

Appendix A2: National Standards – Multiple Food Categories

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National Standards Applicable to Multiple Food Categories

Food Labeling

GB 7718-2011 General Principle for Prepackaged Food Label



National Standards of People's Republic of China

GB 7718-2011

National Food Safety Standard

General Principle for Prepackaged Food Label

Issued on: 2011-04-20

Implemented on: 2012-04-20

Issued by National Health and Family Planning Commission

Foreword

The standard replaces GB 7718-2004 General Principle for Prepackaged Food Label.

As compared to the standard GB7718-2004, key changes are as follows:

- Amended the scope that the standard is applicable to;
- Definitions for prepackaged food and production date were amended, definition for specifications was added, while definition for shelf-life was removed;
- Amended the format for presentation of food additive information;
- Added a section specifying the format for presentation of specification information;
- Amended the format for presentation of manufacturer name, distributor name and their corresponding address and contact information;
- Amended the largest surface area for product packaging or container used when the length of fonts, symbols and numbers used for indicating mandatory label contents is 1.8mm and above;
- Added the requirements on the format of presentation for the recommendation labels indicating presence of potential allergens to consumers;
- Amended the calculation methodology used for computing largest surface area in Appendix A;
- Added Appendix B and Appendix C.

National Standard for Food Safety

General Principle for Prepackaged Food Label

1. Scope

This standard applies to the labels of prepackaged food that are sold/distributed directly and indirectly to the consumers.

This standard does not apply to the labels used on storage & transportation packaging dedicated towards protecting prepackaged food products during storage and the transportation process, labels for bulk food products and labels for food products made and sold on-site.

2. Terms and Definition

2.1 Prepackaged Food

Refers to food that are prepackaged or manufactured in specified fixed amounts, with packaging materials and/or within containers. These include food products consistent in quality and size, maintaining discrepancies within a limited range specified, that are prepackaged and pre-manufactured with packaging materials and/or within containers.

2.2 Food Label

Includes all materials of description, such as words/fonts, pictures/patterns, symbols/numbers.

2.3 Ingredients

Refer to any substance that is present (including substances present in a modified manner) in the food, added and used during food production and processing, including food additives.

2.4 Date of Production (Date of Manufacture)

Refers to the date when food is transformed into its final product format, i.e. the date that the food is packaged or filled into the packaging or container, becoming the final product ready for sales/distribution will constitute the date of production.

2.5 Shelf-life

Refers to the time period that the prepackaged food product maintains quality suitable for consumption, when stored under the recommended storage conditions indicated on its label. Products are suitable for sales/distribution within this stipulated period, and they possesses the unique characteristics that are not required to be indicated explicitly or already indicated in the product labels.

2.6 Specifications

Refer to statements that indicate the net content and the number of smaller packages include in the bigger packaging under the circumstance that the product packaging contains multiple smaller packaging within.

2.7 Main Display Panel

Refers to the panel for labels that is positioned on prepackaged food product packaging or container in an easily observable manner.

3. Basic Requirements

3.1 The label should comply with the provisions of laws and regulations as well as the requirements of corresponding food safety standards.

3.2 The label should be clear, eye-catching, durable, and easy for the consumers to identify and read during the purchasing process.

3.3 The label should be plain, easy to understand, and its content should be scientifically supported. The label should not contain any superstitious, erotic content nor make any attempt to play down on other food products or display any content contrary to general knowledge on nutritional science.

3.4 The label content should be factual and accurate, where false and exaggerated claims should not be used. The label should not contain any deceptive or easily misunderstood messages in manners such as words, patterns, to describe the food products to the consumers. It should not leverage on size difference or color contrasts of fonts to mislead consumers as well.

3.5 The label should not mislead consumers into confusing product or certain aspect of a product that they intend to buy with another product, with the use of direct or suggestive language, words, patterns and symbols.

3.6 The label content should not indicate or suggest that the product has the ability to prevent or cure diseases. Non-health food product label should not indicate or suggest that the product has specific health boosting effect.

3.7 The label should not be separated from the food or its product packaging (container).

3.8 The label should use standard Chinese characters (with the exception of its trademark). Any wordart used meant for decorative purposes should be properly inscribed and easily identifiable.

3.8.1 Pinyin (phonetic illustration of Chinese characters) or characters used by ethnic minorities can be used with Chinese characters, but pinyin used should be smaller than the corresponding Chinese characters used in the label.

3.8.2 Foreign language can be used, but foreign language used must have corresponding relationship with the Chinese characters on the label (with the exception of trademark, name and address of manufacturer of imported food, name and address of foreign distributor, webpage address.) All foreign characters used should be smaller than the corresponding Chinese characters used in the label.

3.9 The length of character, symbol and number used in the mandatory label contents should not be less than 1.8mm when the largest surface area (see Appendix A for the calculation methodology for computing largest surface area) of prepackaged food packaging or container is larger than 35 cm².

3.10 Under the circumstances that the packaging of a single sales unit contains multiple smaller individual packages of products of multiple varieties that can be sold independently, separate food labels should be included for each of the smaller individual packages.

3.11 Under the circumstances that the outer packaging can be opened easily or it is possible to clearly identify all or part of the mandatory label contents of the inner packaging (container) through the outer packaging, corresponding duplicated contents can be excluded from the outer packaging; otherwise, outer packaging should include all mandatory label contents stipulated in this standard.

4. Label Content

4.1 Food Label Content of Prepackaged Food Directly Supplied to Consumers

4.1.1 Basic Requirements

Food label of prepackaged food directly supplied to consumers should include required contents such as food name, ingredient lists, net content and specifications, name, address and contact information of manufacturer and (or) distributor, date of production, shelf-life, storage conditions, serial number of license for food production, product standards code.

4.1.2 Food Name

4.1.2.1 Dedicated term that clearly represent and reflect the true characteristics of the food product should be placed at a striking position on the food label.

4.1.2.1.1 Under the circumstances that national, commercial or local standards had designated one or multiple names as dedicated name(s) for a specific food product, one of these names or its equivalent should be used.

4.1.2.1.2 Common or popular names that do not cause any misunderstanding nor confusion for consumers should be used under the scenario that the choice of name of such products are not regulation by national, commercial or local standards.

4.1.2.2 Names with format specified in 4.1.2.1 should be included on the same main display panel with the displayed name, if one of the following types of displayed names is used: completely new and unknown names, strange/unique names, transliterated names, branded names, names based on regional slang and trademarked names.

4.1.2.2.1 Dedicated name that reflects the true nature of the food product should be shown with the same font as and at a position on the main display panel neighboring the displayed name, when the displayed name – in the form of a completely new and unknown name, strange/unique name, transliterated name, branded name, name based on regional slang or trademarked name, uses words or jargons (phrases) that potentially cause confusion for people.

4.1.2.2.2 Consistent font and color should be used for displayed name to reflect the true nature of the food product, when inconsistent fonts or colors cause people to misunderstand the true nature of food products despite the displayed name actually truly reflect the nature itself.

4.1.2.3 Corresponding words or phrases can be added before or after the dedicated food name for the purpose of preventing misunderstanding or confusion of the true nature, physical state or manufactured methodology of the food product. Examples include terms like “dried”, “concentrated”, “reconstituted”, “smoked”, “fried”, “powdered”, “grain”, etc.

4.1.3 List of Ingredients

4.1.3.1 A list of ingredients should be shown on the prepackaged food product label, where each ingredient listed in the list of ingredients should be labeled in accordance with the requirements of 4.1.2, while food additives should be labeled in accordance with the requirements of 4.1.3.1.4.

4.1.3.1.1 “Ingredients” or “List of Ingredients” (in Chinese) should be used as term of guidance to lead consumer to the actual list of ingredients. When all the raw ingredients used in the processing have changed into other ingredients (in the case of fermented products, e.g. alcohols, soya sauce, edible vinegar), “Raw Ingredients” or “Raw and Supplementary Ingredients” can replace “Ingredients”, “List of Ingredients” as terms of guidance, while complying with the requirements of the corresponding clauses in this standard in labeling the individual raw, supplementary ingredients and food additives. Processing aids are not required to be explicitly listed in the list of ingredients.

4.1.3.1.2 Ingredients should be listed item-by-item in a descending order based on the individual amount added in the product’s manufacturing or processing procedure; ingredients that do not constitute more than 2% of the total product composition can be listed in a random format without following a typical descending order required for other ingredients.

4.1.3.1.3 Compound ingredient made up of 2 or more base ingredients (not inclusive of food additives) should be listed in the list of ingredients with the specific name of the compound ingredient, with its base ingredients listed within parentheses thereafter in descending order based on the amount added. Under the circumstance that the compound ingredient is regulated by national, commercial or local standards and at the same time, it constitutes less than 25% of the total product composition, the act of listing its base ingredients is not required.

4.1.3.1.4 Food additives should be labeled with the specific common name stipulated in the standard GB 2760. Food additives can be labeled either in the format of only its specific name, or in the format of its functional category along with its specific name or International Numbering System (INS) code (See Appendix B for label formats.) One of the label formats shown in Appendix B should be chosen to label food additives on the same prepackaged food label. Under the circumstance that a certain food additive does not have a corresponding INS code or due to labeling requirements for allergens, specific name should be used in favor of INS code along with the food additive’s functional category when the functional category format abovementioned is adopted. Name of food additive does not include its method of production. Food additive used in any compound ingredient constituting less than 25% of total product composition, need not be listed if it fulfills GB 2760’s inductive principle and does not have any impact on the processing of the final product.

4.1.3.1.5 Any water added during food’s manufacturing or processing procedure should be labeled in the list of ingredients. Water or any other ingredients that evaporated during the processing procedure need not be listed.

4.1.3.1.6 Edible packaging or wrapping should also have its raw ingredients listed in the list of ingredients, unless otherwise specified in any other national laws or regulations.

4.1.3.2 Formats of presentation listed in Table 1 can be chosen for the following food ingredients.

Table 1 Formats of Presentation for List of Ingredients

Ingredient Category	Format of Presentation
Any vegetable oil, refined vegetable oil, except for olive oil	"vegetable oil" or "refined vegetable oil"; if the oil is hydrogenated, the oil should be labeled as "hydrogenated" or "partially hydrogenated"
Any starch, except for the chemically modified starch	"starch"
Any spice or spice extract added that constitutes less than 2% of total product composition (individual or total amount)	"spice" "spice category" or "compound spice"
Any formula of gum-based substances used for gum-based candies/sweets	"gum-based agent" "gum-based"
Any preserved, confiture fruits added that constitutes less than 10% of total product composition	"confiture fruit", "preserved fruit"
Edible essence, food fragrance	"edible essence", "food fragrance" "food essence and fragrance"

4.1.4 Labels for Quantity of Ingredients

4.1.4.1 If the food label or food manual specially emphasized that the food is supplemented with or the food contains one or more types of valuable, unique ingredients or components, the amount added or the specific content composition of the emphasized ingredient or component should be indicated clearly on the list of ingredients.

4.1.4.2 If the food label or food manual specially emphasized that the composition of one or more types of ingredients or components is relatively low or insignificant (i.e. zero), the specific composition of the emphasized ingredient or component should be indicated clearly on the list of ingredients.

4.1.4.3 Any food ingredient or component that is mentioned in the food name and at the same time not specially emphasized on the on the corresponding food label, need not have the amount added or its composition labeled on the list of ingredients.

4.1.5 Net Content and Specifications

4.1.5.1 Representation of net content should be made up of the net content value, numbers and its authorized unit of measurement (See Appendix C for reference of format of representation.)

4.1.5.2 Net content of products bundled in packaging (container) should be represented in the following format, according to its authorized unit of measurement:

- Liquid food, use volume units of measurement, such as L or l, mL or ml, or use weight units of measurement such as g or kg
- Solid food, use weight units of measurement such as g or kg
- Semi-solid food or food of sticky nature, use weight units of measurement such as g or kg, or use volume units of measurement such as L or l, mL or ml

4.1.5.3 Net content unit of measurement should be represented according to Table 2.

Table 2 Formats of Representation for Unit of Measurement Used for Net Content

Measurement Format	Range of Net Content Value (Q)	Unit of Measurement
Volume	$Q < 1,000\text{mL}$	mL, ml
	$Q \geq 1,000\text{mL}$	L, l
Weight	$Q < 1,000\text{g}$	g
	$Q \geq 1,000\text{g}$	kg

4.1.5.4 Minimum length of fonts used for representation of net content should comply with the requirements listed in Table 3.

Table 3 Minimum Length of Fonts Used for Net Content

Range of Net Content Value (Q)	Minimum Length of Font
$Q \leq 50\text{mL}; Q \leq 50\text{g}$	2
$50\text{mL} < Q \leq 200\text{mL}; 50\text{g} < Q \leq 200\text{g}$	3
$200\text{mL} < Q \leq 1\text{L}; 200\text{g} < Q \leq 1\text{kg}$	4
$Q > 1\text{kg}; Q > 1\text{L}$	6

4.1.5.5 Net content information should be positioned on the same display panel of the product packaging or container as its food name information.

4.1.5.6 If packaging container contains food substances in both liquid and solid states and the solid food substance within constitute the product's main ingredient, content of the food drained of liquid (i.e. the solid food substance) should be represented in mass or mass proportion format on top of the net content information (See Appendix C for reference of format of representation.)

4.1.5.7 Product specifications should be labeled along with net content information on the larger product packaging when such larger packaging format contains multiple smaller individual prepackaged food products.

4.1.5.8 Product specifications should be made of net content information and quantity of the individual prepackaged food products within the larger packaging, or in the format of just the quantity of smaller packages. "Specifications" need not be shown when presenting product specifications information. Specifications information of individual prepackaged food products is equivalent to the net content of the individual packages (See Appendix C for reference of format of representation.)

4.1.6 Name, Address and Contact Information of Manufacturer and Distributor

4.1.6.1 Name, address and contact information of manufacturer should be presented on the food label. Name and address information of the manufacturer should be the name and address that are legally registered and can be held responsible for the manufacturer's product safety quality standards. Such information should be presented in a format according to the specific requirements under the following scenarios.

4.1.6.1.1 Manufacturer with its independent legal personality, i.e. a group of companies or subsidiary of a group, should use its corresponding name and address on the product label.

4.1.6.1.2 Name and address of the overarching group of companies should be used together with those of the branch office (production base) on the product labels, if manufacturer do not have an independent legal personality, i.e. a branch office or production base of a group of companies; or simply indicating the name and address of the group, along with the production location will suffice, of which the production location should be presented in accordance with the administrative zone guidelines, up to the level of the municipality regions.

4.1.6.1.3 If the processing for the prepackaged food products is subcontracted to another party, name and address of both sides of the subcontracted process should be indicated; or simply indicating the name and address of the party subcontracting the process out, along with the production location will suffice, of which the production location should be presented in accordance with the administrative zone guidelines, up to the level of the municipality regions.

4.1.6.2 Contact information listed for manufacturer/distributor that has an independent legal personality should include at least one of the following content: telephone number, fax number, Internet contact information and their likes, or mailing address listed together with the registered address.

4.1.6.3 Imported prepackaged food products should be labeled with the country or region of origin (e.g. Hong Kong, Macau, Taiwan), and the name, address and contact information of the agent, importer and distributor that are properly registered in China according to relevant laws and regulations. Name, address and contact information of the manufacturer need not be indicated for import food products.

4.1.7 Presentation of Dates

4.1.7.1 Date of production and period of shelf-life of prepackaged food products should be clearly indicated. If “please see certain part of package for ...” format is used for presentation of dates, the date information should be positioned at the specific position mentioned. Date labels should not be separately covered over by another label, reprinted or tampered with (See Appendix C for reference of format of representation.)

4.1.7.2 When a single prepackaged product contains individual smaller prepackaged food products with individual date of production and shelf-life date labels, the earliest expiration date of all the individual packages should be adopted on the outer packaging. The earliest date of production of individual packages or the date when the outer package becomes a typical sales unit (i.e. all individual packages have been packaged into the outer package, ready for sale) will be adopted as the date of production labeled on the outer package; individual packages’ date of production and shelf-life can also be labeled separately on the outer packaging.

4.1.7.3 Date should be presented in the year, month, day order. If the presentation format does not follow the former date order, the specific date order used should be indicated as well (See Appendix C for reference of format of representation.)

4.1.8 Storage Conditions

Prepackaged food product label should include storage conditions (See Appendix C for reference of format of representation.)

4.1.9 Food Production License Number

Food production license number should be indicated on the prepackaged food product label according to format stipulated in relevant regulations.

4.1.10 Code of Product Standards

Prepackaged food products manufactured and sold/distributed in China (excluding imported prepackaged food products) should be labeled with code and serial number of the product standards enforced.

4.1.11 Other Contents

4.1.11.1 Irradiated Food

4.1.11.1.1 Food irradiated with ionization radiation or energy should be labeled with “Irradiated Food” in the proximity of the product name.

4.1.11.1.2 Any ingredient irradiated with ionization radiation or energy should be also be clearly indicated that it is processed in such a way in the list of ingredients.

4.1.11.2 Genetically Modified Food

Label of genetically modified food should comply with the requirements of relevant laws and regulations.

4.1.11.3 Nutrition Label

4.1.11.3.1 Special dietary food and staple, supplementary food dedicated for infants, should indicate the main nutritional contents and their corresponding composition. Labeling format should be in accordance to GB 13432.

4.1.11.3.2 If other types of prepackaged food requires the presentation of nutritional labels, the format of labeling should implemented with reference to relevant laws and regulations.

4.1.11.4 Quality Grade

If the grade of products is clearly defined and regulated in corresponding product standards, the quality grade that the products fall under should be indicated on the food labels.

4.2 Food Label Content of Prepackaged Food Indirectly Supplied to Consumers

Food label of prepackaged food indirectly supplied to consumers should comply with the corresponding requirements specified in clause 4.1, indicating food name, specifications, net content, date of production, shelf-life and storage conditions. Any of the contents not indicated on the label should be specified in the user manual or contract.

4.3 Label Content Exempted

4.3.1 The following prepackaged products can be exempted from indicating the shelf-life period (expiration period): alcoholic beverages with alcohol content $\geq 10\%$; edible vinegar; edible salt; solid sugar category; monosodium glutamate (MSG).

4.3.2 If the largest surface area of the packaging or container used for prepackaged food products is less than 10cm^2 (see Appendix A for the calculation methodology for computing largest surface area), only product name, net content, name and address of manufacturer (or distributor) are required to be indicated on the food label.

4.4 Recommend Content on Label

4.4.1 Batch Number

Batch number can be indicated depending on the requirements of the specific products.

4.4.2 Consumption Method(s)

Helpful instructions for consumers, such as packaging container's method of opening, method of consumption (eating), method of cooking, method of reliquefaction can be included on the food label, according to the requirements of the specific products.

4.4.3 Allergens

4.4.3.1 The following food and its products may cause allergic reaction, thus if they are used as ingredients, easily identifiable names should be used in the list of ingredients, or word of caution should be added near the list of ingredients:

- a) Contains gluten-based grains and its products (e.g. wheat, rye, barley, oats, spelt or hybrid species of a combination of the previously mentioned varieties);
- b) Crustaceans and its products (e.g. shrimp, lobster, crab);
- c) Fishes and its products;
- d) Egg category and its products;
- e) Peanuts and its products;
- f) Soybeans and its products;
- g) Dairy and dairy products (including milk candies);
- h) Nuts and its products.

4.4.3.2 If it is possible to include any of the abovementioned food or its products into the food processing procedure, word of caution should be added near the list of ingredients.

5. Others

Presentation of food labels of products that require special approval according to relevant national regulations should be implemented in accordance to the relevant regulations.

Appendix A

Calculation Methodology Adopted to Compute the Largest Surface Area of Product Packaging and Containers

A.1 Calculation Method for Rectangle-shaped Packaging or Container

Multiply the height (cm) and the width (cm) of the largest side of the rectangle-shaped packaging or container.

A.2 Calculation Method for Cylindrical Packaging or Container, or Almost Cylindrical Packaging or Container

Multiply the height (cm) and 40% of the circumference of the packaging or container.

A.3 Calculation Method for Packaging or Container of Any Other Shapes

Calculate 40% of the total surface area of the packaging or container.

Use the surface area of the main display panel as the largest surface area if such main display panel apparently exists on the product packaging or container.

When calculating the surface area of the packaging or its likes, subtract/eliminate the length/width dedicated for sealing the sides of the packaging from the calculation. Surface area calculation excludes shoulder region, neck region, top region and the convex part at the bottom for bottle or can shaped containers.

Appendix B

Presentation Formats for Food Additives in List of Ingredients

B.1 Specific Name of Food Additives Presented in Descending Order Based on Individual Amount Added

Ingredients: Water, Full cream milk powder, Light cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Phospholipid, Polyglycerol polyricinoleate, Food fragrance, Tartrazine), Glucose syrup, Propanediol fatty acid esters, Carrageenan, Guar gum, Annatto, Maltodextrin, Food essence.

B.2 Functional Category and INS Code of Food Additives Presented in Descending Order Based on Individual Amount Added

Ingredients: Water, Full cream milk powder, Light cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Emulsifier (332, 476), Food fragrance, Coloring agent (102)), Glucose syrup, Emulsifier (477), Thickener (407, 412), Coloring agent (106b), Maltodextrin, Food essence.

B.3 Functional Category and Specific Name of Food Additives Presented in Descending Order Based on Individual Amount Added

Ingredients: Water, Full cream milk powder, Light cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Emulsifier (Phospholipid, Polyglycerol polyricinoleate), Food fragrance, Coloring agent (Tartrazine), Glucose syrup, Emulsifier (Propanediol fatty acid esters), Thickener (Carrageenan, Guar gum), Coloring agent (Annatto), Maltodextrin, Food essence.

B.4 Establishing Consistent Presentation Format for Food Additive Group Parentheses

B.4.1 General Principle

Food additives added directly should be indicated in the food additive group parentheses. Nutritional supplements, food fragrance and essence, base agent and substance in gum-based candies/sweets can be listed outside the food additive group parentheses. Food additives added indirectly will not be indicated within the food additive group parentheses. The order of presentation of food additives within the list of ingredients should be decided based on the total weight of the specific food additives used in the product.

B.4.2 Presentation of All Food Additives in Their Specific Names

Ingredients: Water, Full cream milk powder, Light Cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Phospholipid, Polyglycerol polyricinoleate, Food fragrance, Tartrazine), Glucose syrup, Food additives (Propanediol fatty acid esters, Carrageenan, Guar gum, Annatto), Maltodextrin, Food essence.

B.4.3 Presentation of All Food Additives in Their Functional Category and INS Code

Ingredients: Water, Full cream milk powder, Light Cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Emulsifier (332, 476), Food fragrance, Coloring agent (102)), Glucose syrup, Food additives (Emulsifier (477), Thickener (407, 412), Coloring agent (106b)), Maltodextrin, Food essence.

B.4.4 Presentation of All Food Additives in Their Functional Category and Specific Names

Ingredients: Water, Full cream milk powder, Light Cream, Vegetable oil, Chocolate (Cocoa mass, White sugar, Cocoa butter, Emulsifier (Phospholipid, Polyglycerol polyricinoleate), Food fragrance, Coloring agent (Tartrazine), Glucose syrup, Emulsifier (Propanediol fatty acid esters), Thickener (Carrageenan, Guar gum), Coloring agent (Annatto), Maltodextrin, Food essence.

Appendix C

Recommended Presentation Formats for Some Label Components

C.1 Overview

This Appendix provides the recommended presentation formats for some of the components of the prepackaged food label through the use of examples and illustrations, despite that presentation format is not limited only to these recommended formats for the specific label components. If certain format modifications are required due to unique characteristics of food products or specific nature of the packaging, it is necessary to ensure that the modified format is still consistent with the fundamental meaning of the recommended formats.

C.2 Presentation of Net Content and Specifications

For convenience of illustration, examples for net content information use weight-based units of measurement, with colon symbol as separator. Food label should use the appropriate unit of measurement, and spacing or other symbols can be used as separator according to actual circumstances, so as to facilitate reading for consumers.

C.2.1 Net content (specifications) of single prepackaged food can be presented in the following formats:

Net content (or net content/specification): 450g;

Net content (or net content/specification): 225g (200g+25g complimentary);

Net content (or net content/specification): 200g+25 complimentary;

Net content (or net content/specification): (200+25) g.

C.2.2 Net content of drained product (solid mass) can be presented in the following formats (using example on semi-solid canned pear in syrup):

Net content (or net content/specification): 425g drained (or solid or pear chunks): no less than 225g (or no less than 60%).

C.2.3 When a single prepackaged food packaging contains multiple individual prepackaged food products of the same product category, net content and specifications can be presented in the following formats:

Net content (or net content/specification): 40g×5;

Net content (or net content/specification): 5×40g;

Net content (or net content/specification): 200g (5×40g);

Net content (or net content/specification): 200g (40g×5);

Net content (or net content/specification): 200g (5 pieces);

Net content: 200g Specification: 5×40g;

Net content: 200g Specification: 40g×5;

Net content: 200g Specification: 5 pieces;

Net content (or net content/specification): 200g (100g+50g×2);

Net content (or net content/specification): 200g (80g×2+40g);

Net content: 200g Specification: 100g+50g×2;

Net content: 200g Specification: 50g×2+40g.

C.2.4 When a single prepackaged food packaging contains multiple individual prepackaged food products of different product category, net content and specifications can be presented in the following formats:

Net content (or net content/specification): 200g (product A 40g×3, product B 40g×2)

Net content (or net content/specification): 200g (40g×3+40g×2)

Net content (or net content/specification): 100g product A, 50g×2 product B, 50g product C

Net content (or net content/specification): product A: 100g, product B 50g×2, product C 50g

Net content/specification: 100g (product A), 50g×2 (product B), 50g (product C)

Net content/specification: product A: 100g, product B 50g×2, product C 50g

C.3 Presentation of Dates

Numbers used for individual day, month and year in the dates listed can be separated by spacing, or symbols like slashes, hyphens, period, or not presented without any separator formerly mentioned. Generally, four-digit format is used for presentation of year, although two-digit format can be used for smaller packaging. Two-digit format is used for presentations of month and day.

Dates can be presented in the following formats:

2010.3.20;

2010 03 20; 2010/03/20; 20100320;

(mm/dd/yyyy): 03 20 2010; 03/20/2010; 02302010.

C.4 Presentation of Shelf-life (Expiration Date)

The shelf-life (expiration date) can be presented in following formats:

Eat (or drink) best before...; before... eat (or drink) is best;

Before the date of ... is best; eat (or drink) before the date of ... is best;

The expiration date is...; the expiration is ... months (or days, weeks, years).

C.5 Presentation of Storage Conditions

Storage conditions information can be titled as “Storage Conditions” “Holding Conditions” “Holding

Methods” or the information can be presented un-titled.

Storage conditions can be presented in the following formats:

Store at room temperature (or refrigerated, frozen, in the dark, at shady and dry locations);

Stored at xx - xx°C;

Please store the product at shady and dry locations;

Store at room temperature, but once opened, keep refrigerated;

Temperature: ≤xx°C, humidity: ≤xx%.

GB 28050-2011 Standard for Nutrition Labeling of Prepackaged Foods



National Standards of People's Republic of China

GB 28050-2011

National Food Safety Standard
Standard for Nutrition Labeling of Prepackaged Foods

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Issued by National Health and Family Planning Commission

National Standard for Food Safety

Standard for Nutrition Labeling of Prepackaged Foods

1. Scope

This standard applies to the description and presentation of nutrition information on nutrition labeling of a prepackaged food.

This standard does not apply for the nutrition labeling for health food nor pre-packaged food for special dietary uses.

2. Terms and Definitions

Nutrition labeling

Nutrition labeling is a description intended to inform the consumer of the nutritional information and the nutritional properties of a food, which includes nutrition components table, nutrition claims and nutrient function claims. Nutrition labeling is a part of food labeling.

a) Nutrients

Substances in food with specific physiological role to maintain the body's growth, development, activities, reproduction, and normal metabolism, which include protein, fat, carbohydrates, minerals and vitamin, etc.

b) Nutritional component

Nutritional component refers to nutrients or other component in food in addition to nutrients which have nutritional properties of a food, which includes nutrition components table, nutrition claims and nutrient function claims. Nutrition labeling is a part of food labeling.

c) Core Nutrient

Core nutrients in nutrition labelling include protein, fat, carbohydrates, and sodium, etc.

d) Nutritional Components table

A normative form with the name of nutrient component, the content of nutrient component and % NRV (Nutrient reference value).

e) Nutrient reference value (NRV)

Nutrient Reference Value (NRV) is a reference value especially for food nutrition labelling purpose, and to compare the content levels of nutrient components.

f) Nutrition claim

Nutrition claim refers to a description and declaration of the nutritional properties of a food, such as declaration of energy value and content claim of protein. Nutrition claim includes content claim and comparative claim.

i. Content claim

Content claim means a claim that describes the energy or the nutrient content level in a food. The diction for nutrient content claim includes "contains", "high", "low" or "no", etc.

ii. Comparative claim

Comparative claim means a claim that compares the energy value or the content level of a nutrient in a food with other foods of same type known by consumers. The diction includes "add", "reduce", etc.

g) Nutrient function claim

A claim that describes the role of a nutrient component in normal growth, development and normal physiological function of the body.

h) Rounding interval

Rounding interval is the minimum unit value of a rounded numerical value.

i) Edible parts

Edible parts of a food refer to the remaining parts that can be consumed after the removal of those non-edible parts of the net content of the prepackaged food.

3. General requirements

a) Any nutrition information presented on a nutrition label of a food should be truthful, subjective and not be in any deceptive, not to exaggerate the nutritional or other functions.

b) Nutrition label of the prepackaged food should be written in Chinese. If a foreign language is also adopted, its content should be in correspondence with which in Chinese. The foreign letters shall not larger than the corresponding Chinese characters.

c) The nutrition components of a food should be indicated in form of box table (except in exceptional circumstances) which can be any size and should be perpendicular to the baseline of the packaging. The title of the table should be Nutrition Components Table.

d) The content of a nutrition component in food should be indicated in a special value which may be obtained by calculation using that of raw materials or by product detection. The nutrient reference values (NRV) of the nutrient components are established in the Annex A.

e) The nutrition label formats are specified in the Annex B. The food companies can choose one of them according to the elements such as nutrition property, packaging dimension and shape, etc.

f) The nutrition label should be indicated in the packaging of the minimum sales unit offered to the consumers.

4. Mandatory labeling items

- a) Energy, core nutrients content value and percentage in the nutrient reference values (NRV) are mandatory labeling items on a nutrition label of prepackaging foods. When there are other nutritional components to be claimed, appropriate measures shall be taken to highlight the claims of energy and core nutrients.
- b) When other nutrient components besides energy and core nutrients should be made nutrition claim or nutrient function claim. In the Nutrient Components Table, this nutrient component content and the nutrient reference values (NRV) in which represents should be claimed.
- c) The prepackaged food in which the food nutrition enhancer has been used, in addition to the requirement of the 4.1, in the Nutrient Components Table, this nutrient component content and the nutrient reference values (NRV) in which represents after enhancement should be claimed.
- d) If the food ingredients content hydrogenated and/or partially hydrogenated fats and oils, or they have been used in the production, in the Nutrient Components Table, the content of Trans fat (acid) should be claimed.
- e) The nutrient components whose nutrient reference values (NRV) are not established in the above points only need to claim the content.

5. Optional labeling content

- a) In addition to the above mandatory label content, in the Nutrient Components Table, the other components in the Table 1 could be claimed optional.
- b) When the claimed value of one nutrient component meets the content requirement and the restrictive conditions in the Table C.1, the content claim of this component could be made and the claim form is showed in the Table C.1. When the content of one nutrient component meets the requirements and conditions in the Table C.3, the comparative claim of this component could be made and the claim form is showed in the Table C.3. When one nutrient component meets the requirements of content claim and comparative claim, the two claim forms could be used at the same time, or only use the content claim. The synonymous of the content claim and the comparative claim are in the Table C.2 and Table C.4.
- c) When the claimed value of one nutrient component meets the requirements and conditions of content claim or comparative claim, one or various standard expressions of function claim of the nutrient component in the Annex D could be used. No excision, adjunction and mergence in any form to the expressions of function claim should be made.

6. Nutrient components expression

- a) The content level of energy and nutrient components in prepackaged food shall be expressed in "amount per 100g" and (or) "amount per 100mL" and (or) "Specified numerical values of the edible part per serving". The quantity of per serving should be indicated if "Per serving" expression is adopted and the size of per serving can be defined in according to the features of the food.
- b) The name, regular succession, claim unit, rounding interval, definition of "0" of the nutrient component which need mandatory claim and optional claim in the Nutrient Components Table should be in compliance with the provisions listed in Table 1. When a nutrient component is not claimed, the other items move up in order to the sequence.

c) In addition to the Table 1, when the other nutrient components permitted to be enhanced according to the GB 14880 and the announcements of the Ministry of Health are claimed, they should be listed after the other nutrients in the Table 1.

Table 1: Name, Sequence, Expression units, Rounding interval and Definition of “0” for Energy and Nutritional Components

Name and order of energy/nutritional components	Labeled unit ^a	Rounding interval	Limit value of “0” (Per 100 g or 100ml) ^b
Energy	kJ	1	≤17 kJ
Protein	g	0.1	≤ 0.5 g
Fat	g	0.1	≤ 0.5 g
Saturated fat (fatty acid)	g	0.1	≤ 0.1 g
Trans fat (fatty acid)	g	0.1	≤ 0.3 g
Monounsaturated fat (fatty acid)	g	0.1	≤ 0.1 g
Polyunsaturated fat (fatty acid)	g	0.1	≤ 0.1 g
Cholesterol	mg	1	≤ 5 mg
Carbohydrate	g	0.1	≤ 0.5 g
Sugar (Lactose c)	g	0.1	≤ 0.5 g
Dietary fiber (or monomer of fiber or soluble dietary fiber or insoluble dietary fiber)	g	0.1	≤ 0.5 g
Sodium	mg	1	≤ 5 mg
Vitamin A	μgRE	1	≤ 8μgRE
Vitamin D	Mg	0.1	≤0.1μg
Vitamin E	mg α-TE	0.01	≤0.28mgα-TE
Vitamin K	μg	0.1	≤1.6μg
Vitamin B1	mg	0.01	≤0.03mg
Vitamin B2	mg	0.01	≤0.03mg
Vitamin B6	mg	0.01	≤0.03mg
Vitamin B12	μg	0.01	≤0.05μg
Vitamin C	mg	0.1	≤2.0mg
Nicotinic acid	mg	0.01	≤0.28mg
Folacin/Folic acid	μg or μg DFE	1	≤8μg
Pantothenic acid	mg	0.01	≤0.10mg
Biotin	μg	0.1	≤0.6μg
Choline	mg	0.1	≤9.0mg
Phosphorus	mg	1	≤14mg
Potassium	mg	1	≤20mg
Magnesium	mg	1	≤6mg
Calcium	mg	1	≤8mg
Iron	mg	0.1	≤0.3mg
Zinc	mg	0.01	≤0.30mg
Iodine	μg	0.1	≤3.0μg
Selenium	μg	0.1	≤1.0μg
Copper	mg	0.01	≤0.03mg
Fluorine	mg	0.01	≤0.02mg
Manganese	mg	0.01	≤0.06mg

a Labeled units can be in Chinese or English, or both.

b Define it “0”when content of a certain nutritional component is less than or equals to the limit value of “0”.The regulations on the limit value of “0” (per 100g or per 100ml) shall also be met when “per serving” expression is adopted.

c It can be directly indicated as “lactose” on a nutrition label of milk or milk product.

d) In the whole shelf life, the error range for content of energy and nutritional components shall be judged in according to the provisions listed in Table 2.

Table 2: Allowed error range for content of energy and nutritional components

Nutrients in foods	Allowed error range
Protein, Polyunsaturated fat (fatty acid), Monounsaturated fat (fatty acid), Carbohydrates, Sugars (only lactose), Total dietary fiber (soluble fibre, insoluble fibre), individual component of fibre Vitamins (other than Vitamin D, Vitamin A), Minerals (exclude Sodium), other nutrients enhanced in food	≥80% declared value
Energy, Fat, Saturated fat (fatty acid), Trans fat (fatty acids), Cholesterol, Sodium, Sugars (exclude lactose) in food	≤120% declared value
Vitamin D and Vitamin A in food	80% ~ 180% declared value

7. Prepackaged food exempted for the mandatory nutrient label

Prepackaged foods of following types are exempt from rules on nutrition labeling:

- Fresh food, such as packed raw meat, raw fish, raw vegetables and fruits, fresh eggs, etc;
- Alcohol beverages with greater than or equal to 0.5% of alcohol content;
- Packaged food with total surface area of no more than 100 cm² or the largest surfaces area of the package is no more than 20 cm²;
- Food sold on the site which is usual y bought for immediate consumption;
- Bottled drinking water;
- A prepackaged food that the daily intake amount shall be no more than 10g or 10ml.
- Those prepackaged foods which falling with the criteria of exemptions of food labels according to the laws, regulations and standards.

The exemption will be removed if a nutrition claim is made on prepackaged food listed above. And the nutrition labeling of this product should meet the requirements specified in this standard

Appendix A

Chinese Nutrient Reference Value (NRV) and Methods of Use

1. A.1 Nutrient reference value (NRV)

NRVs for energy and 32 types of specified nutrients are listed in Table A.1.

Table A.1. Nutrient Reference Value (NRV)

Nutrient Components	NRV	Nutrient component	NRV
Energy ^a	8400 kJ	Folic acid	400 µg DFE
Protein	60 g	Pantothenic acid	5 mg
Fat	≤60 g	biotin	30 µg
Saturated fatty acids	≤20 g	Choline	450 mg
Cholesterol	≤300 mg	Calcium	800 mg
Carbohydrate	300 g	Phosphorus	700 mg
Dietary fibre	25 g	Potassium	2000 mg
Vitamin A	800 µg RE	Sodium	2000 mg
Vitamin D	5 µg	Magnesium	300 mg
Vitamin E	14mg α-TE	Iron	15 mg
Vitamin K	80 µg	Zinc	15mg
Vitamin B1	1.4 mg	Iodine	150 µg
Vitamin B2	1.4 mg	Selenium	50 µg
Vitamin B6	1.4 mg	Copper	1.5 mg
Vitamin B12	2.4 µg	Fluorine	1 mg
Vitamin C	100 mg	Manganese	3 mg
Niacin	14 mg		
a 8400kJ of energy is equivalent to 2000 kcal of energy. The energy value contribution of the protein, fat and carbohydrate respectively is 13%, 27% and 60% of total energy.			

2. A.2 Using purpose and method

NRV is used to compare and describe energy level or the content level of nutrients. When nutrition claims and the definition of “0” are adopted for expression, NRV may be used as a standard reference value.

Express nutrient information in percentage of nutrient reference value (% NRV).

The appointed rounding interval for % NRV is 1, such as 1%, 5%, 16% , etc.

3. A.3 Calculation

Calculate NRV% for a nutrient using equation below (A.1):

$$\text{NRV \%} = X / \text{NRV} \times 100\% \dots\dots\dots (\text{A.1})$$

Where: X -- the content of a nutrient in food

NRV -- Nutrition reference value for this item

Appendix B

Format of nutrition label

B.1. This Appendix specifies the format of nutrition label of prepackaged food.

B.2. One of the following six formats of nutrition labels should be adopted.

B.2.1 Only labeling energy and core nutrients format

The nutrition label in which only the energy and core nutrients are claimed refers to the example 1.

Example 1:

Nutrition information Table

Item	Per 100g/100ml or per serving	Nutrient Reference Value % or NRV%
Energy	kJ	%
Protein	g	%
Fat	g	%
Carbohydrate	g	%
Sodium	mg	%

B.2.2 More nutrition composition

The nutrition label with more information refers to the example 2.

Example 2:

Nutrition Information Table

Item	Per 100g/100ml or per serving	Nutrient Reference Value % or NRV%
Energy	kJ	%
Protein	g	%
Fat	g	%
--Saturated fat	g	%
Cholesterol	mg	%
Carbohydrate	g	%
Sugar	g	%
Dietary fiber	g	%
Sodium	mg	%
Vitamin A	μg RE (retinol equivalent)	
Calcium	mg	

Note: the core nutrient should label in appropriate format to make it striking.

B.2.3 Format with foreign language

The nutrition label with foreign language refers to the example 3.

Example 3:

Nutrition Information

Items	100 g or 100 ml per 100g/100ml or per serving	NRV%
Energy	kJ	%
Protein	g	%
Fats	g	%
Carbohydrate	g	%
Sodium	mg	%

B.2.4 Horizontal format

The nutrition label in horizontal format refers to example 4.

Example 4:

Nutrition Information Table

Item	per 100g/100ml or per serving	Nutrient Reference Value % or NRV%	Item	per 100g/100ml or per serving	Nutrient Reference Value % or NRV%
Energy	kJ	%	Protein	g	%
Carbohydrate	g	%	Fat	g	%
Sodium	g	%	--	--	%

* According to the packing characteristics, nutrients can be arranged horizontally from left to right, dividing into two rows or more.

B.2.5 Characters format

For foods whose total area of package less than 100cm², when labeling the nutrition information, the NRV can be omitted. Nutrients can be arranged horizontally from left to right, or vertically up to down according to the packing characteristics. For example:

Example 5:

Nutrition Information /100g: Energy XX kJ, Protein XX g, Fat XX g, Carbohydrate XX g, Sodium XX mg.

B.2.6 Format with nutrition claim and/or nutrition function claim

Nutrition claim and/or nutrition function claim refers to example 6.

Example 6:

Nutrition Information

Item	per 100g/100ml or per serving	Nutrient Reference Value % or NRV%
Energy	kJ	%
Protein	g	%
Fat	g	%
Carbohydrate	g	%
Sodium	mg	%

Nutrition Claim. Such as: Low fat XX

Nutrition Function Claim. Such as: Energy from fat should not exceed 30% of total energy for daily diet.

Nutrition claim and/or nutrition function claim can be labeled on anywhere of the label, but the font size should not exceed the food name and the trademark.

Appendix C

Requirements, Conditions and Synonyms for Energy and Nutrient Content Claim and Comparison Claim

C.1 The Table C.1 provides for requirements and conditions for nutrient content claim and comparative claim of emerge and nutritional components.

C.2 Table C.2 provides for synonyms for nutrient content claim of energy and nutritional components.

C.3 Table C.3 provides for requirements and conditions for nutrient comparative claim of energy and nutritional components.

C.4 Table C.4 provides for synonyms for nutrient comparative claim of energy and nutritional components.

Table C.1 Requirements and conditions for nutrient content claim and comparative claim of emerge and nutritional components

Item	Content Claim Mode	Content demand ^a	Restriction
Energy	No energy	≤17 kJ/100g (solid) or 100ml (Liquid)	Energy from fat ≤50% of total energy
	Low energy	≤170 kJ/100g solid ≤80 kJ/100ml liquid	
Protein	Low protein	Energy from protein ≤ 5% of total energy	Total energy refers to per 100g/ml or per serving
	Origin of protein, or include protein	Content /100 g ≥10% NRV Content /100 ml ≥5% NRV or Content /420 kJ ≥5% NRV	
	High, or rich in protein	Content /100 g ≥20%NRV Content /100 ml ≥10%NRV or Content /420 kJ ≥10%NRV	
Fat	No fat or not including fat	≤0.5 g/100g (solid) or 100ml (liquid)	
	Low fat	≤3 g/100g solid; ≤1.5g/100ml liquid	
	Lean	Fat content ≤10%	Refer to livestock and poultry only
	Skim	Liquid milk and yoghurt: fat ≤0.5%, Milk powder: fat ≤1.5%	Refer to dairy products only.
	No or not including saturated fat	≤0.1 g/ 100g(solid) or 100ml (liquid)	Refer to sum of saturated fat and trans fat
	Low saturated fat	≤1.5 g/100g solid ≤0.75 g /100mL liquid	1. Refer to sum of saturated fat and trans fat 2. energy from saturated fat no more than 10% of total
	No or not including transfat	≤0.3 g/100g (solid) or 100ml (liquid)	
Cholesterol	No or not including cholesterol	≤5 mg/100g (solid) or 100ml (liquid)	Should comply with both demand and restriction for low saturated fat claim
	Low cholesterol	≤20m g /100g solid; ≤10m g /100ml liquid	

Table C.1 (Continued)

Item	Content Claim Mode	Content demand ^a	Restriction
Carbohydrate (Sugar)	Sugar free or sugar excluded	≤ 0.5 g /100g (solid) or 100ml (liquid)	Refer to dairy products only
	Low sugar	≤ 5 g /100g (solid) or 100ml (liquid)	
	Low lactose	Lactose ≤ 2 g/100g (ml)	
	No lactose	Lactose ≤ 0.5 g/100g (ml)	
Dietary fiber	Origin of dietary fiber or including dietary fiber	≥3 g/ 100g (solid) ≥1.5 g/ 100ml (liquid) or ≥1.5 g/ 420 k	Total content of dietary fiber should comply with the demand; or at least one of soluble dietary fiber, insoluble dietary fiber and monomer comply with the demand
	High or rich in dietary fiber or good origin	≥6 g/ 100g (solid) ≥3 g/ 100ml (liquid) or ≥3 g/ 420 kj	
Sodium	No sodium or not including sodium	≤5 mg /100g or 100ml	The “sodium” can be replaced by “salt”, such as “low salt”, “salt reduced”, etc
	Very low sodium	≤40 mg /100g or 100ml	
	Low sodium	≤120 mg /100g or 100ml	
Vitamin	Origin of vitamin X or including vitamin X	/100 g ≥15% NRV /100 ml ≥7.5% NRV or /420 kJ ≥5% NR	Including “multivitamins” refer to 3 or more vitamins, complying with the “including” demand
	High or rich in vitamin X	/100 g ≥ 30% NRV /100 ml ≥15% NRV or /420 kJ ≥ 10% NRV	Rich in “multivitamins” refer to 3 or more vitamins, complying with the “rich in” demand
Mineral	Origin of X or including X	/100 g ≥15% NRV /100 ml ≥7.5% NRV or /420 kJ ≥5% NRV	Including “multi-minerals” refer to 3 or more minerals, complying with the “including” demand
	High or rich in X	/100 g ≥ 30% NRV /100 ml ≥15% NRV or /420 kJ ≥ 10% NRV	Rich in “multi-minerals” refer to 3 or more minerals, complying with the “rich in” demand

a When use per serving as the measuring unit, it also should comply with per 100g(ml) demand.

Table C.2 Synonymous name of content claim

Standard	Synonymous	Standard	Synonymous
Not including, or no	Zero (0), without, 100 % not including, no, 0 %	Including, origin of	Provide, include, have
Very low	very few	Rich in, high	Good origin, with rich XX, rich in xx, provide high xx (content)
Low	less, less oil ^a		

a “less oil” refers to low fat claim only.

Table C.3 Requirements and Conditions for energy and nutrition comparative claim

Comparative Claim Form	Requirements	Conditions
Energy reduced	Compared with the reference food, energy value is reduced by 25% or more	The reference food should be of the same type or genus which is well known by the consumers and easy to be understood.
Protein enhanced or reduced	Compared with the reference food, protein content is enhanced or reduced by 25% or more	
Fat reduced	Compared with the reference food, fat content is reduced by 25% or more	
Cholesterol reduced	Compared with the reference food, cholesterol content is reduced by 25% or more	
Carbohydrate enhanced or reduced	Compared with the reference food, carbohydrate content is enhanced or reduced by 25% or more	
Sugar reduced	Compared with the reference food, sugar content is reduced by 25% or more	
Dietary fiber enhanced or reduced	Compared with the reference food, dietary fiber content is enhanced or reduced by 25% or more	
Sodium reduced	Compared with the reference food, sodium content is reduced by 25% or more	
Mineral (exclude sodium) enhanced or reduce	Compared with the reference food, mineral content is enhanced or reduced by 25% or more	
Vitamin enhanced or reduced	Compared with the reference food, vitamin content is enhanced or reduced by 25% or more	

Table C.4 Synonymous of Comparison Claim

Standard	Synonymous	Standard	Synonymous
Enhanced	Enhanced by X % (X times)	Reduced	Reduced by X % (X times)
	Enhanced, enhanced by X % (X times)		Reduced, reduced by X% (X times)
	Raised, raised by X % (X times)		Subtracted, subtracted by X % (X times)
	Heightened, X % (X times) higher		Lowered, X % (X times) lower
	Increased by X % (X times), etc.		Decreased by X % (X times), etc.
	X% more, X times increased		X% less, X times decreased

Appendix D

Standard Language of energy and nutrition function claim

D.1 The present Appendix specifies the standard language of energy and nutrition function claim.

D.2 Energy

The human body needs energy to maintain life activities.

The body growth, development and all activities need energy.

Appropriate energy could maintain good health.

Over intake of energy and insufficient exercise are related with overweight and obesity.

D.3 Protein

Protein is the main component of body and could provide various kinds of amino acids.

Protein is essential to human life activities, as well as contributing to tissue formation and growth.

Proteins help constituting or repairing of human tissue.

Proteins contribute to tissue formation and growth.

Protein is an essential nutriment for tissue formation and growth.

D.4 Fat

Fat could provide high energy.

Energy from fat should not exceed 30% of total energy for daily diet.

Fat is an essential component for human body.

Fat could help the absorption of fat-soluble vitamins.

Fat could provide the essential fatty acid for human body.

D.4.1 Saturated fat

Saturated fat could facilitate the absorption of cholesterol in foods.

Excessive intake of saturated fat will do harm to health.

Excessive intake of saturated fat will cause increase of cholesterol, so the intake should be less than 10% of total energy every day.

D.4.2 Trans fatty acids

Intake of Trans fatty acid should be less than 2.2g everyday and the excessive intake will do harm to health.

Intake of Trans fatty acids should be less than 1% of total energy every day. Excessive intake will do harm to health.

Excessive intake of trans fatty acid will increase the cholesterol in blood, thereby increasing the risk of Cardiovascular disease

D.5 Cholesterol

For adults, the intake of cholesterol should not exceed 300mg for daily diet.

D.6 Carbohydrate

Carbohydrate is a basic compound for human life, also the main source of energy.

Carbohydrate is the main source of energy for human.

Carbohydrate is the main source for blood sugar formation.

Carbohydrate should take about 60% of total energy in diet.

D.7 Dietary fiber

Dietary fiber can help maintain natural function of intestines.

Dietary fiber is low energy.

D.8 Sodium

Sodium could adjust the water balance of body, hence the acid-base balance.

The daily intake of salt should not exceed 6g for adults.

Excessive intake of sodium will do harm to health.

D.9 Vitamin A

Vitamin A helps maintain the scotopia.

Vitamin A helps maintain the health of skin and mucosa.

D.10 Vitamin D

Vitamin D facilitates the absorption of calcium.

Vitamin D helps maintain the health of bone and tooth.

Vitamin D helps the formation of bone.

D.11 Vitamin E

Vitamin E has anti-oxidation effects.

D.12 Vitamin B₁

Vitamin B₁ is an essential component for energy metabolism.

Vitamin B₁ helps maintain the natural physiological function of neural system.

D.13 Vitamin B₂

Vitamin B₂ helps maintain the health of skin and mucosa.

Vitamin B₂ is an essential component for energy metabolism.

D.14 Vitamin B₆

Vitamin B₆ helps the metabolism and use of protein.

D.15 Vitamin B₁₂

Vitamin B₁₂ helps the formation of RBC.

D.16 Vitamin C

Vitamin C helps maintain the health of skin and mucosa.

Vitamin C helps maintain the health of bone and tooth.

Vitamin C could facilitate the absorption of iron.

Vitamin C has anti-oxidation effects.

D.17 Niacin

Niacin helps maintain the health of skin and mucosa.

Niacin is an essential component for energy metabolism.

Niacin helps maintain the health of neural system.

D.18 Folic acid

Folic acid helps the growth of brain and neural system for embryo.

Folic acid helps the formation of RBC.

Folic acid helps the growth of embryo.

D.19 Pantothenic acid

Pantothenic acid is essential for energy metabolism and tissue formation.

D.20 Calcium

Calcium is the main component for human bone and tooth, also participating many of the physiological function.

Calcium is the main component for bone and tooth, as well as maintaining bone density.

Calcium helps the growth of bone and tooth.

Calcium makes the bone and tooth more firm.

D.21 Magnesium

Magnesium is essential for energy metabolism, tissue formation and bone growth.

D.22 Iron

Iron is factor for RBC formation.

Iron is essential for RBC formation.

Iron is essential for production of hemoglobin.

D.23 Zinc

Zinc is essential element for children growth.

Zinc helps improve of appetite.

Zinc helps maintain the health of skin.

D.24 Iodine

Iodine is essential for natural function of hypothyroid.

**GB 13432-2013 Food Labeling of Prepackaged Foods for Special Dietary
Supplies**



National Standards of People's Republic of China

GB 13432-2013

National Food Safety Standard
Food Labeling of Prepackaged Foods for Special Dietary Supplies

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Foreword

This standard replaces "General Standard for the Labeling of Prepackaged Foods for Special Dietary Uses" (GB 13432-2004).

Compared with GB 13432-2004, this standard has the following main changes:

- The standard name was modified;
- The definition of foods for special dietary supplies was modified, and the food category (scope) was defined;
- The basic requirements were modified;
- Some requirements of the compulsory indication contents were modified;
- The permissible and recommended indication contents were combined and modified into optional indication contents;
- The content claim requirements of energy and nutritional ingredients were modified;
- The comparison claim of energy and nutritional ingredients was deleted;
- The function claim wording of energy and nutritional ingredients was modified;
- Appendix A in the former standard was deleted;

The types of foods for special dietary supplies in Appendix A were added

National Standard for Food Safety

Food Labeling of Prepackaged Foods for Special Dietary Supplies

1. Scope

This standard is applicable to the labeling (including nutrition labeling) of prepackaged foods for special dietary supplies.

2. Terms and Definitions

For the purpose of this standard, the terms and definitions specified in GB 7718 and the following ones apply.

2.1 Foods for special dietary supplies

The foods specially processed or formulated to meet the special dietary demand under special physical or physiologic conditions and (or) disease and disorder. The content of the nutrient and (or) other nutritional ingredients in such foods is significantly different from that in comparable ordinary foods.

See Appendix A for the food categories covered by foods for special dietary supplies.

2.2 Nutrients

The substances which have specific physiological effect, be able to maintain the growth, development, activities, and reproduction of organism, and are necessary for normal metabolism, including protein, grease, carbohydrate, minerals and vitamin, etc.

2.3 Nutritional ingredients

The nutrients in food and other food ingredients (except nutrients) having nutrition and (or) physiological functions.

2.4 Recommended intake

The nutrient intake level which can meet the need of the most individuals in a specific gender, age and physiologic status medium group.

2.5 Adequate intake

A safe intake level of nutrient. The intake of some nutrients for healthy people obtained by observation or experiment.

3. Basic Requirements

The labeling of prepackaged foods for special dietary supplies shall meet the basic requirements specified in GB 7718 and the following the requirements:

- It shall not involve in disease prevention and treatment function;

- It shall meet the relevant requirements of labeling and instructions in corresponding product standards of prepackaged foods for special dietary supplies;
- Content claim and comparison claim shall not be conducted to the essential ingredients in the formula foods for infants aged 0~6 months.

4. Compulsory Indication Contents

4.1 General requirements

The contents indicated on the label of prepackaged foods for special dietary supplies shall meet the requirements of corresponding clauses in GB 7718.

4.2 Food name

Only the foods meeting the requirements defined in 2.1 may use "foods for special dietary supplies" in the name or the corresponding name describing product specificity.

4.3 Indication of energy and nutritional ingredients

4.3.1 Energy, protein, grease, carbohydrate and sodium as well as other nutritional ingredients required in corresponding product standards and their contents shall be indicated in the form of "block table". The block may be of any dimension and vertical to the packed base line, the table title is "Nutrition Information". If optional ingredients are added or some substances are intensified in the product according to relevant laws and regulations or standards, these ingredients and their contents shall also be indicated.

4.3.2 The energy and nutrient contents in prepackaged foods for special dietary supplies shall be indicated by every 100 g and every 100mL and the specific value in the edible part of each portion of food. Where it is indicated by portion, the amount of each portion of food shall be indicated, the size of each portion may be determined according to the characteristics or the recommended amount of the food. If necessary or it is required in corresponding product standards, the content of nutritional ingredients in product per 100 KJ shall also be indicated.

4.3.3 The indication values of energy or nutritional ingredients may be obtained through product testing or raw material calculation. In the quality guarantee period of the products, the actual content of energy and nutritional ingredients shall not be less than 80% of the indicated value, and shall meet the requirements of corresponding product standards.

4.3.4 If the proteins in the prepackaged foods for special dietary supplies are provided by hydrolyzed protein or amino acid, the item "proteins" may be indicated by "proteins", "proteins (equivalent)" or "total amount of amino acid".

4.4 Edible method and suitable group

4.4.1 The edible method, daily intake or intake each time of prepackaged foods for special dietary supplies shall be indicated; if necessary, mixing and rehydration reproduction methods shall be indicated.

4.4.2 The suitable group of prepackaged foods for special dietary supplies shall be indicated.

As for infant formula foods for special medical use and formulated foods for special medical use, the suitable group shall be indicated according to the requirements of product standard.

4.5 Storage conditions

4.5.1 The storage conditions of prepackaged foods for special dietary supplies shall be indicated on label, if necessary, the storage conditions after opening shall be indicated.

4.5.2 Consumers shall be specially warned if the opened prepackaged foods for special dietary supplies should not be stored or should not be stored in the original packaging container.

4.6 Exemption of indication contents

When the maximum surface area of the wrappage or packaging container of the prepackaged foods for special dietary supplies is less than 10cm², it is allowable to only indicate the product name, net content, name and address of the manufacturer (or distributor), production date and quality guarantee period.

5. Optional Indication Contents

5.1 The mass percentage of energy and nutritional ingredients to recommended intake or adequate intake

The energy value and content value of nutritional ingredients shall be indicated, and the mass percentage of energy and nutrient content in 100g and 100ml and (or) each portion of food to DRIs-recommended intake (RNI) or adequate intake (AI) may be indicated according to the suitable group, e.g. X%. The nutrients without recommended intake (RNI) or adequate intake (AI) may not be indicated with mass percentage, or be indicated by "-".

5.2 Content claim of energy and nutritional ingredients

5.2.1 Content claim may be conducted when the content of energy or nutritional ingredients in the product reaches the minimum value of corresponding product standard or the permissible intensified minimum value.

5.2.2 Where there is no minimum value requirement or minimum intensification amount for the nutritional ingredients in product standards, the criterion of other national and (or) international organization's permission to conduct content claim of this nutritional ingredient shall be provided.

5.2.3 The content claim wording includes "contain", "provide", "source", "including" and "have", etc..

5.3 Function claim of energy and nutritional ingredients

5.3.1 As for the prepackaged foods for special dietary supplies meeting content claim requirements, the function claim of energy and (or) nutritional ingredients may be conducted. The wording of function claim shall be the standard wording for function claim specified in GB 28050.

5.3.2 As for the nutritional ingredients for which the standard wording of function claim is not listed in GB 28050, the criterion for the function claim wording of this substance specified by other national and (or) international organizations shall be provided.

Appendix A

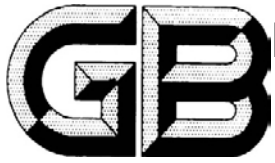
Categories of Foods for Special Dietary Supplies

The categories of foods for special dietary supplies mainly include:

- a) Baby formula foods:
 - 1) Infant formula food;
 - 2) Formula foods for older infant and young children;
 - 3) Infant formula foods for special medical use;
- b) Complementary foods for infants:
 - 4) Cereal-based complementary foods for infants and young children;
 - 5) Canned complementary foods for infants and young children;
- c) Formula foods for special medical use (the variety involved in infant formula foods for special medical use are excluded);
- d) Other foods for special dietary supplies apart from the above-mentioned categories (including complementary nutrition goods, athletic nutrition foods and other foods for special dietary supplies with corresponding national standards).

Contaminants and Bacteria

GB 2762-2012 Maximum Levels of Contaminants in Foods



National Standards of People's Republic of China

GB 2762-2012

National Food Safety Standard Maximum Levels of Contaminants in Foods

Issued on: 2012-11-13

Implemented on: 2013-06-01

Issued by National Health and Family Planning Commission

Foreword

This standard replaces GB 2762-2005 Maximum Levels of Contaminants in Foods.

As compared to GB 2762-2005, key changes are as follows:

- Title of the standard was amended;
- Definition of “edible parts” was added;
- Principle of application was added;
- Removed maximum limits for selenium, aluminium, and fluorin;
- Added maximum limits for tin, 3-chloro-1, 2-propanediol and nitrate;
- Adjusted the limit indicators of maximum levels of N-nitrosamine from N-Nitrosodimethylamine and N-dimethylnitrosamine to N-Nitrosodimethylamine;
- Appendix A was added;

Rare earth indicators were implemented according to the original GB 2762-20

National Standard for Food Safety

Maximum Levels of Contaminants in Foods

1. Scope

This standard stipulates the indicators of maximum levels of lead, cadmium, mercury, arsenic, tin, nickel, chromium, nitrate, nitrite, benzo (a) pyrene, N-nitrosamines, polychlorinated biphenyls (PCBs) and 3-chloro-1, 2-propanediol in foods.

2. Terms and Definition

2.1 Contaminants

Contaminant refers to any materials unintentionally added into the foods during the production (including planting crops, raising animals and applying veterinary medicines), processing, packaging, storage, transportation, sales, consumption of foods or any materials resulting from environmental pollution, the contaminant in this Standard refers to those other than pesticide, veterinary medicines and toxins in fungi.

2.2 Edible Parts

Edible parts refer to the usually edible and drinkable parts obtained from the food raw materials after the inedible parts of the food raw materials are dispelled by mechanical means when bones are removed from meat or fish, the shellfish is shelled, the grains are milled, the fruit is peeled and the nuts are shelled, but excluding the dispelling process that other method is required (such as getting refined vegetable oil from unrefined vegetable oil).

The inedible parts dispelled from the edible parts of the foods are usually based on the processed finished products that meet specified standards, for example, when wheat is made into cereal or whole wheat noodle, the edible part is 100%, when made into flour, it shall be discounted according to flour yield.

