

GBT 16869-2005 Fresh and Frozen Poultry Products



National Standards of People's Republic of China

GB/T 16869-2005

National Food Standard
Fresh and Frozen Poultry Products

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People's Republic of China & China National Standardization Management Committee**

Foreword

Chapter 6 of this standard is voluntary while the rest of the standard is mandatory.

This standard replaces GB 16869-2000 Fresh and Frozen Poultry Products.

As compared to GB 16869-2000, key changes are as follows:

- Detection limits on amount of methamidophos and lenbuterol hydrochloride were removed as part of the requirements;
- Added the criteria that if the surface area of the extravasated blood patch does not exceed 0.5cm², then it can ignored entirely, also the calculation methods and testing guidelines for the number of extravasated blood patches and bristles were added as well;
- Some of the technical requirements were modified;
- Maximum temperature allowed for frozen core portion of frozen poultry products was adjusted to -18oC;
- Maximum loss of moisture allowed during thawing was adjusted to 6%;
- Limits on lead was adjusted to amount not exceeding 0.2mg/kg;
- Residue limits on pesticide beta-Hexachlorocyclohexane (BHC) was adjusted to amount not exceeding 0.1mg/kg (as fraction of total sample mass), 1mg/kg (as fraction of fats);
- Limits on coliform in frozen poultry products was adjusted to amount not exceeding 5×10³ MPN/100g;
- Detection limits on salmonella was adjusted to “0/25g”;
- Detection limits on diarrhea-inducing Escherichia coli was renamed to those for Enterohemorrhagic Escherichia coli (EHEC) (O157:H7), of which limit is 0/25g;
- Determination of diethylstilbestrol was modified to be “in accordance with determination method as required by SN 0672.”

Details in Chapter 6, such as routine tests, settlement inspection, sampling plans and number of general defects allowed, are equivalent to adopting the inspection standards I and inspection standards II of the CAC/RM 42-1969 Sampling Plans for Prepackaged Food Products.

Appendix A is a normative appendix.

This standard was proposed by the National Technical Committee of Food Industry Standardization, Ministry of Health Technical Committee of Food Industry Standardization.

This standard is under the jurisdiction of the National Technical Committee of Food Industry Standardization.

The organizations involved in the drafting of this standard: Food Hygiene Supervision and Inspection Authority of the Ministry of Health, Secretarial Department of the National Technical Committee of Food Industry Standardization, Shanghai City Food Hygiene Supervision and Inspection Authority of the Ministry of Health, Center of Inspection of Slaughtering Process of the State Trade Bureau, Inspection Center for Poultry Products of the Ministry of Agriculture, China Meat Trade Association, Beijing CIQ and Shenzhen CIQ.

Key personnel involved in the drafting of this standard: Yu Hao, Yulian Han, Jingyu Gu, Bingqi Ruan, Linan Lan, Xiaoming Yang, Hong Liu, Suying Liu, Chunfeng Lee, Guoying Tan.

The organizations involved in the drafting of Appendix A of this standard: Institute of Nutrition and Food Hygiene of Chinese Academy of Preventive Medicine and Food Hygiene Supervision and Inspection Authority of the Ministry of Health.

Key personnel involved in the drafting of Appendix A of this standard: Huijing Chan, Xuqin Wang, Dajin Yeung, Guohua Wu.

The standard will replace the previous versions:

- GB 2710-1966, GB 16869-1997, GB 16869-2000

National Food Standard

Fresh and Frozen Poultry Products

1. Scope

This standard specifies the technical requirements, testing methods, inspection guidelines, and the requirements on logo, labeling, packaging and storage of fresh and frozen poultry products.

2. Normative References

Clauses involved in the following documents constitute the ones in this standard through reference in this standard. Any dated reference and the following amendment or revised versions (excluding errata) are not applicable to this standard. However, the study of whether the latest version of these documents can be used by all parties who reach agreement according to this standard is encouraged. Any latest version of the non-dated reference is applicable to this standard.

