



JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

40th Session

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ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX
STANDARDS

1. This document contains the methods of analysis (Appendix I, II and III) proposed by the following Committees:

- Committee on Nutrition and Foods for Special Dietary Uses (methods of analysis for Infant Formula and Formulas for Special Medical Purposes Intended for Infants);
- Committee on Spices and Culinary Herbs (methods of analysis for various spices and culinary herbs);
and
- Committee on Fats and Oils (methods of analysis for name vegetable oils)

CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU40)

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CXS 72-1981)¹

2. The Committee agreed to submit the methods for vitamin K, folic acid and nine minerals and trace elements to CCMAS for review and endorsement and inclusion in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) and request CCMAS to re-type or revoke the related existing methods in CXS 234-1999.

3. The Committee **is invited to endorse** the methods of analysis and consider the retyping or revocation of existing methods in Appendix I.

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH4)

Methods of analysis for provisions in the proposed draft standards for dried or dehydrated garlic, dried oregano, dried roots, rhizomes and bulbs – dried or dehydrated ginger, dried basil, dried floral parts – dried cloves, and saffron²

4. The Committee **is invited to endorse** the methods of analysis in Appendix II.

COMMITTEE ON FATS AND OILS (CCFO26)

Methods of analysis for acid value and free fatty acids for virgin palm oil and crude palm kernel oil³

5. The Committee **is invited to endorse** the methods of analysis in Appendix III.

¹ REP19/NFSDU, para 156, Appendix IX

² REP19/SCH, paras 30, 39, 47, 66, 88, 95 and Appendices II, III, IV, V, VI, VII

³ REP19/FO, para. 67 (ii), Appendix IV, part A

APPENDIX I

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU40)**Methods of analysis for infant formula**

| Commodity | Provision | Method | Principle | Proposed Type |
|-----------|------------|---|---|-------------------|
| | Calcium | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | II III |
| | | AOAC 985.35 | Flame atomic absorption spectrometry | III |
| | Copper | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | AOAC 985.35 | Flame atomic absorption spectrophotometry | II III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Iron | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | AOAC 985.35 | Flame atomic absorption spectrometry | III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Magnesium | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | II III |
| | | AOAC 985.35 | Flame atomic absorption spectrometry | III |
| | Manganese | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | AOAC 985.35 | Flame atomic absorption spectrometry | II III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Phosphorus | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | | AOAC 986.24 | Spectrophotometry (molybdovanadate) | II III |
| | Potassium | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | II III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Sodium | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | II III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Zinc | AOAC 2015.06 / ISO 21424 IDF 243 | ICP mass spectrometry | II |
| | | AOAC 985.35 | Flame atomic absorption spectrometry | II III |
| | | AOAC 984.27 | ICP emission spectroscopy | III |
| | Vitamin K | AOAC 2015.09 / ISO 21446 | HPLC | II |
| | Folic acid | AOAC 2011.06 | LC-MS/MS | II |
| | | AOAC 992.05 / EN 14131 | Microbioassay | II III |
| | | J AOAC Int. 2000:83; 1141-1148 | Optical Biosensor Immunoassay | IV |
| | | J Chromatogr. A., 928, 77-90, | HPLC, incorporating | IV |

| | | | | |
|--|--|------|--|--|
| | | 2001 | immunoaffinity clean-up and conversion to 5-methyltetrahydrofolate | |
|--|--|------|--|--|

APPENDIX II

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH4)*Methods of analysis for provisions in the proposed draft standard for dried or dehydrated garlic***9. METHODS OF ANALYSIS AND SAMPLING****9.1 Methods of analysis¹**

