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REPORT OF THE THIRD SESSION

OF THE

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Berlin, Federal
Republic of Germany
24-27 October 1967

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REPORT OF THE THIRD SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING Berlin, 24-27 October 1967

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its third session from 24 to 27 October 1967 in Berlin under the chairmanship of Professor Dr. R. Franck. There were 53 delegates and observers present, representing 22 countries and 9 international organizations. The Revised Provisional Agenda was adopted and it was agreed that no rapporteur should be appointed and that the draft report should be prepared by the Secretariat. The List of Participants appears as Appendix I and the List of Documents which the Committee had before it as Appendix II to this Report.

COMMENTS ON METHODS OF ANALYSIS AND SAMPLING

2. The Committee agreed that comments from governments on proposed methods of analysis should always be sent directly to the Committee on Methods of Analysis and Sampling; this would be appropriate and would facilitate its work. a/

METHODS OF ANALYSIS FOR HONEY

3. The Committee had before it the document "Honey - Methods of Analysis" (Appendix 4 of ALINORM 66/23), which had been circulated to governments, and comments thereon. The methods of analysis for honey were examined in detail and endorsed by the Committee, with slight amendments, so that appropriate methods be available for the Honey Standard according to present knowledge. This, however, would not prejudice further improvements of methods and the Committee would carry on its work in this field. In this connection the following subjects were discussed:

(a) It was pointed out to the Committee that enzymatic and chromatographic methods of analysis were available which can distinguish between the various sugars present in the honey. In this respect it is important to know which sugars are of particular interest in the description of honey. The delegation of France agreed to prepare a paper for the next session of this Committee on the gas chromatographic determination of sugars in honey. The representative of APIMONDIA similarly undertook to prepare a paper on the enzymatic analysis of sugars in honey.

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a/ Comments should be sent to the Secretariat of the Codex Committee on Methods of Analysis and Sampling with a copy to the Chief, FAO/WHO Food Standards Program, FAO, Rome, to reach them before the end of June 1968.

(b) In view of the fact that various types of honey are mentioned in the Standard, the Committee considered it desirable to use a method such as the pollen analysis for the identification of honeys and asked the representative of APIMONDIA to prepare a paper for the next session of this Committee. It was pointed out that this method had limitations, particularly in the case of multifloral honeys.

(c) The delegation of the United States of America pointed out that no official comments on the methods of analysis for honey had been submitted at Step 3 of the Procedure because uncertainty existed as to whether official comments were being invited on a regional or world-wide basis.

4. The Committee decided upon the following amendments in the document "Honey - Methods of Analysis" (Appendix 4 of ALINORM 66/23):

(c) Moisture content

That the Secretariat be requested to extend the Appendix A (Wedmore Table, 1955) to cover the new provision for 23% moisture content.

(d) Water-insoluble solids content

That the degree of fineness of sintered glass crucible should be from 15-40 micrometers.

(e) Ash content

Procedure

That the second sentence be amended by adding at the end "and overflowing".

(f) Acidity

That the representative of APIMONDIA be requested to supply the method of calculation for inclusion into the method by the end of November 1967 to the Secretariat of the Codex Alimentarius Commission.

(h) Hydroxymethylfurfuraldehyde content

Reagents

1. Barbituric acid solution:

That the first sentence be deleted and the second sentence be amended by inserting "500 mg of barbituric acid" after "Transfer".

2. p-toluidine solution:-

That the penultimate sentence be amended by adding at the end: "for 24 hours".

That the last sentence be amended as follows:

"Read the extinction of the sample against the blank at 550 nm using a 1 cm cell immediately the maximum value is reached."

Procedure

That the first sentence be amended to read:

"Weigh out 10 g of honey sample and dissolve without heating in 20 ml distilled water made free of oxygen by boiling and passing nitrogen through it."

In this connection it was mentioned that another method based on UV spectrophotometric determination was available and the Committee requested the delegation of the Netherlands to make available the description of this method and report on its suitability at the next session of this Committee.

5. With respect to the use of a standard starch for the determination of diastase number the Committee recommended that FAO consider the possibility of creating a stock of standard chemicals, such as standard starch and permitted food colours, for use in comparative tests.

6. It was pointed out that methods for colour grading of honey were available. However, the Committee was of the opinion that the establishment of a standard method of analysis for this purpose should not be undertaken at this time because there were no specifications for colour grading in the Standard for Honey.

7. The Committee endorsed the methods of analysis for honey, as amended, to be sent to the Commission at Step 5 of the Procedure for the Elaboration of Codex Standards. Since in the opinion of the Committee the endorsed methods of analysis for honey can now be regarded as being uncontroversial within the framework of the European Regional Standard, it was recommended that the Codex Alimentarius Commission consider the omission of Steps 6, 7 and 8 and send the amended methods to governments for final acceptance. The Secretariat of the Commission was requested to prepare a working paper for the Commission setting out these methods in the adopted layout for standard methods of food analysis.

METHODS OF ANALYSIS FOR SUGARS

8. The Committee had before it the Report of the Fourth Session of the Codex Committee on Sugars containing the proposed methods of analysis (Appendix X of ALINORM 68/21). During the discussion the following points were raised:

(a) The Committee was informed that there were no agreed methods of analysis for the determination of turbidity of sugar solutions and extraneous insoluble matter in sugars, and that these two criteria were regarded as important by the Codex Committee on Sugars.

(b) In order to cover glucose syrup and dried glucose syrup containing the highest permitted levels of sulphur dioxide, the Committee agreed to add under "Method" and "Accuracy" respectively (p. 22, ALINORM 68/21, Appendix X):

up to 600 mg/kg, test solution of 0.5g/100 ml ± 20 mg/kg

(c) Concerning a proposal to use the method of Monzier-Williams for the determination of sulphur dioxide in sugars, the representative of ICUMSA informed the Committee that this method did not give satisfactory results in the case of high quality sugars.

In the case of the determination of sulphur dioxide in starch conversion products, a number of delegations were of the opinion that the Carruthers method should be replaced by the Tanner modification of the Monzier-Williams method.

(d) The representative of ICUMSA explained that in the case of the proposals by the Codex Committee on Sugars the term "accuracy" reflected the maximum difference between the results obtained in different laboratories on the same sample (see also paragraph 32).

(e) It was brought to the attention of the Committee that the Codex Committee on Food Additives requested that high priority be given to the analysis of sulphur dioxide in food generally (see also paragraph 22).

9. The Committee endorsed the proposals contained in Appendix III of this Report and decided to invite comments from governments at Step 3 of the Procedure (see footnote on page 1).

METHODS OF ANALYSIS FOR FATS AND OILS

10. The Committee had before it a working paper (Codex/Fats and Oils/40) prepared by the Secretariat of the Codex Committee on Fats and Oils containing references, principles of methods, expressions of result and remarks on methods of analysis for edible oils, lard and rendered pork fat, premier jus and edible tallow, margarine and olive oil, proposed by that Committee. During the discussion the following points were raised:

(a) The representative of the International Olive Oil Council (IOOC) informed the Committee that the methods referred to in the above paper had been carefully studied, commented on, adopted and put into commercial practice by the member countries of the IOOC.

The Committee noted the request of the representative of IOOC and the delegate of Spain that in view of the advanced stage of these methods separate consideration should be given to them by the Codex Alimentarius Commission.

(b) The delegation of the United States of America drew the attention of the Committee to the fact that in their country the methods of analysis for margarine follow those for butter, some of which are at present being considered and finalized by the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products. The Secretariat confirmed this statement and undertook to bring the relevant references up to date.

(c) The Committee agreed that scientific methods should be applied whenever reference is made to organoleptic (sensoric) criteria in Standards; it recommended that such methods should be stipulated as they become available. The Committee also noted that ISO had established a working group dealing with this subject.

11. The Committee endorsed the proposals contained in Appendix IV of this Report for comments from governments at Step 3 of the Procedure (see footnote on page 1).

METHODS OF ANALYSIS AND SAMPLING FOR PROCESSED FRUITS AND VEGETABLES

12. The Committee had before it the Report of the Fourth Session of the Codex Committee on Processed Fruits and Vegetables (ALINORM 68/20) containing proposals for the determination of drained weights, for syrup measurements, and for tough string tests, etc. During the discussion of these methods the following points were discussed:

(a) Concerning the determination of drained weight the Committee noted that the relevant specifications in the Standard were based on this method and that therefore they should not be separated.

(b) It was pointed out that no details were provided for the tough string tests. The delegation of the United States of America was requested to provide these details and present them to the next session of this Committee.

(c) The Committee noted that the sieve for the determination of drained weight was an inch-based sieve which may not be generally available in metric countries. It was pointed out that, according to a decision by the Commission at its last session, measurements should always be based on the metric system.

13. The Committee endorsed the above methods (see Appendix V of this Report) with the observations stated above and invited comments from governments at Step 3 of the Procedure, keeping in mind point (a) raised above (see footnote on page 1).

14. The document SP 10/70-SP, "Proposed Sampling Plans for Processed Fruits and Vegetables including Frozen Foods" was discussed but the Committee agreed that the title should be changed to "Proposed Sampling Plans for the Quality Assessment of Processed Fruits and Vegetables" in order to make it clear that the plans are limited to quality assessment.

15. The Committee endorsed the above sampling plans as amended (see Appendix VI of this Report) in the light of government comments and decided that they be submitted to the Codex Alimentarius Commission at Step 5 of the Procedure.

METHODS OF ANALYSIS FOR COCOA PRODUCTS AND CHOCOLATE

16. The Committee considered the "Comparative Synopsis of International Methods of Analysis of Cocoa and Chocolate Products" (Codex ANALYS/67-3) which was circulated to governments for comment, as well as references to methods of analysis for cocoa butter contained in the Report of the Fifth Session of the Codex Committee on Cocoa Products and Chocolate (ALINORM 68/10, Appendix II, pages 4 and 5).

17. The Committee agreed that the methods mentioned above together with the compiled comments received from governments be referred to the Codex Committee on Cocoa Products and Chocolate. The representative of OICC indicated that OICC would prepare a working paper based on the synopsis and the compiled government comments and recommend methods of analysis for the next session of the Codex Committee on Cocoa Products and Chocolate, with the help of experts interested in this matter so that firm proposals can be made by that Codex Committee.

METHODS OF ANALYSIS FOR FRUIT JUICES

18. The Committee had before it the "Synopsis of Methods of Analysis for Fruit Juices", Codex ANALYS/67-2, and comments from governments, delegations and international organizations received by this Committee, and noted the following:

(a) that no methods of analysis for arsenic were mentioned in the synopsis, and

(b) that the Joint ECE/Codex Group of Experts on Fruit Juices should discuss methods for the determination of the concentration of fruit juices.

19. The Committee agreed that the proposals in the synopsis together with a comparative digest of government comments presented to this meeting and any further comments from governments received before 31 December 1967 be referred to the next session of this Committee for further consideration. The comparative digest of all government comments will also be made available to the next session of the Joint ECE/Codex Group of Experts on Fruit Juices.