GB/T 191	<i>Illustration and Logo for Packaging, Storage and Transportation</i>
GB/T 4789.2-2003	<i>Food Hygiene Microbiological Tests: Test for Aerobic Bacterial Count</i>
GB/T 4789.3-2003	<i>Food Hygiene Microbiological Tests: Test for Coliform</i>
GB/T 4789.4-2003	<i>Food Hygiene Microbiological Tests: Test for Salmonella</i>
GB/T 5009.11-2003	<i>Testing Method for Total Arsenic in Foods</i>
GB/T 5009.12-2003	<i>Testing Method for Lead in Foods</i>
GB/T 5009.17-2003	<i>Testing Method for Mercury in Foods</i>
GB/T 5009.19-2003	<i>Testing Method for BHC, DDT Pesticide Residues</i>
GB/T 5009.44-2003	<i>Analysis Method for Hygienic Standard of Meat and Meat Products</i>
GB/T 6388	<i>Transport Packaging and Shipping Mark</i>
GB 7718	<i>General Principle for Prepackaged Food Labels</i>
GB/T 14931.1-1994	<i>Determination Methods for Residues of Oxytetracycline, Tetracycline Aureomycin in Poultry Meat (HPLC-MS/MS)</i>
SN 0208-1993	<i>Determination Methods for Residues of Ten Different Types of Sulfanilamide in Pork Exports</i>
SN/T 0212.3-1993	<i>Determination Methods for Residues of Clopidol in Poultry Exports – Propionylation-Gas Chromatography</i>
SN 0672-1997	<i>Determination Methods for Residues of Diethylstilbestrol in Meat and Meat Products – Radioimmunoassay</i>
SN/T 0973-2000	<i>Test Method of Enterohemorrhagic Escherichia coli (EHEC) in Import and Export Meat</i>

and Meat Products (O157:H7)

3. Terms and Definition

The terms and definition will apply to this standard.

3.1 Fresh Poultry Product

Refers to chilled product that is a result of slaughtering, processing and precooling procedure of live poultry; including whole and eviscerated poultry, segmented portions of whole poultry (poultry meat, poultry wings, poultry drumsticks, etc.), poultry by-products (poultry head, neck, innards, feet (claws), etc.)

3.2 Frozen Poultry Product

Refers to product that is a result of slaughtering, processing and freezing procedure of live poultry; including whole and eviscerated poultry, segmented portions of whole poultry (poultry meat, poultry wings, poultry drumsticks, etc.), poultry by-products (poultry head, neck, innards, feet (claws), etc.)

3.3 Impurity

Refers to visible substances or contaminants, e.g. yellowish skin surface on poultry, poultry excrement taints, bile, other foreign bodies (plastics, metals, feed residues, etc.)

4. Technique Requirements

4.1 Raw Materials

Live poultry should originate from disease-free zones, and should pass the inspection and quarantine standard procedures before being slaughtered.

4.2 Processing

Slaughtered poultry should pass the corresponding inspection and quarantine standard procedures before being processed.

4.2.1 Cleaning

Individual parts of the poultry with external injuries, blood spots, blood taints, roots of feather and their likes should be removed or cut off.

4.2.2 Segmenting (Cutting)

Whole poultry should be precooled before segmenting (cutting into pieces); from bloodletting to packaging, time in the cold storage should not exceed 2 hours.

4.3 Freezing

Products that need to be frozen should ensure that the temperature of its core reaches -18°C or lower within a 12-hour timespan.

4.4 Sensory Conditions

Should comply with the requirements listed in Table 1.

Table 1

Items	Fresh Poultry Products	Frozen Poultry Products (Thawed)
Texture/Structure	Lean meat has elastic texture, part of meat immediately recovered to its original state after being pressed down with a finger	Part of meat recovered at a relatively slower rate after being pressed down with a finger, not likely to recover completely to its original state
Color and luster	Skin surface and lean meat slices possess lustrous appearance, possesses the color that such poultry specie should have	
Smell	Possesses the smell that such poultry specie should have, no unusual odor	
Soup of meat after heating/boiling	Transparent and clear in appearance, fats concentrate on the liquid's surface layer, possesses the taste/flavor that such poultry specie should have	
Extravasated blood [measured by its surface area, (S)] / cm^2 S>1 0.5<S≤1 S≤0.5	Should be detected Number of blood patches ≤2% of total sample quantity Disregarded	
Bristle (feather length >12mm or root of feather diameter >2mm) / (each feather/10kg) ≤	1	
Foreign bodies	Should not be detected	
Note: Surface area of extravasated blood patches refers to area of a single extravasated blood patch found on a single whole poultry or a single segmented portion of poultry.		

4.5 Physical-chemical Indexes

Fresh and frozen poultry products should comply with requirements listed in Table 2.

