PHILIPPINE NATIONAL STANDARD

PNS/FDA 39:2015
ICS 67.020

Code of practice for the processing and handling of fish sauce (patis) and fish flavored sauce

BUREAU OF PRODUCT STANDARDS*

Foreword

This Philippine National Standard for Fish Sauce (patis) and Fish-flavored Sauce was developed under the project of the Food Processing Division, Industrial Technology Development Institute (ITDI) of the Department of Science and Technology (DOST).

The Standard for Fish Sauce (Patis) and Fish-Flavored Sauce and its Code of Practice were reviewed, finalized and endorsed by the Food and Drug Administration (FDA) of the Department of Health (DOH) and approved for adoption as the Philippine National Standard by the Bureau of Philippine Standards.

Public consultation workshops were held in the regions where the product is being manufactured abundantly. Stakeholders from different agencies and offices contributed their expertise for the finalization of the draft.

This Standard was developed in response to the need for high standards of the products, guidance for assurance of its quality and safety, harmonization with export requirements, thus, having Philippine processed food products competitive in the world market.

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Code of practice for the processing and handling of fish sauce (patis) and fish flavored sauce

1 Scope

This Code of Practice is a set of recommended procedures that shall be adopted by processors of fish sauce (patis) and fish flavored sauce to conform to the Standards for Fish Sauce (Patis) and Fish flavored Sauce (PNS/FDA 38:2014).

This code provides a guide in the processing, storage and handling of fish sauce (patis) and fish flavored sauce necessary to maintain their safety and quality from the receipt of raw materials and ingredients up to distribution.

2 References

The titles of the standards and publications referred to in this Standard are listed in the inside back cover.

3 Definition

For the purpose of this standard, the following definitions apply:

3.1
chilled fish
fresh fish, which has been stored at a temperature not exceeding 4°C

3.2
chilling
the process of cooling fish thoroughly to a temperature approaching that of melting ice

3.3
cleaning
the removal of soil, food residues, dirt, grease or other objectionable matter

3.4
contaminant
any biological or chemical agent, foreign matter, or other substances not intentionally added to food which may compromise food safety or suitability

3.5
contamination
the introduction or occurrence of a contaminant in fish, shellfish and their products

3.6
container
any form of packaging material, which completely or partially encloses the food (including wrappers). A container may enclose the food as a single item or several units or types of prepackaged food when such is presented for sale to the consumer

3.7
current Good Manufacturing Practices (cGMP)
a quality assurance system aimed at ensuring that products are consistently manufactured, packed, repacked or held to a quality appropriate for the intended use. It is thus concerned with both manufacturing and quality control procedures

3.8
fermentation
the breakdown of organic substances into simpler components mainly by the action of enzymes produced by microorganisms

3.9
fish
any of the cold-blooded (ectothermic) aquatic vertebrates. Amphibians and aquatic reptiles are not included

3.10
fresh fish
fish or fishery products which have received no preserving treatment other than chilling

3.11
frozen fish
fish which have been subjected to a freezing process sufficient to reduce the temperature of the whole product to a level low enough to preserve the inherent quality of the fish and which have been maintained at this low temperature during transportation, storage and distribution up to and including the time of final sale (Codex, 1991; Codex 2003).

3.12
food
any processed substance which is intended for human consumption and includes drink for man, beverages, chewing gum and any substances, which have been used as an ingredient in the manufacture, preparation or treatment of food

3.13
food additive
any substance not normally consumed as a food by itself and not normally used as a typical ingredient of the food, whether or not it has nutritive value, the intentional addition of which to food for a technological (including organoleptic) purpose in the manufacture, processing, preparation, treatment, packing, packaging, transport or holding of such food results, or may be reasonably expected to result (directly or indirectly), in it or its by-products becoming a component of or otherwise affecting the characteristics of such foods. The term does not include contaminants or substances added to food for maintaining or improving nutritional qualities

3.14
food standard
a regulatory guideline that defines the identity of a given food product (i.e. its name and the ingredients used for its preparation) and specifies the minimum quality

factors and, when necessary, the required fill of container. It may also include specific labeling requirements other than or in addition to the labeling requirements generally applicable to all prepackaged foods

3.15
Hazard Analysis and Critical Control Point (HACCP)
a preventive food quality management system, which identifies, evaluates and controls the hazards significant to food safety specific to a product

3.16
histamine
a biogenic amine formed in fish muscle by decarboxylation of the amino acid histidine by bacteria

3.17
ingredient
any substance including a food additive, used as a component in the manufacture or preparation of a food and present in the final product in its original or modified form

3.18
label
a display of written, printed or graphic matter upon the immediate container of any article and a requirement made by or under authority of existing law that any word, statement, or other information appearing on the label shall not be considered to be complied with unless such word, statement or other information also appears on the outside container or wrapper of the retail package of such article or is easily legible through the outside container or wrapper

3.19
labeling
any written, printed or graphic matter (1) upon any article or any of its container or wrappers or (2) accompanying the packaged food

3.20
lot
quantity of food product produced under essentially the same conditions during a particular production schedule

3.21
packaging
is the process of packing that is part of the production cycle applied to a bulk product to obtain the finished product. Any material, including painted material, employed in the packaging of a product including any outer packaging used for transportation of shipment. Packaging materials are referred to as primary or secondary according to whether or not they are intended to be in direct contact with the product

3.22
pH
the measure of the intensity or degree of acidity of a food material

3.23
pasteurization
a form of heat treatment that kills vegetative pathogens and most but not all spoilage microorganisms in foods

3.24
prepackaged
packaged or made up in advance in a container, ready for sale to the consumer, or for catering purposes

3.25
processed food
the product, resulting from the application of physical, chemical or biological processes to a "primary food commodity" intended for direct sale to the consumer, for direct use as an ingredient in the manufacture of food or for further processing

3.26
proteins
nitrogenousorganic compounds consisting of linked amino acids that are distributed widely in plants and animals. The sequence of amino acids in proteins is determined by the base sequence of their encoding genes. They serve many roles, such as enzymes, structural elements and hormones, and are essential nutrients

3.27
salinometer
a device that measures the approximate salt concentration in the brine. In a saturated brine, the stem will be almost entirely above the level of the salt solution and read 100°S (salinity approximately 23% salt solution). In weaker brines more of the stem will be submerged

3.28
salt content
the amount of salt (as NaCl) in the product, usually expressed as percentage

4 Ingredients and Packaging Material Requirements

4.1 Basic Ingredients

4.1.1 Fish - shall be sound, wholesome and fit for human consumption. Species of fish used may include, but not limited to those listed in Annex 1. This also includes fish parts generated from other fish processing operations, which may include fish heads, flesh/meat, skins, tails, and internal organs.

4.1.2 Salt - shall be of food grade quality and meets the requirements and standards for iodized salt as per R.A. No. 8172: An Act Promoting Salt Iodization Nationwide and for Related Purposes (Annex 2), including Bureau Circular No. 2007-009 Updated Standards for Iodine Level of Salts and/or its future amendments.

4.1.3 Water (to prepare brine) - shall be water fit for human consumption and meets the potability requirements prescribed in the Philippine National Standards for

Drinking Water as per DOH Administrative Order No. 2007-0012 (Annex 3), and/or its future amendments.

4.2 Other ingredients

All other ingredients used for subsequent processes after fermentation shall be of food grade quality and conform to all applicable standards.

4.3 Food Additives

Food additives when used shall be in accordance with the current regulations prescribed by Food and Drug Administration and/or the latest Codex General Standard for Food Additives. The food additives listed but not limited to those in Table 1 may be used for the manufacture of fish sauce (*patis*) and fish flavored sauce.