| Parameter | Method | Principle | Type ⁴ |
|---|--|---|-------------------|
| Moisture | ISO 939 | Distillation | I |
| | AOAC 2001.12 | Titration | II |
| | AOAC 986.21 | Distillation | I |
| | ASTA 2.0 | Distillation | I |
| Total Ash | ISO 928 | Gravimetry | I |
| | AOAC 941.12 | Gravimetry | I |
| | ASTA 3.0 | Gravimetry | I |
| Acid Insoluble Ash | ISO 930 | Gravimetry | I |
| | AOAC 941.12 | Gravimetry | I |
| | ASTA 4.0 | Gravimetry | I |
| Extraneous Matter | ISO 927 | Visual Examination followed by Gravimetry | I |
| | ASTA 14.1 | Visual Examination followed by Gravimetry | I |
| Foreign Matter | ISO 927 | Visual Examination followed by Gravimetry | I |
| | AOAC 960.51 | Sieving followed by Gravimetry | I |
| Insects/ Fragments | Method appropriate for particular spice from AOAC Chapter 16, subchapter 14 | Visual Examination | IV |
| Live Insects | ISO 927 | Visual Examination | IV |
| | AOAC 960.51 | Visual Examination | IV |
| Excreta Mammalian, | Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) AOAC 993.27 (For Ground) | Visual Examination | IV |
| | | Enzymatic Detection Method | IV |
| Cold Water Soluble Extract | ISO 941 | Extraction followed by Gravimetry | I |
| Volatile Organic Sulfur Compounds Content | ISO 5567 | Distillation followed by Titrimetry | IV |
| Mould damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | Visual Examination (For whole) | IV |

¹ Latest edition or version of the approved method should be used

⁴ According to the definition of "types of method of analysis" as per Codex Procedural Manual Section II.

Methods of analysis for provision in the Proposed Draft Standard for Dried Oregano**9. METHODS OF ANALYSIS AND SAMPLING****9.1 Methods of analysis****Table 4.** Methods of analysis¹

| Parameter | Method | Principle | Type ⁵ |
|-----------------------------|--|---|-------------------|
| Moisture | ISO 939 | Distillation | I |
| | ISO 760 | Titration | II |
| | AOAC 2001.12 | Titration | II |
| | ASTA 2.0 | Distillation | I |
| Total ash | ISO 928 | Gravimetry | I |
| | ASTA 3.0 | Gravimetry | I |
| Acid-insoluble ash | ISO 930 | Gravimetry | I |
| | ASTA 4.0 | Gravimetry | I |
| Volatile oils | ISO 6571 | Distillation followed by Volumetric | I |
| | ASTA 5.0 | Distillation followed by Volumetric | I |
| Extraneous vegetable matter | ISO 927 | Visual examination followed by Gravimetry | I |
| | ASTA 14.1 | Visual examination followed by Gravimetry | I |
| Foreign matter | ISO 927 | Visual examination followed by Gravimetry | I |
| Mammalian excreta | Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) | Visual examination | IV |
| | | | IV |
| Other excreta | AOAC 993.27 (For Ground) | Enzymatic Detection Method | IV |
| Whole dead insect | ISO 927 | Visual examination | IV |
| | MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32) | Visual examination | IV |
| Insect fragments | AOAC 975.49 | Flotation method | IV |
| | AOAC 969.44 | Flotation method | IV |

¹ Latest edition or version of the approved method should be used

⁵ According to the definition of "types of method of analysis" as per Codex Procedural Manual Section II.

Methods of analysis in the Proposed Draft Standard for Dried Roots, Rhizomes And Bulbs — Dried Or Dehydrated Ginger

9. METHODS OF ANALYSIS AND SAMPLING

9.1 Methods of analysis¹

| Parameter | Method | Principle | Type ⁶ |
|----------------------------|---|--|-------------------|
| Moisture | ISO 939 | Distillation | I |
| | AOAC 2001.12 | Titration | II |
| | AOAC 986.21 | Distillation | I |
| | ASTA 2.0 | Distillation | I |
| Total Ash | ISO 928 | Gravimetry | I |
| | AOAC 941.12 | Gravimetry | I |
| | ASTA 3.0 | Gravimetry | I |
| Acid Insoluble Ash | ISO 930 | Gravimetry | I |
| | AOAC 941.12 | Gravimetry | I |
| | ASTA 4.0 | Gravimetry | I |
| Volatile Oil | ISO 6571 | Distillation followed by Volumetry | I |
| | AOAC 962.17 | Distillation followed by Volumetry | I |
| | ASTA 5.0 | Distillation followed by Volumetry | I |
| Extraneous Matter | ISO 927 | Visual Examination followed by Gravimetry | I |
| | AOAC 916.01 | Visual Examination followed by Gravimetry | I |
| | ASTA 14.1 | Visual Examination followed by Gravimetry | I |
| Foreign Matter | AOAC 960.51 | Visual Examination followed by Gravimetry | I |
| | ISO 927 | Visual Examination followed by Gravimetry | I |
| Insect Damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) https://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm105731.htm#v-117 | Visual Examination | IV |
| Extractable Colour | American Dried Onion and Garlic Association (ADOGA) method IV.C.5 | Chemical extraction | IV |
| Hot Water Insoluble Solids | ADOGA method IV.C.7 | Chemical extraction followed by Gravimetry | I |
| Insects/Insect Fragments | Method appropriate for particular spice from AOAC Chapter 16, subchapter 14 | Visual Examination | IV |
| Excreta Mammalian, | Macroanalytical Procedure | Visual Examination | IV |