Table 2

Items		Index
Moisture loss of frozen poultry products during thawing / (%)		6
TNB-N / (mg/100g)		15
Mercury (Hg) / (mg/kg)		0.05
Lead (Pb) / (mg/kg)		0.2
Arsenic (As) / (mg/kg)		0.5
BHC / (mg/kg)	Fats composition <10%, BHC as fraction of total sample	0.1
	Fats composition ≥10%, BHC as fraction of total fats	1
DDT / (mg/kg)	Fats composition <10%, DDT as fraction of total sample	0.2
	Fats composition ≥10%, DDT as fraction of total fats	2
DICHLORVOS / (mg/kg)		0.05
Tetracycline / (mg/kg)	Meat	0.25
	Liver	0.3
	Kidney	0.6
Aureomycin / (mg/kg)		1
Oxytetracycline / (mg/kg)	Meat	0.1
	Liver	0.3
	Kidney	0.6
Sulfadimidine / (mg/kg)		0.1
Clopidol / (mg/kg)		0.01
Diethylstilbestrol / (mg/kg)		Should not be detected

4.6 Microbiological Index

Comply with requirements listed in Table 3.

Table 3

Items		Index	
		Fresh Poultry Products	Frozen Poultry Products
Aerobic Bacterial Count / (cfu/g)	≤	1×10 ⁴	5×10 ⁵
Coliform / (MPN/100g)	≤	1×10 ⁴	5×10 ⁵
Salmonella		0/25 g ^a	
EHEC (O157: H7)		0/25 g ^a	
^a 5 samples should be taken.			

5. Testing Methods

5.1 Sensory Conditions

Frozen poultry product should be thawed before inspection.

5.1.1 Texture/Structure, Color and Luster, Smell

Place all the samples that had been used for microbiological tests and inspection under natural light or its equivalent in a room dedicated for sensory inspection. Use sense of touch to evaluate the samples' texture/structure; use sense of sight to observe the color and luster; use sense of smell to identify the smell of the samples.

5.1.2 Soup of meat after heating/boiling

Shred the sample (prepared as in 6.5.2), weigh and take 20g of sample and place them into 200mL beaker. Add 100mL water and cover beaker with a piece of glass, heat up to 50~60°C. Remove the glass lid, use sense of smell to identify the smell of the heated soup. Continue heating till it boils, observe the condition of the soup, and the distribution of the fats within the liquid. Cool the soup back to room temperature, then taste the soup.

5.1.3 Extravasated Blood

Use appropriate means to measure the surface area of extravasated blood patches after completing the tests for texture/structure, color and luster and smell.

The proportion of extravasated blood patches (of surface area $0.5\text{cm}^2 < S \leq 1\text{cm}^2$) of the total quantity of products within a basic box of products can be calculated with formula (1) below:

$$X = \frac{A_1}{A} \times 100 \dots\dots\dots (1)$$

In the formula:

X – Proportion of extravasated blood patches (of surface area $0.5\text{cm}^2 < S \leq 1\text{cm}^2$) of the total quantity of products within a basic box of products (whole poultry measured by each whole poultry, poultry meat measure by each piece of meat, poultry drumstick or wing measure by each piece of drumstick or wing, and as such), %;

A – Total quantity of products in one basic box;

A_1 – Number of extravasated blood patches (of surface area $0.5\text{cm}^2 < S \leq 1\text{cm}^2$).

5.1.4 Bristles

Conduct test concurrently with those for texture/structure, color and luster and smell. Number of bristles in 10kg of products in a basic box can be computed according to formula (2) below, with the help of a pair of Vernier calipers:

$$X_1 = \frac{A_2}{m} \times 10 \dots\dots\dots (2)$$

In the formula:

X_1 – Number of bristles in 10kg of products in a basic box;

A_2 – Actual number of bristles in one basic box;

m – Actual weight of one basic box, unit as kg.

5.1.5 Foreign Bodies

Observe for foreign bodies by the sense of sight, concurrently with tests for texture/structure, color and luster and smell.

5.2 Moisture Loss during Thawing

5.2.1 Apparatus and Tools

Electric Balance: Precision of 1g;

Thermometer: Range of $-10\sim 50^{\circ}\text{C}$, division value of 0.5°C ;

Ceramic Plate/Dish, Metal Mesh.

5.2.2 Test Procedure

Place the metal mesh in the ceramic dish, maintaining a distance of 2cm between the mesh and the bottom of the ceramic dish. Extract 1,000~2,000g of the sample (prepared as in 6.5.2), weigh it with the electric balance and then place them on the metal mesh. Cover the extracted sample with a film of plastic and allow sample to naturally thaw at $15\sim 25^{\circ}\text{C}$. Once the core temperature of the sample increases to $2\sim 3^{\circ}\text{C}$, weigh it again with the electric balance. Place the sample again on the mesh for another 30mins, weigh again. Repeat the 30mins operation mentioned formerly until the difference between 2 consecutive measurements is not more than 2.0g.