2.3 Maximum Levels, MLs

The maximum density of contaminants allowed in edible parts of the food raw materials and/or finished food products.

3. Principle of Application

3.1 Regardless if there is any contamination limit specified, food production and processing parties should take appropriate control measures so as to ensure that the content of contaminants in foods attains the lowest possible level.

3.2 This standard listed all the contaminants that may pose a significant risk to public health and the food categories with limit values specified are representative of foods that have significant effect on the consumer's dietary exposure.

3.3 Descriptions (Appendix A) of food categories (names) used in setting the scope of application for the maximum levels of contaminants in foods are only applicable to this standard. When maximum limit on a specific contaminant is applied towards a specific food category (name), the limit will be applicable for all types of food that fall under the definition of this food category (name), unless otherwise stated.

3.4 Maximum limit of contaminants is computed based on the general edible parts of the food, unless

otherwise stated.

3.5 Maximum limits of contaminants for dried or dehydrated food is adjusted in accordance to the dehydration rate or concentration ratio of the corresponding food raw ingredients. Dehydration rate or concentration ratio can be determined through analysis of food products, information provided by manufacturers and other statistics or information obtainable or available.

4. Index Requirements

4.1 Lead

See Table 1 for indicators of maximum levels of lead in foods.

Table 1 Indicators of Maximum Levels of Lead in Foods

Food Type (Name)	Maximum Level (by Pb) / (mg/kg)
Grains and their products ^a (excluding oatmeal, starch products and gluten)	0.2
Oatmeal, starch products and gluten	0.5
Vegetables and its products	
Fresh vegetables (excluding bulb and stem, leaf vegetables and legume vegetables)	0.1
Bulb and stem, leaf vegetables	0.3
Leguminous vegetables, potatoes	0.2
Vegetable products	1.0
Fruits and their products	
Fruits (excluding berries and grapes)	0.1
Berries and grapes	0.2
Fruit products	1.0
Edible fungi and its products	1.0
Beans and their products	
Beans	0.2
Soy products (excluding soybean milk)	0.5
Soybean milk	0.05
Algae and its product	1.0 (measured in dried form)
Nuts and seeds (excluding coffee nuts)	0.2
Coffee nut	0.5
Meat and meat products	0.2
Meats	
Edible animal offal and its products	0.5
Meat products	0.5
Aquatic products and their products	
Fresh aquatic products (excluding fish, crustaceans, bivalves)	1.0 (Eviscerated)
Fish, crustaceans	0.5
Bivalves	1.5
Aquatic products (excluding dried jelly fish)	1.0
Dried jelly fish and dried seashells	2.0

Food Type (Name)	Maximum Level (by Pb) / (mg/kg)
Milk and milk products	
Raw milk, pasteurized milk, sterilized milk, fermented milk and formulated milk	0.05
Milk powder, non-desalted whey powder	0.5
Other milk products	0.3
Egg and egg products (excluding preserved egg)	0.2
Preserved egg, preserved egg sausages	0.5
Fats and oils, and fat emulsions	0.1
Condiments (excluding edible salt and spices)	1.0
Edible salt	2.0
Spices	3.0
Sugar and starch sugar	0.5
Starch and starch products	
Edible starch	0.2
Starch products	0.5
Bakery products	0.5
Beverages	
Packaged drinking water	0.01 mg/L
Fruit and vegetable juice (excluding condensed fruit and vegetable juice (pulp)	0.05 mg/L
Condensed fruit and vegetable juice (pulp)	0.5 mg/L
Protein beverages (excluding milk drinks)	0.3 mg/L
Milk drinks	0.05 mg/L
Carbonated beverage and tea drinks	0.3 mg/L
Solid beverages	1.0
Other beverages	0.3 mg/L
Alcoholic liquors (excluding distilled wine and traditional Chinese yellow wine)	0.2
Distilled wine and traditional Chinese yellow wine	0.5
Cocoa products, chocolate and chocolate products and candies	0.5
Frozen beverages	0.3

Food Type (Name)	Maximum Level (by Pb) / (mg/kg)
Special nutritious foods	
Infant formulas (excluding liquid form products)	0.15 (measured in powder form)
Liquid form products	0.02 (measured in ready condition)
Infant supplementary foods	
Cereal supplementary foods for infant and babies (excluding fish, liver, vegetables added products)	0.2
Fish, liver, vegetables added products	
Canned supplementary foods for infant and babies (excluding the products made from aquatic products and animal liver)	0.3
Products made from aquatic products and animal liver	0.25
Products made from aquatic products and animal liver	0.3
Other types	
Fruit jelly	0.5
Extruded food	0.5
Tea	5.0
Dried Chrysanthemum	5.0
Ilex	2.0
Honey products	
Honey	1.0
Flower powder	0.5

^a Paddy is calculated based on quantity of brown rice.

4.1.2 Inspection methods: according to GB 5009.12

4.2 Cadmium

4.2.1 See Table 2 for indicators of maximum levels of cadmium in foods.

Table 2 Indicators of Maximum Levels of Cadmium in Foods

Food Type (Name)	Maximum Level (by Cd) / (mg/kg)
Cereal and cereal products	
Cereals (except paddy ^a)	0.1
Grain milling processed (except brown rice, rice)	0.1
Paddy, brown rice, rice	0.2
Vegetables and its products	
Vegetables (excluding bulb and stem, leaf vegetables and legume vegetables)	0.05
Leaf vegetables	0.2
Legume and stem vegetables (excluding celery)	0.1
Celery	0.2
Fruit and its products	
Fresh fruit	0.05
Mushroom and its products	

Fresh mushroom (except mushrooms and Agaricus)	0.2
Mushrooms	0.5
Mushroom products (except Agaricus products)	0.5
Beans and products	
Beans	0.2
Nuts and seeds	
Peanut	0.5
Meat and meat products	
Meat (except poultry offal)	0.1
Poultry liver	0.5
Animal kidneys	1.0
Meat (except liver products, renal products)	0.1
Liver products	0.5
Renal products	1.0
Food Type (Name)	Maximum Level (by Cd) / (mg/kg)
Aquatic animals and their products	
Fresh, frozen aquatic animals	0.1
Fish	0.5
Crustaceans	2.0 (eviscerated)
Bivalves, gastropods, cephalopods, echinoderms	
Aquatic products	
Canned fish (anchovies, swordfish except canned)	0.2
Anchovies, swordfish, canned	0.3
Other fish products (except anchovies, swordfish products)	0.1
Anchovies, swordfish products	0.3
Egg and egg products	0.05
Condiments (excluding edible salt)	
Edible salt	0.5
Fish condiments	0.1
Beverages	
Mineral water (excluding bottled or barreled drinking water)	0.005mg/L
Bottled or barreled drinking water	0.003mg/L
^a Paddy is calculated based on quantity of brown rice.	

4.2.2 Inspection methods: according to GB/T 5009.15

4.3 Mercury

4.3.1 See Table 3 for indicators of maximum levels of mercury in foods.

Table 3 Indicators of Maximum Levels of Mercury in Foods

Food Type (Name)	Maximum Level (by Hg) / (mg/kg)	
	Total Mercury	Methylmercury ^a
Aquatic products (excluding predatory fish)	-	0.5
Predatory fish	-	1.0
Cereal and cereal products Paddy ^b , brown rice, rice, corn, cornmeal (slag, tablets), wheat, wheat flour	0.02	-
Vegetables Fresh vegetables	0.01	-
Edible fungi and its products	0.1	-
Meat and meat products	0.05	-
Milk and milk products Raw milk, pasteurized milk, sterilized milk, fermented milk and formulated milk	0.01	-
Egg and egg products Fresh eggs	0.05	-
Condiments Edible salt	0.1	-
Beverages Mineral water	0.001 mg/L	-
Special nutritious foods Canned supplementary foods for infant and babies	0.02	-
^a Aquatic animals and their products can be first tested for total mercury and if total mercury level does not exceed methylmercury limit, then test for methylmercury is not required; if not, methylmercury determination will be required.		
^b Paddy is calculated based on quantity of brown rice.		

4.3.2 Inspection methods: according to GB/T 5009.17

4.4 Arsenic

4.4.1 See Table 4 for indicators of maximum levels of Arsenic in foods.

Table 4 Indicators of Maximum Levels of Arsenic in Foods

Food Type (Name)	Maximum Level (by As) / (mg/kg)	
	Total Arsenic	Inorganic Arsenic
Cereal and cereal products		
Cereals (except Paddy ^a)	0.5	-
Grain milling processed (except brown rice, rice)	0.5	-
Paddy ^a , brown rice, rice	-	0.2
Aquatic animals and their products (excluding fish and its products)	-	0.5
Fish and its products	-	0.1
Vegetables		
Fresh vegetables	0.5	-
Edible fungi and its products	0.5	-
Meat and meat products	0.5	-
Milk and milk products		
Raw milk, pasteurized milk, sterilized milk, fermented milk and formulated milk	0.1	-
Milk powder	0.5	-
Fat and its products	0.1	-
Condiments (excluding aquatic condiments, algae condiments and spices)	0.5	-
Aquatic condiments (except fish seasoning)	-	0.5
Fish seasoning	-	0.1
Sugar and starch sugar	0.5	-
Beverages		
Packaged drinking water	0.01 mg/L	-
Cocoa products, chocolate and chocolate products and candies		
Cocoa products, chocolate and chocolate products	0.5	-
Foods for special dietary uses		
Cereal supplementary foods for infant and babies (excluding algae added products)	-	0.2
Algae added products	-	0.3
Canned supplementary foods for infant and babies (excluding the products made from aquatic products and animal liver)	-	0.1
Products made from aquatic products and animal liver	-	0.3

^a Paddy is calculated based on quantity of brown rice.

4.4.2 Inspection methods: according to GB/T 5009.11

4.5 Tin

4.5.1 See Table 5 for indicators of maximum levels of tin in foods.

Table 5 Indicators of Maximum Levels of Chromium in Foods

Food Type (Name)	Maximum Level (by Sn) / (mg/kg)
Foods (excluding beverage, infant formula, infant food supplements) ^a	250
Beverage	150
Infant formula, infant food supplements	50

^a only limited to canned foods packaged in containers made of tin-plated sheets.

4.5.2 Inspection methods: according to GB/T 5009.16

4.6 Nickel

4.6.1 See Table 6 for indicators of maximum levels of nickel in foods.

Table 6 Indicators of Maximum Levels of Nickel in Foods

Food Type (Name)	Maximum Level (by Ni) / (mg/kg)
Oil Products	
Hydrogenated vegetable oils and hydrogenated vegetable oil-based products	1.0

4.6.2 Inspection methods: according to GB/T 5009.138

4.7 Cadmium

4.7.1 See Table 7 for indicators of maximum levels of chromium in foods.

Table 7 Indicators of Maximum Levels of Chromium in Foods

Food Type (Name)	Maximum Level (by Cr) / (mg/kg)
Cereal and its products	
Cereal ^a	1.0
Grain milling processed	1.0
Vegetables and their products	
Fresh vegetables	0.5
Beans and their products	
Beans	1.0
Meat and its products	1.0
Aquatic animals and their products	2.0
Milk and milk products	
Raw milk, pasteurized milk, sterilized milk, fermented milk and formulated milk	0.3
Milk powder	2.0

^a Paddy is calculated based on quantity of brown rice.

4.7.2 Inspection methods: according to GB/T 5009.123

4.8 Nitrite, Nitrate

4.8.1 See Table 8 for indicators of maximum levels of nitrite and nitrate in foods.

Table 8 Indicators of Maximum Levels of Nitrite and Nitrate in Foods

Food Type (Name)	Maximum Level / (mg/kg)	
	Nitrite (by NaNO_2)	Nitrate (by NaNO_3)
Vegetables and their products Pickled vegetables	20	-
Milk and milk products Raw milk Milk powder	0.4 2.0	- -
Beverages Packaged drinking water (excluding mineral water) Mineral water	0.005mg/L (by NO_2^-) 0.1mg/L (by NO_2^-)	- 45mg/L (by NO_3^-)
Special nutritious foods Infant formulas Infant formula Older infants and infant formula food Infant formula with special medical purposes Supplementary foods for infant and babies Infant cereal supplementary foods Canned infant supplementary foods	2.0 ^a (by powdered product) 2.0 ^a (by powdered product) 2.0 (by powdered product) 2.0 ^c 4.0 ^c	100 ^b (by powdered product) 100 ^b (by powdered product) 100 (by powdered product) 100 ^b 200 ^b
^a Only applies to dairy based products. ^b Does not apply to products added with vegetable and fruits. ^c Does not apply to products added with beans.		

4.8.2 Inspection methods: Beverage according to GB/T 8538, others according to GB 5009.33

4.9 Benzo(a)pyrene

4.9.1 See Table 9 for indicators of maximum levels of Benzo(a)pyrene in foods.

Table 9 Indicators of Maximum Levels of Benzo(a)pyrene in Foods

Food Type (Name)	Maximum Level / (µg/kg)
Cereal and cereal products Paddy ^a , brown rice, rice, wheat, wheat flour, corn, cornmeal (slag, tablets)	5.0
Meat and meat products Smoked, cooked, roasted meats	5.0
Aquatic animals and its products Smoked, roasted aquatic products	5.0
Fats and oils, and fat emulsions	10
^a Paddy is calculated based on quantity of brown rice.	

4.9.2 Inspection methods: in according to GB/T 5009.27

4.10 N-nitrosamine

4.10.1 See Table 10 for indicators of maximum levels of N-nitrosamine in foods.

Table 10 Indicators of Maximum Levels of N-nitrosamine in Foods

Food Type (Name)	Maximum Level / (µg/kg)
Meat and its products Meat products (excluding canned meat)	3.0
Aquatic products Aquatic products (excluding canned aquatic foods)	4.0

4.10.2 Inspection methods: in according to GB/T 5009.26

4.11 Polychlorinated biphenyls (PCBs)

4.11.1 See Table 11 for indicators of maximum levels of Polychlorinated biphenyls (PCBs) in foods.

Table 11 Indicators of Maximum Levels of Polychlorinated biphenyls (PCBs) in Foods

Food Type (Name)	Maximum Level ^a / (mg/kg)
Aquatic products and their products	0.5
^a Polychlorobiphenyls is accounted as the sum of PCB28, PCB52, PCB101, PCB118, PCB138, and PCB153 and PCB180.	

4.12 3-chloro-1, 2-propanediol

4.12.1 See Table 12 for indicators of maximum levels of 3-chloro-1, 2-propanediol in foods.

Table 12 Indicators of Maximum Levels of 3-chloro-1, 2-propanediol in Foods

Food Type (Name)	Maximum Level / (mg/kg)
Condiments ^a	
Liquid seasoning	0.4
Solid seasoning	1.0
^a Only limited to the products added with hydrolyzed vegetable protein.	

4.12.2 Inspection methods: in according to GB/T 5009.191

Appendix A

Food Category (Name) Descriptions

A.1 See Table A.1 for descriptions of food categories (names).

Table A.1 Food Category (Name) Descriptions

Fruits and products	<p>Fresh fruit (unprocessed, surface-treated, pre-cut or peeled, frozen fruit)</p> <ul style="list-style-type: none"> Arabica berries and other fruits Other fresh fruit (including sugar cane) <p>Fruit Products</p> <ul style="list-style-type: none"> Canned fruit Dried fruit category Vinegar, oil, or salted fruits Jam (mud) Candied preserved fruits (including fruit leather) Fermented fruit products Cooked or fried fruit Fruit Desserts Other fruit products
Vegetables and their products (including potatoes, not including edible fungi)	<p>Fresh vegetables (raw, surface-treated, pre-cut or peeled, frozen vegetables)</p> <ul style="list-style-type: none"> Brassica vegetables Leafy vegetables (including Brassica leafy category) Leguminous vegetables Root and tuber vegetables (such as potatoes, carrots, radish, ginger, etc.) Stem vegetables (including bean sprouts) Other fresh vegetables (including melons, Bulb and aquatic classes, bean sprouts and bamboo shoots perennial vegetables) <p>Vegetable products</p> <ul style="list-style-type: none"> Canned vegetables Dried vegetables Pickled vegetables (for example, marinated, salted, sweet and sour pickle vegetables, etc.) Vegetable puree (sauce) Fermented vegetable products Boiled or fried vegetables Other vegetable products

Mushroom and its products	<p>Fresh mushroom (raw, surface-treated, pre-cut, frozen mushroom)</p> <p>Mushrooms</p> <p>Agaricus</p> <p>Other fresh mushroom</p> <p>Mushroom products</p> <p>Canned mushroom</p> <p>Dried mushroom</p> <p>Pickled mushroom (for example, marinated, salted, sweet and sour mushroom stains, etc.)</p> <p>Boiled or fried mushroom</p> <p>Other mushroom products</p>
Cereal and cereal products (excluding bakery products)	<p>Cereals</p> <p>Paddy</p> <p>Corn</p> <p>Wheat</p> <p>Barley</p> <p>Other cereals [eg, millet (millet), sorghum, rye, oats, buckwheat, etc.]</p> <p>Processed grain milling</p> <p>Brown rice</p> <p>Rice</p> <p>Wheat flour</p> <p>Cornmeal (slag, tablets)</p> <p>Oatmeal</p> <p>Other hulled grains (such as millet, sorghum, rice, pearl barley, millet, etc.)</p> <p>Cereal products</p> <p>Rice products (such as rice, flour and other products, such as rice balls)</p> <p>Wheat flour products</p> <p>Raw wet noodle products (for example, noodles, dumpling skin, wonton skin, burning oatmeal, etc.)</p> <p>Health Dried pastas</p> <p>Fermented flour products</p> <p>Batter (for example, for fish and poultry drag batter), breaded, fried flour</p> <p>Gluten</p> <p>Other wheat flour products</p> <p>Corn Products</p> <p>Other cereal products (for example, with stuffing (material) plane rice products, rice pudding, canned, etc.)</p>
Beans and their products	<p>Legumes (dried beans, dried beans, flour in)</p> <p>Soy products</p> <p>Non-fermented soy products (eg, soy milk, tofu, dried tofu category, yuba classes, cooked beans, soy protein puffed food, vegetarian soy meat, etc.)</p> <p>Fermented soy products (eg, fermented bean curd class, natto, tempeh, tempeh products, etc.)</p> <p>Canned beans</p>

Algae and their products	<p>Fresh algae (unprocessed, surface-treated, pre-cut, frozen algae)</p> <p>Spirulina</p> <p>Other fresh algae</p> <p>Algae Products</p> <p>Algae canned</p> <p>Dried algae</p> <p>After boiled or fried</p> <p>Other algae products</p>
Nuts and seeds	<p>Fresh nuts and seeds</p> <p>Tree nuts (tree nuts)</p> <p>Oil (not including cereals and legumes seed)</p> <p>Sweet drinks and seeds (e.g., cocoa beans, coffee beans, etc.)</p> <p>Nuts and seeds products</p> <p>Cooked nuts and seeds (shelled, shelling)</p> <p>Coated nuts and seeds</p> <p>Canned nuts and seeds</p> <p>Nut and seed purees (sauce), including peanut butter, etc.</p> <p>Other nuts and seeds products (for example, pickled nuts, etc.)</p>
Meat and meat products	<p>Meat (fresh, cooled, frozen meat, etc.)</p> <p>Poultry meat</p> <p>Poultry offal (e.g., liver, kidney, lung, intestine, etc.)</p> <p>Meat (including offal products)</p> <p>Prefabricated meat</p> <p>Conditioning meat (raw material added conditioning)</p> <p>Cured meat products (for example, bacon, bacon, duck, Chinese ham, salami, etc.)</p> <p>Cooked meat</p> <p>Canned meat</p> <p>Sauce braised pork products category</p> <p>Smoked, burned, grilled meat</p> <p>Fried meat</p> <p>Ham (smoked, smoked, cooked ham) category</p> <p>Meat sausage class</p> <p>Fermented meat products category</p> <p>Cooked meat products (eg, floss, dried meat, dried meat, etc.)</p> <p>Other cooked meat</p>
Aquatic animals and their products	<p>Fresh, frozen aquatic animals</p> <p>Fish</p> <p>Non-carnivorous fish</p> <p>Carnivorous fish (such as sharks, tuna, etc.)</p> <p>Crustaceans</p> <p>Molluscs</p> <p>Cephalopods</p> <p>Bivalves</p>

	<ul style="list-style-type: none"> Echinoderms Gastropods Other molluscs Other fresh, frozen aquatic animals Aquatic products Canned fish Surimi products (including fish, etc.) Pickled fish Roe products Dried fish (dried, drying, pressed dry, etc.) Smoked, grilled fish Fermented fish Other aquatic products
Milk and dairy products	<ul style="list-style-type: none"> Milk Pasteurized milk Sterilized milk Modulation milk Fermented milk Condensed milk Milk Whey powder and whey protein powder (including non-desalted whey powder) Cheese Processed cheese Other dairy products
Eggs and egg products	<ul style="list-style-type: none"> Eggs Egg Products Spiced corned egg Bad eggs Preserved egg Salted Dehydrated egg products (eg, protein powder, egg yolk powder, protein tablets, etc.) Thermal coagulation egg products (eg, casein egg yolk, egg intestines, etc.) Frozen egg products (for example, ice and eggs) Other egg products
Oil Products	<ul style="list-style-type: none"> Vegetable oils Animal fats (such as lard, butter, oil, cream, butter, anhydrous butter, etc.) Oil products Hydrogenated vegetable oils and hydrogenated vegetable oil-based products (eg, margarine, shortening, etc.) Cooking oil Other oil products
Condiment	<ul style="list-style-type: none"> Salt Fresh flavor agents and agents

	<p>Vinegar</p> <p>Soy sauce</p> <p>Butter and butter products</p> <p>Flavored wine</p> <p>Spices category</p> <ul style="list-style-type: none"> Spices and flour Spice oils Spice sauce (such as mustard, green mustard sauce, etc.) Other processed spices <p>Aquatic spices</p> <ul style="list-style-type: none"> Fish condiments (eg, fish sauce, etc.) Other aquatic condiments (eg, oyster sauce, shrimp sauce, etc.) <p>Complex seasonings (eg, solid soup, chicken, chicken, mayonnaise, salad dressing, seasoning juice, etc.)</p> <p>Other condiments</p>
Drinks	<p>Packaged Drinking Water</p> <ul style="list-style-type: none"> Mineral water Pure water Other packaged drinking water <p>Juice category (eg, apple juice, apple cider vinegar, juice, hawthorn, hawthorn vinegar)</p> <ul style="list-style-type: none"> Fruit and vegetable juice (pulp) Concentrated fruit and vegetable juice (pulp) Other fruit and vegetable juices (meat) beverages (including fermented products) <p>Protein Drinks</p> <ul style="list-style-type: none"> Milk beverages (fermented milk drinks, milk drinks prepared type, lactobacillus drinks) Plant protein drinks Compound Protein Beverage <p>Carbonated beverages</p> <p>Tea Drinks</p> <p>Coffee Drinks</p> <p>Plants Drinks</p> <p>Flavor Drinks</p> <p>Special purpose beverages (for example, sports drinks, nutrition drinks, etc.)</p> <p>Solid drinks (including instant coffee)</p> <p>Other Drinks</p>
Liquor	<p>Distilled spirits (eg, wine, brandy, whiskey, vodka, rum, etc.)</p> <p>Liqueur</p> <p>Fermented (eg, wine, wine, wine, beer, etc.)</p>
Sugar and Sweeteners	<p>Sugar</p> <ul style="list-style-type: none"> Sugar and sugar products (for example, sugar, cotton, sugar, sugar, sugar, etc.) Other sugars and syrups (eg, brown sugar, brown sugar, borneol sugar, raw

	sugar, molasses, partially invert sugar, maple syrup, etc.) Starch sugar (e.g., fructose, glucose, maltose, invert sugar and other parts)
Starch and starch products (including cereals, legumes and root plant extracts starch)	Edible starch Starch products Noodles, vermicelli Lotus root starch Other starch products (eg, shrimp pieces)
Baked Goods	Bread Pastries (including moon cake) Crackers (eg, cookies, waffles, omelets, etc.) Other baked goods
Cocoa products, chocolate and chocolate products and candies	Cocoa products, chocolate and chocolate products (including cocoa butter and chocolate products) Candy (including gum candy)
Frozen drinks	Ice cream, ice cream category Flavor ice lollies class Edible ice Other frozen drinks
Foods for special dietary uses	Infant formula Infant formula Older infants and infant formula food Infant formula for special medical purposes Infant food supplements Infant cereal food supplements Canned infant complementary food Other foods for special dietary uses
Other categories (in addition to the above food Food)	Jelly Expanded Food Bee (eg, honey, pollen, etc.) Tea Dry Chrysanthemum Ilex

GB 29921-2013 Limits of Pathogenic Bacterium in Food



National Standards of People's Republic of China

GB 29921-2013

National Food Safety Standard
Limits of Pathogenic Bacterium in Food

Issued on: 2013-12-26

Implemented on: 2014-07-01

Issued by National Health and Family Planning Commission

National Standard for Food Safety

Limits of Pathogenic Bacterium in Food

1. Scope

This standard specifies the index limit requirements on pathogenic bacterium and corresponding testing methods.

This standard is applicable to prepackaged food.

This standard is not applicable to canned food.

2. Terms and Definitions

2.1 It is necessary to minimize the level of content of pathogenic bacterium in food and its corresponding probability of risks through establishing control mechanisms and system for the production, processing and distribution of food products, regardless of whether there are existing stipulated limits on pathogenic bacterium.

2.2 Inspection should be implemented in accordance with the testing methods specified in Table 1 after taking samples based on the sampling methodology specified in GB 4789.1.Limits of Pathogenic

3. Requirements on Index

See Table 1 for the limits of pathogenic bacterium in food.

Table 1 Limits of Pathogenic Bacterium in Food

Food Category	Pathogenic Bacterium Index	Sampling Plan and Limit (unit used is 25g or 25ml, unless otherwise specified)				Testing Method	Remarks
		n	c	m	M		
Meat products Cooked meat products Ready-to-eat raw meat products	Salmonella	5	0	0	-	GB 4789.4	-
	Listeria monocytogenes	5	0	0	-	GB 4789.30	
	Staphylococcus aureus	5	1	100 CFU/g	1,000 CFU/g	GB 4789.10 (2 nd Method)	
	Escherichia coli O157:H7	5	0	0	-	GB/T 4789.36	Only applies to beef products
Aquatic products Cooked aquatic products Ready-to-eat raw aquatic products Ready-to-eat algae products	Salmonella	5	0	0	-	GB 4789.4	-
	Vibrio parahaemolyticus	5	1	100 MPN/g	1,000 MPN/g	GB/T 4789.7	
	Staphylococcus aureus	5	1	100 CFU/g	1,000 CFU/g	GB 4789.10 (2 nd Method)	
Ready-to-eat egg products	Salmonella	5	0	0	-	GB 4789.4	-
Grain products Cooked grain products (incl. baked category) Cooked flour/rice products with filling (stuffing) Ready-to-eat flour/rice products	Salmonella	5	0	0	-	GB 4789.4	-
	Staphylococcus aureus	5	1	100 CFU/g	1,000 CFU/g	GB 4789.10 (2 nd Method)	
Instant soy products Fermented soy products Non-fermented soy products	Salmonella	5	0	0	-	GB 4789.4	-
	Staphylococcus aureus	5	1	100 CFU/g	1,000 CFU/g	GB 4789.10 (2 nd Method)	
Chocolate and cocoa products	Salmonella	5	0	0	-	GB 4789.4	

Food Category	Pathogenic Bacterium Index	Sampling Plan and Limit (unit used is 25g or 25ml, unless otherwise specified)				Testing Method	Remarks
		n	c	m	M		
Ready-to-eat fruit and vegetable products (incl. pickled category)	Salmonella	5	0	0	-	GB 4789.4	-
	Staphylococcus aureus	5	1	100 CFU/g (mL)	1,000 CFU/g (mL)	GB 4789.10 (2 nd Method)	
	Escherichia coli O157:H7	5	0	0	-	GB/T 4789.36	Only applies to raw fruit, vegetable products
Beverages (excl. packaged water and carbonated beverage)	Salmonella	5	0	0	-	GB 4789.4	-
	Staphylococcus aureus	5	1	100 CFU/g (mL)	1,000 CFU/g (mL)	GB 4789.10 (2 nd Method)	
Frozen beverages Ice cream category Milk ice category Edible ice and ice lolly category	Salmonella	5	0	0	-	GB 4789.4	-
	Staphylococcus aureus	5	1	100 CFU/g (mL)	1,000 CFU/g (mL)	GB 4789.10 (2 nd Method)	
Ready-to-eat condiments Soy sauce Sauces and sauce products Aquatic product seasoning Compound condiments (salad dressing)	Salmonella	5	0	0	-	GB 4789.4	-
	Listeria monocytogenes	5	2	100 CFU/g (mL)	1,000 CFU/g (mL)	GB 4789.10 (2 nd Method)	
	Staphylococcus aureus	5	1	100 MPN/g (mL)	1,000 MPN/g (mL)	GB/T 4789.7	Only applies to aquatic product seasonings
Nuts and seeds products Nuts and seeds sauce Pickled nuts category	Salmonella	5	0	0	-	GB 4789.4	-

Note1: using food category to define the limit of pathogenic bacterium is only applicable to this standard.

Note2: “n” is for the amount of samples from one batch, “c” is for the maximum amount of samples exceeding the value “m”, “m” is for the limit value acceptable for pathogenic bacterium indicator, “M” is for the maximum safe limit value of pathogenic bacterium indicator.

GB 5009.12-2010 Determination of Lead in Foods



National Standards of People's Republic of China

GB 5009.12-2010

**National Food Safety Standard
Determination of Lead in Foods**

Issued on: 2010-03-26

Implemented on: 2010-06-01

Issued by National Health and Family Planning Commission

Foreword

This standard replaces GB/T 5009.12-2003 “Determination of lead in foods”. The Appendix A in this standard is informative reference

Versions of standard substituted by this standard are:

- GB/T 5009.12-1985, GB/T 5009.12-1996, GB/T 5009.12-2003.

National Standard for Food Safety

Determination of Lead in Foods

1. Scope

This Standard regulates the method for the determination of lead in foods. This Standard applies to the determination of lead in foods.

2. Normative documents

The reference cited in this standard is necessary. For the cited documents which are labeled with date, all their subsequent modification sheets or modified versions are not applicable for this standard. For the cited documents which are not labeled with date, their latest versions are applicable for this standard.

Method 1: Graphite furnace atomic absorption spectrometry

3. Principle

After ashing or acid digestion, the sample is injected into the graphite furnace of atomic absorption spectrophotometer. It then absorbs the resonance line at 283.3 nm after electrothermal atomization. In certain concentration range, the absorption is proportional to lead content, and is used to yield quantitative lead content on the basis of comparison with standard series.

4. Reagents and materials

Unless other specified rules, all the reagents used in this method are pure, and water shall be grade I under the GB/T 6682.

4.1 Nitric acid: GR

4.2 Ammonium persulfate.

4.3 Hydrogen peroxide (30%).

4.4 Perchloric acid: GR.

4.5 Nitric acid (1+1): 50 mL of nitric acid is slowly added into 50 mL of water.

4.6 Nitric acid (0.5 mol/L): 3.2 mL of nitric acid is added into 50 mL of water and then diluted to 100 mL.

4.7 Nitric acid (1 mol/L): 6.4 mL of nitric acid is added into 50 mL of water and then diluted to 100 mL.

4.8 Ammonium phosphate solution (20 g/L): dissolve 2.0 g of ammonium phosphate in water and then diluted to 100 mL.

4.9 Mixed acid: Nitric acid + perchloric acid (9+1). 9 volume of nitric acid is mixed with 1 volume of perchloric acid.

4.10 Standard lead stock solution: weigh 1.000 g of lead (99.99%) accurately, added with a small

amount of nitric acid (1+1) for several times and heated to dissolve. The total volume of nitric acid is not more than 37 mL. Then transfer into a 1000 mL volumetric flask and make up to the mark. Mix well. Lead concentration in the solution is 1.0 mg/mL.

4.11 Standard lead working solution: Pipette 1.0 mL of standard lead stock solution in a 100 mL volumetric flask and make up to the mark with nitrate acid (4.6). Do so several times to obtain standard lead working solution with concentrations of 10.0 ng/mL, 20.0 ng/mL, 40.0 ng/mL, 60.0 ng/mL and 80.0 ng/mL.

5. Equipment and facilities

5.1 Atomic absorption spectrophotometer, with graphite furnace and lead hollow cathode lamp.

5.2 Muffle furnace.

5.3 Balance: sense of balance is 1mg

5.4 Dryer thermostat.

5.5 Porcelain crucible.

5.5 Pressure digestion device, pressure digestion drum or pressure digestion tank.

5.6 Adjustable electric heating plate and adjustable electric furnace.

6. Analysis procedure

6.1 Sample pretreatment

6.1.1 During sampling and preparation, the sample should be prohibited from contamination

6.1.2 After the removal of impurities, grain and beans are ground, pass through a 20-mesh sieve, and are stored in the plastic bottle for use.

6.1.3 Fresh samples with a high water content such as vegetables, fruit, fish, meat and eggs are processed into homogenate by using food processing machine or homogenizers, and then stored in the plastic bottle for use.

6.2 Sample digestion (any digestion method can be selected according to laboratory conditions)

6.2.1 Digestion by pressure digestion tank: 1 g-2 g of sample (nearest to 0.001g, for dry sample and samples with high fat contents, the weight is less than 1 g; for fresh sample, the weight is less than 2 g; or the weight can be determined according to the recommendation in operation instructions of the pressure digestion tank) is weighed, placed in polytetrafluorethylene inner tank, and soaked in 2 mL-5 mL of nitric acid (4.1) overnight. Added 2 mL-3 mL of hydrogen peroxide (4.3) (total volume not exceeding 1/3 of the tank volume). Cover the inner lid and tighten the

stainless steel outer cover, put into dryer thermostat to stand for 3-4 hours under 120°C-150°C, and then cooled to room temperature naturally in the oven. The digestion solution is washed and transferred into or filtered into (depending on the salt content of the sample after digestion) a 10 mL-25 mL volumetric flask using a dropper. A small amount of water is used to wash the tank for many times and then transferred into the volumetric flask, make up to the mark. Mix well. Meanwhile, the reagent blank is prepared.

6.2.2 Dry ashing: weigh 1 g-5 g of sample (nearest to 0.001g, depending on lead content) into porcelain crucible, heated on the adjustable electric heating plate to no smoke. Transferred into muffle furnace and stays for 6 h-8 h at $500 \pm 25^\circ\text{C}$ and cooled down. If certain sample is not completely ashed, add 1 mL of mixed acid (4.9) and heated on the adjustable electric furnace with low power. The process is then repeated for many times until the completion of the digestion happened. After that the sample cools to room temperature and dissolves in nitric acid. The sample digestion solution is washed and transferred into or filtered into (depending on the salt content of the sample after digestion) a 10 mL-25 mL volumetric flask using a dropper. A small amount of water is used to wash the porcelain crucible for many times and then transferred into the volumetric flask, make up to the mark. Mix well. Meanwhile, the reagent blank is prepared.

6.2.3 Ammonium persulfate ashing method: into porcelain crucible weigh 1 g-5 g of sample (nearest to 0.001g), add 2 mL-4 mL of nitric acid (4.1) to soak the sample for more than 1 h. Carbonized under low power at first. After cooling add 2.00 g-3.00 g of ammonium persulfate (4.2) and continues to be carbonized until no smoke is produced. Transfer to muffle furnace to stay for 2 h at 500°C , then 20 min at 800°C and cools down after. Add 2 mL-3 mL of nitric acid (4.7), the sample digestion solution is washed and transferred into or filtered into (depending on the salt content of the sample after digestion) a 10 mL-25 mL volumetric flask using a dropper. A small amount of water is used to wash the porcelain crucible for many times and then transferred into the volumetric flask, make up to the mark. Mix well. Meanwhile, the reagent blank is prepared

6.2.4 Wet digestion method: Into a flask or tall beaker, weigh 1 g-5 g (the nearest to 0.001g) add several glass beads and 10 mL of mixed acid (4.9). The container is then covered to allow the sample to be soaked overnight. After that, digest on a small funnel electric furnace. If become dark brown, more mixed acid should be added until white smoke is produced and the digestion solution is colorless and transparent or a little yellow. Cool down and is washed and transferred into or filtered into (depending on the salt content of the sample after digestion) a 10 mL-25 mL volumetric flask using a dropper. A small amount of water is used to wash the flask or tall beaker for many times and then transferred into the volumetric flask, make up the mark. Mix well. Meanwhile, the reagent blank is prepared.

6.3 Determination

6.3.1 Equipment conditions: adjust equipment to the best situation according to its performance. Reference conditions are as follows: wavelength 283.3 nm, slot 0.2

-1.0 nm, lamp current 5-7 mA, drying temperature 120°C , 20 s; ashing temperature

450°C , 15 s-20 s; atomization temperature 1700°C - 2300°C , 4-5s; the background calibration is based on deuterium lamp or Zeeman Effect.

6.3.2 Preparation of standard curve: pipette 10 μL of each standard lead working solution with a concentration of 10.0, 20.0, 40.0, 60.0 and 80.0 ng/mL (or $\mu\text{g/L}$) respectively and injected into the graphite furnace, measure absorbance and make unary linear regression equation between concentration and absorbance

6.3.3 Sample determination: Pipette 10 μL of sample solution and reagent blank control solution and injected into the graphite furnace to measure absorbance. Then calculate the determination according to unary linear regression equation as the step 6.3.2 shows.

6.3.4 Application of matrix modifier: For samples with interference factors, inject an appropriate amount of matrix modifier of ammonium phosphate solution (4.8), (usually 5 μL or equivalent to the amount of sample), to eliminate the interference. During the preparation of lead standard curve, the matrix modifier ammonium

dihydrogen phosphate solution should also be added with an amount equivalent to that used in sample determination.

7. Calculation of results

The lead content of the sample is calculated on the basis of equation (1).

$$X = \frac{(C1-C0) \times V \times 1000}{m \times 1000} \dots\dots\dots (1)$$

X -- Lead content in the sample, mg/kg or mg/L;

C1 -- Lead content in determination sample solution, ng/mL; C0 -- Lead content in blank control solution, ng/mL;

V -- Total quantitative volume of the sample digestion solution, mL; m -- Weight or volume of the sample, g or mL.

Report the results in terms of arithmetic average from two independent measurements under the repeatable conditions, and possess two significant digits.

8. Degree of precision

The absolute difference between two independent determination results obtained under repeatable conditions is not allowed to exceed 20% of the arithmetic average of them.

Method 2: Hydride generation atomic fluorescence spectrometry 9 Principle

9. Principle

After thermal acid digestion, the sample is placed in the acid medium to allow lead existed in it to react with sodium borohydride (NaBH₄) or potassium borohydride (KBH₄) to yield volatile lead hydride (PbH₄). The hydride is introduced into electrothermal quartz atomizer using carrier gas argon to undergo atomization. Under the illumination of special lead hollow cathode lamp, lead atoms at the ground state are excited to a high energy state. After deactivation, the excited lead atoms get back to the ground state and emit fluorescence light with a characteristic wavelength and with a fluorescent intensity proportional to lead content. Such a relationship is used for the quantitative determination on the basis of standard series.

10. Reagents and materials

- 10.1 Nitric acid and perchloric acid mixture (9+1): mix 900 mL of nitric acid and 100 mL of perchloric acid.
- 10.2 Hydrochloric acid solution (1+1): introduce 250 mL of hydrochloric acid into 250 mL of water
- 10.3 Oxalic acid solution (10 g/L): dissolve 1.0 g of oxalic acid in 100 mL of water and mixed well.
- 10.4 Iron potassium cyanide [K₃Fe(CN)₆] solution (100 g/L): dissolve 10.0 g of iron potassium cyanide in water, diluted to 100 mL and mixed well.
- 10.5 Sodium hydroxide solution (2 g/L): dissolve 2.0 g of sodium hydroxide in 1 L of water and mixed well.
- 10.6 Sodium borohydride [NaBH₄] solution (10 g/L): dissolve 5.0 g of sodium borohydride in 500 mL of

sodium hydroxide solution (2 g/L) and mixed well. This solution should be prepared right before use.

10.7 Standard lead stock solution (1.0 mg/mL)

10.8 Standard lead working solution (1.0 µg/mL): pipette a certain amount of standard lead stock solution (1.0 mg/mL) and diluted to 1.0 µg/mL step by step.

11. Equipment and facilities

11.1 Atomic fluorescence spectrometer

11.2 Lead hollow cathode lamp.

11.3 Electric heating plate.

11.4 Balance: sense of balance is 1 mg

12. Analysis procedure

12.1 Sample digestion

Wet digestion: into 50 mL-110 mL digestion vessel (flask), weigh 0.2 g-2 g of solid sample or 2.00 g (or mL) - 10.00 g (or mL) of liquid sample (nearest to 0.001g), add 5 mL-10 mL of nitric acid and perchloric acid mixture (10.1) shake well and stand for overnight. On the next day, heat the flask on the electric heating plate for digestion until light yellow or colorless (if the color is dark, cool slightly and added a small amount of nitric acid and continue digestion), Cool slightly, add 20 mL of water, and heated again to remove acid until the volume of digestion solution is 0.5 mL-1.0 mL. After cooling down, add a small amount of water to transfer to a 25 mL volumetric flask, added 0.5 mL of hydrochloric acid (10.2) and 0.5 mL of oxalic acid solution (10.3), and mixed well. Then add 1.00 mL of iron potassium cyanide solution (10.4). Then diluted with water to 25 mL accurately, make up to the mark. Mix well. Stand for 30 min. Meanwhile, the reagent blank is prepared.

12.2 Preparation of standard series

Into 25 mL volumetric flasks, accurately added 0.00, 0.125, 0.25, 0.50, 0.75, 1.00 and 1.25 mL of standard lead working solutions (10.8) respectively (lead concentration in each flask will be 0.0, 5.0, 10.0, 20.0, 30.0, 40.0 and 50.0 ng/mL respectively). After diluted with a small amount of water, the solution is added with 0.5 mL of hydrochloric acid (10.2) and 0.5 mL of oxalic acid (10.3), and mixed well. Add 1.0 mL of iron potassium cyanide solution (10.4), diluted with water to 25 mL, and shaken to mix evenly. After staying for 30 min, the solution is ready for determination.

12.3 Determination

12.3.1 Equipment reference conditions

Negative high voltage: 323 V, lead hollow cathode lamp current: 75 mA; atomizer: furnace temperature 750°C-800°C, furnace height: 8 mm; argon flow rate: carrier gas 800 mL/min; shielding gas: 1000 mL/min; duration for the addition of reducing agent: 7.0 s; reading time: 15.0 s; delay: 0.0 s; measurement method: standard curve method; reading method: peak area; injection volume: 2.0 mL.

12.3.2 Measurement method

After the equipment is set under the optimum conditions and the furnace temperature rises to required value progressively and maintains such a value for

10 min-20 min, the measurement may begin. At first, the sample with a concentration of 0 in standard series is introduced to the equipment continuously until the readings are stable. Then other samples in standard series are introduced to the equipment and a standard curve is thus drawn. Finally, the samples, including sample blank control and sample digestion solution, are introduced for the measurement. The lead content of the sample is calculated on the basis of equation (2).

13. Expression of results

The lead content of the sample is calculated on the basis of equation (2).

$$X = \frac{(C_1 - C_0) \times V \times 1000}{m \times 1000 \times 1000} \dots\dots\dots (2)$$

In which,

X -- Lead content in the sample, mg/kg or mg/L;

c1 -- Determination concentration of sample digestion solution, ng/mL;

c0 -- Determination concentration of reagent blank control solution, ng/mL;

V -- Total volume of the sample digestion solution, mL.

m -- Weight or volume of the sample, g or mL;

Report the results in terms of arithmetic average from two independent measurements under the repeatable conditions, and possess two significant digits.

14. Degree of precision

The absolute difference between two independent measurement results obtained under repeatable conditions is not allowed to exceed 10% of the arithmetic average of them.

Method 3: Flame atomic absorption spectrometry

15. Principle

After sample treatment, the lead ion forms a complex with DDTC under a certain pH value, and is introduced to the atomic absorption spectrometer after extraction with 4-methylpentanone-2. After flame atomization, the sample absorbs a resonance line at 283.3 nm with an absorption proportional to lead content. The absorption is thus compared with standard series to yield quantitative results.

16. Reagents and materials

16.1 Nitric acid-perchloric acid (9+1).

16.2 Ammonium sulfate solution (300 g/L): dissolve 30.0 g of ammonium sulfate [(NH₄)₂SO₄] in water and diluted with water to 100 mL.

16.3 Ammonium citrate solution (250g/L): Dissolve 25.0 g of ammonium citrate in water and diluted with

water to 100 mL.

16.4 Bromothymol blue aqueous solution (1 g/L).

16.5 Sodium diethyl dithiocarbamate (DDTC) solution (50 g/L): dissolve 5 g of sodium diethyl dithiocarbamate in water and diluted with water to 100 mL.

16.6 Ammonia (1+1).

16.7 4-Methylpentanone-2 (MIBK).

16.8 Standard lead solution: Operation is the same as those in 10.7 and 10.8. The standard working solution has a lead concentration of 10 µg/mL.

16.9 Hydrochloride acid (1+11): add 10 ml HCl into 110 ml water, mix well

16.10 Phosphoric acid (1+10): add 10 ml phosphoric acid into 100 ml water, mix well

17. Equipment and facilities

17.1 Atomic absorption spectrophotometer with flame atomizer; others are the same as those in 5.2, 5.3, 5.4, 5.5, 5.6 and 5.7.

17.2 Balance: sense of balance is 1 mg

18. Analysis procedure

18.1 Sample treatment

18.1.1 Beverage and liquor: Weigh 10g-20 g (nearest to 0.01g) of well mixed sample in the beaker (The alcohol should be removed from liquor by heating in water bath). The sample is heated on the electric heating plate to evaporate a certain amount of water, add mixed acid (16.1). After full digestion, transfer into a 50 mL volumetric flask and make up to the mark.

18.1.2 The soaking solution of packaging materials can be determined directly.

18.1.3 Cereal: remove impurities and dust, remove the husk if necessary, sieved by a 30-mesh sieve, and mix evenly. Weigh 5g-10 g (nearest to 0.01g) in a 50 mL porcelain crucible, carbonized under low power, and then transfer into muffle furnace, ash for 16 h in a temperature below 500°C. Take crucible out and cool to room temperature. Add a small amount of mixed acid (16.1) and heated under low power so that the mixture does not dry out. The process repeats until there is no charcoal grain in the residue. When the crucible cools slightly, add 10 mL of hydrochloric acid (16.9) to dissolve it. Transfer into a 50 mL volumetric flask. Wash the crucible repeatedly with water and transfer into the volumetric flask. Make up to the mark. This solution should be freshly prepared each time

Prepare reagent blank with same volume of mixed acid and hydrochloric acid (16.9).

18.1.4 Vegetables, melon, fruit and beans: clean and dry the edible part, grind thoroughly and mix evenly. Weigh 10 g-20 g of the sample (nearest to 0.01g) in the porcelain crucible, add 1 mL of phosphoric acid (16.10), and carbonized under low power. The following procedure is the same as that after “and then transferred into muffle furnace” in 18.1.3.

18.1.5 Poultry, eggs, aquatic products and dairy products: mix the edible part evenly. Weigh 5 g-10 g of the sample (nearest to 0.01g) in the porcelain crucible, and carbonized under low power. The following procedure is the same as that after “and then transferred into muffle furnace” in 18.1.3.

For the dairy products, after mixed evenly, take 50.0 mL of milk in the porcelain crucible, add phosphoric acid (16.10), dry out in water bath, and carbonized under low power. The following procedure is the same as that after “and then transferred into muffle furnace” in 18.1.3.

18.2 Extraction and separation

Depending on the sample condition, pipette 25.0 mL-50.0 mL of sample solution prepared from the above-mentioned procedure and blank control solution in 125 mL separation funnels and diluted with water to 60 mL. Add 2 mL of ammonium citrate solution (16.3) and 3-5 drops of bromothymol blue indicator (16.4). Adjust pH with Ammonia solution (16.6) until solution color changes from yellow to blue. Add 10.0 mL of ammonium sulfate solution (16.2), 10 mL of DDTC solution (16.5), shaken to mix well. After stand for about 5 min, add 10.0 mL of MIBK (16.7), shaken vigorously for 1 min, stand for separation. Discard water layer, and MIBK layer is released into a 10 mL graduated tube for use. Pipette 0.00, 0.25, 0.50, 1.00, 1.50 and 2.00 mL (equivalent to 0.0, 2.5, 5.0, 10.0, 15.0 and 20.0 µg of lead respectively) of standard lead working solutions in 125 mL separatory funnels respectively. The following operation procedures are the same as those for the sample.

18.3 Determination

18.3.1 Beverage, liquor and packaging material soaking solution can be introduced to the equipment for determination directly after the extraction.

18.3.2 During the introduction of extraction solution sample, the acetylene gas flow can be reduced appropriately.

18.3.3 Equipment reference conditions: Hollow cathode lamp current 8 mA; resonance line 283.3 nm; slot 0.4 nm; air flow rate 8 L/min; height of burner 6 mm.

19. Expression of results

The content of lead in the sample is calculated according to equation (3).

$$X = \frac{(C_1 - C_2) \times V_1 \times 1000}{m \times V_3 / V_2 \times 1000} \dots\dots\dots (3)$$

In which,

X -- Lead content in the sample, mg/kg or mg/L;

c₁ -- Content of lead in sample solution for determination, µg/mL;

c₀ -- Content of lead in reagent blank control solution, µg/mL;

m -- Weight or volume of the sample, g or mL; V₁ -- Volume of sample extraction solution, mL;

V₂ -- Total volume of sample treatment solution, mL;

V₃ -- Total volume of sample treatment solution for determination, mL.

The results should be reported the mean of independent two results obtain under repeatability condition. The calculation results should express two significant digits.

20. Degree of precision

The absolute value of difference between two independent measurement results obtained under repeatable conditions is not allowed to exceed 20% of the arithmetic average of them.

Method 4: Disulfide hydrazone colorimetry

21. Principle

After sample digestion, the lead ions form a red complex with disulfide hydrazone at pH 8.5-9.0 and then dissolve in chloroform. Add Ammonium citrate, potassium cyanide and hydroxylamine hydrochloride to eliminate the interference brought by iron, copper and zinc ions, compared with standard series to yield quantitative lead content.

22. Reagents and materials

22.1 Ammonia (1+1).

22.2 Hydrochloric acid (1+1): Add 10 hydrochloric into 100 mL of water.

22.3 Phenol red indicator solution (1 g/L): Dissolve 0.10 g of phenol red in a small amount of ethanol for many times, transfer into a 100 mL volumetric flask and diluted.

22.4 Hydroxylamine hydrochloride solution (200 g/L): weigh 20.0 g of hydroxylamine hydrochloride in 50 mL of water, add with 2 drops of phenol red indicator solution and adjust pH to 8.5-9.0 (after the color changes from yellow to red, 2 more drops are added) with ammonia (1+1). Extract with Disulfide hydrazone-chloroform solution (22.10) for several times, until the green color of chloroform layer does not change any more. Wash twice with chloroform until chloroform layer is discarded, acidified water layer with HCl (1+1) and diluted to 100 ml.

22.5 Ammonium citrate solution (200 g/L): Dissolve 50 g of ammonium citrate in

water, add 2 drops of phenol red indicator solution and adjust pH to 8.5-9.0 with ammonia (1+1). Extract with Disulfide hydrazone-chloroform solution (22.10) for several times, each time use 10-20 ml, until the green color of chloroform layer does not change any more. Discard chloroform layer. Water layer is washed twice with chloroform. Each time use 5 ml chloroform. Chloroform layer is discarded, and water layer is diluted to 250 ml.

22.6 Potassium cyanide solution (100 g/L): Dissolve 10.0 g of potassium cyanide in water and diluted to 100 mL.

22.7 Chloroform: shall not contain Oxides.

22.7.1 Inspection method: take 10 mL of chloroform, add 25 mL of freshly boiled water, shaken for 3 min, and stand until full phase separation. Pipette 10 mL of water layer adds several drops of potassium iodide solution (150 g/L) and starch indicator solution. After mixing, the solution shall not appear blue.

22.7.2 Treatment method: A certain amount of chloroform is washed by 1/10-1/20 equivalent volume of sodium thiosulfate solution (200 g/L) and water, dehydrated by a small amount of anhydrous calcium

chloride, and distilled. The initial 1/11 and last 1/10 of the distillate are discarded, and the middle part of it is collected for use.

22.8 Starch indicator solution: dissolve 0.5 g of soluble starch in 5 mL of water, mixed well and slowly poured into 100 mL of boiling water with agitation. After having boiled, the solution is allowed to cool down. It should be prepared right before use.

22.9 Nitric acid (1+99): 1 mL of nitric acid is added into 99 mL of water.

22.10 Disulfide hydrazone chloroform solution (0.5 g/L): It should be stored in the refrigerator and, if necessary, purified by the following method.

Weigh 0.5 g of ground fine disulfide hydrazone dissolve in 50 mL of chloroform. If it does not dissolve completely, filtrate with filter paper, transfer into a 250 mL separatory funnel, and extract with ammonia (1+99) for three times with a volume of 100 mL each time. Filtrate the extract with cotton into a 500 mL separatory funnel, and adjusted pH lower than 7 with hydrochloric acid (1+1). In the acidic system, extract disulfide hydrazone precipitates with chloroform for 2-3 times with a volume of 20 mL each time. Chloroform layers are combined, washed twice with equivalent amount of water, and evaporate in 50°C water bath until all chloroform is evaporated. Refined disulfide hydrazone is then stored in a desiccator with sulphuric acid for future use. Or as an alternative, the disulfide hydrazone precipitate can be extracted by chloroform for three times with a volume of 200, 200 and 100mL, respectively. The chloroform layers are then combined and used as disulfide hydrazone solution.

22.11 Disulfide hydrazone working solution: dilute 1.0 mL of disulfide hydrazone solution To 10 mL with chloroform and mixed evenly. In 1 cm cuvette, measure absorbance

(A) under 510 nm, adjust zero point with chloroform. Calculate the volume (V) of disulfide hydrazone solution required for the preparation of 100 mL of disulfide hydrazone working solution with a transmittance of 70% by formula 4.

$$V = \frac{10 \times (2 - \lg 70)}{A} = \frac{1.55}{A} \dots\dots\dots (4)$$

22.12 Nitric acid-sulphuric acid mixed solution (4 +1).

22.13 Standard lead solution (1.0 mg/ml): Weigh 0.1598 g of lead nitrate and added 10 mL of nitric acid (1+99). After full dissolving, transfer into a 100 mL volumetric flask and make up to the mark

22.14 Standard lead working solution (10.0 ug/ml): Pipette 1.0 mL of standard lead solution in a 100 mL volumetric flask and make up to the mark.

23. Equipment and facilities

23.1 Spectrophotometer

23.2 Balance: sense of balance is 1 mg

24. Analysis procedure

24.1 Sample pretreatment

The same as the operation in 6.1.

24.2 Sample digestion

24.2.1 Nitric acid-sulfuric acid method

24.2.1.1 Grain, bean vermicelli, bean noodle, bean dry product, pastry, tea and other solid food with low moisture content: weigh 5 g-10 g of crushed sample a 250 mL-500 mL nitrogen determination flask, wet the samples with small amount of water, add several glass beads and 10 mL-15 mL of nitric acid, stand for a moment and then heated slowly under low power. After the reaction slows down, cool down naturally. Along the glass wall, and introduced 5 mL or 10 mL of sulphuric acid, heat again. After the liquid in the flask turns brown, introduce nitric acid into the flask along the glass wall continuously until the organic matter decomposes completely. The power is then increased until white smoke is generated. After all the white smoke in the flask has gone, the regeneration of white smoke is an indication of complete digestion. This solution should be transparent and colorless or slightly yellow. Cool down. (During operations, be careful to avoid explosive boiling and explosion) Add 20 mL of water and heated until it boils to remove remaining nitric acid until white smoke generated. This process is repeated two times. Cool down. Transfer into a 50 mL or 100 mL volumetric flask. Wash the nitrogen determination flask with water and transfer into the volumetric flask. Cool down, diluted with water to the mark, and mixed evenly. In the final solution, 1ml is equivalent to 1g of sample and addition of 1ml sulphuric acid. Prepare reagent blank with the same amount of nitric acid and sulphuric acid with the same operation steps

24.2.1.2 Vegetable and fruit: weigh 25.00 g or 50.00 g (nearest to 0.01g) of clean, homogenate sample in a 250 mL-500 mL nitrogen determination flask, and add several glass beads and 10 mL-15 mL of nitric acid. The following procedure is the same as that after “stand for a moment” in 24.2.1.1. But in the final solution here, 10ml is equivalent to 5g of sample and addition of 1ml sulphuric acid.

24.2.1.3 Sauce, soy sauce, vinegar, cold drink, tofu, fermented bean curd and sauce preserved vegetable: weigh 10g or 20g (nearest to 0.01g) of sample or pipette 10.0 mL or 20.0 mL of liquid sample in a 250 mL-500 mL nitrogen determination flask, added several glass beads and 5 mL-15 mL of nitric acid. The following procedure is the same as that after “stand for a moment” in 24.2.1.1. But in the final solution here, 10ml is equivalent to 2g or 2 ml of sample

24.2.1.4 Alcohol beverage or carbon dioxide beverage: pipette 10.00 mL or 20.00 mL of sample in a 250 mL-500 mL nitrogen determination flask, add several glass beads, heated under low power to remove ethanol or carbon dioxide, then add 5 mL-10 mL of nitric acid, and mix evenly. The following procedure is the same as that after “stand for a moment” in 24.2.1.1. But in the final solution here, 10ml is equivalent to 2 ml of sample

24.2.1.5 Food with high sugar content: weigh 5g or 10 g (nearest to 0.01g) of sample in a 250 mL-500 mL nitrogen determination flask, add a little water to wet, add several glass beads and 5 mL-10 mL of nitric acid, and shaken to mix well. Into the flask slowly introduce 5 mL or 10mL of sulphuric acid. After the reaction slows down and the bubbling stops, lowly heat with low power (sugar is subject to carbonize) and add more nitric acid continuously along the glass wall. After all bubbles disappear, the power is increased until the organic matter decomposes completely and white smoke is generated. This solution should be transparent and colorless or slightly yellow. It is then allowed to cool down. The following procedure is the same as that after “add 20 mL of water and heated until it boils” in 24.2.1.1.

24.2.1.6 Aquatic product: weigh 5 g or 10 g (nearest to 0.01g, lower for marine algae and shellfish) homogenized the edible part of the sample in a 250 mL-500 mL nitrogen determination flask, added glass beads and 5 mL-10 mL of nitric acid, and mixed evenly. The following procedure is the same as that after “Along the glass wall introduced 5 mL or 10 mL of sulphuric acid” in 24.2.1.1.

24.2.2 Ashing

24.2.2.1 Grain and other foods with low moisture content: weigh 5 g of the sample (nearest to 0.01g) in a quartz or porcelain crucible, heated until it is carbonized, transfer into muffle furnace to ash 3 h at 500°C. After cooling down, the crucible is taken out, add nitric acid (1+1) to wet the ash, heated with low power to evaporate water, burnt for 1 h at 500°C, then cool down. Then take out the crucible, add 1 mL of nitric acid (1+1) and heat to dissolve the ash content. Transfer into a 50 mL volumetric flask. Wash the crucible into the volumetric flask. Make up to the mark. Mixed evenly for use.

24.2.2.2 Food with high moisture content or liquid sample: weigh 5.0 g or 5.0 mL of the In an evaporating dish, heat in water bath to evaporate water. The following procedure is the same as that after “heated until it is carbonized” in 24.2.2.1.

24.3 Determination

24.3.1 Pipette 10.0 mL of sample solution after digestion and the same volume of reagent blank control solution in 125 mL separatory funnels and diluted with water to 20 mL respectively.

24.3.2 Pipette 0, 0.10, 0.20, 0.30, 0.40 and 0.50 mL (equivalent to 0.0, 1.0, 2.0, 3.0, 4.0

and 5.0 µg of lead) of standard lead working solutions in 125 mL separatory funnels, and diluted with nitric acid (1+99) to 20 mL. Add 2.0 mL ammonium citrate solution (200 g/L), 1.0 mL of hydroxylamine hydrochloride solution (200 g/L) and 2 drops of phenol red indicator solution into the sample digestion solution, reagent blank control solution and standard lead solution. Adjusted by ammonia (1+1) until the color turns red. Add 2.0 mL of potassium cyanide solution (100 g/L), mixed well, then add 5.0 mL of disulfide hydrazone working solution, and shaken vigorously for 1 min. Stand to full phase separation. After filtration with degreased cotton, pipette a certain amount of sample from chloroform layer into a 1 cm cuvette. Chloroform is used for the zero point adjustment and absorbance is measured at a wavelength of 511 nm. After subtracted by the absorbance of sample with a concentration of 0, each absorbance is used for the preparation of standard curve or for the calculation of unary regression equation. The sample absorbance is compared with standard curve.

25. Expression of results

The content of lead in the sample is calculated according to equation (5).

$$X = \frac{(m_1 - m_2) \times 1000}{m_3 \times V_1 / V_2 \times 1000} \dots\dots\dots (5)$$

In which,

X -- Lead content in the sample, mg/kg or mg/L;

m₁ -- Weight of lead in sample solution for determination, µg;

m₂ -- Weight of lead in reagent blank control solution, µg;

m₃ -- Weight or volume of the sample, g or mL;

V₁ -- Total volume of sample treatment solution, mL;

V₂ -- Total volume of sample treatment solution for determination, mL.

Report the mean of two independent results under repeatability condition with two significant digits.

26. Degree of precision

The absolute difference between two independent measurement results obtained under repeatability conditions is not allowed to exceed 11% of the arithmetic average of them.

Method 5: Single-sweep polarography

27. Principle

After sample digestion, in acidified circumstance (Pb^{2+}) and I^- forms PbI_2 -complex ions. The complex possesses electrical activity and generates reduction current on dropping mercury electrode. The peak current varies linearly with lead content and is compared with standard series to yield quantitative lead content.

