codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 7 **CX/PFV 00/7**
June 2000

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

20th Session, 11-15 September 2000
Washington D.C., United States of America

METHODS OF ANALYSIS FOR PROCESSED FRUITS AND VEGETABLES

- REQUEST FOR COMMENTS -

Governments and interested international organizations wishing to submit comments on the attached ***Methods of Analysis for Processed Fruits and Vegetables*** (see ALINORM 99/27, para. 70) as contained in Appendix II and the Recommendations in Appendix I are invited to do so **not later than 15 July 2000** to Ms. Ellen Y. Matten, U.S. Codex Office, Food Safety and Inspection Service, US Department of Agriculture, Room 4861 South Building, 1400 Independence Ave. S.W., Washington, DC, 20250-2700, Fax: +1 202 720 3157, E-mail: uscodex@usda.gov, with a copy to the Secretary, Joint FAO/WHO Food Standards Programme, Viale delle Terme di Caracalla 00100 Rome, Italy (Fax No. + 39.06.5705.4593 or E-Mail codex@fao.org).

BACKGROUND

1. The Codex Committee on Processed Fruits and Vegetables at its 19th Session (March 1998) considered, with assistance of an *ad hoc* working group, methods of analysis necessary to determine compliance of products to the relevant Codex standards for processed fruits and vegetables. The working group revised the list of methods of analysis contained in CX/PFV 98/7. Due to time constraints, the Committee could not consider these methods fully. The Committee agreed to circulate the revised list along with additional information provided by the working group for comments by governments and interested international organizations. It also agreed that the revised list would be considered by the Committee at its 20th Session in light of comments received before being submitted to the Codex Committee on Methods of Analysis and Sampling (CCMAS). The Committee at its 20th Session should agree on the methods of analysis for each standard before they can be sent to the CCMAS for endorsement. After the endorsement of these methods and the adoption of the relevant standards, these methods will be incorporated into Volume 13 of the *Codex Alimentarius*.

2. From the revised list, methods of analysis for vinegar have been removed as the revision of the European Regional Standard has been entrusted to the Codex Coordinating Committee for Europe. Methods of analysis for the draft standards for pickles and kimchi and the proposed draft standard for aqueous coconut products[1] have been added to the list as they are proposed to be developed further by the Committee on Processed Fruits and Vegetables. The names of certain standards in the list have been amended in accordance with the decisions of the Committee made at its 19th Session. The methods in Appendix II are sorted according to alphabetical order of the standards and analytes, except those recommended for processed fruits and vegetables in general.

3. Where there is a specification or labelling requirement in the standard, it is necessary to recommend a method(s) for the provision. However, if there is no specification or labelling requirement, there is no need to select methods of analysis.

4. Governments are invited to comment on the methods of analysis contained in Appendix II and the recommendations in Appendix I.

[1] Pending adoption by the 47th Session of the Executive Committee at Step 5.

APPENDIX I: ADDITIONAL INFORMATION

The following is based on the recommendations made by the working group on methods of analysis made at the 19th Session of the Codex Committee on Processed Fruits and Vegetables. They have been incorporated into the list of methods in Appendix II and methods A-Q in Annex 1, except as otherwise stated.

1. Arsenic in chutney

AOAC 952.13 should be replaced by AOAC 986.15, *Arsenic, Cadmium, Lead, Selenium, and Zinc in Human Food and Pet Foods, a Multi element Method.*

The Committee should review and determine whether or not ISO 6634:1982 is equivalent to AOAC 986.15.

2. Tin in chutney

The current method AOAC 980.19 for *Tin in Food*, should be replaced by AOAC 985.16, *Tin in Food* by atomic absorption spectrophotometry.

The Committee should review and determine whether ISO 2447:1974 is equivalent to AOAC 986.15.

3. Lactic acid in edible fungi and fungus products

AOAC 945.99 *Lactic Acid in Canned Vegetables* by spectrophotometric analysis is recommended.

An enzymatic method for determination of lactic acid may exist, and it should be identified and agreed by the Committee before submitting it to the CCMAS for endorsement.

4. Total ash in chutney

AOAC 940.26, *Ash of Fruits and Fruit Products* is recommended.

The Committee should review and determine whether ISO 5516:1978 is equivalent to AOAC 940.26.

5. pH of processed fruits and vegetables

ISO 11289:1993 and AOAC 981.12 are recommended.

6. Calcium in processed fruits and vegetables

An ISO method and AOAC 968.31 are recommended.

The Committee needs to identify the reference number of the ISO method.

7. Total solids (in ºBrix) in processed fruits and vegetables

ISO 2173:1978 and AOAC 932.12 are recommended.

In addition, AOAC 920.151 is recommended.

8. Sulphite in processed fruits and vegetables

ISO 522:1981 and AOAC 990.26 are recommended.

9. Sodium chloride in processed fruits and vegetables

ISO 3634:1979 and AOAC 971.27 are recommended.

10. Butter or margarine in processed fruits and vegetables

AOAC 970.51 is recommended.

Method A: Determination of Drained Weight - Method II

1. Revise section 2.1 Specifications for Circular Sieves to read: If total quantity of contents is less than ~~1.5 kg. (3 lbs)~~ *1 kg. (2 lbs)* use a sieve. . . .

2. Revise fourth sentence of section 3. Procedure to read: Without shifting the contents, ~~so~~ incline the sieve *approximately 20º from the horizontal* to facilitate drainage . . .

3. Insert new sentence at the end of the paragraph: "This determination should be performed at 20ºC ±5ºC."

4. The instructions omit two important steps: (1) the weighing of the full container; and (2) the weighing of the dry empty container. Both weights are required to calculate the percentage drained weight (solid content) and/or the percent liquid. *The Committee needs to draft appropriate text for these procedures.*

Method B: Tough String Test

Replace this method with French method.

The Committee needs to include either the reference or the text of the method for consideration by the Committee and the CCMAS.

Method C: Determination of Washed Drained Weight:

1. In section 1. Definition, insert the phrase: "For viscous products such as gravy, sauce, and syrup" at the beginning of the paragraph.

2. In section 2.1 Specification for Circular Sieve, revised square opening dimensions from 3.0 mm by 3.0 mm to 2.5 mm by 2.5 mm. *In the original text the square openings are described as 0.30 mm by 0.30 mm.*

Method D: Determination of Proper fill in Lieu of Drained Weight

This method should be eliminated.

