付属資料６　成果品例

１）　年報（Fish Technology Research & Inspection）

第１版～第３版（目次のみ）

FISH TECHNOLOGY RESEARCH & INSPECTION

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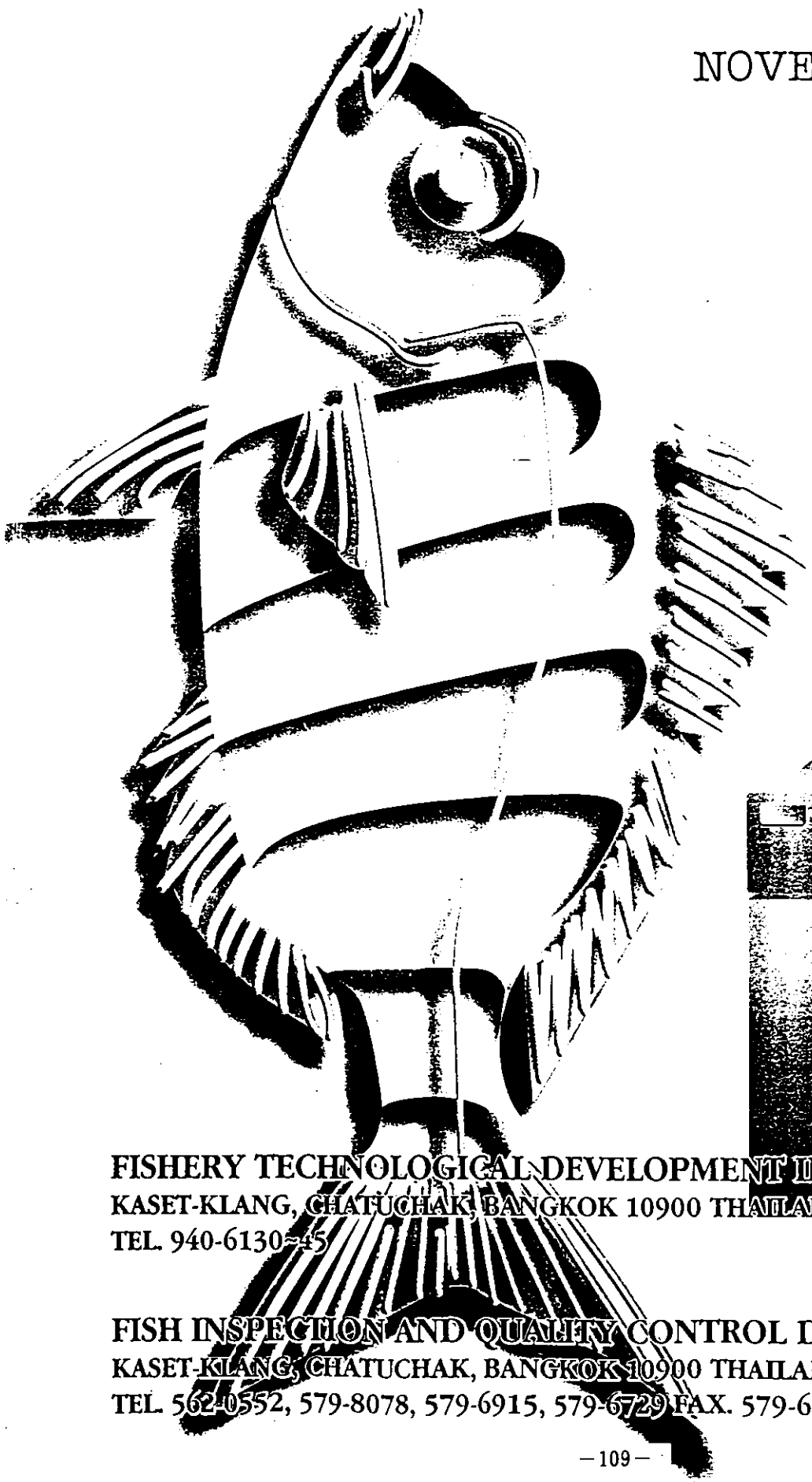