28. Reagents and materials

28.1 Base solution: dissolve 5.0 g of potassium iodide, 8.0 g of potassium sodium tartrate and 0.5 g of ascorbic acid in a 500 mL beaker with 300 mL of water, add 10 mL of hydrochloric acid, transfer into a 500 mL volumetric flask and make up to the mark (it is stored in refrigerator and can be preserved for 2 months).

28.2 Lead standard stock solution (1.0 mg/mL): Weigh accurately 0.1000 g of lead (purity 99.99%) in a beaker, add 2 mL of nitric acid solution (1+1), and heated to dissolve. After cooling down, the solution is transferred into a 100 mL volumetric flask and diluted with water to 100 mL and mixed evenly.

28.3 Lead standard working solution (10.0 µg/mL): Prior to the use of lead solution, pipette

1.00 mL of lead standard stock solution in a 100 mL volumetric flask, make up to the mark and mixed evenly.

28.4 Mixed acid: Nitric acid-perchloric acid (4+1). 80 mL of nitric acid is added with 20 mL of perchloric acid and mixed evenly.

29. Equipment and facilities

29.1 Polarographic analyzer.

29.2 Universal electric furnace with an electronic regulator.

30. Analysis procedure

30.1 Reference conditions for polarographic analysis

Single-sweep polarography (SSP). Initial potential: -350 mV; final potential: -850 mV; sweep speed: 300 mV/s; three electrodes, second derivative, stationary time: 5 s; appropriate measurement range. The peak current of lead is recorded at the peak potential of -470 mV.

30.2 Preparation of standard curve

Pipette 0, 0.05, 0.10, 0.20, 0.30 and 0.40 mL (equivalent to 0, 0.5, 1.0, 2.0, 3.0 and

4.0 µg of lead) of standard lead solutions in 10 mL colorimetric tubes, diluted with base solution to 10.0 mL and mixed evenly. The tubes are transferred into the electrolytic cell one by one and place a three-electrode system. The determination can be carried out under the above-mentioned reference conditions. Record the peak current. Plot the standard curve with peak current against lead content

30.3 Sample treatment

Remove impurities, sieve with 20 mesh sieve and grind for the low moisture content samples like grain, bean ect. Homogenizing the high moisture content samples like vegetable, fruit, fish and meat ect. And store in plastic bottle

30.3.1 Sample treatment (including grain, bean, pastry, tea and meat, except for salt and white sugar): Weigh 1 g-2 g of sample in a 50 mL flask, add 10 mL-20 mL of mixed acid, and soaked overnight with a cover on the top. Then the flask is heated by the universal electric furnace with an electronic regulator with low power. If the color of digestion solution turns darker gradually and appears dark brown, the flask is taken out from the universal electric furnace to cool down, add an appropriate amount of nitric acid, and heat again to continue digestion. When the color of the solution no longer dark, starts to appear transparent and colorless or slightly yellow, and emits white smoke, the solution can be heated with high power to remove residual acid solution. When most of the liquid has evaporated, the system should be heated with low power to yield a white residue, which will be used for determination. Meanwhile, the reagent is used to provide blank control.

30.3.2 Salt and white sugar: weigh 2.0 g of the sample in a beaker for use.

30.3.3 Liquid sample

Weigh 2 g of sample (nearest to 0.1g) in a 50 mL flask (the sample containing ethanol or carbon dioxide should be heated in 80oC water bath to remove them), add 1 mL-10 mL of mixed acid, heated by the universal electric furnace with an electronic regulator at low power. The following procedure is the same as that after "Sample treatment" in 30.3.1. The sample is ready for the determination.

30.4 Sample determination

Add 10.0 mL of base solution into the above sample and reagent blank control bottle, respectively, to dissolve the residue. Transferred into the electrolytic cell. The following procedure is the same as that after "Preparation of standard curve" in 30.2. Record the peak currents. Calculate the lead concentration in the standard curve.

31. Expression of results

The lead content in the sample is calculated on the basis of equation (6).

$$X = \frac{(A-A_0) \times 1000}{m \times 1000} \dots\dots\dots (6)$$

In which,

X -- Lead content in the sample mg/kg or mg/L;

A -- Weight of lead in the sample solution read from standard curve, µg;

A₀ -- Weight of lead in the reagent blank control solution read from standard curve, µg;

m -- Weight or volume of the sample, g or mL;

Report the mean of two independent results under repeatability condition with two significant digits.

32. Degree of precision

The absolute difference between two independent measurement results obtained under repeatability conditions is not allowed to exceed 5.0% of the arithmetic average of them.

33. Other

The detection limit: graphite furnace atomic absorption spectrometry: 0.005 mg/kg; hydride generation atomic fluorescence spectrometry: 0.005mg/kg for solid and 0.001mg/kg for liquid; flame atomic absorption spectrometry: 0.1mg/kg; single-sweep polarography: 0.25 mg/kg ; disulfide hydrazone colorimetry: 0.085 mg/kg.

Appendix A

(Informative reference)

A.1 Polarograms of lead in reagent blank control and standard lead solution are shown in Figures A.1

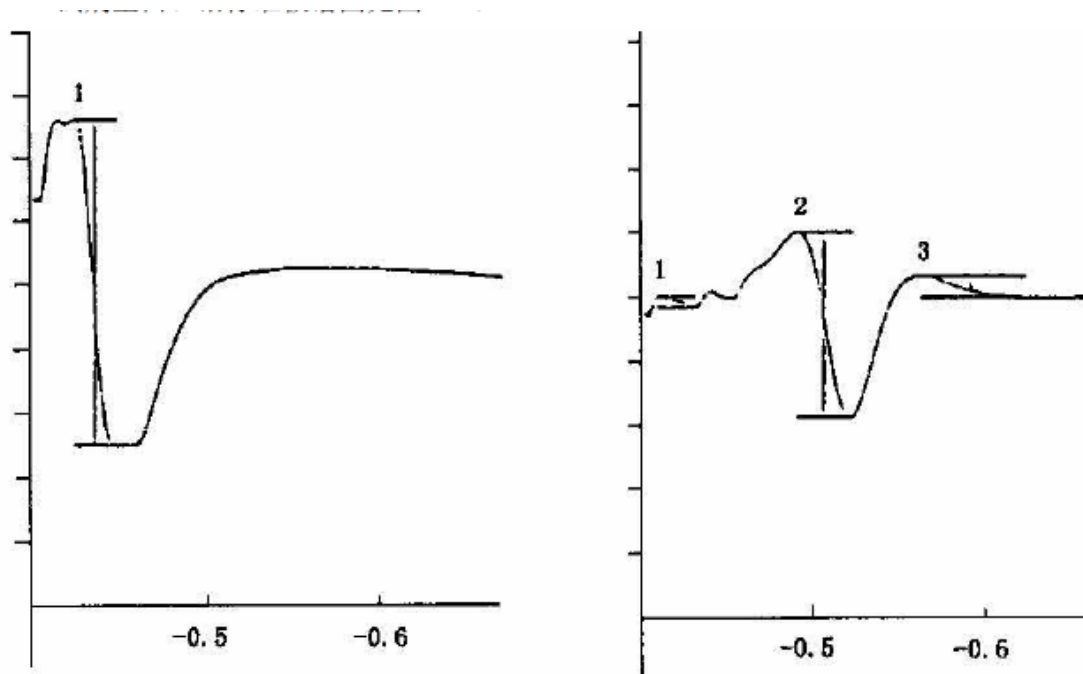


Figure 1 Polarograms of lead in a) reagent blank control; b) standard lead solution

GB 5009.123-2014 Determination of Chromium in Foods



National Standards of People's Republic of China

GB 5009.123-2014

**National Food Safety Standard
Determination of Chromium in Foods**

Issued on: 2015-01-28

Implemented on: 2015-07-28

Issued by National Health and Family Planning Commission

Foreword

This standard will replace GB/T 5009.123-2003 “Determination of Chromium in Foods”.

Compared to GB/T 5009.123-2003, this standard maintains the following major changes:

- Name of the standard is modified into “National Standard for Food Safety - Determination of Chromium in Foods”;
- Specimen pre-treatment is increased with microwave digestion and wet digestion;
- Limit of Quantitation (LOQ) is added;
- Ammonium dihydrogen phosphate instead of ammonium phosphate is used as matrix modifier;
- Method Two Oscillopolarography Law is deleted.

National Standard for Food Safety

Determination of Chromium in Foods

1. Scope

This standard specifies the determination of chromium in foods by graphite furnace atomic absorption spectrometric method.

This standard applies to determination of chromium content in various types of foods.

2. Principle

After the specimen digests, the graphite furnace atomic absorption spectrometric method will be adopted to measure the absorbance at 357.9 nm. Compare the absorbance to limit of qualification of the standard series solution under the given concentration range.

3. Reagents and materials

Note: except otherwise specified, all reagents used in this method are guaranteed pure reagents and water are Class II water specified in GB / T6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid (HClO_4).

3.1.3 Ammonium dihydrogen phosphate ($\text{NH}_4 \text{H}_2\text{PO}_4$)

3.2 Reagent compounding

3.2.1 Nitric acid solution (5 + 95): measure and take 50 ml of nitric acid, slowly pour it into 950 ml of water, and mix it well.

3.2.2 Nitric acid solution (1 + 1): measure and take 250 ml of nitric acid, slowly pour it into 250 ml of water, and mix it well.

3.2.3 Ammonium dihydrogen phosphate solution (20 g/l): weigh and take 2.0 g of ammonium dihydrogen phosphate, dissolve it into water and dilute it to 100 ml, and mix it well.

3.3 Standard products

Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$): purity > 99.5%, or reference materials certified by and granted with reference materials certificate by the State.

3.4 Formulation of standard solution

3.4.1 Chromium standard stock solution: weigh 1.4315 g (accurate to 0.0001 g) of the reference material potassium dichromate (110°C baked for 2h), dissolve it into water and transfer it into 500 ml volumetric flask, dilute it with nitric acid solution (5+ 95) to the mark, and mix it well. This solution contains 1.000 mg of chromium per milliliter. Chromium standard stock solution certified by the State and granted with reference material certificate shall be purchased.

3.4.2 Chromium standard use solution: dilute chromium standard use solution by nitric acid solution (5 + 95) progressively till the content of chromium per ml is 100 ng.

3.4.3 Preparation of standard series solutions: imbibe and take respectively 0 ml, 0.500 ml, 1.00 ml, 2.00 ml, 3.00 ml, and 4.00 ml of chromium standard use solution (100 ng/ml) and transfer it to 25 ml volumetric flask; dilute it with nitric acid solution (5 + 95) to the mark, and mix it well. Chromium content per milliliter in each volumetric flask shall be 0 ng, 2.00 ng, 4.00 ng, 8.00 ng, 12.0 ng, and 16.0 ng respectively. Or, graphite furnace automatic specimen might be adopted for automatic preparation.

4. Equipment and facilities

Note: all glassware applied shall be soaked in nitric acid solution (1 + 4) for 24h or above, washed with water repeatedly, and finally washed and rinsed with deionized water.

4.1 Atomic absorption spectrometer, equipped with graphite furnace atomizer, and attached with chromium hollow cathode lamp.

4.2 Microwave digestion system, equipped with inner digestion tank.

4.3 Adjustable electric furnace.

4.4 Adjustable heating plate.

4.5 Pressure digestion device, equipped with inner digestion tank.

4.6 Muffle furnace.

4.7 Constant temperature drying oven.

4.8 Electronic balance: a sensor volume of 0. 1 mg and 1 mg.

5. Analytical procedures

5.1 Pretreatment of specimen

5.1.1 Crush and load grains, beans and other foodstuffs into a clean container after foreign matters are removed, and use them as specimen. Seal and label clearly; specimens shall be stored at room temperature.

5.1.2 Fresh specimens with higher content of water such as vegetables, fruits, fish, meat and eggs shall be directly refined into homogenized solution, loaded into clean container and used as specimen; seal and label clearly; specimen shall be stored in refrigerator freezer.

5.2 Specimen digestion

5.2.1 Microwave digestion

Accurately weigh and take 0.2g~0.6g (accurate to 0.001g) of specimen and transfer it into microwave digestion tank; add 5 ml nitric acid; digest specimen as per microwave digestion procedure (please refer to A.1 for microwave digestion conditions); take out the digestion tank after it cools down; clear up acid to 0.5 ml~1.0 ml on electric hot plate under the temperature of 140°C~160°C After digestion tank cools down, transfer the digestion solution to 10 ml volumetric flask, wash digestion tank by a small amount of water for 2~3 times; combined the washing liquid, dilute it by water to the mark; conduct reagent blank test at the same time.

5.2.2 Wet digestion

Weigh and take 0.5 g~3 g (accurate to 0.001 g) of specimen and transfer it into digestive tract; add 10 ml of nitric acid and 0.5 ml of perchloric acid; digest on the adjustable electric furnace (reference conditions: 120°C kept for 0.5h~1h, heated up to 180°C for 2h~4h, and heated up to 200°C~220°C). If the digestion liquid is brown in color, add nitric acid into it until white smoke rises; digestion liquid shall be colorless, transparent or slightly yellowish; take out the digestive tract, cool it down and dilute it by water to 10 ml. At the same conduct the blank test for the reagent.

5.2.3 High pressure digestion

Accurately weigh and take 0.3 g~1 g (accurate to 0.001 g) of specimen and transfer it into digestion tank; add 5 ml of nitric acid; replace the inner cover; tighten the stainless steel jacket and put it into the constant temperature oven; maintain it for 4h~5h at 140°C~160°C Cool it down naturally to room temperature within the constant temperature oven; slowly loosen the outer tank and take out the inner digestion tank; place it onto the adjustable electric hot plate and clear up acid to 0.5 ml~1.0 ml at 140°C~160°C After cooling, transfer the digestion liquid into a 10 ml volumetric flask, and wash the inner tank by a small amount of water for 2~3 times. Combine the wash lotion into the volumetric flask and dilute it by water to the mark; at the same, conduct the blank test for the reagent.

5.2.4 Dry ashing

Weigh and take 0.5 g~3 g (accurate to 0.001 g) of specimen and transfer it into a crucible; simmer till it is carbonized and smokeless; transfer it into a muffle furnace; maintain it for 3h~4h at 550°C Take it out and cool down. For specimen that is not fully ashed, add a few drops of nitric acid, simmer, and evaporate carefully; then transfer it into high temperature furnace of 550°C and continue ashing treatment for 1h~2h till the specimen is lime-like; take it out from the high-temperature furnace and cool down; dissolve it by nitric acid solution (1 + 1) and dilute it with water to 10 ml; at the same time conduct the blank test for the reagent.

5.3 Determination

5.3.1 Test conditions of instrument

Calibrate to the optimal state as per performance of the respective instrument. For reference conditions, please refer to A.2.

5.3.2 Standard curve mapping

Take respectively 10 µl (you can select the best injection volume as per the instrument applied) of standard series solution working solution bottom up in terms of concentration, inject it into graphite tube, and measure its absorbance after it is being atomized; take concentration as the horizontal axis and absorbance vertical axis and map the standard curve.

5.3.3 Specimen determination

Under the same experimental conditions under which the standard solution is determined, take respectively 10 µl of the blank solution and specimen solution (the best injection volume might be subject to the instrument applied); inject them into graphite tube; measure its absorbance value after it is being atomized; compare to the limit of quantification of the standard series solution.

Inject 5 µl (the best injection volume can be subject to the instrument applied) of ammonium dihydrogen phosphate solution (20.0 g/l) into the interfered specimen (preparation process of the standard series solution specified in 5.3.3 shall be followed).

6. Statement of analysis result

For calculation of chromium content in the specimen, please refer to Formula (1):

$$X = \frac{(c - c_0) \times V}{m \times 1\,000} \dots\dots\dots (1)$$

In the formula:

X- chromium content in the specimen, in milligram per kilogram (mg/kg);

c- determination of chromium content in the specimen solution, in nanogram per milliliter (ng/ml);

c₀- chromium content in blank solution, in nanogram per milliliter (ng/ml);

V- total constant volume of specimen digestion solution, in milliliter (ml);

m- volume of specimen weighed, in gram (g);

1 000 - conversion coefficient.

When the analysis result ≥1 mg/kg, keep a three-digit valid number; when the analysis result < 1mg/kg, keep a two-digit valid number.

7. Precision

Absolute difference between two independent determination results obtained under the repeatability conditions shall not exceed 20% of their arithmetic mean.

8. Others

Calculate by 0.5 g of the specimen weighed and dilute it to 10 ml; the detection limit in such case shall be 0.01 mg/kg, and the limit of quantification 0.03 mg/kg.

Appendix A

Reference Conditions for Specimen Determination

A.2 Please refer to Table A for reference conditions for microwave digestion

Table A.1 Reference conditions for microwave digestion

Step	Power (1,200w), change/%	Set temperature / °C	Warm-up time /min	Holding time /min
1	0~80	120	5	5
2	0~80	160	5	10
3	0~80	180	5	10

A.3 For reference conditions of graphite furnace atomic absorption spectrometry, please refer to Table A.2.

Table A.2 Reference conditions of graphite furnace atomic absorption spectrometry

Element	Wavelength/nm	Slits /nm	Lamp current /mA	Drying /(°Cs)	Ashing (°Cs)	Atomization (°Cs)
Chromium	357.9	0.2	5~7	(85~120)/(40~50)	900/(20~30)	2,700(4~5)

GB 5009.15-2014 Determination of Cadmium in Foods



National Standards of People's Republic of China

GB 5009.15-2014

National Food Safety Standard
Determination of Cadmium in Foods

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Issued by Ministry of Health of the People's Republic of China

Foreword

This standard replaces GB/T 5009.15-2003 “Determination of cadmium in foods”. The main changes in this standard compared to GB/T 5009.15-2003 are as follows:

- The title of the standard is revised to “National food safety standard determination of cadmium in foods”
- Method 2 Atomic absorption spectrometry, Method 3 Colorimetry, and Method 4 Atomic fluorescence spectrometry, are deleted.

National Food Safety Standard

Determination of Cadmium in Foods

1 Scope

This standard specifies a graphite furnace atomic absorption spectrometric method for determination of cadmium in all kinds of food.

The standard is applicable to the determination of cadmium in all kinds of foods.

2 Principle

The test sample is ashed or digested with acid whereupon a specific amount of the digest is injected into the atomic absorption spectrophotometer graphite furnace and electrothermally atomised, absorbing the 228.8 nm resonance line; within a specific concentration range the absorbance is directly proportional to cadmium content, which is determined by the standard curve method.

3 Reagents and materials

Note 1: Unless otherwise indicated, all reagents used in this method are analytically pure; the water is Class 2 water specified in GB/T 6682.

Note 2: All glassware must be soaked in nitric acid solution (1+4) for not less than 24 h, rinsed repeatedly with water, and finally rinsed with deionised water and dried.

3.1 Reagents

- 3.1.1 Nitric acid (HNO₃): guaranteed reagent.
- 3.1.2 Hydrochloric acid (HCl): guaranteed reagent.
- 3.1.3 Perchloric acid (HClO₄): guaranteed reagent.
- 3.1.4 Hydrogen peroxide (H₂O₂, 30%).
- 3.1.5 Ammonium dihydrogen phosphate (NH₄H₂PO₄).

3.2 Preparation of reagents

- 3.2.1 Nitric acid solution (1%): Add 10.0 mL of nitric acid to 100 mL of water and dilute to 1,000 mL.
- 3.2.2 Hydrochloric acid solution (1+1): Slowly add 50 mL of hydrochloric acid to 50 mL of water.
- 3.2.3 Mixed nitric acid-perchloric acid solution (9+1): Mix 9 parts of nitric acid with 1 part of perchloric acid.
- 3.2.4 Ammonium dihydrogen phosphate solution (10 g/L): Weigh out 10.0 g of ammonium dihydrogen phosphate, dissolve with 100 mL of nitric acid solution (1%) and transfer quantitatively to a 1,000 mL volumetric flask; make up to the graduation mark with nitric acid solution (1%).

3.3 Reference standard material

A standard sample of cadmium metal (Cd) of 99.99% purity or a nationally authenticated reference material holding a reference material certificate.

3.4 Preparation of standard solutions

3.4.1 Cadmium standard stock solution (1,000 mg/L): Accurately weigh 1 g (accurate to 0.0001 g) of the standard sample of cadmium metal into a small beaker, dissolve with 20 mL of hydrochloric acid solution (1+1) added in portions, then add 2 drops of nitric acid; transfer to a 1,000 mL volumetric flask, make up to the graduation mark with water and mix. Alternatively, purchase nationally authenticated reference material holding a reference material certificate.

3.4.2 Cadmium standard intermediate working solution (100 ng/mL): Pipette 10.0 mL of the cadmium standard stock solution into a 100 mL volumetric flask and make up to the graduation mark with nitric acid solution (1%); dilute repeatedly this way to obtain a standard intermediate working solution containing 100.0 ng of cadmium per millilitre.

3.4.3 Standard curve working solutions: Accurately pipette 0 mL, 0.50 mL, 1.0 mL,

1.5 mL, 2.0 mL and 3.0 mL of the cadmium standard intermediate working solution into 100 mL volumetric flasks and make up to the graduation mark with nitric acid solution (1%) to obtain a standard series of solutions containing cadmium 0 ng/mL, 0.50 ng/mL, 1.0 ng/mL, 1.5 ng/mL, 2.0 ng/mL and 3.0 ng/mL, respectively.

4 Instrumentation and apparatus

4.1 Atomic absorption spectrophotometer, with graphite furnace.

4.2 Cadmium hollow cathode lamp.

4.3 Electronic balance: Sensibility reciprocal 0.1 mg and 1 mg.

4.4 Adjustable temperature hotplate, adjustable temperature electric oven.

4.5 Muffle furnace.

4.6 Thermostatic drying cabinet.

4.7 Pressure digester, pressure digestion vessel.

4.8 Microwave digestion system: Fitted with polytetrafluoroethylene or other suitable pressure vessel.

5 Analytical procedure

5.1 Test sample preparation

5.1.1 Dry test samples: For grain and pulses, exclude foreign matter; for nuts remove foreign matter and shells. Grind to a homogeneous sample of particle size not greater than 0.425 mm. Store in a clean plastic bottle and label clearly; hold ready for use at room temperature, or under storage conditions consistent with the sample.

5.1.2 Fresh (wet) test samples: Vegetables, melons, meats, fish, eggs, etc, are homogenised with a food processor or milled to a homogenate; store in a clean plastic bottle, label clearly, and hold in a refrigerator at -16°C to -18°C pending use.

5.1.3 Liquid test samples: Hold under storage conditions consistent with the sample pending use. Degas gas-containing samples before use.

5.2 Digestion of test sample

The sample may be digested using any one of the following methods according to laboratory circumstances. Sample homogeneity should be assured in weighing.

a) Digestion with a pressure digestion vessel: Weigh 0.3 g - 0.5 g (accurate to 0.0001

g) of the dry test sample or 1 g - 2 g (accurate to 0.001 g) of the fresh (wet) test sample into the polytetrafluoroethylene liner vessel, add 5 mL of nitric acid and soak overnight. Then add 2 mL - 3 mL of hydrogen peroxide solution (30%) (the total volume may not exceed one third of the vessel capacity). Cover the liner vessel and tightly screw on the stainless steel case; place in the thermostatic drying cabinet, hold at 120°C-160°C for 4 h - 6 h, and allow to cool naturally to room temperature inside the cabinet. After opening the vessel, heat to near dryness to drive off acid. Wash the digest into a 10 mL or 25 mL volumetric flask, rinse the liner vessel and inner cover 3 times with a little nitric acid solution (1%), combine the washings in the volumetric flask, make up to the graduation mark with nitric acid solution (1%), and mix ready for use. Run a reagent blank test in parallel.

b) Microwave digestion: Weigh out 0.3 g - 0.5 g (accurate to 0.0001 g) of the dry test sample or 1 g - 2 g (accurate to 0.001 g) of the fresh (wet) test sample, place in the microwave digester, and add 5 mL of nitric acid and 2 mL of hydrogen peroxide. The microwave digestion programme can be adjusted to provide the optimum conditions for the instrument model. On completion of digestion, the digestion vessel is left to cool and then opened; the digest is colourless or pale yellow. Heat to near dryness to drive off acid, wash the digestion vessel 3 times with a little nitric acid solution (1%), transfer the solution to a 10 mL or 25 mL volumetric flask, make up to the graduation mark with nitric acid solution (1%), and mix ready for use. Run a reagent blank test in parallel.

c) Wet digestion: Weigh 0.3 g - 0.5 g (accurate to 0.0001 g) of the dry test sample or 1 g - 2 g (accurate to 0.001 g) of the fresh (wet) test sample into a conical flask, introduce several glass beads, add 10 mL of nitric acid-perchloric acid mixture (9+1), cover and soak overnight; with a small funnel added, digest on the electric hotplate. If the digest turns black-brown, add further nitric acid until white fumes are given off and the digest is colourless and transparent or tinged faint yellow, and leave to cool. Then wash the digest into a 10 mL - 25 mL volumetric flask, rinse the conical flask 3 times with a little nitric acid solution (1%), combine the washings in the graduated flask make up to the graduation mark with nitric acid solution (1%), and mix ready for use. Run a reagent blank test in parallel.

d) Dry ashing: Weigh 0.3 g - 0.5 g (accurate to 0.0001 g) of the dry test sample, 1 g - 2 g (accurate to 0.001 g) of the fresh (wet) test sample or 1 g - 2 g (accurate to 0.001 g) of the liquid sample into a porcelain crucible; gently carbonise in the adjustable temperature electric oven until smoke is no longer emitted, then transfer to the muffle furnace, ash for 6 h - 8 h at 500°C and cool. If a sample has not been completely ashed, add 1 mL of mixed acid and heat gently on the adjustable temperature electric oven; once the mixed acid has evaporated to dryness, transfer the sample back into the muffle furnace and continue ashing at 500°C for 1 h - 2 h until fully digested and the ash is greyish white or grey. Leave to cool, dissolve the ash with nitric acid solution (1%), transfer the sample digest to a 10 mL or 25 mL volumetric flask, wash the porcelain crucible 3 times with a little nitric acid solution (1%), and combine the washings in the volumetric flask; make up to the graduation mark with nitric acid solution (1%) and mix ready for use. Run a reagent blank test in parallel.

Note: The experiment must be conducted in a well ventilated fume cupboard. Wherever possible avoid digesting oil/fat-containing samples by wet digestion; digestion by the dry method is best. If digestion by the wet method is essential, the amount of sample taken should not exceed a maximum of 1 g.

5.3 Instrumentation reference conditions

Adjust instruments to the optimum conditions for the model used. The reference operating conditions for the atomic absorption spectrophotometer (with graphite furnace and cadmium hollow cathode fitted) are as follows:

- ☐ Wavelength 228.8 nm, slit 0.2 nm - 1.0 nm, lamp current 2 mA - 10 mA, drying temperature 105°C, drying time 20 s;
- ☐ Ashing temperature 400°C - 700°C, ashing time 20 s - 40 s;
- ☐ Atomisation temperature 1300°C - 2300°C, atomisation time 3 s - 5 s;
- ☐ Background correction is by deuterium lamp or Zeeman effect

5.4 Construction of standard curve

Proceeding from low to high concentration, inject into the graphite furnace 20 µL of each standard curve working solution; measure the absorbance, plot the standard curve with the concentration of the standard curve working solution as horizontal coordinate and the corresponding absorbance as vertical coordinate, and find the simple linear regression equation for the relation of absorbance versus concentration.

The standard series solutions should be cadmium standard solutions of no fewer than 5 different concentrations, and the coefficient of correlation should not be less than 0.995. If an automated sample injection unit is available, programmed dilution may be used to generate the standard series.

5.5 Examination of test sample solution

Under the same experimental conditions as in examination of the standard curve working solutions, pipette 20 µL of the sample digest (the optimum sample volume for the instrument used may be chosen), inject it into the graphite furnace, and measure the absorbance. Substitute into the simple linear regression equation of the standard series to find the content of cadmium in the sample digest, using no fewer than two replicates. If the results of measurement fall outside the range of the standard curve, make the measurements again after dilution with nitric acid solution (1%).

5.6 Use of matrix modifiers

For test samples subject to interference, inject the sample digest into the graphite furnace with 5 µL of ammonium dihydrogen phosphate matrix modifier (10 g/L). The same amount of matrix modifier as in examination of the sample must be added when the standard curve is plotted.

6 Presentation of analytical results

Calculate the cadmium content of the test sample from Equation (1).

$$X = \frac{(C_1 - C_0) \times V}{m \times 1000} \square\square\square\square \dots\dots\dots(1)$$

Where:

X - Cadmium content of test sample in units of milligrams per kilogram or milligrams per litre (mg/kg or mg/L);

C1 - Cadmium content of the test sample digest in units of nanograms per millilitre (ng/mL);

C0 - Cadmium content of the blank solution in units of nanograms per millilitre (ng/mL);

V - Total final volume of the test sample digest in millilitre units (mL);

M - Test sample mass or volume in units of grams or millilitres (g or mL); 1000 ☐ conversion

The arithmetic mean of the results of two independent determinations obtained under repeatable conditions is presented, retaining two significant digits.

7 Precision

The absolute difference between the results of two independent determinations obtained under repeatable conditions shall not exceed 20% of the arithmetic mean.

8 Other particulars

The limit of detection of the method is 0.001 mg/kg and the limit of quantitation is 0.003 mg/kg.

GB 5009.16-2014 Determination of Tin in Foods



National Standards of People's Republic of China

GB 5009.16-2014

**National Food Safety Standard
Determination of Tin in Foods**

Issued on: 2015-01-28

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**Issued by National Health and Family Planning Commission
Ministry of Agriculture**

Foreword

This standard will replace GB / T 5009.16-2003 "Determination of Tin in Foods".

Compared to GB/T 5009.16-2003, this standard maintains the following major changes:

- Name of the standard is modified into "National Standard for Food Safety - Determination of Tin in Foods".
- Standard solution preparation is modified;
- Canned food specimen preparation method is increased;
- Description of the instrument measurement section is modified;
- Limit of quantification is added;
- Detection limit is modified; and
- Calculation equation is modified.

National Food Safety Standard

Determination of Tin in Foods

1. Scope

This standard specifies the determination of hydride of tin in foods by atomic fluorescence spectroscopy and phenylfluoron colorimetry.

This standard applies to determination of tin in canned solid foods, canned drinks, canned jams, and canned infant formula and supplementary foods.

Method I Hydride Generation Atomic Fluorescence Spectroscopy

2. Principle

Tin hydride (SnH_4) will be generated under the effects of sodium borohydride after the specimen digests and will be carried into the atomizer by the carrier gas for atomization. Illuminated by tin hollow cathode lamp, the tin atom in ground state will be activated to high-energy state and fluorescence of characteristic wavelengths will be emitted when such tin atoms are deactivated and return to the ground state. The fluorescence intensity of tin atoms is proportional to its tin content. Compare the limit of quantification to that of the standard series solution.

3. Reagents and materials

Note: except otherwise stated, reagents applied herein shall be the analytical pure reagents, and the water be Class II water specified in GB/T6682.

3.1 Reagents

- 3.1.1 Sulfuric acid (H_2SO_4): guaranteed pure reagent.
- 3.1.2 Nitric acid (HNO_3): guaranteed pure reagent.
- 3.1.3 Perchloric acid (HClO_4): guaranteed pure reagent.
- 3.1.4 Thiourea ($\text{CH}_4\text{N}_2\text{S}$).
- 3.1.5 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$).
- 3.1.6 Sodium borohydride (NaBH_4).
- 3.1.7 Sodium hydroxide (NaOH).

3.2 Reagent compounding

- 3.2.1 Nitric acid - perchloric acid mixture solution (4 + 1): measure and take 400 ml and 100 ml nitric acid perchlorate and mix them well.
- 3.2.2 Sulfuric acid solution (1+9): measure and take 100 ml sulfuric acid and pour it into 900 ml of water, and mix them well.

3.2.3 Thiourea (150 g/l) + a scorbic acid (150 g/l) mixture solution: measure and take respectively 15.0 g thiourea and 15.0 g ascorbic acid and dissolve them in water, dilute them to 100 ml, and place them into a brown bottle for dark preservation or extemporaneous preparation.

3.2.4 Sodium hydroxide solution (5.0 g/l): weigh and take 5.0 g of sodium hydroxide and dissolve it in 1,000 ml of water.

3.2.5 Sodium borohydride solution (7.0 g/l): weigh and take 7.0 g of sodium borohydride, and dissolve it in sodium hydroxide solution for extemporaneous preparation.

3.3 Standard products

Reference material Tin metal (Sn), reference material with a purity of 99.99% or reference material certified and granted by the State with reference material certificate.

3.4 Formulation of standard solution

3.4.1 Tin standard solution (1.0 mg/ml): weigh and take 0.1 g (accurate to 0.000 1 g) Tin metal reference material; place it into a small beaker; add 10.0 ml of sulfuric acid and cover it by pan; heat it up till tin is fully dissolved; remove the pan; continue the heating till thick white smoke occurs; cool it down and slowly add 50 ml of water; transfer it into a 100 ml flask; wash the beaker by sulfuric acid solution (1 +9) repeatedly; pour solution into volumetric flask and dilute it to the mark; mix it well.

3.4.2 Standard tin use solution (1.0 µg/ml): imbibe and take 1.0 ml tin standard solution and transfer it into a 100 ml volumetric flask; dilute it with sulfuric acid solution (1 +9) to the mark. The concentration of this solution is 10.0 µg/ml. Imbibe and take 10.0 ml of this solution and transfer it in 100 ml flask, and dilute it with sulfuric acid solution (1 + 9) to the mark.

4. Equipment and facilities

4.1 Atomic fluorescence spectrometer.

4.2 Electric hot plate.

4.3 Electronic balance: sensor volume of 0.1 mg and 1mg.

5. Analytical procedures

5.1 Specimen preparation

For canned foods, weigh and take edible contents of foods and make them into homogenized or even powder.

5.2 Specimen digestion

5.2.1 Weigh and take 1.0 g ~ 5.0 g specimen and transfer it into the conical flask; add 20.0 ml nitric acid - perchloric acid mixed solution (4 + 1); add 1.0 ml of sulfuric acid and 3 glass beads, and let it stand overnight. On the following day, place it onto electric hot plate to heat up and dissolve it; if the acid is too small, it is ok to supplement an appropriate amount of nitric acid; digest continuously till white smoke rises; take it down and cool it down when the liquid volume is close to 1 ml; transfer by water the digested specimen into the 50 ml volumetric flask, dilute it by water to mark; shake it up for use. At the same time, conduct blank test (if tin content in the specimen solution goes beyond the range indicated by the standard curve, then dilute it with

water and supplement sulfuric acid, so that the concentration of the sulfuric acid solution with constant volume will be identical with that of the standard series solution).

5.2.2 Take 10.0 ml of the specimen diluted to mark in 5.2.1 and transfer it into 25 ml colorimetric tube; add 3.0 ml of sulfuric acid solution (1 + 9); add 2.0 ml of thiourea (150 g/l) + ascorbic acid (150 g/l); mix them well and then dilute it with water to 25 ml; and shake it up for use.

5.3 Reference conditions for equipment

Reference conditions for atomic fluorescence spectrometer analysis:

- Negative high voltage: 380 V;
- Lamp current: 70 mA;
- Atomization temperature: 850°C
- Furnace height: 10 mm;
- Shrouding gas flowrate: 1, 200 ml/min;
- Carrier gas flow rate: 500 ml/ min;
- Measurement mode: standard curve method;
- Reading mode: peak area;
- Delay time: 1s;
- Reading time: 15s;
- Dosing time: 8s;
- Injection volume: 2.0 ml.

5.4 Formulation of standard series solution

Standard curve: respectively imbibe standard tin use solution 0.00 ml, 0.50 ml, 2.00 ml, 3.00 ml, 4.00 ml, 5.00 ml and transfer them to 25 ml colorimetric tubes; add sulfuric acid solution (1 + 9) 5.00 ml, 4.50 ml, 3.00 ml, 2.00 ml, 1.00 ml, and 0.00 ml into it; add 2.0 ml thiourea (150 g/l) + ascorbic acid (150 g/l) mixture solution; dilute it with water to 25 ml. The concentration of the standard series solution: 0 ng / ml, 20 ng / ml, 80 ng / ml, 120 ng / ml, 160 ng / ml and 200 ng / ml respectively.

5.5 Instrument determination

Set up the optimal measurement conditions of instrument as per 5.3 and set the appropriate parameter points as per model of instrument and the workstations applied; ignite and preheat up the instrument; determine the standard curve and specimen solution after it is preheated for 30 minutes.

6. Statement of analysis result

Tin content in the specimens is to be calculated as per Formula (1):

$$X = \frac{(c_1 - c_0) \times V_1 \times V_3}{m \times V_2 \times 1\,000} \dots\dots\dots (1)$$

In the formula:

X- tin content in specimen, in milligram per kilogram (mg/kg);

C₁- determined concentration of digested specimen solution, in nanogram per milliliter (ng / ml);

C₀- concentration of blank digestion solution of the specimen, in nanogram per milliliter (ng / ml);

V₁- constant volume of specimen digestion solution, in milliliter (ml);

V₃- constant volume of solution for purpose of measurement, in milliliter (ml);

m- specimen mass, in gram (g);

V₂- volume of specimen digestion solution taken for purpose of determination, in milliliter (ml);

1000- conversion factor.

When the calculated result is smaller than 10 mg/kg, the hundredth behind the decimal shall be kept; when the calculated result is bigger than less than 10 mg/kg, a two-digit valid number shall be retained.

7. Precision

Absolute difference between two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetic mean.

8. Others

When the specimen volume is 1.0g, the limit of quantification under this method shall be 2.5 mg/kg.

Method II Phenylfluorone Colorimetry

9. Principle

After the specimen is digested, tetravalent tin ions and phenylfluorone form sparingly soluble orange-red complex in weak acid solution, and compares limit of quantification to that of the standard series solution in the presence of protective colloid.

10. Reagents and materials

Note: except otherwise stated, the reagents applied under this method are analytical pure reagents and water is Class III water specified under GB / T6682.

10.1 Reagents

10.1.1 Tartaric acid ($C_4H_4O_6H_2$).

10.1.2 Ascorbic acid ($C_6H_8O_6$)

10.1.3 Phenolphthalein ($C_{20}H_{14}O_4$).

10.1.4 Ammonia (NH_4OH).

10.1.5 Sulfuric acid (H_2SO_4).

10.1.6 Ethanol (C_2H_5OH).

10.1.7 Methanol (CH_3OH).

10.1.8 Phenylfluorone ($C_{19}H_{12}O_5$).

10.1.9 Animal glue (Gelatin).

10.2 Reagent compounding

10.2.1 Tartaric acid solution (100 g/l): weigh and take 100 g of tartaric acid and dissolve it into 1l of water.

10.2.2 Ascorbic acid solution (10.0 g/l): weigh and take 10.0 g of ascorbic acid and dissolve it into 1l of water for purpose of extemporaneous preparation.

10.2.3 Animal glue solution (5.0 g/l): weigh and take 5.0 g of animal glue and dissolve it into 1l of water for purpose of extemporaneous preparation.

10.2.4 Ammonia solution (1+1): weigh and take 100 ml of ammonia water and pour it into 100 ml of water, and mix them well.

10.2.5 Sulfuric acid solution (1+9): weigh and take 10 ml of sulfuric acid, mix and pour it slowly into 90 ml of water and shake them up.

10.2.6 Phenylfluorone solution (0.1 g/l): weigh and take 0.01g (accurate to 0.001 g) of phenylfluorone, add an iota of methanol and a few drops of sulfuric acid to dissolve it, with the methanol being diluted to 100 ml.

10.2.7 Phenolphthalein indicator solution (10.0 g/l): weigh and take 1.0 g of phenolphthalein and dissolve it by ethanol to 100 ml.

10.3 Standard products

Reference material of Tin metal (Sn), reference material with a purity of 99.99% or reference material certified and granted by the State with reference material certificate.

10.4 Formulation of standard solution

10.4.1 Tin standard solution (1.0 mg/ml): weigh and take 0.1 g (accurate to 0.0001 g) of Tin metal; transfer it into a small beaker; add 10 ml of sulfuric acid; cover it with a pan; heat it up till the tin is completely dissolved; remove the pan; continue to heat until thick white smoke occurs; cool it down slowly; add 50 ml of water and transfer it into 100 ml of volumetric flask; wash the beaker by sulfuric acid solution (1 + 9) repeatedly; pour the wash lotion into flask; dilute it to mark and mix it well.

10.4.2 Standard use solution of tin: imbibe and take 10.0 ml of tin standard solution; transfer it into a 100 ml volumetric flask; dilute it by sulfuric acid solution (1 + 9) to the mark and mix it well; dilute it again till each milliliter is equivalent to 10.0 µg tin.

11. Equipment and facilities

11.1 Spectrophotometer.

11.2 Electronic balance: a sensor volume of 0.1 mg and 1 mg.

12. Analytical procedures

12.1 Specimen preparation

12.1.1 Specimen is digested, the same as that described in 5.2.1.

12.1.2 Imbibe and take 1.00 ml ~ 5.00 ml specimen digestion solution and the same amount of reagent blank solution; transfer them into 25 ml colorimetric tube, respectively; add 0.5ml of tartaric acid solution (100 g/l) and 1 drop of phenolphthalein indicator solution (100 g/l) into specimen digestion solution and reagent blank solution and mix it well; add ammonia solution (1 + 1) to each of them till it becomes pink; add 3.0 ml of sulfuric acid solution (1 + 1), 1.0 ml of animal glue solution (5.0 g/l) and 2.5 ml of ascorbic acid solution (10.0 g/l); add water till the volume is 25 ml and mix it well; then add 2.0 ml of phenylfluoron solution (0.1 g/l) and mix it well; stand for 1h and measure.

12.2 Standard curve mapping

Imbibe and take 0.00 ml, 0.20 ml, 0.40 ml, 0.60 ml, 0.80 ml and 1.00 ml of tin standard solution (equivalent to 0.00 µg, 2.00 µg, 4.00 µg, 6.00 µg, 8.00 µg, and 10.00 µg of tin); transfer it into 25 ml colorimetric tube; add 0.5 ml of tartaric acid solution (100 g/l) and 1 drop of phenolphthalein indicator solution (10.0 g/l) into each and mix it well; add ammonia solution (1 + 1) into each till it becomes pink; add 3.0 ml of sulfuric acid solution (1 + 9), 1.0 ml of animal gelatin solution (5.0 g/l) and 2.5 ml of ascorbic acid solution (10.0 g/l); add water then till the volume reaches 25 ml and mix it well; then add 2.0 ml of phenylfluoron solution, mix it well and allow it to stand for 1h; and measure then.

Use 2 cm cuvette to measure absorbance at 490 nm of wavelength; after zero tube absorbance is subtracted from all standard points, take concentration of the standard series solution as the horizontal axis and absorbance as vertical axis to map the standard curve or calculate the linear regression equation.

12.3 Determination of specimen solution

Use 2 cm cuvette to adjust zero point per standard series solution zero tube; measure absorbance of reagent blank solution and specimen solution respectively at 490 nm of wavelength; compare the resulting absorbance to standard curve or substitute it into regression equation to calculate the content.

13. Statement of analysis result

Tin content in the specimen shall be calculated as per Formula (2):

$$X = \frac{(m_1 - m_2) \times V_1}{m_3 \times V_2} \dots\dots\dots (2)$$

In the formula:

X - tin content in the specimen, in milligram per kilogram or milligram per liter (mg/kg or mg/l);

m_1 - tin mass in specimen digestion solution for purpose of determination, in microgram (μg);

m_2 - tin mass in reagent blank solution, in microgram;

V_1 - constant volume of specimen digestion solution, in milliliter (ml);

m_3 - specimen mass, in gram (g);

V_2 - volume of specimen digestion solution for purpose of determination, in milliliter (ml).

Calculation result shall retain two-digit valid number.

14. Precision

Absolute difference between two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetic mean.

15. Others

When 1.0g of such specimen and 5.0 ml of digestion solution are taken for determination, the quantitative limit of this method shall be 20 mg/kg.

GB 5009.190-2014 Determination of Indicative PCB Content in Foods



National Standards of People's Republic of China

GB 5009.190-2014

National Food Safety Standard
Determination of Indicative PCB Content in Foods

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Issued by Ministry of Health of the People's Republic of China

Foreword

This standard has substituted for *GB/T 5009.190—2006 Determination of Indicative PCB Content in Foods* and *GB/T 22331—2008 Measurement of PCB Residue with Gas Chromatography in Aquatic Products*.

Compared with GB/T 5009.190—2006, this standard has the following main changes:

- Modified standard format

National Standard for Food Safety

Determination of Indicative PCB Content in Foods

1. Scope

Method I of this standard specifies the way to measure the content of PCBs (polychlorinated biphenyls) in food, including the indicative PCBs specified in the Global Environmental Monitoring System (GEMS)/food programme (PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and PCB180), as well as PCB18, PCB33, PCB44, PCB70, PCB105, PCB128, PCB170, PCB187, PCB194, PCB195 and PCB199. Method II specifies the way to measure the content of PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and PCB180.

This standard is applicable to the measurement of indicative PCBs in samples of animal-derived food and oil or fat including fish, shellfish, eggs, meat, dairy and their products.

Method I: Gas chromatography-mass spectrography to dilute stable isotopes

2. Principles

The stable isotope dilution technique is applied in the standard to add $^{13}\text{C}_{12}$ -marked PCBs into the samples as the quantitative criterion. Sample solutions going through Soxhlet extraction shall be added to the internal standard of recovery after the chromatographic purification of column chromatography, separation and concentration. Then an analysis shall be made with gas chromatography-low resolution mass spectrometer, quadrupole mass selective ion monitoring (SIM) or ion trap tandem mass spectrometry multiple-reaction monitoring (MRM) and determination with the internal standard method shall be carried out.

3. Reagents and materials

3.1 Reagents

3.1.1. C_6H_{14} : Pesticide Analysis Grade

3.1.2. CH_2Cl_2 : Pesticide Analysis Grade

3.1.3. $\text{C}_3\text{H}_6\text{O}$: Pesticide Analysis Grade

3.1.4. CH_3OH : Pesticide Analysis Grade

3.1.5. C_8H_{18} : Pesticide Analysis Grade

3.1.6. Anhydrous Na_2SO_4 : Top grade pure. We shall put the commercially available anhydrous Na_2SO_4 into the chromatographic column and rinse it twice with normal hexane and dichloromethane. The volume of solvent used every time shall be approximately twice that of anhydrous Na_2SO_4 . After the rinse, we shall transfer the anhydrous Na_2SO_4 into the flask where it dries at 50°C before baking it at 225°C for 8 to 12 hours and storing it in the dryer.

3.1.7. H_2SO_4 : 95%~98%, top grade pure.

3.1.8. NaOH: top grade pure.

3.1.9. AgNO₃: top grade pure.

3.1.10. Silica gel for chromatography (75µm~250µm): We shall put the commercially available anhydrous Na₂SO₄ into the chromatographic column and rinse it twice with normal hexane and dichloromethane. The volume of solvent used every time shall be approximately twice that of silica gel. After the rinse, we shall transfer the silica gel into the flask which will be capped and placed in the oven to dry up at 50 °C. Then we shall bake it at 180°C for 8 to 12 hours and place it in the reagent bottle with ground stopper after cooling, which will be stored in the dryer.

3.1.11. 44% acidated silica gel: Weigh 100 g of activated silica gel and drip 78.6 g of sulfuric acid in. Then shake it until lumps disappear before placing it into the reagent bottle with ground stopper, which will be stored in the dryer.

3.1.12. 33% alkaline silica gel: Weigh 100 g of activated silica gel and drip 49.2 g of 1 mol/L sodium hydroxide solution in. Then shake it until lumps disappear before placing it into the reagent bottle with ground stopper, which will be stored in the dryer.

3.1.13. 10% silver nitrate impregnated silica gel: Dissolve 5.6 g of silver nitrate in 21.5 mL of deionized water and drip it into 50 g of activated silica gel. Then shake it until lumps disappear before placing it into the reagent bottle with ground stopper, which will be stored in the dryer.

3.1.14. Alkaline aluminium oxide for chromatographic analysis: to bake it at 660 °C for 6 hours and place it into the reagent bottle with ground stopper, which will be stored in the dryer.

3.2 Standard solutions

3.2.1. Standard solutions for time window determination: comprising homologues when different PCBs appear at the first and last peaks on the DB-5ms chromatographic column. Please see A.1 at Appendix A.

3.2.2. Internal standard solutions for quantification: Please see A.2 at Appendix A.

3.2.3. Internal standard solutions for recovery: Please see A.3 at Appendix A.

3.2.4. Standard solutions for correction: Please see A.4 at Appendix A.

3.2.5. Standard solutions for accuracy and preciseness: Please see A.5 at Appendix A.

4. Equipment and facilities

4.1 Gas chromatography-quadrupole mass spectrometer (GC-MS) or gas chromatography-ion trap tandem mass spectrometer (GC-MS/MS).

4.2 Chromatographic column: DB-5ms column, 30 m×0.25 mm×0.25 µm, or equivalent chromatographic column

4.3 Tissue homogenizer

4.4 Meat grinder

4.5 Rotary evaporators

4.6 Nitrogen concentrator

4.7 Supersonic cleaner

4.8 Oscillator

4.9 Analytical balance whose sensitivity is 0.1 g

4.10 Preparation for glassware instruments: all re-usable glassware shall be thoroughly rinsed after use in the following procedure.

- a) To rinse the instrument with the last solvent;
- b) To rinse it with normal hexane and acetone;
- c) To rinse it with warm water that contains alkaline detergents;
- d) To rinse it with hot water and deionized water
- e) To rinse it with acetone, normal hexane and dichloromethane.

The combination of supersonic cleaners and warm water that contains alkaline detergents produces very good cleaning effects. Be careful not to damage the internal surface of the glassware instrument when cleansing it with a brush.

5. Analytical procedures

5.1 Specimen preparation

5.1.1. Pre-treatment

5.1.1.1 Samples collected on site shall be packaged with light-proof materials such as aluminum foil and brown glass bottles before being delivered to the lab in a small-sized refrigerator, where they are kept at -10°C or lower.

5.1.1.2 Solid samples such as fish and meat can be freeze-dried or dried with anhydrous Na₂SO₄ and evenly mixed. Oil or fat can directly dissolve in the normal hexane for purification.

5.1.2. Extraction

5.1.2.1 Before extraction, we shall put an empty cellulose or fiberglass socket into the Soxhlet extractor and pre-extract it for 8 hours with normal hexane + dichloromethane (50+50) as the extraction solvent before drying.

5.1.2.2 We shall put 5.0 to 10.0 g of pre-treated samples into the above-mentioned extraction socket in 5.1.2.1 and add ¹³C₁₂-marked quantitative interior standard (3.2.2). Then we shall cover the samples with glass wool which will be put into the Soxhlet extractor after a 30-minute balance. We shall extract it for 18 to 24 hours with normal hexane + dichloromethane (50+50) as the extraction solvent while keeping the back-flow velocity at 3 to 4 times per hour

5.1.2.3 After the extraction, we shall transfer the extracting solution into the eggplant-shaped bottle while evaporating it in a rotatory way until it is almost dry. Where the analysis result must be calculated in terms of fat, fat content is needed to test the samples.

5.1.2.4 Determination of fat content: We shall determine the exact weight of the eggplant-shaped bottle before concentration and dry the solvent before weighing the bottle. The difference value of the two results shall be the fat content of samples. After the determination, we shall add a small amount of normal hexane to dissolve the residue in the bottle.

5.1.3. Purification

5.1.3.1 Purification with acid silica gel column

Decontaminating column filling: We shall fill in 4 g of activated silica gel, 10 g of acid silica gel, 2 g of activated silica gel and 4 g of anhydrous Na_2SO_4 in sequence from bottom to top after blocking the glass column end with glass wool (see the Fig D.1 in Appendix D). Then we shall pre-wash it with 100 ml of normal hexane.

Purification: We shall transfer all the condensed extracting solution to the column and rinse the eggplant-shaped bottle with about 5 mL of normal hexane for 3 or 4 times before transferring the washer liquid to the column.

When the liquid level lowers to the anhydrous Na_2SO_4 layer, we shall add 180 mL of normal hexane for elution and condense the eluent to about 1 mL. When the acidated silica gel layers are all discolored, it means that the fat content in the samples has gone beyond the column load limit. After the condensation of eluent, we shall produce a new acid silica gel purification column and repeat the above procedures until the sulfuric acid silica gel doesn't get discolored.

5.1.3.2 Purification with composite silica gel column

Decontaminating column filling: We shall fill in 1.5 g of silver nitrate impregnated silica gel, 1 g of activated silica gel, 2 g of alkaline silica gel, 1 g of activated silica gel, 4 g of acidated silica gel, 2 g of activated silica gel and 2 g of anhydrous Na_2SO_4 in sequence from bottom to top after blocking the glass column end with glass wool (see the Fig D.1 in Appendix D). Then we shall pre-wash it with 30 mL of normal hexane and dichloromethane (97+3).

Purification: We shall transfer all condensed eluent that is purified in 5.1.3.1 to the column and rinse the eggplant-shaped bottle with about 5 mL of normal hexane for 3 or 4 times before transferring the washer liquid to the column. When the liquid level lowers to the anhydrous Na_2SO_4 layer, we shall add 50 ml of normal hexane and dichloromethane (97+3) for elution and condense the eluent to about 1 mL.

5.1.3.3 Purification with alkaline aluminum oxide column

Decontaminating column filling: We shall fill in 2.5 g of baked alkaline aluminum oxide and 2 g of anhydrous Na_2SO_4 in sequence from bottom to top after blocking the glass column end with glass wool (see the Fig D.1 in Appendix D). Then we shall pre-wash it with 15 mL of normal hexane.

Purification: We shall transfer all condensed eluent that is purified in 5.1.3.2 to the column and rinse the eggplant-shaped bottle with about 5 mL of normal hexane for 3 or 4 times before transferring the washer liquid to the column. When the liquid level lowers to the anhydrous Na_2SO_4 layer, we shall add 30 mL of normal hexane (2×15 mL) for elution. Then when the liquid level lowers to the anhydrous Na_2SO_4 layer, we shall add 25 mL of dichloromethane and normal hexane (5+95), and condense the eluent until it's nearly dry.

5.1.4. Treatment before analysis on the equipment

We shall transfer the purified sample solution into the sample introduction pipe and condense the nitrogen

when it flows down. Then we shall rinse the eggplant-shaped bottle with a small amount of normal hexane for three to four times and transfer the washing liquid into the sample introduction insert-pipe before condensing the nitrogen to about 50 μ l. Then we shall add an appropriate amount of internal standard of recovery (3.2.3) and cap it before analysis on the equipment.

5.2 Reference conditions for equipment

5.2.1. Chromatographic condition

5.2.1.1 Chromatographic column: To carry out chromatographic separation with the 30-m DB-5ms (or other type which is equal to DB-5ms) quartz capillary column. The film thickness shall be 0.25 μ m and the inner diameter 0.25 mm.

5.2.1.2 The temperature at the injection port shall be 300 °C when using splitless injection.

5.2.1.3 The temperature of the chromatographic column rises in the following way: the initial temperature stays at 100 °C for 2 min; then to 180 °C at 15 °C/min; then to 240 °C at 3 °C/min. Finally to 285 °C at 10 °C/min and stay for 10 minutes.

5.2.1.4 High-purity helium (purity > 99.999%) shall be used as the carrier gas.

5.2.2. Mass spectrometric parameters

5.2.2.1 Quadrupole mass spectrometer

Ionization mode: Electron Impact Ion Source (EI); 70 eV. Ion detection mode: selected ion monitoring (SIM); The characteristic ion that is selected when detecting the PCBs is a molecular ion. See Table B.1 at Appendix B.

Ion source temperature shall be 250 °C Transmission line temperature shall be 280 °C Solvent delay will be 10 minutes.

5.2.2.2 Ion trap mass spectrometer

Ionization mode: Electron Impact Ion Source; 70 eV. Ion detection mode: multiple- reaction monitoring (MRM); The parent ion that is selected when detecting the PCBs is a molecular ion (M+2 or M+4) and the daughter ion is a fragment ion (M-2Cl) that is formed when a molecular ion loses two chlorine atoms. See the Table B.2 at Appendix B. The ion trap temperature shall be 220 °C the transmission line temperature shall be 280 °C the manifold temperature shall be 40 °C

5.3 Sensitivity examination

To inject 1 μ L (20 pg) of CS1 solution and detect the GC-MS sensitivity. Detection ion SNR (Signal to Noise Ratio) of all 3-to-7-cl-substitution compounds shall be up to above 3, or the equipment shall be tuned again until it meets the requirement.

5.4 Qualification quantification of PCBs

5.4.1 Confirmation requirement for PCBs' chromatographic peak: The SNR of detected chromatographic peak shall be above 3 (See Fig C.1 or C.3 at Appendix C)

5.4.2 The abundance ratio of the two detected characteristic ion shall be within the theoretical range. Please see Table B.1 and B.2 at Appendix B.

5.4.3 To detect mass spectrum corresponding with the chromatographic peak (See Fig C.2 or C.4 at Appendix C). When the concentration is high enough, there shall be a fragment ion which loses two helium atoms (M-70). See Fig B.1 at Appendix B.

5.4.4 To detect the mass spectrum this corresponds with the chromatographic peak (See Fig C.2 or C.4 at Appendix C). There shall not be any fragment ion with molecular ions and two helium atoms (M+70) See Fig B.1 at Appendix B.

5.4.5 The retention time of determined PCBs shall be located at the time window which determines the standard solution via the analysis window. Time window determination standard solution comprises homologues when different PCBs appear at the first and last peaks on the DB-5ms chromatographic column. The time window shall be determined according to the retention period where different PCBs are by making analysis on window determination solution (1 µL) with the determined chromatographic condition and full-scan mass spectrum acquisition mode. Due to the overlap of retention periods of PCBs from three families in the DB-5ms chromatographic column, characteristic ions of these PCBs shall be detected in a single time window. To ensure the selectivity and sensitivity of the analysis, the detected characteristic ions at one window when determining the time window shall be as little as possible.

5.5 Statement of analysis result

5.5.1 During the standard, PCB28, PCB52, PCB118, PCB153, PCB180, PCB206 and PCB209 are quantified with the isotope dilution technique. Other target compounds shall be quantified with the internal standard method and the recovery calculation of internal standard for quantification shall be done with the internal standard method. The 20 target compounds determined in the standard include most PCBs' industrial products. There are 3 compounds in every family from PCB3 to PCB8. There is one in PCB9 or PCB10. See the Table A.4 in Appendix A. One ¹³C₁₂-marked compound is used as internal standard for quantification in every family. See the Table A.2 in Appendix A. There are two internal standards of recovery to calculate the quantified internal standard recovery. See the Table A.3 in Appendix A. When calculating the quantified internal standard recovery, ¹³C₁₂-PCB101 shall be used as the internal standards of recovery for ¹³C₁₂-PCB28, ¹³C₁₂-PCB52, ¹³C₁₂-1PCB18 and ¹³C₁₂-PCB153 and ¹³C₁₂-PCB194 shall be used as the internal standards of recovery for ¹³C₁₂-PCB180, ¹³C₁₂-PCB202, ¹³C₁₂-PCB206 and ¹³C₁₂-PCB209.

5.5.2 Relative Response Factor (RRF): In this standard, RRF is used for quantitative calculation. The RRF value is calculated with calibration standard solution. See the computational formulas at (1) and (2).

$$RRF_n = \frac{A_n \times c_s}{A_s \times c_n} \dots\dots\dots (1)$$

$$RRF_r = \frac{A_r \times c_s}{A_s \times c_r} \dots\dots\dots (2)$$

In the formula,

RRF_n — the relative response factor of target compounds to internal standard for quantification;

A_n —peak area of the target compounds;

C_s —concentration of internal standard for quantification ($\mu\text{g/L}$);

A_s —peak area of internal standard for quantification;

C_n —concentration of target compounds ($\mu\text{g/L}$);

RRF_r —the relative response factor of internal standard for quantification to internal standard for recovery;

A_r —peak area of the internal standard for recovery;

C_r —solutions of internal standard for recovery ($\mu\text{g/L}$);

The RRF values of the five thickness levels for different compounds shall have the relative standard deviation (RSD) of less than 20%. When the standard is met, we shall use average RRF_n and average RRF_r for quantitative calculation.

5.5.3 Content calculation: see (3) for calculation formula of PCBs content in samples

$$c_n = \frac{A_n \times m_s}{A_s \times RRF_n \times m} \dots\dots\dots (3)$$

In the formula,

C_n —PCBs content in the samples ($\mu\text{g/kg}$)

A_n —peak area of target compounds;

m_s —amount of internal standard for quantification into the samples(ng);

A_s —peak area of internal standard for quantification;

RRF_n —the relative response factor of target compounds to internal standard for quantification;

m —sampling weight(g)

5.5.4 Calculation of quantified internal standard of recovery: quantified internal standard of recovery(R) is calculated according to (4) and the quantitative value is symbolized with %.

$$R = \frac{A_s \times m_r}{A_r \times RRF_r \times m_s} \times 100\% \dots\dots\dots (4)$$

In the formula,

R —quantified internal standard for recovery, %;

A_s —peak area of quantified internal standard;

m_r —quantity of internal standard for recovery in the samples (ng);

A_r —peak area of internal standard for recovery;

RRF_r —the relative response factor of quantified internal standard to internal standard for recovery;

m_s —quantity of internal standard for quantification in the samples,(ng).

Quantitative Results can have two digits after the decimal point.

5.5.5 Limit of detection: it is specified by this standard that the sample detection limit constitutes the sample solution concentration which corresponds with the response that meets the requirement of isotope abundance ratio when the SNR (Signal to Noise Ratio) is 3. See (5) for the computational formula of detection limit.

$$DL = \frac{3 \times N \times m_s}{H \times RRF_n \times m} \dots\dots\dots (5)$$

In the formula:

DL —limit of detection (μg/kg)

N — peak height of the noise

m_s —amount when quantified internal standard is added (ng)

H —peak height of quantified internal standard

RRF_n —the relative response factor of target compounds to internal standard for quantification;

m —sample volume,(g).

