CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

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ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS

Validation studies for the amended version of AOAC 963.15 (report by Indonesia)

Background

At CCMAS36, the Committee noted that CCASIA had agreed to replace the method of analysis for lipid content with ISO 1211|IDF 1:2010 as proposed by CCMAS in order to replace AOAC 983.23 which used chloroform as a reagent. It was pointed out that the scope of the ISO 1211|IDF 1:2010 did not include solid foods, such as tempe and that IDF and ISO did not intend to carry out work to extend the scope at this stage. The Committee agreed to retain the current method AOAC 983.23 for lipid determination in tempe and to request information from, in particular countries in the Asia region as to the applicability of the methods to tempe and whether this method had been tested on tempe products (REP 15/MAS para 39).

At CCMAS37, the delegation of Indonesia confirmed that the method for lipid determination contained in ISO 1211|IDF 1:2010 had not been tested on tempe products in Indonesia. Indonesia shares the same concern raised by CCMAS regarding AOAC 983.23 which used chloroform as a reagent. Indonesia proposes the previous method AOAC 963.15 as stated in Annex I of CL 2012/13-ASIA (Rev) to be further considered as an alternative to replace AOAC 983.23. This is the method for determination of Fat in Cacao Products - Soxhlet Extraction Method.

The Committee, based on the information received, reconfirmed AOAC 983.23 for determination of lipids in tempe. The Delegation of Indonesia informed the Committee that they were using an amended version of the soxhlet extraction method for determination of fat in cocoa products. This method, AOAC 963.15, was considered more applicable for fat content in tempe. The Committee encouraged Indonesia to carry out validation studies for this method in tempe products (REP 16/MAS para 41).

The following are validation studies carried out by Indonesia in response to the decision of the Committee.

Inter-laboratory validation test

The inter-laboratory validation test was performed by 8 participating laboratories in Indonesia of which are accredited under ISO/IEC 17025:2005. The participants come from government and private laboratories. The inter-laboratory program was coordinated by National Accreditation Body of Indonesia (KAN) and KAN appointed Directorate of Standardization and Quality Control, Ministry of Trade, to prepare the inter-laboratory sample. The name of all laboratories are shown in Appendix I.

The test method was conducted according to the AOAC 963.15 (Method of Analysis for Fat in Products). The method performance criteria was defined as described in Codex: Principles for the Establishment of Codex Methods of Analysis. According to this criteria, the HORRAT Value should be ≤ 2 to check the method precision, obtained from collaborative method performance studies.

The inter-laboratory validation test was started by checking laboratories performance to determine fat content in Cacao CRM. Each participant was required to analyse 2-3 replicates of the Cacao CRM.

Before Tempe sample was distributed and analysed by participants, it has been homogenized and stabilized based on ISO 13528:2015. Finally, the Tempe sample was tested 2-3 replicates by each participant for the reproducibility and repeatability criteria of the method with one level concentration.

Results

Table 1 shows the results of fat content analysis performed by 8 laboratories. The data processing was performed under ISO 5725 part 2. The results present 2-3 replicates analysis of each participant with RSD values range between 0,2% - 2,6%.

The results presented in the Table 1 perform the method precision only, while the accuracy was not included as the CRM of Tempe sample has not been provided yet. Codex requires the method precision criteria to meet HORRAT value which is the ratio of the reproducibility relative standard deviation (RSD_R) to the predicted reproducibility relative standard deviation ($PRSD_R$), i.e.:

$$\text{HORRAT} = \frac{\text{RSD}_R, \%}{\text{PRSD}_R, \%}$$

Where $PRSD_R$, % = $2C^{-0.1505}$ and C = the estimated mean concentration. HORRAT values equal or less than 2 may be taken to indicate that the performance value for the method corresponds to historical performance.

The RSD_R was obtained from collaborative method performance studies of 8 participating laboratories, whereas the $PRSD_r$ obtained from Horwitz equation. This study produced HORRAT value of 1,94 which is less than 2. It means that the method of AOAC 963.15 is applicable for determination of fat content in Tempe sample.

Table 1. Results of fat content analysis

Lab	Fat Content (%)	Mean	SD	RSD (%)
01	10,70	10,92	0,196	1,791
	11,08			
	10,97			
02	11,59	11,73	0,146	1,243
	11,71			
	11,88			
03	10,62	10,65	0,031	0,287
	10,68			
	10,66			
04	10,79	11,04	0,290	2,628
	10,98			
	11,36			
05	9,71	9,69	0,035	0,365
	9,66			
06	11,20	11,21	0,061	0,545
	11,28			
	11,16			
07	10,92	10,87	0,062	0,575
	10,89			
	10,80			
08	10,14	10,28	0,118	1,153
	10,34			
	10,35			

Grand mean	10,85
s_r	0,15
RSD_r	1,38
s_R	0,59
RSD_R	5,42
$PRSD_R$	2,79
HORRAT	1,94

Note:

Mean = Average value of each Laboratory

SD = Standar Deviation of each Laboratory

s_r = Standard Deviation of repeatability

RSD_r = Relative Standard Deviation of repeatability

s_R = Standard Deviation of reproducibility

RSD_R = Relative Standard Deviation of reproducibility

$PRSD_R$ = Predicted Value for Relative Standard Deviation of reproducibility (counted as Horwitz value)

HORRAT = Ratio of the reproducibility relative standard deviation to the predicted reproducibility relative standard deviation

Recommendations

Since the HORRAT Value of 1,94 is less than 2, it has fulfilled the method performance criteria as described in Codex: Principles for the Establishment of Codex Methods of Analysis. It means that the method of AOAC 963.15 is applicable for determination of fat content in Tempe sample.

Therefore, Indonesia proposes the CCMAS to endorse AOAC 963.15 (Method of Analysis for Fat in Cacao) for Tempe as Type I Method.

Appendix I. List of Participating Laboratories in Alphabetical Order

1. Bogor Agricultural University Laboratory
2. Centre for Agro Based Industry Laboratory
3. Food Laboratory, Directorate of Standardization and Quality Control
4. National Testing Center for Drug and Food Laboratory
5. PT Indofood CBP Sukses Makmur Laboratory
6. PT MBRIO Food Laboratory
7. PT Saraswanti Indo GeneTech Laboratory
8. Spices Laboratory, Directorate of Standardization and Quality Control