Method E: Determination of Water Capacity of Containers:

1. Delete references to "metal containers".

2. Refer to ISO method 90-1 for determination of water capacity in metal containers.

3. Delete section 4.1.

Method H: Determination of Mineral Oil in Raisins

The method needs some additional clarity: the specification for alumina could be read as alumina in water slurry, which it obviously is not. Also, there is no recommended action with respect to comparisons to the refractive index and IR spectra of a reference sample of mineral oil. It would seem that these comparisons could be deleted from the method. *The Committee needs to elaborate a revised text.*

Method J: Determination of Acidity (Table Olives)

It is recommended to revise the method to require the use of a pH meter for titration to an end point of pH 8.3.

Method K: Determination of pH (Table Olives)

It is recommended that the method be revised to explicitly state that the *brine* is to be measured for pH as opposed to the olives.

Method M: Determination of Broken, Slabs, Dirty, Damaged, and Immature Fruits (Dried Apricot)

1. The method should specify a sample size of 1 kg.
2. The formula for % defective should be:

$$\frac{\text{Weight of defective unites}}{\text{Total weight of sample}} x100 = \%\ \text{defective}$$

Method N: Determination of Volume of Fill (by Displacement)(Pickled Cucumber)

$$\text{percent volume of pickle ingredient} = \frac{V_1 - V_2}{V_1}$$

Revise the equation for percent volume of pickle ingredient:

Method P: Determination of Total Acidity of Extracted Oil (Grated Desiccated Coconut)

Change the method to require the use of a pH meter in the titration to an end point of pH 8.3.

Methods of Sampling [2]

The Committee should request the CCMAS to consider adopting ISO 2859-1:1989 for methods of sampling.

Methods of Sampling C: For Canned Pineapple

Change “600 g” to “500 g” in section 2.(b).

[2] The Codex Committee on Methods of Analysis and Sampling is developing general guidelines for Sampling.

APPENDIX II: METHODS OF ANALYSIS FOR THE STANDARDS FOR PROCESSED FRUITS AND VEGETABLES

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Processed fruits and vegetables	Calcium		AOAC 968.31 ISO method	Complexometric titrimetry			
Processed fruits and vegetables (except canned tomatoes and canned mushrooms)	Drained weight		AOAC 968.30	Sieving	A	E	I
Processed fruits and vegetables (except pickled cucumbers)	Fill of containers (glass containers)		Method E in Appendix	Weighing	A	E	I
Processed fruits and vegetables (except pickled cucumbers)	Fill of containers (metal containers)		ISO 90.1:1986	Weighing	A	E	I
Processed fruits and vegetables	Packing medium		AOAC 932.12 ISO 2173:1978	Refractometry			
Processed fruits and vegetables	pH		AOAC 981.12 ISO 11289:1993	Potentiometry			
Processed fruits and vegetables	Sodium chloride		AOAC 971.27 (Codex general method)	Potentiometry			
Processed fruits and vegetables	Sodium chloride		ISO 3634:1979				
Processed fruits and vegetables	Soluble solids		ISO 2173:1978 AOAC 932.14C	Refractometry		E	I
Processed fruits and vegetables	Sulphite		AOAC 990.26 ISO 522:1981				
Processed fruits and vegetables	Total solids		AOAC 932.12 ISO 2173:1978	Refractometry			
Processed fruits and vegetables	Total solids		AOAC 920.151	Gravimetry			
Processed vegetables	Butter or margarin		AOAC 970.51	Gas chromatography			

[3] Symbols in the left column: B, specification in the body of the standard except those for food additives; F, for food additive(s); and A, specification in Annex of the standard.
Symbol in the center column: E, endorsed by the Codex Committee on Methods of Analysis and Sampling; TE, temporarily endorsed.
Symbol in the right column: I-IV, types of methods.

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Aqueous coconut products	Sampling	-	General Guidelines on Sampling[4]	-			
Aqueous coconut products	Total solids	Varying levels	AOAC 925.23A	Gravimetry (drying at 98-100ºC)	B		
Aqueous coconut products	Total fat	Varying levels	AOAC 945.48G	Röse-Gottlieb method	B		
Aqueous coconut products	Non-fat solids	Varying levels	Subtracting total fats from total solids	-	B		
Aqueous coconut products	Moisture	<=95% m/m	Subtracting total solids from 100	-	B		
Canned applesauce	Total solids	>=9 % (9ºBrix)	AOAC 932.12 ISO 2173	Refractometry	A		
Canned bamboo shoot[5]	pH	>=4.0; 4.0-4.6 (if acid is added)	AOAC 981.12	Potentiometry	B	E	I
Canned berry fruits (raspberry, strawberry)	Packing medium	>=10ºBrix	AOAC 932.12 ISO 2173	Refractometry	A		
Canned berry fruits (strawberry)	Mineral impurities	<=300 mg/kg	AOAC 971.33	Ashing	A	E	I
Canned fruit cocktail	Proportions of fruit		Method L in Appendix	Visual separation			
Canned mushrooms	Washed drained weight	>=27.5% m/m (sauce packs)	Method C in Appendix	Sieving	A	E	I
Canned tomatoes	Drained weight		Method A in Appendix	Sieving	A	E	I
Canned tomatoes	Mould count	Not detected	AOAC 965.41	Howard mould count	A	E	I
Canned tropical fruit salad	Proportions of fruit		Method L in Appendix	Visual separation			
Canned vegetables (green beans and canned wax beans)	Tough string	*No specification* (only definition)	Method B in Appendix	Stretching		E	I
Canned vegetables (green peas)	Alcohol insoluble solids	<= 21 %	AOAC 938.10	Sieving	A	E	I
Canned vegetables (green peas)	Distinguishing types of peas		Method F in Appendix	Visual inspection	A	E	I

[4] Being developed by the CCMAS.

[5] For drained weight the Codex Committee on Methods of Analysis and Sampling at its 21st Session endorsed the same method that had been already endorsed for processed fruits and vegetables.