METHODS OF ANALYSIS FOR PRESERVATIVES

20. The Committee had before it a working paper on the methods of analysis for preservatives in various foods prepared by the delegation of the Netherlands (Codex/ANALYS/67-10) containing references for the determination of sulphur dioxide, sorbic acid, nitrates, nitrites and benzoic acid.

21. Some delegations were of the opinion that the elaboration of methods of analysis for formic acid and parahydroxybenzoic acid and its esters would also be important. The delegation of the United Kingdom drew the Committee's attention to the hazard to health involved using the reagent α-naphthylamine in the estimation of nitrites in meat and meat products.

22. In reply to a request from the Codex Committee on Food Additives (ALINORM 68/12, paragraph 73) the Committee was of the opinion that the method of Tanner should be recommended as useful for the determination of sulphur dioxide in various foods. The Committee based its opinion on paragraph I. 2. of the working paper, Codex ANALYS/67-10.

23. The Commodity Committees were requested to comment on the applicability of the methods mentioned in the above paper (see Appendix VII of this Report) to the commodity standards concerned. The Committee also agreed to invite comments from governments and interested international organizations (see footnote on page 1). The Secretariat pointed out that the Committee of Government Experts on Milk and Milk Products would consider the method of determining nitrite in milk and milk products and government comments thereon.

METHODS OF ANALYSIS FOR ANTIOXIDANTS

24. The Committee had before it a working paper on the methods of analysis for antioxidants in various foods, prepared by the delegation of the Netherlands (Codex/ANALYS/67-8), containing references for the determination of gallates, BHA and BHT.

25. The Commodity Committees were requested to comment on the applicability of the methods mentioned in the above paper (see Appendix VIII of this Report) to the commodity standards concerned. The Committee also agreed to invite comments from governments and interested international organizations (see footnote on page 1). The Secretariat pointed out that the Committee of Government Experts on Milk and Milk Products would consider the method of determining gallates in milk powder and government comments thereon.

SAMPLING TECHNIQUE

26. The Committee had before it a Draft Provisional Standard for the technical procedure of sampling foods, prepared by the delegation of

the Federal Republic of Germany (SP 10/101, Codex/ANALYS/67-5) which is based on the method of the Code of Principles concerning Milk and Milk Products. Since this method has been re-edited, the Committee agreed that the present document should also be revised accordingly.

27. The delegation of Poland suggested that temperatures be recommended for the storage of test samples of deep frozen foods. The delegation of the United States of America suggested that the document be amplified to include a technical procedure for sampling frozen foods. The delegation of Australia recommended that in paragraph 1.3 dealing with sampling report a provision be inserted for the "spray-history" of samples for pesticide residue analysis.

28. The Committee also had before it a note prepared by the delegation of Canada on the subject of sampling. As this document was received too late for proper distribution it was agreed that it should be again presented to the Committee at its next session and that the re-edited paper prepared by the delegation of the Federal Republic of Germany (see Appendix IX of this Report) be submitted to the Codex Commodity Committees and governments for comments at Step 3 of the Procedure (see footnote on page 1).

TRIMETHYLAMINE

29. In the absence of a working paper the subject was only briefly discussed. As doubts had been expressed as to the usefulness of trimethylamine as an indication of quality assessment of fish on an international level, the Committee decided to await further information from the Codex Committee on Fish and Fishery Products. The delegation of the United States of America drew attention to the AOAC methods on volatile acids and the delegation of the Federal Republic of Germany drew attention to a method of analysis for trimethylamine together with trimethylaminaoxide and the Committee requested that they should be made available to the delegation of Canada for information.

ENZYME PREPARATIONS

30. The delegation of the Federal Republic of Germany gave an oral report of the work in progress on the compilation of methods of analysis for commercial enzyme preparations. In collaboration with the U.S.A. useful material has been collected. However, this was not sufficient to make definite proposals for methods as yet. It was pointed out that the Codex Committee on Food Additives has so far only developed the first draft of a general standard for commercial enzyme preparations without reference to individual enzymes requiring methods of analysis. Hence there was no urgency in developing methods for individual enzymes.

The delegation of the Federal Republic of Germany and the delegation of the United States of America were requested to carry on the work on this subject and make definite proposals when required to do so by the Codex Committee on Food Additives.

31. The Committee was informed that the Joint FAO/WHO Expert Committee on Food Additives would deal with enzymes at one of their next meetings. It was also mentioned that the Joint ECE/Codex Alimentarius Group of Experts on Fruit Juices had included filtration enzymes in their standards, which may require a method of analysis for these enzymes. The delegation of the Federal Republic of Germany pointed out that no methods could be proposed at present.

STANDARD LAYOUT

32. The Committee had before it the Standard Layout for a Standard Method of Food Analysis (Appendix 3 of ALINORM 66/23) and the comments received from several governments. During the discussion it was pointed out by the Committee that it would be desirable to define accurately the terms used with reference to the sensitivity, reproducibility, repeatability, etc. of methods of analysis and to draft explanatory notes for the Standard Layout. Furthermore, it was mentioned that provisions for the inclusion of literature citations concerning details of the methods would be useful.

33. The Committee decided that the Standard Layout be held at Step 4 and requested the delegation of the United Kingdom to draft these explanatory notes taking into account the ISO Recommendation R 78, as well as further ISO papers on this subject and the comments received from governments for consideration at the next session of this Committee. Comments available to the delegation of the United Kingdom by 31 December 1967 would also be taken into account.

PROJECTED TABLE OF CONTENTS (for the Codex Alimentarius Methods of Analysis)

34. The delegation of Poland informed the Committee that comments on the document Codex/ANALYS/66-5, presented to the last session of this Committee, had only been received from the delegation of the United Kingdom. The Committee decided to request comments, including comments on the organoleptic methods, from governments and asked the delegation of Poland to prepare an explanatory note to accompany the projected table of contents. a/

INTERNATIONAL COLLECTIONS OF METHODS OF ANALYSIS AND ORGANIZATIONS CONCERNED THEREWITH

35. The Committee had before it document SP 10/101 - Bibliography Codex/ANALYS/66-4 Corr. "International Collections of Methods of Analysis, Legally Codified Methods of Analysis and Organizations Concerned Therewith". It was pointed out that further amendments should be sent to the Secretariat of this Committee and that from time to time the additions should be distributed for the information of governments. It was suggested by the Committee that in due course a revised bibliography should be published and that the Secretariat of the Commission should explore the possibility of FAO publishing such a document.

FUTURE WORK

36. The Committee agreed that, because of the amount of work already under consideration and expected from Codex Commodity Committees, no additional work should be started at this time on standard methods of analysis and sampling.

OTHER BUSINESS

37. Methods for the Detection and Determination of Colours in Foods

The delegation of the United Kingdom informed the Committee that it was not possible to prepare a paper on the methods of analysis of colours at the present time but that such a paper would be presented to the next session of this Committee. In this connection it was pointed out that the Benelux countries have methods elaborated for the detection of colours in foods and the delegation of the Netherlands undertook to make this material available to the United Kingdom. During the

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a/ Note by the Secretariat: The documents in question together with the explanatory notes will be distributed in due course. Comments are requested to be sent to Professor St. Krauze (see List of Participants, Appendix I).

meeting the delegation of France made available to the United Kingdom delegation a document on the isolation and identification of food colours in food. It was understood that the analysis of food colours as such was the responsibility of the Joint FAO/WHO Expert Committee on Food Additives. The Committee considered that as regards not permitted food colours qualitative methods of detection would be sufficient at this time. As regards priorities for the elaboration of methods of analysis of colours, the delegation of Poland suggested that colours which had been endorsed in foods at advanced Steps of the Procedure by the Codex Committee on Food Additives and the Provisional List of Food Colours which has been circulated to governments for comments at Step 3 should be taken into consideration-

38. Referee Methods

The Committee was informed about a proposal by the Executive Committee regarding the question of referee methods and alternate methods of analysis. This proposal was fully supported by this Committee. The Committee was of the opinion that the methods of analysis contained in the Codex Alimentarius should be referee methods. If two or more methods had been proved to be equivalent, these could be regarded as alternatives.

39. General Principles of Codex Methods of Analysis

(a) On the proposal of the delegation of the United States of America the Committee asked the Secretariat of the Commission to prepare a working paper for the next session of this Committee consolidating all provisions, rules, general principles, etc. relating to the work of this Committee and taking into account the decision of the Commission regarding the establishment of Codex Methods of Analysis. In this connection the paper presented by the delegation of the United States of America should also be taken into account.

(b) The Committee was of the opinion that it would be useful to submit these general principles also to the Commodity Committees since, amongst other matters, reference to the layout for the methods of analysis would be contained in the document.

(c) It was pointed out to the Committee that methods of analysis should be in the adopted layout before being considered by the Codex Alimentarius Commission (at Step 5) so that governments have the opportunity to study these in detail before the session of the Commission.

(d) During the elaboration of Codex Methods of Analysis the proposed methods should also be sent to other organizations working in this field.

(Paragraphs 39 (b), (c) and (d) were drafted by the Secretariat upon the request of the Committee after the adoption of the Report.)

DATE AND PLACE OF NEXT SESSION

40. The Chairman suggested as a date and place for the next session of the Committee 4-8 November 1968, in Berlin.

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OICC	Dr. G.F. Schubiger Président de la Commission des Experts Office International du Cacao et du Chocolat Case Postale 88 1814 La Tour de Peilz (Switzerland)
OIV	Prof. P. Jaulmes Professeur de Chimie Analytique et de Toxicologie à la Faculté de Pharmacie de Montpellier Office International de la Vigne et du Vin 11 rue Roquepine Paris 8 (France)
Secretariat	Dr. W. Krönert Wissenschaftlicher Oberrat im Bundesgesundheitsamt 1 Berlin 33, Postfach (Federal Republic of Germany)

LIST OF DOCUMENTS

1.	ALINORM 66/23 (Codex/ANALYS/66-14)	Codex Committee on Methods of Analysis and Sampling, Report of the Second Session
2.	ALINORM 66/4 SP 10/8 - 3rd Session	Report of the Third Session of the Coordinating Committee for Europe
3.	ALINORM/MDS/66/14	
4.	ALINORM 66/19	Draft Provisional Standard for Honey
5.	CL 1967-29 SP 10/101-Bibliography Codex/ANALYS/66-4 Corr.	Distribution, for information purposes, of document entitled "International collections of methods of analysis, legally codified methods of analysis, and organizations concerned therewith"
6.	SP 10/70-SP, July 1966	Proposed Sampling Plans for Processed Fruits and Vegetables including Frozen Foods
7.	Codex/ANALYS/66-5	Table of Contents
8.	SP 10/101 Codex/ANALYS/67-Information	Matters to be brought to the attention of the Codex Committee on Methods of Analysis and Sampling
9.	SP 10/101 Codex/ANALYS/67-Honey	Comments of Governments on the proposed Methods of Analysis for Honey
10.	CL 1967-27 (SP 10/75) (SP 10/101) Codex/ANALYS/67-2	Synopsis of Methods of Analysis for Fruit Juices
		Comments, Observations:
11.	Codex/ANALYS/67-2(1)	OIV
12.	Codex/ANALYS/67-2(2)	United Kingdom
13.	Codex/ANALYS/67-2(3)	Netherlands
14.	Codex/ANALYS/67-2(4)	France
15.	Codex/ANALYS/67-2(5)	U.S. delegate
16.	Codex/ANALYS/67-2(6)	Délégation Suisse
17.	Codex/ANALYS/67-2(7)	Yugoslavia
18.	Codex/ANALYS/67-2(8)	New Zealand
19.	CL 1967-28 SP 10/101 Codex/ANALYS/67-3	Comparative Synopsis of International Methods of Analysis of Cocoa and Chocolate Products
20.	Codex/ANALYS/67-4(1)	United States comments on "Standard Layout for a Standard Method of Food Analysis"

