

## THE PROPOSED DRAFT STANDARD FOR GOCHUJANG

### 1. SCOPE

This standard applies to the product defined in Section 2 below and offered for direct consumption including for catering purposes or for repacking if required. It does not apply to the product when indicated as being intended for further processing. This standard does not apply to chili paste or chili sauce products having red pepper as the main ingredient.

### 2. DESCRIPTION

#### 2.1 PRODUCT DEFINITION

*Gochujang* is a red or dark red pasty fermented food manufactured through the following process:

- (a) Saccharified material is manufactured by saccharifying grain starch with powdered malt, or by naturally occurring microorganisms (which are not pathogenic and do not produce toxin) during fermentation;
- (b) Salt is mixed with the saccharified material obtained in the above (a). Subsequently, the mixture must be fermented and aged;
- (c) Red pepper powder is mixed and other ingredients may be mixed with the mixture before or after the fermentation process (b) above; and
- (d) Processed by heat or other appropriate means, before or after being hermetically sealed in a container, so as to prevent spoilage.

### 3. ESSENTIAL COMPOSITION AND QUALITY FACTORS

#### 3.1 COMPOSITION

##### 3.1.1 Basic Ingredients

- (a) Grains
- (b) Red pepper (*Capsicum annuum* L.) powder
- (c) Salt
- (d) Potable water

##### 3.1.2 Optional Ingredients

- (a) Powdered *meju*\*

\* Fermented material of soybeans or the mixture of soybeans and grains using microorganisms (bacteria, molds and yeasts) in a state of nature

- (b) Soybeans
- (c) Sugars
- (d) Distilled alcohol derived from agricultural products
- (e) Soy sauce
- (f) Fermented soybean paste
- (g) Fish sauce
- (h) Sea food extract
- (i) Fermented wheat protein
- (j) Fermented rice
- (k) Yeast extract
- (l) Hydrolyzed vegetable protein
- (m) Seasoned vegetables
- (n) Vinegar
- (o) Other ingredients

## 3.2 QUALITY FACTORS

### 3.1.3 QUALITY FACTORS

- (a) Capsaicin not less than 10.0 µg/mL (w/w)
- (b) Crude protein not less than 3.0% (w/w)
- (c) Moisture not more than 60.0% (w/w)

3.1.4 *Gochujang* shall have its unique flavour, odour, and the following qualities.

- (a) Colour: The product shall have a red or dark red colour derived from red pepper (*Capsicum annuum* L.).
- (b) Taste: The product shall have a hot and savoury taste. It may also have a somewhat sweet taste and a somewhat salty taste.
- (c) Texture: The product shall have an appropriate level of viscosity.

### 3.3 CLASSIFICATION OF “DEFECTIVES”

Any container that fails to meet the applicable quality requirements, as set out in Sections 3.2, should be considered a “defective”.

### 3.4 LOT ACCEPTANCE

A lot should be considered as meeting the applicable quality requirements referred to in Section 3.2, when the number of “defectives”, as defined in Section 3.3, does not exceed the acceptance number (c) of the appropriate sampling plans.

## 4. FOOD ADDITIVES

The food additives listed below can be used within the scope of a permitted amount.

### 4.1 PRESERVATIVES

INS No.	Name of food additives	Maximum level
200	Sorbic acid	1000mg/kg as sorbic acid, singly or in combination
202	Potassium sorbate	
203	Calcium sorbate	

### 4.2 FLAVOUR ENHANCERS

INS No.	Name of food additives	Maximum level
508	Potassium chloride	limited by GMP
621	Monosodium L-glutamate	limited by GMP
627	Disodium 5'-guanylate	limited by GMP
631	Disodium 5'-inosinate	limited by GMP

### 4.3 ANTIOXIDANT

INS No.	Name of food additives	Maximum level
325	Sodium lactate	limited by GMP

### 4.4 ACIDITY REGULATORS

INS No.	Name of food additives	Maximum level
296	Malic acid (DL-)	limited by GMP
339(i)	Sodium dihydrogen phosphate	5000 mg/kg as phosphorus, singly or in combination
339(ii)	Disodium hydrogen phosphate	
340(i)	Potassium dihydrogen phosphate	
340(ii)	Dipotassium hydrogen phosphate	
452(i)	Sodium polyphosphate	
452(ii)	Potassium polyphosphate	

### 4.5 STABILIZERS

INS No.	Name of food additives	Maximum level
412	Guar gum	limited by GMP
414	Gum arabic (acacia gum)	limited by GMP

415	Xanthan gum	limited by GMP
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## 5. CONTAMINANTS

The products covered by this Standard shall comply with the maximum levels of the *Codex General Standard for Contaminants and Toxins in Foods* (CXS 193-1995).