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Canned vegetables (mature processed peas)	Total solids	>=19.5% of the weight of distilled water at 20 C which the sealed container will hold when completely filled	AOAC 964.22	Vacuum oven	A	E	I
Canned vegetables (palmito)	Mineral impurities	<=0.1% m/m	ISO 762:1982 (confirmed 1992)	Gravimetry	A	E	I
Chutney[6]	Arsenic	<=1.0 mg/kg	AOAC 986.15 (Codex General Method) or ISO 6634:1982	Atomic Absorption Spectrophotometry	B		
Chutney	Ash, insoluble in HCl	<=0.5% m/m	ISO 763:1982	Gravimetry	A	E[7]	I
Chutney	Ash, total	<=5% m/m	AOAC 940.26 ISO 5516:1978	Gravimetry	A		
Chutney	Content of fruits and/or vegetables	>=40%			B		
Chutney	Lead	<=2.5 mg/kg	AOAC 972.25 (Codex General Method) ISO 6633:1984	Atomic Absorption Spectrophotometry	B		
Chutney	Tin	<=250.0 mg/kg	AOAC 985.16 ISO 2447:1974	Atomic Absorption Spectrophotometry	B		
Chutney (mango chutney)	Content of mango fruit	>=40%			B		
Dates	Identification of defects		Method I in Appendix	Visual inspection	B	E	I
Dates	Moisture	<=30 %	AOAC 934.06	Gravimetry (Vacuum oven)	A	E	I
Dried apricots	Identification of defects		Method M in Appendix	Visual inspection	A	E	III
Dried apricots	Moisture	<=25% m/m	AOAC 934.06	Gravimetry (Vacuum oven)	B	E	I
Dried edible fungi	Water	<=13% m/m			B		
Edible fungi and fungus products	Lactic acid	>=1% m/m (fermented fungi)	AOAC 945.99	Spectrophotometry	B		

6 Including mango chutney.

7 For mango chutney.

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Edible fungi and fungus products	Mineral impurities	<=1% m/m (fresh wild growing fungi); <=0.5% m/m (fresh cultivated fungi); <=2% m/m (dried fungi, fungus grits and fungus powder); <=0.1% m/m (pickled fungi; fungi in olive oil and other vegetable oils); <=0.2% m/m (fermented fungi; quick frozen fungi; sterilized fungi); Not detected (fungus extract and fungus concentrate; dried fungus concentrate); <=0.3% m/m (salted fungi)			A		
Edible fungi and fungus products	Sugars	<=2.5% m/m (pickled fungi)			B		
Edible fungi and fungus products	Vinegar	<=2% m/m as acetic acid (pickled fungi)			B		
Edible fungi and fungus products	Water	<=13% (dried fungi, fungus grits & fungus powder); <=13% m/m (fungus grits); <=9% (fungus powder & dried fungus concentrate)			B		
Grated desiccated coconut	Ash	< =2.5 % (m/m)	AOAC 950.49	Gravimetry	A	E	I
Grated desiccated coconut	Extraneous vegetable matter	<=15 fragments per 100 g	Method Q in Appendix	Counting extraneous material with the naked eye	A	E	IV
Grated desiccated coconut	Granularity	Extra fine, fine and medium	ISO 2591-1:1988 Test sieving according to British Standard Mesh Nominal Test Sieves: BS 410-1986	Sieving	A	TE [8]	I
Grated desiccated coconut	Moisture	<= 3 % m/m	AOAC 925.40	Gravimetry, Loss on drying	A	E	I
Grated desiccated coconut	Oil content	>= 55 % m/m	AOAC 948.22	Gravimetry	A	E	I

[8] The Codex Committee on Methods of Analysis and Sampling at its 21st Session extended its temporarily endorsed status.

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Grated desiccated coconut	Total acidity of extracted oil	<=0.3% m/m as lauric acid	Method P in Appendix	Titration of extracted oil	A	E	IV
Jam, jellies and marmalades (jam (fruit preserves) and jellies	Mineral impurities	<= 0.04 % (m/m)	AOAC 971.33	Ashing	A	E	I
Kimchi	Mineral impurities	<=0.03% m/m	AOAC 971.33	Ashing	A	E	I
Kimchi	Sampling	-	Codex Sampling Plans for Prepackaged Foods (AQL 6.5)	-		E	
Kimchi	Total acidity	<=1.0 % m/m	AOAC 942.15	Titrimetry		E	I
Pickled cucumbers	Acidity, total	0.4-3.5% as acetic acid	AOAC 942.15	Titrimetry	A	E	I
Pickled cucumbers	Mineral impurities	<=0.08% m/m	AOAC 971.33	Sedimentation and filtration	A	E	I
Pickled cucumbers	Solids, soluble, salt free (in packing medium)	1.5%-14% (sweet-sour type); >=14% (sweet type)					
Pickled cucumbers	Volume fill by displacement	>=53%	Method N in Appendix	Displacement	A	E	I
Pickles	Acidity	Not specified	AOAC 942.15	Titrimetry		E	I
Pickles	Arsenic	<=1.0 mg/kg	AOAC 952.13 (Codex general method)	Colorimetry, diethyldithiocarbamate	B	E	II
Pickles	Arsenic	<=1.0 mg/kg	ISO 6634:1982	Spectrophotometry, silver diethyldithiocarbamate	B	E	III
Pickles	Benzoic acid	<=250 mg/kg	ISO 5518:1978	Spectrophotometry	F	TE	IV
Pickles	Benzoic acid	<=250 mg/kg	IFU 63 (1995) (suggested by CCMAS for consideration)	Liquid chromatography	F		
Pickles	Benzoic acid	<=250 mg/kg	AOAC 990.28 (Codex general method; suggested by CCMAS for consideration)	Optimized Monier-Williams method	F		
Pickles	Lead	<=1.0 mg/kg	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	B	E	II

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Pickles	Lead	<=1.0 mg/kg	ISO 6633:1984	Flameless atomic abosrption spectrophotometry	B	E	III
Pickles	Salt	Not specified	AOAC 971.27 (Codex general method)	Potentiometry		E	II
Pickles	Salt	Not specified	AOAC 939.10	Volumetry, gravimetry, titrimetry (3 methods)		E	III
Pickles	Sampling		Codex Sampling Plans for Prepackaged Foods (AQL 6.5)	-		E	
Pickles	Sorbates	<=1000 mg/kg	ISO 5519:1978	Spectrophotometry	F	TE	IV
Pickles	Sorbates	<=1000 mg/kg	IFU 63 (1995) (suggested by CCMAS for consideration)	Liquid chromatography	F		
Pickles	Sorbates	<=1000 mg/kg	NMKL 103 (1984)/ AOAC 983.16 (Codex general method; suggested by CCMAS for consideration)	Gas chromatography	F		
Pickles	Sulphur dioxide	<=30 mg/kg	AOAC 990.28 (Codex general method; suggested by CCMAS for consideration)	Optimized Monier-Williams method	F		
Pickles	Tin	<=250.0 mg/kg[9]	AOAC 980.19 (Codex general method)	Atomic absorption spectrophotometry	B	E	II
Pickles	Tin	<=250.0 mg/kg [9]	iso 2447:1974		B	TE	IV
Processed tomato concentrates	Mineral impurities	< 60 mg/kg based on diluted product of 8% solids	AOAC 971.33	Ashing	A	E	IV
Processed tomato concentrates	Tomato soluble solids	>=8%	AOAC 970.59	Refractometry	B	E	I
Raisins	Mineral impurities	Not detected	Method G in Appendix	Ashing			

[9] The CCMAS requested the Committee to consider whether it is necessary to express the provision using four significant figures.

