

C O D E X A L I M E N T A R I U S C O M M I S S I O N



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda item 2

MAS/41 CRD/12

Proposed change to CXS 234-1999
to replace AOAC 2009.01/AACCI 32-45.01
with ICC Standard No. 185/AOAC 2017.16

Comments from IACST(International Association of Cereal Science and Technology)

Accurate RS Measurement is an issue across all the highlighted methods

Methods of analysis for dietary fibre: Guidelines for Use of Nutrition and Health Claims: Table of Conditions for Claims

Standard	Provisions	Method	Principle	Type
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units <=9) ⁽²⁾				
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. (4)	AOAC 985.29 AACC Intl 32-05.01	Enzymatic gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4)	AOAC 991.43 AACC Intl 32-07.01 NMKL 129	Enzymatic gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction, in foods and food products containing more than 10% dietary fibres and less than 2% starch (e.g. fruits) (4)	AOAC 993.21	Gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of Klason lignin (4).	AOAC 994.13 AACC Intl 32- 25.01 NMKL 162	Enzymatic GC/ colorimetry gravimetry	Type I
All foods (1)	Insoluble dietary fibres in food and food products (4)	AOAC 991.42 (Specific for insoluble fibre) AACC Intl 32-20.01	Enzymatic gravimetry	Type I
All foods (1)	Soluble dietary fibres in food and food products (4)	AOAC 993.19 (Specific for soluble fibre)	Enzymatic gravimetry	Type I
General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units <=9) ⁽²⁾				
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight, in food where resistant starches are not present	AOAC 2001.03 AACC Intl 32-41.01	Enzymatic gravimetry and Liquid chromatography	Type I
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches.	AOAC 2009.01 AACC Intl 32-45.01	Enzymatic-Gravimetry High Pressure Liquid Chromatography	Type I
All foods (1)	Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches	AACC Intl 32-50.01 AOAC 2011.25	Enzymatic-Gravimetry High Pressure Liquid Chromatography	Type I

Not commonly used in industry

Note: The Prosky method (AOAC 985.29) and Lee Method (AOAC 991.43) were traditionally employed as methods to measure total dietary fibre but these do not measure SDFS or RS accurately. The Matsutani Method (AOAC 2001.03) allows measurement of SDFS but does not measure RS accurately. It is now known that AOAC 2009.01 also provides inaccurate RS measurement for some samples.

Background: Why is change from AOAC 2009.01 required?

AOAC 2009.01 was developed to provide a method for the measurement of total dietary fibre as defined by CODEX in 2009 (See Appendix 1), to include RS and the option for NDO with DP < 10.

The method was published in 2007 and over the following 8 years a number of limitations were identified:

- ❖ **Incorrect measurement (**overestimation**) of SDFS in highly starchy foods**
Resulting in certain cereal products being claimable as a source of dietary fiber. A problem that needed to be resolved.
- ❖ Incorrect measurement (**underestimation**) of resistant starch (RS₄ and to a lesser extent RS₂)
- ❖ Incorrect measurement (**underestimation**) of FOS (fructo-oligosaccharides, specifically fructotriose)

Comparison of DF Analytical Methods to Physiological Conditions

Parameter	Comparative Incubation Conditions		
	Small intestine	AOAC Method 2009.01 AACC Method 32-45.01	AOAC Method 2017.16 ICC Standard 185
Time	~ 4 h transit time (human body movement)	16 h incubation time (stirring or shaking)	4 h incubation time (stirring or shaking)
pH	6 to 7.5 (in jejunum)	6.0	6.0
Temperature	37°C	37°C	37°C
Digestive enzymes A	Pancreatic α -amylase	Pancreatic α -amylase	Pancreatic α -amylase
B	Mucosal α -glucosidases (maltase-glucoamylase plus sucrase-isoamylase) (to hydrolyze and 'remove' maltose)	Amyloglucosidase (microbial) (Glucoamylase) (to hydrolyze and 'remove' maltose)	Amyloglucosidase (microbial) (Glucoamylase) (to hydrolyze and 'remove' maltose)