21.	Codex/ANALYS/67-4(2)	United Kingdom comments on Standard Method of Food Analysis.
22.	Codex/ANALYS/67-4(3)	Comments by Chemistry Division, Department of Scientific and Industrial Research, Petone, New Zealand.
23.	SP 10/101 Codex/ANALYS/67-5	Draft Provisional Standard for the Technical Procedure of Sampling Foods
24.	CL 1967-37 SP 10/101 Codex/ANALYS/67-6(1), 7(2) 11	Methods of Analysis referred to the Codex Committee on Methods of Analysis and Sampling
25.	Codex/ANALYS/67-6(3)	United Kingdom Step 3 comments on the Processed Sampling Plans for Processed Fruits and Vegetables
26.	Codex/ANALYS/67-7(3)	United States comments on "Comparative Synopsis of International Methods of Analysis of Cocoa and Chocolate Products"
27.	Codex/ANALYS/67-7(4)	United Kingdom comments on Methods of Analysis for Cocoa Products and Chocolate
28.	SP 10/101 Codex/ANALYS/67-8	Antioxidants
29.	SP 10/101 Codex/ANALYS/67-10	Preservatives
30.	Codex/ANALYS/67-13 Codex/FATS and OILS/40	Codex Committee on Fats and Oils Methods of Analysis
31.	Codex/ANALYS/67-19	Note on Sampling prepared by the Canadian Delegation
32.	Codex/ANALYS/67-20	United States suggestions for possible General Principles for use by the Committee on Methods of Analysis and Sampling and by the Commodity Committees
33.	Codex/ANALYS/67-21	Comments of New Zealand on Methods of Analysis for Honey
34.	Codex/ANALYS/67-22	Comments of New Zealand on Codex Document SP 10/70-SP
35.	ALINORM 68/10	Draft Report of the Fifth Session of the Codex Committee on Cocoa Products and Chocolate
36.	ALINORM 68/11	Codex Committee on Fats and Oils Report of the Fourth Session
37.	ALINORM 68/20	Report of the Fourth Session of Codex Committee on Processed Fruits and Vegetables
38.	ALINORM 68/21	Codex Committee on Sugars

1. WHITE SUGAR (Continued)

Analytical Criterion	Method	Accuracy a/	Reference
Sulphur Dioxide (continued)	5-30mg/kg, test solution of 10g/100ml	± 1.0mg/kg	I.S.J.1965 p.364
	10-60mg/kg, test solution of 5g/100ml	± 2.0mg/kg	
Arsenic (As)	Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate		AOAC 1965 24.011
Lead (Pb)	ICUMSA method with wet ashing satisfactory below a level of 0.5mg/kg	± 0.1mg/kg	Ic.M. p.48(c)
Copper (Cu)	ICUMSA method with wet ashing for levels referred to in Codex standards	± 0.5mg/kg	Ic.M. p.106

a/ See paragraph 8(d) of this report

PROPOSED METHODS OF ANALYSIS AND SAMPLING FOR SUGARS

(submitted to governments for comment at Step 3)

I. METHODS OF ANALYSIS

1. WHITE SUGAR

Analytical Criterion	Method	Accuracy a/	Reference
Polarization expressed as sucrose	ICUMSA method for raw sugars (without lead). Lead defecation only if necessary; no correction for 'lead effect'	± 0.1°S	Ic.R1958p.84
Invert Sugar	KNIGHT and ALLEN method for contents below 0.02%	± 0.005%	Ic.M. p. 29
	BERLIN INSTITUTE method for contents between 0.02% and 0.10%	± 0.005%	Ic.M. p. 25
	LANE and EYNON method for contents above 0.1%	± 0.01%	Ic.M. p. 13
Conductivity Ash	ICUMSA method for conductivity ash using 5g/100 ml or 28g/100g solutions; with 5g/100ml, the conductivity expressed in micromhos/cm should be multiplied by the standard C-ratio factor of 1·8 x 10^{-4}	± 0.001%	Ic.R1962 p.12
Loss on drying	ICUMSA method using a minimum sample size of 20g (without grinding)	± 0.005%	Ic.M.p.44
Colour (ICUMSA units)	Measurement by ICUMSA method 4 on a solution of 50g/100g after filtration through a membrane filter of pore size 0.4 µ to 0.6 µ. Results to be expressed as 'ICUMSA units' as defined in Ic.M.p.58	not yet established	IcM.pp 57 and 58 IcR 1958 p.52
Sulphur Dioxide	CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution		
	1-7 mg/kg test solution of 40g/100ml	± 0.3 mg/kg	
	2-15 mg/kg test solution of 20g/100ml	± 0.5 mg/kg	

2. SOFT SUGARS

(a) White to Dark Brown

Analytical Criterion	Method	Accuracy [a]	Reference
Sucrose (saccharose) + invert sugar expressed as sucrose	TATE and LYLE Invertase Modification of LANE and EYNON method		Ic.M.p.71
Invert Sugar	LANE and EYNON method (without inversion)		Ic.M.p.71
Sulphated Ash	Gravimetric double sulphation method		Ic.M.p.36
Loss on drying	ICUMSA method using 10g		Ic.M.p.44
Sulphur Dioxide	CARRUTHERS, HEANEY and OLDFIELD method		
	The range and accuracy depend on the concentration of the test solution		
	1-7mg/kg, test solution of 40g/100ml	± 0.3mg/kg	
	2-15mg/kg, test solution of 20g/100ml	± 0.5mg/kg	
	5-30mg/kg, test solution of 10g/100ml	± 1.0mg/kg	I.S.J.1965 p.364
	10-60mg/kg, test solution of 5g/100ml	± 2.0mg/kg	
Arsenic (As)	Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate		AOAC, 1965 24.011
Lead (Pb)	ICUMSA method with wet ashing, satisfactory below a level of 0.5mg/kg	± 0.1mg/kg	Ic.M.p.48(c)
Copper (Cu)	ICUMSA method with wet ashing for levels referred to in Codex standards	± 0.5mg/kg	Ic.M.p.106

2. SOFT SUGARS (Continued)

(b) Soft White Sugar

Analytical Criterion	Method	Accuracy a/	Reference
Sucrose (saccharose) + invert sugar expressed as sucrose	TATE and LYLE Invertase modification of LANE and EYNON method		Ic.M.p.71
Conductivity Ash	ICUMSA method for conductivity ash using 5g/100ml or 28g/100g solutions; with 5g/100ml the conductivity expressed in micromhos/cm should be multiplied by the standard C-ratio factor of 18×10^{-4}		Ic.R.1962p.12
Colour (ICUMSA units)	Measurement by ICUMSA method 4 on a solution of 50g/100g after filtration through a membrane filter of pore size 0.4/μ to 0.6/μ		Ic.M.pp.57 and 58 Ic.R.1958 p.52

a/ See paragraph 8(d) of this report

3. LACTOSE

Analytical Criterion	Method	Accuracy a/	Reference
Lactose anhydrous	LANE and EYNON method		Ic.M.p.13
Sulphated Ash	Single sulphation		Ic.M.p.100
Loss on drying	U.S.P.method (drying 16 hours at 130°C.) or KARL FISCHER method		U.S.P. 1965 p.336 Angew.Chem. 1935,48,394
pH(10% solution)	by pH meter		Ic.M.p.44
Arsenic (As)	Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate		AOAC,1965 24.011
Lead (Pb)	ICUMSA method with wet ashing satisfactory below a level of 0.5mg/kg	± 0.1mg/kg	Ic.M.p.48(c)
Copper (Cu)	ICUMSA method with wet ashing for levels referred to in Codex standards	± 0.5mg/kg	Ic.M.p.106

a/ See paragraph 8(d) of this report

4. GLUCOSE SYRUP, DRIED GLUCOSE SYRUP

Analytical Criterion	Method	Accuracy a/	Reference
Total solids	Drying in vacuum oven		C.I.R.F.Method E.42
Dextrose Equivalent (reducing sugars expressed as D-glucose	LANE and EYNON method		Ic.M.p.101
Sulphated ash	Single sulphation method		Ic.M.p.100
Sulphur dioxide	CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution, namely		
	1-7 mg/kg test solution of 40g/100ml	± 0.3 mg/kg	
	2-15mg/kg test solution of 20g/100ml	± 0.5 mg/kg	
	5-30mg/kg test solution of 10g/100ml	± 1.0 mg/kg	
	10-60mg/kg test solution of 5g/100ml	± 2.0 mg/kg	I.S.J.1965 p.364
	-600mg/kg test solution of 0.5g/100ml	± 20 mg/kg	
Arsenic (As)	Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyldithiocarbamate		AOAC, 1965 24.011
Lead (Pb)	ICUMSA method with wet ashing satisfactory below a level of 0.5 mg/kg	± 0.1 mg/kg	Ic.M.p.48(c)
Copper (Cu)	ICUMSA method with wet ashing for levels referred to in Codex standards	± 0.5 mg/kg	Ic.M.p.106

a/ See paragraph 8(d) of this report

5. DEXTROSE MONOHYDRATE and DEXTROSE ANHYDROUS

Analytical Criterion	Method	Accuracy[a]	Reference
Dextrose (expressed as D-glucose)	LANE and EYNON method		Ic.M p.101
Total solids	Drying at 100°C for 4 hours under reduced pressure		Ic.M p.113
Sulphated ash	Single sulphation method		Ic.M p.100
Sulphur dioxide	CARRUTHERS, HEANEY and OLDFIELD method. The range and accuracy depend on the concentration of the test solution, namely:		
	1-7mg/kg test solution of 40g/100ml	± 0.3 mg/kg	
	2-15mg/kg test solution of 20g/100ml	± 0.5 mg/kg	
	5-30mg/kg test solution of 10g/100ml	± 1.0 mg/kg	
	10-60mg/kg test solution of 5g/100ml	± 2.0 mg/kg	I.S.J.1965 p.364
Arsenic (As)	Diethyldithiocarbamate method using wet ashing and colorimetric measurement with silver diethyl-dithiocarbamate		AOAC 1965 24.011
Lead (Pb)	ICUMSA method with wet ashing, satisfactory below a level of 0.5 mg/kg	± 0.1 mg/kg	Ic.M p.48(c)
Copper (Cu)	ICUMSA method with wet ashing for levels referred to in Codex standards	± 0.5 mg/kg	Ic.M p.106

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a/ See paragraph 8(d) of this report

II. METHOD OF SAMPLING

(for White and Soft Sugars)

1. Sampling from bags (Ic.M. p.86)

Preferably, samples should be taken from opened bags. When this is not possible samples should be taken by piercing the bags with a trier of the type described in Ic.M. p.80, the punctures being sealed by appropriate means, e.g. with adhesive tape for paper bags. For domestic packets up to 5 kg in weight, the entire package should be taken.

