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 ultrasensitive 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.