Table1 - Food Additives for Fish Sauce (*Patis)* and Fish Flavored Sauce

Functional Class	Codex INS	Food Additive	Maximum Use Level
Colors	150c 150d	Caramel Color Class III and Class IV	50,000 mg/kg 30,000 mg/kg
Sweeteners	950	Acesulfame potassium	1,000mg/kg
	955	Sucralose	120 mg/kg
	951	Aspartame	350 mg/kg
Preservatives		Sodium benzoate	1000 mg/kg
		Potassium sorbate	1000 mg/kg
Preservatives/ Antioxidants	223	Sodium metabisulfite	300mg/kg* (residual)
Flavor Enhancer	621	Monosodium glutamate	300
	630	Inosinic acid	GMP
	631	Disodium Inosine 5' monophophate	GMP
	627	Disodium 5' guanylate	GMP
* Based on the Food Category System No. 12.6 – Sauces and like products			

All other food additives not included in the above list shall be allowed as carry-over, provided they are approved by the FDA regulation and shall be in accordance to Section 5.2 of the "Principle Relating to the Carry-Over of Food Additives into Foods" (CAC/Vol. 1, 1991), and/or its future amendments.

4.4 Packaging Materials

The packaging materials should be appropriate for fish sauce and fish flavored sauce and for the expected conditions of handling during distribution and storage. These should provide adequate protection from contamination for the products and should be sufficiently durable to withstand mechanical, chemical and thermal

stresses encountered during processing and normal distribution. All packaging materials must be clean and free from defects that may affect the product or package integrity. These shall be stored in a clean and sanitary manner.

5 Hygiene

It is recommended that the product covered by the provisions of this code of practice be prepared and handled in accordance with the appropriate sections of the Recommended International Code of Practice – General Principles of Food Hygiene (CAC/RCP 1–1969, Rev 4 (2003) and/or the FDA A.O. No. 153 s. 2004 - Guidelines, Current Good Manufacturing Practices in Manufacturing, Packing, Repacking or Holding Food, and/or their future amendments, covering the plant facilities and operations requirement including the construction and layout of processing plant, hygienic facilities, equipment, utensils and working surfaces.

6 Preparation and Processing

The production of fish sauce (*patis*) and fish flavored sauce is described from the receipt of raw materials up to product storage. The production process should be supervised by personnel with adequate technical training and experience.

6.1 Preparation of ingredients

The raw materials (fish), salt, water and other ingredients must be safe for human consumption and free from spoilage and contamination. Whenever applicable, certificates of analyses (COA) from raw materials and ingredient suppliers shall be secured to confirm their suitability for processing. No ingredients, which have indications of deterioration, decomposition or contamination to an extent which renders them unfit for human consumption, shall be used for processing. Stored stocks of ingredients should be used on a first in - first out (FIFO) or first to expire – first out (FEFO) basis. Ingredients that do not conform to the requirements of Sub-section 4.1 (Ingredient) shall be rejected.

6.1.1 Receipt of Basic Ingredients

6.1.1.1 Fish

Only chilled/iced fresh and/or frozen fresh fish shall be used for processing. The temperature of chilled/iced fish shall not exceed 4°C during holding while frozen fish should be stored at -18°C or lower.

6.1.1.2 Salt

Salt shall conform to Section 4.1.2 of this Code. It shall be pre-packaged, iodized, refined or unrefined in the form of coarse or fine white crystals without any dirt, sand or other foreign matters.

6.1.1.3 Water (to prepare brine)

Water shall conform to Section 4.1.3 of this Code. It shall be obtained only from reliable sources, with adequate supply at all times. It should be clear and, free from any objectionable color, odor or taste.

6.1.2 Receipt of Other Ingredients

All other ingredients shall be inspected before use. They must be packaged properly and free from any signs of quality deterioration, decomposition or contamination.

6.2 Processing Operations

The manufacture of fish sauce and fish flavored sauce shall use standardized formulation and process required to achieve the safety and quality criteria as prescribed in Section 5.2 of the Standards for Fish Sauce (Patis) and Fish Flavored Sauce (PNS/FDA 38:2014).Any modifications introduced must be tested and validated prior to adoption in commercial processing.

Figure 1 shows the general process in the processing of fish sauce (patis) and fish flavored sauce. Processing operations in the production of fish sauce and fish flavored sauce are divided into three (3) stages, namely: a) the fermentation and collection of first extract fish sauce; b) fermentation of fish residues and collection of succeeding fish sauce extracts; and, c) formulating and/or packaging of fish sauce and fish flavored sauce.

6.2.1 Fermentation and Collection of First Extract Fish Sauce

The raw materials should be immediately handled and salted to minimize possible microbial growth.

6.2.1.1 Preparation of fish for salting and fermentation

6.2.1.1.1. Chilled/iced fish should be drained and transferred into appropriate containers.

6.2.1.1.2. Frozen fish should be completely thawed at room temperature. The thawed fish are drained and transferred in appropriate containers.

For both chilled/iced fish and frozen fish, the temperature of the fish prior to use should not exceed 4°C.

6.2.1.2 Sorting and cleaning

The fish is transferred to appropriate working tables and extraneous or objectionable matters are removed. Fish that show any signs of deterioration should be rejected.

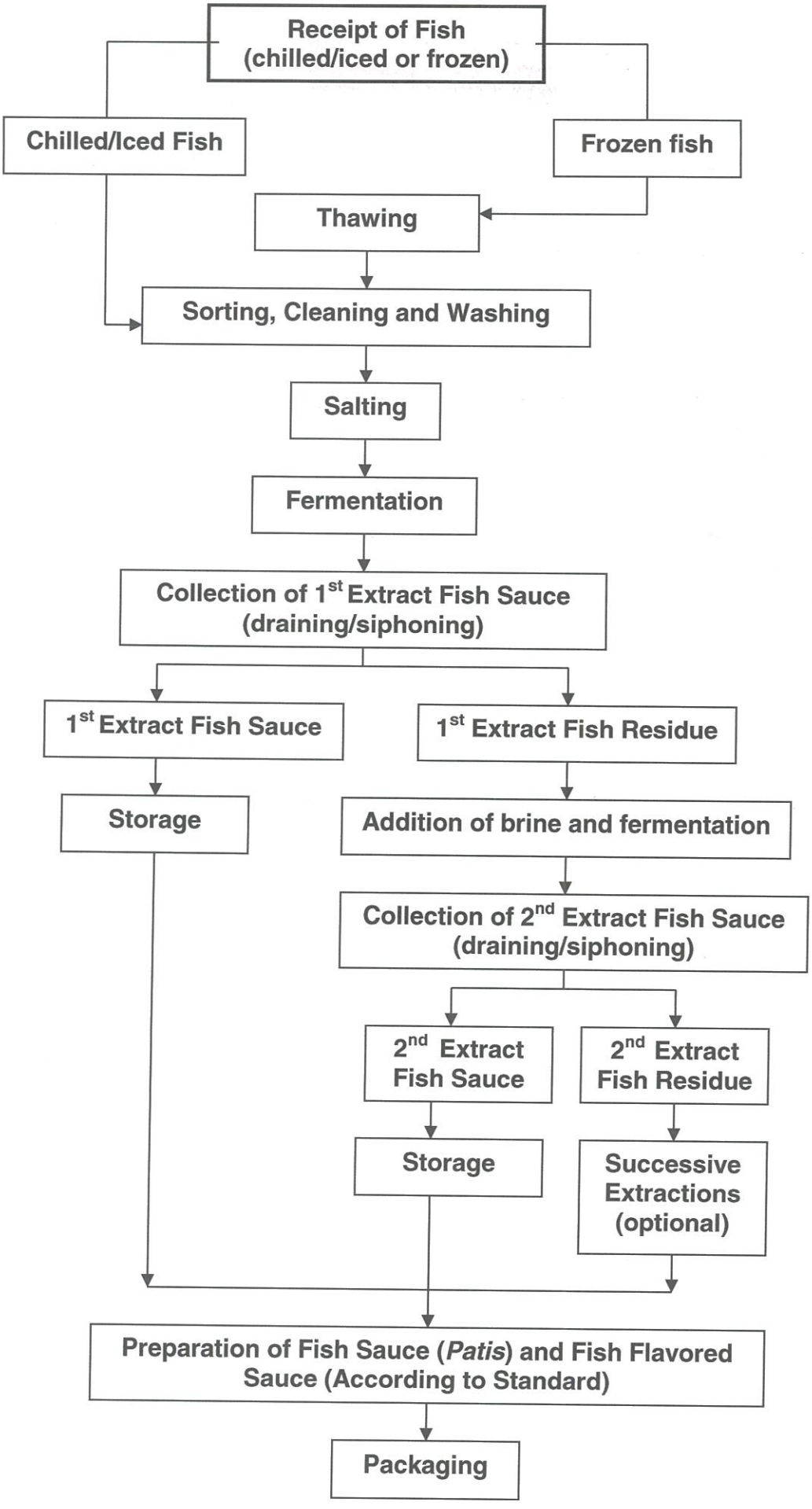

Figure 1. Flow Diagram for the Processing of Fish Sauce (*Patis*) and Fish Flavored

