

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
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Organization

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

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON SPICES AND CULINARY HERBS

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Typing of Methods of Analysis as per the Codex Procedural Manual, Section II

(Submitted by India)

As per the *Principles for the establishment of Codex methods of analysis*¹ and *Methods of analysis and sampling, normal Practice*², the typing of analytical methods referred in the proposed draft standards is prepared.

Methods of Analysis for Spices and Culinary Herbs

Methods are for the proposed draft standards CX/SCH 19/4/3, CX/SCH 19/4/4, CX/SCH 19/4/5, CX/SCH 19/4/6, CX/SCH 19/4/7, CX/SCH 19/4/8, CX/SCH 19/4/9, CX/SCH 19/4/10

SI.No	Spices	Provision	Method	Principles	Type
1	(a) Oregano	Moisture	ISO 939:1980	Distillation	I
			ISO 760:1978	Titration	II
			AOAC 2001.12:2005	Titration	II
			AOAC 986.21:1988	Distillation	I
			AOAC 941.11:1987	Distillation	I
	(b) Dried or Dehydrated ginger	Moisture	ASTA 2.0:2011	Distillation	I
			ISO 928:1997	Gravimetry	I
	(c) Dried and dehydrated Garlic	Total ash	[ISO 3632-2:2010] ^a	Gravimetry	I
			AOAC 941.12:1941	Gravimetry	I
			[AOAC 950.49:1950] ^b	Gravimetry	I
	(d) Dried chilli peppers and paprika	Total ash	ASTA 3.0:1997	Gravimetry	I
			ISO 930:1997	Gravimetry	I
			[ISO 3632-2:2010] ^a	Gravimetry	I
			AOAC 941.12:1941	Gravimetry	I
	(e) Dried Basil	Acid- insoluble ash	ASTA 4.0:1997	Gravimetry	I
			ISO 6571:2008	Distillation followed by Volumetry	I
AOAC 962.17:1996			Distillation followed by Volumetry	I	
ASTA 5.0:2010			Distillation followed by Volumetry	I	
(f) Nutmeg	Volatile oils	ISO 6571:2008	Distillation followed by Volumetry	I	
		AOAC 962.17:1996	Distillation followed by Volumetry	I	
(g) Cloves	Volatile oils	ASTA 5.0:2010	Distillation followed by Volumetry	I	
		ISO 6571:2008	Distillation followed by Volumetry	I	

			ASTA 5.2:2010	Distillation followed by Volumetry	I
		Extraneous vegetable matter	ISO 927:2009	Visual examination followed by Gravimetry	I
			[ISO 3632-2:2010] ^a	Visual examination followed by Gravimetry	I
			AOAC 916.01: 1916	Visual examination followed by Gravimetry	I
			AOAC 960.51: 1996	Sieving followed by Gravimetry	I
			ASTA 14.1:2014	Visual examination followed by Gravimetry	I
		Foreign matter	ISO 927:2009	Visual examination followed by Gravimetry	I
			[ISO 3632-2:2010] ^a	Visual examination followed by Gravimetry	I
			AOAC 960.51: 1996	Sieving followed by Gravimetry	I
		Mammalian excreta	ISO 927:2009 MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32)	Visual examination (for whole)	IV
			[AOAC 993.27:1996] ^c (for ground)	Enzymatic Detection method (for ground)	IV
		Whole dead insect	ISO 927:2009	Flotation method	IV
			AOAC 969.44:1996	Flotation method	IV
		Insect fragments	ISO 927:2009	Flotation method	IV
			AOAC 975.49:2001	Flotation method	IV
		Insect Damage	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32)	Visual examination	IV
2	Dried or dehydrated ginger	Extractable colour	[American Dried Onion and Garlic Association (ADOGA) method 1V .C.5] ^d	Chemical Extraction	IV
		Hot water Insoluble Solids	[American Dried Onion and Garlic Association(ADOGA) method 1V.C.7] ^d	Chemical Extraction followed by Gravimetry	I

3	Dried and dehydrated Garlic	Cold water soluble Extracts	ISO 941:1980	Extraction followed by Gravimetry	I
		Volatile organic sulfur Compounds content	ISO 5567:1982	Distillation followed by Titrimetry	IV
4	Dried chilli peppers and paprika	Natural colouring matter	ISO 7541:1989	Spectrophotometry	IV
		Capsaicin content	ASTA 21.3:2004	HPLC	IV
			ISO 3513:1995	HPLC	IV
5	Dried Basil	Mould damage	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32)	Visual examination (for whole)	IV
6	Nutmeg	Water Insoluble ash	ISO 929:1980	Gravimetry	I
		Calcium content expressed as CaO	[ISO 1003:2008] ^e	Gravimetry	I
			AOAC 941.12C:1941	Gravimetry	I
		Non -Volatile ether extract	ISO 1108:1992	Gravimetry	I
			AOAC 940.29:1940	Gravimetry	I
		Mould visible	ISO 927:2009	Visual examination	1V
			AOAC 960.51:1996	Visual examination	IV
		RSodent contamination	ISO 927:2009	Visual examination	1V
			AOAC 960.51:1996	Visual examination	IV
		Live insect	ISO 927:2009	Visual examination	1V
AOAC 960.51:1996	Visual examination		IV		
7	Saffron	Moisture	AOAC 934.06:1934	Gravimetry	I
			ISO 3623-2:2010	Gravimetry	I
		Total ash	AOAC 941.12:1941	Gravimetry	I
			ISO 928:1997	Gravimetry	I
			ISO 3632-2:2010	Gravimetry	I
		Acid- insoluble ash	AOAC 941.12:1941	Gravimetry	I
			ISO 930:1987	Gravimetry	I
			ISO 3632-2:2010	Gravimetry	I
		Soluble extract in cold water	ISO 941:1980	Solubility	I
ISO 3632-2:2010	Solubility		I		

