CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 5 **ASIA21/CRD2**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

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MATTERS ARISING FROM THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES

Validation data for methods of analysis for Laver products

Comments of Republic of Korea

Background

At CCMAS38, the Committee did not endorse the methods for acid value and agreed to request clarification from CCASIA whether the provision “acid value” applied to the laver product itself, or the extracted oil. If the method was for the extracted oil, it could be endorsed as Type I.

The Committee further noted that the extraction method in the Standard for laver products had been validated for instant noodles and not for laver, and that in this case, a classification as Type IV was recommended, and encouraged CCASIA to submit validation data to CCMAS to reconsider the proposed typing (REP 17/MAS para16).

In response to the decision of the Committee, the following validation test was carried out by Republic of Korea.

Inter-laboratory validation test for Acid value

This inter-laboratory validation test for Acid value was carried out by 3 laboratories from Republic of Korea and 1 laboratory from Japan, which are accredited under ISO/IEC 17025:2005. Korea Food Research Institute (KFRI) coordinated the inter-laboratory program and prepared the samples for validation test. The name of participating laboratories is shown in APPENIDX I.

Seasoned laver produced by the same manufacturer on the same date was used for the test samples in this validation test. Furthermore, after vacuum packing process under the same condition on the same date, each sample was distributed and tested 6 replicates.

Each participating laboratories were required to handle test samples under the following conditions.

1. Store all test samples at room temperature (21 ~ 25℃)

2. Discard remaining test samples. Do not reuse any samples after analysis.

3. Always keep the laboratory at room temperature with relative humidity between 25% and 30% to prevent condensation on the wall.

Extraction of oil

The test method for extraction of oil was conducted according to the Regional standard for Laver products as follows.

Weigh 50 g of test sample into 1000 mL Erlenmeyer flask. Add 500 mL of petroleum ether to the flask followed by replacing air in the flask by N_2 gas. Put a stopper on the flask and let stand for 2 hours. Decant the extracted solution (A) through a filter paper, on which Na_2SO_4 is mounted to remove moisture, on a funnel into 1000 mL round flask-flat bottom. Add additional 250 mL of petroleum ether to residue in the Erlenmeyer flask and decant the extracted solution (B) into the round flask-flat bottom again as done previously. Evaporate the whole extracted solution (mixture of solution A and B) on the rotary evaporator in vacuum less than 40℃.

Determination of acid value

Accurately weigh 10g of extracted oil, conduct determination of acid value according to AOCS Cd 3d-63 (Acid value of Fats and Oils).

Results

The results presented in the Table 1 perform the method precision only, while the accuracy was not included as the CRM of Seasoned Laver Sample has not been provided yet. The results of Acid value analysis are shown in Table 1. The data processing was performed based on ISO 5725 part2. According to Principles for the Establishment of CODEX Methods of Analysis in CODEX PROCEDURAL MANUAL, which defines the method performance criteria, the HORRAT value should be equal or less than 2 to confirm precision of the method conducted by collaborative method performance studies. HORRAT value is the ratio of the reproducibility relative standard deviation (RSD_R) to the predicted reproducibility relative standard deviation ($PRSD_R$).

$$\text{HORRAT value} = \frac{RSD_R,\%}{PRSD_R,\%}$$

This inter-laboratory validation test shows HORRAT value of 1.32. It means that the method of extraction of oil described in the Regional standard for Laver products and the method of determination of acid value (AOCS Cd 3d-63) are applicable for analysis of Acid value of Laver products.

