CODEX ALIMENTARIUS COMMISSION

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World Health Organization

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Agenda Item 2 and 3 **MAS/36 CRD/2**

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

36th Session
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REPORT OF THE WORKING GROUP ON THE ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

The Working Group on the endorsement of methods of analysis and sampling was held on Saturday 21 February 2015, prior to the 36th Session of the Committee on Methods of Analysis and Sampling. The Working Group was chaired by Dr Roger Wood (ICUMSA) and co-chaired by Dr Gregory Noonan (USA). The WG considered the methods of analysis and sampling submitted for endorsement (CX/MAS 15/36/3 and CX/MAS 15/36/3 Add.1) and matters related to methods of analysis referred to the Committee by the CAC and other Codex committees (CX/MAS 15/36/2).

The recommendations are tabulated in the Appendix.

A. Endorsement of methods of analysis and sampling

Codex Committee on Processed Fruits and Vegetables (CCPFV)

Standard for Certain Canned Fruits

Methods of analysis

All the proposed methods were endorsed; these methods had been previously endorsed by CCMAS in similar CCPFV Standards. The WG also considered the request from CCPFV (CX/MAS 15/36/2) to identify an alternative internationally validated method to replace CAC/RM 46 1972 method for fill of glass containers. The WG considered whether the method ISO 90-1:1997 for fill of metal containers was applicable to glass containers. The representative of ISO explained that no validation studies had been conducted to that effect.

The WG recommended that ISO be requested to consider the applicability of ISO 90-1:1997 to glass containers and to report back on their findings to CCMAS37. If not suitable it was requested whether any alternative ISO procedure (e.g. ISO 8106 (Glass containers — Determination of capacity by gravimetric method)) was available and suitable.

Sampling plan

The Committee recommended the endorsement of the attribute sampling plans as proposed by CCPFV. It was noted that the number of units to be taken for analysis was somewhat less than that recommended in CAC/GL 50-2004 but consistent with previous plans adopted by CCPFV.

Standard for Ginseng and Ginseng Products

Methods of analysis

The WG recommended the endorsement of the methods for moisture, solids, ash, water-insoluble solids, water-saturated n-butanol extracts for endorsement as Type I noting the extensive interlaboratory validation studies that had been carried out by the Republic of Korea (ROK) in order to support the re-classification of the methods from Type IV to I.

The WG however recommended the retention of the method for identification ginsenosides Rb1 and Rf as Type IV since the validation studies were only conducted on known ginseng products and were not tested against non-ginseng products to validate its efficiency.

The WG recommended that ROK should formally publish their validation studies in public literature. In the case of the identification of ginsenosides, the typing of the method would be reconsidered upon publication.

Sampling Plans

The WG did not recommend endorsement of the sampling plans since the values in the table did not correspond to those recommended in the Guidelines for Sampling (CAC/GL 50-2004) and proposed that a revised sampling plan should be provided by CCMAS for consideration by CCPFV. Such a plan may be better based on a variables rather than an attribute approach.

Codex Committee on Contaminants in Foods (CCCF)

Sampling Plans

The WG did not recommend endorsement of the sampling plans noting that there were inconsistencies between the number of incremental samples to be taken and the aggregate sample weights specified. If an additional weight reduction stage is to be included in the procedure to obtain the aggregate samples weights specified, then that should be clearly described.

The heading of the first column of Table 1 and 2 is to be “Lot weight (tonnes) and that of the second column of Table 1 is to be “Weight (tonnes) or number of sublots”.

Analytical method

The WG did not recommend endorsement of the performance criteria as these were not considered consistent with those given in the Procedural Manual “Guidelines for establishing numerical values for criteria”. CCCF should consider aligning the numerical values if appropriate, and if it is not appropriate to do so, to provide clarification for the numerical values currently given. It was noted that both the precision values and the recovery values were inconsistent with those given in the Procedural Manual.

Codex Committee on Fats and Oils (CCFO)

Sampling Plan

The WG recommended endorsement of the sampling plan proposed by CCFO.

