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codex alimentarius commission



FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS WORLD HEALTH ORGANIZATION



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Agenda Item 6(a)

CX/PFV 04/22/11 September 2004

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

22nd Session, Washington, DC metro area, U.S.A., 27 September – 1 October 2004

METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

BACKGROUND

CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

21st Session of the Codex Committee on Processed Fruits and Vegetables

1. The 21st Session of the CCPFV (September 2002) considered a number of methods of analysis for endorsement by the 24th Session of CCMAS (November 2002). The Committee endorsed the following recommendations of the Working Group on Methods of Analysis and Sampling intended at reducing the time spent on this matter, facilitating their submission to CCMAS for endorsement, accelerating the process of final adoption of the draft Standards at Step 8, and inclusion of the new methods in Volume 13 of the Codex Alimentarius:

- (a) When a proposed draft Standard or a revised draft Standard is created by the CCPFV, the working group on the project should clearly state the methods of analysis required. This would include both the analysis required for the item and also recommended methods to use.
- (b) When defining the methods to use, the group should include both an ISO method and an AOAC method. The option to use either of these methods would make the Standard more universally acceptable and also easier to use. The exact analysis required and the suggested method of analysis should be included in the draft Standard in the suitable Section.

2. The Committee agreed to forward methods of analysis for processed fruits and vegetables as contained in Appendix VI Parts A, B, and D of ALINORM 03/27 (Report of the 21st Session of the CCPFV) to the 24th Session of CCMAS as follows:

- (a) <u>Appendix VI-Part A</u>: methods of analysis for processed fruits and vegetables in general;
- (b) <u>Appendix VI-Part B</u>: methods of analysis for certain processed fruit and vegetable products namely: aqueous coconut products, canned stone fruits, and pickled products; and,
- (c) <u>Appendix VI-Part D</u>: Codex Recommended Methods (CAC/RMs) for their replacement with compatible references referring to more modern methods and/or their revision/updating as recommended by CCMAS (see para. 3).

Part C of Appendix VI regarding methods of analysis and sampling for those processed fruits and vegetables under consideration at Step 3 namely: proposed draft Codex standards for canned tomatoes; processed tomato concentrates; canned vegetables; jams, jellies, and marmalades; soy sauce; and canned citrus fruits were not considered by CCMAS as they were not yet discussed by the CCPFV. The same applies to those Codex Recommended Methods (CAC/RMs) for the above-mentioned commodities contained in Part D of Appendix VI.

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

20th Session of the Codex Committee on Methods of Analysis and Sampling

3. The 20th Session of CCMAS (October 1995) advised the commodity committees to consider replacing Codex Methods of Analysis and Sampling (CAC/RMs) with more modern methods as appropriate and to replace the CAC/RM numbers with the original literature references, if possible¹. The 21st CCMAS further recommended that when the original reference of a CAC/RM was available, this reference should replace the CAC/RM number, and when the original reference was not available, the full text of the method should be included in *Codex Alimentarius* Volume 13 and the CAC/RM number reference deleted². The Codex Alimentarius Commission at its 22nd Session agreed to the abolition of the CAC/RM Numbering System as recommended by CCMAS³.

23rd Sessions of the Codex Committee on Methods of Analysis and Sampling

4. The 23rd Session of CCMAS (March 2001) noted that it would not be procedurally correct to endorse a method before relevant Codex provisions had been established⁴. In view of this, those methods of analysis corresponding to products which are not being considered by the CCPFV have been deleted from the previous list agreed to by the 20th session of the CCPFV. They will be presented to the Committee as new work is undertaken on the commodities they apply to in the subsequent sessions of the CCPFV.

24th Session of the Codex Committee on Methods of Analysis and Sampling

Processed Fruits and Vegetables

5. The 24th Session of CCMAS (February 2002) asked for clarification from the Committee on the provision and/or commodity concerned by the determination of pH and sulphites. It was noted that a general method for sulphites had been endorsed and that it applied to processed fruits and vegetables. The Committee also recommended that the Commodity Committee consider ISO 1842:1991 as it was specific for pH in processed fruits and vegetables, if the determination of pH was required in a standard under consideration.

6. The Committee asked for clarification on the amendment proposed to AOAC 968.30 for the determination of drained weight, and on how sections 2.1 and 2.2 should be amended.

Aqueous Coconut Products - Coconut Cream and Coconut Milk

7. The Committee did not endorse the methods for moisture, non-fat solids, total fat and total solids for aqueous coconut products as the methods applied to milk.