2. Number of samples to be taken

The maximum size of a lot is 500 tons. The number of packages to be sampled will be $\sqrt[3]{T}$ (where T is the tonnage of the lot) with a minimum of three packages. In all cases, the gross sample set apart will be at least 2 kg: more than one insertion of the trier being made when necessary.

3. Preparation of the sample for analysis (Ic.M. p.83)

From the gross sample collected as described above and mixed, 4 sub-samples, each of at least 500g, should be prepared by the ICUMSA method for raw sugars and sealed in moisture-proof containers.

III. ABBREVIATIONS USED IN THE APPENDIX

Ic.M.	=	ICUMSA Methods of Sugar Analysis (1964)
Ic.R.	=	ICUMSA Report (Report of the Proceedings of the Session)
I.S.J.	=	International Sugar Journal
C.I.R.F.	=	Corn Industries Research Foundation
A.O.A.C.	=	Association of Official Analytical Chemists

PROPOSED METHODS OF ANALYSIS FOR EDIBLE OILS [a]

(Submitted to governments for comment at Step 3)

A. QUALITY CHARACTERISTICS

	Analytical Criterion	References
1.	Acid value	IUPAC (1964), II.D.1; AOCS Official Method Ca 5a-40; and AOAC(1965) 26.060 as amended by JAOAC(1966) 49,231. AOCS Tentative Method Cd 3a-63.
2.	Peroxide value	IUPAC (1964), II.D.13; AOCS Official Method Cd 8-53 and AOAC(1965) 26.024

B. CONTAMINANTS

1.	Matter volatile at 105°C	IUPAC (1964) II.C.1.1.
2.	Insoluble impurities	IUPAC (1964) II.C.2.; AOCS Official Method Ca 3-46
3.	Soap content	BS 684: 1958 page 49
4.	Iron	BS 684: 1958 page 92, alternatively BS 769: 1961
5.	Copper	BS 684: 1958 page 89; AOAC(1965) 24.023; Analyst 1963, 88, 256
6.	Lead	Analyst, 1959, 84, 129; AOAC(1965) 24.053
7.	Arsenic	Analyst, 1960, 85, 639 (Appendix III); AOAC(1965) 24.006; AOAC(1965) 24.011

C. IDENTITY CHARACTERISTICS

1.	Relative density	BS 684:1958 page 10, Method 1; AOCS Official Method Cc 10a-25 and AOAC(1965) 26.003
2.	Refractive index	IUPAC(1964),II.B.2. and AOCS Official Method Cc 7-25 and AOAC (1965) 26.006
3.	Saponification value	IUPAC(1964),II.D.2. and AOCS Official Method Cd 3-25 and AOAC(1965) 26.028
4.	Unsaponifiable matter	IUPAC(1964) II.D.5.3; AOCS Tentative Method Ca 6b-53 and AOAC(1965) 26.071; AOCS Official Method Ca-40
5.	Iodine value (Wijs)	IUPAC(1964)II.D.7.3. and AOCS Official Method Cd 1-25 and AOAC(1965) 26.022
6.	Crismer value	AOCS Official Method Cb.4-35

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a/ Those concerned are requested to consult the paper prepared by the United Kingdom Delegation (Codex/Fats and Oils/40, August 1967) in which the Methods of Analysis are described and discussed. Further copies of this paper can be obtained from the Chief, Food Standards Branch, FAO, Rome.

	Analytical Criterion	References
D. SPECIFIC TESTS		
1.	ARACHIS OIL: Arachidic and Higher Fatty Acids Content	
	(a) Modified Renard Test	AOAC(1965) 26.077
	(b) Arachis Oil Test(Evers)	BS 684:1958, page 97
2.	COTTONSEED OIL Halphen Test	AOCS Official Method Cb 1-25 and AOAC(1956) 26.076
3.	SESAMESEED OIL	
	(a) Modified Villavechia Test	AOCS Official Method Cb 2-40
	(b) Sesame Oil Test (Baudouin)	BS 684:1958, page 96

PROPOSED METHODS OF ANALYSIS FOR LARD AND RENDERED PORK FAT

	Analytical Criterion	References
A. QUALITY CHARACTERISTICS		
1.	Acid value	IUPAC(1964)II.D.I; AOCS Methods Ca5a-40 and Cd 3a-63 and AOAC(1965) 26.060 as amended by JAOAC(1966) 49.231
2.	Peroxide value	IUPAC(1964)II.D.13; AOCS Official methods Cd8-53 and AOAC(1965)26.024
B. CONTAMINANTS		
1.	Matter volatile at 105°C	IUPAC(1964)II.C.I.I.; Draft ISO Recommendation No.1224
2.	Impurities	IUPAC(1964)II.C.2; AOCS Official Method Ca3-46; Draft ISO Recommendation No.1223
3.	Soap Content	BS 684:1958 page 49
4.	Iron	BS 684:1958 page 92; BS 769: 1961
5.	Copper	BS 684:1958 page 89
6.	Arsenic	Analyst 1960,85, 639 (Appendix III); AOAC(1965)24.006; AOAC(1965) 24.011
7.	Lead	Analyst,1959,84,129; AOAC(1965)24.053

Analytical Criterion		References
C. IDENTITY CHARACTERISTICS		
1.	Relative density	BS 684:1958 page 10, Method 1; AOCS Official Method Cc 10a-25 and AOAC(1965) 26.003
2.	Refractive Index	IUPAC (1964)II.B.2. and AOCS Official Method Cc 7-25 and AOAC(1965)26.006 to 26.009
3.	Titre	IUPAC(1964), II.B.3.2; AOCS Tentative Method Cc12-59 and AOAC(1965) 26.013; Draft ISO Recommendation No.1226
4.	Saponification value	IUPAC(1964)II.D.2. and AOCS Official Method Cd3-25 and AOAC(1965) 26.028
5.	Unsaponifiable Matter	IUPAC(1964)II.D.5.3.; AOCS Tentative Method Ca6b-53 and AOAC(1965)26.071; AOCS Official Method Ca-40
6.	Iodine value (Wijs)	IUPAC(1964)II.D.7.3 and AOCS Official Method Cd1-25 and AOAC(1965)26.022

PROPOSED METHODS OF ANALYSIS FOR PREMIER JUS AND EDIBLE TALLOW

A. QUALITY CHARACTERISTICS		
1.	Acid value	IUPAC(1964)II.D.1.; AOCS Methods Ca5a-40 and Cd 3a-63 and AOAC(1965) 26.060 as amended by JAOAC(1966)49,231
2.	Peroxide value	IUPAC(1964)II.D.13; AOCS Official Method Cd 8-53 and AOAC(1965) 26.024
B. CONTAMINANTS		
1.	Matter volatile at 105°C	IUPAC(1964)II.C.I.I.; Draft ISO Recommendation No.1224
2.	Impurities	IUPAC(1964)II.C.2; AOCS Official Method Ca3-46; Draft ISO Recommendation No.1223
3.	Soap Content	BS 684:1958 page 49
4.	Iron	BS 684:1958 page 92; BS 769: 1961
5.	Copper	BS 684:1958 page 89
6.	Arsenic	Analyst 1960,85,639 (Appendix III);AOAC(1965)24.006; AOAC(1965)24.011
7.	Lead	Analyst,1959,84,129; AOAC(1965) 24.053
C. IDENTITY CHARACTERISTICS		
1.	Relative density	BS684:1958 page 10,Method 1; AOCS Official Method Cc10a-25 and AOAC(1965) 26.003

C. IDENTITY CHARACTERISTICS (Continued)

2.	Refractive Index	IUPAC(1964)II.B.2; and AOCS Official Method Cc7-25; and AOAC(1965)26.006 to 26.009
3.	Titre	IUPAC(1964)II.B.3.2; AOCS Tentative Method Cc12-59 and AOAC(1965)26.013; Draft ISO Recommendation No.1226
4.	Saponification value	IUPAC(1964)II.D.2; AOCS Official Method Cd3-25 and AOAC(1965)26.028
5.	Unsaponifiable matter	IUPAC(1964)II.D.5.3; AOCS Tentative Method Ca6b-53 and AOAC(1965)26.071; AOCS Official Method Ca-40
6.	Iodine value (Wijs)	IUPAC(1964)II.D.7.3 and AOCS Official Method Cd1-25 and AOAC(1965)26.022

PROPOSED METHODS OF ANALYSIS FOR MARGARINE

1.	Total fat content	Section II of the Report of the 10th Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products; Official Netherlands Food Analysis (see p. of this Appendix)
	(a) Water content(W)	see above
	(b) Non-fat residue(NFR)	BS 769:1961; see under 1 and 1(a) above
2.	Milk fat content	Analyst,1912,37,183 and AOCS Official Method Cd5-40
	(a) Reichert (R) value (water-soluble volatile fatty acids)	IUPAC (1964) II.D.9; and AOAC (1965), 26.032 and 26.033
	(b) Polenske(P) value (water-insoluble volatile fatty acids)	IUPAC (1964)II.D.9; and AOAC(1965),26.032 and 26.033
	(c) Kirschner (K) value (water-soluble volatile fatty acids that form water-soluble silver salts)	page 70, BS 684: 1958
3.	Vitamin A	AOAC(1965)39.001; J.Assoc.Offic.Anal.Chem.(1959)42,422
4.	Vitamin D	AOAC(1965)39.116
5.	Vitamin E	Analyst,1959, 84, 356
6.	Sodium chloride	(i) FAO/WHO Reference Method for the Determination of Salt (Sodium Chloride) Content of Butter. The text was submitted to FAO and WHO Member Governments for acceptance (see Appendix IV-D of the Report of 10th Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products, August 1967)

	Analytical Criterion	References
6.	Sodium chloride (continued)	(ii) Alternatively: BS 769:1961 para.5, Method 2.
7.	Edible carbohydrate sweetening matters	Not specifically available
8.	Edible proteins	Not specifically available

PROPOSED METHODS OF ANALYSIS FOR OLIVE OIL

	Analytical Criterion	Method suggested by IOOC
1.	Fatty acid composition	IUPAC II.D.19 /Supplement to IUPAC(1964)/
2.	Relative density	IUPAC (1954) page 37
3.	Refractive Index	IUPAC (1964) II.B.2
4.	Iodine value	Wijs Method: IUPAC(1964)II.D.7.3: or Hanus Method: IUPAC(1964)II.D.7.4
5.	Saponification value	IUPAC(1964)II.D.2
6.	Unsaponifiable matter	IUPAC(1964)II.D.5.2
7.	Bellier Index	(see p. 6 of this Appendix)
8.	Semi-siccative (drying) oils test	(see p. 7 of this Appendix)
9.	Residue olive oil test	(see p. 8 of this Appendix)
10.	Cotton seed oil test	(see p.8/9of this Appendix)
11.	Tea Oil test	(see p. 9 of this Appendix)
12.	Sesame oil test	(see p. 9 of this Appendix)
13.	Acidity	IUPAC (1964) II.D.1.
14.	Peroxide value	IUPAC (1964) II.D.13
15.	Specific extinction in ultra violet	(see p.10 of this Appendix)
16.	Moisture and volatile matter	IUPAC (1964) II.C.1.1.
17.	Impurities	IUPAC (1964) II.C.2.
18.	Soap test	(see p.12 of this Appendix)

DIRECT METHOD FOR DETERMINATION OF TOTAL FAT CONTENT OF MARGARINE

(Suggested by the Netherlands)

Melt 50g of the product in a wide-necked bottle with close-fitting stopper at about 40°C and cool whilst shaking vigorously until the mass is homogeneous and viscous. Introduce about 10g into a conical flask, add 20ml 25-% hydrochloric acid (s.g. 1.126) and boil till all non-fatty substances are dissolved. Allow to cool somewhat, transfer the mass to a percolator with the aid of hot water and wash with petroleum ether. Extract in the percolator for about 2 hours. Distil the solvent from the flask, place the flask in a drying oven at 100°C, and weigh. Continue drying in the drying oven until there is no further loss in weight.