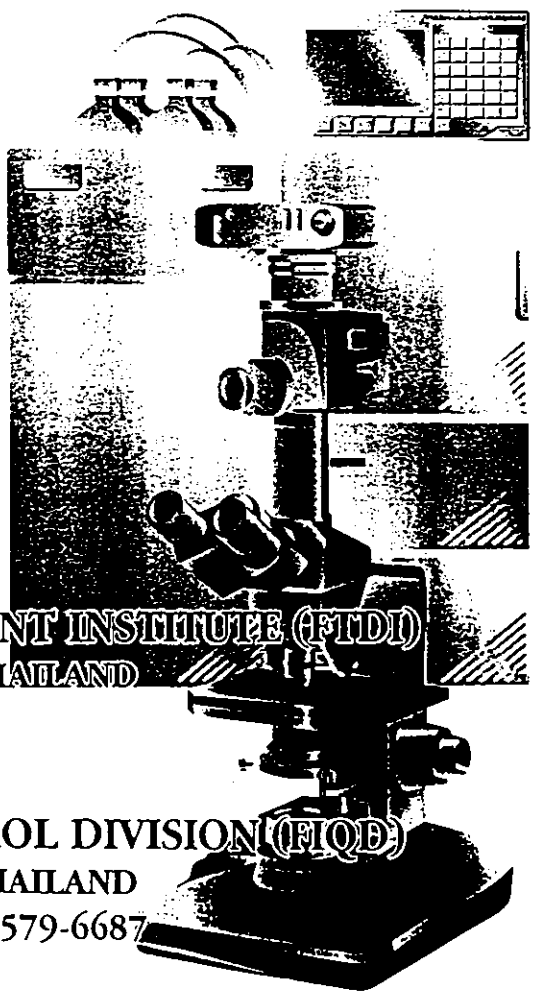

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Volume 1

CONTENTS

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Fish Technology Research and Inspection

Volume II

CONTENTS

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Fish Technology Research and Inspection Volume III

CONTENTS

付属資料６　成果品例

２）　FTDI GLPプログラム（抜粋）

・GLP組織図

・分析手順（水銀検出法）

・試験方法のSOP

・機器管理のSOP

Attached-1

FTDI GLP Program

I Objectives

1. to upgrade the overall quality of laboratory performance.
2. to provide the organization with a model to follow in setting up and operating laboratory management system.
3. to establish laboratory's creditability and reputation.

II General Requirements

1. Premises and equipment

1.1 Availability

The testing laboratory shall be furnished with all items of equipment for correct performance of the tests and measurements which it claims to be competent to carry out. In the exceptional case where the laboratory is obliged to use outside equipment, it shall be ensured the quality of that equipment.

1.2 Premises and environment

The environment in which the test are undertaken shall not be invalidate the test results or adversely affect the required accuracy of measurement. This applies in paticular at sites other than the permanent laboratory premises. The testing premises shall be protected as required from excessive conditions such as heat, dust, moisture, steam, noise, vibration and electromagnetic disturbance or interference, and shall be maintained accordingly. They shall be sufficiently spacious to limit the risk of damage or danger and to allow operators to make practical and precise movements. The premises shall have the equipment and energy sources needed for the testing. When the testing so requires, they shall be equipped with devices to monitor the environment conditions.

Access to and use all test areas shall be controlled in a manner appropriate to their designated purpose and conditions of entry by persons external to the laboratory shall be defined.

Adequate measures shall be taken to ensure good housekeeping in the testing laboratory.

2. Equipment

All equipment shall be properly maintained. Details if maintenance procedures shall be available.

Any item of the equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by calibration or otherwise to be defective, shall be taken out of service, clearly labeled and stored at a specified place until it has been repaired and then shown by test or calibration to be performing its function satisfactorily.

Records shall be maintained of each major item of test and measurement equipment.

Each record shall include:

2.1 The name of the item of equipment;

2.2 The manufacture's name, type identification and serial number;

2.3 Data received and date placed in service;

2.4 Current location, where appropriate;

2.5 Condition when received (e.g. new, reconditioned);

2.6 Details of maintenance carried out;

2.7 History of any damage, malfunction, modification or repair.

Measuring and testing equipment used in the testing laboratory shall be calibrated where appropriate before being put innto service and thereafter according to an established program.

The overall program of calibration of equipment shall be designed and operated so as to ensure that wherever applicable measurements made in the testing laboratory are traceable to national and international standards of measurement where available. Where traceability to national or international standards of measurement is not applicable, the testing laboratory shall provide satisfactory evidence or correlation or accuracy of test results (for example by participation in a suitable program of interlabratory comparisons).

Reference standards of measurement shall be calibrated by a competent body that can provide traceability to a national or international standards of measurement.

Where relevant, testing equipment shall be subjected to in-servvice checks between regular recalibration.