Pickled Products

8. The Committee deleted the methods for acidity, salt and drained weight for pickles as no relevant provisions existed in the Draft Standard. It recalled that the method proposed as Type IV for lead was temporarily endorsed since 1998 and asked the Commodity Committee whether this method was necessary since a general Codex method already existed as Type II. As regards the determination of benzoic acid and sorbates, it was recommended that the Committee consider more modern methods (liquid chromatography) such as NMKL 124 (1997).

25th Session of the Codex Committee on Methods of Analysis and Sampling

General Guidelines on Sampling

9. The 25th Session of CCMAS (March 2004) agreed to advance the draft Guidelines on Sampling to Step 8 for adoption by the 27th Session of the Codex Alimentarius Commission (July 2004). In addition, it was agreed that the draft Guidelines, when adopted, would replace the current Sampling Plans for Prepackaged Foods (AQL 6.5) (CODEX/STAN 233-1969)⁵.

¹ ALINORM 97/23, para. 52.

² ALINORM 97/23A, para. 44.

³ ALINORM 97/37, para. 145.

⁴ ALINORM 01/23, para. 87.

⁵ ALINORM 04/27/23, para. 15 and Appendix III.

10. The 27^{th} Session of the Commission (July 2004) adopted the draft Guidelines on Sampling at Step 8 and agreed that Sampling Plans for Prepackaged Foods (AQL 6.5) (CODEX/STAN 233-1969) should be replaced by the General Guidelines⁶.

11. The Committee is invited to replace the reference to Sampling Plans for Prepackaged Foods (AQL 6.5) (CODEX/STAN 233-1969) in the Codex standards for processed fruits and vegetables to the Codex General Guidelines on Sampling.

The Use of Analytical Results: Sampling Plans, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards

12. The 25th Session of CCMAS (March 2004) recalled that it had agreed to consider the Guidelines redrafted by the *Ad Hoc* Working Group under Agenda Item 12 "Other Business and Future Work"⁷.

13. The Chairperson of the *Ad Hoc* Working Group presented the document and indicated that the text was substantively revised and it should give simple instructions to Commodity Committees regarding sampling, relationship between the analytical results, the measurement uncertainty, recovery factors and provisions in the Codex standards.

14. The Committee amended the title to reflect the view that the use of analytical results related not to sampling as such but rather to sampling plans.

15. The Delegation of New Zealand was of the view that there was a contradiction between the first and last paragraphs of the section on Issues Involved and this required further rewording for clarification purposes. It proposed several substantial amendments as follows. The section on Measurement Uncertainty was not sufficiently general, as this was not the only way to proceed, and should be addressed in a separate paper. The sampling plan should specify whether the specification applied to the average in a lot or the proportion of non-conforming; significant figures should not be addressed in the document as this question relates to reporting not to the use in conformity assessment.

16. The Committee amended the first paragraph in the Recommendations section to clarify that when Commodity Committees discuss and agree on a specification, the concerned analytical methods should also be stated.

17. Different views were expressed regarding the section on Recovery. The Delegation of Ireland drew the attention of the Committee to the fact that recovery was relevant to organic analysis especially when low levels were analysed and proposed to amend the sentence so that the analytical results are to be reported on recovery "where relevant and appropriate".

18. Some delegations proposed to delete this section while other delegations were of the view that the two first sentences from the earlier version of the document better reflected recommendations regarding recovery. The Committee agreed to amend this section as proposed by the Delegation of Ireland and retained it in square brackets for further discussion.

19. It was proposed to forward the document to the Committee on General Principles for their endorsement and subsequent adoption by the Commission and inclusion in the Procedural Manual as guidance to the Codex Commodity Committees. However the Committee noted that although the document was substantively improved, several issues remained to be addressed and there was a need for further consideration therefore decided to request comments on the current version and consider it at the next session of the Committee (see Appendix III to this document). It also agreed that the advice of Commodity Committee would be sought on this document.

20. As requested by CCMAS, the Committee is invited to provide its comments on the Use of Analytical Results: Sampling Plans, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards. For easy of reference, the document is reproduced in Appendix III.

⁶ ALINORM 04/27/41, Appendix V.

⁷ ALINORM 04/27/23, paras. 6 and 13.

CONSIDERATION OF METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

21. Attached is the list of methods of analysis and sampling being recommended for inclusion in processed fruits and vegetables. They are distributed as follows:

- (a) Appendix I:
 - <u>Part I:</u> Methods of analysis and sampling requiring further clarification;
 - <u>Part II</u>: Methods of analysis for those processed fruits and vegetables under study by the Committee (pickled products; processed tomato concentrates; canned tomatoes; canned vegetables; jams, jellies and marmalades; soy sauce; and canned citrus fruits) as proposed by 21st Session of the Committee;
- (b) Appendix II:
 - <u>Part I:</u> Codex Recommended Methods (CAC/RMs) requiring further clarification;
 - <u>Part II</u>: Codex Recommended Methods for those processed fruits and vegetables under study by the Committee (pickled products; processed tomato concentrates; canned tomatoes; canned vegetables; jams, jellies and marmalades; soy sauce; and canned citrus fruits) as proposed by the 21st Session of the Committee;
- (c) **Appendix III**: Use of Analytical Results: Sampling Plans, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards.