The sample base material, sampling volume, injected sample size, quantified internal standard of recovery, chromatographic separation, electrical noises and equipment sensitivity all may affect the sample detection limit, so the noise shall be acquired from the real sample spectrogram. The sample detection limit shall be reported when the outcome of target compounds hasn't come out yet.

6. Quality control and quality guarantee

6.1 Initial precision and accuracy test

The lab shall have the acceptable accuracy and preciseness before analyzing the real samples. Reliability of the analytical methods shall be verified by analyzing marked samples.

We shall use 3 or more blank samples whose base materials are similar to the real samples, in which we respectively add normal experiment solution with the right accuracy and preciseness. See Table A.5 for Appendix A. Then we shall respectively add standard solution of internal standard for quantification before analyzing the formulated marked samples in the same way as the real samples, and calculate the recovery of target compounds and the quantified internal standard of recovery. The estimated value of target PCBs for every sample shall range from 75% to 120% of the addition quantity. $RSD < 30\%$. The average of recovery for quantified internal standard shall range from 50% to 120%. And the quantified internal standard of recovery for single samples shall range from 30% to 130%.

Before being analyzed, real samples shall meet the above requirements. When the way to extract and purify the samples has been modified or the analyzer has been replaced, such experiments shall be repeated until the above requirements are met. The above experiments shall be done in the lab every 6 months to ensure that the above requirements are met.

If we can have standard reference materials which have base materials similar to samples, then we can apply normal reference materials to the accuracy and preciseness experiment instead of marked samples.

6.2 Quantified internal standard of recovery

Quantified internal standard shall be added to rectify the loss of target compounds during the extraction and purification of the samples. Quantified internal standard of recovery shall range from 30% to 130%. If the sample analysis outcome for quantified internal standard of recovery hasn't met the above requirement, samples shall be extracted, purified and analyzed on the equipment again.

6.3 Method blank

A method blank experiment shall be done on every group which has at most 15 samples.

6.4 Quality control sample

There are at most 15 samples for every group with a quality control sample. Quality control samples shall be standard reference material or marked sample with known concentration. The estimated value of target compounds shall range from 75% to 125% of the standard values.

6.5 Retention time window

Time window shall be carried out every week to determine the analysis of standard solution and the correctness of retention time window. When the chromatographic column has been replaced or cut, or the chromatographic parameter has been changed, time window shall be used to determine the standard solution and calibrate the retention time window.

6.6 Calibration of standard solution

Calibration standard solutions with 5 thickness levels shall be used for initial calibration. Calibration is regarded to be successful when the RSD of RRF is less than 20%. During the analysis, confirmatory tests

shall be done every 12 hours. CS3 in the calibration standard solutions shall be used for equipment analysis. The analysis outcome shall be within the scope of 20% of its constant value. Quantified internal standard of recovery shall be within

75% to 125%.

7. Others

Quantification limit of every target compounds shall be 0.5 µg/kg.

Method II Gas chromatographic method

8. Principle

With this method, we add PCB198, the quantified internal standard, into the samples and heat it in the water bath before vibrating extraction. After the sulfuric acid treatment and purification of chromatographic column, we shall make the measurement with the gas chromatographic- electron capture detector method so as to ensure the time qualification and internal standard quantification.

9. Reagent and material

9.1 Reagent

9.1.1 C₆H₁₄:Pesticide Analysis Grade

9.1.2 CH₂Cl₂:Pesticide Analysis Grade

9.1.3 C₃H₆O:Pesticide Analysis Grade

9.1.4 Anhydrous Na₂SO₄: Top grade pure. We shall put the commercially available anhydrous Na₂SO₄ into the glass chromatographic column and rinse it twice with normal hexane and dichloromethane. The volume of solvent used every time shall be approximately twice that of anhydrous Na₂SO₄. After the rinse, we shall transfer the anhydrous Na₂SO₄ into the flask where it dries at 50 °C before baking it at 225°C overnight and storing it in the dryer when it cools down.

9.1.5 H₂SO₄:top grade pure.

9.1.6 Alkaline aluminium oxide for chromatographic analysis: to bake the commercially available chromatographic packing materials at 660 °C for 6 hours and place it into the dryer for storage.

9.2 Standard solution

Indicative PCB standard solutions. See Table A.6 in Appendix A

10. Equipment and facilities

10.1.1 Gas chromatograph: with the electron capture detector(ECD)

10.1.2 Chromatographic column: DB-5ms column,30 m×0.25 mm×0.25 µm,or equivalent chromatographic column

- 10.1.3 Tissue homogenizer
- 10.1.4 Meat grinder
- 10.1.5 Rotary evaporator
- 10.1.6 Nitrogen concentrator
- 10.1.7 Supersonic cleaner
- 10.1.8 Vortex oscillator
- 10.1.9 Analytical balance
- 10.1.10 water bath oscillator
- 10.1.11 Centrifuge
- 10.1.12 Chromatographic column

11. Analytical procedure

11.1 Sample extraction

11.1.1 Solid sample: Weigh 5 g to 10 g of samples (accurate to 0.1 g) and place it into the conical flask with cover. Then add some quantified PCB198 and place it on the water bath oscillator for 2 hours with an appropriate amount of normal hexane + dichloromethane (50+50) as the extract solution. The water bath temperature shall be at 40 °C and the vibrating rate shall be at 200 r/min.

11.1.2 Liquid sample (exclusive of oil and fat sample): Weigh 10 of samples (accurate to 0.1 g) and place it into the conical flask with cover. Then add quantified internal standard PCB198 and 0.5 g of sodium oxalate to be evenly mixed with 10 mL of methyl alcohol. Next, add 20 mL of aether and n-hexane (25+75) to be oscillated for 20 minutes and centrifuged at 3,000 r/min for 5 minutes. Then transfer the supernatant liquor to the glass column which has 5 g of anhydrous Na₂SO₄. Add 20 mL of aether and n-hexane (25+75) in the residue and repeat the above procedure. Finally combine the extracting solution.

11.1.3 Transfer the extracting solution to the eggplant-shaped bottle and rotate to evaporate it until it is nearly dry. Fat content of the sample shall be determined if the analysis result is calculated by fat.

11.1.4 Determination of the sample fat: Get the exact weight of the empty eggplant-shaped bottle before concentration. Then dry the solvent and weigh the bottle and the residue again. The difference value of the two results shall be the fat content of samples.

11.2 Purification

11.2.1 Sulfuric acid purification

Transfer the condensed extracting solution into the 10-mL test tube and rinse the eggplant-shaped bottle for 3 to 4 times with approximately 5 mL of normal hexane before combining it with the concentrated solution. Meter the volume to the scale with normal hexane and add 0.5 mL of concentrated sulfuric acid. Then shake it for 1 minute and centrifuge it at 3,000 r/min, thus separating the sulfuric acid layer from the organic layer. If the upper solution is still colored, that means the fat still remains. So add an appropriate amount of

concentrated sulfuric acid and repeat the operation until the upper solution becomes colorless.

11.2.2 Purification with alkaline aluminum oxide column

Decontaminating column filling: We shall add a small amount of glass wool at the glass column end and fill in 2.5 g of baked alkaline aluminium oxide and 2 g of anhydrous Na₂SO₄ in sequence before rinsing it with 15 mL of normal hexane.

Purification: We shall transfer concentrated solution at 11.2.1 and rinse the eggplant-shaped bottle for 3 to 4 times with 5 mL of normal hexane before transferring it to the chromatographic column. When the liquid level lowers to the anhydrous Na₂SO₄ layer, we shall add 30 mL of normal hexane (2×15 mL) for elution. Then when the liquid level lowers to the anhydrous Na₂SO₄ layer, we shall add 25 mL of dichloromethane and normal hexane(5+95) for elution, and condense the eluent until it's nearly dry.

11.3 Sample solution concentration

Transfer the sample solution in 11.2.2 to the sample introduction bottle and rinse the eggplant-shaped bottle with a small amount of normal hexane for 3 to 4 times. Then add the cleansing solution into the sample introduction bottle. Finally condense it to 1 mL under the nitrogen flow for GC analysis.

12. Determination

12.1 Chromatographic condition

12.1.1 Chromatographic column: DB-5ms column, 30 m×0.25 mm×0.25 μm, or equivalent chromatographic column

12.1.2 Temperature at the injection port: 290 °C

12.1.3 Temperature raising procedure: starting temperature at 90 °C remaining for 0.5 minutes; to 200 °C at 15 °C/min for 5 minutes; to 250 °C at 2.5 °C/min for 2 minutes; then to 265 °C at 20 °C/min for 5 minutes

12.1.4 Carrier gas: high-purity nitrogen (nitrogen>99.999%); column pressure is 67 kPa(10 psi)

12.1.5 Injection Volume: 1 μL of splitless sample introduction

12.1.6 Chromatographic analysis: to keep the qualitative time and compare the quantity with the peak height or peak area of samples and standard.

12.2 Qualitative analysis of PCBs

To make qualitative analysis with retention time or relative retention time. SNR(Signal to Noise Ratio) for the chromatographic peak of the detected PCBs shall be more than 3.

12.3 Quantitative determination of PCBs

12.3.1 Relative Response Factor(RRF)

To make quantitative calculation with RRF by using the internal standard method. To calculate RRF value according to (6) by using the calibrating standard solution sample introduction:

$$RRF = \frac{A_s \times c_s}{A_r \times c_r} \dots\dots\dots (6)$$

In the formula,

RRF—Relative response Factor of target compounds to internal standard of quantification;

A_r—Peak area of target compounds;

C_s—Concentration of internal standard of quantification(μg/L);

A_s—Peak area of internal standard of quantification;

C_r—Concentration of target compounds (μg/L).

In the standard solutions, RSD (relative standard deviation) of the RRF value of all target compounds shall be less than 20%.

12.3.2 Content calculation

To calculate the content of PCBs in samples according to (7):

$$X_r = \frac{A_s \times m_s}{A_r \times RRF \times m} \dots\dots\dots (7)$$

In the formula,

X_r—content of target compounds(μg/kg);

A_r— peak area of target compounds;

m_s—amount of quantified internal standard added to the sample(ng);

A_s—peak area of quantified internal standard;

RRF—relative response Factor of target compounds to quantified internal standard;

m—sampling weight(g).

12.3.3 Limit of detection

It is specified by this standard that the sample detection limit constitutes the sample solution concentration which corresponds with the response that meets the requirement of relative retention time with triple SNR. See the computational formula at (8):

$$DL = \frac{3 \times N \times m_s}{H \times RRF \times m} \dots\dots\dots (8)$$

In the formula:

DL —limit of detection ($\mu\text{g/kg}$)

N — peak height of the noise

m_s —amount when quantified internal standard is added (ng)

H —peak height of quantified internal standard

RRF —the relative response factor of target compounds to internal standard for quantification;

m —sample volume,(g).

The sample base material, sampling volume, injected sample size, chromatographic separation, electrical noises and equipment sensitivity all may affect the sample detection limit, so the noise shall be acquired from the real sample spectrogram. The sample detection limit shall be reported when the outcome of target compounds hasn't come out yet.

13. Accuracy

Absolute differences of the two individual analysis results acquired under repetitive conditions shall be 20% of the arithmetic mean value or less.

14. Others

Quantification limit of every target compounds shall be $0.5 \mu\text{g/kg}$.

Appendix A:

Standard solution for indicative PCB

Standard solutions used for time window determination, quantified internal standard, internal standard of recovery, standard series, accuracy and preciseness to determine the PCB content in food are specified from Table A.1 to A.6.

Table A.1 Standard solution to determine the time window of indicative PCBs tested with the GC-MS method

Compounds	Chlorine atom amount	Concentration mg/L
Biphenyl	0	2.5±0.25
PCB1	1	2.5±0.25
PCB3	1	2.5±0.25
PCB10	2	2.5±0.25
PCB15	2	2.5±0.25
PCB30	3	2.5±0.25
PCB37	3	2.5±0.25
PCB54	4	2.5±0.25
PCB77	4	2.5±0.25
PCB104	5	2.5±0.25
PCB126	5	2.5±0.25
PCB155	6	2.5±0.25
PCB169	6	2.5±0.25
PCB188	7	2.5±0.25
PCB189	7	2.5±0.25
PCB194	8	2.5±0.25
PCB202	8	2.5±0.25
PCB206	9	2.5±0.25
PCB208	9	2.5±0.25
PCB209	10	2.5±0.25

Table A.2 Standard solution for the quantified internal standard of indicative PCBs in the GC-MS method

Compounds	Chlorine atom amount	Concentration mg/L
$^{13}\text{C}_{12}$ -PCB28	3	2.0
$^{13}\text{C}_{12}$ -PCB52	4	2.0
$^{13}\text{C}_{12}$ -PCB118	5	2.0
$^{13}\text{C}_{12}$ -PCB153	6	2.0
$^{13}\text{C}_{12}$ -PCB180	7	2.0
$^{13}\text{C}_{12}$ -PCB202	8	2.0
$^{13}\text{C}_{12}$ -PCB206	9	2.0
$^{13}\text{C}_{12}$ -PCB209	10	2.0

Table A.3 Standard solution for the internal standard of recovery of indicative PCBs in the GC-MS method

Compounds	Chlorine atom amount	Concentration mg/L
$^{13}\text{C}_{12}$ -PCB101	5	2.0
$^{13}\text{C}_{12}$ -PCB194	8	2.0

Table A.4 Standard solution for indicative PCBs in the GC-MS method

Target compounds		Concentration µg/L				
		CS1	CS2	CS3	CS4	CS5
Natural compounds	PCB18	20	50	200	800	2000
	PCB28	20	50	200	800	2000
	PCB33	20	50	200	800	2000
	PCB52	20	50	200	800	2000
	PCB44	20	50	200	800	2000
	PCB70	20	50	200	800	2000
	PCB101	20	50	200	800	2000
	PCB118	20	50	200	800	2000
	PCB105	20	50	200	800	2000
	PCB153	20	50	200	800	2000
	PCB138	20	50	200	800	2000
	PCB128	20	50	200	800	2000
	PCB187	20	50	200	800	2000
	PCB180	20	50	200	800	2000
	PCB170	20	50	200	800	2000
	PCB199	20	50	200	800	2000
	PCB195	20	50	200	800	2000
	PCB194	20	50	200	800	2000
	PCB206	20	50	200	800	2000
	PCB209	20	50	200	800	2000
Quantified internal standard of isotope labeling	¹³ C ₁₂ -PCB180	400	400	400	400	400
	¹³ C ₁₂ -PCB202	400	400	400	400	400
	¹³ C ₁₂ -PCB206	400	400	400	400	400
	¹³ C ₁₂ -PCB209	400	400	400	400	400
	¹³ C ₁₂ -PCB28	400	400	400	400	400
	¹³ C ₁₂ -PCB52	400	400	400	400	400
	¹³ C ₁₂ -PCB118	400	400	400	400	400
	¹³ C ₁₂ -PCB153	400	400	400	400	400
Internal standard for recovery of isotope labeling	¹³ C ₁₂ -PCB101	400	400	400	400	400
	¹³ C ₁₂ -PCB194	400	400	400	400	400

Table A.5 Standard solution for indicative PCBs accuracy and preciseness experiment in the GC-MS method

Compounds	Concentration µg/L	Compounds	Concentration µg/L
PCB18	100	PCB138	100
PCB28	100	PCB128	100
PCB33	100	PCB187	100
PCB52	100	PCB180	100
PCB44	100	PCB170	100
PCB70	100	PCB199	100
PCB101	100	PCB195	100
PCB118	100	PCB194	100
PCB105	100	PCB206	100
PCB153	100	PCB209	100

Table A.6 Standard solution for indicative PCBs in the GC-ECD method

Compounds	Concentration µg/L				
	CS1	CS2	CS3	CS4	CS5
PCB28	5	20	50	200	800
PCB52	5	20	50	200	800
PCB101	5	20	50	200	800
PCB118	5	20	50	200	800
PCB138	5	20	50	200	800
PCB153	5	20	50	200	800
PCB180	5	20	50	200	800
PCB198(Quantified internal standard)	50	50	50	50	50

Appendix B

Characteristic ions and isotopic abundance ratio

Requirements to determine the characteristic ions and isotopic abundance ratio of indicative PCBs in food with the quadrupole mass spectrometer and ion trap mass spectrometer has been specified in Table B.1 and B.2.

Table B.1 Characteristic ions and isotopic abundance ratio of selected ion monitoring (SIM) of the quadrupole mass spectrometer

Homologues	Characteristic ions(m/z)	Ion type	Theoretical abundance	Determining ions
T3CB	256/258	M/M+2	1.03	
T4CB	290/292	M/M+2	0.78	
P5CB	324/326	M/M+2	0.62	
H6CB	358/360	M/M+2	0.52	
H7CB	394/396	M+2/M+4	1.04	
O8CB	428/430	M+2/M+4	0.89	
N9CB	462/464	M+2/M+4	0.78	
D10CB	498/500	M+4/M+6	1.17	
¹² C ₁₂	270	M+2	—	—
¹² C ₁₃	304	M+2	—	—
¹² C ₁₄	338	M+2	—	—
¹² C ₁₅	372	M+2	—	—
¹² C ₁₆	406	M+2	—	—
¹² C ₁₇	442	M+4	—	—
¹² C ₁₈	476	M+4	—	—
¹² C ₁₉	510	M+4	—	—
^a Fragment ions that exist ^b Fragment ions that can't exist ^c These ions are fragment ions(M+35)with one molecular ion and a chlorine. The existence of these ions shows that disturbance of PCBs from adjacent families may occur.				

Table B.2 Characteristic ions and isotopic abundance ratio of Multiple Reaction Monitoring (MRM) of the ion trap tandem mass spectrometer

Homologues	Parent ion (m/z)	Daughter ion	Theoretical abundance
T ₃ CB	258	186/188	2.00
T ₄ CB	292	220/222	1.00
P ₅ CB	326	254/256	0.67
H ₆ CB	360	288/290	0.50
H ₇ CB	396	324/326	1.00
O ₈ CB	430	358/360	0.80
N ₉ CB	464	392/394	0.67
D ₁₀ CB	498	426/428	0.55
¹³ C ₁₂ -T ₃ CB	270	198/200	2.00
¹³ C ₁₂ -T ₄ CB	304	232/234	1.00
¹³ C ₁₂ -P ₅ CB	338	266/268	0.67
¹³ C ₁₂ -H ₆ CB	372	300/302	0.50
¹³ C ₁₂ -H ₇ CB	408	336/338	1.00
¹³ C ₁₂ -O ₈ CB	442	370/372	0.80
¹³ C ₁₂ -N ₉ CB	476	404/406	0.67
¹³ C ₁₂ -D ₁₀ CB	510	438/440	0.55

Appendix C

Mass chromatogram and mass spectrum of PCBs determined with GC-MS

Fig C.1 and C.2 are the SIM chromatogram and mass spectrum to determine PCBs with the quadrupole mass spectrometer. Fig C.3 and C.4 are the MRM chromatogram and mass spectrum to determine the PCBs with the ion trap mass spectrometer.

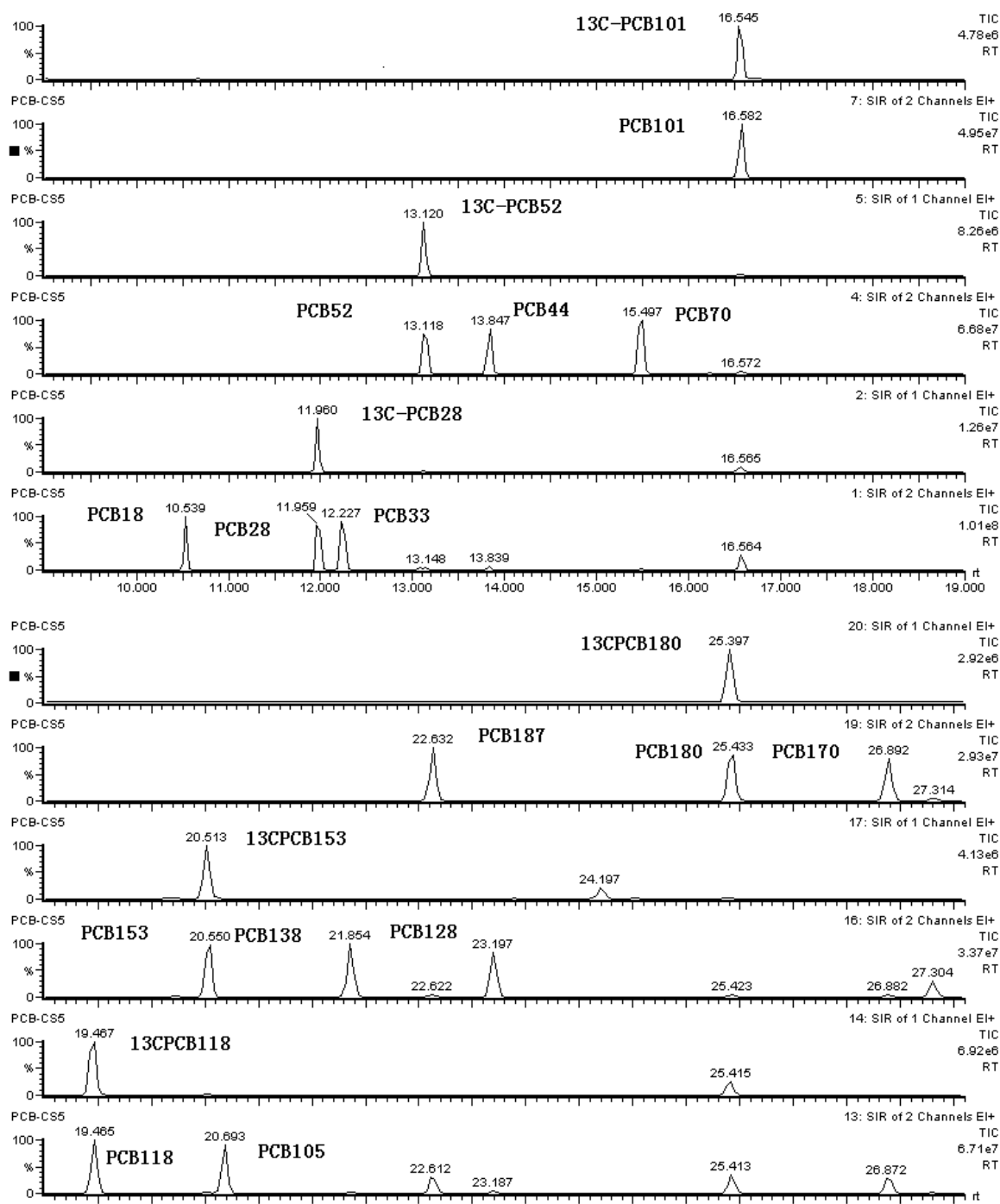
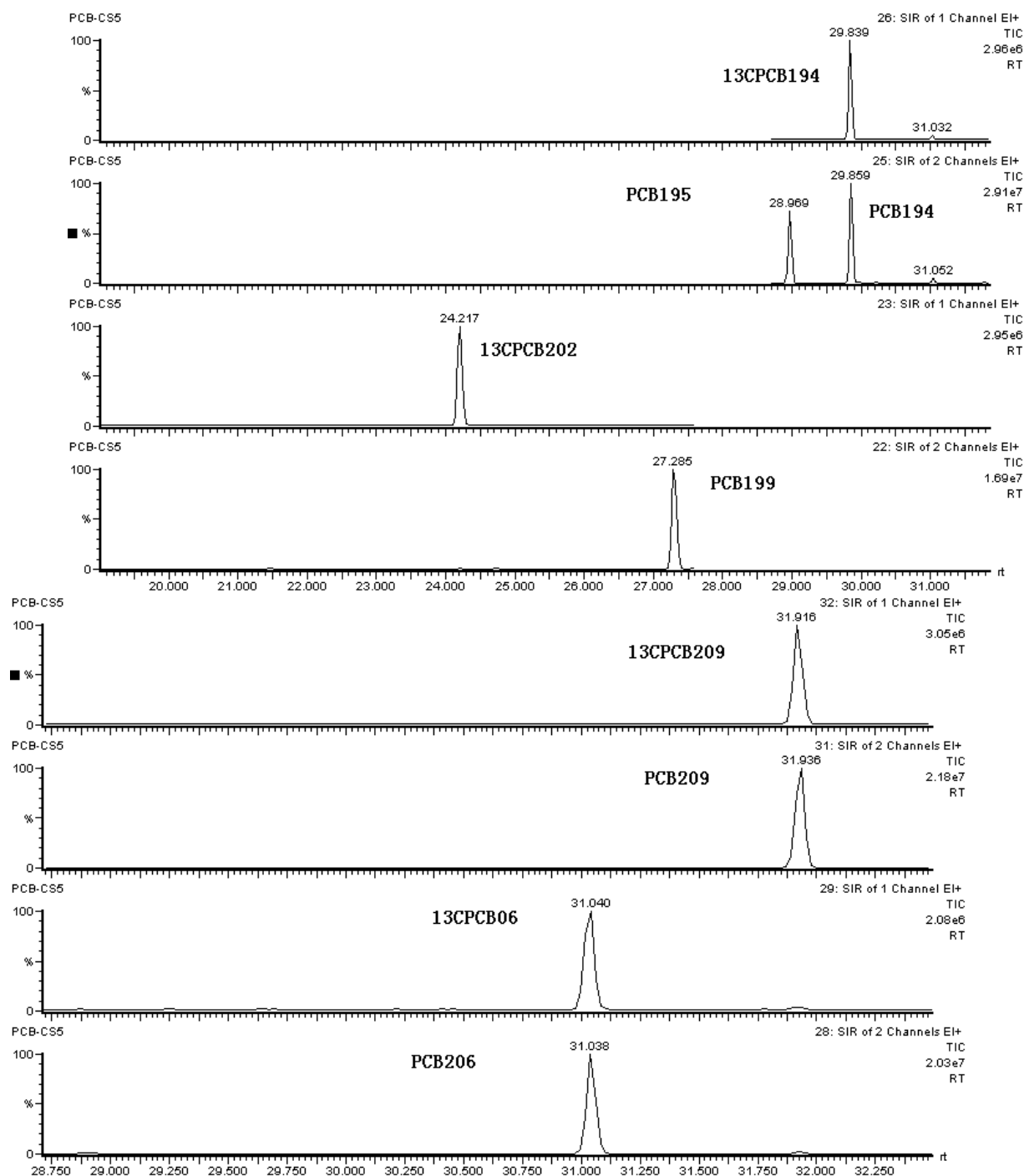


Fig C.1 Mass chromatogram of characteristic ions of target PCBs with SIM



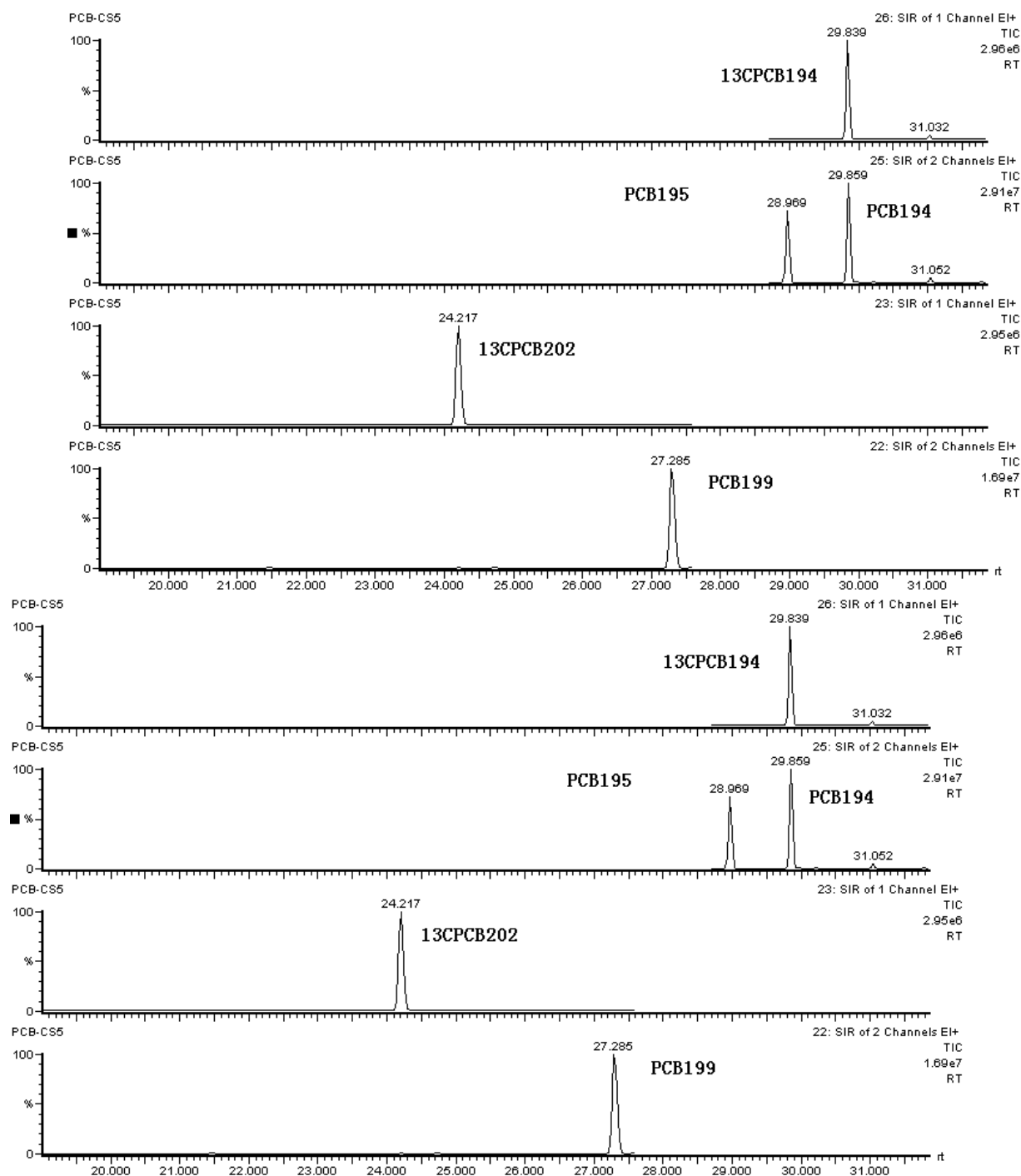


Fig C.1 (to be continued)

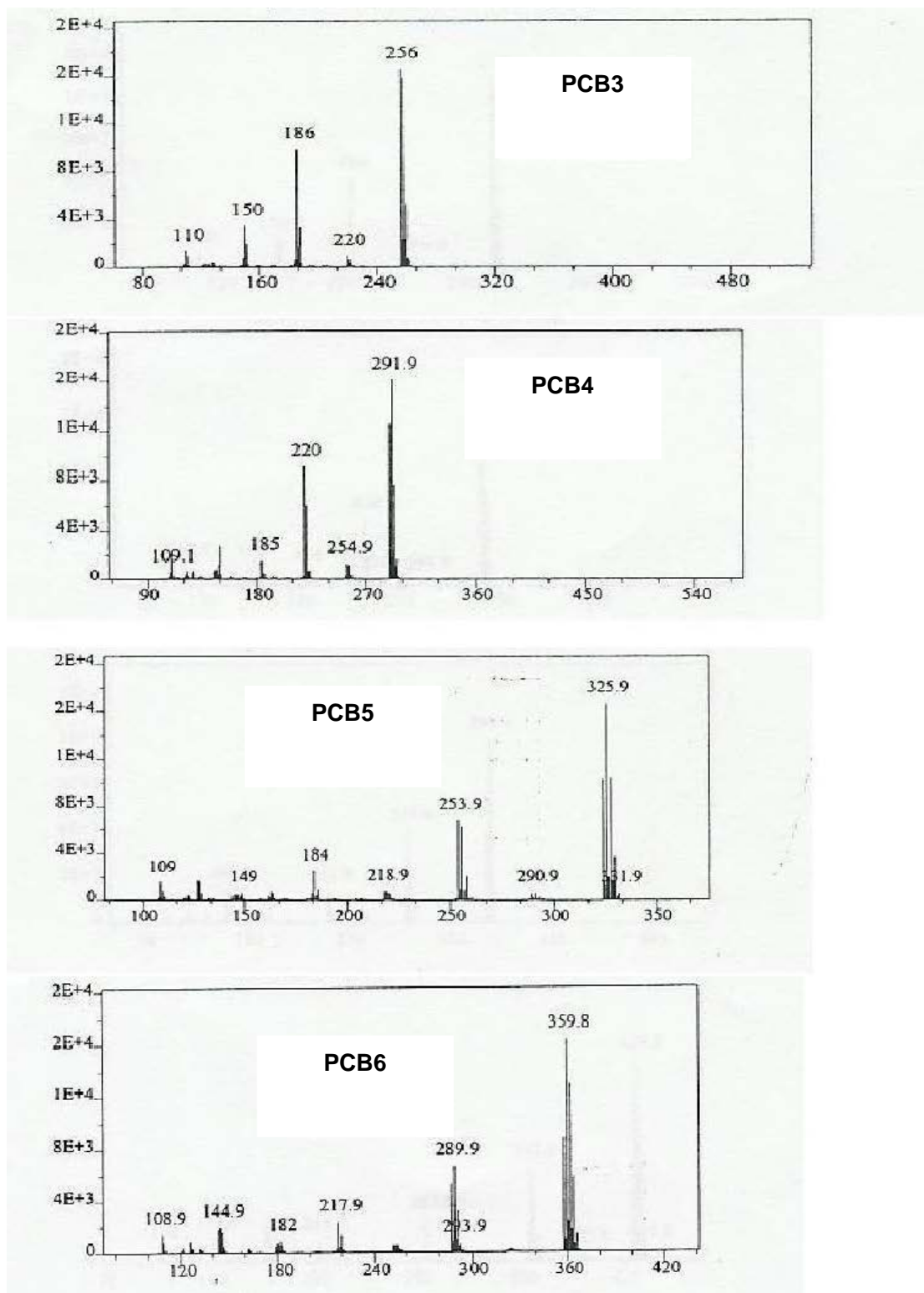


Fig C.2 Mass spectrum of characteristic ions of different PCBs with SIM

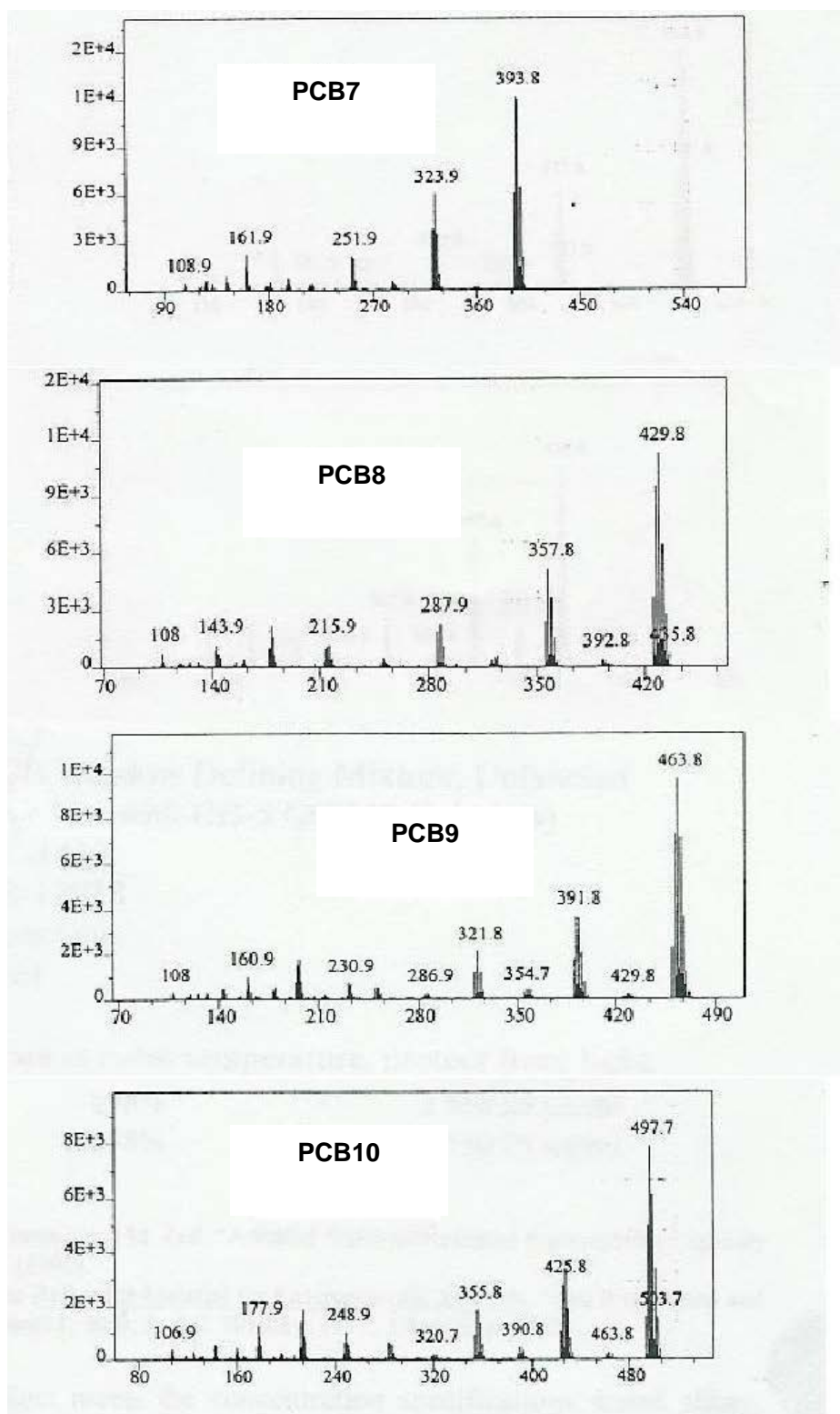


Fig C.2 (to be continued)

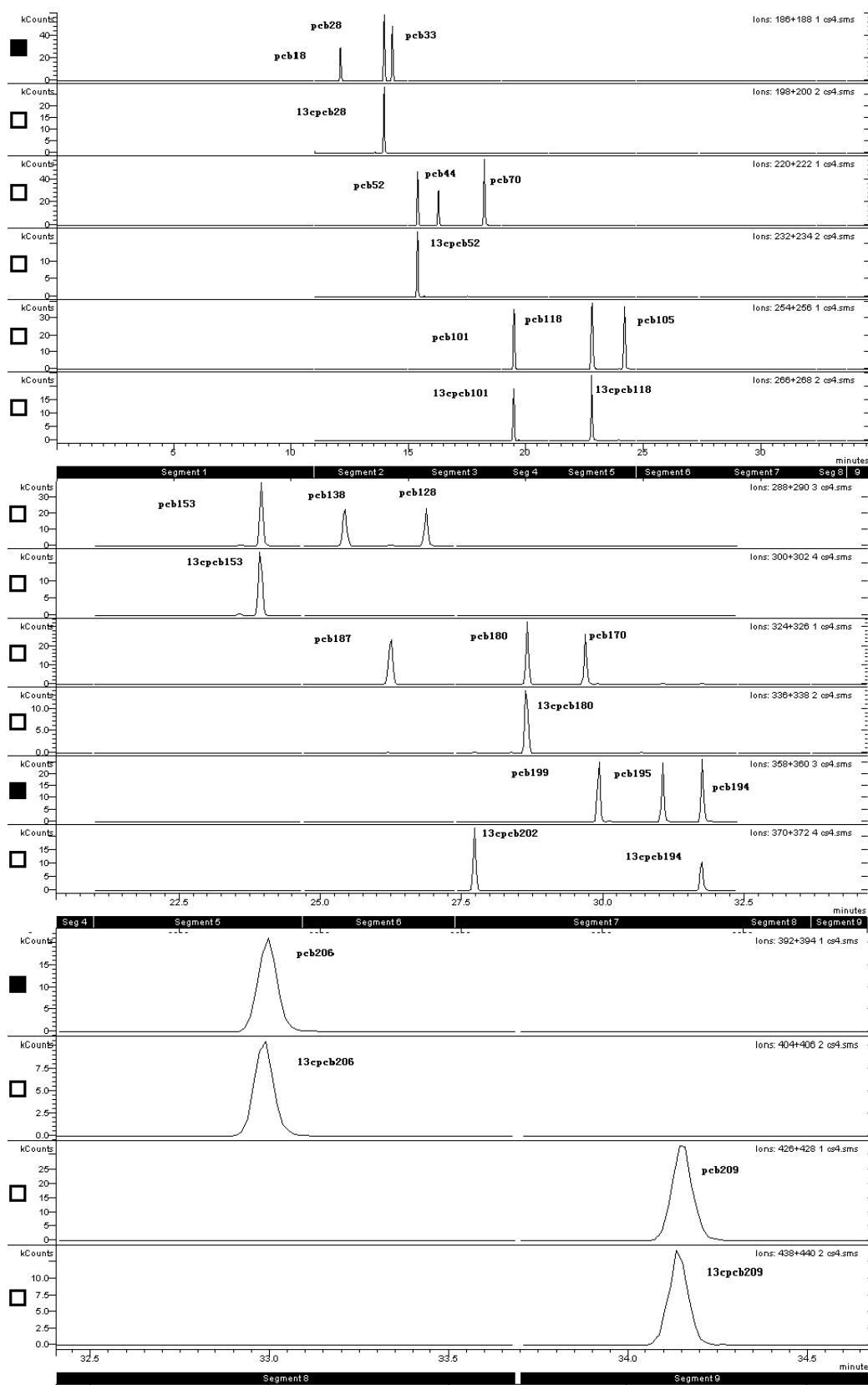


Fig C.3 Recombination ion chromatogram of different target PCBs daughter ions with MRM

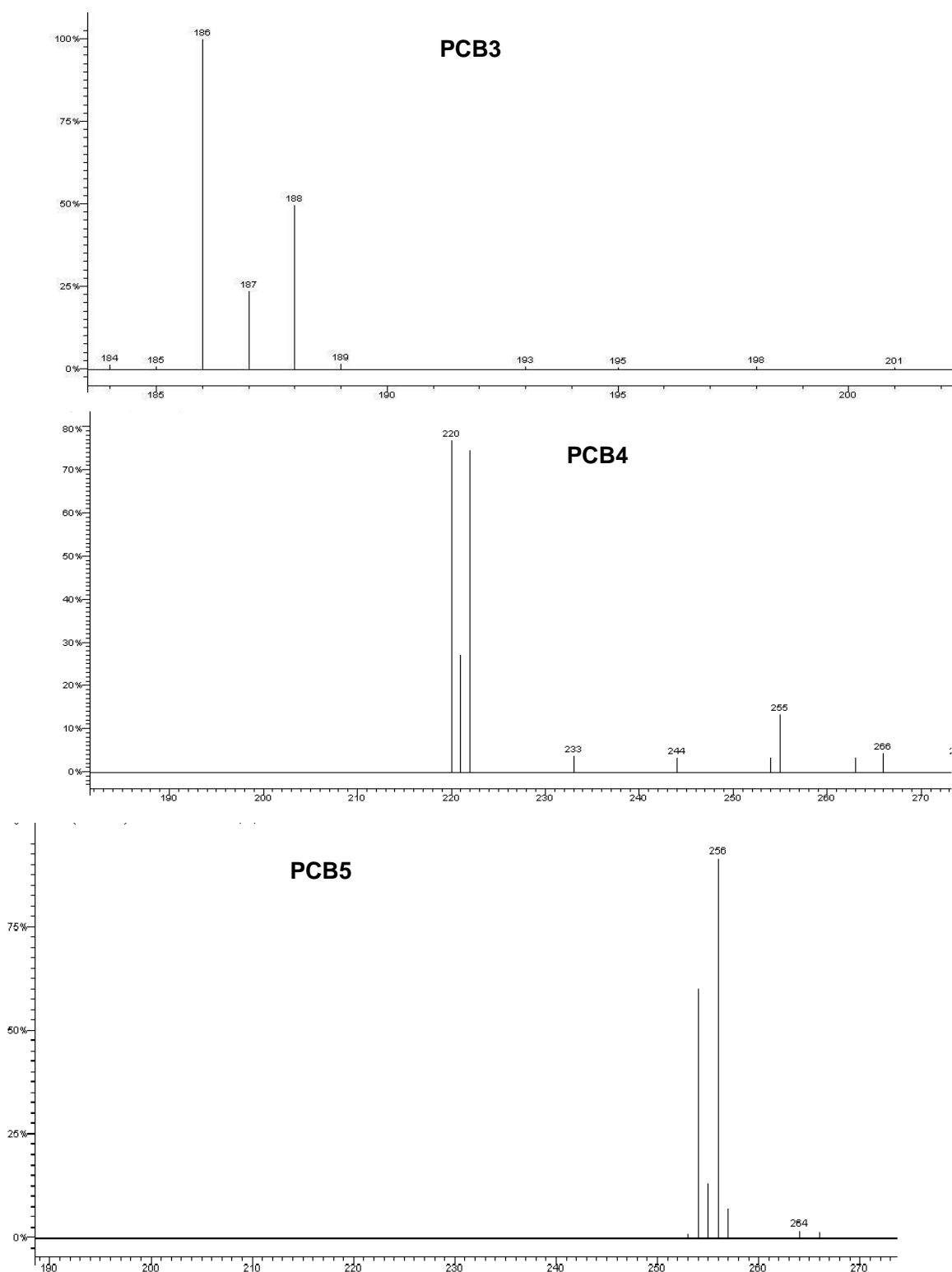


Fig C.4 Mass spectrum of different target PCBs daughter ions with MRM

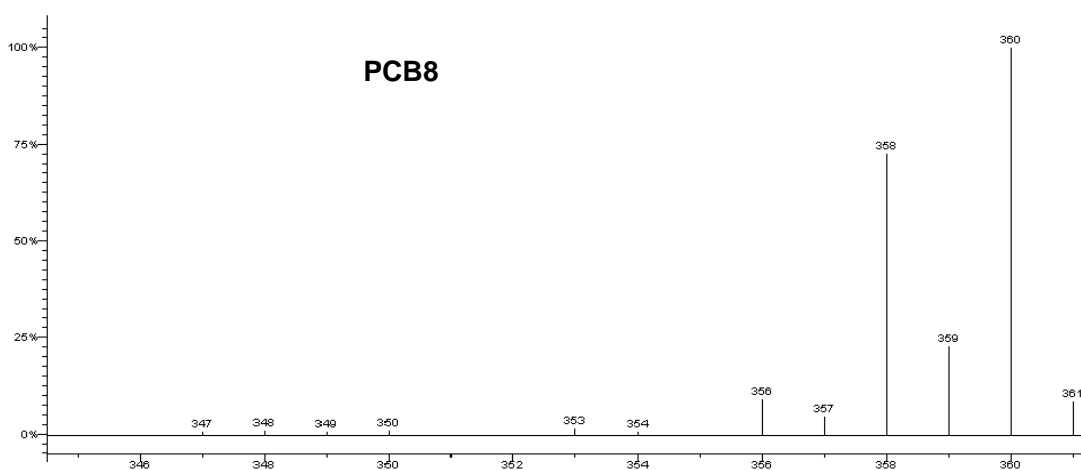
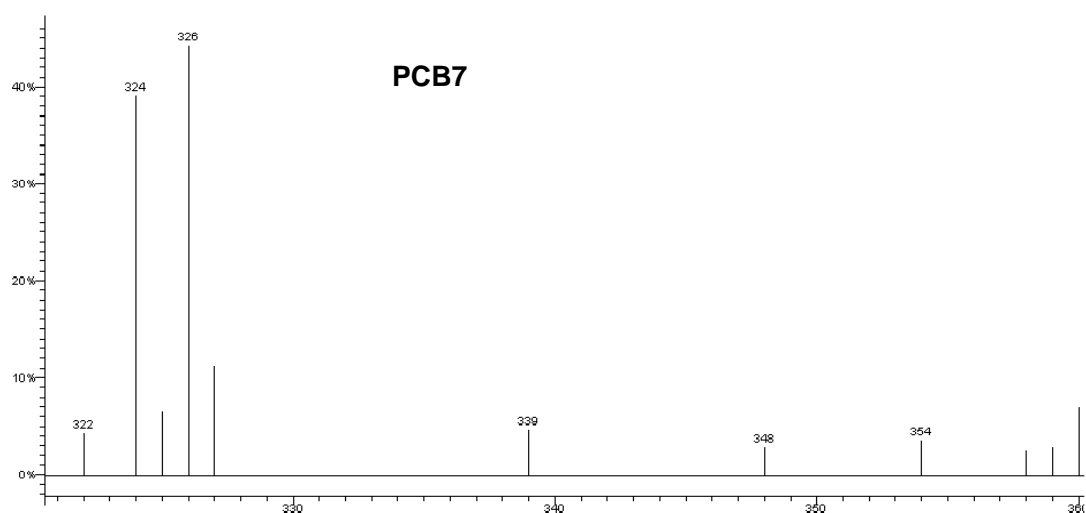
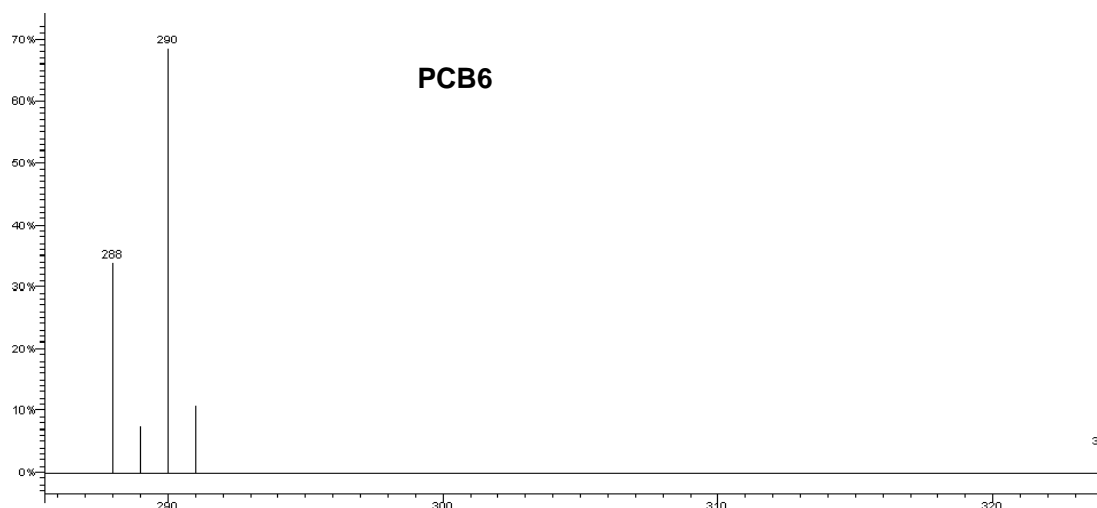


Fig C.4 (to be continued)

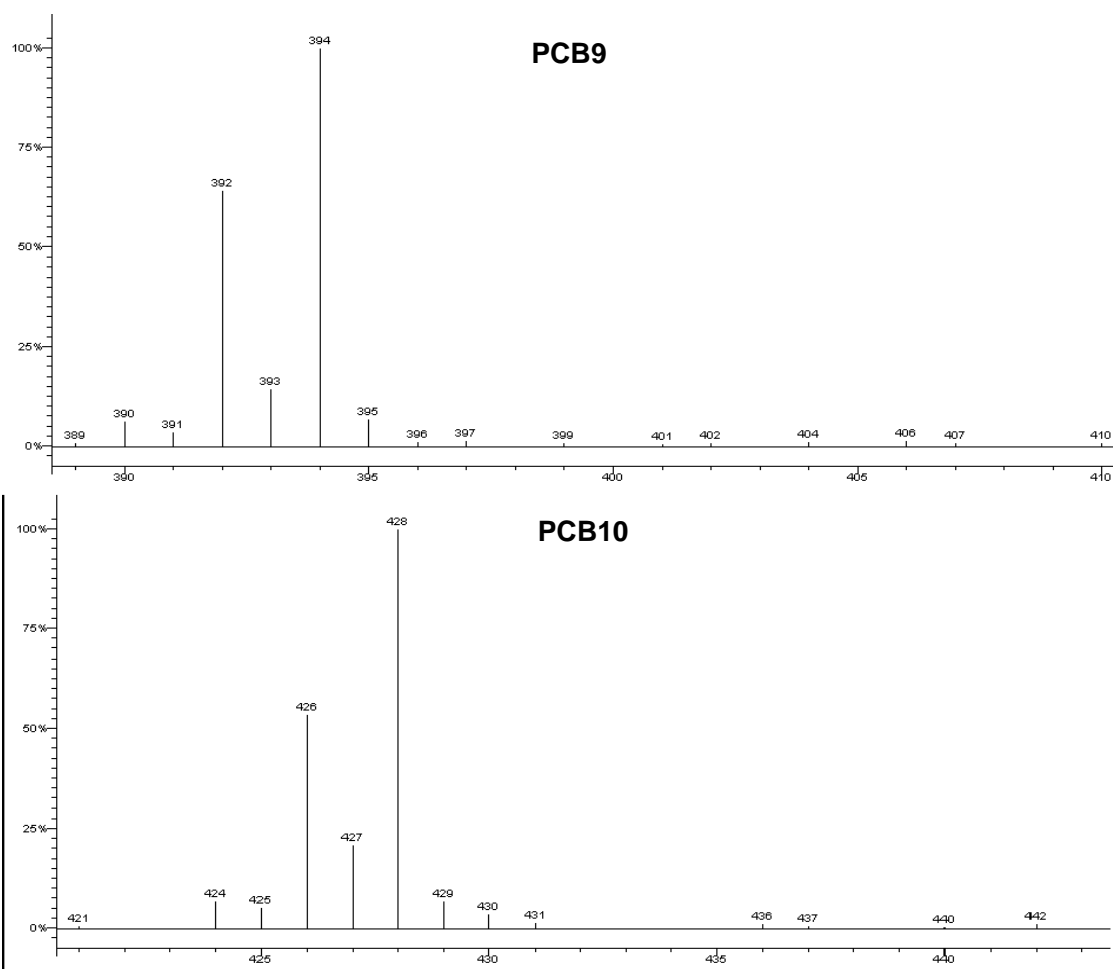


Fig C.4 (to be continued)

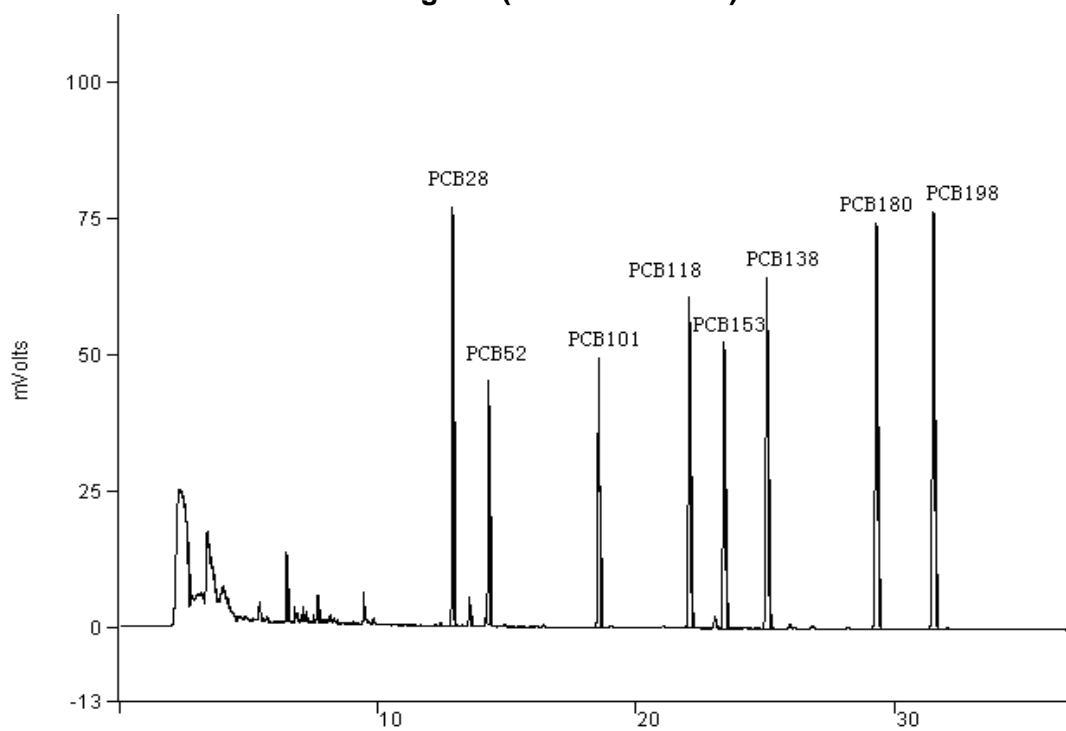


Fig C.5 Chromatogram of indicative PCB standard solution with GC-ECD

Appendix D

Purification flow chart

Fig D.1 is the decontaminating column schematic diagram;

Fig D.2 is GC-MS determination flow chart;

Fig D.3 is GC-ECD determination flow chart

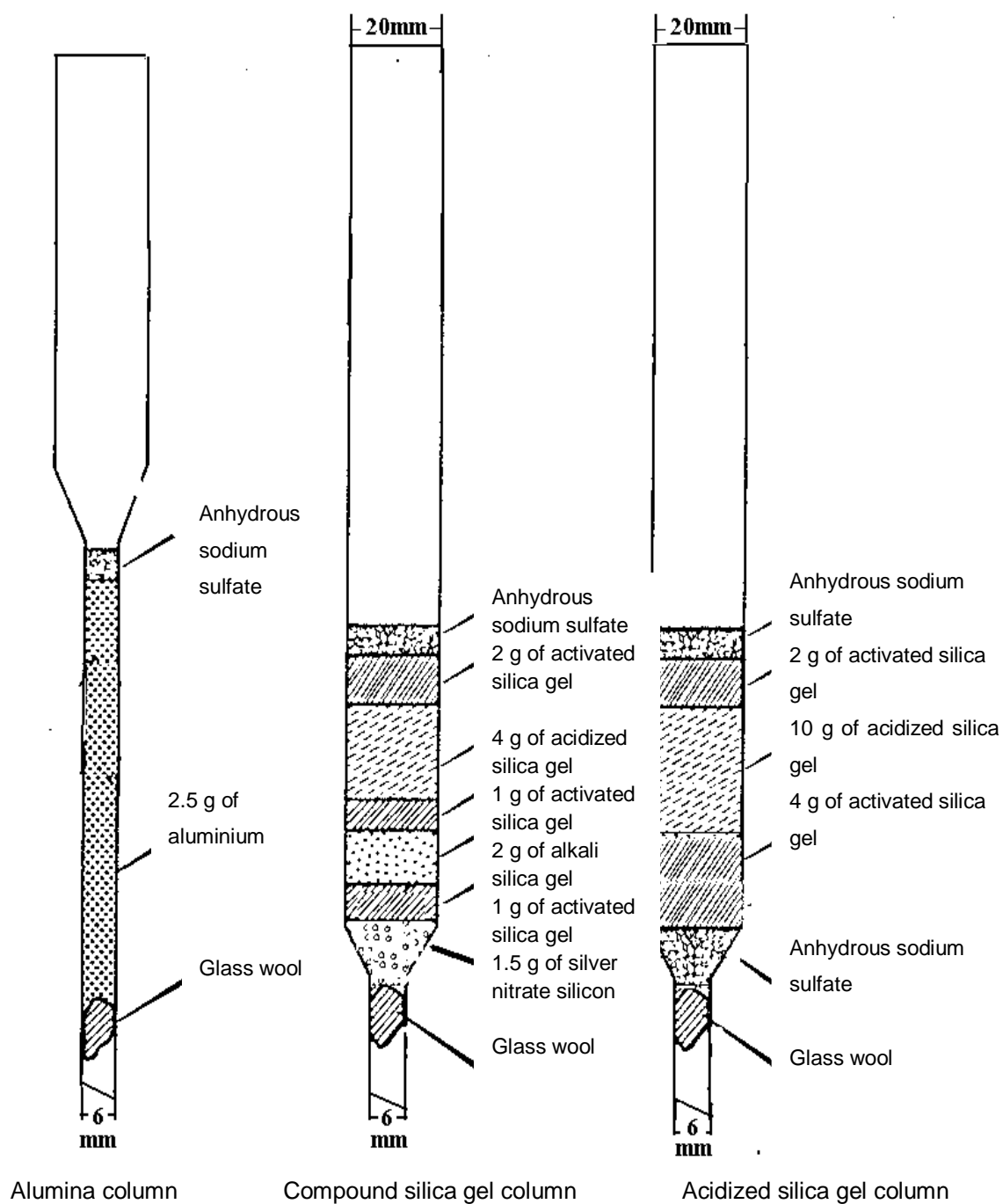


Fig D.1 Decontaminating column schematic diagram with GC-MS

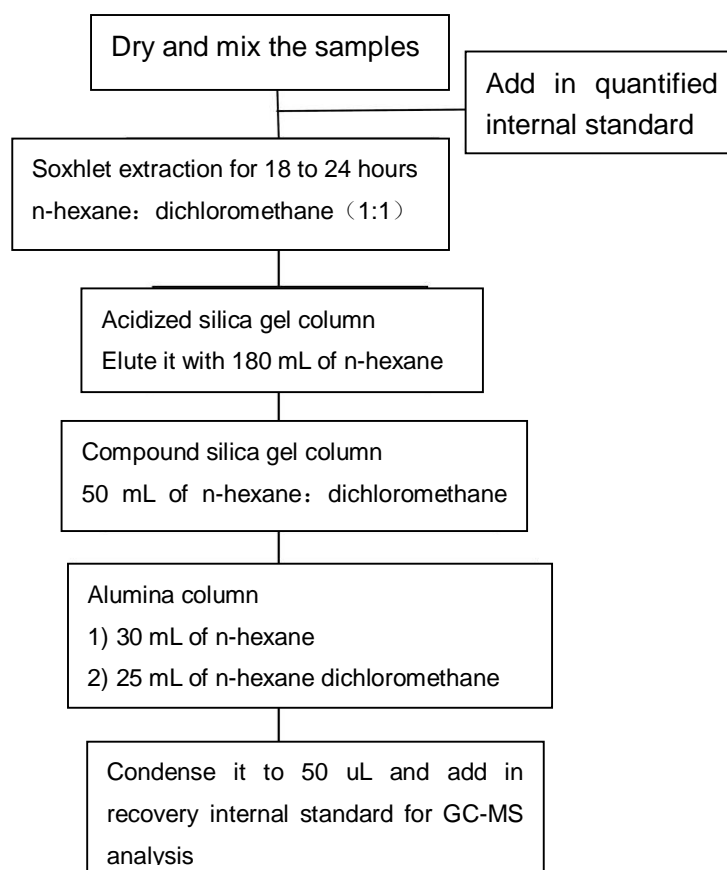


Fig D.2 Flow chart to determine the PCBs in food with isotope dilution gas chromatography-mass spectrometer

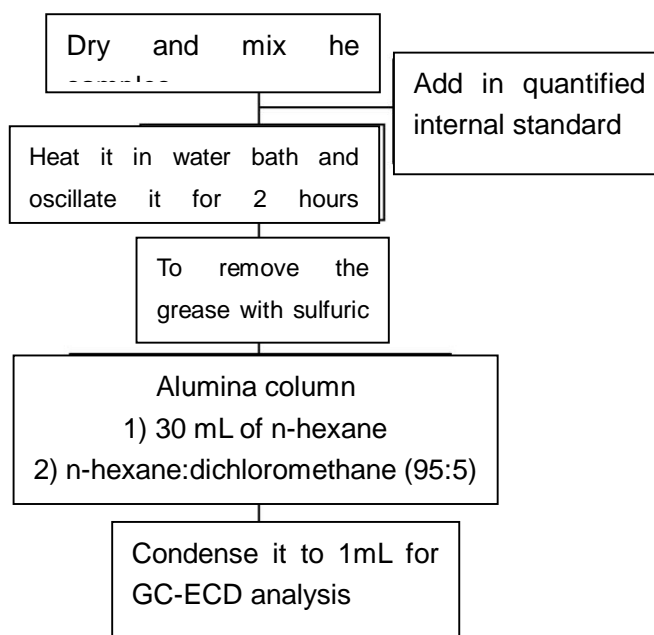


Fig D.3 Flow chart to determine the PCBs in food with the gas chromatography—electron capture detector

GB 5009.204-2014 Determination of Acrylamide in Foods



National Standards of People's Republic of China

GB 5009.204-2014

National Food Safety Standard
Determination of Acrylamide in Foods

Issued on: 2014-12-01

Implemented on: 2015-05-01

Issued by National Health and Family Planning Commission

Foreword

This standard has substituted for GB/T 5009.204—2005 Determination of Acrylamide in Food with the GC-MS method.

