Designation: D 7026 – 04

Standard Guide for Sampling and Reporting of Results for Determination of Biobased Content of Materials via Carbon Isotope Analysis[1]

This standard is issued under the fixed designation D 7026; 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.

INTRODUCTION

The biobased content of a material and the resources consumed in creation of the material, both energy and raw materials are defined in Guide D 6852. These resources are expressed as carbon equivalent. Biobased carbon represents new or recently fixed carbon, opposed to fossil carbon fixed millions of years ago. Test Methods D 6866 presents two methods for experimentally determining the amount of recently fixed carbon in a sample by means of its radioisotope content, allowing direct determination of its biobased content. The following guide represents a companion document to Test Methods D 6866 and defines the sampling and sample handling procedures for the radioisotope methods for determination of biobased content.

There are a great variety of biobased materials that may be tested using one of the radioisotope methods, with a wide range of physical characteristics and special sampling problems.

It is not the intent of this guide to provide specific sample collection and handling instructions for a specific material. Rather, the guide presents general outlines to be followed in sampling procedures and encourages the use of existing material-specific sampling procedures validated by extensive use in industry. The emphasis in the guide is to provide thorough and transparent reporting that allows subsequent evaluation of the validity of the claims regards biobased content.

1. Scope

1.1 This guide provides a framework for collecting and handling samples for determination of biobased content of materials by means of the carbon isotope method described in Test Methods D 6866. Tests for sampling adequacy based on the standard statistical tools are provided. In addition, reporting of the results, including sampling techniques and handling procedures and chain-of-custody issues are discussed.

1.2 This guide is concerned with collecting representative samples within a given material or a lot, not with lot-to-lot variations such as considered in quality control schemes.

1.3 Biobased materials often represent sampling problems specific to a given material, such as heterogeneity, and so forth, which require employment of material-specific sampling methods. The use of specialized sampling methods already accepted and validated by industries that manufacture and/or use the biomaterial is encouraged. However, all sampling techniques, especially non-standard techniques developed for specific materials must be reported in sufficient detail to allow critical assessment of the techniques used.

1.4 Carbon isotope analysis involves thermal processing in presence of oxidants. Compatibility of any given material with Test Methods D 6866 must be assessed. Special attention must be given to materials with potential for explosion hazards, such as peroxides, nitrated compounds, azides, and so forth. Examples of peroxide-forming compounds are ethers, some ketones and a number of other compounds.

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 and health practices and determine the applicability of regulatory requirements prior to use.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

[1] This guide is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

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[2] 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.

D 6852 Guide for Determination of Biobased Content, Resources Consumption and Environmental Profile of Materials and Products

D 6866 Test Methods for Determining the Biobased Content of Natural Range Materials Using Radiocarbon and Isotope Ratio Mass Spectrometry Analysis

E 105 Practice for Probability Sampling of Materials

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process

2.2 *Other Reference:*

Cramer, H., "Elements of Probability Theory," Wiley & Sons, NY, 1961

3. Terminology

3.1 *Definitions:*

3.1.1 *representative sample*—a sample or subunit of material that shows a composition, within statistical limits, that is the same as would be detected if the whole material would be analyzed as a sample.

3.1.2 *biobased content*—in this guide, designates *biobased carbon content*, which can be converted to biobased content as a fraction of weight, based on the molecular weights of the components in the material.

3.1.3 *biobased carbon content*—carbon in a sample that is of recent origin, as evidenced by its ^{14}C isotope content.

3.1.3.1 *Discussion*—^{14}C decays with a half-life of about 5200 years and thus fossil carbon, whose age since fixation is measured in millions of years, does not contain any ^{14}C.

3.1.4 $\mu(0)$—true biobased content of lot.

3.1.5 $\mu(n)$—lot average biobased content based on analysis of n samples from the lot.

3.1.6 $E = I\mu(0) - \mu(n)I$, abs—maximum tolerable error for the sample average, or maximum acceptable difference between average of samples and true average of the lot, $\mu(0)$

3.1.6.1 *Discussion*—The *I____I* are supposed to designate absolute value.

3.1.7 *n(k)*—number of samples that must be tested to provide assurance that the average of these samples lies within E of the true average with a probability defined by k.

3.1.8 k—factor defining the degree of accuracy desired in estimation of the lot average from the average of n samples.

3.1.9 $\sigma(0)$—estimated or experimentally determined standard deviation of the analytical procedure.

3.1.10 *S.D.*—standard deviation (abbreviation used in text).

4. Significance and Use

4.1 The carbon isotope analysis is designed to be an adjunct to other information in determination of biobased content, specifically the manufacturer's records. It is also a means of verifying the authenticity of a disputed lot of material which may be manufactured by different means, from different raw materials. An example would be ethanol, which can be obtained by means of chemical synthesis or by means of fermentation from biobased raw material. This represents a "yes or no" answer, to which the LSC method is ideally suited.

4.2 Representative sampling and handling methods are clearly a prerequisite to obtaining accurate results form the radiocarbon composition determination and any other quantitative analytical method.

4.3 This guide provides for accurate and complete reporting of the sample collection, handling, chain of custody, sample preparation and treatment that allows any independent party to assess the validity of the reported biobased content of the material.

5. Sample Collection

5.1 This guide is designed for materials that can be classified either as solids or liquids.

5.2 If there is a standard sampling technique for the material to be tested that is widely accepted by the industry, such a procedure may be used and the details of sampling recorded, as called for under Reporting.

5.3 The primary requirements for any sampling strategy are that *(a)* the sample be representative of the material to be tested and that *(b)* the quantity or weight of sample be accurately established.

5.4 The biobased content is to be reported based on dry weight. Moisture content of the sample must be controlled carefully. If feasible, the sample should be dried prior to weighing and the sample subsequently kept in dry, controlled environment, such as a desiccator. If there is a possibility of sample decomposition during drying, the sample may be sealed to retain constant moisture level, the water content determined by an appropriate, accepted analytical method and the dry weight of the sample calculated.

5.5 Test Methods D 6866 presents two methods for determining biobased carbon content: *(1)* LSC or Liquid Scintillation Counting, a relatively widely available method, but less accurate of the two, with presently established maximum error (range) of 15 % (which is expected to be reduced as more data is accumulated), and *(2)* AMS/IRMS or Accelerated Mass Spectrometry, coupled with Isotope Ratio mass Spectrometry, with maximum error of about 1 to 2 %, but which can be performed by only a few laboratories in U.S. Standard deviation (S.D.) values for the two methods will be established as more data becomes available. LSC requires a sample that contains 1.0 to 2.0 g of carbon. AMS/IRMS requires a sample that contains 0.5 to 1.0 g of carbon.

NOTE 2—As an example, carbohydrates such as cellulose, starches, and so forth, contain about 40 % wt carbon.

5.6 Samples should be taken from the most homogenous subunit of an object or material. If there are suspected gradual trends in the sample, the material should be subdivided to a set of smaller units or sub lots that can be considered essentially homogenous, except for possible small-scale graininess in some materials, such as particle board and so forth, and these sub lots treated as independent units or lots.

5.7 The sampling should be performed in accordance with the probability sampling methods described in Practice E 105. The lot should be divided into sample size elements. These elements should be assigned numbers and the samples (elements) collected using random numbers, as described in Practice E 105.