6.2.1.3 Washing

The fish are washed thoroughly in water to completely remove adhering dirt and maybe rinsed with brine containing 3-4% salt, then drained. The brine is prepared by dissolving a corresponding amount of salt in a given amount of water. Brine strength should be checked with a salinometer.

6.2.1.4 Salting

Appropriate amount of food grade salt is added and mixed with the fish. The recommended salt to fish ratio is 1 part salt to 3 parts fish by weight. The mixing process may be done manually or using a mechanical mixer.

The salted fish is transferred into appropriate fermenting vats or container, like stainless steel tanks, concrete tanks, plastic drums or earthen jars leaving an adequate headspace. A layer of salt may be added and spread over the salted fish. The fermenting tank is properly covered to prevent the entry of insects, rodents and other foreign matters.

6.2.1.5 Fermentation

The salted fish is allowed to ferment for at least 6 months at room temperature. During fermentation, occasional stirring or mixing is recommended.

During the fermentation process the fish will undergo hydrolysis resulting in the accumulation of a clear, brown liquid hydrolyzate which has a distinctive odor and flavor, which develops progressively as the fermentation progresses.

6.2.1.6 Collection of first extract

When the fermentation process is completed, the supernatant liquid that has accumulated on top of the fermentation tank is collected or extracted by draining the extract thru a drain valve located at the bottom of the tank. This may also be done by siphoning the fish extract using appropriate plastic or stainless steel tubing.

The resulting extract is filtered by passing through cartridge filter or filter bags to remove suspended solids.

6.2.1.7 Storage of first extract

The extract is transferred into appropriate storage containers pending packaging and/or further processing. The headspace should be controlled to a minimum to prevent oxidation which could result in the discoloration of the extract.

6.2.2 Fermentation of Fish Residue and Collection of Succeeding Fish Extracts

To the fish residue resulting from the collection of first extract, brine is added and allowed to ferment to extract the remaining protein and fish flavor.

6.2.2.1 Addition of saturated brine to the fish residue

Brine containing at least 25% salt is added and thoroughly mixed with the fish residue. The volume of brine added is approximately equivalent to the volume of fish sauce extract collected.

The brine is prepared by dissolving a corresponding amount of salt in a given amount of water. Brine strength should be checked with a salinometer and its strength maintained at the required concentration either through the appropriate addition of more salt or by dilution with water.

Annex 4 presents a matrix on the preparation of brine of required strength.

6.2.2.2 Fermentation of fish residue

The brined residue is properly covered and allowed to ferment at room temperature until the acceptable flavor and odor are attained.

6.2.2.3 Collection of second extract

The collection of the second extract is done similar to the extraction of the first extract. The extract is also transferred to appropriate storage containers for packaging and/or further processing.

6.2.2.4 Successive fermentations and collection of fish extracts

Further extractions may be done by following the same procedure in 6.2.2.1.

6.2.3 Preparation and Packing of Fish Sauce (Patis) and Fish Flavored Sauce Products

The fish extracts resulting from the foregoing processing steps are used in the preparation of fish sauce (patis) and fish flavored sauce for commercial distribution.

6.2.3.1 Preparation of fish sauce (patis) and fish flavored sauce products

Fish sauce (patis) and fish flavored sauce are prepared from the resulting fish extracts. Brine solution of at least 25% salt, food ingredients and food additives may be added.

Brine solution is prepared by following the procedure in 5.2.2.1. The brine may be pasteurized below 100°C then filtered and clarified before use.

The prepared fish sauce (*patis*) and fish flavored sauce products should comply with the requirements of the PNS Standard for Fish Sauce (*Patis*) and Fish Flavored Sauce (PNS/FDA 38:2014) as shown on Table 2.

6.2.3.2 Filtration of prepared fish sauce (*patis*) and fish flavored sauce

The products may be filtered using appropriate filtering devices like cartridge filters or filter bags of at least 5 microns mesh size and stored in appropriate containers.

Table 2 - Physico-chemical requirements for fish sauce (*patis*) and fish flavored sauce

Parameter	Fish Sauce		Fish Flavored Sauce
	Special/ Premium	Regular	
Protein, % (min)*	6.0	4.0	1.0
Salt, as NaCl, % (min)	24	24	24
pH	5.0– 6.5	5.0 – 6.5	5.0 – 6.5
Histamine, ppm (max)	400	400	400
* % Protein = Total Nitrogen (g/liter) x PF/actual specific gravity of fish sauce x 10 PF – Protein Factor – 6.25			

6.2.3.3 Pasteurization

The products may be pasteurized below 100°C.

6.2.3.4 Packing in retail and bulk containers

The products are filled into retail containers, like glass and plastic bottles, and flexible packages by manual and/or mechanical filling. No product should adhere on the neck or finish and sealing surface of the bottles and flexible packages.

The products may also be filled in bulk containers.

For both retail and bulk containers, an appropriate headspace should be observed to prevent oxidation of the contents.

6.3 Coding of Packed Products

Coding of packed products in sealed containers shall be made with indelible markers with information details of production, date, batch code, product code, the product line in which the product was packed and other information necessary for product traceability. Whenever the container does not permit the code to be embossed or inked, the label shall be legibly perforated or otherwise marked, and securely affixed to the product package.

6.4 Post-Process Container Handling

Care must be exercised in the handling and distribution of retail or bulk packaged products or as to prevent mechanical damage and spoilage to the products.

7 Labeling

7.1 General Requirements

All fish sauce (patis) and fish flavored sauce products shall be inspected before labeling and casing. All containers of packaged products shall be properly labeled. The label shall conform to current FDA labeling requirements and shall contain the following information:

7.1.1 The name of the product shall be "Fish Sauce (Patis)" or "Fish Flavored Sauce (Patis Flavor)". The products may be called by other common names provided that such names are accepted in the country of distribution.

7.1.2 The name and the address of the manufacturer, packer, distributor, importer, exporter or vendor of the food.

7.1.3 The complete list of ingredients and food additives used in the preparation of the product in descending order of proportion. The common, local or usual name of the raw material used shall be specified.

7.1.4 The net content by weight in the metric system. Other systems of measurement required by importing countries shall appear in parenthesis after the metric system unit.

7.1.5 The words "Use by date"/"Consume Before" indicating end of period at which the product shall retain its optimum quality attributes at defined storage conditions.

7.1.6 Lot identification marked in code identifying product lot.

7.1.7 The words "Product of the Philippines" or the country of origin if imported.

7.1.8 Additional requirement

A pictorial representation of the product(s) on the label should not mislead the consumer with respect to the product so illustrated.

7.2 Labeling of Non-retail, Bulk Containers

The name of the product, lot identification code and the name and address of the manufacturer or packer shall appear in the container. However, the name and address of the manufacturer may be replaced by identification marks provided that such mark is clearly identified with accompanying documents.