Compared with GB/T 5009.204—2005, this standard has the following main changes:

- Method I, Mass-spectrography, gas chromatography to dilute stable isotopes, has been added.
- The GC-MS method has changed the external standard method into stable isotopes dilution method.

National Standard for Food Safety

Determination of Acrylamide in Foods

1. Scope

The standard specifies the method to detect acrylamide in foods.

The standard is applicable to the determination of acrylamide in heat processed (fried, baked, roasted, grilled, etc) foods.

Method I Liquid chromatography-mass spectrography/ mass-spectrography to dilute stable isotopes

2. Principles

The stable isotope dilution technique is applied in the standard to add $^{13}\text{C}_{12}$ -marked PCBs into the samples with water as the extraction solvent. After the solid phase extraction or base material solid phase extraction purification, detection shall be made with the multiple reactions monitoring (MRM) of Liquid chromatography-mass spectrography/ mass-spectrography or selective reaction monitoring (SRM) and determination with the internal standard method shall be carried out.

3. Reagents and materials

Notes: Unless otherwise stated, all reagents in the method shall be analytically pure and the water shall be Grade I water specified in GB/T 6682.

3.1 Reagents

- 3.1.1 Methanoic acid (HCOOH): chromatographically pure
- 3.1.2 Methyl alcohol (CH_3OH): chromatographically pure
- 3.1.3 Normal hexane ($\text{n-C}_6\text{H}_{14}$): analytically pure, used after being re-distilled.
- 3.1.4 Ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$): analytically pure, used after being re-distilled.
- 3.1.5 Anhydrous Na_2SO_4 : baked at 400°C for 4 hours.
- 3.1.6 Ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$.
- 3.1.7 Diatomite: ExtrelutTM 20 or equivalent products.

3.2 Standard products

- 3.2.1 Acrylamide ($\text{CH}_2=\text{CHCONH}_2$) standard products (purity>99%)
- 3.2.2 3.2.2 $^{13}\text{C}_3$ -Acrylamide ($^{13}\text{CH}_2=^{13}\text{CH}^{13}\text{CONH}_2$) standard products (purity>98%)

3.3 Formulation of standard solution

3.3.1 Formulation of acrylamide standard solution

3.3.1.1 Acrylamide standard stock solution(1,000 mg/L):accurately weigh acrylamide standard products and dissolve it with methanol to make the acrylamide concentration reach 1,000 mg/L before storing it in the refrigerator at -20 °C

3.3.1.2 Acrylamide midst solution(100 mg/L): Transfer 1 mL of acrylamide standard stock solution and dilute it to 10 mL with methanol to make the acrylamide concentration reach 100 mg/L before storing it in the refrigerator at -20 °C

3.3.1.3 Acrylamide work solution I (10 mg/L): Transfer 1 mL of acrylamide midst solution and dilute it to 10 mL with 0.1% methanoic acid solution to make the acrylamide concentration reach 10 mg/L. It shall be formulated when being used.

3.3.1.4 Acrylamide work solution II (1 mg/L): Transfer 1 mL of acrylamide work solution I and dilute it to 10 mL with 0.1% methanoic acid solution to make the acrylamide concentration reach 1mg/L. It shall be formulated when being used.

3.3.2 ¹³C₃acrylamide internal standard solution

3.3.2.1 ¹³C₃-acrylamide internal standard stock solution(1,000 mg/L):accurately weigh ¹³C₃-acrylamide standard product and dissolve it with methanol to make the ¹³C₃-acrylamide concentration reach 1,000 mg/L before storing it in the refrigerator at -20 °C

3.3.2.2 Internal standard work solution(10 mg/L):Transfer 1 mL of internal standard stock solution and dissolve it to 100 mL with methanoic acid to make the ¹³C₃-acrylamide concentration reach 10 mg/L before storing it in the refrigerator at -20 °C

3.3.3 Standard curve work solution

Take 6 10-mL volumetric flasks to separately transfer 0.1 mL, 0.5 mL and 1 mL of acrylamide work solution II (1 mg/L), 1 mL and 3 mL of acrylamide work solution I (10 mg/L) and 0.1 mL of internal standard work solution (10 mg/L). Then dilute it with 0.1% methanoic acid solution to the scale. Acrylamide concentration in the standard solution shall be separately 10 µg/L, 50 µg/L, 100 µg/L, 500 µg/L, 1,000 µg/L and 3,000 µg/L. The internal standard concentration shall be 100 µg/L. It shall be formulated when being used.

4. Equipment and facilities

4.1 Liquid chromatogram-mass spectrum/mass spectrometer (LC-MS/MS)

4.2 HLB solid phase extraction column:6 mL, 200 mg, or equivalent products

4.3 Bond Elut-Accucat solid phase extraction column:3 mL, 200 mg , or equivalent products

4.4 Tissue grinder

4.5 Rotary evaporator

4.6 Nitrogen inspissator

4.7 Oscillator

4.8 Glass chromatographic column: column length--30 cm, column internal diameter --1.8 cm

4.9 Turbine mixer

4.10 Ultra-pure water equipment

4.11 Analytical balance whose sensitivity is 0.1 mg

4.12 Centrifuge: rotate speed $\leq 10,000$ r/m.

5. Analytical procedure

5.1 Specimen preparation

5.1.1 Sample extraction

Take 50 g of samples and grind them with the grinder and then store the at -20°C . Accurately weigh 1 g to 2 g of samples (correct to 0.001 g). 10 μL or 20 μL of $^{13}\text{C}_3$ -acrylamide internal standard (10 mg/L) shall be added, which is equal to 100 ng or 200 ng of $^{13}\text{C}_3$ -acrylamide internal standard. Then add 10 mL of ultra-pure water and shake it for 30 minutes to centrifuge it at 4,000 r/m for 10 minutes. Finally acquire the liquid supernatant for purification.

5.1.2 Sample purification

Notes: any of the following methods shall be chosen for purification.

5.1.2.1 Base material solid-phase dispersion extraction method (Selection 1): add 15 g of ammonium sulfate into the liquid supernatant extracted from the samples and shake for 10 minutes to fully dissolve it before centrifuging it at 4,000 r/m for 10 minutes. Get 10 mL of liquid supernatant for use. When the liquid supernatant doesn't reach 10 mL, saturated ammonium sulfate shall be used for complement. Put a little glass wool in the bottom of a pure glass chromatography column and press it tightly. Then fill in 10 g of anhydrous Na_2SO_4 and 2g of diatomite in sequence. Mix 5 g of diatomite ExtrelutTM 20 with the liquid supernatant of the above samples and then fill it in the chromatographic column. Rinse it with 70 mL of normal hexane at 2 mL/min and then remove the normal hexane. Elute the acrylamide with 70mL of ethyl acetate at 2 mL/min. Then collect the ethyl acetate elution solution and place it under the vacuum-rotary evaporation procedure at the 45°C water bath until it's nearly dry. Rinse the residue in the evaporating flask with ethyl acetate for three times (1 mL every time) and then transfer it into the test tube which already has 1 mL of 0.1% methanoic acid solution before vortex oscillation. Blow away the upper organic phase with nitrogen and add in 1 mL of normal hexane before vortex oscillation. Then centrifuge it at 3,500 r/m for 5 minutes. Filter the upper organic phase with the 0.22 μm aqueous phase filter membrane for LC-MS/MS test.

5.1.2.2 Solid phase extraction column (Selection 1): Add 5 mL of normal hexane into the liquid supernatant from the samples for a 10-minute oscillation extraction. Then centrifuge it at 10,000 r/m for 5 minutes. Remove the organic phase and extract it again with 5 mL of normal hexane. Then rapidly get 6 mL of aqueous phase and filter it with the 0.45- μm aqueous phase filter and carry out HLB solid phase extraction column purification. Activate the HLB solid phase extraction column with 3 mL of methyl alcohol and 3 mL of water. Apply 5 mL of the above filter liquor to the HLB solid phase extraction column and

collect the effluent. Then eluate it with 4 mL of 80% methanol water fluid and collect all the eluate. Then mix it with the effluent for Bond Elut-Accucat solid phase extraction column purification; Activate the Bond Elut-Accucat solid phase extraction column with 3 mL of methyl alcohol and 3mL of water. Add all the samples of eluate for the HLB solid phase extraction column purification and make it outflow under the action of gravity. Then collect all the eluate and concentrate it under the nitrogen flow until it's nearly dry. Finally dilute the 0.1% methanoic acid solution to 1.0 mL for LC-MS/MS determination.

5.2 Reference conditions for instruments

5.2.1 Chromatographic conditions

The chromatographic column shall be Atlantis C₁₈ Column (5 µm, 2.1 mm I.D.×150 mm) or equivalent columns.

Pre-column: C₁₈ Guard Column (5 µm, 2.1 mm I.D.×30 mm) or equivalent columns.

Mobile phase: methyl alcohol/0.1% methanoic acid(10:90 , volume fraction)

Flow velocity:0.2 mL/min

Sample injection volume: 25 µL

Column temperature: 26 °C

5.2.2 Mass spectrometric parameters

5.2.2.1 Triple quadrupole tandem mass spectrometer

Detection mode:multiple reaction monitoring(MRM)

Ionization mode:ESI+

Capillary voltage:3 500 V

Cone voltage:40 V

Radio-frequency lens 1 voltage: 30.8 V

Ion source temperature: 80 °C

Desolvation Temperature: 300 °C

Ion collision energy: 6 eV

Acrylamide:parent ion m/z 72, daughter ion m/z 55, daughter ion m/z 44

¹³C₃ acrylamide:parent ion m/z 75, daughter ion m/z 58, daughter ion m/z 45

Quantitative ion: acrylamide, m/z 55

¹³C₃ acrylamide:m/z 58

5.2.2.2 Ion trap tandem mass spectrometer

Detection mode: SRM

Ionization mode: ESI+

Spray voltage: 5,000 V

Heating capillary temperature: 300 °C

Sheath gas: N₂ , 40 Arb

Auxiliary gas: N₂ , 20 Arb

Collision induced dissociation (CID): 10 V

Collision Energy: 40 V

Acrylamide: parent ion m/z 72, daughter ion m/z 55, daughter ion m/z 44

¹³C₃ acrylamide: parent ion m/z 75, daughter ion m/z 58, daughter ion m/z 45

Quantitative ion: acrylamide, m/z 55 ;

¹³C₃ acrylamide: m/z 58

5.3 Mapping of standard curves

Infuse the standard work liquid into Liquid Chromatogram-Mass Spectrum/ Mass Spectrometry System to detect the peak area of corresponding acrylamide and their internal standard. Standard curves shall be mapped by taking the acrylamide sampling concentration of all standard work liquid (μg/L) as the abscissa and the peak area of acrylamide (m/z 55) and ¹³C₃ acrylamide internal standard (m/z 58) as the ordinate.

5.4 Determination of the sample solution

Infuse the sample solution into the Liquid Chromatogram-Mass Spectrum/ Mass Spectrometry System and measure the peak area ratio of acrylamide (m/z 55) and ¹³C₃ acrylamide internal standard (m/z 58). Acrylamide sampling concentration (μg/L) in the to-be-tested liquid is acquired according to standard curves. Parallel determination shall be done twice or more.

5.5 Mass spectrometry

Infuse the samples and standard work liquid into the Liquid Chromatogram- Mass Spectrum/Mass Spectrometer in sequence to record the total ion chromatogram and mass spectrum (see Fig A.1 to A.2 at Appendix A) , as well as the peak area of acrylamide and internal standard and keep the abundance

qualification of time and fragment ions. The chromatographic peak SNR of detected acrylamide shall be more than 3. The retention time of target compounds in detected samples shall be in accordance with that in the standard solution. The abundance ratio of the relevant monitoring ions of the target compounds in tested samples shall be in accordance with that in the standard solution. See the permissible deviation at Table 1.

Table 1 The maximum permissible deviation of relevant ion abundance in qualitative tests

Relevant ion abundance (base line peak)	Permissible relative deviation
>50%	±20%
>20%~50%	±25%
>10%~20%	±30%
≤10%	±50%

6. Expression of the analysis results

The acrylamide content in the samples shall be calculated according to the Formula (1) Internal Standard Method:

$$X = \frac{A \times f}{M} \quad \text{..... (1)}$$

In the formula:

X—acrylamide content in samples (µg/kg)

A—acrylamide weight which is in accordance with the peak area ratio of acrylamide(m/z 55) chromatographic peak and ¹³C₃ acrylamide internal standard (m/z 58) chromatographic peak in the samples (ng)

f—conversion factors of internal standard adding quantity in the samples (f=1 when the internal standard is 10 µL or f=2 when the internal standard is 20 µL)

M—Sample volume added into the internal standard (g)

Calculation results shall be shown with the arithmetic mean value of two individually analysis results acquired on a repetitive basis. The outcome shall be calculate to a three-effective-digit number (or to the first decimal place)

7. Precision

The absolute difference of the two independent analysis results acquired on a repetitive basis shall not exceed the 20% of the arithmetic mean value.

8. Others

Limit of quantitation is 10 µg/kg.

Method II Gas chromatography-mass spectrography to dilute stable isotopes

9. Principles

The standard shall adopt the stable isotope dilution technique and add $^{13}\text{C}_3$ -marked acrylamide internal standard solution into the samples. By taking water as the extraction solvent, the sample extracting solution makes a detection with MRM or SIM after adopting base material solid-phase dispersion extraction purification and bromide reagent derivation. Determination with the internal standard method shall be used.

10. Reagents and materials

Notes: Unless otherwise stated, all reagents in the method shall be analytically pure and the water shall be ultra-pure water.

10.1 Reagents

- 10.1.1 Normal hexane ($n\text{-C}_6\text{H}_{14}$): analytically pure, used after being re-distilled.
- 10.1.2 Ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$): analytically pure, used after being re-distilled.
- 10.1.3 Anhydrous Na_2SO_4 : baked at 400°C for 4 hours.
- 10.1.4 Ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$.
- 10.1.5 Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$).
- 10.1.6 Bromine (Br_2).
- 10.1.7 Hydrobromic acid: content $>48.0\%$.
- 10.1.8 Potassium bromide (KBr).
- 10.1.9 Ultra-pure water, conductivity (25°C) ≤ 0.01 mS/m.
- 10.1.10 Brominating agent.
- 10.1.11 Diatomite: ExtrelutTM 20 or equivalent products.

10.2 Reagent compounding

10.2.1 Saturated bromine water: Measure 100 mL of ultra-pure water and place them in the 200-mL brown reagent bottle. Then add in 8 mL of bromine and keep it out of the sun at 4°C for 8 hours. The upper level is saturated bromine solution.

10.2.2 Brominating agent: Weigh 20.0 g of potassium bromide and add 50 mL of ultra-pure water to totally dissolve it. Then add in 1.0 mL of hydrobromic acid and 16.0 mL of saturated bromine water. Shake it up and dilute it to 100 mL with ultra-pure water. Keep it out of the sun at 4°C .

10.2.3 Sodium thiosulfate solution (0.1 mol/L): Weigh 2.48 g of sodium thiosulfate and add in 50 mL of ultra-pure water to totally dissolve it. The dilute it to 100 mL with ultra-pure water and keep it out of the sun at

4 °C

10.2.4 Saturated ammonium sulfate solution: Weigh 80 g of ammonium sulfate crystals and add in 100 mL of ultra-pure water. Dissolve it with the ultrasound and place it at the room temperature.

10.3 Standard products

10.3.1 Acrylamide ($\text{CH}_2=\text{CHCONH}_2$) standard products: purity>99%

10.3.2 13C 3-acrylamide($^{13}\text{CH}_2=^{13}\text{CH}^{13}\text{CONH}_2$)standard products: purity>98%

10.4 Formulation of standard solutions

10.4.1 Acrylamide and its internal solution: alike to 3.3.1 and 3.3.2.

10.4.2 Standard curve work solution: Take 5 10-mL volumetric flasks to separately transfer 0.1 mL, 0.5 mL and 2 mL of acrylamide work solution I(1 mg/L) and 0.5 mL and 1 mL of crylamide work solution I(1 mg/L), as well as 0.5 mL of internal standard work solution(1 mg/L), with 5 10-mL volumetric flasks. The acrylamide concentration in the standard solution shall be 10 µg/L, 50 µg/L, 200 µg/L, 500 µg/L and 1,000 µg/L. The internal concentration shall be 50 µg/L. It shall be formulated when being used.

11 Equipment and facilities

11.1 Gas chromatograph-quadrupole mass spectrometer(GC-MS)

11.2 Chromatographic column:DB-5ms column (30 m×0.25 mm×0.25 µm) or equivalent chromatographic column

11.3 Tissue grinder

11.4 Rotary evaporator

11.5 Nitrogen concentrator

11.6 Oscillator

11.7 Glass chromatographic column: column length-30 cm, column internal diameter -1.8cm

11.8 Turbine mixer

11.9 Ultra-pure water equipment

11.10 Analytical balance whose sensitivity is 0.1 mg

11.11 Centrifugal machine: revolving speed≤10,000 r/m

12 Analytical procedures

12.1 Specimen preparation

12.1.1 Sample extraction

Grind 50 g of the samples with the grinder and store it at -20°C. Accurately weigh 2 g of samples (correct to 0.001 g). Add 10 µL or 20 µL of 13C3-acrylamide internal standard solution (10.0 mg/L), which is equal to 100 ng or 200 ng of 13C3-acrylamide internal standard solution. Shake it for 30 minutes and centrifuge it at 4,000 r/m for 10 minutes. Collect the liquid supernatant for use.

12.1.2 Sample purification

Add 15 g of ammonium sulfate to the liquid supernatant extracted from the samples and shake it for 10 minutes to fully dissolve it. Centrifuge it at 4,000 r/m for 10 minutes. Collect 10 mL of liquid supernatant for use. Complement the liquid supernatant with saturated ammonium sulfate when the liquid supernatant is less than 10 mL. Fill a little glass wool in the bottom of a clean glass chromatographic column and press it tight. Then fill in 10 g of anhydrous Na₂SO₄ and 2 g of ExtrelutTM 20 diatomite. Weigh 5g of ExtrelutTM 20 diatomite and evenly mix it with the above prepared liquid supernatant samples before filling it in the chromatographic column. Rinse it with 70 mL of normal hexane and keep the flow velocity at 2 mL/min. Then remove the normal hexane eluent. Elute it with 70 mL of ethyl acetate and keep the flow velocity at 2 mL/min. Then collect the ethyl acetate elution and nearly dry it under decompression and rotary evaporation in the 45 °C water bath. Rinse the residues in the evaporating flasks with ethyl acetate for three times (1 mL every time). Transfer it to the test tube with 1 mL of ultra-pure water for vortex oscillation. Blow away the upper organic phase in the nitrogen flow and add 1 mL of normal hexane for vortex oscillation. Then centrifuge it at 3,500 r/m for 5 minutes and collect the lower aqueous phase for derivation.

12.1.3 Derivation

Sample derivation: add 1 mL of brominating agent into the sample extracting solution for vortex oscillation. Place it at 4 °C for at least 1 hour and add in approximately 100 µL of sodium thiosulfate solution (0.1 mol/L) for vortex oscillation to remove the residual derivative agents. Add in 2 mL of ethyl acetate for vortex oscillation for 1 minute. Centrifuge it at 4,000 r/m for 5 minutes. Transfer the upper organic phase to the test tube which contains 0.1 g of anhydrous sodium sulfate. Then repeat the extraction with 2 mL of ethyl acetate and combine the organic phase. Still it for at least half an hour and transfer it to the other test tube before blowing it until it's nearly dry. Then add in 0.5 mL of ethyl acetate to dissolve the residue and leave it for use (Notes: the volume of ethyl acetate which is used to dissolve the residues shall be adjusted according to the sensitivity of instruments. Usually, its dosage shall be 0.5 mL when tandem mass spectrometers are used for detection and 0.1 mL when monopole mass spectrometers are used).

Derivation of standard solution: measure several units of 1.0 mL standard solution and synchronously carry out the above sample derivation method.

13 Reference conditions for instruments

13.1 Chromatographic conditions

Chromatographic column: DB-5ms column (30 m×0.25 mm I.D×0.25 µm) or equivalent chromatographic column

Injection port temperature: remain at 120 °C for 2 minutes and then rise to 240 °C at 40 °C/min where it stays for 5 minutes.

Chromatographic column program temperature: remain at 65 °C for 1 minute and then rise to 200 °C at 15 °C/min and then rise to 240 °C at 40 °C/min where it stays for 5 minutes.

Carrier gas: high-purity helium (purity>99.999%), Pre-column pressure-69 mPa, equal to 10 psi.

Unsplit stream sampling: injection volume-1 μL

13.2 Mass spectrometric parameters

Detection mode: SIM collection

Ionization mode: Electron Impact (EI), energy 70 eV

Transmission line temperature: 250 $^{\circ}\text{C}$

Ion source temperature: 200 $^{\circ}\text{C}$

Solvent delay: 6 min

Mass spectrum acquisition time: 6 min~12 min

Acrylamide monitoring ions: m/z 106, 133, 150 and 152

Quantitative ion: m/z 155

13.3 Formulation of standard curves

Infuse the derivative standard work liquid into Gas Chromatography-Mass Spectrometry System to detect the peak area of corresponding acrylamide and their internal standard. Linearity curves shall be mapped by taking the acrylamide sampling concentration of all standard work liquid($\mu\text{g/L}$) as the abscissa and the peak area of $^{13}\text{C}_3$ acrylamide internal standard detected on the quantitative ion quality chromatogram as the ordinate.

13.4 Determination of sample solution

Infuse the derivative sample solution into the Gas Chromatography-Mass Spectrometry System and measure the peak area ratio of acrylamide and $^{13}\text{C}_3$ acrylamide internal standard. Acrylamide sampling concentration ($\mu\text{g/L}$) in the to-be-tested liquid is acquired according to standard curves. Parallel determination shall be done twice or more.

13.5 Mass spectrometric analysis

Infuse the samples and standard work liquid into the Gas Chromatography/Mass Spectrometer in sequence to record the total ion chromatogram and mass spectrum (see Fig A.3 to A.4 at Appendix A), as well as the peak area of acrylamide and internal standard and keep the abundance qualification of time and fragment ions. The chromatographic peak SNR of detected acrylamide shall be more than 3. The retention time of target compounds in detected samples shall be in accordance with that in the standard solution. The abundance ratio of the relevant monitoring ions of the target compounds in tested samples shall be in accordance with that in the standard solution. See the permissible deviation at Table 1.

14 Expression of the analysis results

The acrylamide content in the samples shall be calculated according to the Formula (1) Internal Standard Method.

Calculation results shall be shown with the arithmetic mean value of two individually analysis results acquired on a repetitive basis. The outcome shall be calculated to a three-effective-digit number (or to the first decimal place).

15 Precision

The absolute difference of the two independent analysis results acquired on a repetitive basis shall not exceed the 20% of the arithmetic mean value.

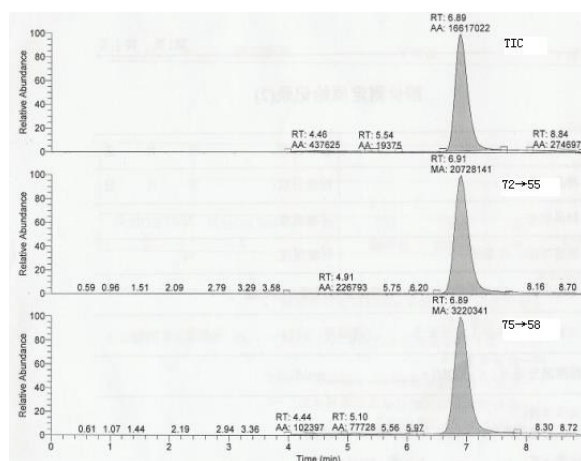
16 Others

Limit of quantitation is 10 µg/kg.

Appendix A:

Chromatogram and mass spectrum

A.1 Fig A.1 and A.2 are mass chromatogram and mass spectrum that are detected with LC-MS/MS.



Notes: From up to down is Total Ion Chromatogram (TIC), Acrylamide Selected Ion Flow Diagram(72→55)and 13C3-acrylamide Internal Standard Selected Ion Flow Diagram(75→58).

Fig A.1 Mass chromatogram of acrylamide and isotope internal standard 13C -acrylamide in potato chips

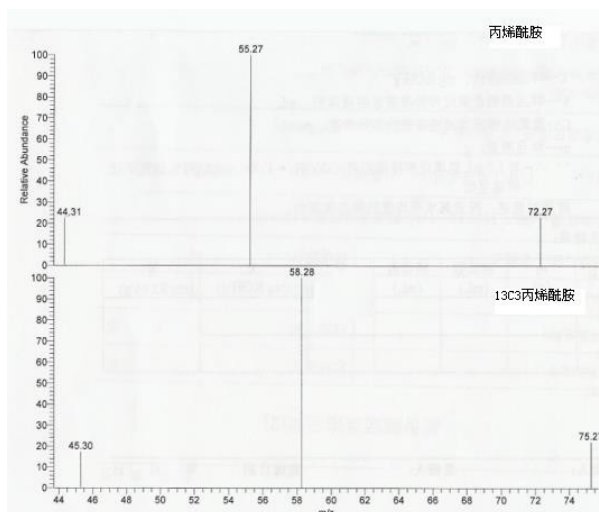
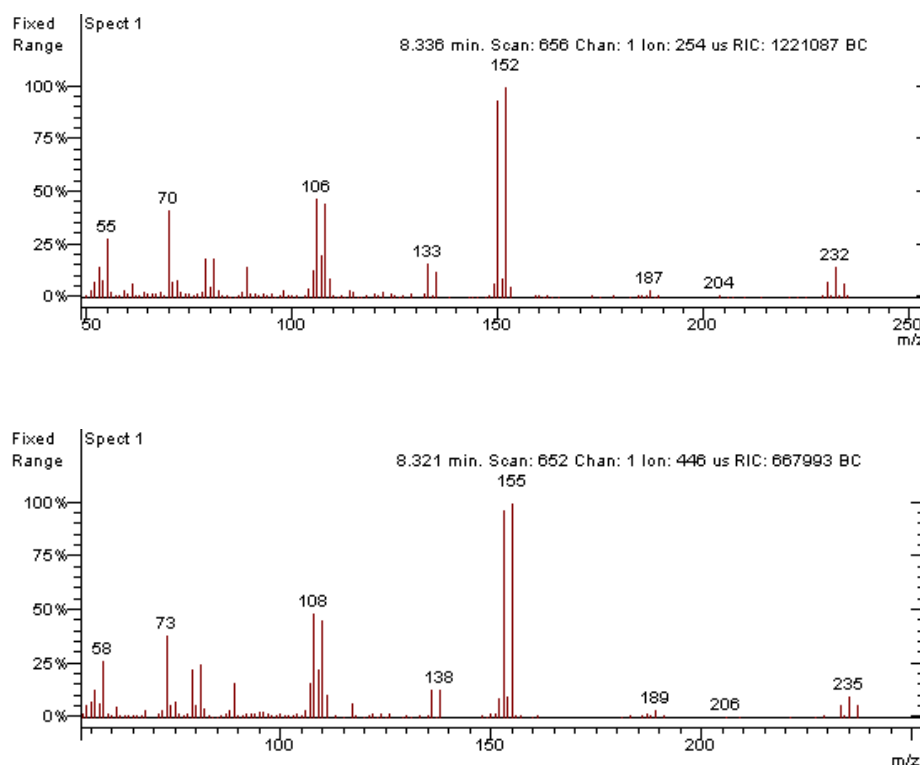
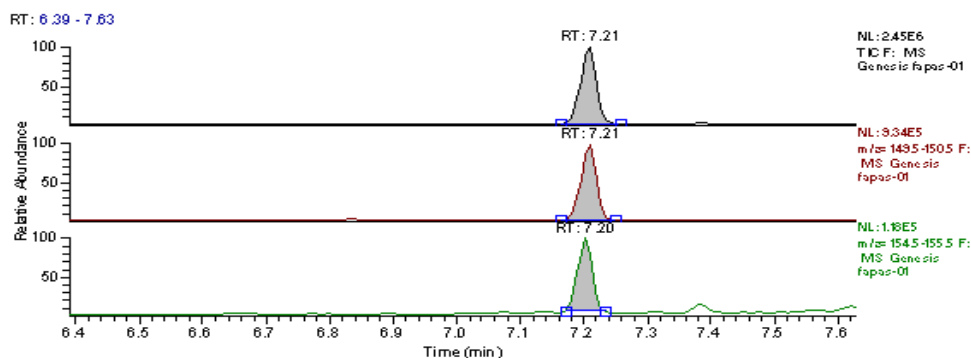


Fig A.2 Mass spectrum of acrylamide and internal standard 13C -acrylamide



Notes: the above figure:acrylamide;the below figure:13C3-acrylamide

Fig A.3 GC-MS full scan mass spectrum of bromo-derivation in the standard solution



Notes: From up to down is Total Ion Chromatogram (TIC) and mass chromatogram of acrylamide derivative m/z 150 and 13C3-acrylamide derivative m/z 155.

Fig A.4 GC-MS mass chromatogram (Quadrupole) of potato chip samples

GB 5009.223-2014 Determination of Urethane in Foods



National Standards of People's Republic of China

GB 5009.223-2014

National Food Safety Standard
Determination of Urethane in Foods

Issued on: 2015-01-28

Implemented on: 2015-01-28

Issued by National Health and Family Planning Commission

National Standard for Food Safety

Determination of Urethane in Foods

1. Scope

This standard specifies the determination of urethane content in beer, wine, rice wine, liquor and other alcoholic as well as in soy sauce by gas chromatography - mass spectrometry.

This standard applies to determination of urethane content in beer, wine, rice wine, liquor and other alcoholic as well as in soy sauce.

2. Principle

After the specimen is added with D5-urethane internal standard substance, it will be purified and eluted by alkaline diatomite SPE column. The elution solution will be calibrated by gas chromatography - mass spectrometry after it is being concentrated. Limit of qualification will be fixed by internal standard method.

3. Reagents and materials

Note: except otherwise specified, all reagents used in this method are analytical pure reagents and water are Class III water specified in GB/T6682.

3.1 Reagents

- 3.1.1 Anhydrous sodium sulfate (Na_2SO_4) .
- 3.1.2 Sodium chloride (NaCl).
- 3.1.3 Hexane (C_6H_{14}): chromatographic pure.
- 3.1.4 Ethyl acetate ($\text{C}_4\text{H}_8\text{O}_2$): chromatographic pure.
- 3.1.5 Ether ($\text{C}_4\text{H}_{10}\text{O}$): chromatographic pure.
- 3.1.6 Methanol (CH_4O): chromatographic pure.
- 3.1.7 Alkaline diatomite SPE column: packing of 4,000 mg, and column capacity of 12 ml.

3.2 Reagent compounding

- 3.2.1 Anhydrous sodium sulfate: baked for 4h at 450°C and stored in a dryer for standby application after it cools down.
- 3.2.2 5% ethyl acetate - ether solution: take 5 ml of ethyl acetate, dilute it with ether solution to 100 ml, and mix it well.

3.3 Standard products

- 3.3.1 Urethane standard product ($\text{C}_3\text{H}_7\text{O}_2\text{N}$, CAS: 51-79-6): with a purity greater than 99.0%.
- 3.3.2 D5-urethane standard product ($\text{C}_3\text{H}_2\text{D}_5\text{NO}_2$, CAS: 73962-07-9): with a purity greater than 98.0%.

3.4 Formulation of standard solution

3.4.1 D5-urethane stock solution (1.00 mg/ml): weigh and take 0.01g (accurate to 0.0001 g) of D5-urethane standard product, dissolve it with methanol solution, dilute it to a constant volume of 10 ml, and keep it at 4°C

3.4.2 D5- urethane use solution (2.00 µg/ml): imbibe 0.10 ml of D5-urethane use solution (1.00 mg/ml), dilute it with methanol to 50 ml, and store it at 4°C or below.

3.4.3 Urethane stock solution (1.00 mg/ml): weigh and take 0.05g (accurate to 0.0001g) of urethane standard product, dissolve it with methanol and dilute it to constant volume of 50 ml, and store at 4°C or below. Shelf life lasts 3 months.

3.4.4 Urethane intermediate solution (10.0 µg/ml): imbibe and take 1.00 ml of urethane stock solution (1.00 mg/ml), dilute it with methanol to a constant volume of 100 ml, and store at 4°C or below. The shelf life lasts 1 month.

3.4.5 Urethane intermediate solution (0.50 µg/ml): imbibe and take 5.00 ml of urethane intermediate solution (10.0 µg/ml), and dilute it by methanol to a constant volume of 100 ml, which is to be prepared right away when needed.

3.4.6 Standard curve working solution: imbibe and take 20.0 µl, 50.0 µl, 100.0 µl, 200.0 µl, and 400.0 µl of urethane intermediate solution (0.50 µg /ml) as well as 40.0 µl and 100.0 µl of urethane intermediate solution (10.0µg /ml) respectively; transfer them into seven 1 ml volumetric flasks; add 100 µl of D5-urethane use solution with a concentration of 2.00 µg/ml respectively and dilute it by methanol to mark; standard curve solutions with a concentration of 10.0 ng/ml, 25.0 ng/ml, 50.0 ng/ml, 100 ng/ml, 200 ng/ml , 400 ng/ml, and 1000 ng/ml result. It shall be prepared right away when needed.

4 Equipment and facilities

4.1 Gas chromatography - mass spectrometer, equipped with electron bombardment source (EI)

4.2 Vortex mixer

4.3 Nitrogen blowing instrument

4.4 Solid phase extraction device, equipped with vacuum pump

4.5 Ultrasonic cleaning machine

4.6 Muffle furnace

4.7 Balances: sensor volume of 0. 1 mg and 1mg.

5 Analytical procedures

5.1 Specimen preparation

Shake up the specimen; weigh and take 2 g (accurate to 0.001 g) of specimen (weigh and take 5min later after beer is ultrasonically degassed); add 100.0 µl of D5-urethane use solution with a concentration of 2.00 µg/ml as well as 0.3 g of sodium chloride (there is no need to add sodium chloride in case of soy sauce); dissolve it ultrasonically, mix it well and add specimen onto alkaline diatomite SPE column; let the specimen seep into the SPE column slowly under the vacuum condition and let it stand for 10 min. After being

sprinkled and washed by 10 ml of n-hexane, elute it by 10 ml of 5% ethyl acetate-ether solution at a flow rate of approx. 1 ml/min. After being dehydrated by a glass funnel containing 2 g of anhydrous sodium sulfate, the resulting elution solution will be loaded into a 10 ml graduated test tube; blow it slowly by nitrogen gas to 0.5 ml or so at room temperature, dilute it to a constant volume of 1.00 ml by methanol, thus getting assay solution prepared, which might be used for GC / MS analysis.

5.2 Reference conditions for equipment

- Reference conditions of gas chromatography - mass spectrometer analysis:
- Capillary column: DB-INNOWAX, 30 m × 0.25 mm (inner diameter) × 0.25 µm (film thickness) or equivalent capillary column
- Injection port temperature: 220°C
- Column temperature: initial temperature of 50°C holding for 1min and rising to 180°C at a rate of 8°C/min; after program running finishes, run 5 min after the temperature reaches 220°C
- Carrier gas: helium gas, purity ≥ 99.999%, a flow rate of 1 ml/min;
- Ionization mode: electron bombardment source (EI), with an energy of 70 eV;
- Quadrupole temperature: 150°C
- Ion source temperature: 230°C
- Transfer line temperature: 250°C
- Solvent delay: 11 min
- Injection mode: splitless
- Injection volume: 1 µl ~ 2 µl
- Detection mode: selected ion monitoring (SIM)
- Urethane selected ion monitoring (m/z): 44, 62, 74, 89, quantitative ion 62;
- D5-urethane selected ion monitoring (m/z) 64, 76, quantitative ion 64.

5.3 Qualitative determination

Determine the standard working solution and specimen as per the conditions applicable to the method; constant volume may be reduced when specimen of low concentration is to be characterized. Allowable tolerance between mass chromatographic peak retention time of the specimen and that of the reference substance shall be less than ± 2.5%; allowable tolerance between the relative abundance and concentration of qualitative ion pairs vs. that of the standard working solution shall not exceed that specified in Table 1.

Table 1 Maximum allowable tolerance of relative ion abundance when quantitation is confirmed

Relative ion abundance /%	> 50	> 20~50	> 10~20	≤10
Maximum allowable tolerance /%	±50	±25	±30	±50

5.4 Quantitative determination

5.4.1 Standard curve mapping

Conduct gas chromatography - mass spectrometer determination over urethane standard working curve solutions of 10.0 ng/ml, 25.0 ng/ml, 50.0 ng/ml, 100 ng/ml, 200 ng/ml, 400 ng/ml, and 1000 ng/ml (containing 200 ng/ml), with urethane concentration being taken as the horizontal axis, and peak area ratio between urethane and D5-urethane in the standard curve working solution as the vertical axis to map the standard curve.

5.4.2 Specimen determination

Determine the specimen solution against the standard curve working solution; calculate urethane content in the specimen as per that in the determination solution of urethane. When the specimen has a low concentration of urethane, it is advised to take the standard curve working solution of 10.0 ng/ml, 25.0 ng/ml, 50.0 ng/ml, 100 ng/ml, and 200 ng/ml map the standard curve.

Please refer to Annex A for mass spectrum of the standard solution.

6 Statement of analysis result

Urethane content in the specimen is to be calculated as per Formula (1):

$$X = \frac{c \times V \times 1\,000}{m \times 1\,000} \dots\dots\dots (1)$$

In the formula:

X- Urethane content in the specimen, in microgram per kilogram (µg/kg)

c- urethane content in determination solution, in nanogram per milliliter (ng/ml)

V- constant volume of specimen determination solution, in milliliter (ml)

m- specimen mass, in gram (g)

1000 - conversion coefficient.

The calculated results shall be represented by the arithmetic mean of two independent measurement results obtained under repeatability conditions and three-digit valid number shall be retained.

7 Precision

As regards the relative deviation of two independent determination results obtained under repeatability conditions, when the content is $\leq 50 \mu\text{g/kg}$, it shall not exceed 15% of the arithmetic mean; when the content is $>50 \mu\text{g/kg}$, it shall not 10% of the arithmetic mean.

8 Others

When 2 g of specimen is taken, the detection limit of urethane in this method shall be $2 \mu\text{g/kg}$ and the limit of quantification be $5.0 \mu\text{g/kg}$.

Appendix A

Mass spectrum of the standard solution

A.1 Total ion of urethane and D5- urethane is shown in Figure A.1.

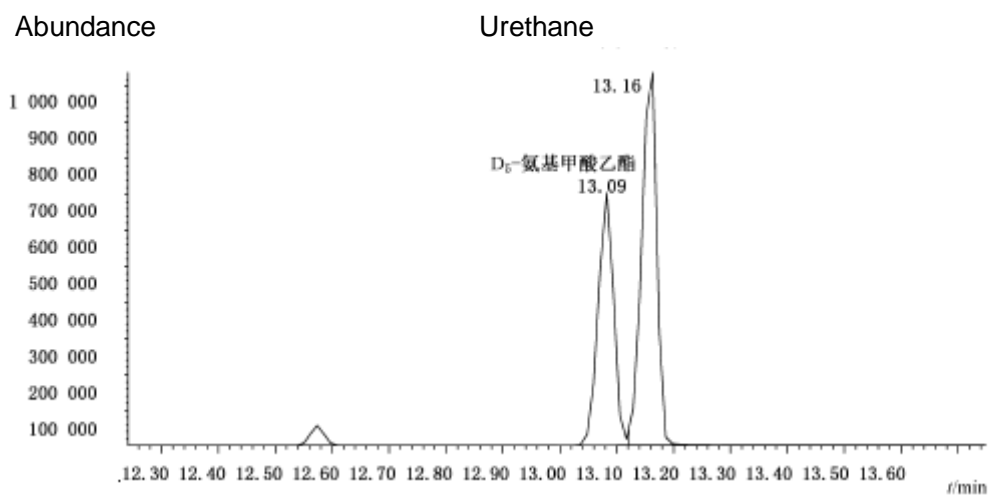


Fig. A.1 Total ion of urethane and D5-urethane

A.2 Urethane mass spectrum is shown in Figure A.2.

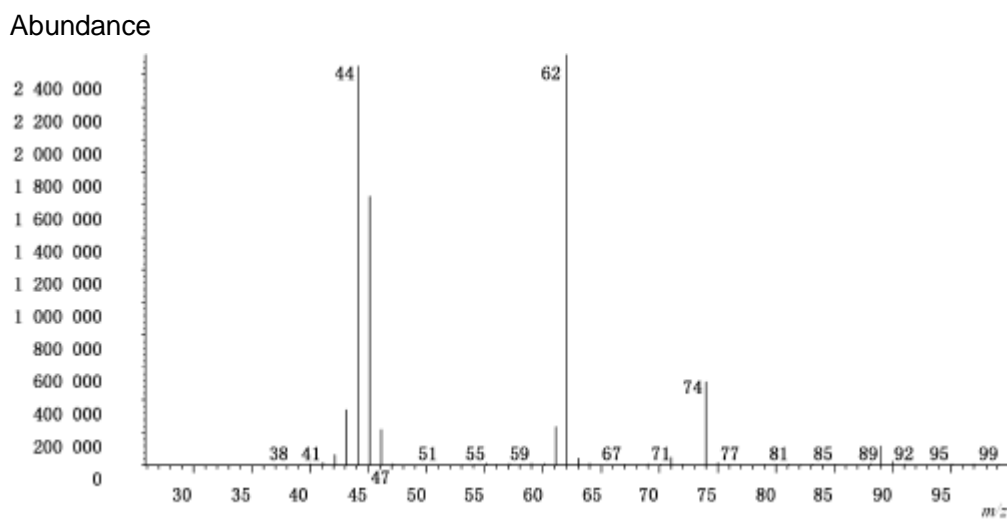


Fig. A.2 Urethane mass spectrum

A.3. D5-urethane mass spectrum is shown in Figure A.3.

Abundance

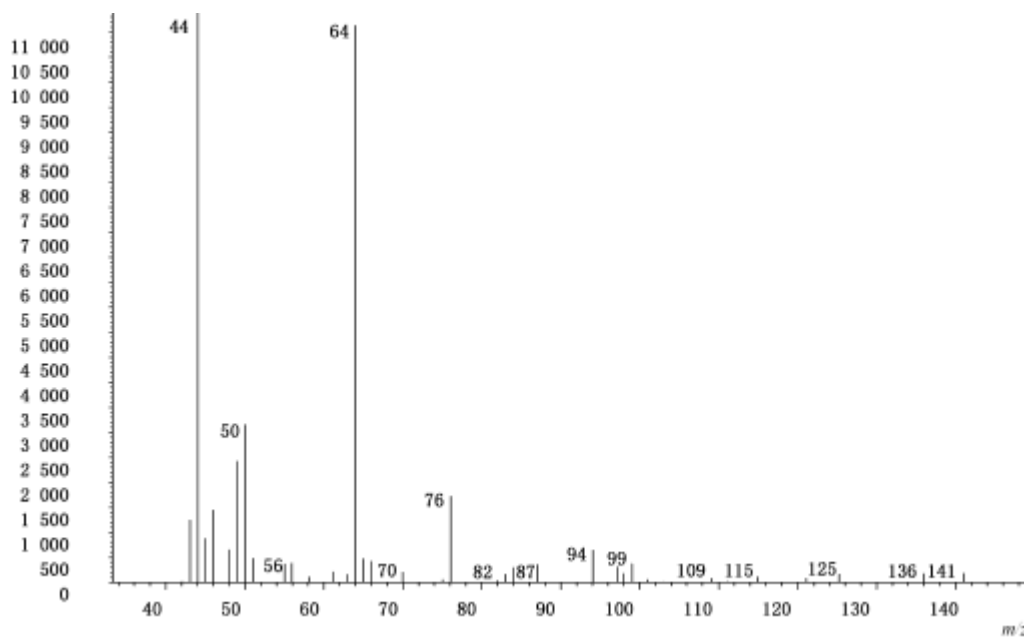
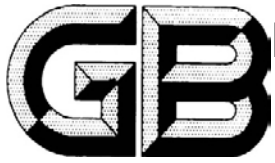


Fig. A.3 D5-urethane mass spectrum

Other Regulations

GB 14881-2013 General Hygienic Regulation for Food Production



National Standards of People's Republic of China

GB 14881-2013

National Food Safety Standard General Hygienic Regulation for Food Production

Issued on: 2013-05-24

Implemented on: 2014-07-01

Issued by National Health and Family Planning Commission

Foreword

This Standard replaces the General Hygiene Regulations for Food Enterprises (GB14881-1994). This standard modifies the GB14881-1994 in the following aspects:

- Changes name of the standard;
- Modifies structure of the standard;
- Adds terms and definitions;
- Emphasizes food safety control requirements in the entire food production process, namely raw material purchase, processing, product storage and transportation; it also lists major control measures for biological, chemical and physical contaminations;
- Modifies sections related to production equipment; the standard sets requirements on layout, materials and design of the production equipment from the perspective of preventing biological, chemical and physical contaminations;
- Adds relevant requirements for the procurement, inspection and acceptance, transportation and storage of raw materials;
- Adds specific requirements on product traceability and recall;
- Adds requirements on record keeping and document management;
- Adds Appendix A: *Guide of Monitoring Procedure for Microorganism in the Food Production Environment*”.

National Standard for Food Safety

General Hygiene Regulations for Food Production

1. Scope

This standard specifies basic requirements and management rules for locations, facilities and personnel of material purchasing, processing, packaging, storage and transportation in the process of food production.

This standard is applicable to production of various kinds of food; if it's necessary to develop a special hygienic regulation for a certain kind of food production, this standard shall be taken as a basis.

2. Terms and Definitions

2.1 Contamination

Process of biological, chemical and physical contamination factors transferred in the process of food production.

2.2 Insect pest

Adverse effect caused by creatures such as insect, bird or rodent including fly, cockroach, sparrow and rat.

2.3 Food processing personnel

Operation personnel directly contacting packaged or unpackaged food, food equipment and instrument and food contact surface.

2.4 Contact surface

Contactable surface of equipment, tools and instruments or human body.

2.5 Separation

Articles, facilities and areas are separated by leaving a certain space between one another instead of arranging physical blockage.

2.6 Partition

Articles, facilities and areas are separated by arranging physical blockage such as wall, hygienic barrier, shade or independent room.

2.7 Food processing location

Building and site for food processing and other buildings, sites and surrounding environment managed in the same way.

2.8 Monitoring

Observation or determination carried out according to the preset way and parameter to evaluate whether the controlling unit is under the controlled state.

2.9 Work clothes

Specialized clothes equipped to reduce the contamination risk of food processing personnel on food according to the requirements of different production areas.

3. Site Selection and Plant Surroundings

3.1 Site selection

3.1.1 For the plant, areas which have large contamination on food shall not be selected. If a place has obviously adverse effect which can't be improved by taking measures on food safety and food edibility, the plant shall not be built in the place.

3.1.2 For the plant, sites where hazardous waste, dust, harmful gas, radioactive substance and other diffusive contaminants can't be eliminated effectively shall not be selected.

3.1.3 For the plant, regions where flood disaster can easily occur should not be selected; if it's difficult to keep away, necessary precaution measures shall be designed.

3.1.4 There should not be potential locations with a large number of insect pest breeding around the plant; if it's difficult to keep away, necessary precaution measures shall be designed.

3.2 Plant surroundings

3.2.1 Potential contamination risk of the surroundings to food production shall be considered and appropriate measures shall be taken to reduce it to the minimum level.

3.2.2 The plant shall be arranged reasonably; each functional area shall be obviously divided with appropriate separation or partition measures to prevent cross contamination.

3.2.3 For the road in the plant, concrete, tar or other hard materials shall be paved; necessary measures shall be taken for vacant land, for example, cement, floor tile or lawn shall be paved to maintain clean surrounding and prevent raising dust and accumulating water under normal weather.

3.2.4 Plant greening shall be kept a proper distance from the production workshop, and vegetation shall be maintained periodically to prevent insect pest from breeding.

3.2.5 The plant shall be provided with proper drainage system.

3.2.6 Living area such as dormitory, canteen or recreation facilities of employees shall be kept a proper distance or partitioned from the production areas.

4. Plant and Workshop

4.1 Design and layout

4.1.1 Internal design and layout of plant and workshop shall meet the operation requirement of the food hygiene to avoid cross contamination during food production.

4.1.2 Design of plant and workshop shall be arranged reasonably according to production process to prevent and reduce the risk of contamination on products.

4.1.3 Operating areas in the plant and workshop shall be divided reasonably according to product

characteristics, production process, production characteristics and the requirements of cleanliness in production process and shall be effectively separated or partitioned. For example: operating areas are generally divided into clean operating area, quasi-clean operating area and general operating area; or clean operating area and general operating area, etc. General operating area shall be partitioned from other operating areas.

4.1.4 Inspection room arranged in the plant shall be partitioned from the production area.

4.1.5 Area and space of the plant shall be corresponding to the productivity to be convenient for equipment arrangement, cleaning and disinfection, material storage and personnel operation.

4.2 Internal structure and materials of the building

4.2.1 Internal structure

The building's internal structure shall be easy for maintenance, cleaning or disinfection and shall be constructed with appropriate durable materials.

4.2.2 Ceiling

4.2.1.1 Ceiling shall be constructed with nontoxic, odorless materials corresponding to the production demand and easy for observing cleaning condition; if coatings are directly coated on the inner-layer of the roof as ceiling, nontoxic, odorless and mold-proof coatings difficult for shedding and easy for cleaning shall be used.

4.2.1.2 Ceiling shall be easy for cleaning and disinfection, and difficult for condensed water to vertically drip in the structure to prevent insect pest and mold from breeding.

4.2.1.3 Pipelines of accessories for steam, water and electricity shall not be arranged above the exposed food; if it's unavoidable, device or measure to prevent dust from scattering and water drop from dripping shall be provided.

4.2.3 Wall

4.2.3.1 Wall surface and partition shall be constructed with nontoxic, odorless and anti-seepage materials; wall surface within the range of operation height shall be smooth, difficult for accumulating dirt and easy for cleaning; if coatings are used, they shall be nontoxic, odorless, mold-proof, difficult for shedding and easy for cleaning.

4.2.3.2 Wall, partition and ground junctions shall be reasonable in structure, easy for cleaning and effectively avoid the accumulation of dirt such as the arrangement of smooth and accessible surfaces.

4.2.4 Doors and windows

4.2.4.1 Doors and windows shall be closed tightly. Door surface shall be smooth, adsorption-proof, anti-seepage and easy for cleaning and disinfection. They shall be made of water proof, solid, and non-deformable materials.

4.2.4.2 Doors of clean operating area, quasi-cleaning operation area and other areas shall be able to timely be shut down.

4.2.4.3 Window glass shall be made of breakage-proof materials. If simple glass is used, necessary

measures shall be taken to prevent contamination on materials, packaging materials and food after glass breakage.

4.2.4.4 If windows are arranged with sills, their structure shall be able to avoid dust accumulation and be easy for cleaning. Windows able to open shall be equipped with insect pest prevention window screen easy for cleaning.

4.2.5 Ground

4.2.5.1 Ground shall be made of nontoxic, odorless, anti-seepage and corrosion-resistant materials. The ground structure shall be conducive to sewage discharge and cleaning.

4.2.5.2 Ground shall be flat, anti-skid, crack-free and easy for cleaning and disinfection and shall be provided with appropriate measures to prevent water accumulation.

5. Facilities and Equipment

5.1 Facilities

5.1.1 Water supply facilities

5.1.1.1 Water supply facilities shall ensure that the water quality, water pressure and water amount meet the production requirements.

5.1.1.2 The quality of food processing water shall meet the requirements of GB 5749. For food with special requirements of processing water quality, corresponding requirements shall be met. The quality of food production water such as indirect cooling water and boiler water shall meet the production requirements.

5.1.1.3 Food processing water and other water such as indirect cooling water, sewage or waste water without contacting with food shall be transported with completely separated pipelines to avoid cross contamination. Each pipeline system shall be marked explicitly for distinction.

5.1.1.4 Self-provided water source and water supply facilities shall meet the relevant requirements. Products used in water supply facilities involving hygienic security of drinking water shall also meet the relevant national requirements.

5.1.2 Drainage facilities

5.1.2.1 Drainage system shall be designed and constructed to ensure unblocked drainage and convenient cleaning and maintenance; it shall adapt to the need of food production and ensure that food, production and clean water be free from contamination.

5.1.2.2 The inlet of drainage system shall be installed with a device such as a floor drain with water seal to prevent solid waste from entering and discharged air from escaping.

5.1.2.3 Outlet of drainage system shall be provided with appropriate measures in order to reduce the risk of insect attack.

5.1.2.4 Indoor drainage shall flow from areas with high cleanliness to those with low cleanliness, and shall be designed to prevent backflow.

5.1.2.5 Sewage shall be disposed by proper ways before discharge to meet the relevant national

requirements about sewage discharge.

5.1.3 Cleaning and disinfection facilities

Sufficient specialized cleaning facilities for food, tools and instruments and equipment shall be provided; where necessary, appropriate disinfection facilities shall be provided. Measures shall be taken to avoid cross contamination brought by tools and instruments for cleaning and disinfection.

5.1.4 Waste storage facilities

Specialized facilities for storing waste which are reasonably designed, anti-seepage and easy for cleaning shall be provided; facilities and containers for storing waste in the workshop shall be marked clearly. Where necessary, facilities for storing waste temporarily shall be arranged in proper site and waste shall be stored in classes according to characteristics.

5.1.5 Personal hygienic facilities

5.1.5.1 Changing room shall be arranged at the entrance of production location or production workshop; where necessary, changing room may be arranged at the entrance of the specific operating area as needed. The changing room shall be designed to ensure that work clothes, personal clothes and other articles be kept apart.

5.1.5.2 Facilities for changing shoes (putting on shoe covers) or disinfection facilities for work shoes or boots shall be arranged as needed at the entrance and necessary place of the production workshop. If disinfection facilities for work shoes or boots are arranged, their specification and size shall meet the requirements of disinfection.

5.1.5.3 Restroom shall be arranged as needed; its structure, facilities and internal materials shall be easy to keep clean; facilities for washing hand shall be arranged at proper place in the rest room. The restroom shall not be directly connected with areas for food production, packaging or storage.

5.1.5.4 Facilities for washing and drying hand and disinfection shall be arranged at the entrance of clean operating area; if necessary, facilities for washing hand and (or) disinfection shall be added in the operating area; for the faucets matched with disinfection facilities, their switches shall be non- manual.

5.1.5.5 Quantity of the faucets for hand washing facilities shall be matched with that of food processing personnel of the same shift; where necessary, mixer of cold and hot water shall be arranged. Wash basins shall be made of smooth, water-proof and easy-to-clean materials and shall be designed and constructed to be easy for cleaning and disinfection. Simple and clear hand washing method shall be marked at visible position adjacent to hand washing facilities.

5.1.5.6 According to the cleanliness of food processing personnel, where necessary, facilities such as air shower and shower room may be arranged.

5.1.6 Ventilation facilities

5.1.6.1 Appropriate natural ventilation or artificial ventilation measures shall be taken; where necessary, natural ventilation or mechanical facilities shall be used to effectively control temperature and humidity of production environment. For ventilation facilities, air shall not flow from operating areas with low requirements on cleanliness to those with high requirements on cleanliness.

5.1.6.2 Air inlet position shall be arranged reasonably, and contamination source such as air inlet, air outlet

and device for storing outdoor garbage shall be kept an appropriate distance and angle. Air inlet and outlet shall be equipped with facilities such as mesh enclosure to prevent insect pest from intruding. Ventilation facilities shall be easy for cleaning, maintenance or replacement.

5.1.6.3 If filtration and purification treatment for air is needed in the production process, air filtration device shall be added and cleaned periodically.

5.1.6.4 According to production requirements, where necessary, de-dusting facilities shall be installed.

5.1.7 Lighting facilities

5.1.7.1 Sufficient natural lighting or artificial lighting shall be provided in the plant; luster and luminance shall meet production and operation requirements; light source shall make it possible that food takes on actual color.

5.1.7.2 If lighting facilities are needed to be installed above the exposed food and materials, safe lighting facilities shall be adopted or protection measures shall be taken.

5.1.8 Storage facilities

5.1.8.1 Storage facilities corresponding to quantity, storage requirements of products shall be provided.

5.1.8.2 Warehouse shall be made of nontoxic and solid materials; warehouse ground shall be flat and convenient for ventilation. Warehouse shall be designed to be easy for maintenance and cleaning to prevent insect pest from hiding and shall be provided with device for preventing insect pest from intruding.

5.1.8.3 Materials, semi-finished products, finished products and packaging materials shall be arranged with different storage sites or placed in different areas according to different properties and shall be marked explicitly to prevent cross contamination. Where necessary, warehouse shall be equipped with control facilities of temperature and humidity.

5.1.8.4 Storing articles shall be kept a proper distance from wall and ground in order to be conducive to ventilation and articles handling.

5.1.8.5 Detergent, disinfectant, pesticide, lubricant or fuel shall be packaged safely and marked explicitly and shall be kept apart from materials, semi-finished products, finished products and packaging materials.

5.1.9 Temperature control facilities

5.1.9.1 Appropriate heating, cooling and freezing facilities and facilities for monitoring temperature shall be equipped according to the characteristics of food production.

5.1.9.2 According to production requirements, facilities for controlling room temperature may be arranged.

5.2 Equipment

5.2.1 Production equipment

5.2.1.1 General requirements

Production equipment corresponding to productivity shall be provided and arranged in order according to process flow to avoid cross contamination.

5.2.1.2 Materials

5.2.1.2.1 Equipment and instruments contacting with materials, semi-finished products and finished products shall be made of nontoxic, odorless, corrosion-resistant materials difficult for shedding and shall be easy for cleaning and maintenance.

5.2.1.2.2 Surface of equipment and tools and instruments contacting with food shall be made of smooth, nonabsorbent materials easy for cleaning, curing and disinfection, and will not react with food, detergent and disinfectant under normal production and shall be kept in perfect condition.

5.2.1.3 Design

5.2.1.3.1 All production equipment shall make it possible in design and structure to avoid parts, metal chip, lubricating oil or other contamination factors being mixed into food and shall be easy for cleaning, disinfection, inspection and maintenance.

5.2.1.3.2 Equipment shall be fixed on the wall or floor without any gap or sufficient space shall remain between it and ground or wall during the installation to be convenient for cleaning and maintenance.

5.2.2 Monitoring equipment

The equipment used for monitoring, controlling and recording such as pressure gauge, thermometer or recorder and shall be calibrated and maintained periodically.

5.2.3 Equipment maintenance and repair

Equipment maintenance and repair system shall be established to strengthen the routine maintenance and curing of equipment; the equipment shall be inspected periodically and the result shall be recorded timely.

6. Hygiene Management

6.1 Hygiene management system

6.1.1 Hygiene management system for food processing personnel, food production and corresponding assessment standard shall be established; post responsibilities shall be determined to carry out post responsibility system.

6.1.2 Monitoring system for key control link significant to guarantee food safety shall be established according to the characteristics of food and hygienic requirements in the production and storage process to be implemented well and inspected periodically. If any problem is found, it shall be timely corrected.

6.1.3 Hygienic monitoring system for production environment, food processing personnel, equipment and facilities shall be established to determine the range, object and frequency of internal monitoring. The monitoring results shall be recorded and filed, and executive condition and effect shall be inspected periodically so that any problem can be rectified if it's found.

6.1.4 Cleaning and disinfection system and management system for cleaning and disinfection instruments shall be established. Equipment and tools and instruments before and after cleaning and disinfection shall be kept apart and safely kept to avoid cross-contamination.