The products covered by this Standard shall comply with the maximum residue limits for pesticides established by the Codex Alimentarius Commission.

## 6. HYGIENE

**6.1** It is recommended that the products covered by the provisions of this Standard be prepared and handled in accordance with the appropriate sections of the *Recommended International Code of Practice - General Principles of Food Hygiene* (CXC 1-1969) and other relevant Codex texts such as Codes of Hygienic Practice and Codes of Practice.

**6.2** The products should comply with any microbiological criteria established in accordance with the *Principles for the Establishment and Application of Microbiological Criteria for Foods* (CXG 21-1997).

## 7. WEIGHTS AND MEASURES

### 7.1 MINIMUM WEIGHT

As for a product whose indicated weight is not more than 1,000g, the tolerance allowed shall be less than 15g. As for a product whose indicated weight is 1,000-5,000g, the net weight of the product shall not be less than 98.5% of the indicated weight. As for a product whose indicated weight is more than 5,000g, the net weight of the product shall not be less than 99% of the indicated weight.

### 7.2 CLASSIFICATION OF "DEFECTIVES"

A container that fails to meet the requirement for minimum weight of Section 7.1 shall be considered a "defective".

### 7.3 LOT ACCEPTANCE

A lot should be considered as meeting the requirements of Section 7.1, when the number of "defectives", as defined in Section 7.2, does not exceed the acceptance number (c) of the appropriate sampling plan.

## 8. LABELLING

In addition to the provisions of the *Codex General Standard for the Labelling of Prepackaged Foods* (CXS 1-1985), the following specific provisions apply.

### 8.1 PRODUCT NAME

**8.1.1** The name of product shall be "Gochujang".

**8.1.2** The name of product can be labelled in accordance with domestic laws, so that its characteristics may be expressed

### 8.2 LABELLING OF NON-RETAIL CONTAINERS

Information for non-retail containers shall be given on the container or in accompanying documents, except that the name of the product, lot identification and the name and address of the manufacturer, packer or distributor, as well as storage instructions, shall appear on the container. However, lot identification, and the name and address of the manufacturer, packer or distributor may be replaced by an identification mark, provided that such mark is clearly identifiable with the accompanying documents.

## 9. METHODS OF ANALYSIS AND SAMPLING<sup>1</sup>

For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.<sup>2</sup>

### 9.1 SAMPLING

<sup>1</sup> The listing of methods of analysis and sampling will be removed when the standard is adopted by CAC and included in CXS 234-1999.

<sup>2</sup> The Codex Secretariat proposed to insert the texts and footnote 2 according to the Procedural Manual.

Sampling shall be conducted as follows:

- (a) Samples shall be stored in such a way as materials may not be heated up;
- (b) Great care shall be taken so that samples, sampling equipment, and sampling containers may be protected from outside pollution;
- (c) Samples shall be kept in a clean and dry container with its lid. The container shall carry detailed descriptions about sampling such as sampling date, seller's name, and other particulars of consignment sale.

## **9.2 METHODS OF ANALYSIS**

### **9.2.1 Determination of Capsaicin**

According to AOAC 995.03 or the method described in Annex.

### **9.2.2 Determination of Crude protein**

According to AOAC 984.13 (Nitrogen conversion factor: 6.25).

### **9.2.3 Determination of Moisture**

According to AOAC 934.01.

## Determination of Capsaicin in *Gochujang* using Gas Chromatography (GC) Detection

### 1. SCOPE

This method is suitable for the determination of capsaicin in *Gochujang* using gas- chromatographic detection. The method uses squalene as an internal standard. The concentration of capsaicin is expressed as µg/mL.