5.2.3 Presentation of Test Results

The formula for computing moisture loss during thawing:

$$X_2 = \frac{m - m_1}{m} \times 100 \dots\dots\dots (3)$$

In the formula:

X_2 – Percentage of moisture lost during the thawing process, %;

m – Weight of frozen product before thawing, g;

m_1 – Weight of frozen product after thawing, g.

Result of calculation presented in the nearest whole number.

5.3 TVB-N

Refer to test method specified in 4.1 of GB/T 5009.44-2003.

5.4 Mercury

Refer to test method specified in GB/T 5009.17-2003.

5.5 Arsenic

Refer to test method specified in GB/T 5009.11-2003.

5.6 Lead

Refer to test method specified in GB/5009.12-2003.

5.7 BHC & DDT

Refer to test method specified in GB/5009.19-2003.

5.8 DICHLORVOS

Refer to test method specified in Appendix A of this standard.

5.9 Tetracycline, Chlortetracycline and Terramycin

Refer to test method specified in GB/T 14931.1-1994.

5.10 Sulfadimidine

Refer to test method specified in SN 0208-1003.

5.11 Clopidol

Refer to test method specified in SN/T 02012.3-103.

5.12 Diethylstilbestrol

Refer to test method specified in SN 0672-1997.

5.13 Total Aerobic Bacterial Count

Refer to test method specified in GB/T 4789.2-2003.

5.14 Coliform

Refer to test method specified in GB/T 4789.4-2003.

5.15 Salmonella

Refer to test method specified in GB/T 4789.4-2003.

5.16 EHEC

Refer to test method specified in SN/T 0973-2000.

5.17 Core Temperature of Product

5.17.1 Thermometer

Non-mercury glass thermometer or other apparatus for temperature measurement with a range of -20~50°C.

5.17.2 Procedure

Use a drill with diameter slightly longer than that of the thermometer to drill a hole to the center (core area) of the lean meat. Remove the drill, then immediately insert the non-mercury glass thermometer (or any other appropriate apparatus for temperature measurement) into the center of the meat, wait for readings to stabilize and then record the core temperature reading off the thermometer used.

6. Inspection Guidelines

6.1 Inspection Classification

6.1.1 Routine Inspection

6.1.1.1 Routine inspection should be carried out in any of following situation:

- a) Once-off submission of an isolated batch of products for inspection;
- b) Origin of production of live poultry changes;
- c) First batch of products processed at newly established plant/factory;
- d) Continuous processing operation for 6 months, or resume processing operations after operations had been shut down previously;
- e) Obvious discrepancies between inspection for delivery and the last routine inspection;
- f) As required by the relevant quality and hygiene supervisory organizations.

6.1.1.2 Routine inspection test items should include those test items specified in Table 1, Table 2 and Table 3.

6.1.2 Inspection for Delivery

6.1.2.1 All the product should undergo inspections for delivery before being shipped out from the factory of production.

6.1.2.2 Test items in inspection for delivery should include all test items listed in Table 1, as well as tests for moisture lost during thawing of frozen poultry products, TVB-N, the total aerobic bacterial count and coliform.

6.2 Batch

6.2.1 Continuous Batches

Products that are manufactured under the same processing conditions, derived from the same part of the poultry's body (e.g. whole poultry, poultry meat, wings, drumsticks, head, feet, innards), with the same

packaging and delivered together in a single occurrence will be classified as a single batch. Batch quantity will be measured by number of basic packaging boxes (thereinafter simply referred to as basic boxes.)

6.2.2 Isolated Batches

Products that are derived from the same part of the poultry's body (e.g. whole poultry, poultry meat, wings, drumsticks, head, feet, innards), with the same packaging and sent together for tests and inspection in a single occurrence will be classified as a single batch. Batch quantity will be measured by number basic boxes.

6.3 Sampling

6.3.1 Routine Inspection Sampling

Samples should be randomly drawn according to the batch quantity with reference to Table 4.

Table 4

Batch Quantity (in basic boxes)	Samples Quantity (in basic boxes)	Defect Quantity Allowed (in basic boxes)
600 or below	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
Above 42,000	200	19