BELLIER INDEX

I. Definition of the Bellier index

The Bellier index of an oil is the temperature at which precipitation of salts of the fatty acids of this oil commences, when the oil has been saponified and made into solution as described under "procedure".

II. Analysis

1. Apparatus

1.1 Test tubes 26-27mm in diameter and 22cm long.
1.2 A condenser consisting of a glass tube with stopper.
1.3 A thermometer graduated in ¼ degrees from 8 to 25°C, fixed in a stopper.
1.4 Graduated pipettes.

2. Reagents

2.1 A hydro-alcoholic solution of alcoholic potash (42.5g of pure KOH is dissolved in 72ml of distilled water: bring up to 500ml with 95% alcohol).
2.2 70% ethyl alcohol solution (use pure ethyl alcohol or "fine" alcohol).
2.3 An aqueous solution of acetic acid, 1+2 (by volume) so adjusted that 1.5ml exactly neutralizes (phenolphthalein indicator) 5 ml of the hydro-alcoholic solution of potassium hydroxide (2.1).

3. Preparation of sample

The oil is deprived of water by decantation and filtering through paper at a temperature slightly above the point of fusion of certain solid constituents which could separate from the fluid fatty matter.

4. Procedure

4.1 Place 1ml of fatty matter and 5ml of potash solution in a test tube.
4.2 Connect to condenser and heat moderately, agitating by rotation from time to time until saponification is complete, that is to say, until a perfectly clear solution is obtained.

BELLIER INDEX (continued)

4.3 Allow to cool. Disconnect condenser. Add 1.5m. of the acetic acid solution and 50ml of the alcohol solution. Attach thermometer and homogenize.

4.4 Place test tube in a beaker of water at 23-25°C. If a flocculent precipitate forms, leave standing for an hour at the same temperature and filter into a test tube.

4.5 Attach thermometer to the test tube containing the clear solution. Place for a moment in a beaker of water at about 10°C less than the estimated Bellier index.
Withdraw and ensure even temperature by inverting a number of times (cooling should be at the rate of about 1°C per minute). Repeat this operation until cloudiness appears. Note temperature.

4.6 Allow the temperature to increase a few degrees to dissolve the precipitate. Homogenize by inverting test tube over. Cool. The cooling should be slow and shaking more frequent as the temperature approaches that noted the first time. The temperature at which the cloudiness reappears is: i.e. t° = Bellier index.

5. Permitted tolerances

Two parallel determinations may not differ by more than 0.25°C.

SEMI-SICCATIVE OILS TEST

Apparatus:

- Stoppered 50ml Erlenmeyer flask
- Bath of melting ice

Reagents:

- Hexane, or in default, light petroleum with distillation point between 40° and 60° and bromine value less than 1, free of residues.

- Bromine reagent obtained by adding drop by drop and shaking 4ml of chemically pure bromine (the presence of chlorine prevents the reaction) into 100ml of hexane or light petroleum, chilled to 0°C and kept in the melting ice until required.

Procedure:

The oil to be tested must have previously been filtered and had the moisture removed. Place 1 ml of the oil in the previously dried Erlenmeyer flask. Dissolve it in 10ml of hexane. Place the stoppered Erlenmeyer flask in the melting ice. After 5 minutes, add 10ml of the bromine reagent in small quantities at a time, while shaking and maintaining the temperature at 0°C. The colour of the solution must clearly indicate excess bromine. Leave the Erlenmeyer flask in the melting ice for one hour, after which note appearance of solution. If semi-siccative oil is present a flocculent precipitate will be present, varying in quantity according to the percentage of adulteration and the nature of the adulterating oil.

The solution remains limpid and transparent in the case of genuine olive oils

RESIDUE OLIVE OIL TEST

Apparatus:

- 100ml balloon-flask equipped with reflux condenser
- 5ml pipette, graduated in tenths
- 5ml pipette
- 50ml test tube
- Heating arrangement to keep balloon-flask at about 80°C
- Thermometer graduated from 15° to 60°C

Reagents:

As for Bellier index (see p.7)

Preparation of sample:

As for Bellier index (see p.7)

Procedure:

Place about 1g of the oil prepared as above in the balloon-flask. Add 5ml of alcoholic solution of potash. Attach condenser and bring to boil holding at this temperature for 10 minutes, shaking from time to time. Allow to cool to ambient temperature. Add 1.5ml of acetic acid solution and 50ml of ethyl alcohol solution previously heated to 50°C. Mix by shaking, introduce thermometer and allow to cool, noting the appearance of the solution once 45°C is reached. If a flocculent precipitate forms at a temperature above 40°C the test is positive.

Allow to cool to ambient temperature (not lower than 18°C) over at least 12 hours. Observe solution again: the formation of a flocculent precipitate, floating in the middle of the liquid, indicates that the test is also positive. A cloudiness not forming into flakes does not indicate the presence of residue olive oil.

NOTE: On rare occasions some virgin olive oils, obtained by second pressing, yield a positive result.

COTTONSEED OIL TEST

Apparatus:

- 250mm x 25mm test tubes
- Water bath in which temperature can be regulated
- Heating arrangement to keep the tubes at 110° - 115°C

Reagents:

Mix equal volumes of amyl alcohol and a solution of 1g of sulphur in 100ml of carbon disulphide.

Procedure:

Place about 10ml of the oil under examination in a test tube, add the same volume of reagent; shake and keep in water bath at 70°-80°C, shaking until the carbon

COTTONSEED OIL TEST (Continued)

disulphide has completely evaporated (generally 5 minutes are enough), which is confirmed by the appearance of slight fuming above the liquid. Transfer the test tube to the heating apparatus and keep at 110°-120°C for 2 hours.

A red, or rose, colour betrays the present of cottonseed oil. The appearance of an orange colour must not be interpreted as proving the presence of cottonseed oil.

NOTE: The heating of the cottonseed oil to temperature above 170°C brings about a progressive destruction of the cycle-propenoic acids responsible for the colouring. This destruction is practically complete at 200°C.

TEA OIL TEST

Apparatus:

- 150mm x 15mm test tube
- 2ml pipette graduated in tenths
- Dropper so calibrated that 7 drops of oil weigh approximately 0.22g
- Water bath at 5°C

Reagents:

- Analytically pure chloroform
- Concentrated sulphuric acid (d = 1.84) analytically pure
- Analytically pure acetic anhydride
- Analytically pure diethyl oxide

Procedure:

Using the graduated pipette, place 0.8ml acetic anhydride, 1.5ml of chloroform and 0.2ml of sulphuric acid in a test tube. Cool to 5°C, then add about 0.22g of oil. If cloudiness appears, add, drop by drop while shaking, a quantity of acetic anhydride until the solution is clear. Keep at 5°C for 5 minutes. Add 10ml of diethyl oxide previously cooled to 5°C. Stopper the test tube and mix up immediately by inverting it twice. Return the test tube to the bath at 5°C and observe the colour.

After about one minute a red colour will appear if tea oil is present.

NOTE: A rose colour cannot be regarded as proof of the presence of tea oils, for some olive oils yield this colour.

SESAME OIL TESTS

A. Detection of Sesamoline

Apparatus:

- Graduated 50ml stoppered test tube

Reagents:

- Concentrated hydrochloric acid (d = 1.18)
- Solution of 2% freshly distilled furfurol in 95° alcohol

SESAME OIL TESTS (Continued)

Procedure:

Place 10ml of the oil and 10ml of hydrochloric acid in the graduated test tube. Stopper and shake energetically for 30 seconds. Leave to rest. Add 0.5ml of the solution of furfurol. Stopper and shake again. Leave to rest until decantation. If the bottom layer does not turn red, the test is negative.

If a red colouring appears, add 10ml of water and shake gently. Let the liquid settle. If the colouring disappears, the test is negative. If the colouring remains the test is positive. Refined sesame oils do not always give a positive reaction by this method.

B. Detection of Sesamine

Apparatus:

- 25ml, stoppered graduated test tube
- Decanting beaker of about 50ml
- Flat-bottomed porcelain capsule about 60mm in diameter

Reagents:

- Concentrated sulphuric acid (d = 1.84)
- Solution of 0.35 per mil freshly distilled furfurol in acetic anhydride

Procedure:

Place 10ml of the oil and 5ml of the solution of furfurol in the test tube. Stopper and shake vigorously for about one minute. Pour the mixture into the decanting beaker and allow to settle. Transfer a portion of the deposit into the capsule and add 6 or 7 drops of sulphuric acid. Mix by shaking the capsule gently.

The test is positive if a greenish-blue colour appears.

Sesame oils, even when refined, give a positive reaction.

SPECIFIC EXTINCTION IN ULTRA-VIOLET

Apparatus:

- Ultra-Violet spectrophotometer for measurements between 210 and 300mm
- Quartz cells of 1cm thickness
- 50ml and 500ml gauged phials
- Pipettes

Adjustment of equipment:

Dissolve 0.2g of dry potassium chromate in exactly 1 litre of a 0.05 N solution of potassium hydroxide. Place 25ml exactly measured, of this solution in a 500ml phial and bring up to 500ml mark with the 0.05N solution of potassium hydroxide. Determine the optical density of this latter solution by comparison with the 0.05N solution of potassium hydroxide as a reference solution, in a 1-cm cell. This, at 275nm should be 0.200 ± 0.005.

SPECIFIC EXTINCTION IN ULTRA-VIOLET (Continued)

Reagent:

- Spectrophotometrically pure cyclohexane: minimum transmittance at 220nm; 40% and minimum transmittance at 250nm: 95% by comparison with distilled water.