Reference materials shall where possible be traceable to national or international standards reference materials.

3. Personnel

3.1 The testing laboratory shall have sufficient personnel, having the necessary education, training, technical knowladge for their assigned functions.

3.2 The testing laboratory shall ensure that the training of its personnel is kept up-to-date.

3.3 Information on the relevant qualifications, training and experience of the technical personnel shall be maintained by the laboratory.

Ⅲ Organization Chart of FTDI Laboratory

See the Organization Chart

Ⅳ Organization's Responsibility

1. Laboratory director

a. Direct the scientific side of laboratory operation.

b. To ensure the laboratory's quality performance, must direst the preparation of the GLP, monitor its development and its implementation, and ensure its continued operation.

c. Must understand and be able to apply personnel management concepts and techniques.

d. As part of the quality assurance program, take prompt corrective action when innovation are recommended by manager of QAU who has observed error, deficiency, or out-of-control situation.

e. Must prepare and keep the documents that defined the responsibilities of division supervisor, section chief and analysts.

f. Approve preparation and amendment of SOPs.

g. Prepare training program for division supervisor, section chief and analysts, implement its and keep its records.

2. Division supervisor

a. Support position to the laboratory director in the management of the laboratory, including planning and policy formulation.

b. Provide direction of the work flow to section chief and analysts on a daily basis to perform laboratory operation, and confirm whether all of the operation

is carried out according to SOPs.

c. Prepare and amendment of SOPs and keep its.

d. Manage facilitied and instruments (equipment).

e. Confirm sampling and treatment of samples.

f. Decide the methords of analysis and ensure the proper validation of methords and results.

g. May direct and participation in validation and intra-laboratory proficiency program.

h. May participate in keeping specimen, data and copy of certificate.

3. Section chief and analysts

a. Carry out laboratory operation according to SOPs.

b. Who is designated as inspector in charge of instrument (equipment), reagent, test animals or hazardous materials management must check and record according to each SOPs.

c. Understand the importance of the quality assurance program (GLP) and the importance of the role of employee in making it succeed.

d. Should recognize potencial sources of error, report situation in which the quality of work falls below expected levels, ans abide by safety and house keeping rules in the performance of day-to-day assignments.

e. Understand the principles of methors used, foolow methors as written or carefully documents and deviations, keep accurate records, and have a basic knowledge of statistics and its application in laboratory quality control.

4. Quality assurance unit manager

a. Monitor quality assurance activities to determine conformance with policy, procedures, and sound practices.

b. Make recommendation for correction and improvement in the program.

c. Evaluate analytical data, equipment management records, reagent management records and other records generated under the GLP.

d. Advise management with regard to changing technology, new analytical methods, and new analytical equipment.

e. Coordinate or conduct quality program investigation.

f. Participate laboratory audits.

g. Participate intra-laboraotry proficiency tests.

GLP Organization Chart of FTDI Laboratory

Director

GLP Commitee
Chair Person : Pensri
Rerngrudee
Jirawan
Niracha
Attaya
Varatip
Porathip
Orawan
Sunee
Sumate
S. Saito (Advisor)

Quality Assuarance Unit (QAU)
Manager : Preeda
Porathip
Panatip
Bordin
Supaporn

Microbiological Division
Supervisor : Niracha
Bacteriology : Supaporn*
Virology : Nitacha*

Analysts :
Bordin (1) (2) (3) (4) (5)
Supaporn (2) (3)
Jiraporn (1) (2) (3)
Janissata (5)
Rachada (1) (3) (4) (5)
Daungchai (1) (2) (3) (4) (5)

Chemical Division
Supervisor : Rerngrude
Freshness : Sumate*
Quality Index : Varatip*
Food Additive : Jirawan
NCR : Pantip*
Contaminant : Niracha*
Biotoxin : Attaya*

Analysts :
Orawan (1)
Varatip (2)
Pantip (1)
Poratip (5)
Sumate (1) (2)
Sunee (3)
Bordin (2)
Jiraporn (4)
Somyos (5)
Rachada (4)
Sirirat (3) (5)
Janissata (3) (5)
Pawared (4)
Danai (3) (4)

Sensory Division
Supervisor : Jirawan
Sample : Rerngrudee*
Panel : Sumate*
Testing : Jirawan*

Analysts :
Rerngrudee (1) (2) (3)
Jirawan (1) (2)
Poratip (2) (3)
Attaya (1) (4)
Sumate (1) (2) (3) (4)
Amonrat (1) (2) (3) (4)
Jiraporn (1) (4)