### 2. PRINCIPLE

To extract capsaicin, the mixture is blended to a homogeneous consistency. Capsaicin in *Gochujang* is extracted with 100% methanol, followed by methanol – hexane fractionation to remove hydrophilic and hydrophobic interfering substances by a separating funnel. Capsaicin in methanol layer is extracted with dichloromethane (DCM) and the saturated NaCl, concentrated by a rotary evaporator. A portion of the concentrated sample extract is then taken and completely solved with DCM containing squalene as an internal standard for analysis using gas chromatographic detection.

### 3. REAGENT AND MATERIALS

During the analysis, unless otherwise stated, use only reagent of recognized analytical grade and water of at least grade 3 as defined in ISO 3696.

#### 3.1 REAGENTS

- 3.1.1 Capsaicin (99 + %, C<sub>18</sub>H<sub>27</sub>NO<sub>3</sub>, Fw 305.42, CAS 404-86-4)
- 3.1.2 Squalene (CAS 111-02-4)
- 3.1.3 Hexane
- 3.1.4 Methanol
- 3.1.5 Methanol + Water (80 + 20)
- 3.1.6 Dichloromethane
- 3.1.7 Sodium chloride
- 3.1.8 Sodium sulfate

#### 3.2 PREPARATION OF STANDARD SOLUTION

##### 3.2.1 Capsaicin Stock solution (A)

Weigh approximately 100 mg of capsaicin, making up to 100 ml in a volumetric flask with DCM to give solution (A) of approximate 1000 µg/mL.

##### 3.2.2 Capsaicin working solution (B)

Prepare 100 mL intermediate solution B by dilution of 10 ml solution A (3.2.1) with 100 mL of DCM to exactly 100 µg/mL in DCM.

##### 3.2.3 Squalene internal standard working solution (C)

Weigh approximately 100 mg squalene and make up to 250 mL in a volumetric flask with DCM to give a solution (C) of approximately 400 µg/mL in DCM.

#### 3.3 CALIBRATION SOLUTIONS OF CAPSAICIN

Dispense volumes of the 100 µg/mL solution (B, 3.2.2) into 50 mL round flask, dried up and add 2 mL of internal standard working solution (C, 3.2.3) to give 10.0, 50.0, 100.0, 300.0, 500.0 µg/mL capsaicin.

### 4. APPARATUS

4.1 Gas chromatograph with flame ionization detector (FID) The following conditions have been found to be suitable:

4.1.1 Injector / Detector temperature : 320°C / 350°C

4.1.2 Oven temperature program: 220°C for 1 minute, ramp at 5°C/min to 250°C, hold for 13 minutes and raise to 280°C holding 5 min by 20°C/min. Helium carrier gas at 1.5 mL/minute

4.1.3 Make split injection of 1.0µL with split ratio 1:5

4.1.4 GC column, 30 m x 0.32 µm, 0.25 µm film thickness, HP-1 or equivalent

4.2 Analytical balance, capable of weighing to 4 decimal places

- 4.3 Shaker, capable of attaining 2,000 rpm
- 4.4 Centrifuge, capable of attaining 3,500 rpm
- 4.5 Filter paper (Waterman No. 2 or equivalent)

## 5. LABORATORY SAMPLES

On receipt, samples are given a unique sample number. *Gochujang* sample is stored at below 4°C. All other samples are stored at room temperature in an air tight container prior to analysis.

## 6. PROCEDURE

### 6.1 LABORATORY SAMPLE

Samples should be minced or grated to a homogeneous mixture. All samples should be stored in the air-tight container and at room temperature prior to analysis. All samples should be mixed thoroughly to a homogeneous mixture before analysis.