STANDARD	PROVISION	LEVEL	METHOD	PRINCIPLE	Note [3]		
Raisins	Mineral oil	<= 5 g/kg	Method H in Appendix	Extraction and separation on alumina	F	E	II
Raisins	Moisture	<=31%	AOAC 972.20	Moisture meter	B	E	I
Raisins	Sorbitol	<= 5 g/kg	AOAC 973.28	Gas chromatography	F	E	II
Table olives	Acidity of brine	>=0.4% m/m as lactic acid (fermented product)	Method J in Appendix	Titrimetry	B	E	IV
Unshelled pistachio nuts	Moisture	<= 7 % m/m	AOAC 925.40	Gravimetry (Loss on drying)	B	E	I
Unshelled pistachio nuts	Specific defects		Method O in Appendix	Visual separation			

ANNEX 1

METHODS OF ANALYSIS PREVIOUSLY RECOMMENDED AS CAC/RMS OR STATED IN THE STANDARDS

A. DETERMINATION OF DRAINED WEIGHT - METHOD II

1. DEFINITION

Drained weight expresses % solid content as determined by the procedure described below.

2. SPECIFICATIONS FOR CIRCULAR SIEVES

2.1 If the quantity of the total contents of the container is less than 1 kg (2 lb) use a sieve with a diameter of 20 cm (8 in).

2.2 If the quantity of the total contents of the container is 1.5 kg (3 lb) or more, use a sieve with a diameter of 30 cm (12 in).

2.3 The meshes of such sieves are made by so weaving wire as to form square openings of 11.2 mm by 11.2 mm[10].

3. PROCEDURE

Remove lid from container, but in the case of a container with lid attached by double seam, do not remove or alter the height of the double seam. Tilt the opened container so as to distribute the contents over the meshes of a circular sieve which has previously been weighed or for which a tare has been established. Without shifting the contents, incline the sieve approximately 20º from the horizontal to facilitate drainage of the liquid. Allow to drain for two minutes. At the end of the two minutes draining period, ascertain the weight of the material while still on the sieve, allowing for the tare (or weight of the sieve). This determination should be performed at 20º±5ºC.

4. CALCULATION AND EXPRESSION OF RESULTS

From weights thus obtained determine % m/m liquid and %m/m drained weight (solid content).

B. TOUGH STRING TEST

1. DEFINITION

A tough string is a string that will support the weight of 250 g for five seconds or longer when tested in accordance with the procedure described below.

2. PRINCIPLE

Strings are removed from individual pods, fastened through a clamp assembly weighing 250 g, and hung so that the string supports the entire weight. If the string supports the weight for five seconds or more it is considered a tough string.

3. APPARATUS

3.1 Weighted clamp

Use battery clamp (with teeth filed off or turned back), spring operated clothes pin, or binder clip which presents a flat clamping surface. Attach weight so that entire assembly of weight and clamp weighs 250 g. See Figure 1. A bag containing lead pellets is convenient as a weight.

4. PROCEDURE

4.1 From the drained product select a representative sample of not less than 285 g. Record the weight of this test sample.

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[10] Ref. ISO Recommendation R 565; such sieves may be replaced by U.S. sieves 2 mesh (size of opening 11.3 mm).

4.2 Break the individual bean units and set aside those that show evidence of tough strings. Remove the strings from the pods and retain the pod material for weighing.

4.3 Fasten the clamp assembly to one end of the string. Grasp the other end of the string with the fingers (a cloth may be used to aid in holding the string) and lift gently.

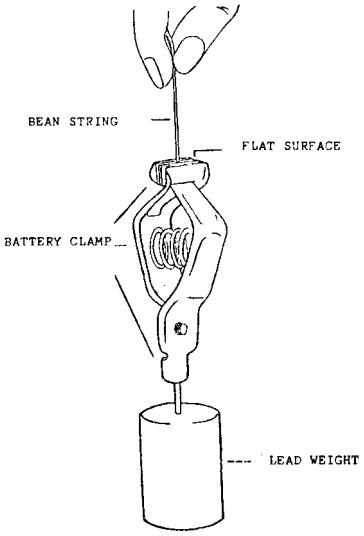
4.4 If the string supports the 250 g assembly for at least five seconds consider the bean unit as containing tough string. If the string breaks in less than five seconds, retest the broken parts that are 13 mm or longer to determine if such portions are tough.

4.5 Weigh the bean units which contain tough strings.

5. CALCULATION AND EXPRESSION OF RESULTS

$$\%\ m/m \text{ pods containing tough strings} = \frac{\text{pods containing tough strings (g)}}{\text{test sample (g)}} \times 100$$

Figure 1 - Tough String Tester for Green or Wax Beans



C. DETERMINATION OF WASHED DRAINED WEIGHT

1. DEFINITION

For viscous prducts such as gravy, sauce and syrup, washed drained weight expresses % m/m solid contents after washing, with hot water, as determined by the procedure described below.

2. MATERIALS

2.1 Specifications for circular sieves

Fine mesh U.S. sieve No. 50[11] 20 cm (8 inches) diameter. The meshes of such sieves are made by so weaving wire as to form square openings of 0.30 mm by 0.30 mm.

3. PROCEDURE

3.1 Weigh the unopened can.

3.2 Open the can and wash the contents on to a tared fine mesh sieve.

3.3 Wash the contents of the sieve under the running cold water and then wash with running hot water until free of adhering substances.

3.4 Spread the mushrooms after washing over the bottom of the sieve and drain for 5 minutes and then weigh.

3.5 Weigh the empty dried can and determine the net contents (or total product weight).

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[11] To be replaced by the corresponding ISO sieve when ISO international standard is available.