7.3 Nutrition Labeling

Nutrition labeling shall conform to established regulations by the FDA.

8 Quality Assurance

8.1 Inspection of Finished Products

All processed products shall be inspected and should pass the quality criteria prescribed in Section 4 of the Standards forFish Sauce (Patis) and Fish Flavored Sauce (PNS/FDA 38:2014) and the type of defects, as defined in Section 6, in the lot examined must not exceed the acceptable number based on the appropriate sampling plan in Annex 5and/or its future amendments (FAO/WHO Codex Alimentarius Sampling Plans for Prepackaged Foods - CAC/RM 42-1969, Codex Alimentarius Volume 13, 1994).

8.2 Record Keeping

Permanent and legible dated records of production batches, code marks and other pertinent details shall be kept concerning each load. Such records are essential as a check on processing operations.

Written records of all package examinations shall specify the code lot and the date of package inspections, the measurements obtained and all the corrective actions taken.

Records identifying initial distribution of the finished product to facilitate, if necessary, the segregation of specific food lots that may have been contaminated or otherwise unfit for intended use, shall be kept and maintained.

All process deviations involving failure to satisfy the minimum requirements of the process shall be recorded detailing those deviations and the actions taken

8.3 Hazard Analysis and Critical Control Points (HACCP)

Appropriate HACCP plan must be developed for the product. Prior to the development of HACCP plan, establishments shall have developed, documented and implemented prerequisite programs (PRPs) based on FDA's Current Good Manufacturing Practices (cGMP) and Hygiene Control. Guidelines for the Application of the Hazard Analysis Critical Control Point (HACCP) System (CAC/GL 18-1993) present the recommended sequence and document formats for the application of the HACCP systems.

9 Storage and Transportation of Finished Products

Storage and transport conditions of the fish sauce (patis) and fish flavored sauce shall be such that the integrity of the product container is protected, and the safety and quality of the product are not adversely affected.

Cases and cartons must be thoroughly dry. They must be of proper size so that the containers fit snugly and are not subject to damage from movement within the case. They must be strong enough to withstand normal transport and distribution conditions.

Extreme temperature fluctuations, during storage and transport of the product must be avoided to prevent product deterioration.

10 Laboratory Control Procedures

Each food processing establishment shall have access to laboratory analyses and control of both the processes used and the finished products. All food ingredients and food products declared unfit for human consumption by the laboratory shall be rejected.

Representative samples for each lot or batch shall be randomly taken to assess the safety and quality of the product.

The microbiological laboratory shall be separated from the processing area. No pathogens shall be handled within the premises of the manufacturing plant.

Laboratory procedures for quality control of the processes and the product must follow recognized or standard methods for easy interpretation and recognition of the results.

11 End Product Specifications

Appropriate methods shall be used for sampling and analyses fish sauce (patis) and fish flavored sauce to meet the following specifications:

11.1 To the extent possible in good manufacturing practices, the products shall be free from any objectionable matter and parasites harmful to humans.

11.2 The product shall be free from micro-organisms in amounts harmful to humans and should not contain any substances originating from microorganisms in amounts which may represent a hazard to health.

11.3 The product shall be free from chemical contaminants in amounts which may represent hazard to health.

11.4 The product shall comply with the requirements set forth by the Food and Drug Administration (FDA) and the Codex Alimentarius Commission on Pesticide Residues and Food Additives.

Annex 1
(informative)

Species of Finfishes Utilized in the Production of Fish Sauce (Patis) and Fish-Flavored Sauce

English Name	Local Name	Scientific Name
1. Anchovy	*Dilis*	*Stolephorusspp*
2. Barracuda	*Tursillo*	*Spyraena spp.*
3. Big-eyed scad	*Matangbaka*	*Selarcrumenophthalmus*
4. Croaker	*Alakaak*	*Penahiaspp/*
5. Fimbriated sardines	*Tunsoy*	*Sardinellafimbriata*
6. Frigate tuna	*Tulingan*	*Auxisthazard*
7. Fusilier	*Dalagangbukid*	*Caesio spp.*
8. Indian mackerel	*Alumahan*	*Rastrelligerkanagurta*
9. Indian oil sardines	*Tamban*	*Sardinellalongiceps*
10. Japanese scad	*Galunggong*	*Decapterusmaruadsi*
11. Lizard fish	*Kalaso*	*Saurida spp.*
12. Mackerel scad	*Galunggong*	*Decapterusmacarellus*
13. Parrotfish	*Loro, Molmol*	*Leptuscarus sp.*
14. Round sardinella	*Lapad*	*Sardinellaaurita*
15. Short bodied mackerel	*Hasa-hasa*	*Rastrelligerbrachysoma*
16. Short finned scad or roundscad	*Galunggong*	*Decapterusmacrosoma*
17. Slipmouth	*Sap-sap*	*Leignatuhs spp.*
18. Spotted sardinella	*Tamban, tunsoy*	*Amblygastersirm*
19. Smooth belly sardinella	*Tamban*	*Amblygasterleiogaster*
20. Striped mackerel	*Alumahan*	*Rastrelligerkanagurta*
21. Threadfin bream	*Bisugo*	*Nemipterus spp.*
22. White sardinella	*Tunsoy*	*Sardinellaalbella*
23. Whiting, common	*Asuhos*	*Sillagosihama*

Reference:
Ganaden, S.R. and F. Lavapie-Gonzalez. 1999. Common and Local Fishes Names of Marine Fishes of the Philippines. Bureau of Fisheries and Aquatic Resources, Philippines. 386 pp

Annex 2

Standard for Iodized Salt

8. SCOPE

This standard applies to iodized salt used as condiment or an ingredient in the preparation of food in households, food service and food manufacturing establishments.

9. DESCRIPTION

Iodized salt is food grade salt that contains the prescribed level of iodine. It shall be produced refined or unrefined (crude) salt obtained from underground rock salt deposits or by evaporation of seawater or natural brine. The finished product shall be in the form of solid crystal or powder, white in color, without visible spots of clay, sand, gravel or other foreign matter.

10. IODIZATION PROCESS

Salt may be iodized with potassium iodate (KIO_3) or potassium iodide (KI) by means of any of the following methods:

a) dry mixing of salt in powdered form
b) dip feeding or spray mixing if salt is in crystal form
c) submersion of ice crystals in iodated brine

11. ESSENTIAL COMPOSITION AND QUALITY FACTORS

To ensure the stability of iodine, salt to be iodized must conform with the following quality requirements:

Moisture, minimum	4 % for refined salt
	7 % for unrefined salt
NaCl minimum	97 % dry basis
Calcium and magnesium, maximum	2 %
Water insolubles, maximum	0.2 %
Heavy meal contaminants	
Arsenic as As	0.5 mg/kg
Cadmium as Cd	0.5 mg/kg
Lead as Pb	2.0 mg/kg
Mercury as Hg	0.1 mg/kg

4.1 Naturally Present Secondary Products and Contaminants in Raw Salt

Notwithstanding the purity requirements in section 4.1. the raw salt may naturally contain secondary products, which are present in varying amounts depending on the origin and method of production of salt, and which are composed mainly of calcium, potassium, magnesium and sodium sulphates, carbonates, bromides and of calcium, potassium chlorides as well as natural contaminant may also be present in amounts varying with the origin and method of production of the salt.

5. LABELLING

5.1 Iodized salt for commercial distribution shall carry appropriate labeling in accordance with BFAD rules and regulations on labeling of prepackaged foods. Specifically, the following information shall be declared in every container of iodized salt whether in bulk or retail package.