22. When considering methods of analysis, the Committee should give due consideration to the provisions contained in the Procedural Manual of the Codex Alimentarius Commission in particular the *General Criteria for the Selection of Methods of Analysis* as set out in the *Principles for the Establishment of Codex Methods of Analysis*⁸ and the *Relations between Commodity Committees and General Committees (Methods of Analysis and Sampling*⁹.

23. It is also noted that 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.

24. A separate document CX/PFV 04/22/11-Add.1 containing methods of analysis and sampling for certain processed fruits and vegetables and individual commodities falling within this category, including Codex methods of analysis and sampling (CAC/RMs), in force up to date will be presented as a follow-up on the status of the endorsement process vis-à-vis the work of the CCPFV.

ACTIONS TO BE TAKEN BY THE CCPFV

25. The Committee is invited to revise the methods of analysis listed in Appendices I and II Parts 1 and 2 and:

- (a) propose methods of analysis for the combinations of standard/provision (specification and/or labelling requirement) requiring them. In doing so, the Committee should clearly indicate if the revision corresponds to an update of the reference or to a new method which replaces the current one in force;
- (b) provide further clarification on those methods of analysis which were temporarily endorsed or not endorsed by CCMAS (see paras. 5, 6, 7 and 8);
- (c) identify which CAC/RMs should be deleted or replaced by the original reference available and report to CCMAS accordingly;
- (d) incorporate the methods of analysis into the relevant standards under study.

The methods agreed to be used for the revised standards will need to be submitted to CCMAS for endorsement and will supersede the methods currently in force for the products to which they apply.

⁸ Procedural Manual of the Codex Alimentarius Commission, 13th Edition, pages 73-82.

⁹ Procedural Manual of the Codex Alimentarius Commission, 13th Edition, pages 97-98.

METHODS OF ANALYSIS AND SAMPLING REQUIRING FURTHER CLARIFICATION 24th CCMAS (November 2002), ALINORM 03/23, Appendix VI/H.

1. General Methods of Analysis for Processed Fruits and Vegetables

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Processed fruits and vegetables	рН	AOAC 981.12 ISO 11289:1993	Potentiometry	The CCPFV should identify the provisions and the standards concerned and consider ISO 1842:1991 for processed fruits and vegetables. See also para. 5 of CX/PFV 04/22/11.	IV	NE

2. Methods of Analysis for specific commodities: Aqueous Coconut Products and Pickled Products

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Aqueous Coconut Products	Moisture	Subtracting total solids from 100	Calculation	See para. 7 of CX/PFV 04/22/11.		NE
Aqueous Coconut Products	Non-fat solids	Subtracting total fats from total solids	Calculation	See para. 7 of CX/PFV 04/22/11.		NE
Aqueous Coconut Products	Total fats	AOAC 989.05, IDF/AOAC method to be checked	Ether extraction	This method applies to milk and the CCPFV should clarify whether it is applicable to coconut products. See also para. 7 of CX/PFV 04/22/11.		NE
Aqueous Coconut Products	Total solids	AOAC 990.20	Oven extraction	This method applies to milk and the CCPFV should clarify whether it is applicable to coconut products. See also para. 7 of CX/PFV 04/22/11.		NE
Aqueous Coconut Products	Sampling	CAC/GL 50-2004				

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Pickles	Benzoic acid	NMKL 103 (1984) AOAC 983.16	Gas Chromatography	The CCPFV should consider more modern methods (LC method) such as NMKL124 (1997). See also para. 8 of CX/PFV 04/22/11.	II	Е
Pickles	Lead	ISO 6633:1984	Flameless atomic absorption spectrophotometry	The CCMAS recalled that the method proposed as Type IV for lead was temporarily endorsed since 1998 ¹ and asked the CCPFV whether this method was necessary since a general Codex method AOAC 972.25 ¹ already existed as Type II.	IV	TE
Pickles	Sorbate	NMKL 103 (1984) AOAC 983.16	Gas Chromatography	The CCPFV should consider more modern methods (LC method) such as NMKL124 (1997).	II	Е
Pickles	Sulphur Dioxide			See General Method for sulphites* (food additives/processed fruits and vegetables).		
Pickles	Tin ≤ 250.0 mg/kg	ISO 2447:1998	Spectrophotometry	The CCPFV should consider using the General Codex Method AOAC 980.19 and clarify why this method is proposed.		NE

¹ 22nd CCMAS, November 1998, ALINORM 99/23, App. III Part 1/B.