⁶ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II.

| | | | |
|---------------|---|----------------------------|----|
| | Manual, USFDA, Technical Bulletin V.39 B (For whole) | | |
| Excreta Other | AOAC 993.27 (For Ground) | Enzymatic Detection Method | IV |
| Mould visible | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | Visual examination | IV |
| Live Insect | ISO 927 | Visual Examination | IV |
| | AOAC 960.51 | Visual Examination | IV |

¹ Latest edition or version of the approved method should be used.

Methods of analysis in the Proposed Draft Standard for Dried Leaves – Dried Basil

9. METHODS OF ANALYSIS AND SAMPLING

9.1 Methods of Analysis*

| Parameter | Method | Principle | Type⁷ |
|----------------------------------|---|--|-------------------------|
| Moisture | ISO 760 | Titration | II |
| | ISO 939 | Distillation | I |
| | AOAC 2001.12 | Titration | II |
| | ASTA 2.0 | Distillation | I |
| | AOAC 941.11 | Distillation | I |
| Total Ash | AOAC 986.21 | Distillation | I |
| | ISO 928 | Gravimetry | I |
| | AOAC 950.49 | Gravimetry | I |
| Acid Insoluble Ash | ASTA 3.0 | Gravimetry | I |
| | ISO 930 | Gravimetry | I |
| Volatile Oil | ASTA 4.0 | Gravimetry | I |
| | ISO 6571 | Distillation followed by Volumetry | I |
| | AOAC 962.17 | Distillation followed by Volumetry | I |
| Extraneous Matter | ASTA 5.0 | Distillation followed by Volumetry | I |
| | ISO 927 | Visual Examination followed by Volumetry | I |
| Foreign Matter | ISO 927 | Visual Examination followed by Volumetry | I |
| Insect Damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) | Visual Examination | IV |
| Insects/Excreta/Insect Fragments | Method appropriate for particular spice from AOAC Chapter 16, subchapter 14 [ISPM 08 Determination of Pest Status in an area] | Visual Examination | IV |
| Mould damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) | Visual examination (for whole) | IV |

⁷ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II.

| | | | |
|--------------------|--|----------------------------|----|
| Colour | Consider the use of Chroma Meters Measuring Head for color measurement | Colourimetry | IV |
| Excreta Mammalian, | Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) | Visual Examination | IV |
| Excreta Other | AOAC 993.27 (For Ground) | Enzymatic Detection Method | IV |

* Latest edition or version of the approved method should be used.

Methods of analysis for provisions in the Proposed Draft Standard for Dried Floral Parts – Dried Cloves

9. METHODS OF ANALYSIS AND SAMPLING

9.1 Methods of analysis¹

| Parameter | Method | Principle | Type⁸ |
|----------------------------------|---|---|-------------------------|
| Moisture | ISO 939 | Distillation | I |
| | [AOAC 2001.12] | Titration | II |
| | [ASTA 2.0] | Distillation | I |
| Total Ash | ISO 928 | Gravimetry | I |
| | ISO 3632-2 | Gravimetry | I |
| | AOAC 950.49 | Gravimetry | I |
| | ASTA 3.0 | Gravimetry | I |
| Acid Insoluble Ash | ISO 930 | Gravimetry | I |
| | ISO 3632-2 | Gravimetry | I |
| | ASTA 4.0 | Gravimetry | I |
| Volatile oil | ISO 6571 | Distillation followed by Volumetry | I |
| | AOAC 962.17 | Distillation followed by Volumetry | I |
| | ASTA 5.0 | Distillation followed by Volumetry | I |
| Extraneous Matter | ISO 927 | Visual examination followed by Gravimetry | I |
| | ISO 3632-2 | Visual examination followed by Gravimetry | I |
| | ASTA 14.1 | Visual examination followed by Gravimetry | I |
| Foreign Matter | ISO 927 | Visual examination followed by Gravimetry | I |
| | ISO 3632-2 | Visual examination followed by Gravimetry | I |
| Insect Damage | ISO 927 | Visual Examination | IV |
| | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | Visual Examination | IV |
| Insects/Excreta/Insect Fragments | ISO 927 Method appropriate for particular spice from AOAC Chapter 16, subchapter 14 | Visual examination | IV |
| Crude Fibre | AOAC – 920.169 | Gravimetry | I |
| | ISO 5498 | Gravimetry | I |