Methods of Analysis

Determination of fatty acid composition

The WG recommended endorsement of the ISO methods as Type III. The WG noted that while ISO 5508 had been superseded by ISO 12966-2, it was still maintained, as this method was still effective and would be used until it is withdrawn.

Determination arsenic and lead

The WG noted that there were many other methods that could be used as for the determination of these heavy metals and recommended that the criteria be developed once the specification level is finalised. Suitable methods which are expected to be satisfactory are listed (see Appendix I).

Determination of Acid Value

The WG recommended endorsement of the AOCS and ISO methods as Type I as these methods are equivalent. The WG did not recommended endorsement of the European Pharmacopoeia 2.5.1 as it was unclear whether this method was equivalent to the other methods proposed and that clarification was needed before this method could be endorsed.

Determination of peroxide value

The WG recommended not to endorse the European Pharmacopoeia 2.5.5 as this method uses chloroform as a reagent noting the previous decision of CCMAS not to endorse such methods.

Determination of Vitamin A

It was proposed that the European Pharmacopoeia reference be clarified and an alternative CEN procedure be included. The two methods are:

- European Pharmacopoeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29.
- EN 12823-1(Determination of vitamin A by high performance liquid chromatography - Part 1: Measurement of all-E-retinol and 13-Z-retinol)

Determination of Vitamin D

It was proposed that the European Pharmacopoeia reference be clarified, and its applicability. Alternative CEN and NMKL procedures be included. The two methods are:

- European Pharmacopoeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29.
- EN 12821 (Determination of vitamin D by high performance liquid chromatography - Measurement of cholecalciferol (D3) or ergocalciferol (D2))
- NMKL 167 (Cholecalciferol (vitamin D3) and Ergocalciferol (vitamin D2). Determination by HPLC in foodstuffs).

The applicability of the European Pharmacopoeia method was questioned as it only determined vitamin D3 and CCFO should therefore be asked to clarify if that was sufficient. The WG therefore recommended temporarily endorsement.

Determination of phospholipids

The WG recommended the endorsement of the methods proposed as Type III if expressed as phospholipids.

B. Matters referred

Methods for determination of marine biotoxins

The WG noted the divergent views on the typing of the MBA to retain the current Typing or to reclassify the methods.

Recommendations were made to consider this matter in light of the discussions on procedures for determining equivalency to Type I methods (Item 5) and the discussion paper on the criteria approach for methods which use a sum of components (Item 6).

The WG agreed that this matter would be better dealt with in plenary and made no recommendations.

Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU)

Methods for dietary fibre

The WG recommended that AACC Intl 32-50.01 be adopted as proposed by CCNFSDU.

The WG retained the AOAC methods 2009.01 and 2011.25 as Type I methods, noting that these methods were still equivalent to the related AACC 32-45.01 and 32.50.01, respectively since AOAC had not modified these methods.

Trans fatty acids

The WG considered the request from CCNFSDU on the lowest level of TFAs that current analytical methods can accurately detect as well as consistently reproduce.

The WG noted that it would be difficult to provide such information to CCNFSDU as the levels obtained would depend on the matrix of the product, and in addition at total TFA levels below 0.1g/100g product, results become variable. It would be more appropriate for CCNFSDU to provide the CCMAS with the levels for total TFA and the matrix to which the level applies.

The WG noted that analysis in some matrices had been carried out by ISO, IDF and AOAC, and give some preliminary results (see CRD16), and that the method will be published by the end of 2015.

Method for detection of the toxic fraction in gluten harmful for individuals

The WG noted that it was not possible to have two Type I methods and that if ELISA G12 method were to be added, the provision in the Standard would need to be differentiated to allow for both methods to be included as Type I methods. The WG noted that ELISA G12 had been validated for gluten free foods, rice matrices, whereas R5 had been validated for gluten-free foods, maize matrices. Both methods had recently been fully validated by collaborative trial and are published by AACCI as:

R5 method: AACC Intl 38-50.01 (immunoassay procedure (validated using maize matrices)) and

G12 method: AACC Intl 38-52.01 (immunoassay procedure (validated using rice matrices))

The WG recommended that decision in this regard should be taken by CCNFSDU.