6.3.2 Inspection for Delivery Sampling

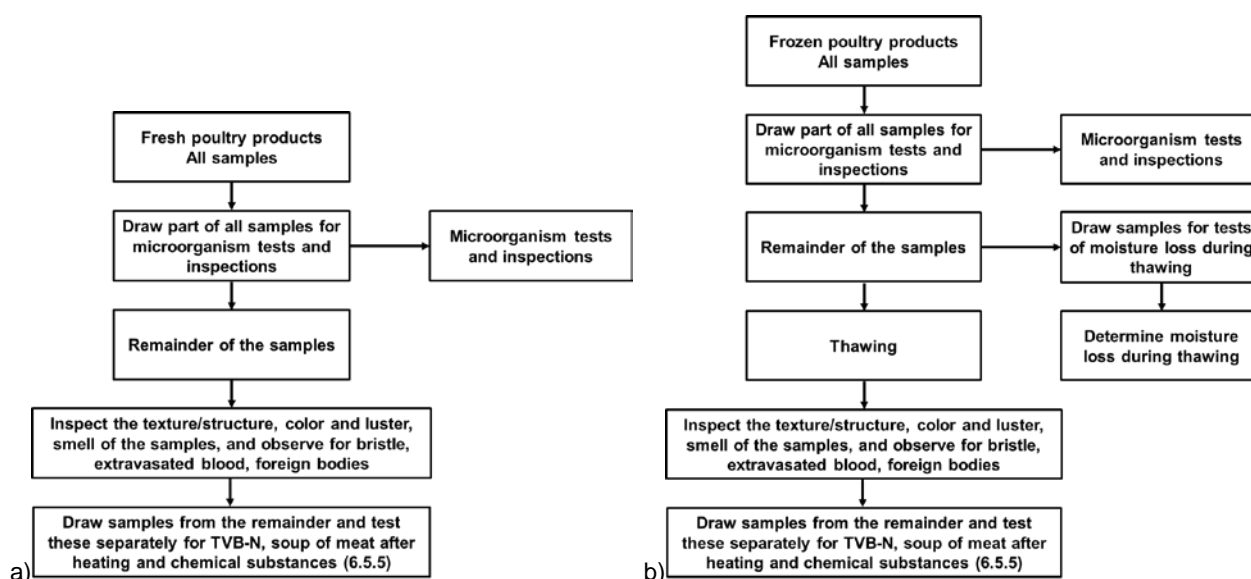
Samples should be randomly drawn according to the batch quantity with reference to Table 5.

Table 5

Batch Quantity (in basic boxes)	Samples Quantity (in basic boxes)	Defect Quantity Allowed (in basic boxes)
600 or below	6	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
Above 42,000	126	13

6.4 Sampling and Test Procedures

Fresh and frozen poultry products should be sampled and tested according to the process flow illustrated in Picture 1 below.



Picture 1 Fresh and Frozen Poultry Products Sampling and Inspection Procedures

6.5 Sampling Methods

The samples described in the following sections should not come with extravasated blood patches, bristles and foreign bodies.

6.5.1 Samples Drawn for Microorganism Tests

Randomly draw (3~5) basic boxes of products from all the samples drawn, extract approximately 100g of samples from each basic box under sterile conditions. Mix the extracted samples.

Note: Extract 5 portions (25g per portion) from the sample mixture for salmonella test samples; similarly extract another 5 portions (25g per portion) for EHEC test samples.

6.5.2 Samples for Tests of Moisture Loss during Thawing

Randomly draw (3~5) basic boxes of products from all the samples drawn, extract approximately 500g of samples from each basic box. Mix the extracted samples and then place the mixture in an insulated container.

6.5.3 Samples for TVB-N Tests

Randomly draw 3 basic boxes of products from all the samples drawn, extract approximately 100g of fatless and boneless samples from each basic box. Mix the extracted samples.

6.5.4 Samples for Preparation of Soup of Meat

Randomly draw 3 basic boxes of products from all the samples of whole poultry, poultry meat, wings or drumsticks drawn, extract 100g of meat from each basic box. Mix the extracted samples.

6.5.5 Test Samples for Chemical Substances (incl. 12 types of chemical listed in Table 2)

Randomly draw 3 basic boxes of products from all the samples drawn, extract approximately 200g of edible parts of the poultry from each basic box. Mix the extracted samples.

6.6 Decision Guidelines and Re-test/inspection

6.6.1 Defect Classifications

6.6.1.1 Typical Defects: Refer to number of extravasated blood patches and bristles did not fulfill the requirements in this standard.

6.6.1.2 Serious Defects: Refer to situations where results of the tests on texture/structure, color and luster, smell, soup of meat heated and individual items specified in Table 2 and Table 3 did not fulfill the requirements in this standard, and there are visible foreign objects observed.

6.6.2 Judgment of Individual Test Results

6.6.2.1 Judgment of test results of extravasated blood patches and bristles inspection: Base unit for the result of the extravasated blood patches and bristles inspection will be measured in units of single basic boxes.

Illustration 1:

Total samples are equivalent to 6 basic boxes of products, each numbered according to their respective order.

Inspection Results: Number of extravasated blood patches in Box No.1 and number of bristles in Box No.3 did not meet the requirements of the standard.