5.1.1 For locally produced iodized salt

a) The name of the product, "IODIZED SALT", printed in bold capital letters
b) Name and address of manufacturer
c) Net weight
d) Iodine compound used
e) Chemical additives, e.g. anti-caking agents, emulsifiers
f) Open date marking, e.g. "Best Before" or "Consume Before" Date
g) Lot identification code (replacers must use manufacturer's lot i.d code)
h) Storage Instruction: STORE IN COOL DRY PLACE

5.1.2 For imported Iodized salt

a) same as 5.1.1 (a), (c) to (h)
b) Name and address of Importer/Local Distributor
c) Country of Origin

5.2 Labeling of Non-retail Containers

In the case of non-retail containers of at least 25 kg of iodized salt, the labelling information required in sections 5.1.1. (b), (d)or in 5.1.2 (b) may not be declared if such bulk packages are intended for delivery to distributors of food manufacturers/institutional users, provided every shipment or delivery is accompanied by a document containing all information in 5.1.1. or 5.1.2.

5.3 Iodine levels based on WHO recommendation

In order to meet national needs, the prescribed levels of iodized salt be indicated below:

	Type of Container	Packages
Sampling point	Bulk (>2 kg)	Retail (<2 kg)
Production site	70-150 g/kg	60-100 mg/kg
Port of entry*	70-150 mg/kg	60-100 mg/kg
Retail site	> 50 mg/kg	> 40 mg/kg
* For imported iodized salt, also at importer's/distributor warehouse		

12. FOOD ADDITIVES

12.1 All additives used, including KIO and KI, and shall be of food grade quality and shall conform to the specifications prescribed by JECFA of the Food Chemicals Codex.

	Maximum Level in the Final Product
6.1.1 Anti-caking Agents	
6.1.1.1 Coating agents; Carbonate. Calcium/magnesium, Magnesium oxide; Phosphate, Tricalcium; Silicon dioxide, amorphous; Silicates, calcium , magnesium, sodium alumino or sodium or sodium calcium alumino	20 g/kg singly or in Combination
6.1.1.2. Coating hydrophobic agents, aluminum, calcium, magnesium, potassium or sodium salts of myristic, palmitic or stearic acid	GMP
6.1.1.3 Crystal modifiers: ferrocyanide, calcium, potassium combination or sodium	10 mg/kg singly or in combination, expressed as {Fe(CN)}
6.1.2. Emulsifiers Polysorbate 80	10 mg/kg
6.1.3 Processing Aid Dimethylpolysiloxane	10 mg of residue/kg

7. PACKAGING

All iodized salt shall be packed in woven propylene bags, clean and unused jute bags, or other non-porous material with a lining of high density polyethylene to ensure the retention of appropriate iodine level at the time of consumption.

8. STORAGE, TRANSPORT AND DISPLAY AT RETAIL

In order to minimize avoidable losses of iodine, iodized salt shall not be exposed to any of the following conditions during storage, transport and display at retail outlets:

a) direct sunlight or near source of strong light
b) high temperature and humidity
c) contamination with moisture, e.g. rain, flood, etc.
d) contamination with dust or filth from the environment

October 10, 2007

Bureau Circular
No. 2007-009

Subject: Updated Standards for Iodine level of Salts

I. RATIONALE

Rule VI, Section 1 a) of the Revised Implementing Rules and Regulations (RIRR) of Republic Act (RS) No. 8172 also known as "An Act Promoting Salt Iodization Nationwide and for other Purposes" identifies Department of Health (DOH), as the lead agency in the implementing the said Act, and that through the Bureau of Food and Drugs (BFAD), the DOH shall set and enforce standards for food-grade iodized salt and monitor the compliance thereof by the food-grade manufacturers/importers, distributors and traders as specified in Section 2 Rule VIII.

The Food Nutrition and Research Institute (FNRI) on 26 May 2007 referred to the BFAD its recommendation on the possible levels of iodine across distribution stages. In particular, the FNRI proposed the following standard for iodine content:

	Type of containers/packaging	
	Bulk (<2 kilograms)	Retail (<2 kilograms)
Iodine Content	40-70 mg/kg	15-40 mg/kg

Also, attached with said letter are syntheses of studies conducted in other countries that provided empirical basis for regulatory decision.

It is emphasized that lowering the standard will harmonize the iodine level with other countries, will reduce cost and will encourage compliance. Also emphasized in the attachments is the international iodine standard which is 15-20 mg/kg.

II. DIRECTIVE

In view of the foregoing considerations, and ease of administration of regulatory standards, the BFAD hereby adopts the following standard for iodine content in pursuant of its mandate provided for in RA 8172.

Iodine Content	:20-70 mg/kg	across distribution channels, whether bulk or retail, imported or local

III. REPEALING CLAUSE

Provisions of previous issuances which are contrary to those reflected hereon are modified, and/or repealed accordingly.

IV SEPARABILITY

If any provisions of this Order is declared as unconstitutional, or not valid, the rest of the provisions thereon shall still subsist given their effect in entirety.

V. EFFECTIVITY

This Order shall be effective within fifteen (15) days after publication.

Annex 3

Standard Parameters and Values for Drinking Water

Philippine National Standards for Drinking Water 2007 (DOH AO 2007-0012)

Table 1. Standard values for bacteriological quality

Parameter	Value/Unit	Point of Compliance
Total Coliform	< 1.1 MPN/100 ml	Service Reservoir Water treatment works Consumers' taps Refilling stations Water haulers Water vending machines
Fecal Coliform	< 1.1 MPN/100 ml	Service Reservoir Water treatment works Consumers' taps Refilling stations Water haulers Water vending machines Point sources - Level 1
Heterotropic Plate Count	< 500 CFU/ml	Service Reservoir Water treatment works Consumers' taps nearest meter Refilling stations Water vending machines

Table 2. Standard values for Physical and Chemical Quality for Acceptability Aspects for Drinking Water

Constituents	Maximum Level (mg/L) or Characteristic	Constituents	Maximum Level (mg/L) or Characteristic
Taste	No objectionable taste	Hydrogen Sulfide	0.05
Odor	No objectionable odor	Iron	1.0
Color	Apparent = 10 color units True = 5 color units	Manganese	0.4
Turbidity	3 NTU	pH	6.5 – 8.5
Aluminum	0.2	Sodium	200
Chloride	250	Sulfate	250
Copper	1.0	Total Dissolved Solids	500
Hardness	300 as CaCO3	Zinc	5.0

Table 3. Standard Values for Organic and Inorganic Chemical Constituents of Health Significance in Drinking Water

Inorganic Chemical	Constituents	Maximum Level (mg/L)	Constituents	Maximum Level (mg/L)
	Antimony	0.02	Fluoride	1.0
	Arsenic	0.05	Lead	1.01
	barium	0.7	Mercury (total)	0.001
	Boron	0.5	Nickel	0.02
	Cadmium	0.003	Nitrate	50
	Chromium (Total)	0.05	Nitrite	3.0
	Cyanide (Total)	0.07	Selenium	0.01
Organic Chemical	**Constituents**	**Maximum Level (mg/L)**	**Constituents**	**Maximum Level (mg/L)**
	Benzene	0.01	Ethylbenzene	0.30
	Carbon tetrachloride	0.004	Nitrilotriacetic acid (NTA)	0.20
	1,2-Dichlorobenzene	0.1	Polyaromatic hydrocarbons (PAHs)	0.20
	1,4-Dichlorobenzene	0.5	Polynuclear aromatic	0.0007
	1,2-Dichloroethane	0.003	Tetrachloroethene	0.02
	1,1-Dichloroethene	0.05	Styrene	0.04
	1,2-Dichloroethene	0.07	Tetrachloroethene	0.70
	Dichloromethane	1.0	Trichloroethene	0.07
	Di(2-ethyhexyl) phthalate	1.01	Vinyl chloride	0.0003
	Edetic Acid (ADTA)	0.001	Xylene	0.5