Aminal Testing Division
Supervisor : Attaya
Sample : Attaya*
Tested animal : Bordin*
Waste : Pawared*

Analysts :
Attaya (1) (2) (3) (4)
Bordin (2) (3) (4)
Sirirat (1) (2)
Pawared (2) (4)

Physics Division
Supervisor : Orawan
AW : Janisata*
Color measurement :Pantip*
Texture : Orawan*
Temperature : Sumate*
Heat Penetration : Varatip*
Packaging Quality-
Management : Varatip*
Filth :

Analysts :
Orawan (1) (2) (3) (4)
Pantip (1) (2) (3)
Varatip (2) (4)
Sumate (1) (2) (3) (4)
Janissata (1) (2) (3) (4)
Pawares (1) (2) (4)

* Chief of Section

(1) ~ (5) : Job responsibility

Mercury in Fish

Simplified Method by Mercury Analyzer

Principles

Sample is digested with HNO_3 and H_2SO_4 with a help of urea and potassium permanganate.

Ion Hg is reduced by $SnCl_2$ to metal Hg. Determine absorption of vaporized Hg using Mercury Analyzer

Apparatus

(1) Mercury Analyzer

Operating conditions: Wavelength 253.7 nm, slit width 160 um, lamp current 3 ma and sensitivity scale 2.5

(2) 100 ml volume Erlenmeyer flask

(3) Eye drop shaped glass stopper See figure 1.

Reagents

(1) Reducing solution

Mix 50 ml H_2SO_4 with ca 300 ml H_2O. Cool to room temperature and dissolve 15 g NaCl, 15 g hydroxylamine sulfate, and 25 g $SnCl_2$ in solution. Dilute to 500 ml.

(2) Diluting solution

To 1 L volumetric flask containing 300 - 500 ml H_2O, add 58 ml HNO_3 and 67 ml H_2SO_4. Dilute volume with H_2O.

(5) Sulfric acid(H_2SO_4)

(6) Nitric acid(HNO_3)

(7) 10 % Potassium permanganate solution

(8) 10 % Urea solution

(9) Hydroxylamine sulfate(Hydroxyl Ammonium Sulfate) $(NH_2OH)_2SO_4$

(10) Tin(II) Chloride (heavy metal analysis grade)

(11) Mercury Standard Solution

a. Stock Solution(1,000 μ g/ml), Dissolve 0.1354 g $HgCl_2$ in 100.0 ml distilled water

b. Working Solution(100 ng/ml), Dilute 10 ml stock soln. to 100 ml with 1 N H_2SO_4, and dilute 10 ml dilluted soln. above to 100 ml with 1 N H_2SO_4, repeat diluting 10 ml to 100 ml twice times again to obtain 100 ng/ml of solution. Prepare fresh daily.

Determination

Weigh 1.0 ~ 2.0g sample into 100 ml volume erlenmeyer flask

Rinse neck of flask with <5 ml H_2O, if necessary

← 5 ml H_2SO_4
← 20 ml HNO_3

Quickly placce a eye drop shaped stopper on the flask

Swirl to mix

Heat on the hot plate at 160 ℃ for 60 mims(or 180 ℃ for 30 mins)

Swirl flask intermittently during digestion

Remove flask from heat

← 5 ml 10 % Urea soln.
← 5 ml 10 % Potassium permanganate soln.

Heat on the hot plate for 10 mins

Cool digested sample solution to room temperature

Rinse glass stopper with H_2O

Transfer digest to 100 ml volumetric flask

Ignore solidified fat(it does not interfere)

Carefully rinse digestion flask with several portions H_2O

Dilute to volume with rinse H_2O

(Transfer 25.0 ml of digesting solution to another 100 ml volumetric flask)

Dilute to volume with diluting solution if its necessary

Measure Hg by Mercury Analyzer

Preparation of calibration curve

Add 0, 5, 10, 15 and 20 ml of working standard soln. to series of 100 ml volumetric flasks.
To each flask, dilute to volume with soln.
Measure Hg by Mercury Analyzer.

Plot standard curve from least squares linear regression of A against ng Hg.

Determine ng Hg in aliquot from curve. If ug Hg determined falls outside range of calibration, repeat determination with smaller aliquot of sample solution to bring ng Hg into region of standard curve.

From size of aliquot used, detemine total μ g Hg in original sample.