		Taste strength (expressed as picrocrocin) A ^{1%} 1 cm 257 nm	ISO 3632-2:2010	Absorbance	IV
		Aroma strength (expressed as safranal) A ^{1%} 1 cm 330 nm	ISO 3632-2:2010	Absorbance	IV
		Coloring strength (expressed as crocin) A ^{1%} 1 cm 440 nm	ISO 3632-2:2010	Absorbance	IV
		Artificial colorants	ISO 3632-2:2010	Chromatography	II
		Extraneous matter	ISO 927:2009	Visual examination followed by gravimetry	I
			ISO 3632-2:2010	Visual examination followed by gravimetry	I
		Foreign matter	ISO 927:2009	Visual examination followed by gravimetry	I
			ISO 3632-2:2010	Visual examination followed by gravimetry	I

Notes for table entries: ^aMethod is specific for saffron, ^bMethod is for nut and nut products, ^cMethod is specified for ground black pepper, ^dMethod is specific for onion and garlic, ^eISO 1003:2008 is specifications for ginger

References

I. AOAC Methods

1. AOAC 916.01:1916 – Adulterants in spices – Microscopic examination method
2. AOAC 934.06:1934 – Loss on dried (moisture) in dried fruits
3. AOAC 940.29:1940 – Methylene chloride extract (Volatile and non volatile) of spices – Gravimetric method
4. AOAC 941.11:1987 - Moisture in spices – Distillation method
5. AOAC 941.12:1941 Ash of spices – Gravimetric Method
6. AOAC 950.49:1950 - Ash of nut and nut products– Gravimetric Method
7. AOAC 960.51:1996 – Foreign matter in spices and condiments – sieving method
8. AOAC 962.17:1996 – Volatile oil in spices
9. AOAC 969.44:1996 – Light filth in oregano – Floatation method
10. AOAC 975.49:2001 - Light filth in Spices and condiments - Floatation method
11. AOAC 986.2:1988 - Moisture in spices – Distillation method
12. AOAC 993.27: 1996 – Mammalian feces in ground black pepper – Alkaline Phosphatase Detection method
13. AOAC 2001.12:2005 – Water/ dry matter (moisture) in animal feed, grain and forage - Karl Fischer titration Method.

II. ASTA Methods

1. ASTA 2.0:2011 Moisture (Distillation Method)
2. ASTA 3.0:1997 Total ash
3. ASTA 4.0:1997 Acid insoluble ash
4. ASTA 5.0:2010 Steam Volatile oil (Modified Clevenger Method)
5. ASTA 5.2:2010 Steam Volatile oil in spices
6. ASTA 14.1:2014 Extraneous matter in spices
7. ASTA 21.3:2004 Pungency of capsicum and oleoresins (HPLC method – preferred)

III. ISO Methods

1. ISO 760:1978 Determination of water – Karl Fischer Method
2. ISO 927:2009 - Spices and condiments – Determination of extraneous matter and foreign matter
3. ISO 928:1997 Spices and condiments – Determination of total ash
4. ISO 929:1980 Spices and condiments – Determination of water insoluble ash
5. ISO 930:1987 Spices and condiments – Determination of acid insoluble ash
6. ISO 939:1980 Spices and condiments – Determination of moisture content/ Entrainment method
7. ISO 941:1980 Spices and condiments – Determination of cold water extracts
8. ISO 1003:2008 Ginger (*Zingiber officianale*) - specification
9. ISO 1108:1992 Spices and condiments – Determination of non volatile ether extracts
10. ISO 3513:1995 chillies – Determination of Scoville Index
11. ISO 3632-2:2010 Spices – Saffron (*Crocus sativus L*) – Part 2 Test methods
12. ISO 5567:1982 Determination of volatile organic sulfur compounds
13. ISO 6571:2008 Spices and condiments and herbs – Determination of volatile oil content (Hydrodistillation method)
14. ISO 7541:1989 Ground (powdered) paprika – Determination of total natural coloring matter content

IV. Others

1. MPM V-8 Spices, Condiments, Flavours and Crude Drugs, Macroanalytical procedure manual, A. General methods for spices herbs and botanicals (V 32) , FDA Technical Bulletin Number 5 (for whole)

¹Principles for the Establishment of Codex Methods of Analysis, Section 2, Page 77, CODEX Alimentarius Commission Procedural Manual, 26th Edition.

²Elaboration of Codex Texts, Methods of Analysis and Sampling, Normal Practice, Section 2, Page 53, CODEX Alimentarius Commission Procedural Manual, 26th Edition.