35th Joint Arab Codex Contact Points and CCNE Coordination Meeting

PREPARATION FOR THE 44th SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

(CCMAS44)

Eng. Narjes Mhajbi, Tunisia

April 20, 2025

Agenda Item 4

Matters pending from CCMAS43

4.1 - Methods of analysis for protein in quinoa (Comments in reply to CL 2024/91–MAS) - *CX/MAS 25/44/4*

4.2- Determination of moisture content in whey powder - *CX/MAS 25/44/5*

4.1. METHODS OF ANALYSIS FOR PROTEIN IN QUINOA

(Comments in reply to CL 2024/91-MAS)

submitted by

Argentina, Bolivia, Egypt, EU, Indonesia, Peru, Saudi Arabia

BACKGROUND

- CCMAS43 endorsed ISO 1871 for the determination of protein in quinoa **as a Type IV method.**
- CCMAS43 noted that the **typing could be reconsidered** if more information were provided.
- Seven (7) countries had provided validation data for ISO 1871 for determination of protein in quinoa.

- Additional information were required for re (typing). Therefore, Codex Members and Observers were invited to submit information on:

  - ❖ the specific chemicals used for the catalysts;
  - ❖ the different reagents and their concentrations; and
  - ❖ conditions of the method corresponding to the validation data provided.

METHODS OF ANALYSIS FOR PROTEIN IN QUINOA

Method ISO 1871
Determination of Nitrogen by the Kjeldahl Method

ISO 1871 gives **general guidance** for the determination of protein according to the Kjeldahl principles and **does not offer validation data**.

- **Option 1**: Elaboration of a standard for determination of protein by the principles described in ISO 1871
- **Option 2**: The scope of ISO 20483 Cereals and pulses — Determination of the nitrogen content and calculation of the crude protein content — Kjeldahl method could be extended to quinoa.
- **Option 3**: Describing the validated method for the determination of protein in quinoa in an Annex to CXS 234

4.2. DISCUSSION PAPER
DETERMINATION OF MOISTURE CONTENT IN WHEY POWDER

(Prepared by New Zealand with assistance from Australia, Brazil, EU, Uruguay and IDF)

CXS 234-1999 58

Milk and milk products

Commodity	*Provisions*	*Method*	*Principle*	*Type*
Whey powders	Ash	ISO 5545 | IDF 90	Gravimetry (ashing at 825 M °C)	IV
Whey powders	Lactose	ISO 5765-1/2 | IDF 79-1/2	Enzymatic method: Part 1– Glucose moiety or Part 2– Galactose moiety	II
Whey powders	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Whey powders	Milk protein	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Whey powders	Water[xlii] (moisture)	ISO 5537 | IDF 26	Gravimetry (drying at 87 °C)	I

INTERNATIONAL STANDARD **ISO 5537:2004(E)**
IDF 26:2004(E)

Dried milk — Determination of moisture content (Reference method)

Proposal to endorse the 102°C method as Type IV for whey powders, and with explanatory notes on use conditions as footnotes in CXS 234-1999.

Note "Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix III is listed as Type IV".

DISCUSSION PAPER

DETERMINATION OF MOISTURE CONTENT IN WHEY POWDER

- There was **no consensus reached**, and two proposals are tabled for consideration by CCMAS44, depending on CCMAS44's decision on whether the data provided is adequate to support the endorsement.

RECOMMENDATION

CCMAS44 is invited to:

i. consider the proposal presented in paragraph 13 based on the information provided in Appendix I Table 1: Key points for each proposal; and

ii. decide whether the data provided is adequate to support the endorsement.

REMINDER!

Description of Method Typing from Procedural Manual

Methods of Analysis

Definition of types of methods of analysis

(a) Defining Methods (Type I)

Definition: A method which determines a value that can only be arrived at in terms of the method per se and serves by definition as the only method for establishing the accepted value of the item measured.

Examples: Howard Mould Count, Reichert-Meissl value, loss on drying, salt in brine by density.

b) Reference Methods (Type II)

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Example: Potentiometric method for halides.

(c) Alternative Approved Methods (Type III)

Definition: A Type III Method is one which meets the criteria required by the Committee on Methods of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

Example: Volhard Method or Mohr Method for chlorides

(d) Tentative Method (Type IV)

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined.

Examples: chlorine by X-ray fluorescence, estimation of synthetic colours in foods.

KEY POINTS OF DISCUSSION

Supporting Endorsement	Not Supporting Endorsement
Use of the 102 °C method for national and control purposes	The current multi-laboratory trial data for the 102°C method is inadequate for drawing definitive conclusions
Coexisting Type I and Type IV methods is requested only as an exceptional case	Outlier exclusion criteria!
Outlier exclusion criteria!	Comparison with the ISO5537 | IDF 26 method is required due to the potential bias of the 102°C method for whey powders
	The 102 °C method has issues when testing products containing lactose at levels greater than those found in whole milk and skim milk powders: • Temperatures greater than 90°C cause color changes and the lactose starts to lose its water of crystallization.
	Issues of coexistence of Type I and Type IV methods

Comparative studies of both methods?


thank you
danke
謝謝
ngiyabonga
рахмат
teşekkür ederim
Баярлалаа
спасибо
faafetai lava
vinaka
спасибі
blagodaram
dank je
misaotra
matondo
paldies
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mahalo
tapadh leat
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shukriya
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감사합니다
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ευχαριστώ
merci
شكرا
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