6.2 Hygiene management of plant and facilities

6.2.1 Facilities in the plant shall be kept clean and repaired or renewed timely in case of any problem; in case of any damage of plant ground, roof, ceiling and wall, it shall be repaired timely.

6.2.2 Equipment and tools and instruments for production, packaging and storage, pipeline for production and contact surface of exposed food shall be cleaned and disinfected periodically.

6.3 Health management and hygienic requirement for food processing personnel

6.3.1 Health management for food processing personnel

6.3.1.1 Health management system for food processing personnel shall be established and carried out.

6.3.1.2 Personnel involved in food processing shall undergo an annual physical examination check and obtain a health certificate; they shall accept hygienic training before taking posts.

6.3.1.3 Food processing personnel who suffer from infectious disease of digestive tract such as dysentery, typhoid, viral hepatitis A and viral hepatitis E, diseases affecting food safety such as active pulmonary tuberculosis and suppurative or exudative dermatosis, or the personnel whose skin injury has not been healed shall be transferred to other posts without affecting food safety.

6.3.2 Hygiene requirements of food processing personnel

6.3.2.1 The personnel shall handle personal hygiene before entering food production site to avoid contaminating food.

6.3.2.2 The personnel shall wear clean work clothes when entering the operating area, wash hand and disinfect as needed; hair shall be hidden in work cap or restraint by hairnet.

6.3.2.3 The personnel shall not wear jewelry and watch and shall not make up, dye fingernails and spray perfume; they shall not carry or store personal articles irrelevant to food production.

6.3.2.4 After going to the rest room, contacting articles which may contaminate food or engaging in other activities irrelevant to food production, they shall wash hand and disinfect before engaging in activities. Insect pest control measures shall be prepared and carried out for periodical inspection. Effective measures such as yarn curtain, gauze, rat guard, fly prevention lamp or wind screen shall be taken in production workshop and warehouse to prevent rodent or insects from intruding. If trail of insects or rodent is found, its source shall be traced to eliminate hidden danger.

6.3.3 Visitors

Those who are not food processing personnel shall not enter food production site; if they enter the food production site under special circumstances, they shall abide by the same hygienic requirements with food processing personnel.

6.4 Insect pest control

6.4.1 The building shall be kept in perfect condition and tidy to prevent insect attack from intruding and breeding.

6.4.2 Insect pest control measures shall be prepared and carried out for periodical inspection. Effective measures such as yarn curtain, gauze, rat guard, fly prevention lamp or wind screen shall be taken in production workshop and warehouse to prevent rodent or insects from intruding. If trail of insects or rodent is

found, its source shall be traced to eliminate hidden danger.

6.4.3 Plan drawing for insect pest control shall be exactly drawn to mark the positions of mousetrap, glue board, fly-killing lamp, outdoor bait and killing device of biochemical pheromone.

6.4.4 Pest control shall be carried out periodically in the plant.

6.4.5 During the treatment by physical, chemical or biological agent, food safety and the proper food quality shall not be affected and food contact surface, equipment, tools and instruments and packaging material shall not be contaminated. Pest control shall be recorded correspondingly.

6.4.6 Before using various kinds of pesticides or other drugs, preventive measures shall be taken to avoid contamination on persons, food, equipment and tools; in case of contamination carelessly, contaminated equipment or tools shall be cleaned thoroughly in time to eliminate contamination.

6.5 Waste disposal

6.5.1 System for waste storage and elimination shall be prepared; for waste with special requirements, its disposal shall meet the relevant requirements. Waste shall be eliminated periodically; corruptible waste shall be eliminated as soon as possible; where necessary, waste shall be eliminated timely.

6.5.2 Waste location outside the workshop shall be isolated from food processing site to prevent contamination; smelly or harmful, toxic gas shall be prevented from escaping; insect pest shall be prevented from breeding.

6.6 Work clothes management

6.6.1 The personnel shall wear work clothes while entering the operating areas.

6.6.2 Specialized clothes such as coats, pants, shoes, caps and hairnet shall be equipped according to the food characteristics and the requirements of production process; where necessary, mask, apron, sleeve or glove may be equipped.

6.6.3 Cleaning system for work clothes shall be prepared, where necessary, work clothes shall be replaced timely; during the production, work clothes shall be kept clean and in perfect condition.

6.6.4 Work clothes shall be designed and made to adapt to the requirements of different operating areas to reduce the risk of cross contamination; position of work clothes pocket and connection fastening shall be reasonably selected to reduce the contamination risk caused by content or fastening dropping.

7. Food Material, Food Additives and Products Relevant to Food

7.1 General requirements

Purchasing, acceptance, transportation and storage management system for food material, food additives and products relevant to food shall be established to ensure that the food materials, food additives and products relevant to food meet the relevant national requirements. Any substance which may damage human health and life safety shall not be added to food.

7.2 Food material

7.2.1 License and qualified certificate of the Supplier for the purchased food materials shall be checked; food materials without qualified certificate shall be inspected according to food safety standard.

7.2.2 Food materials can't be used until they pass the acceptance. Food materials without passing the acceptance shall be kept apart from the qualified materials in designated areas and marked obviously and shall be returned and replaced timely.

7.2.3 Sensory inspection should be conducted before processing and where necessary, laboratory inspection shall be conducted; once the item indexes involving food safety are found to be abnormal, the food materials shall not be used and only the verified applicable ones shall be used.

7.2.4 During transportation and storage, the food materials shall be kept away from direct sunlight and shall be equipped with rainproof and dustproof facilities; according to the characteristics and hygiene requirements of food materials, they shall also be provided with facilities for insulation, cold storage and fresh keeping.

7.2.5 Transportation tools and vessels of food materials shall be kept clean and be maintained in good condition and be disinfected where necessary. The food materials shall not be shipped together with toxic and harmful substance to avoid contamination on food materials.

7.2.6 For warehouse of food materials, management system shall be established and it shall be managed by specific personnel who are responsible for periodically inspecting the quality and hygienic condition and timely cleaning bad food materials or those exceeding quality guarantee period. The distribution order of warehouse shall comply with the principle of "first in first out"; where necessary, it shall be determined according to the characteristic of different food materials.

7.3 Food additives

7.3.1 License of the Supplier and qualified certificate of products shall be inspected where food additives are purchased. The food additives can't be used until they pass the acceptance.

7.3.2 The transportation tools and containers of food additives shall be kept clean and be maintained in good condition and shall be provided with necessary protection to avoid contamination on the food additives.

7.3.3 Storage of food additives shall be managed by specific personnel who are responsible for periodically inspecting the quality and hygienic condition and timely cleaning the bad food materials or those exceeding quality guarantee period. The distribution order of warehouse shall comply with the principle of "first in first out"; where necessary, it shall be determined according to the characteristic of food additives.

7.4 Products relevant to food

7.4.1 Products relevant to food such as purchased food packaging materials, containers, detergent and disinfectant shall be inspected for qualified certificate; those which are carried out with license management shall also be inspected for the license of the Supplier and those such as food packaging materials can't be used until they pass the acceptance.

7.4.2 The transportation means and vessels of products relevant to food shall be kept clean and be maintained in good condition and shall be provided with necessary protection to avoid contamination on food materials and-cross contamination.

7.4.3 Storage of relevant products relevant to food shall be managed by specific personnel who are

responsible for periodically inspecting the quality and hygienic condition and timely cleaning the bad food materials or those exceeding quality guarantee period. The distribution order of warehouse shall comply with the principle of "first in first out".

7.5 Other

For packaging or containers of food materials, food additives and packaging materials directly contacting food, their materials shall be stable, nontoxic, harmless, and difficult to be contaminated and meet hygienic requirements.

Food materials, food additives and food packaging materials shall be provided with a certain buffer or cleaning measures for external packaging to reduce the contamination risk.

8. Food Safety Control in Production Process

8.1 Contamination risk control of product

8.1.1 Hazard analysis method shall be used to define the key link of food safety during production process and control measures for the key link of food safety shall be taken. In the area of the key link, relevant documents such as list of ingredients (feeding) and post specifications shall be prepared to implement control measures.

8.1.2 Hazard Analysis and Critical Control Point system is encouraged to be adopted for the food safety control during production process.

8.2 Control of biological contamination

8.2.1 Cleaning and disinfection

8.2.1.1 The effective cleaning and disinfection system shall be developed for production equipment and environment to reduce the risk of microbial contamination according to the characteristics of material, product and process.

8.2.1.2 Cleaning and disinfection system shall include: cleaning and disinfection area and name of equipment or instruments; responsibilities of cleaning and disinfection work; detergent and disinfectant; cleaning and disinfection method and frequency; verification of cleaning and disinfection effect and treatment for those failing to meet the requirements; cleaning and disinfection work and monitoring record.

8.2.1.3 The cleaning and disinfection system shall be guaranteed to be implemented and recorded faithfully; the disinfection effect shall be timely verified and it shall be corrected timely in case of any problem.

8.2.2 Microbial monitoring of food processing

8.2.1.4 The key control link is determined according to the product characteristics to carry out microbial monitoring; where necessary, the microbial monitoring procedure of food processing shall be established, including microbial monitoring of production environment and process product.

8.2.1.5 The microbial monitoring procedure of food processing shall include: microbial monitoring indexes, sampling points, monitoring frequency, sampling and inspection method, evaluation principles and rectification measures. The specific items may be developed by reference to the requirements of Appendix A in combination with production process and product characteristics.

8.2.1.6 The microbial monitoring shall include pathogenic bacteria monitoring and indicator bacteria monitoring, and the microbial monitoring result of food processing shall be able to reflect the control level of microbial contamination during food processing.

8.3 Control of chemical contamination

8.3.1 The management system to avoid chemical contamination shall be established; the possible contamination source and contamination way shall be analyzed and the proper control plan and control procedure shall be developed.

8.3.2 Use system of food additives and processing aids for food industry shall be established and the food additives shall be used according to the requirements of GB 2760.

8.3.3 Any non-edible chemical composition except food additives and other substances which may hazard human health shall not be added during food processing.

8.3.4 On the production equipment, if the movable components which may directly or indirectly contact food need lubrication, the edible oil or other oil meeting requirements of food safety shall be adopted.

8.3.5 The use system of chemicals such as detergent and disinfectant is established. Except for the cleaning and disinfection requirement and process demand, the chemicals which may contaminate food shall not be used and stored in the production site.

8.3.6 All food additives, detergents and disinfectants shall be preserved in proper container and shall be stored with obvious mark and in classes; during the receiving, they shall be exactly measured and recorded.

8.3.7 Hazardous substances resulting from food production must be monitored and effective measures must be encouraged and taken to reduce risk.

8.4 Control of physical contamination

8.4.1 The management system to avoid contamination of foreign matters shall be established; the possible contamination source and contamination way shall be analyzed and the corresponding control plan and control procedure shall be developed.

8.4.2 The measures such as equipment maintenance, hygiene management, site management, outsider management and processing supervision shall be taken to reduce the contamination risk of foreign matters such as glass, metal and plastic cement in maximum extent.

8.4.3 Effective measures such as arrangement of screen mesh, collector, magnet and metal checker shall be taken to reduce the risk of metal or other foreign matters to contaminate food.

8.4.4 During site repair, maintenance and construction, the proper measures shall be taken to avoid foreign matters, unpleasant smell and chips to contaminate food.

8.5 Packaging

8.5.1 The food packaging shall be able to protect the food safety and quality in maximum extent under normal storage, transportation and marketing (wholesale and retail) conditions.

8.5.2 Identification shall be checked to avoid misuse where the packaging material is used; and the use condition of packaging material shall be recorded truthfully.

9. Inspection

9.1 The inspection shall be carried out for material and product through self-inspection or by the consignable food inspection institution with corresponding qualification and the recording system for delivery inspection of food is established.

9.2 For self-inspection, the corresponding inspection room and inspection capability to inspection items shall be provided with; the inspection is carried out by the inspection personnel with corresponding qualification according to required inspection method; the inspection instruments and equipment shall be inspected periodically.

9.3 The inspection room shall be provided with sound management system to properly preserve the original record and inspection report of each inspection. Products sampling system shall be established to timely keep sample.

9.4 Comprehensive consideration shall be taken for factors such as product characteristics, process characteristics, material control condition to reasonably determine inspection items and frequency so as to effectively verify control measures during production process. The inspection frequency of net content, sensory requirements and other inspection items easy to change due to effect of production process shall be greater than that of other inspection items.

9.5 For the same variety of product with different packaging, inspection items free from effect of packaging specification and packaging type may be inspected together.

10. Storage and Transportation of Food

10.1 Proper storage and transportation conditions are selected according to requirements of food characteristics and hygiene; where necessary, the facilities shall be equipped for insulation, cold storage and fresh keeping. The food shall not be stored and transported together with toxic, harmful or smelly articles.

10.2 Suitable storage system shall be established and carried out and in case of any abnormality, it shall be timely treated.

10.3 The containers, tools and instruments and equipment to store, transport and load and unload the food shall be safe, harmless and clean to reduce the risk of food contamination.

10.4 During the storage and transportation, the direct sunlight, rain, notable temperature and humidity change and violent impact shall be avoided to prevent the adverse effect on food.

11. Product Recall Management

11.1 The product recall system shall be developed according to the relevant national regulations.

11.2 Where the produced food is unconformable with the food safety standard or other inedible conditions are found, the production shall be stopped immediately and the food already sold in market shall be recalled; the relevant production operators and consumers shall be notified and the recall and notification condition shall be recorded.

11.3 The recalled food shall be safely disposed or be destroyed to avoid them flowing into the market again. For food that is recalled due erroneous labeling, identification, or directions for use that is not in conformity with food safety standards, corrective measures shall be taken to guarantee the safety of the product, and explain the situation to consumers once the product is re-launched for sale.

11.4 Production batch shall be reasonably divided and recorded and identification shall be carried out such as product batch No. to be convenient for product traceability.

12. Training

12.1 Training system for relevant post of food production shall be established and the corresponding training about food safety knowledge shall be carried out for food processing personnel and practitioners of relevant post.

12.2 The awareness and responsibility of the practitioner to comply with relevant laws, regulations and standards of food safety and implement management system of food safety shall be promoted and the corresponding knowledge level shall be improved through the training.

12.3 The annual training plan of food safety shall be developed and implemented according to the actual demand of different posts of food production and the assessment is carried out; the training record is made.

12.4 Where the relevant laws, regulations and standards of food safety is updated, the training shall be timely developed.

12.5 The training plan shall be examined and revised periodically and the training effect is evaluated; and the routine inspection is carried out to ensure the effective implementation of training plan.

13. Management System and Personnel

13.1 The professional technical personnel and management personnel of food safety shall be allocated and the management system to guarantee food safety shall be established.

13.2 The management system of food safety shall be corresponding to the production scale, process level and variety characteristics of food and shall be continuously improved according to actual production and implementation experience.

13.3 The management personnel shall know about the basic principles and operation specifications of food safety and shall be able to judge the potential risks and take suitable preventive and corrective measures to ensure the effective management.

14. Record and Document Management

14.1 Record management

14.1.1 The recording system shall be established to record links of food production such as purchasing, processing, storage, inspection and marketing (sales) in detail. The record contents shall be complete and true to ensure that all links from material purchasing to production, to marketing of the product may be traced effectively.

14.1.1.1 The contents such as name, specification, quantity, supplier's name and contact, and purchase date of products relevant to food such as food materials, food additives and food packaging materials shall be recorded truthfully.

14.1.1.2 The contents such as food processing (including process parameter and environmental monitoring), storage condition of food and inspection batch No., inspection date, inspection personnel, inspection method and inspection result of the product shall be recorded truthfully.

14.1.1.3 The contents such as name, specification, quantity, production date, production batch No., Purchaser's name and contact, quality certificate and marketing (sales) date of delivery product shall be recorded truthfully.

14.1.1.4 The contents such as name, batch, specification, quantity, recall reason and subsequent rectification program of recalled food shall be recorded truthfully.

14.1.2 The purchasing inspection record of products relevant to food such as food materials, food additives and food packaging materials and delivery inspection record of food shall be rechecked and signed by the record personnel and examiner; the record contents shall be complete. The preservation period shall not be less than 2 years.

14.1.3 The customer complaint handling mechanism shall be established. As for the written or verbal advice and complaint proposed by customers, the related management departments of the enterprise shall make records, find out the reasons and handle them carefully.

14.2 The management system of document shall be established to effectively manage documents so as to ensure that documents at each relevant location are valid.

14.3 The advanced technology and means (such as information system of electronic computer) are encouraged to be adopted to carry out record and document management.

Appendix A

Microbial Monitoring Procedure Guide of Food Processing

Note: this Appendix gives key points which shall be considered where the environmental microbial monitoring procedure in food processing is developed, and they may be referred to in actual production according to factors such as product characteristics and technical level of production process.

A.1 The microbial monitoring during food processing is important means to ensure the food safety and the tool to verify or evaluate effectiveness of target microorganism control procedure and to ensure the continuous improvement of whole food quality and safety system.

A.2 This Appendix proposes the key points which shall be considered where the microbial monitoring procedure of food processing is developed.

A.3 The microbial monitoring of food processing mainly includes the environmental microbial monitoring and microbial monitoring of process product. The environmental microbial monitoring is mainly used to judge the hygiene control condition of processing and find out the potential contamination source. Generally, the environmental monitoring objects include food contact surface, adjacent contact surface to food or food contact surface and environmental air. The microbial monitoring of process product is mainly used to evaluate the hygiene control capacity of processing and hygienic condition of product.

A.4 The microbial monitoring of food processing covers microbiology evaluation and evaluation of cleaning and disinfection effect and microorganism control effect of each link during processing. During development, the following contents shall be considered:

- a) The microbial monitoring of processing shall include the microbial monitoring indexes, sampling points, monitoring frequency, sampling and inspection method, evaluation principles and treatment for non-conformance condition.
- b) The microbial monitoring indexes of processing shall take the indicator microorganism (such as aerobic bacteria count, coliform bacteria, yeast or other indicator bacteria) which is able to evaluate the hygienic condition of processing environment and process control capacity as priority. Where necessary, the pathogenic bacteria may also be adopted as the monitor index.
- c) The microbial monitoring sampling points of processing: sampling points of environmental monitoring shall be places which are contaminated due to the possible existence or entrance of microorganism. The sampling points may be determined according to the relevant literature information, experience or accumulated historical data. The sampling points of process product monitoring plan shall cover all process products whose microorganism level may change and may affect the product safety and (or) food quality in the whole processing link, for example, the one behind the key control point controlled by microorganism. The specific contents may refer to examples detailed in Table A.1.
- d) The microbial monitoring frequency of processing: monitoring frequency shall be developed based on the possible risk of contamination. The reasonable monitoring frequency may be determined according to the relevant literature information, relevant experience and professional knowledge or accumulated historical data. The specific contents may refer to examples detailed in Table A.1. The microbial monitoring of processing shall be dynamic, adjusted according to the data change and contamination risk of processing and periodically evaluated. For example, where the indicator microorganism monitoring result is on the high side, the pathogenic bacteria is found in final product, after the significant

maintenance construction activities are completed, or downtrend appears for hygienic condition, the sampling points and monitoring frequency are needed to be increased; where the monitoring result meets the requirements all the time, the sampling points or the monitoring frequency may be properly reduced.

e) The sampling and inspection method: generally, coating sampling is the primary of environmental monitoring and the direct sampling is adopted for process product monitoring. The selection of inspection method shall be based on the monitor index.

f) The evaluation principles: the judgment shall be carried out according to the certain monitor index limit and the limit may be determined based on the microorganism control effect and its influence on the product quality and food safety.

g) The treatment requirements for inconformity condition of microbial monitoring: the monitoring result of each monitoring point shall meet the monitor index limit and remain stable; where the slight inconformity appears, measures such as increasing sampling frequency may be adopted to strengthen monitoring; where the severe inconformity appears, correction shall be carried out immediately and the reason leading to problem shall be found out at the same time to determine whether the corresponding corrective measures are taken for microorganism control procedure.

Table A.1 Microbial Monitoring Example of Food Processing

Monitoring items		Suggested sampling points ^a	Suggested monitoring microorganism ^b	Suggested monitoring frequency ^c	Suggested monitor index limit
Environmental microbial monitoring	Food contact surface	Hands and work clothes of food processing personnel, surfaces of glove conveyors, tools and instruments and other equipment directly contacting food	Bacterial colony, coliform etc.	The verification of cleaning effect shall be carried out after the cleaning and disinfection and others may be carried out every week, every two weeks or every month	Determined in combination with actual situation of production
	Adjacent contact surface to food or food contact surface	External surface of equipment, support surface, control panel and contact surface of part car	Indicator microorganism for hygienic condition of bacteria colony and coliform; where necessary, the pathogenic bacteria is monitored	Every two weeks or every month	Determined in combination with actual situation of production
Monitoring items		Suggested sampling points ^a	Suggested monitoring microorganism ^b	Suggested monitoring frequency ^c	Suggested monitor index limit
	Environmental air of processing area	Position close to exposed product	Bacteria colony, yeast etc.	Every week, every two weeks or every month	Determined in combination with actual situation of production
Microbial monitoring of process product		Process product whose microorganism level may change and may affect the food safety and (or) food quality during processing link	Indicator microorganism for hygienic condition such as bacteria colony, coliform, yeast or other indicator bacteria	Every week (every two weeks or every month) for the product produced in the first time of shift beginning and subsequent continuous production process	Determined in combination with actual situation of production
^a Sampling points may be selected according to the food characteristics and actual situation of processing. ^b One or more hygiene indicator microorganism may be selected to implement monitoring according to the requirements. ^c Monitoring frequency may be determined according to the risk of specific sampling points.					

GB 15193.1-2014 Procedures for Toxicological Assessment of Food



National Standards of People's Republic of China

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National Food Safety Standard
Procedures for Toxicological Assessment of Food

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National Standard for Food Safety

Procedures for Toxicological Assessment of Food

1. Scope

This Standard specifies the procedure for toxicological assessment of food.

This Standard is applicable to the assessment of the safety of chemical, biological and physical factors that may cause harm to health concerned during food production, processing, storing, transport and selling process. Assessment objects include food and its raw material, food additives, new food raw materials, irradiated food, food related products (used for food packaging materials, containers, detergent, disinfectant; and the tools and equipment used for food production and marketing), and food pollutants.

2. Requirements for test substances

2.1 It shall provide the test substance's name, batch number, content, storage conditions, and sources of raw materials, production processes, quality specifications, character, human recommended (possible) intake, and other relevant information.

2.2 For single-ingredient chemical substance, the physical, chemical property (including chemical structure, purity, stability, etc.) of test substance (including impurities when necessary) shall be provided. For mixed substances (including formulated products), the composition of test substance shall be provided; when necessary, physical property, chemical property (including chemical name, structure, purity, stability, solubility, etc.) of all compositions of test substances and relevant data shall be provided.

2.3 If the test substance is formulated product, it shall be the standardized product; its composition ingredients, proportion and purity shall be the same as the actual application. If the test substance is an enzyme preparation, it shall, before other compound ingredients are added, use the product as the test substance.

3. Contents of toxicological assessment for food safety

3.1 Acute oral toxicity test

3.2 Genetic toxicity test

3.2.1 Genetic toxicity test contents. Bacterial reverse mutation test, mammalian erythrocyte micronucleus test, mammalian bone marrow chromosome aberration test, mouse spermatogonia or spermatocytes chromosome aberration test, in-vitro mammalian cell HGPRT gene mutation test, in-vitro mammalian cell TK gene mutation test, in-vitro carcinogenicity studies (or chronic toxicity and carcinogenicity merge test).

3.2.2 Genetic toxicity test combinations. Generally speaking, these combinations shall comply with the principle of integrating prokaryotic cells and eukaryotic cells together and integrating in-vitro tests and in-vivo tests together. The following combinations are recommended in accordance with properties of the test material and the test purpose:

Combination 1: Bacterial Reverse Mutation Assay: Mammalian Erythrocyte Micronucleus Test or Mammalian Bone Marrow Cell Chromosome Aberration Test, Mouse Spermatogonial / Spermatocyte Chromosome Aberration Test or Rodent Dominant Lethal Test.

Combination 2: Bacterial Reverse Mutation Assay: Mammalian Erythrocyte Micronucleus Test or Mammalian

Bone Marrow Cell Chromosome Aberration Test, In Vitro Mammalian Cells Chromosome Aberration Test or In Vitro Mammalian Cell TK Gene Mutation Test.

Other Optional Genotoxicity Tests: Sex-linked Recessive Lethal Test in *Drosophila melanogaster*, In-vitro Mammalian Cells Repair of DNA Damage (Unscheduled DNA Synthesis (UDS)) Test, In-vitro Mammalian Cells HGPRT Gene Mutation Test.

3.3 28-day Oral Toxicity Test

3.4 90-day Oral Toxicity Test

3.5 Teratogenicity Test

3.6 Reproductive Toxicity Test and Reproductive & Developmental Toxicity.

3.7 Toxic kinetic Test

3.8 Chronic Toxicity Test

3.9 Carcinogenesis Test

3.10 Chronic Toxicity and Carcinogenicity Test

4. Principle for Selection of Toxicity Tests

4.1 For substances initiated in China, especially those substances whose chemical structures indicate potential chronic toxicity, genotoxicity or carcinogenicity, or those substances of large output, wide range of application and large human intake, systematical toxicity tests will be required, including acute oral toxicity test, genotoxicity test, 90-day oral toxicity test, malformation test, reproductive & developmental toxicity test, toxicokinetic test, chronic toxicity test and carcinogenesis test (or chronic toxicity and carcinogenicity test).

4.2 For derivatives or analogs of which the chemical structure is basically same as that of known substances (refer to those that have passed safety evaluation and are allowed for use), or the substances that have safe-use history in some countries and regions, then it may firstly conduct the acute oral toxicity studies, genetic toxicity test, 90-day oral toxicity test, and teratogenicity test. According to the test results, determine whether it needs to conduct the toxicokinetic test, reproductive toxicity test, chronic toxicity test and carcinogenicity test, etc.

4.3 For substances that are known or have use history in several countries, at the same time, the application organization has the data to prove that the quality specifications of the declared test substance are consistent with foreign products, then it may firstly conduct the acute oral toxicity test, genetic toxicity test, and 28-day oral toxicity test. According to the test results, determine whether it needs to further conduct toxicity test.

4.4 Selection of the safety toxicology assessment test for food additives, new resources of food and ingredients, food related products, pesticide residue, veterinary drug residue

4.4.1 Food additives

4.4.1.1 Flavoring

4.4.1.1.1 All the flavorings that have been approved for use or have been formulated for daily acceptable

intake by World Health Organization (WHO), and allowed by two or more organizations of WHO, FEMA, COE and IOFI, then it generally is not required to conduct test.

4.4.1.1.2 If the data is incomplete or is only approved by one of the international organizations, then acute toxicity test and one of genetic toxicity test combination shall be conducted first; decide whether further test is needed after preliminary assessment.

4.4.1.1.3 If no data can be referred to and it is not yet allowed to use by international organization, then conduct the acute toxicity test first, genetic toxicity test and 28-day oral toxicity test. Decide whether further test is needed after preliminary assessment.

4.4.1.1.4 For single high-purity natural flavoring that is extracted from edible part of animal or plant, if the chemical structure and relevant data do not show un-safety, then toxicity test is generally not required.

4.4.1.2 Enzyme preparations

4.4.1.2.1 For enzyme preparations that have long history of safe consumption and that are produced from edible parts of animals or plants, if the World Health Organization has announced the acceptable daily intake (ADI) or ADI is not required to be specified or several countries have approved the use, on the basis of providing relevant proving.

4.4.1.2.2 For enzyme preparations obtained from other sources: if relatively complete toxicology data is available, and WHO has defined the acceptable daily intake or it is not necessary to define the acceptable daily intake, or the preparation has been approved for use in several countries, it is required to carry out the acute oral toxicity test and the genotoxicity test in the case the preparation's quality complies with corresponding international quality standards, and it is required to carry out the acute oral toxicity test, the genotoxicity test, and the 28-day oral toxicity test in the case the preparation's quality does not comply with corresponding international quality standards, and the question whether to execute other toxicity tests depends on the results of the tests required.

4.4.1.2.3 For enzyme preparations obtained from other sources: if the preparation is a new species, it is required to carry out the acute oral toxicity test, the genotoxicity test, the 90-day oral toxicity test and the malformation test first for preliminary assessment, and whether to carry out further tests depends on the results of the preliminary assessment; if the preparation has been approved for use in one country and the WHO has not defined the acceptable daily intake or no complete data available, it is required to carry out the acute oral toxicity test, the genotoxicity test, the 28-day oral toxicity test, and whether to carry out further tests depends on the results of the tests required.

4.4.1.2.4 Testing on enzyme preparations produced by transgene should comply with applicable regulations on management of transgenesis of the state.

4.4.1.3 Other Food Additives

4.4.1.3.1 If relatively complete toxicology data is available, and WHO has defined the acceptable daily intake or it is not necessary to define their acceptable daily intake, or the additive has been approved for use in several countries, it is required to carry out the acute oral toxicity test and the genotoxicity test in the case the additives' quality complies with corresponding international quality standards, and it is required to carry out the acute oral toxicity test, the genotoxicity test, and the 28-day oral toxicity test in the case the additives' quality does not comply with corresponding international quality standards, and the question whether to execute other toxicity tests depends on the results of the tests required.

4.4.1.3.2 If the additives have been approved for use in one country and the WHO has not defined the acceptable daily intake or no complete data available, it is required to carry out the acute oral toxicity test, the genotoxicity test, the 28-day oral toxicity test, and the teratogenicity test, and whether to carry out further tests depends on the results of the tests required.

4.4.1.3.3 For single components prepared from animals, plants, and microorganisms, and high purity food additives, if the component/additive is a new species, it is required to carry out the acute oral toxicity test, the genotoxicity test, the 90-day oral toxicity test and the teratogenicity test first for preliminary assessment, and whether to carry out further tests depends on the results of the preliminary assessment; if the component/additive has been approved for use by an international organization or in one country it is required to carry out the acute oral toxicity test, the genotoxicity test and the 28-day oral toxicity test, and whether to carry out further tests depends on the results of the tests required

4.4.2 New food raw materials

It shall be evaluated in accordance with "New food raw material declaration and acceptance provisions" (State-Health-Food-Announcement [2013] 23).

4.4.3 Food related products

It shall be evaluated in accordance with "Food related product new varieties declaration and acceptance provisions" (Health-Supervision-Announcement [2011] 49).

4.4.4 Pesticide residue

It shall be evaluated in accordance with GB15670.

4.4.5 Veterinary drug residue

It shall be evaluated in accordance with "Veterinary drug before-clinical toxicology evaluation test guidelines" (The People's Republic of China, Ministry of Agriculture Bulletin No. 1247).

5. Objective of toxicological assessment of food and result judgment

5.1 Objectives of toxicological test

5.1.1 Acute toxicity test

Understand the degree of toxicity, property and possible target organ of test substance; provide basis for selection of dose and toxicity observation index for further toxicity test; classify the acute toxicity dosage according to LD50.

5.1.2 Genetic toxicity test

Screen the genetic toxicity of test substance and whether it has potential carcinogenesis and cell mutagenicity.

5.1.3 28-day oral toxicity test

On the basis of the acute toxicity test, understand more about the nature of the test substance's toxicity, the dose-response relationship and possible target organs, so as to obtain the 28-day oral not-observed adverse effect dose; preliminarily evaluate the safety of the test substance; and provide the basis for

selecting longer-term toxicity and chronic toxicity test dose, observation indicators, toxicity endpoint for next step.

5.1.4 90-day oral toxicity test

Alternative tests (at least one is in-vivo test). If the additional 2 alternative tests are negative, it may proceed to the next toxicity test; if 1 item is positive, it shall give up that the test substance can be used in food.

5.1.5 Teratogenicity Test

The test is to determine whether a test substance has teratogenicity and development toxicity, and define the test substance's NOAEL(No Observed Adverse Effect Level) for teratogenicity and development toxicity.

5.1.6 Reproductive Toxicity Test and Reproductive & Developmental Toxicity

The test is to understand how a test substance toxic affects the laboratory animal breeding and its offspring growth, in terms of gonadal function, estrous cycle, mating behaviour, pregnancy, childbirth, breastfeeding and weaning, and its offspring reproductive and development. After obtaining the unobserved harmful effects dose levels by the test substances, it provides the scientific evidence of developing the preliminary population safe lifting limits standard.

5.1.7 Toxicokinetics Test

The test is to explain how a test substance is absorbed and distributed in vivo and how soon will be excreted and other related information, to provide basis for selection of suitable germine of experimental animals used in the chronic toxicity test, and to reveal the formation of metabolites.

5.1.8 Chronic Toxicity Test and Carcinogenesis Test

The test is to explain a test substance's toxicity and carcinogenicity of long-term exposure, to define the test substance's NOAEL, and to provide basis for the final assessment for application in foods and the determination of HBGV (health-based guidance values).

5.2 Analysis on Results of Various Toxicology Tests

5.2.1 Acute Toxicity Test

Generally speaking, if a test substance's LD50 (Lethal Dose, 50%) is less than 100 times of the human recommended (possible) intake, the substance should not be used in food, and no further toxicology tests is necessary.

5.2.2 Genotoxicity Tests

5.2.2.1 It is very likely that the test substance has genotoxicity and carcinogenicity if the results of two or more tests in the genotoxicity test combination are positivethe, and the substance should not be used in food if so.

5.2.2.2 If the result of one test in the genotoxicity test combination is positivethe, two more optional tests (one in vivo test at least) shall be selected and carried out. If the results of the tow optional tests are both positive, further toxicity tests shall be carried out; if the result of one optional test is positive, the test substance should not be used in food.

5.2.2.3 If the results of the three tests are positive, further toxicity tests shall be carried out.

5.2.3 28-day Oral Toxicity Test

For test substances only the acute toxicity test, genotoxicity test and 28-day oral toxicity test are required to be carried out, a preliminary assessment could be obtained combining with results of other tests, if no evident toxicity is observed in the acute toxicity test, genotoxicity test and 28-day oral toxicity test; and further toxicity tests should be carried out if evident toxicity is observed, especially a dose-response relationship exists.

5.2.4 90-day oral toxicity test

According to the not-observed adverse effect dose obtained by the test to conduct assessment, the principle is:

- a) If not-observed adverse effect dose is less than or equal to 100 times of recommended (possible) human-intake, it indicates stronger toxicity; it shall give up that the test substance can be used in food;
- b) If not-observed adverse effect dose is more than 100 times but less than 300 times, it shall conduct chronic toxicity test;
- c) If not-observed adverse effect dose is more than or equal to 300 times, then chronic toxicity test is not required; it may proceed to safety assessment.

5.2.5 Teratogenicity Test

According to the test results, evaluate if the test substance is the teratogenic substance to laboratory animals. If teratogenic test result is positive, then reproductive toxicity and reproductive-developmental toxicity test shall not be continued. For other developmental toxicities that are observed in teratogenicity test, it shall combine with 28-day and (or) 90-day oral toxicity test results to conduct the assessment.

5.2.6 Reproductive toxicity test and reproductive-developmental toxicity test

According to the not-observed adverse effect dose obtained by the test to conduct assessment, the principle is:

- a) If the NOAEL is lower than or equal to 100 times of the human recommended (possible) intake, the test substance should not be used in foods.
- b) If the NOAEL is higher than 100 times but lower than 300 times of the human recommended (possible) intake, the chronic toxicity test should be carried out.
- c) If the NOAEL is higher than 100 times or equal to 300 times of the human recommended (possible) intake, a safety assessment could be obtained without carrying out the chronic toxicity test.

5.2.7 Chronic Toxicity Test and Carcinogenesis Test

5.2.7.1 The principle for assessment according to the NOAEL obtained in the chronic toxicity test is as follows:

- a) If the NOAEL is lower than or equal to 50 times of the human recommended (possible) intake, the

test substance could have strong toxicity and should not be used in foods.

- b) If the NOAEL is higher than 50 times but lower than 100 times of the human recommended (possible) intake , the question whether the test substance could be used in foods depends on the safety assessment.
- c) If the NOAEL is higher than 100 times of the human recommended (possible) intake, the test substance allowed to be used in foods.

5.2.7.2 The principle to analyse the result of the carcinogenesis test according to the incidence, incubation period and multiplicity determined in the carcinogenesis test is as follows (If one or more of the following conditions are met , the result of the carcinogenesis test could be regarded as positive; if a dose-response relationship exists, it could be more certain about the positive result.) :

- a) Cancer only occurs among animals of the test group.
- b) Cancer occurs among animals both of the test group and the control group, but the incidence of the test group is higher.
- c) The multiplicity of cancer is evident in the test group, while no multiplicity exists or only seldom animals have gotten multiple cancer in the control group.
- d) There is no evident difference in the incidence between the test group and the control group, but cancer occurred earlier in the test group.

5.2.8 Others

If the highest dosage of the test substance used as feed additive(no more than 10% of the feed, in principle) or the dosage after concentration (liquid test substances)is lower than the required times of the human recommended (possible) intake specified according to the NOAEL, a safety assessment could be made combining results of other toxicity tests and the actual intake.

6. Factors to Consider During Food Safety Assessment

6.1 Test indicators' statistical significance, biological significance and toxicological significance

For the abnormal changes of some indicators in experiment, it shall, according to if there are statistical differences between experimental-group and control-group's indicators; if there is dose-response relationship; the horizontal comparison of similar indicators; and consistency of both sexes and of the historic control value range of that laboratory; etc., comprehensively consider if the indicator difference has biological significance. And further judge if there is toxicological significance. In addition, if some tumor is found in experimental-group but not in control-group, even it has no statistical significance with the control-group, it still needs to be paid attention to.

6.2 Test substance of larger recommended (possible) human-intake

It shall consider that, when the given test substance is too high, it may affect nutrient intake and bioavailability, so as to cause some toxicology performance, rather than that the toxicity is caused by the test substance.

6.3 Time-toxic effect relationship

When conducting analysis and assessment to toxic effects of experimental animals that are caused by the test substance, it shall consider that, at the same dose level, the toxic effects change along the time.

6.4 Special human-groups and vulnerable human-groups

For the foods eaten by pregnant women, nursing mothers or children, it shall specially pay attention to its embryo toxicity or reproductive-developmental toxicity, neurotoxicity and immunotoxicity etc.

6.5 Human-group information

Due to the species difference between human being and animal, while assessing the food safety, response data of human being after contacting test substances shall be collected as much as possible, such as occupational contact and accidental contact, etc. Under the condition of ensuring safety, human tasting test can be considered in accordance with relevant regulations. And volunteer testees' toxicokinetics or metabolism data is of important significance to deduct the animal test result to human being.

6.6 Animal toxicity test and in-vitro test data

The various animal toxicity tests and in-vitro test systems listed in this Standard are the most important data that can be obtained under the current management (regulations) toxicological assessment level; it is also the main basis for safety assessment. When test result is positive, and the result judgment is involved in whether the test substance could be applied in food, the repeatability of result and dose-response relationship shall be considered.

6.7 Uncertainty coefficient

It is the safety coefficient. When deducting the animal toxicity test result to human being, because there are biological differences among animal, human being, and human individual, uncertainty coefficient is usually 100; however, it may comprehensively consider the number of safety coefficient according to the test substance's raw material source, physiochemical property, degree of toxicity, metabolism characteristic, accumulation, contacted human-group scope, usage amount in food and possible intake for human being, application scope and function, and other factors.

6.8 Data of toxicokinetics test

Toxicokinetic test is an important aspect for toxicological assessment to chemical substances, because different chemical substances or dose amount often have significant impact to the differences of toxicokinetic or metabolism. In toxicity test, the animal species with the same metabolism method and mode as human being shall be, in principle, applied as much as possible for test. Studying the difference of test substance's absorption, distribution, excretion and biotransformation on animal and human being has important significance on deducting the animal test results to human being and reducing the uncertainty.

6.9 Comprehensive assessment

While conducting final assessment, the physiochemical property, structure, degree of toxicity, metabolism characteristic, accumulation, contacted human being scope, usage amount and usage scope in food, recommended (possible) intake for human being, and other factors of test substance shall be comprehensively considered; For the substances that have been applied in food for a relative long-time, they are of great significance to the epidemiology investigation for contact people; however, it is usually difficult to obtain the reliable data on dose-response relationship. For new test substances, only animal test

and other test study data can be relied on. However, even if there are complete and detailed animal test data and some human being contact epidemiology study data, it is still hard to make assessment that it could guarantee every person's safety due to the different species of human being and individuals. The so-called absolute safety actually does not exist. It shall comprehensively balance between that it may possibly cause harm to human-body health and it may provide benefits; on premise of food safety, the basis of safety assessment is not only the result of safety toxicology test, but also relates to the then-level of science, technological conditions, socio-economy, and cultural factors. Thus, along the time, the development of social economy, scientific and technological progress, it is necessary to re-assess the test substances that have previously passed the assessment.

GB 5009.3-2010 Determination of Moisture in Foods



National Standards of People's Republic of China

GB 5009.3-2010

**National Food Safety Standard
Determination of Moisture in Foods**

Issued on: 2010-03-26

Implemented on: 2010-06-01

Issued by National Health and Family Planning Commission

Foreword

This standard will replace GB/T5009.3-2003 *Determination of Moisture in Foods* and GB/T14769-1993 *Testing Methods for Moisture in Foods*.

Comparing this standard and GB/T5009.3-2003, key changes are as follows;

- Added the Karl Fischer method as the “fourth method”;
- Amended the range of temperature for the direct drying method;
- Clarified the units of measurement used in the first and second method calculation formula;
- Amended the scope of application for depressurized drying method.

This article will supersede earlier versions, including the following:

- GB/T5009.3-1985, GB/T5009.3-2003
- GB/T14769-1993.

National Standard for Food Safety

Determination of Moisture in Foods

1. Scope

This standard specifies the testing methods for moisture in foods.

The direct drying method in this standard is applicable for the determination of moisture in foods without or with minimal volatile substances under the 101°C~105°C temperature range and it is not applicable for samples with moisture level less than 0.5g/100g. Applicable food categories include grains and its products, aquatic products, soy products, dairy products, meat products, preserved (brine) vegetables, etc.

Depressurized drying method applies to the determination of moisture in foods that can be easily broken down, e.g. sugar, monosodium glutamate (MSG), but it is not applicable to testing of candies with supplementary ingredients added during production, e.g. milk candies, gummies as well as samples with moisture content lower than 0.5g/100g.

Distillation method applies to the determination of moisture in foods with relatively higher volatile substance content, e.g. fats, spices, but it is not applicable to samples with moisture content lower than 1g/100g.

Karl Fischer method applies to the determination of moisture in foods; specifically it is applicable to foods with samples with moisture content higher than 1.0×10^{-3} g/100g, while Karl Fischer coulometry applies to samples with moisture content higher than 1.0×10^{-5} g/100g.

Method I Direct Drying Method

2. Principle

Leveraging on the physical properties of moisture in foods, determine the sample's loss of mass (includes hygroscopic water, partially crystalized water and substances that may evaporate under certain conditions) during the drying process, adopting the evaporation method at temperature 101°C~105°C, 101.3kPa (1 Atm). Calculate the moisture content by comparing the sample mass values before and after the drying process.

3. Reagents & Materials

All reagents used in this method are analytically pure (AR) unless otherwise stated.

3.1 Hydrochloric Acid: Superior Purity.

3.2 Sodium Hydroxide (NaOH): Superior Purity.

3.3 Hydrochloric Acid Solution (6mol/L): Measure and extract 50mL hydrochloric acid, then dilute by adding water till 100mL volume.

3.4 Sodium Hydroxide Solution (6mol/L): Weigh and extract 24g sodium hydroxide, add water to fix and dilute till 100mL volume.

3.5 Marine Sand: Take marine sand or river sand washed off of mud/soil with water and boil the sand in hydrochloric acid (prepared as in 3.3) for 0.5h. Thereafter, neutralize the acidity with water, then boil again

with sodium hydroxide solution (prepared as in 3.4) for another 0.5h. Neutralize with water, dry at 105oC and set aside for use later.

4. Apparatus & Equipment

4.1 Flat-shaped Aluminum or Glass Weighing Bottle.

4.2 Electric Heating Thermostatic Drying Oven.

4.3 Dryer: With effective desiccant.

4.4 Weighing balance: Sensitivity 0.1mg.

5. Analysis Procedures

5.1 Solid Sample: Take a clean aluminum or glass flat-shaped weighing bottle and place it in a 101oC~105oC drying oven for 1.0h with its cap slanted around the side of the bottle. Thereafter, place the bottle in a dryer to cool for 0.5h, weigh it and repeat the dry/cool and weigh process until the difference between 2 consecutive measurements do not exceed 2mg; set this as the equilibrium mass. Weigh and extract 2g~10g (precision 0.0001g) of samples that have been quickly grinded into fine particles of size less than 2mm (samples that cannot be properly grinded should be cut into finer pieces as much as possible) and evenly mixed them, then place samples into the abovementioned bottle. Samples with thickness not exceeding 5mm are considered loose samples and further processing is not required. However, for samples with thickness more than 10mm, cover these with cap, make record of the precise mass of the bottle with the samples and then place it into 101oC~105oC drying oven for 2h~4h with its cap slanted around the side of the bottle. Once drying is completed, cover with cap, remove from oven, then place it into the dryer to cool for 0.5h and weigh again. Thereafter, place the bottle into 101oC~105oC drying oven for about 1h, remove, place it into the dryer to cool for 0.5h and weigh. Repeat the oven, dryer and weigh operation until the difference between 2 consecutive measurements do not exceed 2mg; set this as the equilibrium mass.

Note: Use the final weighing value recorded for the final calculation with the 2 equilibrium mass values.

5.2 Semi-solid or Liquid Sample: Take a clean weighing bottle, add 10g marine sand and a small glass rod within, then place bottle into 101oC~105oC drying oven for 1.0h, remove, place it into the dryer to cool for 0.5h and weigh; repeat operations till equilibrium mass is attained. Thereafter, weigh and extract 5g~10g samples (precision 0.0001g) and place them into an evaporating dish. Use a small glass rod to stir and mix evenly and then place the dish on a boiling water bath so as to evaporate all its water content, stirring and wiping the bottom of dish of water droplets whenever necessary. Place dish in 101oC~105oC drying oven for 4h, cap and remove, then transfer into a dryer to cool for 0.5h and weigh thereafter. Operations after this will be similar to instructions in 5.1 from "Thereafter, place the bottle into 101oC~105oC drying oven for about 1h....." onwards.

6. Presentation of Analysis Results

Moisture content in samples can be calculated based on formula (1).

$$X = \frac{m_1 - m_2}{m_1 - m_3} \times 100 \dots\dots\dots (1)$$

In formula:

X – Moisture content in samples, units in g/100g;

m1 – Mass of weighing bottle (with marine sand and glass rod) and samples, units in g;

m2 – Mass of weighing bottle (with marine sand and glass rod) and samples after the drying process, units in g;

m3 – Mass of weighing bottle, units in g.

Calculation results should retain 3 significant figures when moisture content $\geq 1\text{g}/100\text{g}$; calculation results should retain 2 significant figures when moisture content $< 1\text{g}/100\text{g}$.

7. Precision

Absolute discrepancies between 2 independent test results conducted under iterative conditions should not exceed 5% of their arithmetic mean value.

Method II Depressurized Drying Method

8. Principle

Leveraging on the physical properties of moisture in foods, determine the sample's loss of mass during the drying process, adopting the depressurized drying method, heating up to temperature $60\text{oC}\pm 5\text{oC}$ after reaching $40\text{ kPa}\sim 53\text{ kPa}$. Calculate the moisture content by comparing the sample mass values before and after the drying process.

9. Apparatus and Equipment

9.1 Vacuum Drying Oven.

9.2 Flat-shaped Aluminum or Glass Weighing Bottle.

9.3 Dryer: With effective desiccant.

9.4 Weighing balance: Sensitivity 0.1mg .

10. Analysis Procedures

10.1 Preparation of Samples: Weigh and extract powdered and crystal form samples directly for tests; larger hard candy samples should be crushed into finer chunks using a mortar and mixed evenly before use.

10.2 Test: Take a weighing bottle that has attained equilibrium mass and place approximately $2\text{g}\sim 10\text{g}$ (precision 0.0001g) samples into the bottle. Thereafter, place the bottle with samples into the vacuum drying oven and connect the oven to a vacuum pump, drawing out the air inside the vacuum oven (required pressure at $40\text{kPa}\sim 53\text{kPa}$) while increasing the temperature simultaneously to the required temperature of $60\text{oC}\pm 5\text{oC}$. Close the piston of the vacuum pump, stop the vacuuming process but maintain the vacuum drying oven at a certain temperature and pressure. After 4h, open the piston and allow air to gradually fill the vacuum drying oven through the dryer equipment, opening the oven only when the pressure inside the oven restores itself to normal conditions. Remove the weighing bottle, place it into a dryer for 0.5h and weigh it. Repeat the previous dryer operation until the difference between two consecutive measurements do not exceed 2mg ; set this as equilibrium mass.

11. Presentation of Analysis Results

Same as Section 6.

12. Precision

Absolute discrepancies between 2 independent test results conducted under iterative conditions should not exceed 10% of their arithmetic mean value.

Method III Distillation Method

13. Principle

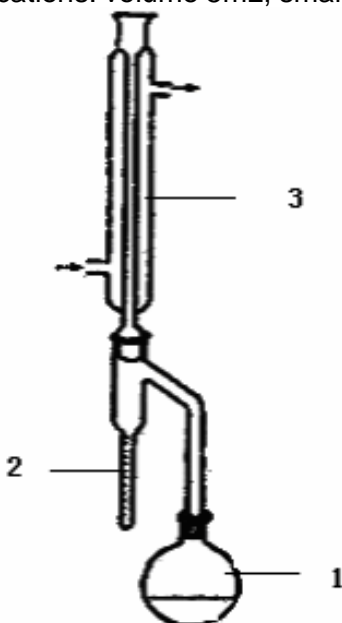
Leveraging on the physical and chemical properties of moisture in foods, determine the moisture content in samples according to the volume of moisture and toluene or xylene in foods extracted during the distillation extraction process using a moisture measuring device. This method applies to food that contains relatively higher content of other volatile substances, e.g. fats, spices.

14. Reagents and Materials

Toluene or Xylene (Chemically Pure): Take toluene or xylene, saturate with water and remove the layer of water after the saturation process. Thereafter, conduct distillation and collect the distillate for use later.

15. Apparatus and Equipment

15.1 Moisture Measuring Device: Illustrated as in Diagram 1 (attached with adjustable electro-heating jacket). Moisture receiving tube specifications: volume 5mL, smallest scale 0.1mL, volume error smaller than 0.1mL.



1. 250mL Distillation Bottle; 2. Moisture Receiving Tube, with Scales; 3. Condensation Tube

Diagram 1 Moisture Measuring Device

15.2 Weighing balance: Sensitivity 0.1mg.

16. Analysis Procedures

Accurately weigh and extract samples (should ensure the final water distillate should be 2mL~5mL, but maximum volume of samples used should not exceed 2/3 of that of the distillation bottle), and place them in a 250mL conical flask. Add 75mL newly distilled toluene (or xylene), connect the condensation tube and the moisture receiving tube. Channel the toluene from the top end of the condensation tube into the device until the moisture receiving tube is full.

Slowly heat up the distillation liquid, producing 2 drops of distillates per second. Increase the distillation speed to 4 drops per second when most of the moisture have been distilled out of the distillation liquid. Add toluene from the top end of the condensation tube to flush and clean the tube when the moisture have been fully distilled and the volume of water in the receiving tube does not increase anymore over time. If there is water droplets formed on the walls of the condensation tube, a copper wire with a small rubber head can be used to wipe these droplets off. Continue the distillation process for a short while until the water droplets stopped forming on the top end of the receiving tube and condensation tube. Distillation end point will be set when the level of distillate remains constant for 10mins. Read off the volume marking.

17. Presentation of Analysis Results

Moisture content in samples can be calculated based on formula (2).

$$X = \frac{V}{m} \times 100 \dots\dots\dots (2)$$

In formula:

X – Moisture content in the samples, units in mL/100g (or the weight of water at 20oC, density 0.998, 20g/mL);

V – Volume of water in receiving tube, units in mL;

m – Mass of samples, units in g.

Present the arithmetic mean value of 2 independent test results under iterative test conditions as the final result, in 3 significant places.

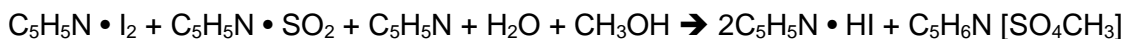
18. Precision

Absolute discrepancies between 2 independent test results conducted under iterative conditions should not exceed 10% of their arithmetic mean value.

Method IV Karl Fischer Method

19. Principle

According to the chemical reaction that can happen between iodine and water, sulfur dioxide; when pyridine and methanol coexist, 1 mol of iodine will react with 1 mol of water, as illustrated by the chemical formula below:



Karl Fischer moisture determination method can be further distinguished into Coulomb's method and titration method. Iodine used in Coulomb's method is produced through chemical reaction, i.e. if the electrolyte solution contains water, the iodine produced from the process will chemically react according to the chemical reaction formula with a 1:1 ratio. Once all the water molecules have went through the abovementioned chemical reaction, excess iodine will form around the anode region and the reaction will stop entirely. Iodine used in the titration method is added as a titrant, with the concentration of the iodine solution known. From there, moisture content in the tested substance can be calculated based on the amount of iodine consumed in accordance to the volume of titrant solution consumed during the process.

20. Reagents & Materials

20.1 Karl Fischer Reagent.

20.2 Anhydrous Methanol: Superiorly pure.

21. Apparatus & Equipment

21.1 Karl Fischer Moisture Determination Device.

21.2 Weighing balance: Sensitivity 0.1mg.

22. Analysis Procedures

22.1 Karl Fischer Reagent Calibration (Titration Method)

Add a certain volume of methanol (so as to fully immerse the platinum electrodes) into the reaction bottle, then titrate with Karl Fischer reagent while stirring till the predetermined titration end point. Add 10mg water (precision 0.0001g), titrate till the predetermined titration end point and make record of the volume (V) of Karl Fischer reagent used in the process. Karl Fischer reagent titer can be calculated according to formula (3):

$$T = \frac{M}{V} \dots\dots\dots (3)$$

In formula:

T – Karl Fischer reagent titer, units in mg/mL;

M – Mass of water, units in mg;

V – Volume of Karl Fischer reagent consumed by the water in the titration process, units in mL.

22.2 Preparation of Samples

Crushable solid samples should be crushed into finer particles as much as possible, and mixed evenly before use. Samples that are hard to crush/grind should be cut into smaller pieces.

22.3 Determination of Water in Samples

Add a certain volume of methanol or the solvent specified for use in the Karl Fischer device into the reaction bottle so as to fully immerse the platinum electrodes and then use the Karl Fischer reagent while stirring to titrate to the predetermined titration end point. Swiftly add the samples that can dissolve in the abovementioned solution with ease into the titration cup; for samples that may not dissolve easily in the solution, they should be added into the titration cup under heated condition or if other solvents that assist the dissolving process have been added. Titrate to the predetermined end point. The Coulomb's method is recommended for samples with moisture content more than 10µg and volume more than 100µg. For samples that may require a substantially longer time for the titration process, the potential drift value should be deducted from the results.

22.4 Determination of Drift Values

Add a certain amount of solvent consistent with the amount of samples added to the titration cup and titrate to the predetermined titration end point. Set it aside and let it settle for not less than 10mins before titrating to the end point again. The volume change between the two titration operations per unit time is the drift value (D).

23. Presentation of Analysis Results

Moisture content in solid samples can be calculated based on formula (4), while moisture content in liquid samples can be calculated based on formula (5).

$$X = \frac{(V_1 - D \times t) \times T}{M} \times 100 \dots\dots\dots (4)$$

$$X = \frac{(V_1 - D \times t) \times T}{V_2 \rho} \times 100 \dots\dots\dots (5)$$

In formula:

X – Moisture content in samples, units in g/100g;

V1 – Volume value of Karl Fischer reagent in the titration process, units in mL;

T – Accurate value of the titer of Karl Fischer reagent, units in g/mL;

M – Mass value of samples, units in g;

V2 – Volume value of liquid samples, units in mL;

D – Drift value, units in mL/min;

t – Time taken for the titration process, units in min;

ρ – Concentration of liquid samples, units in g/mL.

Calculation results should retain 3 significant figures when moisture content $\geq 1\text{g}/100\text{g}$; calculation results should retain 2 significant figures when moisture content $< 1\text{g}/100\text{g}$.

24. Precision

Absolute discrepancies between 2 independent test results conducted under iterative conditions should not exceed 10% of their arithmetic mean value.

GB 4789.2-2010 Food Microbiological Examination: Aerobic Plate Count



National Standards of People's Republic of China

GB 4789.2-2010

**National Food Safety Standard
Food Microbiological Examination
Aerobic Plate Count**

Issued on: 2010-03-26

Implemented on: 2010-06-01

Issued by Ministry of Health of the People's Republic of China

Foreword

The standard substitutes the GB/T4789.2-2008 'Microbiological Examination in Foods: Aerobic Plate Count'

Compared with the GB /T4789.2-2008 main changes are following:

- The Chinese and English names of the standard are revised.
- The explanation of the calculation formula of Aerobic Plate Count is revised.
- The culture mediums and reagents are revised.
- The 2nd method: Petrifilm™ for aerobic plate count is deleted.

The appendix A of the standard is the normative appendix.

The replaced former editions are:

- GB 4789.2-1984, GB/T 4789.2-1994, GB/T 4789.2-2003, GB/T 4789.2-2008.

National Food Safety Standard

Food Microbiological Examination: Aerobic Plate Count

1. Scope

This Standard defines the determination method of aerobic plate count in foods.

This Standard is applicable to the determination method of aerobic plate count in all kinds of foods.

2. Terms and Definitions

2.1 Aerobic plate count

The aerobic plate count obtained from 1ml (or 1g) of sample under certain cultivation conditions (such as the ingredients of culture medium, cultivation temperature and time, pH, and aerobic, etc) after proper treatment.

3. Equipment and Materials

In addition to conventional sterilization and cultivation equipment in microbiological laboratory, other equipment and materials are as follows:

3.1 Thermostatic cultivator: $36\pm 1^{\circ}\text{C}$, $30\pm 1^{\circ}\text{C}$

3.2 Refrigerator: 2°C – 5°C .

3.3 Thermostatic water bath: $46\pm 1^{\circ}\text{C}$

3.4 Balance: accuracy of 0.1g.

3.5 Homogenizer.

3.6 Oscillator.

3.7 Sterile pipette: 1ml (with a scale of 0.01ml), 10ml (with a scale of 0.1ml) or micropipette and tips.

3.8 Sterile conical beaker: 250ml, 500ml.

3.9 Sterile culture plate: with a diameter of 90mm.

3.10 pH meter or pH colorimetric tube or precise pH indicator paper.

3.11 Magnifying glass or/ and bacterial colony counter.

4. Culture Medium and Reagents

4.1 Agar Culture Medium for plate count: please refer to Appendix A.1.

4.2 Phosphate buffer solution: please refer to Appendix A.2.

4.3 Sterile normal saline solution: please refer to Appendix A.3.3

5. Examination Procedures

For the examination procedures of aerobic plate count, please refer to Fig.1.

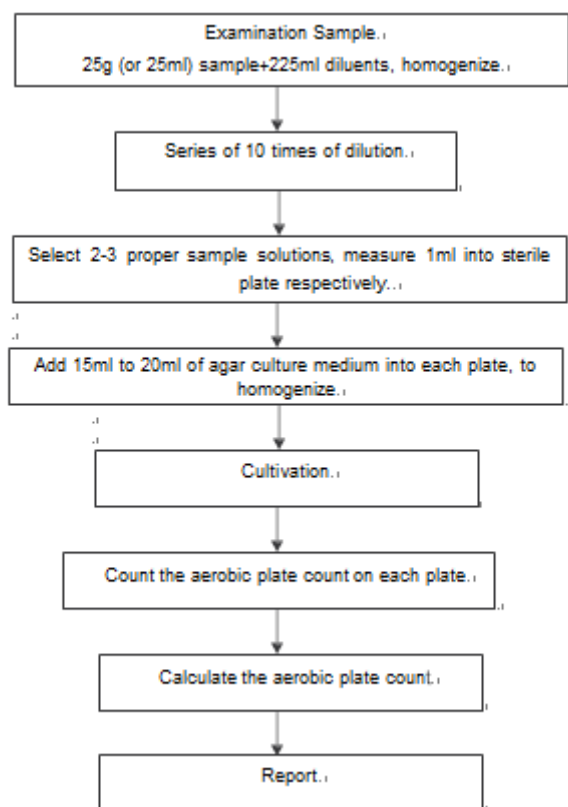


Figure 1 The Examination Procedures of plate count

6. Operation Procedures

6.1 Dilution of samples

6.1.1 Solid and semi solid samples: measure 25g sample into a sterile homogenizing cup containing 225ml phosphate buffer solution or normal saline solution, homogenize with 8000-10000r/min for 1-2min, or place into sterile homogenizing bag containing 225ml diluent, beating with slaping type homogenizer for 1min to 2min, and then formulate into 1:10 sample solution.

6.1.2 Liquid Sample: Measure 25ml sample with sterile pipette into a sterile conical beaker containing 225ml phosphate buffer solution or normal saline solution (proper amount of sterile beads are placed in the beaker in advance), and then homogenize and formulate into 1:10 sample solution.

6.1.3 Absorb 1ml of 1:10 sample solution with 1ml sterile pipette or micropipettor, drip the solution into the sterile tube containing 9ml diluent along the wall of the tube (it is noted that the tip of the pipette shall not touch the diluent solution surface), shake up the test tube or place a piece of sterile pipette, blow repeatedly to homogenize, and then formulate into 1:100 sample solution.

6.1.4 Follow the operation procedures in 6.1.3, formulate the sample solution with series of dilution of 10 times. For each dilution, one piece of 1ml sterile pipette or tip is replaced.

6.1.5 As per the estimation of contamination status of samples, select 2 to 3 sample solutions with proper dilution (for liquid sample, original liquid shall be applied), when carrying out the escalating 10 times series of dilution, for each dilution, 1ml of sample solution is placed into two sterile plates. At the same time, measure 1ml of diluents into two sterile plate respectively to serve as blank controls.