Organic Pesticide	Constituents	Maximum Level (ug/L)	Status in the Philippines
	Aldrin and Dieldrin (combined)	30.0	Banned
	Atrazine	0.03	Registered
	Carbofuran	2.0	Registered
	Chlordane	7.0	Banned
	DDT **	0.2	Banned
	1,2-Dibromo-3-chloropropane (DBCP)	1.0	Banned
	2,4-Dichlorophenoxyacetic acid (2,4-D)	1.0	Registered
	Endrin	30.	Banned
	1,2-Dibromomethane (Ethylene dibromide)	0.6	Banned
	Heptachlor and Heptachlor epoxide (combined)	0.03	Banned
	Lindane	2.0	Restricted
	MCPA (4-(2-methyl-4-chloro) phenoxyl acetic acid	2.0	Registered
	Pendimethalin	20.0	Registered
	Pentachlorophenol (PCP)	9.0	Banned

Annex 4

Preparation of Brine of Required Strength

The amount of salt to be dissolved in water to obtain required brine strength: brine strength measured at 16°C (61°F)

Specific Gravity	Salt by Weight (%)	Salinometer Reading (°S)	Salt (kg) to be Dissolved in 100 L Water
1.007	1	3.8	1.0
1.014	2	7.6	2.0
1.022	3	11.4	3.1
1.029	4	15.2	4.3
1.037	5	19.0	5.3
1.044	6	22.7	6.4
1.051	7	26.5	7.5
1.058	8	30.3	8.7
1.066	9	34.1	9.9
1.073	10	37.9	11.1
1.081	11	41.7	12.4
1.089	12	45.5	13.6
1.096	13	49.3	14.9
1.104	14	53.1	16.3
1.112	15	56.8	17.6
1.119	16	60.6	19.0
1.127	17	64.6	20.5
1.135	18	72.0	22.0
1.143	19	75.8	23.5
1.151	20	79.6	25.0
1.159	21	83.4	26.6
1.168	22	87.2	28.2
1.176	23	91.0	29.9
1.184	24	94.8	31.6
1.192	25	98.5	33.3
1.201	26	100.0	35.1
1.204	26.4	-	35.9

Source: Codex Recommended International Code of Practice for Smoked Fish (CAC/RCP 26-1979).

Annex 5

Codex Alimentarius Sampling Plans for Prepackaged Foods (AQL 6.5) (CAC/RM 42-1969)
Sampling Plan No. 1 – Normal Operations
Inspection Level 1, AQL 6.5)

1. Net weight: ≤ 1 kg

Lot Size (N)	Sample size	Acceptance Number (C)
4,800 or less	6	1
4,801 – 24,000	13	2
24,001 – 48,000	21	3
48,001 – 84,000	29	4
94,001 – 144,000	48	6
144,001 – 240,000	84	9
More than 240,000	126	13

2. Net weight: >1 kg ≥ 4.5 kg

Lot Size (N)	Sample size	Acceptance Number (C)
2,400 or less	6	1
2,401 – 15,000	13	2
15,001 – 24,000	21	3
24,001 – 42,000	29	4
42,001 – 72,000	48	6
72,001 – 120,000	84	9
More than 120,000	126	12

3. Net weight > 4.5kg

Lot Size (N)	Sample size	Acceptance Number (C)
600 or less	1	1
601 – 2,000	13	2
2,001 – 7,200	21	3
7,201 – 15,000	29	4
15,001 – 24,000	48	6
24,001 – 42,000	84	9
More than 42,000	126	13

Sampling Plan 2 - In Case of Disputes
Inspection Level 2, AQL 6.5)

1. Net weight: ≥ 1kg

Lot Size (N)	Sample size	Acceptance Number (C)
4,800 or less	13	2
4,801 – 24,000	21	3
24,001 – 48,000	29	4
48,001 – 84,000	48	6
94,001 – 144,000	84	9
144,001 – 240,000	126	13
More than 240,000	200	19

2. Net weight: >1 kg ≥ 4.5 kg

Lot Size (N)	Sample size	Acceptance Number (C)
2,400 or less	13	2
2,401 – 15,000	21	3
15,001 – 24,000	29	4
24,001 – 42,000	48	6
42,001 – 72,000	84	9
72,001 – 120,000	126	13
More than 120,000	200	19

3. Net weight > 4.5kg

Lot Size (N)	Sample size	Acceptance Number (C)
600 or less	13	2
601 – 2,000	21	3
2,001 – 7,200	29	4
7,201 – 15,000	48	6
15,001 – 24,000	84	9
24,001 – 42,000	126	13
More than 42,000	200	19

Source: Codex Alimentarius Sampling Plans for Prepackaged Foods - CAC/RM 42-1969, Codex Alimentarius Volume13.

Annex 6

Determination of Total Nitrogen (TN) for Protein Determination (Improved Kjeldahl Method)

1. Reagents:

a. Sulfuric acid. – 93-98% H_2SO_4, N-free
b. Mercuric oxide or metallic mercury. – HgO or Hg, reagent grade, N-free
c. Potassium sulfate (or anhydrous sodium sulfate). Reagent grade, N-free
d. Salicylic acid, Reagent grade, N-free
e. Sulfide or thiosulfate solution. – Dissolve 40g com. K_2S in 1L H_2O. (Soln of 40 g Na_2S or 80 g $Na_2S_2O_3.5H_2O$ in 1L may be used.)
f. Sodium hydroxide.- Pellets or solution, nitrate-free. For soln, dissolve ca 450g solid NaOH in H_2O, cool, and dilute to 1L. (Specific gravity should be ≥1.36)
g. Zinc granules. – Reagent grade.
h. Zinc dust. – Impalpable powder.
i. Methyl red indicator. – Dissolve 1g methyl red in 200 ml alcohol.
j. Hydrochloric or sulfuric acid standard solution. – 0.5or0.1M when amount of N is small, or (sulfuric acid – 0.25 or 0.05M when amount of N is small)
k. Sodium hydroxide standard solution. –0.1M

Standardize each standard solution with primary standard and check one against the other. Test reagents before use by blank determination with 2 g sugar, which ensures partial reduction of any nitrates present.

[*Caution*: Use only freshly opened H_2SO_4 or add dry P_2O_5 to avoid hydrolysis of nitrites and cyanates. Ratio of salt to acid (w:v) should be ca 1:1 at the end of digestion for proper temperature control. Digestion maybe incomplete at lower ratio; nitrogen may be lost at higher ratio. Each g fat consumes 10 mL H_2SO_4 and each g carbohydrates 4 mL during digestion]

2. Apparatus:

a. For digestion. Use Kjeldahl flask or hard moderately thick, well-annealed glass with total capacity ca 500-800ml. Conduct digestion over heating device to bring 250 mL H_2O at 25C to rolling boil in ca 5 min or other time as specified in method. To test heaters, preheat 10 min if gas or 30min if electric. Add 3-4 chips to prevent superheating.

b. For distillation. Use 50-800 Kjeldahl or other suitable flask, fitted with rubber stopper through which passes lower end of efficient scrubber bulb or trap to prevent mechanical carryover of NaOH during distillation. Connect upper end of the bulb tube to condenser tube by rubber tubing. Trap outlet of condenser in such a way as to ensure complete absorption of NH_3 distilling over into acid in receiver.

3. Determination

a. Place weighed test portion of fish sauce (0.7-2.2 g) in a digestion flask. Add 0.7g HgO*or*0.65metallic mercury, 15 g powdered K_2SO_4 or anhydrous Na_2SO_4 , and 25ml H_2SO_4. If test portion > 2.2g is used, increase H_2SO_4 by 10 ml for each g test portion. Place flask in inclined position and heat gently until frothing ceases (if necessary, add a small amount of paraffin to reduce frothing); Boil briskly until solution clears and then add then ≥ min longer (2h for materials containing organic material).

b. Cool, add 200 ml H_2O, cool below 25°add 25 ml of the sulfide or thiosulfate solution, and mix to precipitate Hg. Add few Zn granules to prevent bumping, tilt flask and add layer of NaOH without agitation. (For each 10ml H_2SO_4 used, or its equivalent in diluted H_2SO_4, add 15 g solid NaOH or enough solution to make contents strongly alkaline.) (Thiosulfate or sulfide solution may be mixed with the NaOH solution before addition to flask.) Immediately connect flask to condenser and, with the tip of condenser immersed in standard acid and 5-7 drops indicator in receiver, rotate flask to mix contents thoroughly; then heat until all (NH_3) has distilled (≥150 ml distillate). Remove receiver, wash tip of condenser, and titrate excess standard acid in distillate with standard (NaOH) solution. Correct for blank determination on reagents.