Procedure:

If the oil is not completely limpid at ambient temperature, filter before attempting measurements. Place approximately 0.5g, weighed accurately, of the oil in the 50ml gauged phial. Add the cyclohexane up to gauge mark and shake. Fill a cell with this solution and measure the optical density using the cyclohexane as reference solution. Make determinations at 232 and 270nm. Determine, in the region of 270nm, the m wavelength of maximum absorption and determine the optical density at m, m - 4 and m + 4, establishing for m - 4 and m + 4 the wavelengths 4nm above and below the maximum.

Expression of the result:

(a) Calculation of specific extinction

$$E\,{}^{1\%}_{1cm}\,\lambda = \frac{A\lambda}{cl}$$

where "$E\,{}^{1\%}_{1cm}\,\lambda$" marks the specific extinction at wavelength λ, "$A\,\lambda$" the optical density read on the spectrophotometer, "c" the concentration of the solution in g/100ml and "l" the thickness of the cell in centimeters.

If the optical density read is less than 0.2, re-measure with a more concentrated solution. If it is more than 0.8, re-measure with a weaker solution.

(b) Calculation of $\triangle E$ - Calculate the value of $\triangle E$ on the basis of:

$$\triangle E = E\lambda_m - \frac{E\lambda_{m-4} + E\lambda_{m+4}}{2}$$

(c) Determination of the specific extinction after passage through alumina

Place 30g of alumina for chromatography Brockman specification (loss at 300°C about 5%) in a chromatography column about 35mm in diameter and 45cm long, furnished with a draining tube of about 10mm diameter. Tamp the alumina mechanically by repeatedly tapping the column, held vertically, on a wooden surface. Place in the column thus prepared 100ml of a solution of 10% oil in hexane. Collect the drainings and evaporate the solvent in a vacuum at less than 25°C.

Using the oil so obtained, immediately determine the specific extinction at 270nm, as previously described.

NOTE: Measurement of the specific extinction in Ultra-Violet is essentially a measurement of the state of alteration of the oil. It is not specifically a measurement of the refining. In some particular cases, abnormally altered virgin oils can show spectral characteristics close to those of refined oils.

SOAP TEST

Apparatus:

- 150mm x 15mm test tube

Reagents:

- Solution of 0.1% of bromophenol blue in 96° ethyl alcohol
- Freshly distilled acetone, 2% water content

A few drops of the solution of bromophenol blue should give a yellow to yellow-green colour to the acetone with 2% water.

Procedure:

Place 10ml of the acetone and 1 drop of the solution of bromophenol blue in a test tube. The solution should have a yellow colour. If not, rinse the test tube with acetone until the blue colour disappears. Place 10g of the oil in the test tube, stopper with a clean stopper, shake and allow to settle. The lower acetonic layer should not have a blue colour, which would indicate the presence of soap.

A. PROPOSED METHODS OF ANALYSIS FOR PROCESSED FRUIT AND VEGETABLES
(Submitted to governments for comment at Step 3)

1. (a) Drained Weights (for: Canned Green Beans, Wax Beans, Peaches, Grapefruit, Asparagus, Pineapple and Green Garden Peas)

In accordance with the applicable Drained Weight Method for Processed Fruit and Vegetable Products of the "Methods of Analysis of the Association of Official Analytical Chemists" (latest edition) or in accordance with any other standardized method which gives equivalent results.

(b) Drained Weight (for: Canned Tomatoes only)

(i) 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.

(ii) 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.

(iii) Without shifting the tomatoes, so incline the sieve as to facilitate drainage of the liquid.

(iv) Allow to drain for two minutes.

(v) At the end of the two minutes draining period, ascertain the weight of the tomato material while still on the sieve, allowing for the tare (or weight of the sieve).

Specifications for circular sieves

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

(ii) 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).

(iii) The meshes of such sieves are made by so weaving wire of 0.054 inch (1.3716 mm) diameter as to form square openings of 0.446 inch (11.3284 mm) by 0.446 inch.

2. Alcohol Insoluble Solids (for: Canned Green Garden Peas)

In accordance with the applicable Alcohol Insoluble Solids Method for Processed Fruit or Vegetable Products in the "Methods of Analysts of of the Association of Official Analytical Chemists" (latest edition) or in accordance with any other standardized method which gives equivalent results.

3. Total Soluble Solids (for: Canned Applesauce)

Total soluble solids and/or Brix determination to be made by the refractometric method without corrections for insoluble solids or acidity but with corrections for temperatures to the equivalent at 20°C.

4. Tough String Test (for: Canned Green Beans and Canned Wax Beans)

A tough string is a string that will support the weight of 250 g (8 ozs) for five (5) seconds or longer.

5. Test for Calcium Salts (for: Canned Tomatoes)

Determined in accordance with:

(a) Method(s) outlined in the Official Methods of Analysis of the Association of Official Analytical Chemists (U.S.); or

(b) Method(s) which give comparable results such as those being developed by ISO TC 34/SC 3.

6. Syrup Measurements (for: Canned Grapefruit, Pineapple, Peaches)

Syrup measurements of "Cut-out" Brix shall be determined on the finished canned product in accordance with standardized methods by hydrometer or by refractometer.

B. METHODS OF SAMPLING FOR PROCESSED FRUIT AND VEGETABLES

1. Sampling (for: Canned Tomatoes, Green Beans, Wax Beans, Peaches, Grapefruit, Asparagus, Pineapple, Green Garden Peas)

Sampling shall be in accordance with the 'Proposed Sampling Plans for the Anality Assessment of Processed Fruit and Vegetables' (see para 14 of this Report and Appendix VI)

2. Size of Sample Unit (for Canned Pineapple)

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

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) 600 g of drained fruit (of a representative mixture) when the container holds more than 1.0 litre.

PROPOSED SAMPLING PLANS FOR THE QUALITY ASSESSMENT OF PROCESSED FRUITS AND VEGETABLES

(Submitted to the Codex Alimentarius Commission at Step 5)

SCOPE

The attached sampling plans provide tables for sampling and inspection of Processed Fruits and Vegetables. The plans include (1) Inspection Levels; (2) Sample Sizes in relation to lot size and container size; and (3) Acceptance (or Rejection) Criteria. These plans apply to the examination or testing of individual containers (or sub samples) drawn from a specific lot. They do not include detailed inspection procedures on how to examine the sample after it has been drawn. However, once the sample has been examined in accordance with the requirements of a standard or specification and a decision made, that the containers or sample units are either acceptable or "defective", the attached plans include acceptance criteria by which the lot can be classified as either meeting or failing the quality requirement of the standard.

The sampling plans are intended primarily to cover the quality provisions of the commodity standard. For the purposes of this instruction quality refers to those factors or product characteristics that are evaluated by organoleptic means or physical measurements. Examples of such characteristics are color, flavor, texture, defects, size and appearance. The plans may also be used for other determinations, such as Brix measurement, net weight or drained weight, provided acceptance criteria is based on an AQL of 6.5. They are not intended, however, to cover factors that might present a hazard to health or be unwholesome or highly objectionable to the consumer. Examples of these latter categories are pesticide residue, contaminants, blown cans, foreign material.

DEFINITIONS

Acceptable Quality Level (AQL) - the maximum percent defective units permitted in a lot that will be accepted approximately 95% of the time. For example, a sampling plan at an AQL of 6.5 will accept a lot or production that has 6.5% defective approximately 95% of the time.

Acceptance Number (c) - the number in a sampling plan that indicates the maximum number of "defectives" permitted in the sample in order to consider the lot as meeting the requirements of a standard or specification.

Consumer's Risk - the risk a consumer takes that a lot will be accepted by a sampling plan even though it may fail to conform to prescribed requirements.

Defective - a sample unit or container that fails to conform with the requirements of a standard or specification. For the purpose of processed fruit and vegetable acceptance inspection a defective is further qualified as follows:

(a) when the standard provides optional quality levels or classification a "defective" is a sample unit that fails the quality level specified but meets the next lower quality classification.

(b) when the standard provides a minimum or single quality level a "defective" is a sample unit that fails to meet such minimum quality requirements but only to the extent that is slightly below and would not be objectionable to the consumer.

Inspection - the process of measuring, examining, testing or otherwise comparing a container or unit of product with the requirements of a standard or specification.

Inspection level - the term used to indicate the relative amount of sampling performed on lots of a given product or class of products.

Lot - the term also means "Inspection lot" - collection of primary containers, or units, of the same size, type and style which has been manufactured or processed under essentially the same conditions.

Lot Size (N) - the number of primary containers, or units, in the lot.

Sample Unit - the individual container (primary container), a portion of the contents of the primary container or a composite mixture of product that is examined or tested as a single unit.

Sample - any number of sample units which are used for inspection. Generally the sample comprises all of the containers or sample units drawn for examination or testing purposes from a particular lot.

Sampling - the process of drawing or selecting containers or sample units from a lot or production.

SampleSize (n) - the number of containers, or units, comprising the total sample drawn from a lot or production.

Sampling Plan - a sampling scheme which includes samples sizes, inspection levels, acceptance and rejection numbers so that a decision can be made to accept or reject the lot or production based on the results of inspection and testing of the sample.

METHOD

In using the attached plans the following information must be known:

Container size
Inspection level
Lot size - number of primary containers in lot
Requirements of the standard or specification with respect to product quality

Knowing the above information the following steps are taken:

(1) Refer to the attached Sampling Table
(2) Select the Inspection Level deemed most appropriate for the situation, keeping in mind the following recommendations -

Level I - Screening procedure or small lots at retail level

Level II - Normal sampling

Level III - Disputes, enforcement or need for better lot estimate

(3) Convert lot size to number of primary containers.
(4) Determine number of sample units to draw from the inspection lot, consideration being given to container size, lot size and inspection level.
(5) Draw at random the required number of sample units from the lot giving proper consideration to code or other identifying marks in selection of the sample.
(6) Examine the product according to requirements of the standard or specification. Classify any container or sample unit that fails to meet the specified quality level of the standard as a "defective".
(7) Consider the lot acceptable if the number of "defectives" does not exceed the acceptance number (c) of the appropriate sampling plan.
(8) Consider the lot as failing if the number of "defectives" exceeds the acceptance number of the plan.

Example 1: "Screening" procedure (Inspection Level I)

A lot consists of 300 cans 28 ounces each. It is desired to sample the lot for general information as to label, color type, size of units and composition of syrup. Inspection Level I is selected in order to keep sample size as small as possible.

Lot Size - 300 containers
Container Size - 28 ounces (Table of less than 2.2 pounds)
Inspection Level - I
Sample Size - 1
Acceptance Number (c) - 0

If in testing the product it is found satisfactory, no further examination may be required. If, on the other hand, the container is mislabeled, the wrong color type, or otherwise unsatisfactory, the should be rejected without further testing.

NOTE: A sample size of 1 is not considered satisfactory for most purposes. However, there are times when decisions must be made on samples of 3 or less containers.

Example 2: Increased inspection (Inspection Level III)

A lot consists of 1200 cases packed 12 - $2\frac{1}{2}$ pound cartons per case. A decision is made to take a larger than normal sample because of possible dispute over product quality.