6.1.6. Timely cool down the agar culture medium plates with 15-20ml content in each plate to 46C(which are placed into 46±1C water bath), decant the plates, and then rotate the plates to homogenize.

6.2 Cultivation

6.2.1 After the solidification of agar, turn the plates up-side-down, cultivate at 36±1C for 48h±2h. For aquatic products, cultivate at 30±1C for 72h±3h.

6.2.2 If the samples possibly contain bacteria that could spread growing on the surface of agar culture medium, a thin layer of agar culture medium is covered on the agar surface after solidification (about 4ml), and then turn the plate up-side-down after solidification, and cultivate it as per 6.2.1.

6.3 Plate Count

It could be observed with naked eyes, apply magnifying glass or bacteria colony counter when necessary, and record the dilution times and corresponding plate count. Plate count number is represented by colony-forming units (CFU).

6.3.1 Select the plates for total plate count with colony number between 30-300CFU, and without spreading growth on the plate. For plate with plate count under 30CFU, the number of colony is recorded, while for plate count over 300, it shall be recorded as uncountable. For each dilution degree, the average number of two plates shall be applied.

6.3.2 For those plates with large piece of colony growing, they shall not be applied. However, the plates without large piece of colony growth shall be applied for plate count; If the piece of colony covers less than one half of the plate area, and the colonies on the remaining half of the plate area scatter evenly, it shall be counted of this half of the plate and then multiply by 2, to represent the entire plate count.

6.3.3 When there occurs chain like growth on the plate without evident border line between colonies on the plate, each chain shall be calculated as one colony.

7. Results and reports

7.1 Calculation method for aerobic plate count

7.1.1 If there is only one dilution degree whose plate count fall in the proper counting scope, the average plate count of both plate shall be calculated, and then multiply the average value by corresponding dilution times, to serve as the total plate count in one gram (or ml) of sample.

7.1.2 If there are two continuous dilution degrees whose plate count falls in the proper counting scope, they shall be calculated as in Formula (1):

$$N = \sum C / (n_1 + 0.1n_2)d \dots\dots\dots (1)$$

Where,

N - Plate Count in sample;

$\sum C$ - The total number of colonies on the plates (including the plates within the range of proper plate count;

N1 - The number of colonies on the plates of the first proper dilution degree;

N2 - The number of colonies on the plates of the second proper dilution degree;

d - Dilution Factor (the first dilution degree).

Example:

Dilution degree	1:100 (the first dilution degree)	1:1000 (the second dilution degree)
Number of colonies	232,244	33,35

$$N = \sum C / (n_1 + 0.1n_2)d$$

$$= \frac{232+224+33+35}{[2+(0.1 \times 2)] \times 10^{-2}} = \frac{544}{0.022} = 24727$$

The values mentioned above are round-up, and then represented as 25000 or 2.5×10^4 .

7.1.3 If the colony numbers on the plates of all dilution degrees are all over 300CFU, count the plates with the maximum dilution degree. For other plates, they shall be recorded as uncountable, and the results shall be obtained by multiplying the average colony number by the maximum dilution times.

7.1.4 If the colony numbers on the plates of all dilution degree are all less than 30CFU, it shall be calculated by multiplication of average colony number on the minimum dilution degree plates by the dilution times.

7.1.5 If, for plates of all dilution degrees (including the original liquid samples), there is no colony growth, then it shall be calculated as multiplying the minimum dilution degree by a factor smaller than 1.

7.1.6 If, for plates of all dilution degrees, the colony number falls outside the range between 30CFU and 300CFU, part of which are less than 30CFU or more than 300CFU, then it shall be calculated for the plates whose colony number is closest to 30CFU or 300CFU, as the average colony number multiply by dilution times.

7.2 Reports of plate count

7.2.1 When the plate count falls within 100CFU, it shall be rounded up and reported as interger.

7.2.2 When the plate count is larger than or equal to 100CFU, the third digit shall be rounded up, and take the first two digits, while the following digits are replaced by 0; it could also be indicated as exponential of 10CFU, round-up and then take the two significant digits.

7.2.3 When all the plates are covered by spreading colonies, making it unable to calculate, it shall be reported as colony spreading.

7.2.4 When there are colonies growing on the blank control, the examination result is invalid.

7.2.5 For sampling by weight, CFU/g is applied as the report unit, while for sampling by volume, CFU/ml is applied as the report unit.

Appendix A (Normative Appendix)

Culture Mediums and Reagents

A.1 Plate count agar (PCA) culture medium

A.1.1 Ingredients

Tryptone	5.0g
Yeast Extract	2.5g
Glucose	1.0g
Agar	15.0g
Distilled Water	1000ml

pH 7.0±0.2

A.1.2 Formulation method

Add the above mentioned ingredients into distilled water, boil for dissolving, and then adjust the pH value. Distribute into tubes or conical beakers, autoclave at 121C for 15 min.

A.2 Phosphate Buffer Solution

A.2.1 Ingredients:

KH ₂ PO ₄	34.0g
Distilled Water	500ml

pH 7.2

A.2.2 Formulation Method

Stock Solution: Measure 34.0 g KH₂PO₄ to dissolve in 500ml distilled water, adjust the pH value to 7.2 with about 175ml of 1mol/L NaOH solution, then dilute with distilled water to a volume of 1000ml, and then store in the refrigerator.

Diluent Solution: Measure 1.25ml of the Stock Solution, dilute with distilled water to 1000ml, distribute into proper containers, and then autoclave at 121C for 15min.

A.3 Sterile normal saline solution

A.3.1 Ingredients

NaCl	8.5g
Distilled Water	1000ml

A.3.2 Formulation Method

Measure 8.5g NaCl to dissolve in 1000ml distilled water, and autoclave at 121C for 15 minutes.

GB 4789.3-2010 Food Microbiological Examination: Enumeration of Coliforms



National Standards of People's Republic of China

GB 4789.3-2010

**National Food Safety Standard
Food Microbiological Examination
Enumeration of Coliforms**

Issued on: 2010-03-26

Implemented on: 2010-06-01

Issued by Ministry of Health of the People's Republic of China

Foreword

The standard substitutes the GB/T4789.2-2008 'Microbiological Examination in Foods: Aerobic Plate Count'

Compared with the GB /T4789.2-2008 main changes are following:

- The Chinese and English names of the standard are revised.
- The explanation of the calculation formula of Aerobic Plate Count is revised.
- The culture mediums and reagents are revised.
- The 2nd method: Petrifilm™ for aerobic plate count is deleted.

The appendix A of the standard is the normative appendix.

The replaced former editions are:

- GB 4789.2-1984, GB/T 4789.2-1994, GB/T 4789.2-2003, GB/T 4789.2-2008.

This standard substitutes GB/T 4789.3-2008 Food hygiene microbiological examination: Enumeration of coliforms

Compared with GB/T 4789.3-2008, the main modifications of this standard are as follows:

- Modify Chinese-English titles in the standard;
- The scope of plate colony counts in "Method 2 Coliform Plate Counts" is modified to "15 CFU ♦ 150 CFU";
- Delete "Method 3 Coliform Petrifilm™ Method"

The Annex A and Annex B here of the standard are informative annex. The releases of all editions substituted by this Standard are as follows:

- GB 4789.3-1984, GB 4789.3-1994, GB /T 4789.3-2003, GB /T 4789.3- 20080

National Food Safety Standard

Food Microbiological Examination: Enumeration of Coliforms

1. Scope

This standard provides the method for enumeration of coliforms in foods

This standard is applicable to enumeration of coliforms in various foods.

2. Terms and Definitions

The following terms and definitions are applicable to this standard.

2.1 Coliforms

A cluster of concurrently aerobic and anaerobic gram negative sporeless bacilli which can ferment lactose and generate acid and gas if cultured in 360 for 24 hours.

2.2 Most probable number; MPN

A Poisson distribution-based indirect counting method

3. Devices Requirements

Devices and materials except conventional sterilizing and culturing devices for microbiological laboratory are as follows:

3.1 Thermostatic incubator: $36\text{C}\pm 1\text{C}$

3.2 Refrigerator: $20\sim 5\text{C}$

3.3 Thermostatic water bath: $46\text{C}\pm 1\text{C}$

3.4 Balance: sensitive to 0.1g

3.5 Homogenizer

3.6 Oscillator

3.7 Aseptic suction tube: 1ml (with 0.01ml graduation), 10ml (with 0.1ml graduation) or micro pipettor and sucker

3.8 Aseptic conical flask: 500ml in volume

3.9 Aseptic culture dish: 90mm in diameter

3.10 pH meter or pH colorimetric tube or precision pH test paper 3.11 Colony counter

4. Culture Media and Reagents

4.1 Lauryl sulfate tryptose (LST) broth: See Chapter A.1 of Annex A

- 4.2 Brilliant green lactose bile (BGLB) broth: See Chapter A.2 of Annex A
- 4.3 Violet red bile agar (VRBA): See Chapter A.3 of Annex A
- 4.4 Phosphate buffer solution: See Chapter A.4 of Annex A
- 4.5 Aseptic physiological saline: See Chapter A.5 of Annex A
- 4.6 1mol/L Sodium hydroxide (NaOH): See Chapter A.6 of Annex A
- 4.7 1mol/L Hydrochloric acid (HCL): See Chapter A.7 of Annex A
- 4.8 Petrifilm™ coliform examination test wafer and plate

The First Method: Coliform MPN Counts

5. Examination Procedures

See Figure 1 for Coliform MPN counts examination procedures.

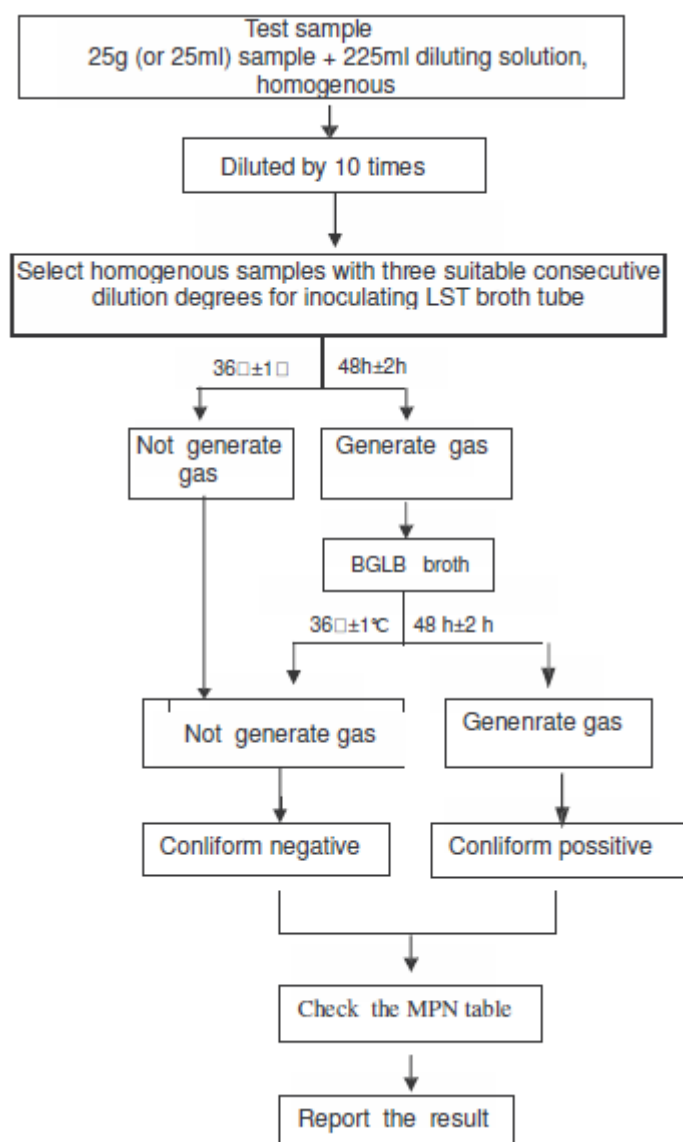


Figure 1: Coliform MPN counts examination procedures

6. Operating Steps

6.1 Diluting the samples

6.1.1 Solid and semi-solid samples: Weigh and take 25g sample, put it in an aseptic homogenizing cup which contains 225ml phosphate buffer solution or physiological saline, and homogenize it 8000r/min to 10000r/min for 1 to 2 minutes; or put it in an aseptic homogenizing bag which contains 225ml phosphate buffer solution or physiological saline and homogenize it by flapping with a smack type homogenizer for 1 to 2 minutes to get 1:10 homogenous sample liquor.

6.1.2 Liquid samples: Suck 25ml sample with an aseptic suction tube, put it in an aseptic conical flask (with a certain number of aseptic glass beads placed inside beforehand) which contains 225ml phosphate buffer solution or physiological saline, and blend the solution properly to get 1:10 homogenous sample liquor.

6.1.3 pH value of the homogenous sample liquor should be between 6.5 and 7.5. Regulate its pH value with 1mol/L sodium hydroxide (NaOH) or 1mol/L hydrochloric acid (HCL) respectively, when necessary.

6.1.4 Suck 1ml 1:10 homogenous sample liquor with a 1ml aseptic suction tube or micro pipettor, empty it in an aseptic test tube (attention: the pointed end of test tube or sucker should not touch the diluting liquid) which contains 9ml phosphate buffer solution or physiological saline slowly along the tube wall, jolt the test tube or beat upon it with a 1ml aseptic suction tube so that it will be homogenized properly to get 1:100 homogenous sample liquor.

6.1.5 According to estimation of sample pollution, make homogenous sample liquor series diluted by 10 times and above as per the above-stated operating steps. For every increased diluting degree, replace one 1ml aseptic suction tube or sucker. From preparation of homogenous sample liquor to completion of inoculation, the whole process should be within 15 minutes.

6.2 Primary fermentation test

For every sample, select homogenous sample liquors with three suitable consecutive dilution degrees (stock solution may be chosen in case of liquid sample), and for every dilution degree, inoculate 3 tubes of lauryl sulfate tryptone (LST) broth, 1ml each tube (if more than 1ml is inoculated, double LST broth should be adopted). Make them cultured in 360 ± 10 for $24\text{h} \pm 2\text{h}$ and observe whether bubbles are generated in the tubes; if there is no any bubble, make them cultured for $48\text{h} \pm 2\text{h}$ in total. Tubes without bubbles are coliform negative and tubes with bubbles go through secondary fermentation test.

6.3 Secondary fermentation

Take 1 circle of cultures from each of all LST broth tubes which ferment and generate gas within $48\text{h} \pm 2\text{h}$ respectively with an inoculation ring, transfer-inoculate them to brilliant green lactose bile (BGLB) broth, culture them in 360 ± 10 for $48\text{h} \pm 2\text{h}$, observe bubble- generation. Tubes which generate bubbles are recorded as coliform positive.

6.4 Reporting most probable number (MPN) of coliforms

According to the number of tubes which are coliform positive verified through 6.3, search the MPN Table (see Annex B) to report coliform MPN counts in every gram (or ml) of sample.

The Second Method: Coliform Plate Counts

7. Examination Procedures

See Figure 2 for coliform plate counts examination procedures.

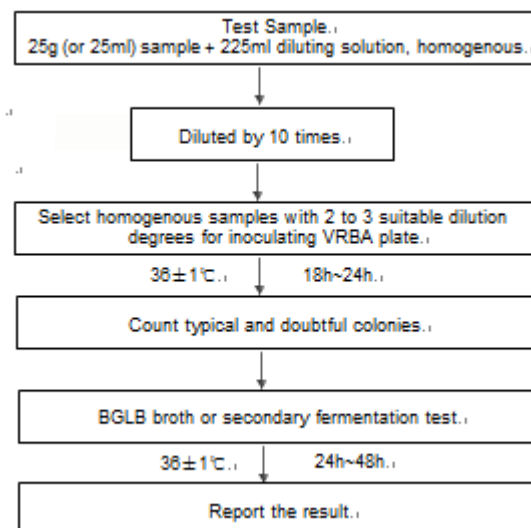


Figure 2: Coliform plate counts examination procedures

8. Operating Steps

8.1 Diluting the samples

Dilute the samples as per clause 6.1.

8.2 Plate count

8.2.1 Select 2 to 3 suitable consecutive dilution degrees, for each of which, inoculate two aseptic flat dishes, 1ml per dish, and at the same time, add 1ml physiological saline in the two aseptic flat dishes for blank control.

8.2.2 Pour 15ml to 20ml violet red bile agar (VRBA) which is cooled to 46C in each of the flat dishes in time, turn the flat dishes carefully to blend the culture medium with the sample liquor properly. After agar is coagulated, add 3ml to 4ml VRBA to cover the plate surface. Flip the plate and put it in 36 ± 10 for 18h to 24h.

8.3 Selecting colony plate counts

Select plates with colony counts between 15 to 150 and count typical and doubtful coliforms appearing on plates. Typical coliforms are in purple, 0.5mm in diameter or bigger, surrounded by red bile salt deposit circle.

8.4 Verification test

Select 10 typical and doubtful coliforms of different types on VRBA plates, transfer- inoculate them in BGLB broth tubes respectively, and culture them in 360 ± 10 for 24h to 48h and observe bubble generation. All BGLB broth tubes which generate gas are reported as coliform positive.

8.5 Reporting coliform plate counts

The percentage of coliform positive test tubes finally confirmed are multiplied by the coliform plate counts in clause 8.3 and dilution multiples to get the number of coliforms per gram (or ml) sample. E.g.: For 10^{-4} diluted sample liquor, 100 typical and doubtful coliforms exist on VRBA plates, and 10 of them are selected and inoculated to BGLB broth tubes, and 6 positive tubes are confirmed. According to the foregoing, it's determined that the number of coliforms in this sample is: $100 \times 6/10 \times 10^4/\text{g (ml)} = 6.0 \times 10^5 \text{ CFU/g (CFU/ml)}$.

Appendix A (Regulatory annex)

Culture Media and Reagents

A.1 Lauryl sulfate tryptose (LST) broth

A.1.1 Ingredients

Typtone or Trypticase	20.0g
Sodium chloride	5.0g
Lactose	5.0g
Dipotassium hydrogen phosphate	2.75g
Monopotassium phosphate	2.75g
Lauryl sodium sulfonate	0.1g
Distilled water	1000ml
pH 6.8±0.2	

A.1.2 Preparation method

Dissolve the above-stated ingredients in distilled water and regulate the pH. Separately fill the solution in test tubes having small glass backward tubes, 10ml each, and sterilize them under high pressure in 121C for 15 minutes.

A.2 Brilliant green lactose bile (BGLB) broth

A.2.1 Ingredients

Peptone	10.0g
Lactose	10.0g
Oxgall or gxbile solution	200.0ml
0.1% brilliant green water solution	13.3ml
Distilled water	1000ml
pH 7.2±0.1	

A.2.2 Preparation method

Dissolve peptone and lactose in about 500ml distilled water, add in 200ml oxgall solution (dissolve 20.0g dehydrated oxgall powder in 200ml distilled water, regulate pH 7.0 to 7.5), dilute it with distilled water to 975ml, regulate its pH to 7.4, and then add in 13.3ml 0.1% brilliant green water solution, dilute it with distilled water to 1000ml, filter the solution, and separately fill the filtrate in test tubes having small glass backward tubes, 10ml each. Sterilize them under high pressure in 1210 for 15 minutes.

A.3 Violet red bile agar (VRBA)

A.3.1 Ingredients

Peptone	7.0g
Yeast cream	3.0g
Lactose	10.0g
Sodium chloride	5.0g
Bile salt or No. 3 bile salt	1.5g
Neutral red	0.03g
Crystal violet	0.002g
Agar	15g-18g
Distilled water	1000ml
pH 7.4±0.1	

A.3.2 Preparation method

Dissolve the foregoing ingredients in distilled water, put the solution in stillness for several minutes, blend it fully, and regulate the pH. Boil it for 2 minutes, cool the culture medium to 450~500 pour plate. Prepare it for immediate use no more than 3 hours later.

A.4. Buffer phosphate

A.4.1 Ingredients

Monopotassium phosphate (KH ₂ PO ₄)	34.0g
Distilled water	500ml
pH 7.2	

A.4.2 Preparation method

Stock solution: Weigh and take 34.0g monopotassium phosphate and dissolve it in 500ml distilled water. Regulate its pH to 7.2 with about 175ml 1mol/L sodium hydrochloride solution, dilute with distilled water to 1000ml, and store it in refrigerator.

Diluted solution: Take 1.25ml stock solution, dilute it with distilled water to 1000ml, separately fill the solution in a suitable container, and sterilize it under high pressure in 1210 for 15 minutes.

A.5 Aseptic physiological saline

A.5.1 Ingredients

Sodium chloride	8.5g
Distilled water	1000ml

A.5.2 Preparation method

Weigh 8.5g sodium chloride, dissolve it in 1000ml distilled water, and sterilize it under high pressure in 1210 for 15 minutes.

A.6 1 mol/L Sodium hydroxide

A.6.1 Ingredients

Sodium hydroxide	40.0g
Distilled water	1000ml

A.6.2 Preparation method

Weigh 40g sodium hydroxide, dissolve it in 1000ml distilled water, and sterilize it under high pressure in 1210 for 15 minutes.

A.7 1 mol/L hydrogen chloride

A.7.1 Ingredients

Hcl Hydrogen chloride	90ml
Distilled water	1000ml

A.7.2 Preparation method

Take 90ml concentrated hydrochloric acid, dilute it with distilled water to 1000ml, and sterilize it under high pressure in 1210 for 15 minutes.

Appendix B (Regulatory annex)

Most Probable Number (MPN) of Coliform Retrieval Table

B.1 Most Probable Number (MPN) of Coliform Retrieval Table

See Table 1 for the most probable number (MPN) of coliforms per gram (or millimeter) test sample.

Table 1: Most probable number (MPN) of coliforms per gram (or millimeter) test sample

Number of positive tubes			MPN	95% confidence		Number of positive tubes			MPN	95% confidence	
0.10	0.01	0.001		Lower limit	Upper limit	0.10	0.01	0.001		Lower limit	Upper limit
0	0	1	<	0.15	9.6	2	2	1	28	8.7	94
0	1	0	3.0	0.15	11	2	2	2	35	8.7	94
0	1	1	3.0	1.2	18	2	3	0	29	8.7	94
0	2	0	6.1	1.2	18	2	3	1	36	8.7	94
0	3	0	6.2	3.6	38	3	0	0	23	4.6	94
1	0	0	9.4	0.17	18	3	0	1	38	8.7	110
1	0	1	3.6	1.3	18	3	0	2	64	17	180
1	0	2	7.2	3.6	38	3	1	0	43	9	180
1	1	0	11	1.3	20	3	1	1	75	17	200
1	1	1	7.4	3.6	38	3	1	2	120	37	420
1	2	0	11	3.6	42	3	1	3	160	40	420
1	2	1	11	4.5	42	3	2	0	93	18	420
1	3	0	15	4.5	42	3	2	1	150	37	420
2	0	0	16	1.4	38	3	2	2	210	40	430
2	0	1	9.2	3.6	42	3	2	3	290	90	1000
2	0	2	14	4.5	42	3	3	0	240	42	1000
2	1	0	20	3.7	42	3	3	1	460	90	2000
2	1	1	15	4.5	42	3	3	2	1100	180	4100
2	1	2	20	8.7	94	3	3	3	> 1100	420	
			27								

Note 1: This table adopts three dilution degrees [0.1g (or 0.1ml), 0.01g (or 0.01ml) and 0.001g (or 0.001ml)], for which of which, three tubes are inoculated.

Note 2: If the tested amounts as shown in this table are changed to 1g (or 1ml), 0.1g (or 0.1ml) and 0.01g (or 0.01ml), figures in this table should be decreased by 10 times accordingly; if the tested amounts are changed to 0.01g (or 0.01ml), 0.001g (or 0.001ml) and 0.0001g (or 0.0001ml), figures in this table should be increased by 10 times accordingly, so on and so forth.

GB 22255-2014 Determination of Sucralose in Foods



National Standards of People's Republic of China

GB 22255-2014

National Food Safety Standard
Determination of Sucralose in Foods

Issued on: 2015-01-28

Implemented on: 2015-07-28

Issued by National Health and Family Planning Commission

Foreword

This standard will replace GB/T 22255-2008 Determination of Sucralose in Foods from the implementation date of this standard.

Compared with GB 2760—2007, the major changes in this standard are as follows:

- This standard changed the standard title into National Standard for Food Safety ----Determination of Sucralose in Foods
- This standard introduced the application of refractive index detectors.
- This standard added the solid-phase extraction column purification and isolation steps in test sample preparation.
- This standard added preparation steps for samples of fermented alcoholic drinks, integrated alcoholic drinks and protein free beverages.

National Standard for Food Safety

Determination of Sucralose in Foods

1. Scope

This standard specifies the method to determine sucralose in foods.

This standard applies to the determination of sucralose in foods.

2. Principle

By extracting sucralose from a food sample using methanol aqueous solution, removing protein and fat contained in the extract, purifying and enriching the extract using a solid-phase extraction column, separating the extract using a high performance liquid chromatograph and a C18 reverse-phase chromatographic column, then analyzing the extract using an evaporative light scattering detector or a refractive index detector, the qualitative and the quantitative analyses could be carried out based on the retention time and peak height/peak area respectively.

3. Reagents and materials

Unless otherwise specified, reagents used for the determination method specified herein are all analytical reagents, and water used is water for analytical laboratory use----Class 1 as specified in GB/T6682.

3.1. Reagents

- 3.1.1 Methanol (CH_3OH)
- 3.1.2 Acetonitrile (CH_3CN): chromatographic grade
- 3.1.3 Hexane (C_6H_{14})
- 3.1.4 Zinc acetate: $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$
- 3.1.5 Potassium Ferrocyanide: $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$
- 3.1.6 Neutral Alumina: 100~200mesh

3.2. Reagent compounding

- 3.2.1 Zinc acetate solution (219g/L): mix 21.9g Zinc acetate and 3mL acetic acid and then dilute with water to 100mL.
- 3.2.2 Potassium ferrocyanide solution (106 g/L): Dilute 10.6g potassium ferrocyanide with water to 100mL.
- 3.2.3 Methanol-water solution (75+25): Mix up 25mL methanol and 75mL water.
- 3.2.4 Acetonitrile-water solution (11+89): Mix up 11mL acetonitrile and 89mL water.

3.3. Standard products

Standard Sucralose ($C_{12}H_{19}C_{13}O_8$): CAS No.: 56038-13-2, purity $\geq 99\%$

3.4. Formulation of standard solution

3.4.1 Sucralose Standard Stock Solution (10.0mg/mL): Transfer 0.25g (accurate to 0.0001g) standard sucralose to a 25mL volumetric flask, dilute with water to volume and mix up, the concentration will be 10.0mg/mL. Shelf life of the solution stored in a lab refrigerator at 4°Cs 6 months.

3.4.2 Sucralose Inter-mediate Standard Solution: Transfer 5.00 mg/mL sucralose standard stock solution prepared in 3.4.1 to a 50mL volumetric flask, dilute with water to volume and mix up, the concentration will be 1.0mg/mL. Shelf life of the solution stored in a lab refrigerator at 4°Cs 3 months.

3.4.3 Sucralose Standard Working Solutions: Transfer 0.200mL, 0.500mL, 1.00mL, 2.00mL and 4.00mL inter-mediate standard solution prepared in 3.4.2 to five 10mL volumetric flasks, respectively. Dilute each with water to volume and mix up, the concentration will be 0.0200mg/mL, 0.0500mg/mL, 0.100mg/mL, 0.200mg/mL, and 0.400mg/mL respectively.

3.5. Materials

Solid-phase extraction columns (200mg, using hydrophile-lipophile balanced N-Vinyl-2-pyrrolidone and divinylbenzene as packing) should be activated by 4mL methanol and 4mL water successively before use.

4. Equipment and facilities

4.1 High Performance Liquid Chromatograph: equipped with a refractive index detector/evaporative light scattering detector.

4.2 Balance Scale: a sensor volume is 0.1mg/1mg

4.3 Vortex Mixer

4.4 Centrifuge: rotating speed $\geq 3,000$ r/min

4.5 Centrifuge: rotating speed $\geq 10,000$ r/min

4.6 Ultra-sonic Cleaner: operating frequency: 35KHz

4.7 Water Bath

5. Analytical procedures

5.1 Specimen preparation

5.1.1 Test Samples Containing Protein and Fat

5.1.1.1 Transfer a piece of 1~2g (accurate to 0.001g) solid sample crushed evenly and 1~5g liquid sample (accurate to 0.001g) to a 50mL centrifuge tube, add 5mL water, vortex on the vortex mixer for 3 min, then add 15mL methanol, and vortex on the vortex mixer again for 30 s; after an ultrasonic extraction for 20 min, centrifugate at 3000r/min for 10 min, then transfer the supernatant to a 50mL centrifuge tube. Add 5.0mL methanol-water solution (75+25) into the sediment, stir up with a glass rod, vortex on the vortex mixer for 30s, centrifugate at 3000r/min for 10 min, and do the extraction operation again, then collect and transfer all supernatants to a 150mL separatory funnel.

5.1.1.2 Add 30mL Hexane into the 150mL separatory funnel mentioned above, shake for 2 min, allow the mixture to stand for 20 minutes and separate into two layers, transfer the lower layer water phase into a 50mL evaporation dish; put the evaporation dish into the water bath for evaporation by boiling until only about 1mL liquid remained. Wash the evaporation dish with 9mL water totally in three flushing, collect and transfer the used water for the three flushing to a 15mL centrifuge tube. After an ultra-sonic treatment of 5min, centrifugate the used flushing water at 3000r/min for 10 min.

5.1.1.3 Inject all supernatants mentioned in 5.1.1.1 into an activated solid extraction column at a flow rate no more than 1 drop/second, add 1mL water when the supernatant level in the column reaches about 2mm, drain the extraction column keeping the flow rate at 1 drop/second. Then elute the extraction column with 3mL methanol, collected and transfer the used methanol eluent to a 50mL evaporation dish, put the evaporation dish into the water bath for evaporation by boiling until the used methanol eluent contained is dried up, dilute the evaporation residue with 1.0mL acetonitrile-water solution(11+89), and filter the solution with a 0.45µm membrane filter, the filtered solution is the prepared test sample.

Note: For jelly products, the supernatants extracted should be heated in the water bath at 50°C and pass through the extraction column when it is still hot to avoid plugging.

5.1.2 Sauce and Sauce Pickled Products, Soy Sauce & Vinegar

Transfer a 2g evenly mixed product sample (accurate to 0.001g) to a 50mL centrifuge tube, add 1.0g neutral alumina and 3mL water, vortex on the vortex mixer for 3min, then add 15mL methanol, and complete the procedure specified from 5.1.1.1 “and vortex on the vortex mixer again for 30 s; after an ultrasonic extraction for 20 min, centrifugate at 3000r/min for 10 min” to 5.1.1.3 “the filtered solution is the prepared test sample” in order.

5.1.3 Alcoholic Samples(fermented alcoholic drinks and integrated alcoholic drinks)

Transfer a 2g evenly mixed product sample (accurate to 0.001g) to a 50mL evaporation dish, put the evaporation dish into the water bath for evaporation by boiling until the sample is dried up, dilute the evaporation residue with 1.0mL acetonitrile-water solution(11+89), and filter the solution with a 0.45µm membrane filter, the filtered solution is the prepared test sample.

5.1.4 Non-alcoholic Beverages

Transfer a 2g evenly mixed product sample (accurate to 0.001g) to a 15mL centrifuge tube, add 5mL water, vortex on the vortex mixer for 30s, centrifugate at 3000r/min for 10 min, then complete steps specified in 5.1.1.3.

5.1.5 Flavored Fermented Milk and Milky Tea Products

Transfer a 1~5g evenly mixed product sample (accurate to 0.001g) to a 50mL centrifuge tube, add 5mL water, vortex on the vortex mixer for 3min, add 15mL methanol, 0.50 mL zinc acetate solution, and 0.50 mL potassium ferrocyanide solution, centrifugate at 3000r/min for 10 min, and complete the procedure specified from 5.1.1.1 “and vortex on the vortex mixer again for 30 s; after an ultrasonic extraction for 20 min, centrifugate at 3000r/min for 10 min” to 5.1.1.3 “the filtered solution is the prepared test sample” in order.

For various test samples, sample pre-treatment shall be done simultaneously with a blank test.

5.2 Reference conditions for equipment

5.2.1 Chromatographic Column: C18, (4.6×150mm, 5µm) or equivalent product

5.2.2 Mobile Phase: acetonitrile-water solution(89+11)

Note: In order to avoid influences of strong retention substances on subsequent testing steps, an elution procedure (applicable to evaporative light scattering detectors) could be to adopt after testing a sample with complex matrix.

5.2.3 Flow Rate: 1.0mL/min

5.2.4 Column Temperature: 35°C

5.2.5 Condition of Refractive Index Detector

----Temperature in Detection Cell: 35°C

----Detection Sensitivity: 16

5.2.6 Condition of Evaporative Light Scattering Detector:

The detector should be set according to its brand-specific requirements for high water mobile phase.

For example, a Sedex 75 evaporative light scattering detector should be set as follows:

Atomization Pressure: 3.5Bar, Gain:8,Evaporation Temperature:80°C

Otherwise, a detector with equivalent performances should be used.

5.2.7 Injection Volume: 20.0µL

5.3 Standard curve mapping

5.3.1 Refractive Index Detector

Inject 20.0µL of the 5 kinds of sucralose standard working solutions, respectively. Determine the peak area under the chromatographic conditions mentioned above, and establish the peak area-sucralose concentration (mg/mL) standard curve. As shown in Formula (1), the curve equation complies with working principles of the refractive index detector.

$$y = ax + b \quad \text{.....(1)}$$

In the formula:

y----refers to the peak area

a, b---constants related to detection cell temperature, mobile phase properties and other experimental conditions

x---sucralose concentration (mg/mL)

5.3.2 Evaporative Light Scattering Detector

Inject 20.0µL of the 5 kinds of sucralose standard working solutions, respectively. Determine the peak area under the given chromatographic conditions, and establish the peak area-sucralose concentration (mg/mL) standard curve. As shown in Formula (2), the curve equation complies with working principles of the evaporative light-scattering detector.

$$y = bx^a \quad \text{..... (2)}$$

In the formula:

y----refers to the peak area

a, b---constants related to evaporation chamber temperature, mobile phase properties and other experimental conditions

x---sucralose concentration (mg/mL)

Depending on the data processing method of software processing data collected by experimental instruments, the above mentioned Equation may be converted into a logarithmic equation, i.e. $\lg y = b + a \lg x$.

5.4 Testing of Sample Solutions

Inject 20.0µL sample solution and 20.0µL blank solution into a high performance liquid chromatograph , respectively and carry out high-performance liquid chromatographic evaluations. As specified in Annex B, the qualitative and the quantitative analyses shall be carried out based on the retention time and the peak area external reference method, respectively.

6. Statement of analysis result

The sucralose content in a test sample could be expressed by Formula (3)

$$X = \frac{(c - c_0) \times V \times 1\,000}{m \times 1\,000} \quad \text{..... (3)}$$

In the formula:

X---refers to the sucralose content in a test sample

c---refers to the sucralose concentration positioned on the standard curve, in the liquid injected as sample, (mg/mL)

c₀---refers to the sucralose concentration positioned on the standard curve, in the liquid injected as blank sample, (mg/mL)

V---refers to the test sample's constant volume, (mL)

m---mass of the test sample, (g)

1000---conversion coefficient

The calculation results should be reserved 3-digit decimals.

7. Precision

The absolute difference between two independent determination results obtained based on the repeatability of the determination procedure should not exceed 10% of these two results' mathematic average.

8. Others

If dilute a sample of 2g with water to 1.00 mL, the detection limit is 0.0025 g/kg, and the quantitation limit is 0.0075 g/kg.

Appendix A

Elution Procedure of the Liquid Chromatograph under Complex Conditions (applicable to evaporative light scattering detectors)

In order to avoid influences of strong retention substances on subsequent testing steps, an elution procedure could be adopted after testing a sample with complex matrix. Elution parameters are as shown in Table A.1.

Table A.1 Elution Procedure of the Liquid Chromatograph under Complex Conditions (applicable to evaporative light scattering detectors)

Time (min)	Ultra-pure Water (Volume Fraction) %	Acetonitrile (Volume Fraction) %
0	89	11
14	89	11
15	10	90
22	10	90
23	89	11
26	89	11

Appendix B

High Performance Liquid Chromatogram of Sucralose

Refer to Fig B.1 for liquid chromatogram (evaporative light scattering detector) of standard sucralose solution (0.200mL).

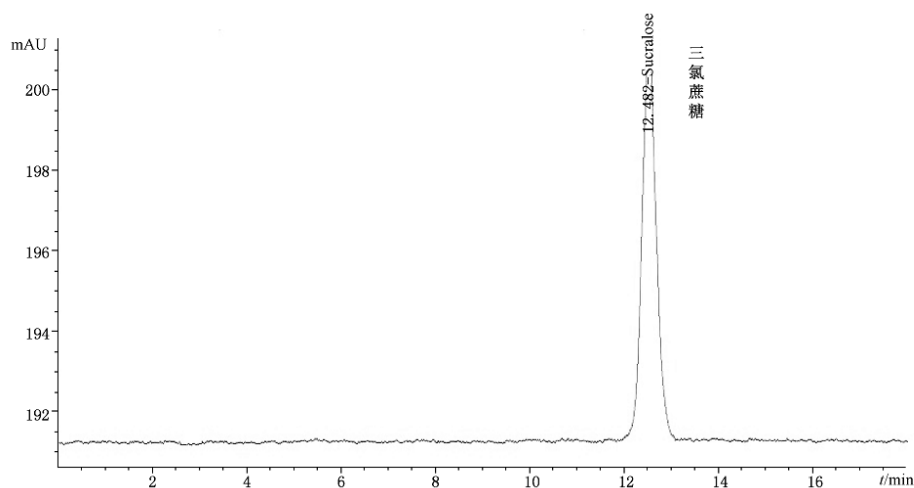


Fig B.1 Liquid Chromatogram(evaporative light scattering detector) of Standard Sucralose Solution

Refer to Fig B.2 for liquid chromatogram (refractive index detector) of standard sucralose solution (0.200mL).

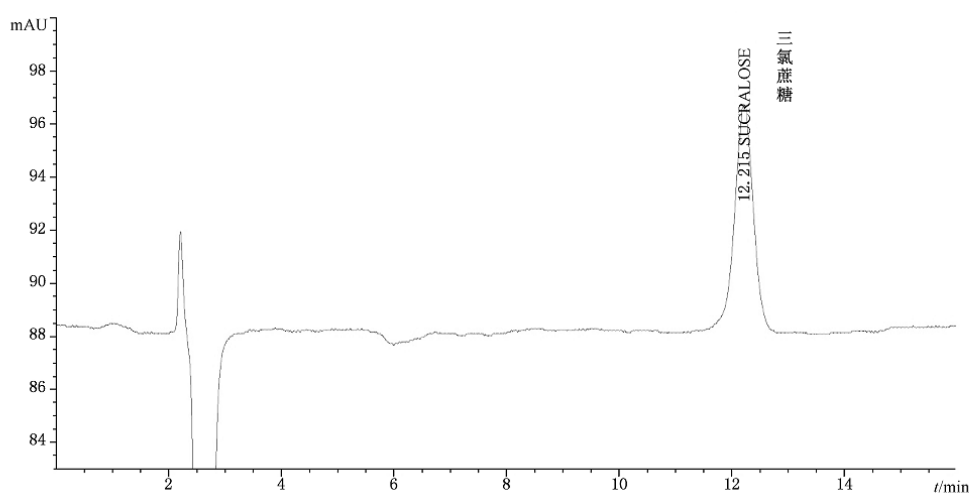


Fig B.2 Liquid Chromatogram (refractive index detector) of Standard Sucralose Solution

**GB 14880-2012 Standard for the Use of Nutritional Fortification Substances in
Foods**



National Standards of People's Republic of China

GB 14880-2012

National Food Safety Standard
Standard for the Use of Nutritional Fortification Substances in Foods

Issued on: 2010-03-15

Implemented on: 2013-01-01

Issued by National Health and Family Planning Commission

Foreword

This standard referred to the standards of Codex Alimentarius Commission (CAC) 'Codex Stan CAC/GL 09-1987 (amended 1989,1991) General Principles for the addition of Essential Nutrients to Foods' and CAC/GL 10-1979 (revised 2008) Advisory Lists of Nutrient Compounds for Use in Foods for Special Dietary Uses for Infants and Young Children. This standard also referred to the regulation of European Union 'No. 1925/2006 Regulation on the Addition of Vitamins and Minerals and of certain other substances to foods'.

This standard replaced the previous version: the standard GB 14880-1994 < Hygiene Standard for the use of nutritional fortification substances in foods>

Compared to the standard GB 14880-1994, this standard has some changes as follows:

- More technical terms and definitions are added.
- The principles of nutritional fortification, the principles of using nutritional fortification substances and the principles of selecting carriers for nutritional fortification substances are added.
- Based on the risk assessment, the food classification system is combined into this standard. The application of varieties, scope and amount of nutritional fortification substances are adjusted and integrated. Some food categories, which are not suitable for nutritional fortification, are deleted.
- The allowed sources of nutritional fortification substances are increased
- The sources of nutritional fortification substances for food for special dietary uses are increased and the application amounts of some of the nutrients are increased
- The food classification system is added
- The Appendix A 'Rules for implementation of health standards for nutritional fortification substances is deleted. (The Appendix A, Appendix B and Appendix C are standardized appendices. Appendix D is information Appendix)>

The previous issued version replaced by this standard is

- GB 14880-1994

National Standard for Food Safety

Standard for the Use of Nutritional Fortification Substances in Foods

1. Scope

This Standard stipulates the principles for the fortification of nutritional fortification substances in food, the principles for the use of nutritional fortification substances and the principles for selecting carrier of nutritional fortification substances. This standard also stipulates the application of varieties, scope and amount of the nutritional fortification substances.

This standard is applicable to all nutritional fortification substances.

2. Terminology and definitions

2.1. Nutritional fortification substances

Nutritional fortification substances are natural, synthetic nutrients and other nutritional components, which are added into food to increase the nutritional value of food.

2.2. Nutrients

A nutrient is a substance that an organism needs to live, grow, develop, reproduce and metabolize. It has special physiological functions. Nutrients include proteins and amino acids, fats and fatty acids, carbohydrates, minerals, vitamins, and etc.

2.3. Other nutritional components (excluding nutrients mentioned in 2.2)

Nutritional components are food ingredients, which have nutritional and physiological functions.

2.4. Fortified food

Fortified foods are those foodstuffs in which a certain amount of nutritional fortification substances are added.

2.5. Food for special dietary uses

These foods must supply a special dietary need that exists by reason of a physical or physiological condition or by reason of a specific disease or disorder. The ingredients of these foods should be significantly different from those of conventional or natural foods.

3. Principles of nutritional fortification

3.1. Nutritional fortification substances can be used in the following situations:

3.2. Nutritional fortification substances can be used to compensate the nutrient loss during food processing or storage.

3.3. There is sufficient evidence to show that a certain type of nutrient deficiency exists in a specific group of people with a population of considerable size in a certain geographical area; and the low intake level of the nutrients and adverse health effects caused by nutrient deficiency can be improved by nutritional fortification; and the nutrients can be provided to the people with the nutrient deficiency through fortified foods.

3.4. There is sufficient evidence to show that in certain geographic areas, the low intake of certain/some nutrients or nutrient deficiencies are caused by dietary habits or other factors; and the low intake level of the nutrients and adverse health effects caused by the nutrient deficiency can be improved by nutritional fortification and the nutrients can be provided to the people with nutrient deficiency through fortified foods.

3.5. To increase the nutritional content of substitutes for traditional food.

3.6. Supplement and modify the nutrients and the content of other nutritional substances in food for special dietary uses.

4. Principles for the use of nutritional fortification substances

4.1. The use of nutritional fortification substances should comply with the following principles:

4.2. The use of nutritional fortification substances must not lead to excessive intake of the nutrients or/and nutrient imbalance; and must not lead to metabolic disorders.

4.3. Nutritional fortification substances should remain stable (in terms of quality) in foods during storage, transport, and consumption.

4.4. Additional of nutritional fortification substances must not lead to undesirable changes of foods in terms of colors, taste, flavor, cooking property etc.

4.5. The use of nutritional fortification substances should not cause misleading information and misconception to consumers through exaggerating the content of a certain nutritional ingredient.

4.6. The use of nutritional fortification substances should not encourage or lead to food consumption models that are contrary to the country's nutrition regulations.

5. The principles of selecting carriers for nutritional fortification substances

5.1. The selection of carriers for nutritional fortification substances should comply with the following principles:

5.2. Should select foods that are easy to purchase and obtain by the target population.

5.3. The consumption of food carriers should be stable, thus to help calculate the additive amount of nutritional fortification substances, and should be able to avoid excessive levels of nutrients and other nutritional substances in the human body due to excessive intake of the food carriers.

5.4. The natural foods, which are already good source of a certain nutrients, should not be used as carriers of this nutrient.

6. The regulation of the use of nutritional fortification substances

6.1. The application scope, application amount of nutritional fortification substances should comply with the requirement of Appendix A, the sources of chemicals should comply with the regulation of Appendix B;

6.2. The contents of nutrients and other nutritional substances used in foods for special dietary uses should comply with the National Food Safety Regulation, the allowed sources of nutritional fortification substances should comply with the requirement of Appendix C.

7. Food classification system

The food class system is used to define the application scope of nutritional fortification substances. It is only applicable for this standard, please refer to Appendix D If a certain nutritional fortification substances is allowed to use in a certain food category, then this substances can be used in all foodstuffs under this category, except otherwise provided.

8. The standard of quality specification

The uses of nutritional fortification substances should comply with the standard of quality specification and corresponding regulations.

Appendix A
Standardized Appendix
Regulation on the use of nutritional fortification substances in foods

Nutrients	Classification of food		Application amount/kg
	Food Codes	Classification/Name	
Vitamin A	01.01.03	Modified milk powder	600-1000 µg/kg
	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	3000-9000 µg/kg
		Modified milk powder (for children)	1200-7000 µg/kg
		Modified milk powder (for pregnant women)	2000-10000 µg/kg
	02.01.01.01	Vegetable oil	4000-8000 µg/kg
	02.02.01.02	Margarine and similar products	4000-8000 µg/kg
	03.01	Ice cream and ice confectionery	600-1200 µg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	3000-7000 µg/kg
	04.04.01.08	Soybean	600-1400 µg/kg
	06.02.01	Rice	600-1200 µg/kg
	06.03.01	Wheat/flour	600-1200 µg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	2000-6000 µg/kg
	07.02.02	Foreign pastry	2330-4000 µg/kg
	07.03	Biscuits	2330-4000 µg/kg
	14.03.01	Milk containing drinks	300-1000 µg/kg
	14.06	Solid beverage	4000-17000 µg/kg
	16.01	Jelly	600-1000 µg/kg
	16.06	Puffing food	600-1500 µg/kg
B-carotene	14.06	Solid beverages	3-6 mg/kg
Vitamin D	01.01.03	Modified milk powder	10-40 µg/kg
	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	63-125 µg/kg
		Modified milk powder (for children)	20-112 µg/kg
		Modified milk powder (for pregnant women)	23-112 µg/kg
	02.02.01.02	Margarine and similar products	125-156 µg/kg
	03.01	Ice cream and ice confectionery	10-20 µg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	15-60 µg/kg
	04.04.01.08	Soybean	3-15 µg/kg
	06.05.02.03	Lotus root starch	50-100 µg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	12.5-37.5 µg/kg
	07.03	Biscuits	16.7-33.3 µg/kg
	07.05	Other baked products	10-70 µg/kg/kg
	14.02.03	Fruit and vegetable juices (Include fermented products)	2-10 µg/kg
	14.03.01	Milk containing drinks	10-40 µg/kg
	14.04.02.02	Flavored beverages	2-10 µg/kg
	14.06	Solid beverages	10-20 µg/kg
	16.01	Jelly	10-40 µg/kg
	16.06	Puffing food	10-60 µg/kg
Vitamin E	01.01.03	Modified milk powder	12-50 mg/kg

	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	100-310 mg/kg
		Modified milk powder (for children)	10-60 mg/kg
		Modified milk powder (for pregnant women)	32-156 mg/kg
	02.01.01.01	Vegetable oils	100-180 mg/kg
	02.02.01.02	Margarine and similar products	100-180 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	30-70 mg/kg
	04.04.01.08	Soybean	5-15 mg/kg
	05.02.01	Gum candy	1050-1450 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	50-125 mg/kg
	14.0	Beverage (excluding beverages covered under 14.01, 14.06)	10-40 mg/kg
	14.06	Solid beverages	76-180 mg/kg
	16.01	Jelly	10-70 mg/kg
Vitamin K	01.03.02	Modified milk powder (for children)	420-750 mg/kg
		Modified milk powder (for pregnant women)	340-680 mg/kg
Vitamin B ₁	01.03.02	Modified milk powder (for children)	1.5-14 mg/kg
		Modified milk powder (for pregnant women)	3-17 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	6-15 mg/kg
	04.04.01.08	Soybean	1-3 mg/kg
	05.02.01	Gum candy	16-33 mg/kg
	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	3-5 mg/kg
	06.03	Wheat flour and derived products	3-5 mg/kg
	06.04	Grain flour (soybean flour only)	3-5 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	7.5-17.5 mg/kg
	07.01	Bread	3-5 mg/kg
	07.02.02	Foreign pastry	3-6 mg/kg
	07.03	Biscuits	3-6 mg/kg
	14.03.01	Milk containing beverages	1-2 mg/kg
	14.04.02.02	Flavored beverages	2-3 mg/kg
	14.06	Solid beverages	9-22 mg/kg
	16.01	Jelly	1-7 mg/kg
Vitamin B ₂	01.03.02	Modified milk powder (for children)	8-14 mg/kg
		Modified milk powder (for pregnant women)	4-22 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	6-15 mg/kg
	04.04.01.08	Soybean	1-3 mg/kg
	05.02.01	Gum candy	16-33 mg/kg
	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	3-5 mg/kg
	06.03	Wheat flour and derived products	3-5 mg/kg
	06.04	Grains flour (including soybean flour) and derived products (soybean flour only)	3-5 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	7.5-17.5 mg/kg

	07.01	Bread	3-5 mg/kg
	07.02.02	Foreign pastry	3.3-7.0 mg/kg
	07.03	Biscuits	3.3-7.0 mg/kg
	14.03.01	Milk containing drinks	1-2 mg/kg
	14.06	Solid beverages	9-22 mg/kg
	16.01	Jelly	1-7 mg/kg
Vitamin B ₆	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	8-16 mg/kg
		Modified milk powder (for children)	1-7 mg/kg
		Modified milk powder (for pregnant women)	4-22 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	10-25 mg/kg
	07.03	Biscuits	2-5 mg/kg
	07.05	Other baked products	3-15 mg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	0.4-1.6 mg/kg
	14.06	Solid beverages	7-22 mg/kg
	16.01	Jelly	1-7 mg/kg
Vitamin B ₁₂	01.03.02	Modified milk powder (for children)	10-30 µg/kg
		Modified milk powder (for pregnant women)	10-66 µg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	5-10 µg/kg
	07.05	Other baked products	10-70 µg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	0.6-1.8 µg/kg
	14.06	Solid beverages	10-66 µg/kg
	16.01	Jelly	2-6 µg/kg
Vitamin C	01.02.02	Favored fermented product	120-240 mg/kg
	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	300-1000 mg/kg
		Modified milk powder (for children)	140-800 mg/kg
		Modified milk powder (for pregnant women)	1000-1600 mg/kg
	04.01.02.01	Canned fruits	200-400 mg/kg
	04.01.02.02	Puree	50-100 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	400-700 mg/kg
	05.02.01	Gum candy	630-13000 mg/kg
	05.02.02	Sweets other than gum candy	1000-6000 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	300-750 mg/kg
	14.02.03	Fruits and vegetable (pulp) juices	250-500 mg/kg
	14.03.01	Milk containing drinks	120-240 mg/kg
	14.04	Water-based flavored beverages	250-500 mg/kg
	14.06	Solid beverage	1000-2250 mg/kg
	16.01	Jelly	120-240 mg/kg
Niacin (or nicotinamide)	01.03.02	Modified milk powder (for children)	23-47 mg/kg
		Modified milk powder (for pregnant women)	42-100 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	60-120 mg/kg
	04.04.01.08	Soybean	10-30 mg/kg

	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	40-50 mg/kg
	06.03	Wheat flour and derived products	40-50 mg/kg
	06.04	Grain flour (including soybean flour) and derived products (soybean flour only)	40-50 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	75-218 mg/kg
	07.01	Bread	40-50 mg/kg
	07.03	Biscuits	30-60 mg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	3-18 mg/kg
	14.06	Solid beverages (excluding soy milk powder)	110-330 mg/kg
Folic acid	01.01.03	Modified milk powder	400-1200 µg/kg
	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	2000-5000 µg/kg
		Modified milk powder (for children)	420-3000 µg/kg
		Modified milk powder (for pregnant women)	2000-8200 µg/kg
	06.02.01	Rice (washing-face rice only)	1000-3000 µg/kg
	06.03.01	Wheat flour	1000-3000 µg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	1000-2500 µg/kg
	07.03	Biscuits	390-780 µg/kg
	07.05	Other baked products	2000-7000 µg/kg
	14.02.03	Fruits and vegetable (pulp) juices	157-313 µg/kg
	14.06	Solid beverages	600-6000 µg/kg
	16.01	Jelly	50-100 µg/kg
Pantothenic acid	01.03.02	Modified milk powder (for children)	6-60 mg/kg
		Modified milk powder (for pregnant women)	20-80 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	30-50 mg/kg
	14.04.01	Carbonated drinks	1.1-2.2 mg/kg
	14.04.02.02	Flavored drinks	1.1-2.2 mg/kg
	14.05.01	Tea drinks	1.1-2.2 mg/kg
	14.06	Solid beverages	22-80 mg/kg
	16.01	Jelly	2-5 mg/kg
Biotin	01.03.02	Modified milk powder (for children only)	38-76 µg/kg
Chlorine	01.03.02	Modified milk powder (for children only)	800-3000 mg/kg
		Modified milk powder (for pregnant women only)	1600-3400 mg/kg
	16.01	Jelly	50-100 mg/kg
Inositol	01.03.02	Modified milk powder and modified cream powder (including flavored milk powder and flavored cream powder)	210-250 mg/kg
	14.02.03	Fruits and vegetable (pulp) juices	60-120 mg/kg
	14.04.02.02	Flavored beverages	60-120 mg/kg
Minerals			
Iron	01.01.03	Modified milk powder	10-20 mg/kg
	01.03.02	Modified milk powder (excluding milk	60-200 mg/kg

		powder used for pregnant women and children)	
		Modified milk powder (for children)	25-135m mg/kg
		Modified milk powder (for pregnant women)	50-280 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	46-80 mg/kg
	05.02.02	Sweets other than gum candy	600-1200 mg/kg
	06.02	Rice and derived products (Rice, rice vermicelli, rice cake)	14-26 mg/kg
	06.03	Wheat flour and derived products	14-26 mg/kg
	06.04	Grain flour (soybean flour only)	14-26 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	35-80 mg/kg
	07.01	Bread	14-26 mg/kg
	07.02.02	Foreign pastry	40-60 mg/kg
	07.03	Biscuits	40-60 mg/kg
	07.05	Other baked products	50-200 mg/kg
	12.04	Soy sauce	180-260 mg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	10-20 mg/kg
	14.06	Solid beverages (excluding the food mentioned in 14.06.02)	95-220 mg/kg
	16.01	Jelly	10-20 mg/kg
Calcium	01.01.03	Modified milk powder	250-1000 mg/kg
	01.03.02	Modified milk powder (for children)	3000-7200 mg/kg
		Modified milk powder (for pregnant women)	3000-6000 mg/kg
	01.06	Cheese	2500-10000 mg/kg
	03.01	Ice cream and ice cream cake products	2400-3000 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	1600-8000 mg/kg
	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	1600-3200 mg/kg
	06.03	Wheat flour and derived products	1600-3200 mg/kg
	06.04	Grain flour (Including soybean flour) and derived products (soybean flour only)	1600-3200 mg/kg
	06.05.02.03	Lotus root starch	2400-3200 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	2000-7000 mg/kg
	07.01	Bread	1600-3200 mg/kg
	07.02.02	Foreign pastry	2670-5330 mg/kg
	07.03	Biscuits	2670-5330 mg/kg
	07.05	Other baked products	3000-15000 mg/kg
	08.03.05	Meat sausage	850-1700 mg/kg
	08.03.07.01	Dry meat floss	2500-5000 mg/kg
	08.03.07.02	Bak kwa products	1700-2550 mg/kg
	10.03.01	Dehydrated egg products	190-650 mg/kg
	12.03	Vinegar	6000-8000 mg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	160-1350 mg/kg
	14.02.03	Fruits and vegetable (pulp) juices	1000-1800 mg/kg
	14.06	Solid beverages	2500-10000 mg/kg
	16.01	Jelly	390-800 mg/kg

Zinc	01.01.03	Modified milk powder	5-10 mg/kg
	01.03.02	Modified milk powder (excluding milk powder used for pregnant women and children)	30-60 mg/kg
		Modified milk powder (for children)	50-175 mg/kg
		Modified milk powder (for pregnant women)	30-140 mg/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	29-55.5 mg/kg
	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	10-40 mg/kg
	06.03	Wheat flour and derived products	10-40 mg/kg
	06.04	Grain flour (soybean flour only)	10-40 mg/kg
	06.06	Ready-to-eat cereals, including oats and rolled oats	37.5-112.5 mg/kg
	07.01	Bread	10-40 mg/kg
	07.02.02	Foreign pastry	45-80 mg/kg
	07.03	Biscuits	45-80 mg/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	3-20 mg/kg
	14.06	Solid beverages (excluding soy milk powder)	60-180 mg/kg
	16.01	Jelly	10-20 mg/kg
Selenium	01.03.02	Modified milk powder (excluding children)	140-280 µg/kg
		Modified milk powder (for children only)	60-130 µg/kg
	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	140-280 µg/kg
	06.03	Wheat flour and derived products)	140-280 µg/kg
	06.04	Grain flour (Including soybean flour) and derived products (soybean flour only)	140-280 µg/kg
	07.01	Bread	140-280 µg/kg
	07.03	Biscuits	30-110 µg/kg
	14.03.01	Milk containing drinks	50-200 µg/kg
Magnesium	01.03.02	Modified milk powder (excluding pregnant women and children)	300-1100 mg/kg
		Modified milk powder (for pregnant women only)	300-2800 mg/kg
	01.03.02	Modified milk powder (for children only)	300-2300 mg/kg
	14.0	Beverages (excluding the food mentioned in 14.01 and 14.04.01)	30-60 mg/kg
	14.06	Solid beverages	1300-2100 mg/kg
Copper	01.03.02	Modified milk powder (excluding pregnant women and children)	3-7.5 mg/kg
		Modified milk powder (for children only)	2-12 mg/kg
		Modified milk powder (for pregnant women only)	4-23 mg/kg
Manganese	01.03.02	Modified milk powder (excluding pregnant women and children)	0.3-4.3 mg/kg
		Modified milk powder (for children only)	7-15 mg/kg
		Modified milk powder (for pregnant	11-26 mg/kg

		women only)	
Potassium	01.03.02	Modified milk powder (excluding pregnant women and children)	7000-14100 mg/kg
Phosphorous	04.04.01.07	Grain flour (soybean flour only) and derived products	1600-3700 mg/kg
	14.06	Solid beverages	1960-7040 mg/kg
Others			
L-Lysine	06.02	Rice and derived products (rice, rice vermicelli, rice cake)	1-2 g/kg
	06.03	Wheat flour derived products	1-2 g/kg
	06.04	Grain flour (Including soybean flour) and derived products (soybean flour only)	1-2 g/kg
	07.01	Bread	1-2 g/kg
Taurine	01.03.02	Modified milk powder	0.3-0.5 g/kg
	04.04.01.07	Grain flour (soybean flour only) and derived products	0.3-0.5 g/kg
	04.04.01.08	Soybean	0.06-0.1 g/kg
	14.03.01	Milk containing beverages	0.1-0.5 g/kg
	14.04.02.01	Beverage for special uses	0.1-0.5 g/kg
	14.04.02.02	Flavored beverages	0.4-0.6 g/kg
	14.06	Solid beverages	1.1-1.4 g/kg
L-carnitine	01.03.02	Modified milk powder (excluding children usage)	300-400 mg
		Modified milk powder (for children only)	50-150 mg/kg
	14.02.03	Fruit and vegetable (pulp) juices	600-3000 mg/kg
	14.03.01	Milk containing drinks	600-3000 mg/kg
	14.04.02.01	Products with special uses (for sports beverages only)	100-1000 mg/kg
	14.04.02.02	Flavored beverages	600-3000 mg/kg
	14.06	Solid beverages	6000-30000 mg/kg
γ-linoleic acid	01.03.02	Modified milk powder and modified cream powder (including flavored milk powder and flavored cream powder)	20-50 g/kg
	02.01.01.01	Vegetable oil	20-50 g/kg
	14.0	Beverages (excluding packaged drinking water mentioned in 14.01)	20-50 g/kg
Lutein	01.03.02	Modified milk powder (for children only, measurements of liquids will be done after dilution)	1620-2700 µg/kg
Oligofructose	01.03.02	Modified milk powder (For children and pregnant women)	≤64.5 g/kg
1,3-dioleoyl-2-palmitoyl-glycerol	01.03.02	Modified milk powder (for children only, measurements of liquids will be done after dilution)	24-96 g/kg
Arachidonic acid (AA or ARA)	01.03.02	Modified milk powder (for children only)	≤1% (percentage of total fatty acids)
DHA	01.03.02	Modified milk powder (for children only)	≤0.5% (percentage of total fatty acids)
		Modified milk powder for pregnant women only)	300-1000 mg/kg
Lactorferrin	01.01.03	Modified milk products	≤1.0 g/kg

