

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD
HEALTH
ORGANIZATION



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Agenda Item 5

CX/MAS 10/31/5

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Thirty-first Session

Budapest, Hungary, 8 - 12 March 2010

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS¹

This document contains the methods of analysis proposed by the following Committees in draft standards and related texts under elaboration or as update of current methods:

- Codex Committee on Fish and Fishery Products
- Codex Committee on Nutrition and Foods for Special Dietary Uses

A. CODEX COMMITTEE ON FISH AND FISHERY PRODUCTS

Draft Standard for Sturgeon Caviar (At Step 8) (ALINORM 10/33/18, Appendix V)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Sturgeon Caviar	Salt content ($\geq 3\text{g}/100\text{g}$ and $\leq 5\text{g}/100\text{g}$ in the end product)	As in CODEX STAN 167-1989 (see below)	Titrimetry	I

1. Principle

The salt is extracted by water from the preweighed sample. After the precipitation of the proteins, the chloride concentration is determined by titration of an aliquot of the solution with a standardized silver nitrate solution (Mohr method) and calculated as sodium chloride.

2. Equipment and chemicals

- Brush
- Sharp knife or saw
- Balance, accurate to 0.01 g
- Calibrated volumetric flasks, 250 ml
- Erlenmeyer flasks
- Electric homogenizer
- Magnetic stirrer
- Folded paper filter, quick running
- Pipettes

¹ Methods of Analysis for Milk and Milk Products will be presented in CX/MAS 10/31/5-Add.1

- Funnel
- Burette
- Potassium hexacyano ferrate (II), $K_4Fe(CN)_6 \cdot 3H_2O$, 15% w/v (aq)
- Zinc sulphate, $ZnSO_4 \cdot 6H_2O$, 30% w/v (aq)
- Sodium hydroxide, NaOH, 0.1 N, 0.41% w/v (aq)
- Silver nitrate, $AgNO_3$, 0.1 N, 1.6987% w/v (aq), standardized
- Potassium chromate, K_2CrO_4 5% w/v (aq)
- Phenolphthalein, 1% in ethanol
- distilled or deionized water

3. Procedure

- (i) Five gram of homogenized subsample is weighted into a 250 ml volumetric flask and vigorously shaken with approximately 100 ml water.
- (ii) Five millilitre of potassium hexacyano-ferrate solution and 5 ml of zinc sulphate solution are added, the flask is shaken.
- (iii) Water is added to the graduation mark.
- (iv) After shaking again and allowing to stand for precipitation, the flask content is filtered through a folded paper filter.
- (v) An aliquot of the clear filtrate is transferred into an Erlenmeyer flask and two drops of phenolphthalein are added. Sodium hydroxide is added dropwise until the aliquot takes on a faint red colour. The aliquot then diluted with water to approximately 100 ml.
- (vi) After addition of approximately 1 ml potassium chromate solution, the diluted aliquot is titrated under constant stirring, with silver nitrate solution. Endpoint is indicated by a faint, but distinct, change in colour. This faint reddish-brown colour should persist after brisk shaking.
To recognize the colour change, it is advisable to carry out the titration against a white background.
- (vii) Blank titration of reagents used should be done.
- (viii) Endpoint determination can also be made by using instruments like potentiometer or colorimeter.

4. Calculation of results

In the equation of the calculation of results the following symbols are used:

A= volume of aliquot (ml)

C= concentration of silver nitrate solution in N

V= volume of silver nitrate solution in ml used to reach endpoint and corrected for blank value

W= sample weight (g)

The salt content in the sample is calculated by using the equation:

$$\text{Salt concentration (\%)} = (V \times C \times 58.45 \times 250 \times 100) / (A \times W \times 1000)$$

Results should be reported with one figure after the decimal point.

B. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (ALINORM 10/33/26, APPENDIX II)

Methods of analysis for dietary fibre: Guidelines for Use of Nutrition and Health Claims: Table of Conditions for Claims (reproduced below)

Commodity	Provisions	Method	Principle	Type proposed
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units ≤ 9)⁽²⁾				
All foods ⁽¹⁾	Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, lignin, and plant cell wall. ⁽⁴⁾	AOAC 985.29	Enzymatic gravimetric	III
All foods ⁽¹⁾	Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, lignin, and plant cell wall. ⁽⁴⁾	AOAC 991.43	Enzymatic gravimetric	III
All foods ⁽¹⁾	Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, lignin, and plant cell wall. ⁽⁴⁾	AOAC 992.16	Enzymatic gravimetric	III
All foods ⁽¹⁾	Dietary fibre in food and food products with less than 2% starch. ⁽⁴⁾	AOAC 993.21	Non-enzymatic gravimetric	III
All foods ⁽¹⁾	Dietary fibre based on precipitation in 4 parts alcohol and 1 part water, quantitated as component neutral sugars, uronic acids, plus Klason lignin. ⁽⁴⁾	AOAC 994.13	Enzymatic chemical	III
General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units ≤ 9)⁽²⁾				
All foods ⁽¹⁾	Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, resistant malto-dextrins, lignin, and plant cell wall. ⁽³⁾	AOAC 2001.03	Enzymatic gravimetric and Liquid chromatography	III
All foods ⁽¹⁾	Dietary fibre (Soluble + insoluble polysaccharides + lignin + resistant starch + oligosaccharides).	AOAC 2009.01	Enzymatic-Gravimetric-High Pressure Liquid Chromatography Method	III