Lot Size - 1200 x 12 or 14,400 cartons
Container Size - 2½ pounds (Table of 2.2 to 10 pounds)
Inspection Level - III
Sample Size - 29
Acceptance Number (c) - 4

In the examination of the product if 4 or less cartons are classified as "defective" the lost is considered acceptable. If more than 4 cartons are "defective" the lot fails to meet acceptance criteria of the plan.

Example 3:- Normal inspection (Inspection Level II)

A lot consists of 800 cases packed 6 - 6½ pound containers per case.

Lot Size - 800 x 6 or 4,800 containers
Container Size - 6½ pounds (Table of 2.2 to 10 pounds)
Inspection Level - II
Sample Size - 6
Acceptance Number (c) - 1

In this example the lot is being offered and presented as Quality Level A in accordance with a standard that has three quality classifications, namely A, B and C. If in testing the product all of the containers are quality A, or 5 containers are quality A and 1 container quality B, the lot is considered acceptable for quality A requirements. If, on the other hand, 2 or more containers fail to meet quality A requirements; or, if any container falls below quality B requirements, the lot is considered unacceptable for quality A requirements. It would then be considered for the next lower level (quality B) and the same acceptance procedures would be applied for this lower quality level. If the lot fails quality B it would then be tested to determine if it would meet quality C requirements.

SAMPLING PLANS AND INSPECTION LEVELS
PROCESSED FRUITS AND VEGETABLES
(AQL 6.5)

LOT SIZE (Primary Containers)	INSPECTION LEVELS I		II		III	
	n	c	n	c	n	c
Net Weight Equal to or Less Than 2.2 Pounds (1 Kg.)						
2,400 or less	1	0	3	0	13	2
2,401-12,000	3	0	6	1	21	3
12,001-24,000	6	1	13	2	29	4
24,001-48,000	13	2	21	3	48	6
48,001-84,000	21	3	29	4	84	9
84,001-144,000	29	4	48	6	126	13
144,001-240,000	48	6	84	9	200	19
over 240,000	84	9	126	13	315	28
Net Weight Greater Than 2.2 Pounds but Not More Than 10 Pounds (4.5 Kg.)						
	n	c	n	c	n	c
1,200 or less	1	0	3	0	13	2
1,201-7,200	3	0	6	1	21	3
7,201-15,000	6	1	13	2	29	4
15,001-24,000	13	2	21	3	48	6
24,001-42,000	21	3	29	4	84	9
42,001-72,000	29	4	48	6	126	13
72,001-120,000	48	6	84	9	200	19
over 120,000	84	9	126	13	315	28
Net Weight Greater Than 10 Pounds (4.5 Kg.)						
	n	c	n	c	n	c
300 or less	1	0	3	0	13	2
301-1,200	3	0	6	1	21	3
1,201-2,000	6	1	13	2	29	4
2,001-7,200	13	2	21	3	48	6
7,201-15,000	21	3	29	4	84	9
15,001-24,000	29	4	48	6	126	13
24,001-42,000	48	6	84	9	200	19
over 42,000	84	9	126	13	315	28

n = number of primary containers in sample c = acceptance number

LIST OF METHODS OF ANALYSIS FOR PRESERVATIVES REFERRED TO THE CODEX COMMODITY COMMITTEES CONCERNED

Note by the Secretariat

This list has been taken from a paper prepared by the Delegation of the Netherlands, SP 10/101, Codex Analys/67-10 which has been circulated to Codex Contact Points. Those concerned wishing to comment on the Methods of Analysis listed in this Appendix are requested to consult the original paper in which the various methods are fully discussed and compared. Copies of the Netherlands paper can be obtained from the Chief, Food Standards Branch, FAO, Rome.

I. Sulfur dioxide (total sulfurous acid)

I,1 Qualitative

I,1.a. Kaplan E., J.Am.Offic.Agric. Chemists 44, 485 (1961)

Meat — "Malachite Green Test for presence of sulfite in meat"

I,1.b. Roemmele O., Arch.Levenam.Hyg. 7, 278 (1956)

"Simple, rapid and sure method for the detection of sodium sulfite in ground meat with potassium iodate-starch paper"

I,2 Quantitative

Fruit pulp and Fruit dried	I,2,a. Principle Monier-Williams, modification of Zonneveld, procedure described by Tanner
Fruit dried	I,2.b. Nury F.S. and H.R. Bohm, J.Assoc. Offic.Anal.Chemists 48, 796 (1965)
Fruit juice chemically preserved for direct consumption	I,2.c. Principle Monier-Williams, procedure described by Tanner
Fruit pectin liquid for domestic use Jams, jellies and marmalades	I,2.d. Principle Monier-Williams, modification Zonneveld, procedure described by Tanner
Glucose syrup for manufacturing purposes. Sugar white	I,2.e. Principle Monier-Williams, procedure described by Tanner
Vegetables dried Potatoes dried	I,2.f. Principle Monier-Williams, modification Zonneveld, procedure described by Tanner

I. Sulfur dioxide (continued) — I,2 Quantitative

Beer and wines

I,2.g. Principle Monier-Williams, procedure described by Tanner

II. Sorbic acid — II,1 Qualitative

II,1.a. Genest Chr. and D.G. Chapman, J.Assoc.Offic.Agric.Chemists 43, 438 (1960)

Fruit products
margarine
cheese
preserved fish

"Qualitative extraction of certain antimicrobial preservatives from foods"

II,1.b. Gosselé J.A.W. et al., J.Chromatog. 23. 305 (1966)

"Thin-layer chromatographic separation of preservatives"

II,2 Quantitatives

II,2.a. Roose J.B. and A. Versnel, Chem. Weekblad 55, 521 (1959)

Margarine

"Spectrophotometric determination of sorbic acid in margarine and butter"

II,2.b. Schmidt H, Deuts.Lebensm.Rundschau 58. 1 (1962)

Wine
Fruit wine

"Colorimetric determination of sorbic acid in wine"

Non-alcoholic foods, such as fruit juices, dried fruit, jams and fruit juice bases

II,2.c. Schmidt H, Z.Anal.Chem. 178, 173 (1960)

"A specific method for the determination of sorbic acid"

II,2.d. Nury F.S. and H.E. Bolin, J.Food Sci. 27, 370 (1962)

"Colorimetric assay for potassium sorbate in dried fruits"

II,2.e. Carr W. and G.A. Smith, J.Assoc. Public Analysts 2, 37 (1964)

"Determination of sorbic acid in dried prunes and prunes in syrup.

III. Nitrate and nitrite	III,1 Qualitative	
Meat and meat products	III,1.a. Diemair W, in "Laboratoriumsbuch für den Lebensmittelchemiker" 8th ed.(1963)	
	III,2 Quantitative	
	III,2.a. International Organization for Standardization (ISO)	
Meat and meat products	"Determination of the nitrite content of meat and meat products"	
	III,2.b. International Organization for Standardization (ISO)	
	"Determination of the nitrate content of meat and meat products"	
Fish and fishery products	With regard to the analysis of nitrate and nitrite in fish and fishery products, further information is needed. No methods can be suggested at this moment.	
	III,2.c. Hänni H, Mitt.Geb.Lebensm.Unters. u Hyg. 42, 114 (1951)	
Milk and milk products a/	"Method for the determination of the nitrite content of milk and milk products"	
	III,2.d.Hänni H, Mitt.Geb.Lebensm.Unters u Hyg. 42, 114 (1951)	
	"Method for the determination of the nitrate content of milk and milk products"	
IV. Benzoic acid	IV,1 Qualitative	
Non-alcoholic and alcoholic liquids Solid and semi-solid substances	IV,1.a. Official methods of Analysis of the Association of Official Agricultural Chemists, 10th Ed., 1965,p.450 (27.002 - 27.004)	
	IV,1.b. Gosselé J.A.W. et al., J.Chromatog. 23, 305 (1966)	
	"Thin-layer chromatographic separation of preservatives"	

a/ Referred to the Committee on Milk and Milk Products (see para 23)

IV. Benzoic acid (continued) IV,2 Quantitative

IV,2.a. Roos J.B. and A.Versnel, Chem. Weekblad 55, 67 (1959)

Margarine

Sorbic acid can be determined in the same way (see II,2). A method for the analysis of both sorbic acid and benzoic acid is given by the same analysts in Deuts. Lebensm.Rundschau 56, 128 (1960). The concentration of the acids is determined by using the absorption peaks at 228 nm (benzoic acid), 258 nm (sorbic acid) and 233,3 nm (isobestic point).

IV,2.b. Stanley R.L., J.Assoc.Offic.Agric. Chemists 42, 486 (1959); ibid 43, 587 (1960)

Tomato products, jam, jellies, beverages, soft drinks and fruit juices

"Benzoic acid in foods"
The method is adopted by the AOAC and published in the Official Methods of Analysis of the Association of Official Agricultural Chemists, 10th ed.,1965,p.451(27.007-27.009).

IV,2.c. Official Methods of Analysis of the Association of Official Agricultural Chemists, 10th ed.,1965, p.450 (27.005 - 27.006).

Ibid and salted or/and dried fish

IV,2.d. Stanley R.L., J.Assoc.Offic.Agric. Chemists 46, 616 (1963)

Meat

"Rapid screening procedure for benzoates in meat"

LIST OF METHODS OF ANALYSIS FOR ANTIOXIDANTS REFERRED TO THE CODEX COMMODITY COMMITTEES CONCERNED

Note by the Secretariat

This list has been taken from a paper prepared by the Delegation of the Netherlands, SP 10/101, Codex Analys/67-8, which has been circulated to Codex Contact Points. Those concerned wishing to comment on the methods of analysis listed in this Appendix are requested to consult the original paper in which the various methods are fully discussed and compared. Copies of the Netherlands paper can be obtained from the Chief, Food Standards Branch, FAO, Rome.

I. Gallates

I,1. Qualitative

I,1.a. Schwien W.G., and H.W. Conroy, J.Am. Offic.Agric.Chemists 48, 489 (1965)

Fats and Oils

"Qualitative analysis of propyl gallate, nordihydroguaiaretic acid, butylated hydroxy anisol and butylated hydroxy toluene in fats and oils"

The method is adopted by the AOAC (Official first action) and published in the Official Methods of Analysis of the Association of Official Agricultural Chemists, 10th Edition, 1965, p.443.

I,1.b. Scheidt S.A. and H.W.Conroy, J.Am. Offic.Anal.Chemists 49, 807 (1966)

"Detection of PG, NDGA, BHT and BHA in fats and oils by thin-layer chromatography"

I,1.c. The extraction of the gallates (propyl- octyl- and dodecyl gallate) can be done in a way as suggested by Vos H.J. et al., The Analyst 82, 362 (1957)(see I,2.a). Subsequent separation and detection of the gallates as described by Salo T. and K.Salminen, Z.Lebansm. Unters.u Forsch. 125, 167 (1964).

I,1.d. Frequently used is the method by which the gallates are extracted from fat or oil with ethanol (72%). Biefer K.W., Mitt.Lebensm.Hyg. 53, 243 (1962) Mahon J.H. and R.A.Chapman, Anal.Chem. 23, 1116 (1951).