Table 1. Results of Acid value analysis

Laboratory	Samples	Results			
		Acid Value	Mean(%)	SD(%)	RSD(%)
1	1	0.18	0.18	0.004	2.289
	2	0.18			
	3	0.18			
	4	0.17			
	5	0.18			
	6	0.18			
2	1	0.16	0.18	0.013	7.027
	2	0.18			
	3	0.20			
	4	0.18			
	5	0.18			
	6	0.18			
3	1	0.18	0.19	0.016	8.507
	2	0.20			
	3	0.20			
	4	0.20			
	5	0.19			
	6	0.16			
4	1	0.17	0.17	0.004	2.425
	2	0.17			
	3	0.17			
	4	0.17			
	5	0.17			
	6	0.16			

Grand Mean	0.178
S_r	0.009
RSD_r	5.152
S_R	0.012
RSD_R	6.864
$PRSD_R$	5.183
HORRAT	1.32

Mean	Average value of each Laboratory
SD	Standard 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
HORRAT	Ratio of the reproducibility relative standard deviation to the predicted reproducibility relative standard deviation

Recommendations

The Republic of Korea would like to confirm that the provision “acid value” applied to the extracted oil.

This collaborative method performance studies by 4 participating laboratories show the HORRAT value of 1.32, which meets the method performance criteria required by CODEX according to Principles for the Establishment of Codex Methods of Analysis in CODEX PROCEDURAL MANUAL.

Accordingly, the Republic of Korea would like to propose CCASIA to submit this validation test report to CCMAS to endorse Method of Analysis for Acid value described in the Regional standard for Laver products as Type I (or Type II).

Additional Proposals

Korea Food Research Institute (KFRI) coordinated the inter-laboratory test by the same 4 participating laboratories for endorsement of the methods for moisture content as Type I (or Type II).

Each participating laboratories were required to handle test samples under the following conditions.

1. Store all test samples at room temperature (21 ~ 25℃)

2. Discard remaining test samples. Do not reuse any samples after analysis.

3. Always keep the laboratory at room temperature with relative humidity between 25% and 30% to prevent condensation on the wall.

4. To prevent moisture change, each laboratory was required to conduct determination of moisture content soon after grinding.

Three types of Laver products (e.g. Dried laver, Roasted laver and Seasoned laver) were used for the test samples in this validation test. The same types of laver were produced by the same manufacturer on the same date each. Furthermore, after vacuum packing process under the same condition on the same date, each sample was distributed and tested 6 replicates.

Preparation of test sample

Test sample was prepared as follows.

Remove packaging materials from the test sample. Grind the sample with a grinder. To prevent moisture change, each laboratory was required to conduct determination of moisture content soon after grinding.

Determination of Moisture content

With 5g of homogenized test sample, the test method for determination of moisture content was conducted according to *Part B. Drying at Atmospheric Pressure*, AOAC 925.45.

The results of moisture content analysis are shown in APPENDIX II. The data processing was performed based on ISO 5725 part2. HORRAT value of Dried laver, Roasted laver and Seasoned laver were 1.10, 1.62 and 1.71 respectively. It means that each HORRAT value meets the method performance criteria required by CODEX according to Principles for the Establishment of Codex Methods of Analysis in CODEX PROCEDURAL MANUAL.

Accordingly, the Republic of Korea would like to propose CCASIA to submit the validation test report to CCMAS to endorse Method of Analysis for moisture content described in the Regional standard for Laver products as Type I (or Type II).

Furthermore, the Republic of Korea proposes the editorial amendments in the provision of Method of Analysis for clarity. (See APPENDIX III)

APPENDIX I

List of Participating Laboratories in Alphabetical Order

1. Japan Food Research Laboratory
2. Korea Food Research Institute
3. Korea Health Supplements Institute
4. Suwon Women’s University Food Analysis Research Center

APPENDIX II

Results of Moisture content analysis of three types of laver

Table 1 : Dried laver

Table 2 : Roasted laver

Table 3 : Seasoned laver

The name of participating laboratories is shown in APPENIDX I.