In addition, the CCMAS deleted the methods for the determination of <u>acidity</u>, <u>salt</u>, and <u>drained weight</u> as these provisions are not specified in the draft Standard for Pickles.

COMMODITY	PROVISION	МЕТНОД	PRINCIPLE	Note	Туре	Status
Pickles	Acidity	AOAC 942.15	Titrimetry	The 21 st CCPFV repealed ISO 750:1981 in view of the decision of CCMAS that there can only be one Type I method for the same provision ² . The 22 nd CCMAS endorsed ¹ AOAC 942.15 as Type I. The 24 th CCMAS deleted this methods as no relevant provisions existed in the draft Standard.	Ι	DELETED
Pickles	Drained weight	AOAC 968.30	Gravimetry	The 22 nd CCMAS endorsed this methods as Type I ¹ . The 24 th CCMAS deleted this methods as no relevant provisions existed in the draft Standard.	Ι	DELETED
Pickles	Salt	AOAC 971.27 (Codex General Method)	Potentiometry (Determination of chloride, expressed as sodium chloride)	The 22 nd CCMAS endorsed ¹ this methods as Type II. The 24 th CCMAS deleted this methods as no relevant provisions existed in the draft Standard.	Π	DELETED
Pickles	Salt	AOAC 939.10	Volumetry, gravimetry, titrimetry (3 methods) (Determination of chloride, expressed as sodium chloride)	The 22 nd CCMAS endorsed ¹ this methods as Type III. The 24 th CCMAS deleted this methods as no relevant provisions existed in the draft Standard.	III	DELETED

² ALINORM 03/27, Appendix VI-Part B.

* <u>Sulphites</u>

Codex Committee on Food Additives and Contaminants

Food Additives

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Individual Foods ³	Sulphites	EN 1988-1 : 1998-02 AOAC 990.28	Part 1: Optimized Monier-Williams method		III	Е
Individual Foods ⁴	Sulphites	EN 1988-2:1998 -02 NMKL 135 (1990)	Part 2: Enzymatic method		III	Е

Codex Committee on Processed Fruits and Vegetables

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Processed fruits and vegetables	Sulphites	EN 1988-1: 1998-02 AOAC 990.28	Optimized Monier- Williams method	General method for sulphites as endorsed for food additives (see CCFAC/Food Additives above). See also para. 5 of CX/PFV 04/22/11.	III	Е

³

Hominy, fruit juice, sea food Wine, dried apples, lemon juice, potato flakes, sultanas, beer 4

NOTE: In view of the adoption of the General Guidelines on Sampling and the consequent revocation of CX/STAN 233-1999, the CCPFV is invited to clarify whether the additional provisions to CX/STAN 233-1999 in the Codex Standard for Kimchi still applies and if so to submit to CCMAS for endorsement.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Туре	Status
Kimchi	Sampling		 CODEX STAN 233-1969 CAC/GL 50-2004 [In addition, the following applies: (a) Samples shall be taken and stored in a protected cool place - from 0°C to 4°C so as to prevent deterioration of the sample. (b) Precautions shall be taken to protect the sample, the material being sampled, the sampling instruments, and the sample containers from extraneous contamination. (c) The sample shall be placed in clean dry glass containers with air tight stoppers or closures. It shall be marked with full details of sampling, date of sampling, name of the vendor and other particulars of the consignment.] 	This provision was endorsed ⁵ by the 22 nd CCMAS (November 1998). However, in view of the revocation of CX/STAN 233-1969 and the adoption of the newly CAC/GL 50- 2004 General Guidelines on Sampling, the CCPFV should clarify whether the additional provision still need to remain in addition to CAC/GL 50-2004.		

⁵ 22nd CCMAS, November 1998, ALINORM 99/23, App. III Part 2/A.

METHODS OF ANALYSIS AND SAMPLING FOR CERTAIN PROCESSED FRUITS AND VEGETABLES¹

STANDARD	PROVISION	Level	METHOD	PRINCIPLE	Туре	Status	Note
Canned tomatoes	Mould count	Not detected	AOAC 965.41	Howard mould count	Ι	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).
Canned tomatoes	Calcium		AOAC 968.31	Complexometry Titrimetry	Π	Ε	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 24 th CCMAS endorsed AOAC 968.31 (Type II) as a general method for the determination of calcium in processed fruits and vegetables ² . This method replaces CAC/RM 38-1970.
Canned vegetables (green peas)	Alcohol insoluble solids	≤ 21 %	AOAC 938.10	Sieving	Ι	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 21 st CCPFV agreed to recommend CCMAS to replace CAC/RM 47-1972 with AOAC 938.10 (see CX/PFV 04/22/11-App. II-Part II).