Judgment: Typical defects are found in 2 basic boxes of products.

Illustration 2:

Total samples are equivalent to 13 basic boxes of products, each numbered according to their respective order.

Inspection Results: Number of extravasated blood patches in all of the boxes, Box No.1~13 and the number of bristles in Box No.8 did not meet the requirements of the standards.

Judgment: Typical defects are found in 13 basic boxes of products.

6.6.2.2 Judgment of test results of texture/structure, color and luster, smell, soup of meat heated and the test items listed in Table 2 and 3: If any of the test results did not meet the requirements of this standard, all the samples drawn will be judged as having serious defects.

6.6.3 Judgment for Routine Inspection and Re-test/inspection

6.6.3.1 If test results of all the routine inspection items (6.1.1.2) all meet the requirements of this standard, then the whole batch of products will be judged as qualified.

6.6.3.2 If test results of routine inspection items (6.6.1.2) found to have one serious defect, then the whole batch of products will be judged as unqualified and re-test/inspection are not allowed.

6.6.3.3 If test results of routine inspection items (6.6.1.1) found to have general defects but number did not exceed the number allowed in Table 4, then the whole batch of products will be judged as qualified; however if number exceed the number allowed in Table 4, re-test/inspection can be conducted according to Table 4, then the re-test/inspection results can be compared again with Table 4 (Defect Quantity Allowed) to judge if

the whole batch of products qualify or do not qualify in each case.

6.6.4 Judgment for Inspection for Delivery and Re-test/inspection

6.6.4.1 If test results of all the inspection for delivery items (6.1.1.2) all meet the requirements of this standard, then the whole batch of products will be judged as qualified.

6.6.4.2 If test results of inspection for delivery items (6.6.1.2) found to have one serious defect, then the whole batch of products will be judged as unqualified and re-test/inspection are not allowed.

6.6.4.3 If test results of inspection for delivery items (6.6.1.1) found to have general defects but number did not exceed the number allowed in Table 4, then the whole batch of products will be judged as qualified; however if number exceed the number allowed in Table 4, re-test/inspection can be conducted according to Table 4, then the re-test/inspection results can be compared again with Table 4 (Defect Quantity Allowed) to judge if the whole batch of products qualify or do not qualify in each case.

7. Labeling, Logo, Packaging and Storage

7.1.1 Labeling

Label provided directly to consumers should comply with the requirements listed in GB/7718.

7.1.2 Transport Packaging Logo

Transport packaging logo and shipping marks should comply with the requirements listed in GB/T 191 and GB/T 6388.

7.2 Packaging

Fresh and frozen poultry product should be packaged with new packaging materials that comply with relevant hygienic standards.

7.3 Storage

Frozen poultry products should be stored in frozen room of -18°C or lower in temperature, where temperature should not fluctuate above the range of 1°C overnight.

Appendix A

(Normative Appendix)

Determination of Amount of Multicomponent Residues of Organophosphorus Pesticides in Animal Food Products

The appendix applies to the determination of amount of residues of organophosphorus pesticides (methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion, ethion) in poultry, dairy and dairy products, egg and egg products.

The respective limits on amount of residues of each type of organophosphorus pesticides are as follows: methamidophos 5.7, dichlorvos 3.5, acephate 10.0, monocrotophos 12.0, dimethoate 2.6, disulfaton 1.2, parathion-methyl 2.6, fenitrothion 2.9, pirimiphos methyl 2.5, malathion 2.8, fenthion 2.1, parathion 2.6, ethion 1.7.

A.1 Summary of Method

Samples will undergo extraction, purification, enrichment, volume fixing, separation (separation using capillary chromatographic column) and then they will be tested using a flame photometric detector, so as to ensure that the results are time-consistent and standardized externally.

Order of peaks on diagram of test results: methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion, ethion

A.2 Reagent

Reagents used throughout this test are all analytically pure reagents unless otherwise regulated; water used in the experiments should comply with the requirements on grade 2 water listed in the standard GB/T 6682.

A.2.1 Acetone: Double distillation.

A.2.2 Dichloromethane: Double distillation.

A.2.3 Ethyl Acetate: Double distillation.

A.2.4 Cyclohexane: Double distillation.

A.2.5 Sodium chloride.

A.2.6 Anhydrous sodium sulfate.

A.2.7 Gel: Bio-Beads S-X3 (or gel equivalent to Bio-Beads S-X3); 200~400 pieces.

A.2.8 Standard organophosphorus pesticide: Purity of methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion, ethion should be no less than 90%.