4. CALCULATION AND EXPRESSION OF RESULTS

Calculate the % m/m drained weight on the net contents (or total product weight).

E. DETERMINATION OF WATER CAPACITY OF CONTAINERS

1. SCOPE

This method applies to glass containers.

2. DEFINITION

The water capacity of a container is the volume of distilled water at 20°C which the sealed container will hold when completely filled.

3. PROCEDURE

3.1 Metal containers

3.1.1 Select a container which is undamaged in all respects.

3.1.2 Wash, dry and weigh the empty container after cutting out the lid without removing or altering the height of the double seam.

3.1.3 Fill the container with distilled water at 20°C to 4.8 mm vertical distance below the top level of the container, and weigh the container thus filled.

3.2 Glass containers

3.2.1 Select a container which is undamaged in all respects.

3.2.2 Wash, dry and weigh the empty container.

3.2.3 Fill the container with distilled water at 20°C to the level of the top thereof, and weigh the container thus filled.

4. CALCULATION AND EXPRESSION OF RESULTS

Subtract the weight found in 3.2.2 from the weight found in 3.2.3. The difference shall be considered to be the weight of water required to fill the container. Results are expressed as ml of water.

F. METHOD FOR DISTINGUISHING TYPE OF PEAS

1. DEFINITION

This method is based on differentiation between starch granules of the wrinkled-seeded types and starch granules of the smooth-seeded types.

2. REAGENTS AND MATERIALS

2.l Compound microscope - 100 to 250 magnification.
- Phase contrast.

2.2 Microscope slide and cover glass.

2.3 Spatula.

2.4 Ethanol - 95% v/v.

2.5 Glycerine.

3. PROCEDURE

3.l Preparing mount

3.1.1 Remove a small portion of the endosperm and place on glass slide;

3.1.2 Using a spatula grind the material with 95% v/v ethanol;

3 l.3 Add a drop of glycerine, place cover glass on material and examine under microscope.

3.2 Identification

Starch granules of the wrinkled-seeded types (garden peas, sweet) show up as clear cut, well defined, generally spherical particles.

Starch granules of the smooth-seeded types (round, early, Continental) show up as an amorphous mass with no well defined geometric shape.

G. DETERMINATION OF MINERAL IMPURITIES (SAND TEST) IN RAISIN

1. PRINCIPLE OF METHOD

Because of harvesting and drying methods, raisins are exposed to potential contamination by sand or particles of soil. The objective of the "sand test" is to separate sand and similar inorganic material from the raisin material through a combination of screens, agitation and water spray. After the sand has been separated from the raisin tissue, it is collected on a fine mesh screen, transferred to a crucible, incinerated to eliminate any organic matter and then weighed. A large test sample is used in order to provide a representative cross section on the product and also provide sufficient "mineral impurities" or sand.

2. MATERIALS

- Beakers - Pyrex - 2,000 ml.
- Beakers - 800 ml.
- Hot Plate or Stove
- Muffle - 550° to 600°C
- Crucibles for incineration of residue
- Screens - 20 cm (8 inch) diameter - 8 mesh; pore openings 2.38 mm.
- Screens - 20 cm (8 inch) diameter - 24-25 mesh; pore openings 0.70 mm.
- Screens 20 cm (8 inch) diameter - 250-270 mesh; pore openings 50 µm.

NOTE: The fine 250-270 mesh screen may be reduced to 7.5 to 10.0 cm (3 or 4 inch) diameter with a tapered adapter or funnel to collect washings from 20 cm (8 inch), 24-25 mesh screen.

3. REAGENT

NaCl solution (15%) 15 g NaCl are diluted to 100 ml water.

4. PROCEDURE

4.1 Weigh 200 g of raisins into a 2,000 ml beaker; add 1,000 ml of water.

4.2 Add 5 drops of detergent (a secondary alkyl sulphate or any household detergent), bring to the boil and simmer for about 20 minutes.

4.3. Wash through nested screens with the 8 mesh on top, the 25 mesh in the middle and the 270 mesh on the bottom. Using about one-third of the raisins at a time, use a combination of water spray and vigorous rubbing to break down the tissue and release sand or other earthy material.

4.4 Remove the 8 mesh screen and thoroughly wash the residue on the 25 mesh screen.

4.5 Collect all material that passes through the 25 mesh screen on the 270 mesh sieve.

4.6 Carefully transfer the material remaining on the 270 mesh screen to an 800 ml beaker using a small stream of water.

4.7 Let stand for about 5 minutes permitting the heavier material to settle to the bottom of the beaker and the lighter raisin tissue to float.

4.8 Decant most of the water and the floating raisin material, retaining the heavier sand on the beaker.

4.9 It this point most of the organic material should be eliminated. If there appears to be any appreciable amount in the beaker add about 400 ml of hot 15% NaCl solution, let stand for 5 minutes and again decant the water and the lighter material. Remove NaCl by washing with hot water. Removal can be verified by testing the washings with $AgNO_3$.

4.10 Filter the residue remaining in the beaker through a fast ashless filter and transfer to a tared crucible.

4.11 Dry and ignite in muffle at 550°-600°C for about 2 hours.

4.12 Cool and weigh residue.

5. EXPRESSION OF RESULTS

Results are expressed as mg/100g of the product.

6. LITERATURE REFERENCE

Adapted from "AOAC 971.33 Residue (Acid-Insoluble)(Soil) in Fruits and Vegetables (Frozen)".

H. DETERMINATION OF MINERAL OIL IN RAISINS

1. PRINCIPLES OF METHOD

Raisins contain a certain amount of natural oil which will be extracted along with mineral oil in a normal solvent extraction procedure. The first step, therefore, if to remove any oil, whether vegetable or mineral, from the product using a suitable solvent such as chloroform. After evaporation of the chloroform, the residue containing the oil is then passed through an alumina column to separate the unsaponifiable mineral oil from vegetable oil based upon the solubility differential between the two oils. The vegetable oil remains attached to the alkaline alumina column whereas the non-polar mineral oil if carried through by petroleum ether. Evaporation of the petroleum ether leaves a residue of unsaponifiable oil which is considered mineral oil after verification of purity using refractive index: value and the Irtran plate spectrum.