¹ These methods of analysis and sampling correspond to those standards that are being considered at Step 4 by the 22^{nd} Session of the Committee. Most of the methods were proposed at the 21^{st} Session of the CCPFV as contained in ALINORM 03/23, Appendix VI-C. A few methods have been introduced as they were missing from Appendic VI-C but contained in CX/STAN 234-1999 List of Methods of Analysis and Sampling in force in Codex standards.

² ALINORM 03/23, Appendix VI/H.

STANDARD	PROVISION	Level	METHOD	PRINCIPLE	Туре	Status	Note
Canned vegetables (green peas)	Calcium		AOAC 968.31	Complexometry Titrimetry	II	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).
							The 24 th CCMAS endorsed AOAC 968.31 (Type II) as a general method for the determination of calcium in processed fruits and vegetables ² . This method replaces CAC/RM 38-1970.
Canned vegetables (mature processed	Total solids	\geq 19.5% of the weight of distilled	AOAC 964.22	Vacuum oven	Ι	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).
peas)		water at 20°C which the sealed container will hold when completely filled					The 24 th CCMAS endorsed AOAC 920.51 (Gravimetry, Type I) as a general method for the determination of total solids in processed fruits and vegetables ² .
Canned vegetables (palmito)	Mineral impurities	≤0.1% m/m	ISO 762:1982 (confirmed 1992)	Gravimetry	Ι	E	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).
							The 21 st CCPFV agreed to recommend CCMAS to replace CAC/RM 49-1972 with AOAC 971.33 for the determination of mineral impurities (sand) (see CX/PFV 04/22/11-App. II-Part II).
Citrus marmalade	Calcium		AOAC 968.31	Complexometry Titrimetry	II	E	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).
							The 24 th CCMAS endorsed AOAC 968.31 (Type II) as a general method for the determination of calcium in processed fruits and vegetables ² . This method replaces CAC/RM 38-1970.

STANDARD	PROVISION	Level	METHOD	PRINCIPLE	Туре	Status	Note
Jam, jellies and marmalades (jam (fruit preserves) and jellies	Calcium		AOAC 968.31	Complexometry Titrimetry	Π	Ε	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 24 th CCMAS endorsed AOAC 968.31 (Type II) as a general method for the determination of calcium in processed fruits and vegetables ² . This method replaces CAC/RM 38-1970.
Jam, jellies and marmalades (jam (fruit preserves) and jellies	Mineral impurities	≤ 0.04 % (m/m)	AOAC 971.33	Ashing	Ι	E	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 21 st CCPFV agreed to recommend CCMAS to replace CAC/RM 49-1972 with AOAC 971.33 for the determination of mineral impurities (sand) (see CX/PFV 04/22/11-App. II-Part II).
Processed tomato concentrates	Mineral impurities	< 60 mg/kg based on diluted product of 8% solids	AOAC 971.33	Ashing	IV	Ε	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 21 st CCPFV agreed to recommend CCMAS to replace CAC/RM 49-1972 with AOAC 971.33 for the determination of mineral impurities (sand) (see CX/PFV 04/22/11-App. IV).
Processed tomato concentrates	Mould Count		AOAC 945.90	[]	[]		Proposed by the Drafting Group on Processed Tomato Concentrates/Canned Tomatoes.

STANDARD	PROVISION	Level	METHOD	PRINCIPLE	Туре	Status	Note
Processed tomato concentrates	Sodium chlorine		AOAC 971.27 (Codex General Method)	Potentiometry	Ι	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1). The 24 th CCMAS endorsed ISO 3634:1979 (Potentiometry, Type III) as a general method for the determination of sodium chlorine in processed fruits and vegetables ^{2.}
Processed tomato concentrates	Tomato soluble solids	≥ 8%	AOAC 970.59	Refractometry	Ι	Е	Already in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1).