Calculation

Hg(ppm) = ng Hg x D x 100(/25) x 1/ S x 1/1000

ng Hg : Obtained ng Hg in aliquot
D : Dilutiong times
S (g) : Original sample size

Worksheet for Process check
Analytical items : Mercury in Fishy by Flameless AAS

Samples No. :
Commodity :

Date Start (/ /)
Date finish(/ /)

Samples

	Samples No.	Name	Notes
(1)	( )	( )	( )
(2)	( )	( )	( )
(3)	( )	( )	( )
(4)	( )	( )	( )
(5)	( )	( )	( )
(6)	( )	( )	( )
(7)	( )	( )	( )
(8)	( )	( )	( )
(9)	( )	( )	( )
(10)	( )	( )	( )

Sampling

	Sample size(5g)	Details of sample portion
(1)	(g) ☐ Mix whole ☐ Portion(	)
(2)	(g) ☐ Mix whole ☐ Portion(	)
(3)	(g) ☐ Mix whole ☐ Portion(	)
(4)	(g) ☐ Mix whole ☐ Portion(	)
(5)	(g) ☐ Mix whole ☐ Portion(	)
(6)	(g) ☐ Mix whole ☐ Portion(	)
(7)	(g) ☐ Mix whole ☐ Portion(	)
(8)	(g) ☐ Mix whole ☐ Portion(	)
(9)	(g) ☐ Mix whole ☐ Portion(	)
(10)	(g) ☐ Mix whole ☐ Portion(	)

Digestion

◎ H_2SO_4 ☐ 5 ml, ☐ Other(ml)
◎ HNO_3 ☐ 20 ml, ☐ Other(ml)
◎ Heating time ☐ 30 mins, ☐ 60 mins, ☐ Other(mins)
◎ 10 % Urea soln. ☐ 5 ml, ☐ Other(ml)
◎ 10 % $KMnO_4$ soln. ☐ 5 ml, ☐ Other(ml)
◎ Second heating time ☐ 10 mins, ☐ Other(mins)

Measurement

◎ Date of standard preparation(/ /)
◎ Concentration of stock standard sol. ☐ 1,000 ppm ☐ others(ppm)
◎ Concentration of working standard sol. ☐ 100 ppb ☐ others(pp)
◎ Use auto-sampler ☐ Yes ☐ No

	Sample No.	Bial No.	Dilution of Sample sol.
(1)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(2)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(3)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(4)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(5)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(6)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(7)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(8)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(9)	( )	( )	☐ No ☐ Yes(ml → ml Times)
(10)	( )	( )	☐ No ☐ Yes(ml → ml Times)

Calculation

Conc. in Sample solution (ng/ml) | Dilution(Times) | Volume(100ml)(/25) | Sample size(g)

[ ] × [ ] × [ ] × [1/]
× 1/1000 = [Hg ppm]

※ Comment s on data ☐ No ☐ Yes
If yes, write comments

Analyst _____

Supervisor _____

Worksheet for calculation

Analytical items : Mercury in Fish by Simplified Method

Samples Number	Name of fish	Conc. in Sample Sol. (ng/ml) × Dilution (Times) × Volume(ml)(/25) (ml)/25 × 1/Sample size(g) 1/(Sg) × 1/1000	Result (ppm)	Notes

Standard	Consenration(ng/ml)	Peak high or area
STD-1	0	
STD-2	5	
STD-3	10	
STD-4	15	
STD-5	20	
STD-6		

Date of Analysis

Analyst

Supervisor

Standard Operational Procedures of Surimi Test

By Japan Frozen Food Inspection Corp.
Research Division

1. Moisture

Weigh accurately 5 g ca(A) of duplicate half thawed Surimiin pre-dry and weighed Alminium Cup(B)

|

Dry in oven at 105 ℃ for 20 hours

|

Keep in decicator for at least 10 minutes

|

Weigh(C)

|

Calculation

$$\text{Moisture}(\%) = \frac{(A + B) - C}{A} \times 100$$

2. Impurities

Weigh 40 g of Surimi in plastic bag

|

After completely thawed, Spread in 1 millimeter(mm) thickness

|

Count black skin and bone residue

Counting :

Impurities larger than 2 mm in size as one(1)
Impurities 1 to 2 mm in size as half(0.5)
Impurities less than 1 mm in size are ignored as insignificant.