4. Calculation of Percent Nitrogen (N) of Fish Sauce

a. When standard HCl is used:

Percent N = (mL of standard acid x molarity of acid) - (ml of standard NaOH x molarity of NaOH)] x 1.4007/ g fish sauce sample

b. When standard H_2SO_4 is used:

Percent N = (mL of standard acid x 2 x molarity of acid) - (ml of standard NaOH x molarity of NaOH)] x 1.4007/ g fish sauce sample

c. Calculation of Nitrogen Content from Percent to g/liter is computed as follows:

Nitrogen (g/liter) = % N x 10/gpecific gravity of fish

Specific gravity of fish sauce – 1.20

5. Calculation of Total Nitrogen to Percent Protein

% Protein = Nitrogen (g/liter) x PF/specific gravity x 10

PF = Protein Factor needed to convert nitrogen concentration to protein. For fish sauce and other fishery products, a PF of 6.25 is used. This is equivalent to 0.16 g nitrogen per gram of protein.

The conversion of total nitrogen (g/liter) to percent protein is provided in Table 1.

Table 1. Calculated percent protein from total nitrogen (TN) in g/liter

Total Nitrogen (gram/liter)	Protein (%)	Total Nitrogen (gram/liter)	Protein (%)	Total Nitrogen (gram/liter)	Protein (%)
0.5	0.26	10.5	5.47	21.0	10.94
1.0	0.52	11.0	5.73	21.5	11.20
1.5	0.78	11.5	5.99	22.0	11.46
2.0	1.04	12.0	6.25	22.5	11.72
2.5	1.30	12.5	6.51	23.0	11.98
3.0	1.56	13.0	6.77	24.0	12.50
3.5	1.82	13.5	7.03	24.5	12.76
4.0	2.08	14.0	7.29	25.0	13.02
4.5	2.34	14.5	7.55	25.5	13.28
5.0	2.60	15.0	7.81	26.0	13.55
5.5	2.86	15.5	8.07	26.5	13.81
6.0	3.13	16.0	8.33	27.0	14.07
6.5	3.39	16.5	8.59	28.0	14.56
7.0	3.65	17.0	8.85	28.5	14.85
7.5	3.91	17.5	9.11	29.0	15.11
8.0	4.17	18.0	9.38	30.0	15.63
8.5	4.43	18.5	9.64	30.5	15.89
9.0	4.69	19.0	9.90	31.0	16.15
9.5	4.95	20.0	10.42	31.5	16.41
10.0	5.21	20.5	10.68	32.0	16.67

References:

1. AOAC. 2005. Official Methods of Analyses of AOAC: Method 940.25, 955.04. AOAC International 18th Edition
2. *JAOAC* 38, 56(1955)

Annex 7

Determination of Salt (Chlorine as Sodium Chloride) (Volumetric Method)

1. Reagents

a. Silver nitrate standard solution -0.1 N. Prepare by dissolving slightly more than theoretical weight of $AgNO_3$ (Equivalent weight, 169.87) in halogen-free H_2O and dilute to volume. Thoroughly clean glassware, avoid contact with dust, and keep prepared solution in amber glass-stoppered bottles away from the light. Standardize against 0.1N NaCl containing 5.844g of dry NaCl/L.

b. Ammonium thiocyanate standard solution – 0.1N. Prepare ca 0.1N solution from reagent that shows no chlorine, using 7.612 g NH_4SCN or KSCN/L.

c. Determine working titer by accurately measuring 40-50mL standard $AgNO_3$ solution adding 2 mL ferric alum solution and 5mL HNO_3 (1 + 1), and titrating with thiocyanate solution until solution appears pale rose after vigorous shaking.

d. Ferric indicator – Saturated solution of $FeNH_4(SO_4)_2 \cdot 12H_2O$.

e. Nitric acid solution – 6N. Transfer 380 mL concentrated HNO_3 in 1L volumetric flask. Volume to 1L with distilled H_2O. Transfer to amber bottle.

2. Determination

a. Weigh 10 gram triplicate samples into 250 mL Erlenmeyer flask.

b. Add 20 ml of the standardized 0.1 N $AgNO_3$ or more than enough to precipitate all Cl as AgCl.

c. Add 20 ml of HNO_3. Boil gently on a hot plate or sand bath until all solids except AgCl solids dissolve (usually 15 min).

d. Cool, add 50 mL H_2O and 50 mL indicator and titrate with 0.1*N* NH_4SCN solution until solution becomes permanent light brown.

e. Subtract mL 0.1*N* NH_4SCN used from mL 0.1*N* $AgNO_3$ added and calculate difference as NaCl. With 10 g sample each mL 0.1*N* $AgNO_3$ = 0.058% NaCl.

References :

1. AOAC. 2005. Official Methods of Analyses of the AOAC: Official Method 937.09. AOAC International 18th Edition, 2005
2. JAOAC 20, 410 (1937); **23**, 589(1940)
3. CAS-7647-14-5 (Sodium Chloride)

Annex 8

Determination of pH

1. Principle

pH is measurement of H ion activity and indicates acidity. It may be measured by determining electric potential between glass and reference electrodes, using commercial apparatus standardized against primary standard pH buffers.

2. Apparatus and Reagents

(a) pH meter. Commercial instrument with scale graduated in ≤0.1 pH unit and repeatability of ≤0.05 unit. Some instruments permit expansion of any 2 pH unit range to cover entire scale and have accuracy of ca ⁊ 0.01 pH unit and repeatability of 0.005 pH unit. Other instruments have digital read-outs with similar capabilities. Operate meter in accordance with manufacturer's instructions.

(b) Standard buffer solutions – pH 4 buffer and pH 7 buffer.

(c) Electrodes – Glass membrane indicator electrode and calomel reference electrode (single or combination). Keep calomel electrodes filled with saturated KCl solution.

(d) Balance – With capacity of ≤2 kg and sensitivity of 0.1 g.

3. Standardization and Operation of pH meter

(a) Switch instrument on and let electronic components warm up and stabilize before proceeding.

(b) Standardize specific instrument according to manufacturer's instructions, using NIST SRM2 buffers. Equilibrate electrodes, buffers, and samples at same temperature (ca 2 °C) before pH measurements. Set temperature compensator control of instrument at observed temperature. When determining pH of either unknown sample or buffer, gently stir solution before testing.

4. pH Determination

(a) Obtain test portions of fish sauce for pH determination.

(b) Dilute fish sauce to 1:10 with distilled water by weight. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce (Codex Standard for Fish Sauce (Codex Stan 302-2011).

(c) Mix diluted sample and allow to equilibrate to ca 25°C. If pH meter is equipped with temperature compensator, then it may be used in lieu of equilibrating samples to specified temperature, provided it is ± 15 – 25°C.

(d) pH measurement. Rinse and blot electrodes. Immerse electrodes in sample and read pH, letting meter stabilize 1 min. Rinse and blot electrodes and repeat on fresh portion of sample. Determine 2 pH values on each sample. Readings in close agreement indicate that sample is homogenous. Report values to 2 decimal places.

Reference:

AOAC. 2005. Official Methods of Analysis of the AOAC: Method 981.12. AOAC International 18th Edition, 2005

Annex 9

Determination of Histamine (Fluorometric Method)

1. Principle

Product is extracted with 75 (v/v) methanol. Extract is passed through ion exchange column.) *o*-Phthaldialdehyde solution is added to eluate to form fluorescent histamine derivatives. Fluorescent intensity of derivatives is measured using fluorometer and histamine is quantified using external standards.