A.2.9 Solution of organophosphorus pesticide

A.2.9.1 Standard stock solution of monomer organophosphorus pesticides: Accurately weigh and extract 0.0100g of each of the organophosphorus pesticides, place each extract into 25ml volumetric flask, and dissolve the extracts with ethyl acetate to a constant volume (maintaining that solution concentration is 400ug/ml).

A.2.9.2 Mixture solution of organophosphorus pesticides for standard application: Before the determination tests, weigh and extract each standard stock solution of monomer organophosphorus pesticides of different volume (A.2.9.1) into 10ml volumetric flask, channel nitrogen gas into the solutions. Perform extraction according to methods in A.5.1.3 and A.5.2, then dilute with purified fresh milk extracts and fixed to certain volume. The concentration ($\mu\text{g/ml}$) of mixture solutions of individual organophosphorus pesticides should be as follows: methamidophos 16, dichlorvos 80, acephate 24, monocrotophos 80, dimethoate 16, disulfaton 24, parathion-methyl 16, fenitrothion 16, pirimiphos methyl 16, malathion 16, fenthion 24, parathion 16, ethion 8.

Note: Standard stock solution and mixture solution for standard application are only need to be prepared for dichlorvos if it was only meant to test for dichlorvos.

A3 Apparatus

A.3.1 Gas Chromatograph: Come with a flame photometric detector and capillary chromatographic columns

A.3.2 Rotatory Evaporator

A.3.3 Gel purification column: the height is 30cm, inner diameter is 2.5cm, has piston glass layer column, glass wool at the bottom of column, soak the gel with ethyl acetate and cyclohexane eluent (1:1), pour them into column with wet process, the height of column is about 26cm, the gel should be always in the eluent.

A.4 Preparation of Samples

A.4.1 Egg and egg products: Deshelled and then prepared as homogenates.

A.4.2 Meat and meat products: Remove the tendons and bones, cut into small pieces (chunks), and then prepared as meat paste.

A.4.3 Milk and milk products: Mix uniformly.

A.5 Analysis Procedure

A.5.1 Extraction, Distribution and Enrichment

A5.1.1 Egg and egg products: Weigh and extract 20g (0.01g precision) and place extract into 100mL conical flask with a stopper. Add 5mL water (add water correspondingly to the water content of each extract, ensuring total water within solution maintains at ~20g; typically water content in egg ~75%, thus adding 5mL should suffice), 40mL acetone and then shake for 30 mins. Add 6g sodium chloride, mix uniformly then add 30mL dichloromethane and shake for another 30mins. Extract 35mL supernatant, filter anhydrous sodium sulfate into a rotary evaporation bottle, and enrich the content till it reaches 1mL. Add 2mL solution of ethyl acetate cyclohexane (1:1) and repeat enrichment process. Repeat this process 3 times until it reaches 1mL.

A.5.1.2 Meat and meat products: Weigh and extract 20g (0.01g precision) and place extract into 100mL conical flask with a stopper. Add 5mL water (add water correspondingly to the water content of each extract, ensuring total water within solution maintains at ~20g; typically water content in meat ~70%, thus adding 6mL should suffice). Thereafter, process similar to A.5.1.1.

A.5.1.3 Milk and milk products: Weigh and extract 20g (0.01g precision) and place extract into 100mL conical flask with a stopper (adding water is not required for fresh milk, extract directly with acetone will suffice). Thereafter, process similar to A.5.1.1.

A.5.2 Purification

Elute the prepared solution concentrate (A.5.1) with solution of ethyl acetate cyclohexane (1:1) through the use of gel purification columns. Collect 35mL~70mL of distillates and enrich the distillates with the rotatory evaporator till it reaches 1mL. Purify again with the gel purification columns, collect another 35mL~70mL of distillates and enrich the distillates with the rotatory evaporator till it reaches 1mL. Transfer it into test tube with 5mL graduation markings and then elute with solution of ethyl acetate cyclohexane multiple times through the use of gel purification columns, transferring the result of the process into the same test tube. Channel nitrogen gas into the solution till it reaches below 1mL in volume, add solution of ethyl acetate cyclohexane till it reaches 1mL and set it aside for chromatography analysis.

A.5.3 Conditions of Chromatographic Analysis

A.5.3.1 Chromatographic columns: Elastic capillary columns made of quartz, with inner column diameter is 0.32mm, the length is 30m; coated with SE-54, with thickness at 0.25µm

A.5.3.2 Column temperature: Programmed temperature changes

60°C / 1 min $\xrightarrow{40^{\circ}\text{C/min}}$ 110°C $\xrightarrow{5^{\circ}\text{C/min}}$ 235°C $\xrightarrow{40^{\circ}\text{C/min}}$ 265°C

A.5.3.3 Temperature at Sample's Entry Point: 270°C.