CODEX RECOMMENDED METHODS (CAC/RMs) REQUIRING FURTHER CLARIFICATION 24th CCMAS (November 2002), ALINORM 03/23, Appendix VI/H.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Туре	Status	Note
Processed fruits and vegetables	Determinatio n of Drained Weight - Method I	CAC/RM 36- 1970 should be replaced by AOAC 968.30 plus some modification to the AOAC method	Weighing		NE	 The 21st CCPFV (September 2002) agreed to the following for endorsement by CCMAS: (a) Replaces CAC/RM 36-1970. (b) The following changes are proposed to the AOAC method: 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. Revise second sentence of Section 3. Procedure to read: Without shifting the contents, so incline the sieve approximately 20° from the horizontal to facilitate drainage Insert new sentence at the end of the paragraph: "This determination should be performed at 20°C ±5°C." 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 AOAC 968.30 has been endorsed by CCMAS as a Type I Method for the determination of drained weight in Codex standards for processed fruits and vegetables (CX/STAN 234/1999, see CX/PFV 04/22/11-Add.1) including the Codex Standards for Bamboo Shoots¹ and Kimchi² and in the draft Codex Standard for Pikeled Products². The CCPFV should provide clarification on whether: (a) CAC/RM 36-1970 should be replaced by AOAC 968.30 as a general Codex method for the determination of drained weight in processed fruits and vegetables as recommended by the 20th CCMAS (see para. 3) and consequently, replace CAC/RM 36-1970 by AOAC 968.30 or (b) CAC/RM 36-1970 should be replaced by AOAC 968.30 and, in addition, AOAC 968.30 or should be amended as proposed by the 21st CCPFV. If so, the CCPFV should provide clarification on by the 21st CCPFV. Should provide clarification on how sections 2.1 and 2.2 should be amended.

¹

^{21&}lt;sup>st</sup> CCMAS, March 1997, ALINORM 97/23A, App. V-Part 2/D. 22nd CCMAS, November 1998, ALINORM 99/23, App. III Part 1/B 2

METHODS OF ANALYSIS PREVIOUSLY RECOMMENDED AS CAC/RMS OR STATED IN THE STANDARDS CAC/RM 36/1970 DETERMINATION OF DRAINED WEIGHT

METHOD I - (BASED ON AOAC METHOD)

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 2.8 mm by 2.8 mm³.

3. **PROCEDURE**

Weight full can, open, and pour entire contents on circular sieve for which a tare has been established. Without shifting product, incline sieve so as to facilitate drainage. Drain 2 minutes, weight either drained solids or free liquid direct, and weight dry empty can.

4. CALCULATION AND EXPRESSION OF RESULTS

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

5. LITERATURE REFERENCE

AOAC (1965), 30.001: Drained weight.

³ Ref. ISO Recommendation R 565; such sieves may be replaced by US sieves with No 8 Standard screen (size of opening 2.38 mm)

CODEX RECOMMENDED METHODS (CAC/RMS) FOR CERTAIN PROCESSED FRUITS AND VEGETABLES¹

CAC/RM Reference	Method	Current Reference	21 st CCPFV Recommendations	Note
Reference CAC/RM 37-1970	Determination of Drained Weight - Method II	-	 Recommendations Add to the method title (for Canned Tomatoes Only) Include the following text changes as recommended by the Working Group on Methods of Analysis and Sampling: 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. Revise third sentence of Section 3. Procedure to read: Without shifting the contents, so incline the sieve approximately 20° from the horizontal to facilitate drainage Insert new sentence at the end of the paragraph: "This determination should be performed at 20°C ±5°C." 	The 3 first indents have been already introduced in CAC/RM 37- 1970.
			- 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.	

¹ These Codex Recommended Methods correspond to those standards that are being considered at Step 4 by the 22nd Session of the Committee. The methods were revised at the 21st Session of the CCPFV (ALINORM 03/23, Appendix VI-D) in response to the request of CCMAS "...to consider replacing Codex Methods of Analysis and Sampling (CAC/RMs) with more modern methods as appropriate and to replace the CAC/RM numbers with the original literature references, if possible..." (see para. 3 of CX/PFV 04/22/11).

CAC/RM Reference	Method	Current Reference	21 st CCPFV Recommendations	Note
CAC/RM 39-1970	Tough String Test	-	This will remain the same until the French Method is reviewed.	
			Text for French Method which has not appeared in previous literature for review by CCPFV and CCMAS is as follows:	
			The percentage of tough string beans is determined on the drained weight of the product.	
			For containers \leq 850ml all beans must be tested.	
			For containers > 850ml, the test will be made on 500g of drained beans.	
			Each bean will be broken in its middle, between two fingers.	
			Keep only the beans when appears a tough string longer than 3cm.	
			Weigh the beans for which a tough string has been detected.	
			Calculate the percentages of the tough string beans in relation to the drained weight.	
CAC/RM 45-1972	Determination of Proper Fill in lieu of Drained Weight	-	Add to the method title (for Canned Peas Only).	This amendment has been already introduced in CAC/RM 45-1972
CAC/RM 47-1972	Determination of Alcohol Insoluble Solids	AOAC 938.10	Remove reference CAC/RM 47-1972 and replace with current reference AOAC 938.10.	AOAC 938.10 is already contained in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1) for canned green peas (Type II).
CAC/RM 48-1972	Method of Distinguishing Type of Peas	-	Retain.	