### 6.2 TEST SAMPLE

- 6.2.1 Thoroughly mix the sample. Weigh, to the nearest 0.01 g, and 10 g portion of *Gochujang* into a 250 mL centrifuge bottle.
- 6.2.2 Add 50 mL of methanol and shaking for 2 hours, extracting capsaicin.
- 6.2.3 Filter the extract with Watman No. 2 filter paper into a 250 mL flat bottom flask (Ext-A).
- 6.2.4 Add additional 30 ml of methanol to residue and shaking for 1 hour, extracting capsaicin (Ext-B).
- 6.2.5 Repeat step 6.2.3 to 6.2.4 (Ext-C)
- 6.2.6 Combine Ext-A, Ext-B and Ext-C in 250 mL flat bottom flask, concentrating up to approximately 5 ml.
- 6.2.7 Solve the concentrate with 20 mL of 80% methanol and 20 mL of hexane.
- 6.2.8 Transfer the solution into a 250 mL separating funnel.
- 6.2.9 Shake and separate into two layers, methanol layer (M1-layer, upper) and hexane layer (H1-layer, lower)
- 6.2.10 Reserve H1-layer in 100 mL flask and transfer M1-layer (6.2.9) into a separating funnel and add additional 20 mL of hexane.
- 6.2.11 Repeat step 6.2.9 to 6.2.10 (M2-layer and H2-layer)
- 6.2.12 Repeat step 6.2.9 to 6.2.10 (M3-layer and H3-layer)
- 6.2.13 Combine H1-layer, H2-layer and H3-layer (HC-layer) in the 250 mL separating funnel, adding 20 ml 80% methanol, shaking and separating into two layers, methanol layer (M'1-lower layer) and hexane layer (H'1-upper layer).
- 6.2.14 Reserve M'1-layer in the new 250 mL flat bottom flask.
- 6.2.15 Add 20 mL of 80% methanol into the separating funnel containing HC-layer, shaking and separating into two layers (M'2-layer and H'2-layer)
- 6.2.16 Combine the all M-layer in the new separating funnel (250 mL), adding 20 mL of saturated NaCl and 20 mL of DCM.
- 6.2.17 Shake and separate into two layers (D1-layer and WM1-layer) in the 250 mL separating funnel.
- 6.2.18 Transfer D1-layer into the new 250 mL flat bottom flask.
- 6.2.19 Add additional 20 mL DCM into the separating funnel (6.2.16), shaking and separating into two layers (D2-layer and WM1-layer)
- 6.2.20 Repeat step 6.2.16 (D3-layer and WM1-layer)
- 6.2.21 Combine D1-layer, D2-layer and D3-layer into the 250 mL flat bottom flask, concentrating it (C-D)
- 6.2.22 Transfer the concentrate (C-D, 6.2.21) into a 100 ml round flask, solving it completely with DCM.
- 6.2.23 Mount approximate 3 g of sodium sulfate on the filter paper and dehydrate C-D by passing through sodium sulfate
- 6.2.24 Collect the dehydrated C-D layer in 50 mL round flask and concentrate to dryness by the rotary evaporator
- 6.2.25 Solve the concentrate with 2 mL of DCM containing squalene as the internal standard solution (C, 3.2.3)
- 6.2.26 Analyze the sample solution by GC

## **7. CALCULATION – INTERNAL STANDARD METHOD**

- 7.1 Measure the area of the capsaicin and squalene peaks.
- 7.2 Calculate the ratio of the capsaicin and squalene peak areas.
- 7.3 Construct a calibration graph for the standards by plotting the peak area ratio against the weight in microgram of capsaicin in the vial.
- 7.4 Calculate the slope of the calibration line.
- 7.5 Divide the peak area ratio of the unknowns by the value of the slope to give the weight of capsaicin per vial for the unknown samples.

## **8. FINAL PRESENTATION OF RESULTS**

Results are expressed as ppm and quoted to 2 significant digits.

## **REFERENCES**

1. W. Hawer and J. Ha et al. : Effective separation and quantitative analysis of major heat principles in red pepper by capillary GC, Food Chemistry, 49, pp.99-103, 1994.
2. J. Jung and S. Kang : A new method for analysis of capsaicinoids content in microcapsule, Korean J. Food Sci. Technol., Vol.32, No. 1, pp.42-49, 2000.
3. C.A. Reilly et al.: Quantitative analysis of capsaicinoids in fresh peppers, oleoresin capsicum and pepper spray products, J. of Forensic Science, Vol.43, No. 3, pp.502-509, 2001.
4. Ha et al.: Gas Chromatography Analysis of Capsaicin in Gochujang, Journal of AOAC International Vol. 91. No. 2, 2008.