A.5.3.4 Detector: Flame Photometric Detector (FPD-P), temperature at 270°C.

A.5.3.5 Carrier gas: Nitrogen gas, rate of flow at 1mL/min, make-up gas at 50mL/min.

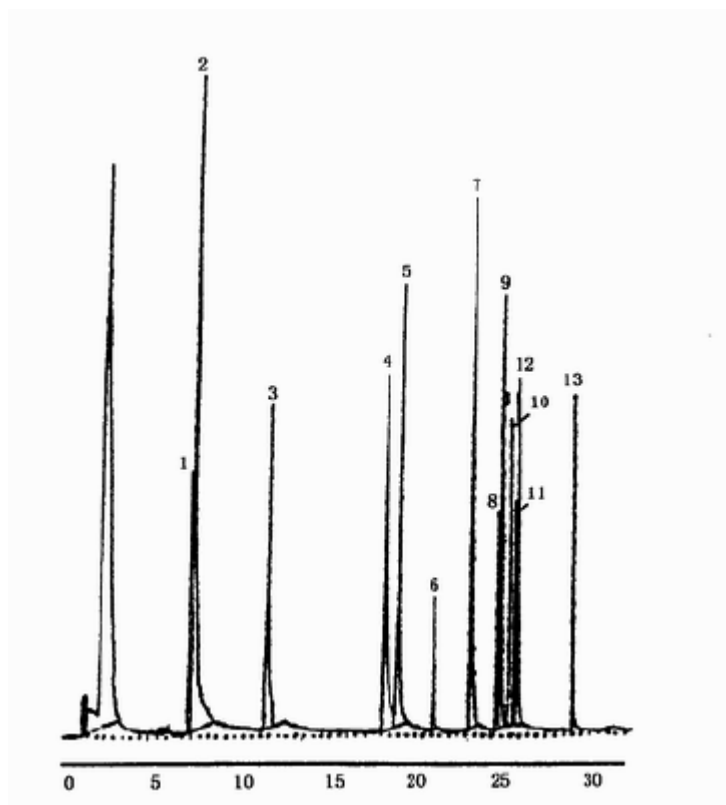
A.5.3.6 Rate of flow of hydrogen and air: Hydrogen at 50mL/min, air at 500mL/min.

A.5.4 Determination

Measure and extract 1µL of mixture solution of organophosphorus pesticides for standard application (A.2.9.2) and purified sample solution (A.5.2) respectively into the chromatographic instrument. To ensure time-consistency, perform quantitative comparisons of both the peak and peak area between the sample solution and standard application solution.

A.5.5 Chromatogram of 13 Types of Organophosphorus Pesticides

See Picture A.1 below for the chromatography diagram for 13 different types of organophosphorus pesticides.



- 1 – methamidophos;
- 2 – dichlorvos;
- 3 – acephate;
- 4 – monocrotophos;
- 5 – dimethoate;
- 6 – disulfaton;
- 7 – parathion-methyl;
- 8 – fenitrothion;
- 9 – pirimiphos methyl,
- 10 – malathion;
- 11 – fenthion;
- 12 – parathion;
- 13 – ethion.

Picture A.1 Chromatography Diagram for 13 Different Types of Organophosphorus Pesticides

A.6 Representation of Analysis Results

Amount of residual of organophosphorus pesticides within sample can be calculated by the following formula (A.1):

$$X = \frac{m_1 \times V_2 \times 1,000}{m \times V_1 \times 1,000} = \frac{m_1 \times V_2}{m \times V_1} \dots\dots\dots (A.1)$$

In the formula:

X – Amount of residual of organophosphorus pesticides within sample, unit in mg/kg;

M – Weight of sample, unit in g;

m₁ – Content of organophosphorus pesticide in test solution, unit in ng;

V₁ – Volume of samples introduced, unit in µL;

V₂ – Final fixed volume of test solution, mL.

A.7 Tolerance for Errors

Determination error during two test iterations should not exceed 20% of the average value of the results of the two iterations.

A.8 Precision

Precision is presented with recovery rate.

If required, add standard application solution (A.2.9.2) into poultry (livestock), egg or milk for tests on recovery rate, range of results should lie within 70%~100%.

Recovery rate can be calculated by the following formula (A.2):

$$Y = \frac{m_1 - m_2}{m} \times 100 \dots\dots\dots (A.2)$$

In the formula:

Y – Recovery rate, %;

m₁ – Detectable amount after adding standard application solution in sample;

m₂ – Content of certain component in sample;

m – Amount of certain component added.