2. APPARATUS

- Beakers - 1000 ml, 800 ml, 30-50 ml
- Separating Funnels - 800 ml
- Steam bath
- Filter paper, rapid flow
- Chromatographic tube, 250 ml dispensing burette; or 30 x 450 mm chromatographic tube fitted with stopcock

3. REAGENTS

- 6N HCl (1+1)
- Alumina (Al_2O_3) Brookman Activity I, basic, 80-200 mesh pH 9-11 in 10% aqueous slurry. (Fisher Scientific Co. No. A 540,6-941 J.T. Baker No. (0539)).[12]
- Chloroform, Analytical Grade
- Petroleum ether, Analytical Grade, B.P. 30° - 60°C
- Na_2SO_4 (anhydrous)

4. SAMPLE PREPARATION

4.1 Weigh 200 g of raisins into a 1 litre beaker.

4.2 Add with stirring 50 ml 6N HCl; let stand one hour with occasional stirring.

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[12] The nature of the alumina was found to be important.

4.3 Add 200 ml chloroform to the raisin mass, stir and decant chloroform and aqueous extract into 800 ml beaker; retaining the raisins in the 1000 ml beaker.

4.4 Repeat Step 4.3 extraction two more times using 200 ml portions of chloroform for each extraction.

4.5 Transfer the combined extractions to a separating funnel, allow to stand sufficiently long to separate chloroform and water layers. Draw off the heavier chloroform layer into an 800 ml beaker.

4.6 Add about 100 g of anhydrous Na_2SO_4 to the chloroform extract and decant through a rapid filter into another 800 ml beaker.

4.7 Wash the Na_2SO_4 with a 50 ml portion of chloroform and decant through filter into beaker, combining the chloroform extracts.

4.8 Evaporate to near dryness on a steam bath under a gentle steam of air.

4.9 Transfer residue quantitatively to a 50 ml beaker using small portions of chloroform, again evaporate this time to dryness.

4.10 Dry residue 2 to 3 hours at 100°C and cool.

5. PREPARATION OF ALUMINA COLUMN

5.1 Pack constricted tube of the column with a small wad of glass wool.

5.2 Add through a powder funnel, 175 g of alumina, tapping tube to ensure uniform packing. Level the surface and cover surface with disc cut from rapid filter paper slightly smaller in diameter than inside of tube.

5.3 Pre-wash column with about 200 ml petroleum ether. Just before last of the petroleum ether settles into alumina, stop flow.

6. PROCEDURE

6.1 Take up the dried residue in 5-10 ml petroleum ether.

6.2 Pour carefully into alumina column, open stopcock, and collect eluate at rate “less than" 5 ml/min.

6.3 Close stopcock when ether-oil mixture has settled to just above surface of alumina. Rinse sample beaker with two 5 ml portions petroleum ether, rinsing sides of column with each rinse.

6.4 Open stopcock and let ether settle almost to surface of alumina. Fill column with petroleum ether.

6.5 Continue adding petroleum ether to column until total of 400 ml collects.

6.6 Evaporate petroleum ether to small volume on steam bath, using gentle stream of dry air to aid solvent removal. Stirring rod placed in flask will help prevent superheating and possible boiling over.

6.7 Transfer quantitatively to small weighed beaker, using small portion of petroleum ether.

6.8 Evaporate to dryness on warm surface using gentle stream of air. Dry in convection oven for 1 hour at 100°C.

6.9 Carry out Step 6.5 to 6.8 using a total of 400 ml petroleum ether as a blank determination.

7. CALCULATION AND EXPRESSION OF RESULTS

Calculate the percent by mass of this unsaponifiable oil in relation to the original mass of raisins (200 g).

Calculation:

$$\frac{m_1}{m_2} \times 100 = \frac{\text{mineral oil}}{\text{100 g of the product}}$$

where
m_1 = residue in g
m_2 = mass in g of the sample at 4.1

8. IDENTIFICATION AND PURITY OF MINERAL OIL

8.1 Transfer approximately 2 drops residue oil to face of NaCl or Irtran plate. Cover with another plate and prepare IR spectrum. Prepare similar curve, using USP (or equivalent) mineral oil. If volume of residue oil is too small to transfer to plate directly, transfer with aid of CS_2. Evaporate solvent completely before covering plate with second plate. Peaks occur at 3.4, 6.82 and 7.25 nm.

8.2 Obtain refractive index on another drop or two of residue oil and compare with refractive index of USP (or equivalent) mineral oil read at temperature.

9. LITERATURE REFERENCE

Adapted from "AOAC 966.11 Mineral Oil in Baked Products".

I. DETERMINATION OF INTERNAL DEFECTS (DATES)

Examine each date carefully for internal defects using a strong light. If the dates are pitted, open up the flesh so that the internal cavity can be viewed. If the dates are unpitted, slit the date open so as to expose the pit, remove the pit and examine the pit cavity.

J. DETERMINATION OF ACIDITY (TABLE OLIVES)[13]

Transfer 25 ml of the brine by pipette to a 250 ml conical flask. Titrate the solution with 0.1N sodium hydroxide solution to pH 8.3 using a pH meter. The sodium hydroxide solution may be standardized against dried A.R. grade potassium hydrogen phthalate, and any necessary factor applied.

1 ml 0.1N NaOH = 0.0090 g lactic acid.

K. DETERMINATION OF PH (TABLE OLIVES)[13]

Set up and adjust a pH meter and the glass and calomel electrodes according to the manufacturer's operating instructions for use at 20°C. Calibrate the instrument with a recognized buffer solution of pH 4.0 at 20°C. Rinse the electrodes free from buffer solution with copious amounts of distilled water. Dip the electrode into the brine contained in a beaker and adjusted to 20°C. Read the pH to the nearest 0.05 units.

L. ASCERTAINING PROPORTIONS OF FRUIT(CANNED FRUIT COCKTAIL; CANNED TROPICAL FRUIT SALAD)

1. PROCEDURE

1.1 Determine drained weight and keep liquid and fruit separate.

1.2 Separate individual fruit ingredients, removing those fruits present in lesser amount (such as cherries, pineapple, grapes).

1.3 Weigh the individual fruit ingredients to the nearest gramme.

1.4 Record the weight of each fruit and add all of these weights.

2. CALCULATION AND EXPRESSION OF RESULTS

Calculate the percentage of fruit proportions:

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[13] The presence of acidic food additives affects the interpretation of the results.

$$\frac{\text{weight of each fruit}}{\text{*sum of all fruit weights}} \text{x} 100 = \% \text{ of the weight of each fruit}$$

* Do not use the original drained weight of the product before separation of the fruits.