Methods that measure individual specific components (monomeric units: the whole range for each type of components is covered)⁽²⁾				
All foods ⁽¹⁾	Insoluble dietary fibres in food and food products	AOAC 991.42	Enzymatic gravimetric	III
All foods ⁽¹⁾	(1→3)(1→4) <i>Beta</i> -D-Glucans	AOAC 992.28	Enzymatic	III
All foods ⁽¹⁾	Soluble dietary fibres in food and food products	AOAC 993.19	Enzymatic gravimetric	III
All foods ⁽¹⁾	(1→3)(1→4) <i>Beta</i> -D-Glucans	AOAC 995.16	Enzymatic	III
All foods ⁽¹⁾	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides)	AOAC 997.08	Enzymatic & HPAEC-PAD	III
All foods ⁽¹⁾	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides)	AOAC 999.03	Enzymatic & colorimetric	III
All foods ⁽¹⁾	Polydextrose	AOAC 2000.11	HPAEC-PAD	III
All foods ⁽¹⁾	Trans-galacto-oligo saccharides	AOAC 2001.02	HPAEC-PAD	III
All foods ⁽¹⁾	Resistant starch (Recommended for RS2 & RS3)	AOAC 2002.02	Enzymatic	III
Other methods⁽²⁾				
All foods	Insoluble glucans and mannans of yeast cell wall (for yeast cell wall only)	Eurasyp (European association for specialty yeast product) – LM Bonanno. Biospringer- 2004 – online version : http://www.eurasyp.org/public.technique.home.screen .	Chemical & HPAEC-PAD	IV
All foods	Fructo-oligosaccharides (monomeric units<5)	Ouarné et al. 1999 in <i>Complex Carbohydrates in Foods</i> . Edited by S. Sungsoo, L. Prosky & M. Dreher. Marcel Dekker Inc, New York	HPAEC-PAD	IV

All foods	Non-starch polysaccharides (NSP) ⁽³⁾	Englyst H.N, Quigley M.E., Hudson G. (1994) Determination of dietary fibre as non-starch polysaccharides with gas-liquid chromatographic high performance liquid chromatographic or spectrophotometric measurement of constituent sugars – Analyst 119, 1497-1509	Enzymatic Gas-Liquid Chromatography Method	IV
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⁽¹⁾ Users should consult the description of each method for the food matrices that were the subject of interlaboratory study in the Official methods of Analysis of AOAC International.

⁽²⁾ Two issues are left for national authorities: to include monomeric units 3-9 and which isolated or synthetic compounds have physiological benefit.(Refer to GL 2-1985)

⁽³⁾ Quantitation lost for resistant starch. Refer to specific methods.

⁽⁴⁾ Quantitation lost for inulin, resistant starch, polydextrose and resistant maltodextrins. Refer to specific methods.

**GUIDELINES FOR USE OF NUTRITION AND HEALTH CLAIMS:
TABLE OF CONDITIONS FOR NUTRIENT CONTENTS (PART B) DIETARY FIBRE**

Component	Claim	Conditions: not less than
B.		
Dietary Fibre	Source	3 g per 100 g* or 1.5 g per 100 kcal or 10 % of daily reference value per serving**
	High	6 g per 100 g* or 3 g per 100 kcal or 20 % of daily reference value per serving**

* Conditions for nutrient content claims for dietary fibre in liquid foods to be determined at national level.

** Serving size and daily reference value to be determined at national level.

Definition:

Dietary fibre means carbohydrate polymers² with ten or more monomeric units³, which are not hydrolysed by the endogenous enzymes in the small intestine of humans and belong to the following categories:

- Edible carbohydrate polymers naturally occurring in the food as consumed,
- carbohydrate polymers, which have been obtained from food raw material by physical, enzymatic or chemical means and which have been shown to have a physiological effect of benefit to health as demonstrated by generally accepted scientific evidence to competent authorities,
- synthetic carbohydrate polymers which have been shown to have a physiological effect of benefit to health as demonstrated by generally accepted scientific evidence to competent authorities

² When derived from a plant origin, dietary fibre may include fractions of lignin and/or other compounds when associated with polysaccharides in the plant cell walls and if these compounds are quantified by the AOAC gravimetric analytical method for dietary fibre analysis : Fractions of lignin and the other compounds (proteic fractions, phenolic compounds, waxes, saponins, phytates, cutin, phytosterols, etc.) intimately "associated" with plant polysaccharides are often extracted with the polysaccharides in the AOAC 991.43 method. These substances are included in the definition of fibre insofar as they are actually associated with the poly- or oligo-saccharidic fraction of fibre. However, when extracted or even re-introduced into a food containing non digestible polysaccharides, they cannot be defined as dietary fibre. When combined with polysacchrides, these associated substances may provide additional beneficial effects (pending adoption of Section on Methods of Analysis and Sampling).

³ Decision on whether to include carbohydrates from 3 to 9 monomeric units should be left to national authorities.