3. pH

Weigh 10 g of duplicate thawed Surimi

|

Add 90 milliliter(ml) of distilled water

|

Homogenize using high speed blender

|

Measure by pH meter(Glass-electrod Horiba F-13 pH meter)

|

Two sample of results are averaged

4. Gel-Forming Ability

4-1. Gel Preparation(Kamaboko Process)

Cut frozen Surimi into two Surimi blocks of weight about 2.2 to 2.5 Kgs with one corner of original block

↓

Keep one piece of the said Surimi block in the cold storage, and the other piece to be used for the first test

Thawed frozen Surimi in refrigerator with temperature of 2 to 5 ℃ (Leave cut frozen Surimi block in refrigerator at around 5 pm the day before when gel-forming test performed)

↓

Weigh 2.0 Kgs of thawed Surimi

↓

Slice in 1 to 2 centimeter(cm) thickness

↓

The temperature of the Surimi must be below - 5 ℃ before Grinding

↓

Grind by Silent Cutter

↓

Add 60 grams(3 % equivalant) after 4 to 6 minutes

↓

During grinding, scrape meat in the bowl and cover of Silent Cutter twice

↓

Finish grinding
Final temperature must be betwen 6 to 8 ℃ as well as the grinding time must be more than 13 minutes.
If temperature rises to six(6) ℃ and time runs over 13 minutes, finish grinding.
In case that temperature rises over eight(8) ℃ but grinding time is shorter than 13 minutes, the retest is performed using the other Surimi block said above which has being kept in the cold storage.

↓

Slam meat to bowl of Silent Cutter for 7 to 8 times

↓

Stuff Surimi paste into 3 pieces of 30 mm-diameter plastic casings for without Suwari tests.

↓

Link the plastic casings in about 25 cm length using Stapling Machine

↓

Boil at 90 ℃ for 40 minutes for without Suwari immediately after stuffing

Incubate at 35 ℃ for 60 minutes for high temp. Suwari process
incubate at 10 ℃ for 18 hours for low temp. Suwari process
Incubate at 60 ℃ for 30 minutes for Modori process

↓

Heat at 90 ℃ for 40 minutes

↓

Cool in running water for 5 minutes and then ice water for 15 minutes

↓

Stand at 20 ℃ for not less than 18 hours and not more than 48 hours

4-2. Gel Strength Tests

Cut in 25 mm height of four test specimens from one Kamaboko piece to get twelve(12) specimens for each process(without Suwari, with Suwari at high temp. and with Suwari at low temp.)

|

Measure W-value(Yield Stress-grams) and L-value (Indentation Depth-cm) for 12 specimens using Rheometer

Conditions of Rheometer:

Range	02000 g (2000 g)
Test speed	06 cm/min.
Sweep speed	006 cm/min.
Adapter diameter	05.0 mm (5 mm)
Sample height	025.0 mm (25 mm)
Detecter	02 K
(Plunger shape	Round)

Calculation :

First, eliminate abnomal test data caused by the presence of air bubble(s) which is determined by JEFFIC staff based on the chart record.

Second, eliminate the heighest and the lowest value for each of Yield Stress(W) and Indentation Depth(L) independently.

Average value for W and L, yielding Jelly Strength

$JS(g \cdot cm) = W \times L$

4-3. Folding Test

Slice Kamaboko piece in 3 mm thickness

|

Fold between thumb and index fingers

Counting :

AA = No crack occurs after folding twice

A = Crack occurs after folding twice, but no crack occurs after folding once

<A = Crack occurs after folding once

4-4. Hunter Whiteness Test

Measure Z value in the tri-stimulus XYZ system for test specimens prepared at the Gel Strength tests using Colorimeter.
The test is performed in triplecate and the results are averaged.

4-5. Colour Tone

Measure L*, a*, b* value for test specimens prepared at the Gel Strength tests using Colorimeter
The test is performed in triplecate and the results are averaged.

4-6. Whiteness

$$\text{Whiteness} = 100 - \sqrt{(100 - L^*)^2 + a^{*2} + b^{*2}}$$

SOP for Maintenance of Equipment
[Incubator]

1 Name of equipment
Incubator

2 Methods of check for maintenance at the use
Person(s) who use the equipment(hereinafter refered to as "Operator") must check following items before start and each time at the use after that. For consecutive use with same condition, follow frequency described in each items.
Person who is responssible for the equipment(hereinafter refered to as "Supervisor") must confirm that maintenance check had been done properly at the end of work day.
All these check data and confirm data must be recorded on the log book form 1(Incubator log book at use).