M. DETERMINATION OF BROKEN, SLABS, DIRTY, MOULDY, DAMAGED AND IMMATURE FRUITS (DRIED APRICOT)

Examine the fruits (sample size: 1 kg) visually and weigh the defective items. Calculate the percentage of defects:

$$\frac{\text{Weight of defective unites}}{\text{Total weight of sample}} x100 = \% \text{ defective}$$

N. DETERMINATION OF VOLUME FILL (BY DISPLACEMENT) (PICKLED CUCUMBER)

METHOD I

(1) This method may be used for all sub-types of pickles. Use a four to eight litre size can with an overflow spout constructed from 0.5 cm to 1 cm inside diameter metal tubing (see figure 1). The tubing is soldered to an opening in the side of the can 2 to 3 cm from the bottom and is bent upward parallel to sides. The tube is bent over and slightly downward from the can at the upper end to form a spout about 4 cm below the top of the can. The lower tip end of the spout is lower than the inside lower curve of the spout (point A). The upper tip end of the spout is higher than the inside lower curve of the spout (point A). The upper tip end of the spout is slightly shorter than the lower tip end of the spout. A brace near the top of the can holds the tubing firmly in place. A woven wire basket made from screen wire with about eight meshes to the inch with a handle is used for lowering the pickle ingredient into the overflow can.

(2) Place overflow can on a level table so that overflow will discharge into a sink. Fill the overflow can with water at room temperature (approximately 20°C or 68°F). Place the empty basket into the filled overflow can.

(3) When overflow ceases, place a beaker or graduated cylinder under the spout.[14]

(4) Remove basket and place drained pickle ingredient (at room temperature) in basket and lower slowly into the overflow can. When overflow ceases, measure the volume of the fluid overflow. The percent volume of pickle ingredient (volume occupied) is calculated as follows:

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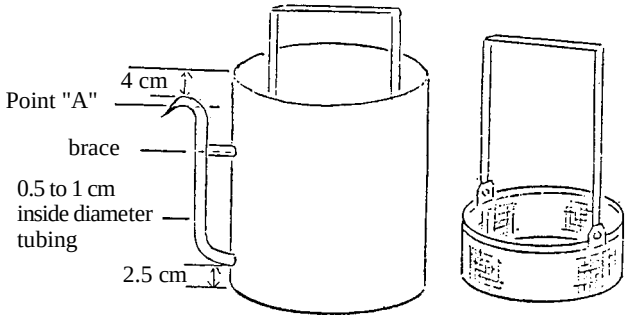
[14] NOTE: Prior to determining the percent volume of pickle ingredient for mustard pickles, the drained pickle ingredient is prepared as follows: Empty the contents of the container upon an ISO Recommendation R 565 or a U.S. Standard No. 8 sieve of proper diameter so as to distribute the product evenly. Wash off all adhering sauce under a spray of water at a temperature of approximately 20°C (680F). Incline the sieve to facilitate drainage and allow to drain for two minutes. Proceed with (4).

$$\frac{\textit{Overflow Volume}}{\textit{Total capacity (volume) of container (see Method E)}} \textit{ x100 = percent volume of pickle ingredient}$$

Figure 1



Overflow can

METHOD 2

(1) Use water to partially fill a graduated cylinder (or other technical measuring device) large enough so that the pickle ingredient from one container may be completely submerged.

(2) Prior to adding the pickle ingredient, record the volume of water in the partially filled cylinder.[15]

(3) Add all the drained pickle ingredient from one container so that it is entirely submerged.

(4) Measure the volume of liquid and pickle ingredient after submersion of pickle ingredient.

(5) Subtract the value in (2) from the value in (4) to obtain the pickle ingredient displacement.

(6) To determine Volume Fill, calculate:

$$\frac{\textit{Pickle Ingredient Displacement}}{\textit{Total Capacity (volume) of Container (see Method E)}} \textit{ x100 = percent volume of pickle ingredient}$$

METHOD 3

(1) Remove and collect the packing medium from the container for other quality determinations - 2.2.3.[16]

(2) With the pickle ingredient in the container fill it to capacity (9.2.5) with water.

(3) Drain, collect and measure the water.

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15 NOTE: Prior to determining the percent volume of pickle ingredient for mustard pickles, the drained pickle ingredient is prepared as follows: Empty the contents of the container upon an ISO Recommendation R 565 or a U.S. Standard No. 8 sieve of proper diameter so as to distribute the product evenly. Wash off all adhering sauce under a spray of water at a temperature of approximately 20°C (68°F). Incline the sieve to facilitate drainage and allow to drain for two minutes. Proceed with (3) above.

16 NOTE: Prior to determining the percent volume of pickle ingredient for mustard pickles, the drained pickle ingredient is prepared as follows: Empty the contents of the container upon an ISO Recommendation R 565 or a U.S. Standard No. 8 sieve of proper diameter so as to distribute the product evenly. Wash off all adhering sauce under a spray of water at a temperature of approximately 20 C (68 F). Incline the sieve to facilitate drainage and allow to drain for two minutes. Proceed with (2) above.

$$\text{percent volume of pickle ingredient} = \frac{V_1 - V_2}{V_1}$$

(4) To determine Volume Fill, calculate:

Where,

V1=Total capacity (volume) of container (Method E); and
V2=Volume of drained water from (3) above

O. DETERMINATION OF SPECIFIC DEFECTS (UNSHELLED PISTACHIO NUTS)

1. DETERMINATION OF CLOSEDNESS

(a) Weigh 500 g of the pistachios and count the number.
(b) Separate all the closed pistachios.
(c) Count the closed pistachios.
(d) Divide the number of closed pistachios by the number of pistachios in the sample to determine the percentage of closedness (x100).

2. DETERMINATION OF EMPTINESS AND UNRIPENESS

(a) Mix the closed pistachios with the rest of the weighed sample.
(b) Open all the pistachios in the sample. Count the empty ones and unripe ones separately.
(c) Divide the number of empty ones and unripe ones by the number of pistachios in the sample to determine the percentage of emptiness and unripeness (x100).

3. DETERMINATION OF PEST AND DISEASE DAMAGE

(a) Examine all the kernels of the above sample individually for pest and disease damaged kernels.
(b) Count the damaged kernels.
(c) Divide the number of pest and disease damaged pistachios by the number of pistachios in the sample to determine the percentage of pest and disease damaged pistachios (x100).