⁸ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II.

| Parameter | Method | Principle | Type ⁸ |
|--------------------|---|--|-------------------|
| Mould visible | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | Visual examination | IV |
| Live Insect | ISO 927 AOAC 960.51 | Visual Examination Visual Examination | IV IV |
| Excreta Mammalian, | Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) | Visual Examination | IV |
| Excreta Other | AOAC 993.27 (For Ground) | Enzymatic Detection Method | IV |

¹ Latest edition or version of the approved method should be used

Methods of analysis for provisions in the Proposed Draft Standard for Saffron

9. METHODS OF ANALYSIS AND SAMPLING

9.1 Methods of analysis^{1,2}

| Parameter | Method | Principle | Type ⁹ |
|---|--------------------------------------|--|-------------------|
| Moisture | AOAC 934.06 ISO 3632-1 | Gravimetry Gravimetry | I I |
| Total Ash | AOAC 941.12 ISO 928 ISO 3632-2 | Gravimetry Gravimetry Gravimetry | I I I |
| Acid Insoluble Ash | AOAC 941.12 ISO 930 ISO 3632-2 | Gravimetry Gravimetry Gravimetry | I I I |
| Soluble extract in cold water | ISO 941 ISO 3632-2 | Extraction | I I |
| Taste strength (expressed as picrocrocin) $A_{1\%}^{1\text{cm}}$ 257 nm | ISO 3632-2 | Absorbance | IV |
| Aroma strength (expressed as safranal) $A_{1\%}^{1\text{cm}}$ 330 nm | ISO 3632-2 | Absorbance | IV |
| Coloring strength (expressed as crocin) $A_{1\%}^{1\text{cm}}$ 440 nm | ISO 3632-2 | Absorbance | IV |
| Artificial colorants | ISO 3632-2 | Chromatography | II |

⁹ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II.

| Parameter | Method | Principle | Type ⁹ |
|--------------------------|---|---|-------------------|
| Extraneous Matter | ISO 927 | Visual Examination followed by Gravimetry | I |
| | ISO 3632-2 | Visual Examination followed by Gravimetry | I |
| Foreign Matter | ISO 927 | Visual Examination followed by Gravimetry | I |
| | ISO 3632-2 | Visual Examination followed by Gravimetry | I |
| Insect Damage | ISO 927 | Visual Examination | I |
| Insects/Insect Fragments | ISO 927 | Visual Examination | I |
| Mould visible | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | Visual examination | IV |
| Excreta Mammalian, | Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) | Visual Examination | IV |
| Excreta Other | AOAC 993.27 (For Ground) | Enzymatic Detection Method | IV |

¹ Note: The minimum laboratory sample according to ISO 3632-2 (Table 1) for duplicate analysis is: filament saffron: 11.5 g x 2 = 23 g, powdered saffron: 6.75 g x 2 = 13.5 g

² Latest edition or version of the approved method should be used.

APPENDIX III

REPLACEMENT OF ACID VALUE WITH FREE FATTY ACIDS FOR VIRGIN PALM OIL AND INCLUSION OF FREE FATTY ACIDS FOR CRUDE PALM KERNEL OIL

(CONSEQUENTIAL AMENDMENT TO CXS 210-1999, APPENDIX, SECTION 5: METHODS OF ANALYSIS AND SAMPLING, TO INCLUDE AN ADDITIONAL METHOD FOR THE DETERMINATION OF ACIDITY)

(Proposed changes to CXS 234)

| <i>Commodity Standard</i> | <i>Provision</i> | <i>Method</i> | <i>Principle</i> | <i>Type</i> |
|------------------------------------|--|--|--------------------------|--------------------|
| <u>Named Vegetable Oils</u> | <u>Acidity:</u> <u>acid value</u> | ISO 660 or AOCS Cd 3d-63 or AOCS Ca 5a-40 | <u>Titrimetry</u> | ! |
| | <u>free fatty acids</u> | ISO 660 or AOCS Ca 5a-40 | <u>Titrimetry</u> | ! |