

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

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PROPOSAL FOR INTRODUCTION OF ICC STANDARD No. 185 (AOAC Method 2017.16) TO REPLACE AOAC Method 2009.01

Prepared by ICC

In the spirit of our close collaboration in the past, today ICC would like to introduce ICC Standard No. 185 (AOAC Method 2017.16) and propose a discussion in the agenda of the 41st Session under agenda item 13 “Other Business and Future Work” about a replacement of AOAC Method 2009.01 as currently presented in CXS 234-1999.

Scope and field of application of ICC Standard No. 185

This method is an update of AOAC Method 2009.01 (also published as AACC Method 32-45.01) for the determination of total dietary fiber in foods and food ingredients as defined by the Codex Alimentarius Commission. Enzymatic digestion with pancreatic α -amylase plus amyloglucosidase at 37°C for 4 h is used to simulate human intestinal digestion. Higher molecular weight dietary fiber, including insoluble dietary fiber and fiber which precipitates in the presence of 78% ethanol is measured gravimetrically. Resistant starch is captured in the insoluble dietary fiber fraction. Dietary fiber that is soluble in 78% aqueous ethanol (SDFS) is recovered and measured by high-performance liquid chromatography. Total dietary fiber is the sum of higher molecular weight dietary fiber and SDFS. The method measures the complete range of dietary fiber components from resistant starch to digestion-resistant oligosaccharides (SDFS). Reducing the incubation with pancreatic α -amylase plus amyloglucosidase from 16 h to 4 h makes the assay more physiologically relevant, resulting in more accurate measurement of resistant starch (RS), including RS₂ and RS₄, and complete hydrolysis of non-resistant starch and starch derived oligosaccharides. In this update of AOAC Method 2009.01, sodium azide is removed from buffers; desalting of samples for high-performance liquid chromatography is simplified; accurate measurement of fructo-oligosaccharides and all other oligosaccharides is achieved using TSKgel® G2500PWXL HPLC columns (in place of the Waters Sugar-Pak® column); and glycerol is employed as the internal standard in place of D-sorbitol (which occurs in some food products). Diethylene glycol serves as an alternative internal standard for food samples suspected of containing glycerol (as a humectant).

Main improvements compared to AOAC Method 2009.01

ICC Standard No. 185 (AOAC Method 2017.16) has addressed the various issues identified in the use of 2009.01, such as:

1. Incubation time with pancreatic alpha-amylase/amyloglucosidase has been reduced from 16 h to 4 h (in line with average time of residence of food in the human small intestine)
2. Sodium azide has been removed from the incubation buffer (this is possible with the shorter incubation time)
3. Preparation of low molecular weight dietary fibre components for HPLC has been greatly simplified.
4. Columns used for HPLC have been changed from Sugar-Pak^R to TSK^R gel permeation. This results in correct chromatography and measurement of fructo-triose (which is present in hydrolysed inulin materials, and on Sugar-Pak^R columns chromatographs as a disaccharide and thus is not included in the SDFS value).
5. More accurate measurement of all resistant starch fractions, especially RS3 and RS4.

Relevance to CODEX

This method has been rapidly adopted in Australia and the interest in numerous other countries is rapidly growing. The main feature of this method is that it allows the accurate measurement of ALL dietary fibre

components, both from natural sources and as ingredients. Currently, there are fifteen CODEX methods for the measurement of dietary fiber, six of these measure specific dietary fiber components, while nine are defined as CODEX Type I for more general application. The endorsement of nine Type I methods is confusing in the regulatory, analytical and commercial arenas. An urgent need exists for a single method that accurately and reliably measures all dietary fiber components. ICC Standard 185 (AOAC Method 2017.16) fulfils this need. Significantly, this method was developed by the same research team that developed AOAC Method 2009.01, and this was in response to feedback from the analytical community concerning issues related to AOAC Method 2009.01. This method complements the more traditional and established fibre methods (AOAC Methods 985.29 and 991.43) that measure specific fiber component mixtures.

Information on the relation between the proposal and other existing CODEX documents

The introduced ICC Standard No. 185/AOAC Method 2017.16 shall replace AOAC Method 2009.01 as currently presented in CXS 234-1999.

As required, we can support further discussion with published scientific papers, and hope that this topic can be introduced at the 41st Session of CCFSPU in Dusseldorf, Germany, 24-29 November 2019 under agenda item 13 "Other Business and Future Work".

Attachments as reference:

1. [McCleary, B. V., Sloane, N. and Draga, A. 2015. *Determination of total dietary fibre and available carbohydrates: A rapid integrated procedure that simulates in vivo digestion*. **Starch/Starke**, **67**:860-883.](#)
2. [McCleary, B. V. and Cox, J. \(2017\) *Evolution of a definition for dietary fiber and methodology to service this definition*. **Luminacoids Research \(Japan\)**, **21**, 9-21.](#)
3. [ICC Draft Standard 185, \(2017\) *Measurement of total dietary fibre in cereals, ingredients and food products using the rapid integrated TDF procedure \(RINTDF\)*. **International Association for Cereal Science and Technology**. Approved April, 2107 \(final approval 2018\).](#)
4. [McCleary, B. V., Cox, J., Ivory, R. and Delaney, E. 2018. *Definition and analysis of dietary fiber in grain products*. In **Grain-based Functional Foods: Carbohydrate and Phytochemical Components \(Food Chemistry, Function and Analysis\)** \(T. Beta & M. E. Camire Eds\). **Royal Society of Chemistry**. Chapter 6, pp. 103-126.](#)
5. [McCleary, B. V. 2018. *Total Dietary Fiber \(CODEX Definition\) in Foods and Food Ingredients by a Rapid Enzymatic-Gravimetric Method and Liquid Chromatography: Collaborative Study, First Action 2017.16*. **J. AOAC International**, **102**:196-207.](#)