4. SIZE CLASSIFICATION

(a) Weigh 500 g of the above pistachios, the foreign matter of which has been separated.
(b) Count the number of pistachios.
(c) Divide the number of pistachios counted in 500 g by 5 and match the result with the figures in Section 1.4 of Annex for size classification.

P. DETERMINATION OF TOTAL ACIDITY OF EXTRACTED OIL (GRATED DESICCATED COCONUT)

PRINCIPLE

The sample is extracted by ethyl ether at room temperature. The acidity of the extracted oil is determined by titrations with alkali and the results expressed as percent of lauric acid.

REAGENTS

(1) Anhydrous ethyl ether, peroxide free
(2) Ethyl ether and ethyl alcohol 95% (1:2) mixture
(3) Sodium hydroxide 0.1N

PROCEDURE

50 g of the sample is extracted at room temperature in 500 ml Erlenmeyer flask with 300 ml of ethyl ether (reagent 1) for one hour with mechanical agitation. The extract is filtered through Whatman No. 542 filter paper and further undergoes dry evaporation in rotary evaporator with nitrogen flow at the maximum temperature of 40°C.

20 g of the extracted oil is weighed and dissolved with addition of 100 ml of ethyl alcohol mixture (Reagent 2) and further titrated with 0.1N sodium hydroxide (Reagent 3) to pH 8.3 using a pH meter.

EXPRESSION OF RESULTS

Acidity is calculated as below:

$$\text{Acidity} = \frac{VxNx20}{m}$$

V = Volume (ml) of NaOH
N = Normality of NaOH solution
m = mass of the sample in grammes

The results, as obtained above, are expressed in percent lauric acid m/m.

Q. DETERMINATION OF EXTRANEOUS VEGETABLE MATTER (GRATED DESICCATED COCONUT)

The determination is carried out by spreading 100 g of the sample in a thin layer against a white background and counting the extraneous material with the naked eye.

ANNEX II

METHODS OF SAMPLING

Although sampling matters are outside of the scope of this paper and that the CCMAS is elaborating sampling plans which should be applicable to all Codex commodity standards, it is worthwhile to note the following:

- Except for the Standard for Grated Desiccated Coconut, all the other standards use the Codex Sampling Plans for Pre-packaged Foods (AQL 6.5)(1969)[17] as amended in 1981[18];
- Standard for Grated Desiccated Coconut uses ISO 2170-1980 (Cereals and Pulses) or ICC Method of Sampling No. 101-1960, and Instructions on Codex Sampling Procedures[19];
- A number of standards include some additional sampling instructions/information in the text of the standards, which are extracted here.

A. CANNED FRUIT COCKTAILS

SIZE OF SAMPLE UNIT

1. For ascertaining proportions of fruits and fill of container (including drained weight) the entire container shall be the sample unit.

2. For ascertaining compliance with percentage requirements for size and shape of fruits and defects, the sample unit shall be:

(a) the entire container when it holds 1 litre or less; or

(b) 500 g of drained fruit (of a representative mixture) when the container holds more than 1 litre.

B. CANNED MANGO

for net weight, analytical requirements - need to be elaborated.

SIZE OF SAMPLE UNIT

1. For ascertaining fill of container and drained weight the sample unit shall be the entire container.

2. For ascertaining compliance with the requirements for styles and defects the sample unit shall be:

(a) the entire container when it holds 1 litre or less; or

(b) 500 g of drained fruits (of a representative mixture) when the container holds more than 1 litre.

C. CANNED PINEAPPLE

SIZE OF SAMPLE UNIT

1. In ascertaining the quality requirements for all styles other than Tidbits, Cubes, Crushed or ships styles, the entire container shall be the sample unit.

2. In ascertaining the quality requirements for Tidbits, Cubes, Crushed or Chips styles, the sample unit shall be:

(a) the entire container when it holds 1.0 litre or less; or

(b) 500 g of drained fruits (of a representative mixture) when the container holds more than 1.0 litre.

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17 Codex Alimentarius Volume 13, pp. 115-125. Formerly CAC/RM 42-1969.
18 ALINORM 83/20, pp. 44-45.
19 CX/MAS 1-1987.

D. DATES

1. GROSS SAMPLE

Select at random not less than 2 individual packages per each 1,000 kg portion of the lot. From each individual package draw a sample of 300 g and in any case sufficient to obtain a gross sample of not less than 3,000 g. Use the gross sample for checking carefully for live infestation and general cleanliness of the product prior to its examination for compliance with other provisions of the standard.

2. SUB-SAMPLES FOR EXAMINATION AND TESTING

Mix the gross sample well and take small quantities at random from many different places as follows:

For moisture test	- 500 g
For pits (in pitted style)	- 100 dates
For specified defects and size requirements	- 100 dates

E. GRATED DESICCATED COCONUT

1. Instructions for drawing primary samples according to ISO 2170-1980 (Cereals and Pulses) or ICC Method of Sampling No. 101-1960 (Sampling of Milled Products).

2. The size of the sample to be undertaken from a homogeneous lot should be in accordance with Table 3 of the Instructions on Codex Sampling procedures (CX/MAS 1-1987, Appendix V).

3. For all determinations the laboratory sample should be prepared according to the variables plan for proportion defective (CX/MAS 1-1987, Appendix IV)

4. For all determinations, except granularity, analysis should be performed on the "blended bulk sample".

5. For verification of granularity, i.e., size grade as declared on the label, the determinations in consignments of prepackaged products should be on individual packages.

F. MANGO CHUTNEY

For net contents, analytical requirements - need to be elaborated

G. UNSHELLED PISTACHIO NUTS

1. GROSS SAMPLING

Select at random not less than 2 individual packages per each 1000 kg portion of the lot. From each individual package draw a sample of 150 g and in any case, sufficient to obtain a gross sample of not less than 1500 g. Where the product is packed in bulk containers select at random from various parts of the containers and per each portion of 1000 kg of the lot, not less than two samples of 150 g, and in any case sufficient to obtain a gross sample of not less than 1500 g. Use the gross sample for checking carefully for live infestation, mouldy pistachios and general cleanliness of the product prior to its examination for compliance with other provisions of the standard.

2. SUB-SAMPLES FOR EXAMINATION AND TESTING

Mix the gross sample well and take small quantities at random from many different places as follows:

(a) Moisture Test - 50 g
(b) General Requirements - 500 g
(c) Specific Defects - 600 g