Recovery of Polysaccharides and Oligosaccharides; A comparison of AOAC Methods 2009.01 and 2017.16

Oligosaccharides (SDFS)	Original Sample	AOAC 2009.01	AOAC 2017.16
Galacto-oligosaccharides (GOS)	76.0	70.6	72.0
Polydextrose [®]	84.3	85.1	82.5
Fibersol 2 [®]	88.5	83.4	82.4
Fructo-oligosaccharides (Raftilose P95)*	89.0	76.2*	88.2
AdvantaFibre [®] (Isomalto-oligosaccharides)	65.4	29.0	10.8

* The FOS analyzed had a high content of inulinotriose. Analyzed using Sugar Pak column.

Polysaccharides	Original Sample	AOAC 2009.01	AOAC 2017.16
Beta-glucan	~ 100	96.0	96.2
Wheat arabinoxylan	~ 100	94.5	96.0
Citrus pectin	~ 90	87.0	84.5
Hylon VII (High amylose maize starch) (RS ₂)	~ 100	46.5	58.8
Fibersym (Phosphate cross-linked starch (RS ₄))	~ 100	29.7	60.2

Conclusion

In accordance with the best principles of CODEX to adopt analytical methods that reflect the most up-to-date scientific knowledge, it is recommended that **AOAC 2009.01/AACCI 32-45.01** is replaced with **AOAC 2017.16/ICC Standard 185** as a Type 1 analytical method for the measurement of total dietary fibre.

Further Information:

Enter the following into your internet search engine: "MAS-CRD/03"



A one-stop shop - CX234

Codex recommended methods of analysis are particularly important for checking compliance with provisions in Codex standards. "It is important to keep them updated, relevant and easily accessible - this is what we intend to achieve with our one-stop shop on methods", said Verna Carolissen, Food Standards Officer in the Codex Secretariat. "This could only have been possible with the support and commitment of Members and SDOs and with our new guidance to support this work, consensus should become easier", she said.

Measurement of Dietary Fiber – Which Method to Use

Samples Not Containing RS Analytical Data Required	AOAC Method to use	Megazyme Test Kit
IDF + SDFP together (HMWDF) (Prosky Fiber)	985.29	K- TDFR
IDF and SDFP separately (Lee Fiber)	991.43	K-TDFR
IDF, SDFP and SDFS separately (Lee + Matsutani)	991.43 + 2001.03	K-TDFR
Unknown Samples and Samples Containing RS Analytical Data Required	AOAC Method to use	Megazyme Test Kit
Total Dietary Fiber (HMWDF + SDFS)	2017.16	K- RINTDF
IDF, SDFP and SDFS separately (allowing separate measurement of soluble & insoluble DF)	2017.16*	K-RINTDF

*Modified filtration and alcohol precipitation according to AOAC 2011.25 to allow for soluble/insoluble DF determination