Table 1. Summary of repeatability test for trial proper samples ( $\mu\text{g/mL}$ )

Test No.	<i>Gochujang - K</i>
1	64.7
2	69.0
3	70.6
4	71.8
5	70.5
Mean	69.3
RSD,%	3.99

Table 2. Summary of recovery test for trial proper samples (%)

Test No.	<i>Gochujang - K</i>
1	80.47
2	77.29
3	87.97
4	91.00
5	95.18
Mean	86.38
RSD,%	8.56



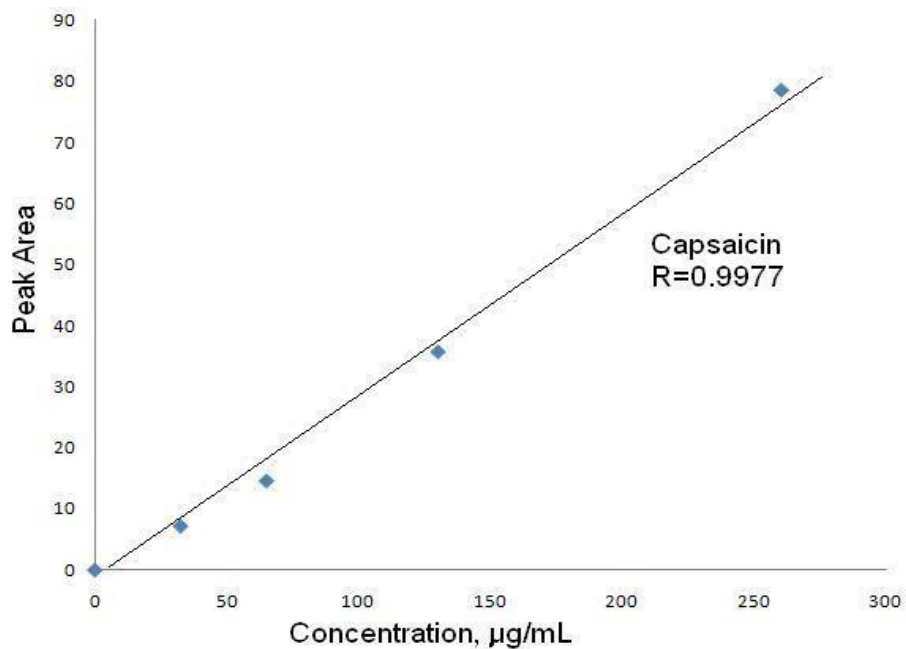


Fig.1. Calibration curve of capsaicin by GC method.

Fig. 2. GC chromatogram of capsaicin standards.

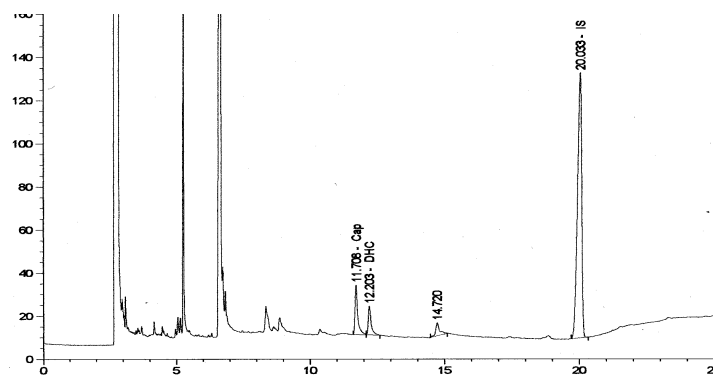
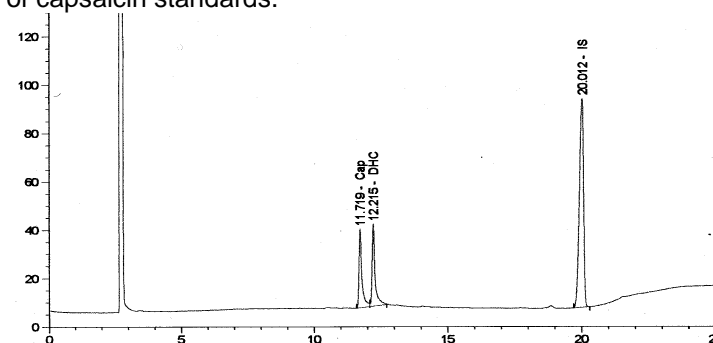


Fig. 3. GC chromatogram of capsaicin in *Gochujang*.