CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 3.1 **CX/MAS 26/45/3-Add.1**

March 2026

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Forty-fifth Session

9-13 March 2026

Budapest, Hungary

ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS – METHODS SUBMITTED BY CCFO29

1. This document contains the methods of analysis proposed by the 29th session of the Codex Committee on Fats and Oils (CCFO) (REP26/FO)[1].

2. Appendix I of this document contains the method of analysis for the determination of gamma oryzanol in crude rice bran oil transcribed in the *Standard for named vegetable oils* (CXS 210-1999). The method exists in CXS 210-1999 as Type IV but CCFO29 requested CCMAS to retype the method as Type III when endorsing the method for inclusion in the *Recommended methods of analysis and sampling* (CXS 234-1999).

3. Appendix II of this document contains two parts:

   - Part 1: Methods of analysis for provisions (other than for the determination of moisture and volatile matter) in the proposed draft standard for microbial omega-3 oils; and
   - Part 2: Methods of analysis for the determination of moisture and volatile matter in the proposed draft standard for microbial omega-3 oils

4. CCFO29 noted that two type I methods had been proposed for the determination of moisture and volatile matter in the proposed draft standard for microbial omega-3 oils, and requested CCMAS to review the method typing.

5. CCMAS45 is invited to:

   i. **endorse** the method of analysis contained in Appendix I for inclusion in CXS 234-1999;

   ii. **endorse** the methods of analysis contained in Appendix II (Part 1) for inclusion in CXS 234-1999;

   iii. **review** the method typing for the methods of analysis in Appendix II (Part 2).

[1] REP26/FO paragraphs 18(i), 75 and 82(ii), Appendix II

APPENDIX I

Method of analysis for the determination of gamma oryzanol in crude rice bran oil (for endorsement)

Fats and oils				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Crude rice bran oil	Gamma oryzanol	See Appendix **	Absorption in ultraviolet	III

Appendix ** of CXS 234-1999

DETERMINATION OF GAMMA ORYZANOL CONTENT IN CRUDE RICE BRAN OIL

Definition

This method is used to determine gamma oryzanol content (percentage) in oils from spectrophotometer absorption measurements at the wavelength of maximum absorption near 315 nm.

Scope

Applicable to crude rice bran oil.

Apparatus

- Spectrophotometer – for measuring extinction in the ultraviolet between 310 nm and 320 nm
- Rectangular quartz cuvettes – having an optical light path of 1 cm
- Volumetric flask – 25 ml
- Filter paper – Whatman No. 2, or equivalent

Reagents

- n-Heptane – spectrophotometrically pure.

Procedure

(i) Before using, the spectrophotometer should be properly adjusted to a zero-reading filling both the sample cuvette and the reference cuvette with n-Heptane.

(ii) Filter the oil sample through filter paper at ambient temperature.

(iii) Weigh accurately approximately 0.02 g of the sample so prepared into a 25 ml volumetric flask, make up to the mark with n-Heptane.

(iv) Fill a cuvette with the solution obtained and measure the extinction at the wavelength of maximum absorption near 315 m, using the same solvent as a reference.

(v) The extinction values recorded must lie within the range 0.3–0.6. If not, the measurements must be repeated using more concentrated or more diluted solutions as appropriate.

Calculation

Calculate gamma oryzanol content as follows:

$$\text{Gamma oryzanol content, \%} = 25 \times (1 / W) \times A \times (1 / E)$$

Where: W = mass of sample, g

A = extinction (absorbance) of the solution

E = specific extinction $E^{1\%}_{1\ cm} = 359$

APPENDIX II

Part 1: Methods of analysis for provisions in the proposed draft standard for microbial omega-3 oils (except for moisture and volatile matter) (for endorsement)

Fats and oils				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Microbial omega-3 oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Preparation of FAME* and determination by GC-FID	III
Microbial omega-3 oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1i-07	Preparation of FAME* and determination by GC-FID	II
Microbial omega-3 oils	EPA and DHA	Ph.Eur. 2.4.29 / USP 401	GC-FID	II
Microbial omega-3 oils	EPA and DHA	AOCS Ce 1i-07	GC-FID	III
Microbial omega-3 oils	Peroxide Value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158 / European Pharmacopoeia 2.5.5	Titrimetry (colorimetric)	I
Microbial omega-3 oils	Anisidine Value	European Pharmacopoeia 2.5.36 / AOCS Cd 18-90/ ISO 6885	Spectrophotometry	I
Microbial omega-3 oils	Acid Value	AOCS Ca 5a-40 / AOCS Cd 3d-63 / ISO 660 / NMKL 38 / USP 401, Method 1	Titrimetry	I
Microbial omega-3 oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry and Titrimetry	I
Microbial omega-3 oils	Moisture	ISO 8534	Titrimetry	II
Microbial omega-3 oils	Moisture	AOCS Ca 2e-84	Titrimetry	II

*FAME = Fatty Acid Methyl Esters

Part 2: Methods of analysis for the determination of moisture and volatile matter in the proposed draft standard for microbial omega-3 oils (for review of method typing and endorsement)

Fats and oils				
Commodity	**Provision**	**Method**	**Principle**	**Type**
Microbial omega-3 oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Microbial omega-3 oils	Moisture and volatile matter	AOCS Ca 2c-25	Gravimetry	I