(1) Appearence
Check damage and stain of main body and thermometer

(2) Cleaning and disinfection
Spray antiseptic alcohol and wipe with cloth sorked in alcohol inside of incubator once a week. Record on the log book form 1. In case of dirt, filth or stain is appeared on, clean immediatly.

(3) Temperatute check
Measure temperature inside of incubator at 900 AM and 1300 PM(allow 30 inutes before and after), and record max-min temperature for past 24 hours by reading the auto-thermo-recorder at 900 AM. Temperature should be kept ±1.0℃ of setting temperature. If there are no auto-thermo-recorder equiped, check max and min temperature for past 24 hours using max-min thermometer and record on the log book form 3 (Incubator log book for temperature management).

(3) Temperature differences
Using standard termometer, measure temperature defferences by equipment once a week and record log book form 3.

(4) Changing condition(temperature)
Any change of condition(temperature) should be recorded on the log book for form 1(Incubator log book at the use) with name and date changed.

3 Methods of regular inspection for maintenance
Supervisor must check following items by the frequency of

twice a year(in August and February).

(1) Confirmation of check at the use
Proper check at the use and maintenance was conducted, and of all these data were recorded on the log book form 1.
(2) Confirmation of temperature inside incubator
Conduct caribration using standard thermometers to assure the accuracy of indicated temperature inside of incubator.
(3) Temperature differnces
Measure and culcurate temperature differences between actual temperature and indicated temperature on equipment, auto-thermo-recorder and amx-min thermometer using standard thermometers.

4 The measurement for trouble shooting

(1) Report immediately to supervisor and unit chief whenever trouble is happened, and record the facts on the log book for occurance of troubles(Form 3).
(2) Never use trouble caught equipment until repaired.
(3) The supervisor who received trouble report, should try to repair according to instruction possibly. If it is unable to repair by him or her self and concluded necessity of calling serviceperson(s), report to unit chief.
(4) The unit chief should investigate into troubles, order to repair to the supervisor to contact with service company after recognized the needs of repair.
(5) The supervisor must inspect the conditionsof equipment after completion of repair and record on the log book form 3.
(6) Operator must inspect with attendance of the supervisor at the first use after repair. All the evidences forrepair must be kept in file.
(7) Address and phone number of servisce company must be written on the log book Form 1. (prefer to attach a name card of serviceperson)
(8) If any trouble is happened on this incubator being occupied, contens must be moved to another incubator to prevent any subsequent detarioration.

5 The other requirments

(1) Equipment should be installed at the fixed place and indicated a name of the equipment and a name of the supervisor.

(2) Set temperature must be indicated on it.(Prefer to attach card or lable)

(3) Equipment should not be moved without proper reason(s) except necessity to be used at the other place indispansably. In that case, it should be put back to where it is used be immediately after finishing, and record on the log book form 1.

6 Recording methods of check for maintenance on the log book

(1) Recording on the log book at the use

Operator must check each items of chapter 2 describedabove at the use and mark or write in each column of the incubator log form 1.

If adjustment was done according to the caribration, record adjusted values in the notes.

Supervisor must review of checked results for each items of log book and sign on it.

Unit chief must confirm and sign at the end of the page.

(2) Recording on the log book for regular inspection

Supervisor must examine each items of chapter 3 described above and mark or write in each colmn of the incubator log book form 2, and write the data inspected and sign on it.

Judgement should be done as "Pass" for nothing problem or "Reject" for there some problems.

Unit chief must confirm and sign at each regular check.

7 Established November 25, 1997 by S. Saito, Leader JICA Project

Aproved November 30, 1997 by

Amendments:

付属資料６　成果品例

３）　クロスチェック実施要領

Attached-2

Cross-check

I Objectives :

Since starting of this project, a several tens of items of analytical methods have been transfered to both of Fishery Technology Development Institute (FTDI) and Fish Inspection and Quality Control Division (FIQD) of Department of Fisheries.

Until the last year of this project, any evaluation have not been conducted. According to the answers from questionaire on how they understand the technology of analysis that project have transfered, a few items were found to be acceptable for cross-check.

Originally, purposes of cross-check are to assure tho laboratory quality assurance system conducted through distributing referece materials by intenationally recognized institutes.

Therefore, a series of cross-check were planned and conducted for evaluation of technology transfer and for evaluation of proficiency of intra and inter laboratory among FTDI and FIQD.