18 Appendix II-Part II

CAC/RM Reference	Method	Current Reference	21 st CCPFV Recommendations	Note
CAC/RM 49-1972	Determination of Mineral Impurities (Sand)	AOAC 971.33	Remove reference CAC/RM 49-1972 and replace with current reference AOAC 971.33.	AOAC 971.33 is already contained in CX/STAN 234/1999 (see CX/PFV 04/22/11-Add.1) for jams, jellies, and marmalades and processed tomato concentrates (Type I and IV respectively).

APPENDIX II-PART II METHODS OF ANALYSIS PREVIOUSLY RECOMMENDED AS CAC/RMS OR STATED IN THE STANDARDS CAC/RM 37/1970 DETERMINATION OF DRAINED WEIGHT (for canned tomatoes only)

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 lbs) 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^2 .

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 20° 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 $20^{\circ}C \pm 5^{\circ}C$

4. CALCULATION AND EXPRESSION OF RESULTS

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

2

Ref. ISO Recommendation R 565; such sieves may be replaced by US sieves 2 mesh (size of opening 11.3 mm).

CAC/RM 39-1970 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 <u>Weighted clamp</u>

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.

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 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



CAC/RM 45-1972

DETERMINATION OF PROPER FILL IN LIEU OF DRAINED WEIGHT (for canned peas only)

1. DEFINITION

The method for determination of proper fill is an alternative method for determining a fill of canned peas in lieu of the drained weight.

2. **PROCEDURE**

2.1 Pour the contents of one container into an empty container of the same kind and size and return the contents completely to its original container.

2.2 Level off the contents thus returned irrespective of the quantity of liquid 15 seconds after the contents are so returned.

3. EXPRESSION OF RESULTS

3.1 A container with lid attached by double seam shall be considered to be completely filled when it is filled to the level 4.8 mm vertical distance below the top of the double seam.

3.2 A glass container shall be considered to be completely filled when it is filled to the level 12.7 mm vertical distance below the top of the container.

CAC/RM 47-1972 DETERMINATION OF ALCOHOL INSOLUBLE SOLIDS (Based on AOAC Method)

1. DEFINITION

The alcohol insoluble solids content of peas is in relation to their texture and maturity.

2. MATERIALS

2.1 Specifications for circular sieves

2.1.1 If the quantity of the total contents of the container is less than 1.5 kg (3 pounds) use a sieve with a diameter of 20 cm (8 inches)

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

2.1.3 The meshes of such sieves are made by so weaving wire as to form square openings of 2.8 mm by 2.8 mm.³

3. **PROCEDURE**

3.1 Pour the sample on circular sieve. Spread peas evenly and let drain. Transfer peas to white pan and remove any foreign material. Add volume H_2O equal to double volume original sample.

3.2 Pour peas back on sieve, spreading evenly, tilt sieve as much as possible without shifting peas, and drain 2 minutes. With cloth wipe surplus moisture from lower surface of sieve. Grind drained peas in food chopper until cotyledons are reduced to smooth homogeneous paste, stir and weigh 20 g ground material into 600 ml beaker. Add 300 ml 80% (v/v) alcohol, stir, cover beaker, and bring to boil. Simmer slowly 30 minutes.

3.3 Fit into Büchner filter paper of appropriate size (previously prepared by drying in flat-bottom dish 2 hours at temperature of boiling H_2O , covering with tighfit cover, cooling in desiccator, and weighing at once). Apply suction and transfer contents of beaker to Büchner so as to avoid running over edge of paper. Suck dry and wash material on filter with 80% (v/v) alcohol until washings are clear and colourless.

3.4 Transfer paper and alcohol-insoluble solids to dish used in preparation of paper, dry uncovered 2 hours at temperature of boiling H₂O, place cover on dish, cool in desiccator and weigh at once. From this weight deduct weight of dish, cover and paper.

4. CALCULATION AND EXPRESSION OF RESULTS

Calculate % m/m of alcohol-insoluble solids.

5. LITERATURE REFERENCE

3

AOAC (1965) 30.015 - Alcohol Insoluble Solids in Canned Peas (6). Official.

Ref. ISO Recommendation R 565. Such sieves could be replaced by US sieves with No 8 Standard screen (size of opening 2.38 mm).

CAC/RM 48-1972 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

2.1 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**
- 3.1 <u>Preparing mount</u>
- 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;
- 31.3 Add a drop of glycerine, place cover glass on material and examine under microscope.
- 3.2 Identification

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

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

CAC/RM 49-1972 DETERMINATION OF MINERAL IMPURITIES (SAND)

1. APPARATUS

Blender or macerator (Atomix, Turmix, Waring or equivalent).

Beakers - 2,000 ml capacity.

Funnels.