Table 1. Results of moisture content analysis of Dried laver

Laboratory	Samples	Results			
		Moisture content	Mean(%)	SD(%)	RSD(%)
1	1	3.50	3.33	0.186	5.586
	2	3.50			
	3	3.40			
	4	3.30			
	5	3.00			
	6	3.30			
2	1	3.40	3.42	0.098	2.878
	2	3.50			
	3	3.30			
	4	3.30			
	5	3.50			
	6	3.50			
3	1	3.54	3.44	0.100	2.921
	2	3.52			
	3	3.44			
	4	3.48			
	5	3.29			
	6	3.34			
4	1	3.42	3.37	0.096	2.841
	2	3.51			
	3	3.41			
	4	3.36			
	5	3.26			
	6	3.27			
Grand Mean		3.389			
S_r		0.120			
RSD_r		3.546			
S_R		0.124			

RSD_R	3.669
$PRSD_R$	3.328
HORRAT	1.10

Table 2. Results of moisture content analysis of Roasted laver

Laboratory	Samples	Results			
		Moisture content	Mean(%)	SD(%)	RSD(%)
1	1	1.50	1.50	0.063	4.216
	2	1.40			
	3	1.50			
	4	1.50			
	5	1.50			
	6	1.60			
2	1	1.50	1.57	0.052	3.296
	2	1.60			
	3	1.60			
	4	1.60			
	5	1.60			
	6	1.50			
3	1	1.61	1.53	0.045	2.967
	2	1.48			
	3	1.54			
	4	1.51			
	5	1.50			
	6	1.53			
4	1	1.45	1.41	0.113	8.024
	2	1.51			
	3	1.47			
	4	1.41			
	5	1.40			
	6	1.19			
Grand Mean		1.500			
S_r		0.068			
RSD_r		4.550			
S_R		0.092			
RSD_R		6.104			
$PRSD_R$		3.763			
HORRAT		1.62			

Table 3. Results of moisture content analysis of Seasoned laver

Laboratory	Samples	Results			
		Moisture content	Mean(%)	SD(%)	RSD(%)
1	1	1.10	1.10	0.000	0.000
	2	1.10			
	3	1.10			
	4	1.10			
	5	1.10			
	6	1.10			
2	1	1.10	1.20	0.110	9.129
	2	1.20			
	3	1.20			
	4	1.20			
	5	1.40			
	6	1.10			
3	1	1.11	1.19	0.066	5.567
	2	1.19			
	3	1.16			
	4	1.22			
	5	1.15			
	6	1.30			
4	1	1.20	1.19	0.063	5.321
	2	1.26			
	3	1.12			
	4	1.13			
	5	1.26			
	6	1.15			
Grand Mean			1.169		
S_r			0.060		
RSD_r			5.109		
S_R			0.078		
RSD_R			6.682		
$PRSD_R$			3.906		
HORRAT			1.71		

APPENDIX III

The provisions of Method of Analysis in the Regional standard for Laver products

10.3 Method of Analysis

10.3.1 Determination of Moisture

10.3.1.1 Preparation of Test Sample

Remove packaging materials from the test sample. Weigh 20g of test sample and grind the sample with a grinder. To prevent moisture change, conduct determination of moisture content soon after grinding.

10.3.1.2 Determination

Accurately weigh 5g of homogenized test sample, conduct determination of moisture content according to *Part B. Drying at Atmospheric Pressure*, AOAC 925.45.

10.3.2 Extraction of oil from Seasoned Laver

Weigh 50 g of test sample into 1000 mL Erlenmeyer flask. Add 500 mL of petroleum ether to the flask followed by replacing air in the flask by N_2 gas. Put a stopper on the flask and let stand for 2 hours. Decant the extracted solution (A) through a filter paper, on which Na_2SO_4 is mounted to remove moisture, on a funnel into 1000 mL round flask-flat bottom. Add additional 250 mL of petroleum ether to residue in the Erlenmeyer flask and decant the extracted solution (B) into the round flask-flat bottom again as done previously. Evaporate the whole extracted solution (mixture of solution A and B) on the rotary evaporator in vacuum less than 40℃.

10.3.3 Determination of acid value

Accurately weigh 10g of extracted oil, conduct determination of acid value according to AOCS Cd 3d-63.