2. Apparatus

(a) *Chromatographic tube* – 200 x 7 id mm propylene tube fitted with small plastic stopcocks and ca 45 cm Teflon tubing. Control flow rate at >3mL/min by adjusting height of column relative to tubing outlet. Alternatively, use 2-way valve in place of tubing.

(b) *Photofluoremeter*– With medium pressure Hg lamp with excitation at 350 nm and measuring emission at 444 nm.

(c) *Repipets* – 1 or 5 mL

3. Reagents:

(a) *Ion exchange resin* - - Bio-Rad AG 1-X8, 50-100 mesh (Bio-RadLaboratories,1000 Alfred Nobel Dr. Hercules, CA 94547, USA:www.biorad.com)) or Dowex 1-X8, 50-100 mesh. Convert to –OH form by adding ca 15mL 2MNaOH/g resin to beaker. Swirl mixture and let stand <30 min. Decant liquid and repeat with additional base. Thoroughly wash resin with H_2O, slurry into fluted paper, and wash again with H_2O. Prepare resin fresh weekly and store under H_2O. Place glass wool plug in base of tube, **B(a),** and slurry in enough resin to form 8 cm bed. Maintain H_2O level above top of resin bed at all times. Do not regenerate resin in packed column; rather, use batch regeneration in beaker when necessary. Wash column with ca 10 mL H_2O before applying each extract.

(b) *Phosphoric Acid* – 1.19M as H_3PO_4. Dilute 121.8 mL 85% H_3PO_4 to L. For other concentration H_3PO_4, the volume required for 1L 1.19M H_3PO_4 = 17493/(densityH_3PO_4 x % H_3PO_4). Standardize 5.00 mL by titration with 1.00M NaOH to phenolphthalein end point and adjust concentration if necessary.

(c) *o-Phthaldialdehyde (OPT) solution* – 0.1%. Dissolve 100 mg OPT in100 mL distilled-in-glass methanol. Store in amber bottle in refrigerator. Prepare fresh weekly.

(d) *Histamine standard solutions.* Store in refrigerator.

(1) *Stock solution.* – 1 mg/mL as free base. Accurately weigh ca 169.1 mg Histamine·2HCl (98%) into 100 mL volumetric flask, and dissolve and dilute to volume with 0.1M HCl. Prepare fresh weekly.

(2) *Intermediate solution* - 10µg/mL.Pipet 1mL stock solution into 100 mL volumetric flask, and dilute to volume with 0.1M HCl. Prepare fresh weekly.

(3) *Working solutions* - 0.5, 1.0 and 1.5µg/mL. Pipet 1,2 and 3 mL intermediate solution into separate 100 mL volumetric flasks, and dilute each to volume with 0.1M HCl. Prepare fresh daily.

4. Preparation of Standard Curve

(1) Pipet duplicate 5 mL aliquots of each working standard solution into separate 50mL glass or polypropylene Erlenmeyers. Pipet in 10 mL 0.1M HCl to each flask and mix. Pipet in 3 mL 0.1M HCl and mix. Pipet in 3mL 1M NaOH and mix. Within 5 min, Pipet in 1mL OPT solution and mix immediately.

(2) After exactly 4 min. pipet in 3 mL 3.57 N H_3PO_4 and mix immediately. It is important to mix thoroughly after each addition and at least once during OPT reaction. (Run 6-10 OPT reactions simultaneously by adding reagents to Erlenmeyers in set order).

(3) Prepare blank by substituting 5 mL 0.1M HCl for histamine solution. Within 1.5 h record fluorescence intensity (*I*) of working standard solutions with H_2O in reference cell, using excitation wavelength 350 nm and emission wavelength of 444 nm. Plot (*I*) (corrected for blank) using µg histamine/5mL aliquot.

5. Determination

(1) .Extract prepared 10g test portion with 75% (v/v) methanol as in 957.07C , paragraph 1. (Transfer 10 g prepared test portion in semimicro container of high speed blender , add ca. 50mL methanol, blend in high-speed blender for ca 2 min.

(2) Transfer to 100mL glass-stoppered volumetric flask, rinsing lid and blender jar with methanol and adding rinsing to flask. Heat in H_2O bath to 60°C and let stand for 15 min at this temperature. Cool to 25°C, dilute to volume with methanol, and filter through folded paper. Alcohol filtrate may be stored in refrigerator several weeks.) (Light powdery precipitate separating on storage may be ignored).

(3) Pass 4-5 mL H_2O through column, **B(a)**,and discard eluate. Pipet 1mL extract onto column and add 4-5 mL H_2O. Immediately initiate column flow into 50 mL volumetric flask containing 5.00mL 1.00M HCl. When

liquid level is ca 2 mm above resin, add 5 mL H_2O and let elute. Follow with H_2O in larger portions until ca 35 mL has eluted. Stop column flow, dilute to volume with H_2O, stopper, and mix. Refrigerate eluate.

(4) Pipet 5 mL eluate into 50 mL Erlenmeyer, add pipet in 10 mL 0.1M HCl. Proceed as in preparation of calibration curve beginning "Pipet in 3 mL 1M NaOH....."

If test sample contains >15mg histamine/100g fish sauce, pipet 1 mL test solution-OPT mixture into 10 mL beaker containing exactly 2 mL blank-OPT mixture, and mix thoroughly. Read fluorescence of new solution. Dilute and mix aliquots with blank-OPT mixture as needed to obtain measurable reading. This approximation indicates proper dilution of eluate required prior to second OPT reaction needed for reliable quantitation of test solution. Alternatively, use sensitivity range control of fluorometer (if instrument has one) to estimate dilution. Use these approximations to prepare appropriate dilutions of aliquot of eluate with 0.1M HCl, and proceed as in the Preparation of Standard Curve, beginning "Pipet in 3mL 1M NaOH....."

6. Calculation

Plot of I (measured by meter of deflection or recorder response and corrected for blank) against µg histamine/5mL test solution should be straight line passing through origin with slope= $m = [(I_a/1.5) + I_b + 2I_c]/3$.

mg Histamine/100 g Fish sauce = $(10)(F)(1/m)(I_s)$

µg Histamine/g Fish sauce = 10 x (mg histamine/100g fish sauce)

where I_s, I_a, I_b and I_c = fluorescence from samples, 1.5, 1.0, and 0.5 µg histamine standards respectively; and F= dilution factor = (mL eluate = mL 0.1M HCl)/mL eluate. F = 1 for undiluted eluate.

If calibration plot is not linear, use standard curve directly from quantitation. Each subdivision on abscissa should be ≤ 0.1µg histamine/5 mL test solution.

Read all values from curve to nearest 0.05 µg histamine/5 mL test solution

mg Histamine/100 g Fish sauce = (10)(F)(W)

where W = µg histamine/5 mL test solution as determined from standard curve.

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1. AOAC. 2005. Official Methods of Analysis of the AOAC: Method 977.13. AOAC International 18th Edition
2. JAOAC 60, 1125, 1131 (1977)
3. CAS-51-45-6 (histamine)

Annex 10

Determination of Net Weight

1. **Apparatus:**

   Weighing balance (sensitivity: 0.10 gram)

2. **Procedure:**

   (a) Weigh the sample unit on its original sample packed container. This is the gross weight.

   (b) Open and pour out the contents of each individual package. Wash the empty package and blot dry.

   (c) Weigh out the washed empty package. This is the weight of the packaging material.

   (d) Subtract the weight of the empty package from the gross weight. The resulting figure is the net weight of the individual package (net weight = gross weight – weight of packaging).

   (e) Average the results from all package of a sample representing a lot. Report result as the average net weight of the product.

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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FOOD STANDARDS FORMULATING BODY

Development of Standards for Fish Sauce (*Patis*) and Fish Flavored Sauce

Food and Drug Administration - Department of Health

FDA Committee on Philippine National Standards for Processed Food Products

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