Filter Paper, Whatman No. 1, or equivalent.

Porcelain or Platinum crucibles.

Air oven or bunsen burner.

Muffle furnace (600°C).

Desiccator with active desiccant.

Analytical balance.

2. **REAGENTS**

NaCl solution (15%)

HCl

AgNO₃

3. PREPARATION OF TEST SAMPLE

3.1 Containers of 500 g, or less - use the entire contents including strawberries and packing medium. Comminute in blender and use entire portion for the analytical sample.

3.2 Containers larger than 500 g - thoroughly comminute the contents of the entire container. Quickly remove a 500 g for the analytical sub sample (sub).

4. **PROCEDURE**

4.1 Transfer the analytical sub to a 2-L beaker taking care to include any sand that might settle out.

4.2 Nearly fill the beaker with water and mix contents by swirling, using a stirring rod if needed.

4.3 Let stand about 10 minutes and decant supernatant material and water into a second 2-L beaker.

4.4 Refill the first beaker with water, repeat the mixing and swirling operation and again let set 10 minutes.

4.5 Fill the second beaker with water, mix and swirl, and let stand 10 minutes.

4.6 At the end of the 10 minute period decant beaker No. 2 into beaker No. 3. Likewise decant beaker No. 1 in beaker No.2.

4.7 Repeat the sequence carefully decanting supernatant from beaker No. 3 into sink, until all fruit tissue is removed from the sample.

4.8 Finally collect the residue from all the beakers in beaker No. 3.

4.9 Remove any seeds or fruit tissue that settle out by treating the residue in beaker No. 3 with hot 15% NaCl solution.

4.10 Remove NaCl by washing with hot water. Removal can be verified by testing the washings with $AgNO_3$.

4.11 Finally transfer residue remaining in Step 4.10 to funnel fitted with ashless filter paper. Use small portion of water to assure transfer of all residue. Discard filtrate.

4.12 Transfer filter paper to a weighed crucible. Dry in air oven or oven bunsen burner. Ignite in muffle furnace for about 1 hour at 600°C.

4.13 Cool, add 5 ml HCl and heat to boiling. Again cool, add 10 ml H₂O and heat to boiling.

4.14 Filter, and wash free of acid.

4.15 Ignite the filter by an initial drying and incineration in muffle furnace at 600°C.

4.16 Cool in desiccator, and weight.

5. CALCULATION AND EXPRESSION OF RESULTS

5.1 The weight of acid insoluble residue is determined by subtracting the weight of the empty crucible from the weight of the crucible plus incinerated residue (expressed as mg).

5.2 Express the residue, i.e. mineral impurities as mg/kg of the total product.

- (a) If the test sample is 500 g, multiply the value obtained in Step 5.1 by two (2).
- (b) If the test sample is other than 500 g, use the following formula:

$$X = \frac{1000}{W} (R)$$

where:

X = mineral impurities

W = weight of test sample (grammes)

R = residue remaining after incineration (milligrammes)

6. LITERATURE REFERENCE

Journal of the AOAC, Vol. 54, No. 3, 1971 (pages 581-583)

ALINORM 04/27/23 APPENDIX VII

THE USE OF ANALYTICAL RESULTS: SAMPLING PLANS, RELATIONSHIP BETWEEN THE ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND PROVISIONS IN CODEX STANDARDS

ISSUES INVOLVED

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards. In particular, different approaches may be taken regarding sampling procedures, the use of measurement uncertainty and recovery corrections.

At present there is no official guidance on how to interpret analytical results across the Codex Community. Significantly different decisions may be taken after analysis of the "same sample". For example some countries use an "every-item-must-comply" sampling regime, others use an "average of a lot" regime, some deduct the measurement uncertainty associated with the result, others do not, some countries correct analytical results for recovery, others do not. This interpretation may also be affected by the number of significant figures included in any commodity specification.

It is essential analytical results are interpreted in the same way if there is to be equivalence across the Codex Community.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results and various Guides prepared dealing with Measurement Uncertainty.

RECOMMENDATIONS

It is recommended that when a Codex Commodity Committee discusses and agrees on a commodity specification and the analytical methods concerned, it states the following information in the Codex Standard:

1. Sampling Plans

The appropriate sampling plan to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, to the average in a lot or the proportion nonconforming;
- the appropriate acceptable quality level to be used;
- the acceptance conditions of a lot controlled, in relation to the qualitative/quantitative characteristic determined on the sample.

2. Measurement Uncertainty

That an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens.

3. Recovery

[Where relevant and appropriate the analytical results are to be reported on a recovery corrected basis and that the recovery should be quoted in any analytical report.]

4. Significant Figures

The units in which the results are to be expressed and the number of significant figures to be included in the reported result.