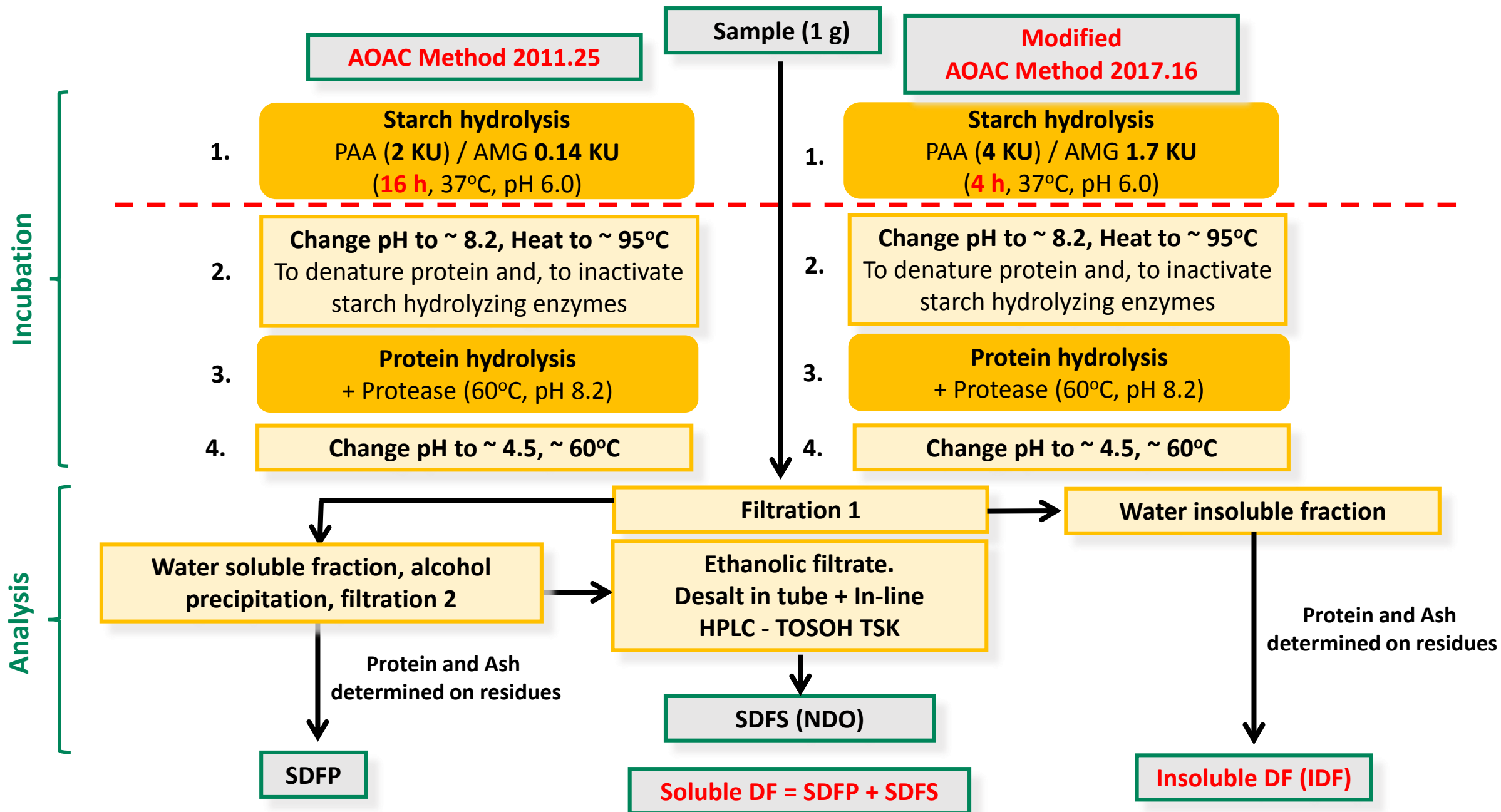
HMWDF: High MW dietary fiber = IDF + SDFP.

IDF: Water insoluble dietary fiber (e.g. cellulose).

SDFP: Water soluble fiber which precipitates in 78 % ethanol (e.g. β -glucan).

SDFS: Water soluble fiber that remains soluble in 78% ethanol = NDO.

Modification of AOAC 2017.16 to allow Separate Measurement of Insoluble & Soluble DF



Acronyms used in this presentation and their meanings:

Acronym	Definition	Comments/Examples
IDF	insoluble dietary fibre	DF insoluble in water, eg. cellulose
SDFP	soluble dietary fibre that precipitates in the presence of 78% v/v aqueous ethanol	eg. β -glucan, galactomannan, arabinoxylan
SDFS	soluble dietary fibre that is soluble in 78% v/v aqueous ethanol	eg. FOS, GOS, Polydextrose (part thereof), Fibersol 2 (part thereof)
TDF	total dietary fibre	IDF + SDFP + SDFS
HMWDF	high molecular weight dietary fibre	IDF + SDFP
RS	resistant starch	Starch types include: RS ₁ (physically inaccessible), RS ₂ (granular), RS ₃ (retrograded), RS ₄ (chemically modified)
NDO	non-digestible oligosaccharides	Equivalent to SDFS