) correction shall be made using the delta 13C ($\delta^{13}C$) measured by IRMS, CRDS (cavity ring down spectroscopy) or other equivalent technology that can provide precision to ±0.3 per mil. Reference standard must be traceable to Vienna Pee Dee Belemite (VPDB) using NIST SRM 8539, 8540, 8541, 8542 or equivalent.

9.3 Zero pMC represents the entire lack of measurable ^{14}C atoms in a material above background signals thus indicating a fossil (for example, petroleum based) carbon source. One hundred pMC indicates an entirely modern carbon source. A pMC value between 0 and 100 indicates a proportion of carbon derived from fossil vs. modern source.

9.4 The correction factor is based on the ^{14}C activity in the atmosphere at the time of testing. The first version of this standard (ASTM D6866-04) in 2004 referenced a value of 107.5 pMC based on measurements of CO2 in the air in a rural area of the Netherlands (Lutjewad, Groningen) and the ASTM D6866-10 version (2010) cited 105 pMC. These data points equated to a decline of 0.5 pMC per year. From the period of 2004 through 2019 the annual decrease in the REF was maintained at the projected 0.5 pMC per year, based on then available data through 2010. From the existing historical data and recently published long term ^{14}C atmospheric data compiled at sites from around the world "*Atmospheric Radiocarbon for The Period 1950—2019 Quan Hua et al.*," it has been

shown that the projected decrease of 0.5 pMC per year slowed to approximately 0.3 pMC per year on average beginning in about 2014. Therefore, on January 2 of each year, the values in Table 1 are used as REF for the years 2024 through 2026 reflecting a 0.3 pMC decrease per year. The REF (pMC) values for 2019 through 2024 were determined to be 100.0, based on continued measurements at The Netherlands (Lutjewad, Groningen) through 2022 and recently published long term ^{14}C atmospheric data compiled at sites from around the world "*Atmospheric Radiocarbon for The Period 1950—2019 Quan Hua et al.*," which is reflective of a slowing in the dilution of excess ^{14}C in the atmosphere. References for reporting carbon isotopic ratio data are given in Refs. (**15**, **26**) for ^{14}C and ^{13}C, respectively.

TABLE 1 Percent Modern Carbon (pMC) Reference

Year	REF (pMC)
2015	102.0
2016	101.5
2017	101.0
2018	100.5
2019	100.0
2020	100.0
2021	100.0
2022	100.0
2023	100.0
2024	99.7
2025	99.4
2026	99.1

9.5 Calculation of % biobased carbon content is made by dividing pMC by REF and multiplying the result by 100. (for example, [102 (pMC) / 102 (REF)] × 100 = 100 % biobased carbon. Results are reported as % biobased carbon content or % biogenic carbon content rounded to the nearest 1 unit with an applied error of 3 % absolute (see 4.2).

9.6 See 22.7 for calculating and reporting results for materials which calculate to greater than 100 % biobased carbon content.

9.7 As stated in 4.1, this testing standard is applicable to materials whose carbon source was directly in equilibrium with CO_2 in the atmosphere at the time of cessation of respiration or metabolism. See 22.11 for calculating and reporting results for materials from marine and aquatic environments.

METHOD C: Liquid Scintillation Counting

10. Detailed Requirements

NOTE 4—Acceptable tolerance levels of ±5 % are standard to this method unless otherwise stated.

10.1 Low level LSCs with active shielding that can produce consistent background counts of less than 5 dpm.

10.2 Anti-coincidence systems such as two and three PMTs (multidetector systems).

10.3 Coincidence circuits.

10.4 Software and hardware that include thresholds and statistics, pulse rise and shape discrimination, and three-dimensional spectrum analysis.

10.5 Use of external and internal standards must be used in LSC operation.

10.6 Optimized counting regions to provide very low background counts while maintaining counting efficiency greater than 60 % of samples 0.7 to 1.5 g in clean, 3-mL, 7-mL or 20-mL low potassium glass counting vials. Alternatively, clean PTFE or quartz counting vials may be used in this method.

10.7 No single LSC is specified for this method. However, minimum counting efficiency and control of background interference is specified. Like all analytical instruments, LSCs require study as to their specific components and counting optimization.

10.8 Standardization of sample preparation is required.

10.9 Standardization and optimization of clean sample vials, which must be made of either PTFE, quartz, or low-potassium glass with PTFE tops. Sample vials may be either 3-mL, 7-mL or 20-mL in volume. Plastic vials must not be used for this method.

10.10 Counting efficiency and background optimization should be performed using a suitable reference standard (for example, NIST SRM-4990B or SRM-4990C oxalic acid) using the same reagents and counting parameters as the samples.

10.11 Counting efficiency (E) shall be determined by dividing the measured cpm by the known dpm, and multiplying this by 100 to obtain the counting efficiency as a percentage. For example, for the Oxalic Acid I standard, E = (cpm/g Oxalic Acid/ 14.27 dpm/g) × 100, where E = counting efficiency in %, cpm/g Oxalic Acid is the net activity per gram measured for the oxalic acid after subtracting background, and 14.27 dpm/g is the absolute value of the NIST "OxI" reference standard. (SRM 4990B). The NIST "OxII" standard (SRM 4990C) has a slightly different ^{14}C activity level. ANU sucrose (NIST SRM 8542) can be used as a suitable standard in place of oxalic acid.

10.12 Counting interference concerns that must be addressed as part of specific instrument calibration and normalization include luminance, chemical or color quench, static electricity, random noise, temperature, and humidity variability (**27**).

10.13 Alternate regions of interest parameters may be used based upon testing of 20, or more, 6-h counts of the same reference (STDCT) standard that record the raw data and spectrum for keV regions of interest 4 through 96. Optimal counting conditions should be established by maximizing the Figure of Merit (E^2/bkg) values to obtain the highest count efficiency and the lowest background and other interference. Counting efficiency of less than 60 % is unacceptable and can be improved by LSC instrument optimization and sample/reagent compatibility or shielding improvements.

10.14 Samples will be equilibrated with reference standards under identical conditions of time and temperature.

10.15 Samples will be counted for a minimum of 10 h with region of interest (ROI) channels including ROI energy levels of 0-155 keV such that E^2/B is 1,000 or higher in 20 to 120-min subsets with raw data saved to disk for later statistical analysis and documentation of stable counting conditions.

10.16 Before commercial testing, laboratories that intend to implement this method must participate in an inter-laboratory comparison study to assess between laboratory reproducibility.