Other foods

Extraction of the fat with petroleum ether followed by I,1.a., I.1.b. (propyl gallate only) or I,1.c.(PG, OG and DG)

I. Gallates (continued)

I,2. Quantitatives

I,2.a. Vos H.J. et al., The Analyst 82, 362 (1957)

Fats and Oils

"The quantitative determination of the antioxidants propyl-, octyl-, dodecyl gallate in fats and oils"

I,2.b. Sahasrabudhe M.R., J.Assoc.Offic. Agric.Chemists 47, 888 (1964)

"Application of thin-layer chromatography to the quantitative estimation of antioxidants: BHA, BHT, PG and NDGA".

I,2.c. Raadsveld C.W. and E.G. Kooy, Neth. Milk and Dairy J. 15, 282 (1961)

Milk powder a/

"Quantitative determination of dodecyl gallate and BHA in milk powder"

II. Butylated hydroxy anisol (BHA)

II,1. Qualitative

II,1.a. Schwien W.G. and H.W. Conroy, J.Am. Offic.Agric.Chemists 48, 489 (1965)

"Qualitative analysis of propyl gallate, nordihydroguaiaretic acid, butylated hydroxy anisol and butylated hydroxy toluene in fats and oils".

The method is adopted by the AOAC (official first action) and published in the Official Methods of Analysis of the Association of Official Agricultural Chemists, 10th Edition, 1965, p.443.

II,1.b. Scheidt S.A. and H.W. Conroy, J.Am. Offic.Anal.Chemists 49, 807 (1966)

"Detection of PG, NDGA, BHT and BHA in fats and oils by thin-layer chromatography"

After extraction of the antioxidants as suggested by Schwien (see II,1.a.) or Sahasrabudhe, J.Am,Offic.Agric.Chemists 47, 888 (1964), subsequent separation and detection of BHA can be done as described by:

a/ Referred to the Committee on Milk and Milk Products (see para.25)

II. Butylated hydroxy anisol (BHA)(Continued)

II,1.c. Salo T. et al., Z.Lebensm.Unters. u Forsch. 125, 450 (1964)

"Thin-layer chromatography of antioxidants.

II.BHA, BHT, NGDA and tocoferol"

II,1.d. Davidek J. et al., Z.Lebensm,Unters u Forsch. 131, 345 (1967)

"Thin-layer chromatography of antioxidants"

II,1.e. Giannone L. Ind.Conserva 38, 209 (1963) via Chem.Abstr. 62, 13762 (1965)

Bouillon preparations — "Detection of BHA in bouillon preparations"

II,2 Quantitative

II,2.a. Sloman et al. J.Am.Offic.Agric. Chemists 45, 76 (1962)

Fats and Oils and other foods — "Trace analysis of BHA and BHT in food products"

II,2.b. Nordisk Metodik-Komité for Levneds-midler. Nr.50, 1963

"Quantitative determination of the anti-oxidants BHA and BHT in fats"

II,2.c. Sahasrabudhe M.R., J.Assoc.Offic. Agric.Chemists 47, 888 (1964)

"Application of thin-layer chromatography to the quantitative estimation of anti-oxidants: BHA, BHT, PG and NDGA"

III. Butylated hydroxy toluene (BHT)

III,1 Qualitative

III,1.a. Schwien W.G. and H.W. Conroy, J.Am. Offic.Anal.Chemists 48, 489 (1965)

Fats and Oils — "Qualitative analysis of PG, NDGA, BHA and BHT in fats and oils"

The method is adopted by the AOA (Official first action) and published in the Official Methods of Analysis of the Association of Official Agricultural Chemists, 10th Edition, 1965, p.443.

III. Butylated hydroxy toluene (BHT)(continued)

III.1 Qualitative (continued)

III,1.b. Scheidt S.A. and H.W. Conroy, J.Am. Offic.Anal.Chemists 49, 807 (1966)

"Detection of PG, NDGA, BHT, and BHA in fats and oils by thin-layer chromatography"

After separation of the antioxidants as suggested by Schwien (III,1.a.) or Sahasrebudhe (see I,2.b.) subsequent separation and detection of BHT can be done as described by

III,1.c. Salo T. et al., Z.Lebensm.Unters.u Forsch. 125, 450 (1964)

"Thin-layer chromatography of antioxidants II.BHA, BHT, NDGA and Tocoferol".

III,1.d. Davidek J. et al., Z.Lebensm. Unters. u Forsch. 131, 343 (1967)

"Thin-layer chromatography of antioxidants".

III,2. Quantitative

III,2.a. Szalkowski C.R. and J.B. Garber, J.Agr.Food Chem. 10, 490 (1962)

Fats and Oils

"Determination of BHT: application to edible fats and oils".

III,2.b. Sahasrabudhe M.R., J.Assoc.Offic. Agric.Chemists 47, 888 (1964)

Fats and Oils

"Application of thin-layer chromatography to the quantitative estimation of antioxidants BHA, BHT, PG and NDGA".

III,2.c. Sloman K.G. et al.J.Assoc.Offic. Agric.Chemists 45,76 (1962)

"Trace analysis of BHA and BHT in food products"

III,2.d. Nordisk Metodik-Komité for Levneds-midler. Nr.50, 1963

"Quantitative determination of the anti-oxidants BHA and BHT in fats".

DRAFT PROVISIONAL STANDARD FOR THE TECHNICAL PROCEDURE OF SAMPLING FOODS

(Submitted to governments for comment at Step 3)

1. SCOPE

Procedures are described for sampling foods to obtain from a unit (e.g. bulk container, small retail container, etc...) a portion (sub-sample) which is as representative as possible of that unit.

2. GENERAL INSTRUCTIONS

2.1 Instructions of an administrative character

2.1.1. Sampling should be performed by an authorized or sworn independent agent, properly trained in the appropriate technique. The agent should be free from any infectious disease.

2.1.2. If possible, representatives of the parties concerned should be given the opportunity to be present when sampling is performed.

2.1.3. Samples should be accompanied by a report, signed by the sworn or authorized sampling agent and counter-signed by any witnesses present.
This report should give particulars of the place, date and time of sampling, the name and designation of the agent and of any witnesses, the precise method of sampling which is followed if this deviates from the prescribed standard method, the nature and number of the units constituting the consignment together with their batch code markings where available, the number of samples duly identified as to the batches from which they were drawn, and the place to which the samples will be sent.
When appropriate, the report should also include any relevant conditions or cirsumstances, for example the condition of the packages and their surroundings, temperature and humidity of the atmosphere, method of sterilization of the sampling equipment, whether a preservative substance has been added to the samples, and any other special information relating to the material being sampled.

2.1.4. Each sample should be sealed and labelled to give the nature of the product, the identification number and any code markings of the batch from which the sample has been taken, the date of sampling, and the name and signature of the sampling agent.
When necessary, additional information may be required, as, for example, the mass of the sample and the unit from which it was taken.

2.1.5. All samples should be taken at least in duplicate, one set being held, if necessary, in cold storage and put as soon as possible at the disposal of the second party.
It is recommended that, when previously agreed between the parties, additional sets of samples be taken and retained for independent arbitration if necessary.
The samples should be dispatched immediately after sampling to the testing laboratory.

2.2 Technical instructions

2.2.1. Sampling equipment

2.2.1.1. Specifications: as laid down for each product to be sampled.

2.2.1.2. Sampling for chemical purposes: the sampling equipment and sample containers should be dry and clean.

2.2.1.3. Sampling for bacteriological purposes: all sampling equipment should be clean and should be treated by one of the following methods:

(a) Exposure to hot air at 170°C for two hours (may be stored if kept under sterile conditions).

(b) Exposure to steam at 120°C (autoclave) for fifteen to twenty minutes (may be stored if kept under sterile conditions).

(c) Exposure to steam at 100°C for one hour (equipment should be used the same day).

(d) Immersion in water at 100°C for one minute (equipment should be used immediately).

(e) Immersion in 70% ethanol and flaming to burn off the ethanol immediately before use.

(f) Exposure to hydrocarbon (propane, butane) torch flame so that all working surfaces contact the flame immediately before use.

The choice of the treatment will depend upon the nature, shape and size of the equipment and upon the conditions of sampling. Sampling equipment should be sterilized wherever possible by one of the methods (a) or (b).

Methods (c), (d), (e) and (f) should be regarded as secondary methods only.

2.2.2. Sample containers

2.2.2.1. For liquids

Containers should be made of suitable, waterproof, greaseproof material (glass, stainless metal, suitable plastic material), of a quality suitable for sterilization by the methods given in 2.2.1.3 if necessary and of a suitable shape and capacity for the material to be sampled (as defined in each particular case). They should be clean and dry.
Containers should be securely closed either by means of a suitable rubber or plastic stopper or by a screw cap of metal or plastic having, if necessary, a liquid-tight plastic liner which is insoluble, non-absorbent, greaseproof, and which will not influence odour, flavour or composition of the sample.
If rubber stoppers are used these should be covered with non-absorbent, flavourless material (such as a suitable plastic) before pressing into the sample container. Suitable plastic bags may also be used.

2.2.2.2. For solids or semi-solids

Containers should be wide mouth, cylindrical receptacles of suitable, waterproof, greaseproof material (glass, stainless metal, suitable plastic material), of a quality suitable for sterilization by the methods given in 2.2.1.3 if necessary, and of a capacity suited to the size of the sample to be taken (as defined in each particular case). They should be clean and dry. They should be made air-tight by one of the means defined in 2.2.2.1. Suitable plastic bag may also be used.

2.2.2.3. Small retail containers

The contents of the intact and unopened containers should constitute the samples.

2.2.3. Sampling technique

The precise method of sampling and the mass or volume of product to be taken as a sample vary with the nature of the products and the purpose for which sampling is required, and are defined for each particular case.

2.2.4. Preservation of samples

2.2.4.1. To samples of liquid products or cheese required for chemical analysis a suitable preservative may be added. Such preservatives should not interfere with the subsequent analysis and the nature and quantity of the addition should be indicated on the label and in any report. Preservatives should not be added to samples of semi-solid, solid (except cheese) or dried products intended for chemical analysis. Samples should be rapidly cooled and stored in a refrigerator at a temperature between 0°C and +5°C.

2.2.4.2. Preservatives should not be added to samples intended for bacteriological or organoleptic examination. Instead, they should be held at a low temperature (0°C to + 5°C) except for conserved products when the sample comprises unopened hermetically sealed containers in which the products are sold. Liquid and semi-liquid products should be kept cold and bacteriological examination of liquid products should start as soon as possible and never later than twenty-four hours after sampling.

2.2.5. Transport of samples

Samples shall be transported to the laboratory as quickly as possible after sampling. Precautions should be taken to prevent, during transit, exposure to direct sunlight, to temperatures below 0°C, or to high temperatures, which should not exceed 10°C in the case of perishable products. For samples intended for bacteriological examination, an insulated transport container capable of maintaining a low temperature (0°C to + 5°C) should be used, except for samples of conserved products in unopened containers, or in the case of very short journeys.