II Items of analysis for cross-check

1. Food additives
   - a Bezoic acid
   - b Sorbic acid
   - c EDTA
   - d Poly phosphate
   - e Sulfur dioxide
2. Heavy metals
   - a Cadmium
   - b Lead
   - c Mercury
3. Biotoxins
   - a Paralytic shellfish poison (PSP)
   - b Amnestic shellfish poison (ASP) (Domoic acid)
4. Antibiotics
   - a Oxolenic acid
   - b Oxytetracycline
5. Histamine
6. Pesticides residue
   - a BHC, DDT and Drins
   - b Trichlorfon, Dichlorvos
7. Nutritional components

   a Moisture, b Protein, c Fat, d Ash, e Phosphorus, f Iron, g Calcium, h Iodine, i Retinol, j Catotane, k Vitamine B1, l Vitamine B2, m Niacin, n Vitamine E, o Cholesterol, p Fatty acid compositions

8. Microbiological test

a Aerobic Plate Count

b Detection of Coliform group, E. coli, Salmonella group, Staphyrococcus aureus and Bacillus cereus

Among these items, EDTA, Poly phosphate, Oxolenic acid, oxytetracycline have not been conducted for some reasons finally.

Ⅲ Samples

1. Bezoic acid , Sorbic acid, Cadmium, Lead

To prepare fake food, the mixture of tomato ketchup and worcester sauce, spiked with each standards to make following concentrations.

Analytical items	Concentration (ppm)	
	Sample blank	Spiked sample
Bezoic acid	0	360
Sorbic acid	0	240
Cadmium	0	2.5
Lead	0	6.25

2. Mercury

Chop and mix fresh shark meat. As a reference data, request to OMIC to analyse according to AOAC's official method fo mercury for fish (AOAC Final Method 974.14 Mercury in Fish)

Obtained data are as follows.

	1	2	3	Average
Mercury contents (ppm)	0.64	0.54	0.60	0.59

3. EDTA and Poly phosphate

Add EDTA · 2Na, EDTA · 2NaFe, and EDTA · 2NaCa and Potassium phosphate, Pyropolyphosphate, Trypolyphosphate, Hexapoluphosphate to mixture of fresh meat of Sawara and Seabass to prepare following concentrations.

Analytical items	Concentration (ppm)		
	Sample blank	Sample 1	Sample 2
EDTA	0	230	505
Polyphosphate as phosphorus (P)	0	90	180

4. Histamine, Oxolenic acid and Oxytetracycline

Add Histamine dihydrochloride, Oxolenic acid and Oxytetracycline hydrochloride to fresh

meat of shrimp to prepare following concentrations.

Analytical items	Concentration (ppm)		
	Sample blank	Sample 1	Sample 2
Histamine	0	50	100
Oxolenic acid	0	0.5	1.5
Oxytetracycline	0	0.8	2.4

5. Sufur dioxide

Add Sidium sulfite to fresh meat of shrimp to prepare following concentrations as Sulfur dioxide.

Analytical items	Concentration (ppm)		
	Sample blank	Sample 1	Sample 2
Sulfur dioxide	0	30	90

6. Biotoxins

6-1. Paralytic shellfish poison (PSP)

Mix fresh scalop meat with digestive gland in which known concentration of PSP is contained.

6-2. Amnestic shellfish poison (ASP) -Domoic acid

Add domoic acid (Standard for HPLC) to fresh scalop meat to prepare concentration of 5 μ g/g as domoic acid.

Analytical results of PSP and ASP by Japan Frozen Foods Inspection Corporation as reference laboratory data are as follows.

Analytical items	Sample 1	Sample 2	Sample 3
PSP (MU/g)	29	30	31
ASP (ppm)	4.2	4.4	4.2

7. Nutritional components and Fatty acid conpositions

Process fish powder from Lizard fish at the commercial plant.

As the reference data, order to analyse from Japan Frozen Foods Inspection Corporation. Obtained data are contained in results table.

8. Microbiological tests

8-1. Aerobic plate count

Using commercial filter paper slips absorbing 1 million of spores of Bacillus subtilis.

8-2. Detection of Coliform group, E. coli, Salmomella, Staphlococcus aureus and Bacillus cereus.

Prepare using 10 kinds of type culture, enriched in nutrient broth individually and absorbed in gouge. Detail of samples is contained in results table.

IV Results

Results are shown in Table 1, 2, 5, 6, and 7, and Table 3 and 4 showed the results of statistical anlysis for BA, SoA, Cd, Pb, Hg and Aerobic plate count.