	01.02.02	Flavored fermented products	≤1.0 g/kg
	14.03.01	Milk containing beverages	≤1.0 g/kg
Calcium casein peptide	06.0	Rice and derived products (rice, rice vermicelli, rice cake) (excluding products covered under 06.01 and 07.0)	≤1.6 g/kg
	14.0	Beverages (Excluding beverages covered in 14.01)	≤1.6 g/kg (Usage can be increased after dilution for solid beverages)
Casein phosphopeptides	01.01.03	Modified milk products	≤1.6 g/kg
	01.02.02	Flavored fermented products	≤1.6 g/kg
	6.0	Rice and derived products (rice, rice vermicelli, rice cake) (excluding products covered under 06.01 and 07.0)	≤1.6 g/kg
	14.0	Beverages (excluding the foodstuffs mentioned in 14.01 and 14.06)	≤1.6 g/kg (Usage can be increased after dilution for solid beverages)
A The use of table A.1 is in accordance to the categorization number and name of product			

Appendix B
Standardized Appendix
List of allowed source of nutritional fortification substances

Nutrients	The source of nutritional compounds
Vitamin A	Retinol acetate (Vitamin A acetate) Retinol palmitate (Vitamin A palmitate) B-carotene
B-carotene	B-carotene
Vitamin D	Ergocalciferol (Vitamin D ₂) Cholecalciferol (Vitamin D ₃)
Vitamin E	d-α tocopherol dl-α tocopherol dl-α tocopheryl acetate dl-α tocopheryl acetate Mixed tocopherol concentrate Vitamin E succinate calcium d-α-tocopherol succinate dl-α-tocopherol succinate
Vitamin K	Phytonadione
Vitamin B ₁	Thiamine hydrochloride Thiamine mononitrate
Vitamin B ₂	Riboflavin Riboflavin-5'-phosphate
Vitamin B ₆	Pyridoxine phosphate 5-pyridoxine phosphate
Vitamin B ₁₂	Cyanocobalamine Cyanocobalamine hydrochloride Hydroxocobalamin
Vitamin C	L-ascorbic acid Calcium L-ascorbate Vitamin C magnesium phosphate Sodium L-ascorbate L-ascorbic acid, L-ascorbic acid, potassium -6 - palmitate (ascorbyl palmitate)
Niacin	Niacin Nicotinamide
Folic acid	Folic acid
Pantothenic acid	D-calcium pantothenate D-sodium pantothenate
Biotin	D-biotin
Choline	Choline Choline bitartrate
Inositol	Inositol

Iron	<p> Ferrous sulfate Ferrous gluconate Ferric ammonium citrate Ferrous fumarate Ferric citrate Ferrous lactate Hemin Ferric pyrophosphate Iron porphyrin Ferrous glycine Ferrum redactum Sodium iron EDTA Carbonyl iron Ferrous carbonate Ferrous citrate Ferrous fumarate Ferrous succinate Heme iron Electrolytic iron </p>
Calcium	<p> Calcium Carbonate Calcium gluconate Calcium citrate Lactate L-lactate Dicalcium L-threonate Bisglycinatocalcium Calcium Aspartate Calcium citrate malate Calcium acetate (calcium acetate) Calcium chloride Tricalcium phosphate (phosphate) Vitamin E succinate calcium Glycerol phosphate Calcium oxide Calcium sulfate Bone powder (superfine fresh bone meal) </p>
Zinc	<p> Zinc sulfate Zinc gluconate Zinc glycinate Zinc oxide Zinc lactate Zinc citrate Zinc chloride </p>

	Zinc acetate Zinc carbonate
Selenium	Selenite Selenium Sodium Selenoproteins Se mushroom powder L-Se - methyl selenocysteine selenide carrageenan (only for 14.03.01 milk drinks) selenium yeast (only for 14.03.01 milk drinks)
Magnesium	Magnesium Magnesium chloride Magnesia Magnesium carbonate Magnesium hydrogen phosphate Magnesium gluconate
Copper	Copper sulfate Copper gluconate Copper citrate Copper carbonate
Manganese	Manganese sulfate Manganese chloride Manganese carbonate Manganese citrate Manganese gluconate
Potassium	Potassium gluconate Potassium citrate Potassium dihydrogen phosphate Dipotassium hydrogen phosphate Potassium chloride
Phosphorus	Calcium phosphate Calcium hydrogenphosphate
L-Lysine	L-lysinehydrochloride L-lysine-aspartate
Taurine	Aminoethanesulfonic acid
L-carnite	L-carnitine-L-tartrate L-carnitine
γ-linoelic acid	γ-linoelic acid
Lutein	Lutein (source: marigold)
Oligofructose	Oligofructose (source: chicory)
1,3-dioleoyl-2-palmitoyl-glycerol	1,3-dioleoyl-2-palmitoyl-glycerol
Arachidonic acid (AA or ARA)	Arochidonic acid oil (source: Mortierella alpine)
DHA	DHA single cell oil, Source: schizochytrium sp., Ulkenia amoeboida, crypthecodinium cohnii, tuna oil
Calcium casein peptide	Calcium casein peptide
Casein phosphopeptides	Casein phosphopeptides

Appendix C

Standardized Appendix

List of source of nutritional fortification substances for food for special dietary uses

C.1: List of source of nutritional fortification substances for food for special dietary uses

Nutrients	The source of nutritional compounds
Vitamin A	Retinol acetate (Vitamin A acetate) Retinol palmitate (Vitamin A palmitate) B-carotene All-trans-retinol
Vitamin D	Ergocalciferol (Vitamin D ₂) Cholecalciferol (Vitamin D ₃)
Vitamin E	d-α tocopherol dl-α tocopherol dl-α tocopheryl acetate dl-α tocopheryl acetate Mixed tocopherol concentrate d-α-tocopherol succinate
Vitamin K	Phytonadione
Vitamin B ₁	Thiamine hydrochloride Thiamine mononitrate
Vitamin B ₂	Riboflavin Riboflavin-5'-phosphate
Vitamin B ₆	Pyridoxine phosphate 5-pyridoxine phosphate
Vitamin B ₁₂	Cyanocobalamine Cyanocobalamine hydrochloride
Vitamin C	L-ascorbic acid Sodium L-ascorbate Calcium L-ascorbate L-ascorbic acid, potassium ascorbate -6 – palmitate (ascorbyl palmitate)
Niacin	Niacin Nicotinamide
Folic acid	Folic acid
Pantothenic acid	D-calcium pantothenate D-sodium pantothenate
Biotin	D-biotin
Choline	Choline chloride Choline bitartrate
Inositol	Inositol
Sodium	Sodium bicarbonate Sodium dihydrogen phosphate Sodium citrate Sodium

	Disodium hydrogen phosphate
Potassium	Potassium gluconate Potassium citrate Potassium dihydrogen phosphate Dipotassium hydrogen phosphate Potassium chloride
Copper	Copper sulfate Copper gluconate Copper citrate Copper carbonate
Magnesium	Magnesium sulfate Magnesium chloride Magnesium oxide Magnesium carbonate Magnesium hydrogen phosphate Magnesium gluconate
Iron	Ferrous sulfate Ferrous gluconate Ferric ammonium citrate Ferrous fumarate Ferric citrate Ferric pyrophosphate Sodium iron EDTA (only for food supplement nutritional supplements)
Zinc	Zinc sulfate Zinc gluconate Zinc oxide Zinc lactate Zinc citrate Zinc chloride Zinc acetate
Manganese	Manganese sulfate Manganese chloride Manganese carbonate Manganese citrate Manganese gluconate
Calcium	Calcium carbonate Calcium gluconate Calcium citrate L- calcium lactate Calcium hydrogenphosphate Tricalcium phosphate Calcium phosphate Calcium glycerophosphate Calcium oxide

	Calcium sulfate
Phosphorus	Calcium phosphate Calcium hydrogenphosphate
Iodine	Potassium iodide Sodium iodide Potassium iodate
Selenium	Selenium Sodium selenite
Chromium	Chromium sulfate Chromic chloride
Molybdenum	Sodium molybdate Ammonium molybdate
Taurine	Taurine (aminoethyl sulfonic acid)
L-methionine (L-methionine)	Non animal origin
L-tyrosine	Non animal origin
L-tryptophane	Non-animal origin
L-carnitine	L-carnitine L-carnitine-tartrate
DHA	DHA single cell oil, Source: schizochytrium sp., Ulkenia amoeboida, cryptocodinium cohnii, tuna oil
Arachidonic acid (AA)	Arachidonic acid oil (source: Mortierella alpine)

C.2 Other nutrients and their application amount used in formula for infants and young children

Name	Product number	category	Name of product type	Application amount
Galacto-Oligosaccharides	13.01 13.02.01		Infant formula Infant cereal food supplements	Used alone or in combination, the total amount of these substances cannot exceed 64.5g/kg
Fructo-oligosaccharide				
Polyfructose (contain Fructo-oligosaccharide)				
Raffinose				
Polydextrose	13.01		Infant formula	15.6 g/kg ~ 31.25 g/kg
1,3-dioleoyl-2-palmitoyl-glycerol	13.01.01		Infant formula	32-96 g/kg
	13.01.02		Older infants and infant formula food	24-96 g/kg
	13.01.03		Infant formula for special medical purposes	32-96 g/kg
Lutein	13.01.01		Infant formula	300-2000 µg /kg
	13.01.02		Older infants and infant formula food	1620-4230 µg/kg
	13.01.03		Infant formula for special medical purposes	300-2000 µg/kg
DHA	13.02.01		Infant cereal food supplements	≤1150 mg/kg
Arachidonic acid (AA or ARA)	13.02.01		Infant cereal food supplements	≤2300 mg/kg
Nucleotie Sources including the following compounds: Adenosine-5'-monophosphate Adenosine'-monophosphate disodium salt Cytidine-5'-monophosphate Cytidine-5'-monophosphate disodium salt Guanosine-5'-monophosphate Guanosine-5'-monophosphate disodium salt Inosine-5'-monophosphate Inosine-5'-diphosphoric acid disodium salt Urdine-5'-monophosphate Urdine-5'-monophosphate disodium salt	13.01		Infant formula	0.12-0.58g/kg (in terms of total nucleotides)
Lactoferrin	13.01		Infant formula	≤1.0 g/kg
Casein calcium peptide	13.01		Infant formula	≤3.0 g/kg
	13.02		Infant food supplements	≤3.0 g/kg
Casein phosphopeptides	13.01		Infant formula	≤3.0 g/kg
	13.02		Infant food supplements	≤3.0 g/kg
a. The application amount shown above is limited in powdered infant formula. In the liquid infant formula, the amount should be calculated based on the corresponding dilution factor.				

Appendix D

Information Appendix

Classification of food as carriers of nutritional fortification substances

Food codes	Classification/name
01.0	Milk and dairy products (excluding food for special dietary uses mentioned in 13.0)
01.01	Milk and modified milk
01.01.01	Pure milk (whole milk, partly skimmed, skimmed), including reconstituted milk
01.01.02	Sterilized milk
01.01.03	Modified milk
01.02	Fermented milk
01.02.01	Plain fermented milk (whole milk, partly skimmed, skimmed)
01.02.02	Flavored fermented milk and fruit fermented milk
01.03	Milk powder (including milk powder with sugar added), cream powder and its modified products
01.03.01	Milk powder (whole milk, skimmed, partly skimmed) and cream powder
01.03.02	Modified milk powder and modified cream powder (including flavored milk powder and flavored cream)
01.04	Condensed milk and its modified products
01.04.01	Condensed milk (plain)
01.04.02	Modified condensed milk (including sweet condensed milk, flavored sweet condensed milk and other modified condensed milk manufactured by non-milk ingredients)
01.05	Cream (cream) and similar products
01.06	Cheese and processed cheese
01.07	Ready-to-eat flavored deserts or their pre-made products manufactured by using milk as the main ingredient (excluding ice cream and flavored yogurt)
01.08	Other milk derived products (whey powder, casein powder etc.)
02.0	Fats, oils and emulsified fat products
02.01	Anhydrous fats and oils
02.01.01	Vegetable oils
02.01.01.01	Vegetable oils
02.01.01.02	Hydrogenated vegetable oils
02.01.02	Animal fats (lard, butter, fish oil and other animal fats)
02.01.03	Anhydrous butter, anhydrous cream,
02.02	Water-in-oil or oil-in-water emulsified product
02.02.01	Emulsified products with fat content more than 80%
02.02.01.01	Butter and condensed butter
02.02.01.02	Margarine and similar products (e.g. mixture of butter and margarine)
02.02.02	Emulsified products with fat content less than 80%
02.03	Emulsified products, including the mixed and/or flavored emulsified products (excluding the products mentioned in 02.02)
02.04	Fat-based desserts
02.05	Other fats for fat-based products
03.0	Frozen drinks
03.01	Ice-cream
03.02	Ice-cream
03.03	Flavored ice, popsicles
03.04	Edible ice
03.05	Other frozen drinks
04.0	Fruits, vegetables (including root vegetables), beans, edible mushrooms, algae, nuts, seeds etc.
04.01	Fruits
04.01.01	Fresh fruits

04.01.02	Processed fruits
04.01.02.01	Canned fruits
04.01.02.02	Puree
04.02	Vegetables
04.02.01	Fresh vegetables
04.02.02	Processed vegetables
04.03	Edible mushroom and algae
04.03.01	Fresh edible mushroom and algae
04.03.02	Processed edible mushroom and algae
04.04	Bean products
04.04.01	Non-fermented bean products
04.04.01.01	Tofu (north tofu, south tofu, lactone tofu, frozen tofu)
04.04.01.02	Dried tofu
04.04.01.03	Processed dried tofu
04.04.01.04	Bean curd sticks (bean curd sticks, oily bean curd sheets)
04.04.01.05	New-type bean products (puffed food made from, veg-meat)
04.04.01.06	Cooked bean products
04.04.01.07	Grain flour (soybean flour only) and derived products
04.04.01.08	Soybean
04.04.02	Fermented bean products
04.04.02.01	Pickled bean curd
04.04.02.02	Fermented soya beans and derived products (including natto)
04.04.03	Other bean products
04.05	Nuts and seeds
04.05.01	Fresh nuts and seeds
04.05.02	Processed Nuts and seeds
05.0	Cocoa products, chocolate and derived products (chocolate substitutes) and candy
05.01	Cocoa products, chocolate and derived products including chocolate substitutes
05.01.01	Cocoa products using cocoa as the main ingredient (cocoa butter, powder, spread, paste, filling)
05.01.02	Chocolate and derived products, cocoa products (excluding cocoa products mentioned in 05.01.01)
05.01.03	Chocolate substitutes and chocolate similar products made from cocoa substitutes
05.02	Candy
05.02.01	Hard candy
05.02.02	Hard bonbons
05.03	Candy and chocolate coated products
05.04	Coating of candy and chocolate products
06.0	Decorative candy (e.g. garnish, cake decoration), topping (non-fruit materials) and syrup
06.01	Cereals and derived products, including rice, flour, coarse grain, root crops, pulse and corn starch (excluding the Bakery products mentioned in 07.0)
06.02	Cereals
06.02.01	Rice and derived products (rice, rice vermicelli, rice cake)
06.02.02	Rice
06.02.03	Rice products
06.02.04	Rice flour (including glutinous rice flour)
06.03	Rice vermicelli
06.03.01	Wheat flour and derived products
06.03.02	Other special-purpose flour
06.04	Batter (e.g. batter used in cooking fish and poultry), fried coating powder, frying powder
06.04.01	Coarse grain flour
06.04.02	Coarse grained flour derived products
06.04.02.01	Caned mixed congee
06.04.02.02	Other coarse grain derived products
06.05	Starch and derived products
06.05.01	Edible starch

06.05.02	Starch derived products
06.05.02.01	Vermicelli, rice noodle
06.05.02.02	Prawn chips
06.05.02.03	Lotus root starch
06.05.02.04	Tapioca ball
06.06	Ready-to-eat cereals, including oats and rolled oats
06.07	Instant rice or instant noodle
06.08	Frozen rice or noodle
06.09	Cereal and starch dessert (e.g. rice pudding, cassava pudding)
06.10	Fillings made from cereals
07.0	Bakery products
07.01	Bread
07.02	Pastry
07.02.01	Chinese pastry
07.02.02	Western pastry
07.02.03	Moon cake
07.02.04	Topping
07.03	Biscuit
07.03.01	Sandwich and decorative crackers
07.03.02	Waffles
07.03.03	Egg rolls
07.03.04	Other biscuits
07.04	Fillings of bakery products
07.05	Other bakery products
08.0	Meat and meat products
08.01	Raw fresh meat
08.02	Pre-made meat products
08.03	Cooked meat products
08.03.01	Stewed meat products
08.03.02	Smoked, barbecued, roasted meat
08.03.03	Deep-fried meat
08.03.04	Western ham (roasted, smoked, steamed ham)
08.03.05	Sausages
08.03.06	Fermented meat products
08.03.07	Cooked dried meat products
08.03.07.01	Dried meat floss
08.03.07.02	Dried meat
08.03.07.03	Fried meat crisps
08.03.08	Canned meat
08.03.09	Edible sausage casings
08.03.10	Other meat and derived products
09.0	Aquatic products and derived products (including fish, crustacean, shellfish, mollusk, echinoderms and derived products)
09.01	Fresh aquatic products
09.02	Frozen aquatic and derived products
09.03	Pre-made aquatic products (semi-manufactured products)
09.04	Cooked dried aquatic products (ready-to-eat)
09.05	Canned aquatic products
09.06	Other aquatic products and derived products
10.0	Eggs and derived products
10.01	Fresh eggs
10.02	Processed eggs (the physical properties are not changed)
10.03	Egg product (the physical properties are changed)
10.03.01	Dehydrated egg products (e.g. egg white powder, egg yolk powder, egg white tablet)
10.03.02	Thermo-coagulated egg products (egg cheese, thousand-year egg sausages)
10.03.03	Frozen egg products

10.03.04	Liquid eggs
10.04	Other egg products
11.0	Sugar, including honey
11.01	Sugar
11.01.01	Sugar and sugar products (e.g. sucrose, beet sugar, crystal sugar, fructose etc.)
11.01.02	Other sugar and syrup (brown sugar, brown granulated sugar, maple syrup)
11.02	Starch sugar (fructose, glucose, maltose; partially invert sugar, including malaises)
11.03	Honey and pollen
11.04	Table sugar
11.05	Flavored syrup
11.06	Other sweetener
12.0	Condiment
12.01	Salt and salt substitutes
12.02	Flavor enhancer
12.03	Vinegar
12.04	Soy sauce
12.05	Sauces and derived products
12.06	--
12.07	Cooking wine and derived products
12.08	--
12.09	Spices
12.10	Mixed seasonings
12.10.01	Solid mixed seasonings
12.10.02	Semi-solid mixed seasonings
12.10.03	Liquid mixed seasoning (excluding 12.03, 12.04)
12.11	Other seasonings
13.0	Food for special dietary uses
13.01	Formula for infant and young children
13.01.01	Infant formula
13.01.02	Formula for older infant and young children
13.01.03	Infant formula for special medical purpose
13.02	Complementary foods for infants and young children
13.02.01	Infant cereal food supplements
13.02.02	Canned infant complementary food
13.03	Formula for special medical purpose (excluding the products mentioned in 13.01)
13.04	Other foods for special dietary uses except for the products mentioned in 13.01-13.03)
13.05	Food for special dietary uses (excluding products mentioned in 13.01-13.04)
14.0	Beverages
14.01	Packaged drinking water
14.02	Fruit and vegetable juices
14.02.01	Fruit and vegetable juices (squash)
14.02.02	Condensed fruit and vegetable juice (squash)
14.02.03	Fruit and vegetable (pulp) juices
14.03	Protein containing drinks
14.03.01	Milk containing drinks
14.03.02	Vegetable protein containing drinks
14.03.03	Compound protein beverages
14.04	Water-based flavored beverages
14.04.01	Carbonated drinks
14.04.02	Non-carbonated beverages
14.04.02.01	Beverages for special purpose (including sports drinks, nutritional drinks etc.)
14.04.02.02	Flavored beverages (fruit flavored drinks, flavored milk, tea flavored drinks and other flavored beverages)
14.05	Tea, coffee, botanical beverages
14.05.01	Tea
14.05.02	Coffee

14.05.03	Botanical beverages (excluding fruit and vegetable juices)
14.06	Solid beverages
14.06.01	Fruit flavored solid beverages
14.06.02	Protein containing solid beverages
14.06.03	Instant coffee
14.06.04	Other kinds of solid beverages
14.07	--
14.08	Other beverages
15.0	Liquors
15.01	Distilled spirits
15.02	Mixed liquors
15.03	Fermented liquors
16.0	Others (excluding those mentioned in 01.0-15.0)
16.01	Jelly
16.02	Tea, coffee
16.03	Collagen casings
16.04	Yeast products
16.05	Fried food
16.06	Puffing foods
16.07	Others

GB 22570-2014 Complementary Food Supplement



National Standards of People's Republic of China

GB 22570-2014

**National Food Safety Standard
Complementary Food Supplement**

Issued on: 2010-04-29

Implemented on: 2014-11-01

Issued by National Health and Family Planning Commission

Foreword

This standard supersedes GB/T 22570-2008 Complementary Food Supplement National Standard

Compared with GB/T 22570-2008, the following changes have been made to the Standard:

- Standard name are modified;
- Standard structure are modified;
- Technical demanding are modified;
- Labeling requirements are modified;
- Delete Annex A and B in the previous standard.

National Standard for Food Safety

Complementary Food Supplement

1. Scope

This Standard applies to complementary food supplement for infants from 6 months to 36 months, and children from 37 months to 60 months.

2. Terms and Definitions

2.1 Food supplement:

Food supplement refers to additional foods to meet nutritional needs for more than 6-month-old infants and young children who are continuing to breast feed. The foods come by both family prepared and factory production

Refer to persons of 0 ~ 12 months old.

2.2 Complementary food supplement:

A kind of supplements full of micronutrients (vitamins and minerals), with or without the food matrix and other complementary food, add in the instant food supplement for infants from 6 months to 36 month old, and young children from 37 months to 60 months old as well. Now the common types are: complementary feed nutrient supplements, complementary micronutrient supplements and complementary micronutrients reagents.

2.2.1 Complementary feed nutrient supplements

Food matrix based on one or more of soybean, soy protein, milk and milk protein products, made from complementary feed nutrient supplements by adding micronutrients and (or) other supplements. Food shape can be powder, granular or semi-solid, and the food matrix could provide part of high quality protein.

2.2.2 Complementary micronutrient supplement tablets

Food matrix based on one or more of soybean, soy protein, milk and milk protein products, made from flake complementary food supplements by adding micronutrients and (or) other supplements. The supplements are breakable or easily dispersed.

2.2.3 Complementary micronutrients supplement sprinkles

Powdered or granular complementary nutritional supplements mixed by multiple-micronutrient, may without food matrix.

3. Technical Requirements

3.1 Daily recommended amount of complementary food supplement

Complementary feed nutrient supplements are of 10.0g ~ 20.0g, complementary micronutrient supplement tablets are of 1.5g ~ 3.0g, and complementary micronutrients sprinkles are of 0.8g ~ 2.0g

3.2 Requirements for raw materials

3.2.1 Food matrix shall be take instant edible food as raw materials, and its quality shall meet the relevant standards and (or) regulations

3.2.2 Soybean and soybean-based products shall be under the process of high-temperature treatment to eliminate the anti-nutritional factors such as trypsin inhibitors, etc.

3.2.3 Food accessories shall meet the relevant standards and (or) regulations

3.3 Sensory requirement

The color, taste, smell and organization status of complementary food supplements shall conform to the relevant product specifications, and shall be no visible extraneous matter

3.4 Essential components

In complementary foods supplement, protein content shall be no less than 25g/100g, test method shall followed by GB 5009.5, and protein content calculation shall be Nitrogen (N) * 6.25

Other nutrients content in complementary food supplement shall convert into daily content and be consistent with the requirements of Table 1

Table 1: Required components

Nutrition	Daily content			Test method
	6 – 12 months	13 – 36 months	37 – 60 months	
Calcium/(mg) ^a	120 ~ 240	180 ~ 360	180 ~ 360	GB 5413.21
Iron/(mg)	3.0 ~ 9.0	3.6 ~ 10.8	3.6 ~ 10.8	GB 5413.21
Zinc/(mg)	2.0 ~ 6.0	2.0 ~ 7.0	2.0 ~ 7.0	GB 5413.21
Vitamin A/(μg RE) ^b	120 ~ 360	150 ~ 450	150 ~ 450	GB 5413.9
Vitamin D/(μg) ^c	3.0 ~ 9.0	3.0 ~ 9.0	3.0 ~ 9.0	GB 5413.9
Vitamin B ₁ /(mg) ≥	0.12	0.24	0.24	GB 5413.11
Vitamin B ₂ /(mg) ≥	0.2	0.24	0.24	GB 5413.12
a. Applies only to food supplementary nutrition in supplement foods b. RE equivalent to retinol. 1 μg RE = 3.33 IU, Vitamin A = 1 μg all-TRANS retinol (Vitamin A). Vitamin A only includes preformed retinol, and when in calculating and claiming vitamin A activity shall exclude any component of β-carotene c. Calciferol, 1 μg vitamin D = 40 IU vitamin D				

3.5 Optional components

In addition to the essential components in 3.4, if one or more nutrients listed in Table 2 can be selected to add or claimed on label, whereas the content of such nutrients converted in to daily content shall meet the specification of Table 2

Table 2: Optional components

Nutrition	Daily content			Test method
	6 – 12 months	13 – 36 months	37 – 60 months	
Calcium/(mg) ^a	120 ~ 240	180 ~ 360	180 ~ 360	GB 5413.21
Vitamin K ₁ /(μg)	3.0 ~ 9.0	4.5 ~ 13.5	4.5 ~ 13.5	GB 5413.10
Hydrochloric acid (Nicotinamide) /(mg) ^b	1.2 ~ 6.0	2.4 ~ 6.0	2.4 ~ 6.0	GB 5413.15
Vitamin B ₆ /(mg) ≥	0.12	0.20	0.20	GB 5413.13
Folic acid/(μg)	18.8 ~ 150	35.3 ~ 150	35.3 ~ 150	GB 5413.16
Vitamin B ₁₂ /(μg) ≥	0.2	0.36	0.36	GB 5413.14
Pantothenic acid/(mg) ≥	0.72	0.8	0.8	GB 5413.17
Choline/(mg) ≥	60	80	80	GB 5413.20
Biotin/(μg) ≥	2.4	3.2	3.2	GB 5413.19
Vitamin C/(mg) ≥	20	24	24	GB 5413.18
22 decosahexaenoic acid/(mg)	30 ~ 90	30 ~ 90	30 ~ 90	GB 5413.27
a. Applicable to complementary nutrients sub agent and food supplement nutrition supplements				
b. Niacin does not include precursors				

3.6 Limits of contaminants

The limit of contaminants shall meet the specification of Table 3

Table 3: Indices of Contaminants

Item	Index	Test Method
Lead/(mg/kg) ≤	0.5	GB 5009.12
Total arsenic ≤	0.5	GB/T 5009.11
Nitrate (based on NaNO ₃) ^a ≤	100	GB 5009.33
Nitrate (based on NaNO ₂) ^b ≤	2	GB 5009.33
a. Not applicable for products with fruits and vegetables		
b. Only applicable to milk-based infant formulas		

3.7 Limit of mycotoxin

The limit of mycotoxin shall meet the specification of Table 4

Table 4: Limit of Mycotoxin

Item	Index	Test Method
Aflatoxin M ₁ ^a (μg/kg) ≤	0.5	GB 5009.24
Aflatoxin B ₁ ^b (μg/kg) ≤	0.5	
a. Aflatoxin M1 only for milk-based products		
b. Aflatoxin B1 only for cereal, peanuts and soybean-based products		

3.8 Limit of microorganisms

The limit of microorganisms shall meet the specification of Table 5

Table 5: Limit of Microorganisms

Item	Sampling plan ^a and limit (Unless specified otherwise, it shall be expressed in cfu/g or cfu/ml)				Test Method
	n	c	m	M	
Total plate count	5	2	1000	10000	GB 4789.2
Coliform bacteria	5	2	10	100	GB 4789.3 plate count method
Salmonella	5	0	0/25g	-	GB 4789.4
a. Sample analysis and handling shall comply with GB 4789.1					

3.9 Food additives and nutrition fortifiers

3.9.1 The use of food additives shall conform to GB 2760 requirements

3.9.2 The use of nutrition fortifiers shall conform to GB 14880 requirements, among which the ethyl diamine tetra sodium acid daily dosage in terms of iron shall not exceed 2.8mg

3.9.3 Quality of food additives and nutrition fortifiers shall be consistent with relevant safety standards and requirements

3.10 Urease activity

The urease activity of products containing soy component shall be consistent with the provisions of Table 6

Table 6: Indices of Urease Activity

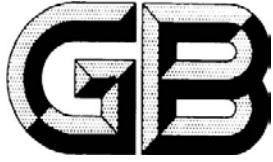
Item	Index	Test Method
Qualitative determination of urease activity	Negative	GB 5413.31

4. Labels

4.1 Product labels shall comply with the provisions of GB 13432, and mark 'food supplements' and (or) the corresponding category of 'complementary feed nutrient supplements', 'complementary micronutrient supplement tablets' and 'Complementary micronutrients supplement sprinkles'

4.2 Label shall be identified by the different month, and mark 'this product add micronutrients, please pay attention to eat with other similar products'. If the products face to the infants from 6 ~ 36-month-old, the label shall be marked 'this product is not a substitute for breast milk and infant food aid'

GB 29922-2013 General Rule on Formulated Foods for Special Medical Purposes



National Standards of People's Republic of China

GB 29922-2013

National Food Safety Standard
General Rule on Formulated Foods for Special Medical Purposes

Issued on: 2013-12-26

Implemented on: 2014-07-01

Issued by National Health and Family Planning Commission

National Standard for Food Safety

General Rule on Formulated Foods for Special Medical Purposes

1. Scope

This standard is applicable to formulated foods for special medical purposes which are suitable for people above 1 year.

2. Terms and definitions

2.1 Formulated foods for special medical purposes

As especially formulated foods that are produced to meet the special requirements for nutrient or meals of people who suffer from eating limitation, disorder of digestion and absorption, metabolic disorders or special disease state, these products shall be eaten individually or with other foods under the guidance of doctors or clinical dietitians.

2.1.1 Full nutritional formula foods

Formulated foods for special medical purposes that can meet the requirements of target groups for nutrition as a single nutrition source.

2.1.2 Specific full nutritional formula foods

Formulated foods for special medical purposes that can meet the requirements of target groups for nutrition under the condition of specified diseases or medical conditions as a single nutrition source.

2.1.3 None-full-nutritional formula foods

Formulated foods for special medical purposes that can meet the requirements of target groups for nutrition and are not suitable to be used as a single nutrition source.

3 Technical requirements

3.1 Basic requirements

Formulated foods for special medical purposes shall be based on the medical and/or nutritional research results with scientifically verified security and clinical effects. Their production condition shall also be in accordance with relevant national regulations.

3.2 Requirements for materials

Formulated foods for special medical purposes shall be made of raw materials which meet the requirements of relevant standards and/or regulations. Those which jeopardize consumers' health shall be forbidden.

3.3 Sensory requirements

The colors, tastes, smells, textures and dissolving ability of formulated foods for special medical purposes shall be in accordance with their characteristics and include no visible extraneous matters.

3.4 Nutritional ingredients

3.4.1 Full nutritional formula foods suitable for people aged from 1 to 10 years

3.4.1.1 Full nutritional formula foods suitable for people aged from 1 to 10 years shall contain 250 kJ (60 kcal) of energy or more in every 100 mL of their liquid products or reconstituted foods under their immediately-edible condition, or in every 100 g of their immediately edible non-liquid products. To calculate the energy, we can multiply the content of protein, fat and carbohydrate in every 100 mL or 100 g of products by their respective energy coefficients, i.e. 17 kJ/g, 37 kJ/g and 17 kJ/g (energy coefficients of dietary fiber, to be calculated with 50% of carbohydrate energy coefficient). Their sums are the values of kJ/100mL or kJ/100g, which can be divided by 4.184 to be the values of kcal/100mL or kcal/100g.

3.4.1.2 Full nutritional formula foods suitable for people aged from 1 to 10 years shall contain 0.5g/100kJ (2g/100kcal) of protein or more, in which quality protein shall account for 50% or more. Please see GB 5009.5 for the way to test protein.

3.4.1.3 In the full nutritional formula foods suitable for people aged from 1 to 10 years, the energy supply ratio of linoleic acid shall be 2.5% or more and that of α -linolenic acid shall be 0.4% or more. Please see GB 5413.27 for the way to test aliphatic acid.

3.4.1.4 In the full nutritional formula foods suitable for people aged from 1 to 10 years, the content of vitamins and mineral substances shall be in accordance with Table 1.

3.4.1.5 Except for the ingredients specified in Table 1, if one or more ingredients in Table 2 are added or shown in the products, their content shall be in accordance with Table 2.

Table 1 Vitamin and mineral substance index (people aged from 1 to 10 years)

Nutrient	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Vitamin A/(μ g RE) ^a	17.9	53.8	75	225	GB 5413.9 or GB/T 5009.82
Vitamin D/(μ g) ^b	0.25	0.75	1.05	3.14	GB 5413.9
Vitamin E/(mg α -TE) ^c	0.15	N.S. ^e	0.63	N.S.	GB 5413.9 or GB/T 5009.82
Vitamin K1 /(μ g)	1	N.S.	4	N.S.	GB 5413.10 or GB/T 5009.158
Vitamin B1/(mg)	0.01	N.S.	0.05	N.S.	GB 5413.11 or GB/T 5009.84
Vitamin B2/(mg)	0.01	N.S.	0.05	N.S.	GB 5413.12
Vitamin B6 /(mg)	0.01	N.S.	0.05	N.S.	GB 5413.13 or GB/T 5009.154

Nutrient	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Vitamin B12 /(µg)	0.04	N.S.	0.17	N.S.	GB 5413.14
Nicotinic acid (nicotinamide) /(mg) ^d	0.11	N.S.	0.46	N.S.	GB 5413.15 or GB/T 5009.89
Folic acid/(µg)	1.0	N.S.	4	N.S.	GB 5413.16 or GB/T 5009.211
Pantothenic acid/(mg)	0.07	N.S.	0.29	N.S.	GB 5413.17 or GB/T 5009.210
Vitamin C/(mg)	1.8	N.S.	7.5	N.S.	GB 5413.18
Biotin/(µg)	0.4	N.S.	1.7	N.S.	GB 5413.19
Sodium/(mg)	5	20	21	84	GB 5413.21 or GB/T 5009.91
Potassium/(mg)	18	69	75	289	GB 5413.21 or GB/T 5009.91
Copper/(µg)	7	35	29	146	GB 5413.21 or GB/T 5009.13
Magnesium/(mg)	1.4	N.S.	5.9	N.S.	GB 5413.21 or GB/T 5009.90
Iron/(mg)	0.25	0.5	1.05	2.09	GB 5413.21 or GB/T 5009.90
Zinc /(mg)	0.1	0.4	0.4	1.5	GB 5413.21 or GB/T 5009.14
Manganese/(µg)	0.3	24	11	100.4	GB 5413.21 or GB/T 5009.90
Calcium/(mg)	17	N.S.	71	N.S.	GB 5413.21 or GB/T 5009.92
Phosphorus/(mg)	8.3	46.2	34.7	193.5	GB 5413.22 or GB/T 5009.87
Iodine/(µg)	1.4	N.S.	5.9	N.S.	GB 5413.23
Chlorine/(mg)	N.S.	52	N.S.	218	GB 5413.24
Selenium/(µg)	0.5	2.9	2.0	12.0	GB 5009.93

^a RE is the retinol equivalent. 1µg RE =3.33 IU of Vitamin A=1µg of alltrans retinol (Vitamin A). Vitamin A only includes preformed retinol. No carotenoid ingredients are included when the activity of Vitamin A is calculated and claimed.

^b Calciferol, 1µg of Vitamin D=40 IU of Vitamin D

^c 1 mgα-TE (α-tocopherol equivalent)=1 mg of d-α-tocopherol

^d Nicotinic acid doesn't include the precursor form.

^e N.S. means no specification.

Table 2 Selectable ingredient index (people aged from 1 to 10 years)

Selectable ingredient ^a	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Chromium/(μg)	0.4	5.7	1.8	24	GB/T 5009.123
Molybdenum/(μg)	1.2	5.7	5	24	—
Fluorine/(mg)	N.S. ^b	0.05	N.S.	0.2	GB/T 5009.18
Choline/(mg)	1.7	19.1	7.1	80	GB/T5413.20
Inositol/(mg)	1	9.5	4.2	39.7	GB 5413.25
Taurine/(mg)	N.S.	3.1	N.S.	13	GB 5413.26 or GB/T 5009.169
L-carnitine/(mg)	0.3	N.S.	1.3	N.S.	—
Docosahexaenoic Acid(%Total fatty acid c)	N.S.	0.5	N.S.	0.5	GB 5413.267 or GB/T 5009.1698
Eicosatetraenoic Acid(%Total fatty acid c)	N.S.	1	N.S.	1	GB 5413.27
Nucleotide/(mg)	0.5	N.S.	2	N.S.	—
Dietary fiber/(g)	N.S.	0.7	N.S.	2.7	GB 5413.6 or GB/T 5009.88
^a Compound origins of fluorine are sodium fluoride and potassium fluoride. Please see the allowed origin in C.2 of GB 14880 for nucleotide and dietary fiber. For other compound origins, please see GB 14880.					
^b N.S. means no specification.					

3.4.2 Full nutritional formula foods suitable for people aged above 10 years

3.4.2.1 Full nutritional formula foods suitable for people aged above 10 years shall contain 295 kJ (70 kcal) of energy or more in every 100 mL of their liquid products or reconstituted foods under their immediately-edible condition, or in every 100 g of their immediately edible non-liquid products. To calculate the energy, we can multiply the content of protein, fat and carbohydrate in every 100 mL or 100 g of products by their respective energy coefficients, i.e. 17 kJ/g, 37 kJ/g and 17 kJ/g (energy coefficients of dietary fiber, to be calculated with 50% of carbohydrate energy coefficient). Their sums are the values of kJ/100mL or kJ/100g, which can be divided by 4.184 to be the values of kcal/100mL or kcal/100g.

3.4.2.2 Full nutritional formula foods suitable for people aged from 1 to 10 years shall contain 0.7g/100kJ (3g/100kcal) of protein or more, in which quality protein shall account for 50% or more. Please see GB 5009.5 for the way to test protein.

3.4.2.3 In the full nutritional formula foods suitable for people aged above 10 years, the energy supply ratio of linoleic acid shall be 2.0% or more and that of-linolenic acid shall be 0.5% or more. Please see GB 5413.27 for the way to test aliphatic acid.

3.4.2.4 In the full nutritional formula foods suitable for people aged above 10 years, the content of vitamins and mineral substances shall be in accordance with Table 3.

3.4.2.5 Except for the ingredients specified in Table 3, if one or more ingredients in Table 4 are added or shown in the products, their content shall be in accordance with Table 4.

Table 3 Vitamin and mineral substance index (people aged above 10 years)

Nutrient	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Vitamin A/(μg RE) ^a	9.3	53.8	39.0	225	GB 5413.9 or GB/T 5009.82
Vitamin D/(μg) ^b	0.19	0.75	0.80	3.14	GB 5413.9
Vitamin E/(mg α-TE) ^c	0.15	N.S. ^e	0.80	N.S.	GB 5413.9 or GB/T 5009.82
Vitamin K1/(μg)	1	N.S.	4.40	N.S.	GB 5413.10 or GB/T 5009.158
Vitamin B1/(mg)	0.02	N.S.	0.07	N.S.	GB 5413.11 or GB/T 5009.84
Vitamin B2/(mg)	0.02	N.S.	0.07	N.S.	GB 5413.12
Vitamin B6/(mg)	0.02	N.S.	0.07	N.S.	GB 5413.13 or GB/T 5009.154
Vitamin B12/(μg)	0.043	N.S.	0.13	N.S.	GB 5413.14
Nicotinic acid (nicotinamide)/(mg) ^d	0.05	N.S.	0.20	N.S.	GB 5413.15 or GB/T 5009.89
Folic acid/(μg)	5.3	N.S.	22.2	N.S.	GB 5413.16 or GB/T 5009.211
Pantothenic acid/(mg)	0.07	N.S.	0.29	N.S.	GB 5413.17 or GB/T 5009.210
Vitamin C/(mg)	1.3	N.S.	5.6	N.S.	GB 5413.18
Biotin/(μg)	0.5	N.S.	2.2	N.S.	GB 5413.19
Sodium/(mg)	20	N.S.	83	N.S.	GB 5413.21 or GB/T 5009.91
Potassium/(mg)	27	N.S.	111	N.S.	GB 5413.21 or GB/T 5009.91
Copper/(μg)	11	120	44	500	GB 5413.21 or GB/T 5009.13

Nutrient	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Magnesium/(mg)	4.4	N.S.	18.3	N.S.	GB 5413.21 or GB/T 5009.90
Iron/(mg)	0.20	0.55	0.83	2.30	GB 5413.21 or GB/T 5009.90
Zinc /(mg)	0.1	0.45	0.4	2.2	GB 5413.21 or GB/T 5009.14
Manganese/(µg)	6.0	2146.0	25.0	611.0	GB 5413.21 or GB/T 5009.90
Calcium/(mg)	13	N.S.	56	N.S.	GB 5413.21 or GB/T 5009.92
Phosphorus/(mg)	9.6	N.S.	40.0	N.S.	GB 5413.22 or GB/T 5009.87
Iodine/(µg)	1.6	N.S.	6.7	N.S.	GB 5413.23
Chlorine/(mg)	N.S.	52	N.S.	218	GB 5413.24
Selenium/(µg)	0.8	5.3	3.3	22.0	GB 5009.93

^a RE is the retinol equivalent. 1µg RE =3.33 IU of Vitamin A=1µg of alltrans retinol (Vitamin A). Vitamin A only includes preformed retinol. No carotenoid ingredients are included when the activity of Vitamin A is calculated and claimed.

^b Calciferol, 1µg of Vitamin D=40 IU of Vitamin D

^c 1 mgα-TE (α-tocopherol equivalent) =1 mg of d-α-tocopherol

^d Nicotinic acid doesn't include the precursor form.

^e N.S. means no specification.

Table 4 Selectable ingredient index (people aged above 10 years)

Selectable ingredient ^a	Every 100kJ		Every 100kca		Test method
	Minimum	Maximum	Minimum	Maximum	
Chromium/(μg)	0.4	13.3	1.8	55.6	GB/T 5009.123
Molybdenum/(μg)	1.3	12.0	5.6	2450.0	—
Fluorine/(mg)	N.S. ^b	0.05	N.S.	0.20	GB/T 5009.18
Choline/(mg)	5.3	39.8	22.2	166.7	GB/T5413.20
Inositol/(mg)	1.0	33.5	4.2	140.0	GB 5413.25
Taurine/(mg)	N.S.	4.8	N.S.	20.0	GB 5413.26 or GB/T 5009.169
L-carnitine/(mg)	0.3	N.S.	1.3	N.S.	—
Nucleotide/(mg)	0.5	N.S.	2.0	N.S.	—
Dietary fiber/(g)	N.S.	0.7	N.S.	2.7	GB 5413.6 or GB/T 5009.88

^a Compound origins of fluorine are sodium fluoride and potassium fluoride. Please see the allowed origin in C.2 of GB 14880 for nucleotide and dietary fiber. For other compound origins, please see GB 14880.

^b N.S. means no specification.

3.4.3 Specific full nutritional formula foods

The energy and nutrient content of specific full nutritional formula foods shall be based on the full nutritional formula foods in 3.4.1 or 3.4.2, but can be properly adjusted according to the special requirements of diseases or medical condition for nutrients to meet the nutrition requirements of target groups. Please see the common specific full nutritional formula foods in Appendix A.

3.4.4 None-full-nutritional formula foods

The common none-full-nutritional formula foods include nutrient ingredients, electrolyte formula, thickening ingredients, liquid formula and formula of amino acid metabolism disorder. Technical indicators of all products shall be in accordance with the requirements of Table 5. Unable to satisfy the nutrition requirements of target groups as a single nutrition source, such products shall be consumed with other foods. So their nutrient content shall not be required. None-full-nutritional formula foods shall be consumed in accordance with the special condition or requirements of individual patients under the guidance of doctors or clinical dietitians.

Table 5 Key technical requirements for common none-full-nutritional formula foods

Product category		Main technical requirements for formulas
Nutrient ingredients	Protein (amino acid) ingredients	1. It comprises protein and/or amino acid; 2. One or more amino acids, protein hydrolysates, peptides or quality integral protein can be chosen as protein sources.
	Fat (aliphatic acid) ingredients	1. It comprises fat and/or aliphatic acid; 2. LCT, MCT or other fat (aliphatic acid) origins in accordance with laws and regulations can be chosen.
	Carbohydrate ingredients	1. It comprises carbohydrate; 2. Monosaccharide, disaccharide, oligosaccharide or polysaccharide, maltodextrin, glucose polymers or other raw materials in accordance with laws and regulations shall be chosen as the origins of carbohydrate.
Electrolyte formula		1. It shall be based on carbohydrate; 2. An appropriate amount of electrolyte shall be added.
Thickening ingredients		1. It shall be based on carbohydrate; 2. One or more thickeners shall be added; 3. Dietary fibers can be added.
Liquid formula		1. It shall be based on carbohydrate and protein; 2. Various vitamins and mineral substances can be added; 3. Dietary fibers can be added.
Formula of amino acid metabolism disorder		1. It is mainly made of amino acid but contains little amino acid which is related to amino acid. See the amino acid types and content requirements limited in common amino acid metabolism disorder formula foods at Table 6. 2. An appropriate amount of fat, carbohydrate, vitamins, mineral substance and/or other substances; 3. It shall meet patients' requirement for some of the vitamins and mineral substances while meeting their requirement for some of the protein (amino acid).

Table 6 Amino acid types and content limited in common amino acid metabolism disorder formula foods

Common amino acid metabolism disorder	Amino acid types limited in formulated foods	Amino acid types limited in formulated foods mg/g
Phenylketonuria	Phenylalanine	≤1.5
Maple syrup urine disease	Leucine, isoleucine, valine	≤1.5 ^a
Propionic acidemia/methylmalonic acidemia	Methionine, threonine, valine	≤1.5 ^a
	Isoleucine	≤1.5
Tyrosinemia	Phenylalanine, tyrosine	≤1.5 ^a
homocystinuria	Methionine	≤1.5
Glutaric Acidemia Type I	Lysine	≤1.5
	Tryptophan	≤18
Isovaleric acidemia	Leucine	≤1.5
Urea cycle disorders	Non-essential amino acid (alanine, arginine, aspartic acid, asparaginate, glutamic acid, glutamine, glycine, proline, serine)	≤1.5 ^a
^a means content of single amino acids		

3.5 Limited quantity of pollutants

Limited quantity of pollutants shall be in accordance with Table 7.

Table 7 Limited quantity of pollutants (to be calculated with solid products)

Items	Index		Test method
Lead/(mg/kg) ≤	0.15	0.5 ^a	GB 5009.12
Nitrate(calculated by NaNO3) /≤ (mg/kg) ^b	100		GB 5009.33
Nitrite(calculated by NaNO2)≤ /(mg/kg) ^c	2		
^a Products only suitable for people aged above 10 years.			
^b Not suitable for products containing vegetables and fruits.			
^c Only suitable for dairy-based products (containing no soybean)			

3.6 Limited quantity of mycotoxin

Mycotoxin shall be in accordance with Table 8.

Table 8 Limited quantity of mycotoxin (to be calculated with solid products)

Items	Index	Test method
Aflatoxin M1 (μg/kg) a ≤	0.5	GB 5009..24
Aflatoxin B1 (μg/kg) b ≤	0.5	
a Only suitable for dairy and lactoprotein products		
b Only suitable for soybean and soybean protein products		

3.7 Limited quantity of microorganism

Limited quantity of microorganism in solid formulated foods for special medical purposes shall be in accordance with Table 9. Microbiological indicators for liquid formulated foods for special medical purposes shall be in accordance with commercial standard of sterility and be tested according to GB/T 4789.26.

Table 9 Limited quantity of microorganism

Items	Sampling plana quantity limit(demonstrated with CFU/g if not being specified)				Test method
	n	c	m	M	
Aerobic bacterial count ^{b,c}	5	2	1000	10000	GB 4789.2
Coliform	5	2	10	100	GB4789.3 plate counting method
Salmonella	5	0	0/25g	-	GB 4789.4
Staphylococcus aureus	5	2	10	100	GB 4789.10 plate counting method
^a Samples shall be analyzed and processed in accordance with GB 4789.1. ^b Products which are not suitable to include active bacteria (aerobiotic and anaerobic probiotics) (viable count of activated probiotics in products shall be 10 ⁶ CFU/g (mL) or more) ^c Products only suitable for people aged from 1 to 10 years.					

3.8 Food additives and nutrition enhancers

3.8.1 The usage of food additives in products suitable for people aged from 1 to 10 years shall be in accordance with additive types and usage for infant formula foods in GB 2760. The usage of food additives in products suitable for people aged above 10 shall be in accordance with additive types and usage for the same or similar products in GB 2760.

3.8.2 Nutrient supplements shall be applied in accordance with GB 14880.

3.8.3 Specification and quality of food additives and nutrient supplements shall be in accordance with relevant standards and regulations.

3.8.4 One or more amino acids can be added to the formulated foods for special medical purposes according to people's special requirement for nutrition. The origin of amino acids shall be in accordance with Appendix B and/or GB 14880.

3.8.5 Other substances that are added to formulated foods for special medical purposes shall be in accordance with the relevant national regulations.

4 Others

4.1 Labels

4.1.1 Product labels shall be in accordance with GB 13432. The label "every 100 kJ (/100kJ)" shall be added to the label of nutrient and selectable ingredients.

4.1.2 The formula or nutritional features of products shall be described in the label, as well as the product types, target users and the warning "unsuitable for non-target people".

4.1.3 The warning "to be used under the guidance of doctors or clinical dietitians" shall be placed in the striking area of the label.

4.1.4 The warning "this product shall not be used for parenteral nutrition support or intravenous injection" shall be shown in the label.

4.2 Instructions for use

4.2.1 The usage, explanation and diagram of formulation, and the storage condition of relevant products shall be definitely specified on the label. Such a diagram may not be used when the largest superficial area of the package is less than 100 cm² or the product weight is less than 100 g.

4.2.2 Hazard to health due to improper formulation or misuse shall be demonstrated in the instructions.

4.3 Packages

Food-grade carbon dioxide and/or nitrogen whose purity is 99.9% or more can be used as the packing medium.

Appendix A

Common specific full nutritional formula foods

- A.1 Full nutritional formula foods for diabetes
- A.2 Full nutritional formula foods for diseases of respiratory system
- A.3 Full nutritional formula foods for nephrosis
- A.4 Full nutritional formula foods for tumors
- A.5 Full nutritional formula foods for liver disease
- A.6 Full nutritional formula foods for the muscle attenuation syndrome
- A.7 Full nutritional formula foods for trauma, infection, surgery and other stress situations
- A.8 Full nutritional formula foods for inflammatory bowel diseases
- A.9 Full nutritional formula foods for food protein allergy related
- A.10 Full nutritional formula foods for intractable epilepsy
- A.11 Full nutritional formula foods for gastrointestinal malabsorption and pancreatitis
- A.12 Full nutritional formula foods for fatty acid metabolism disorder
- A.13 Full nutritional formula foods for obesity and defatting surgery

Appendix B Amino acids that can be used for formulated foods for special medical purposes

See the amino acids that can be used in formulated foods for special medical purposes at Table B.1.

Table B.1 Amino acids that can be used for formulated foods in special medical purposes

S/R Number	Amino acid ^{a,b}	Compound source	Chemical name	Molecular formula	Molecular weight	Specific rotation [α] _{D,20°C}	pH	Purity % ≥	Moisture % ≤	Ash % ≤	Lead mg/kg ≤	Arsenic mg/kg ≤
1	Aspartic acid	L-aspartic acid	L-Asparaginic acid	C ₄ H ₇ NO ₄	133.1	+24.5~+26.0	2.5~3	98.5	0.2	0.1	0.3	0.2
		L-magnesium	L-magnesium aspartic	2(C ₄ H ₆ NO ₄) Mg	288.49	+20.5~+23.0	—	98.5	0.2	0.1	0.3	0.2
2	Threonine	L-threonine	L-2-amino-3-hydroxyb	C ₄ H ₉ NO ₃	119.12	-26.5~-29.0	5.0~6	98.5	0.2	0.1	0.3	0.2
3	Serine	L-serine	L-2-amino-3-hydracryli	C ₃ H ₇ NO ₃	105.09	+13.6~+16.0	5.5~6	98.5	0.2	0.1	0.3	0.2
4	Glutamic acid	L-glutamic acid	α-aminoglutaric acid	C ₅ H ₉ NO ₄	147.13	+31.5~+32.5	3.2	98.5	0.2	0.1	0.3	0.2
		L-potassium	α-potassium	C ₅ H ₈ KNO ₄ ·H ₂ O	203.24	+22.5~+24.0	—	98.5	0.2	0.1	0.3	0.2
		L-calcium	α-calcium	C ₁₀ H ₁₆ CaN ₂ O ₈	404.39	+27.4~+29.2	6.6	98.5	0.2	0.1	0.3	0.2
5	Glutamine	L-glutamine	2-amino-4-butanoic	C ₅ H ₁₀ N ₂ O ₃	146.15	+6.3~+7.3	—	98.5	0.2	0.1	0.3	0.2
6	Proline	L-proline	Pyrrolidine-2-carboxyli	C ₅ H ₉ NO ₂	115.13	-84.0~-86.3	5.9~6	98.5	0.2	0.1	0.3	0.2
7	Glycine	Glycine	Amino acetic acid	C ₂ H ₅ NO ₂	75.07	—	5.6~6	98.5	0.2	0.1	0.3	0.2
8	Alanine	L-alanine	L-2-aminopropionic	C ₃ H ₇ NO ₂	89.09	+13.5~+15.5	5.5~7	98.5	0.2	0.1	0.3	0.2
9	Cystine	L-cystine	L-3,3'-dithiobis (2-aminopropionic	C ₆ H ₁₂ N ₂ O ₄ S ₂	240.3	-215~-225	5.0~6.5	98.5	0.2	0.1	0.3	0.2
		L-cysteine	L-α-amino-β-thiohydra	C ₃ H ₇ NO ₂ S	121.16	+8.3~+9.5	4.5~5	98.5	0.2	0.1	0.3	0.2
		L-cysteine	L-cysteiny	C ₃ H ₇ NO ₂ S·HCl·H	175.63	+5.0~+8.0	—	98.5	0.2 b	0.1	0.3	0.2
		N-acetyl-L-cysteine	N-acetyl-L-α-amino-β-thiohydracrylic acid	C ₅ H ₉ NO ₃ S	163.20	+21~+27	2.0~2.8	98.0	0.2	0.1	—	—
10	Valine	L-valine	L-2-amino-3-isovaline	C ₅ H ₁₁ NO ₂	117.15	+26.7~+29.0	5.5~7	98.5	0.2	0.1	0.3	0.2
11	Methionine	L-methionine	2-amino-4-methyl	C ₅ H ₁₁ NO ₂ S	149.21	+21.0~+25.0	5.6~6	98.5	0.2	0.1	0.3	0.2
		N-acetyl-L-met	N-acetyl-2-amino-4-m	C ₇ H ₁₃ NO ₃ S	191.25	-18.0~ -22.0	—	98.5	0.2	0.1	0.3	0.2
12	Leucine	L-leucine	L-2-amino-4-methyl	C ₆ H ₁₃ NO ₂	131.17	+14.5 ~+16.5	5.5~6	98.5	0.2	0.1	0.3	0.2

Table B.1 (to be continued)

S/R Number	Amino acid a,b	Compound source	Chemical name	Molecular formula	Molecular weight	Specific rotation [α] _{D,20°}	pH	Purity % ≥	Moisture % ≤	Ash % ≤	Lead mg/kg ≤	Arsenic mg/kg ≤
13	Isoleucine	L-isoleucine	L-2-amino-3-methylpentanoic acid	C ₆ H ₁₃ NO ₂	131.17	+38.6~+41.5	5.5~7.0	98.5	0.2	0.1	0.3	0.2
14	Tyrosine	L-tyrosine	S-amino-3-(4-hydroxycyclohexyl)phenyl ketone)-propionic acid	C ₉ H ₁₁ NO ₃	181.19	-11.0~-12.3	—	98.5	0.2	0.1	0.3	0.2
15	Phenylalanine	L-phenylalanine	L-2-amino-3-phenylpropionic acid	C ₉ H ₁₁ NO ₂	165.19	-33.2~-35.2	5.4~6.0	98.5	0.2	0.1	0.3	0.2
16	Lysine	L-lysine hydrochloride	L-lysine monohydrochloride	C ₆ H ₁₄ N ₂ O ₂ ·HCl	182.65	+20.3~+21.5	5.0~6.0	98.5	0.2	0.1	0.3	0.2
		L-lysine acetate	L-Lysine monoacetate	C ₆ H ₁₄ N ₂ O ₂ ·C ₂ H ₄ O ₂	206.24	+8.5~+10.0	6.5~7.5	98.5	0.2	0.1	0.3	0.2
		L-lysine	L-2,6-diamino caproic acid	C ₆ H ₁₄ N ₂ O ₂ ·H ₂ O	164.2	+25.5~+27.0	9.0~10.5	98.5	0.2	0.1	0.3	0.2
		L-lysine-L-glutamic acid	L-2,6-diamino caproic acid α-amino glutarate	C ₁₁ H ₂₃ N ₃ O ₆ ·2H ₂ O	329.35	+27.5~+29.5	6.0~7.5	98.0	0.2	0.1	0.3	0.2
		L-lysine-aspartic acid	L-2,6-diamino caproic acid L- amino succinate	C ₁₀ H ₂₁ N ₃ O ₆	279.30	+24.0~+26.5	5.0~7.0	98.0	0.2	0.1	0.3	0.2
17	Arginine	L-arginine	L-2-amino-5-guanidyl valeric acid	C ₆ H ₁₄ N ₄ O ₂	174.2	+26.0~+27.9	10.5~12.0	98.5	0.2	0.1	0.3	0.2
		L-arginine hydrochloride	L-2-amino-5-guanidyl valeric acid hydrochloride	C ₆ H ₁₄ N ₄ O ₂ ·HCl	210.66	+21.3~+23.5	—	98.5	0.2	0.1	0.3	0.2
		L-arginine-aspartic acid	L-2-amino-5-guanidyl valeric acid- L-aspartic acid	C ₁₀ H ₂₁ N ₅ O ₆	307.31	+25.0~+27.0	6.0~7.0	98.5	0.2	0.1	0.3	0.2

GB 29923-2013 Good Manufacturing Practice of Foods for Special Medical Purposes



National Standards of People's Republic of China

GB 29923-2013

National Food Safety Standard
Good Manufacturing Practice of Foods for Special Medical Purposes

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National Standard for Food Safety

Good Manufacturing Practice of Foods for Special Medical Purposes

1. Scope

The standard specifies the basic requirements and administration rules for the sites, facilities and personnel during the raw material purchase, processing, packaging, storage and transportation of formulated foods for special medical purposes.

The standard is applicable to manufacturers of formulated foods for special medical purposes including infant formula foods for special medical purposes.

2. Terms and definitions

The following terms and definitions specified in GB 14881--National Standard for Food Safety, General Hygienic Regulation of Food Production are applicable to the standard.

2.1 Formulated foods for special medical purposes

As especially formulated foods that are produced to meet the special requirements for nutrient or meals of people who suffer from eating limitation, disorder of digestion and absorption, metabolic disorders or special disease state, these products shall be eaten individually or together with other foods under the guidance of doctors or clinical dietitians. Formulated foods for special medical purposes shall be based on the medical and/or nutritional research results with scientifically verified security and clinical effects.

2.2 Clean work area

Work areas with high requirements for cleanliness, such as the processes where liquid products are in contact with the air environment (such as the weighing and compounding), bottling rooms and workshops for storage, filling and inner packing of semi-finished products where powder products are exposed for packaging.

2.3 Sub-clean work area

Work areas with requirements for cleanliness lower than clean work area, such as the raw material pre-treatment plants.

2.4 Ordinary work area

Work areas with lower requirements for cleanliness than sub-cleaning work area, such as milk collection rooms, warehouses for raw materials and packaging materials, workshops for external packing and warehouses for finished products, etc.

2.5 Commercial sterilization

A condition where products, after proper sterilization, contain neither pathogenic microorganisms nor non-pathogenic microorganisms that can breed in the normal temperature.

2.6 Sterile filling

The process to put the foods which are sterilized and meet the commercial sterilization requirements into the pre-sterilized container (with a lid) before sealing it in an aseptic environment.

3. Site selection and the plant area environment

It shall be in accordance with GB 14881.

4. Workshop and factory

4.1 Design and layout

4.1.1 It shall be in accordance with GB 14881.

4.1.2 Facilities and equipment related to manufacture shall be properly designed, built and planned at the workshop and factory so as to prevent the breeding and pollution of microorganisms, especially the pollution of salmonella. As to infant products, pollution of enterobacter sakazakii (belonging to Cronobacter) shall be especially prevented. Their hideouts or breeding shall be prevented or reduced as much as possible. The following shall be considered during the design:

- a) Dry area shall be separated from wet area. Cross contamination due to personnel, equipment and material flow shall be effectively controlled.
- b) Work material shall be properly piled to prevent the sites from being untidy due to improper piling.
- c) The enclosure and sealing between the perforations and various pipes cables which go through the building floors, ceilings and walls shall be ensured.
- d) The wet cleaning procedure shall be properly designed to prevent the breeding and spreading of microorganisms in dry areas due to improper wet cleaning procedures.
- e) Proper facilities or measures shall be utilized or taken to remain dry while avoiding and timely removing water residue, thus preventing relevant microorganisms from growing and spreading.

4.1.3 Cleanliness levels for work zones shall be determined according to the requirements for manufacturing techniques, sanitation and quality. In principle, work zones can be divided into ordinary zones, sub-clean zones and clean zones.

4.1.4 Work without follow-up sterilization in dry processing areas shall be done in clean work areas, such as the work from drying (or post-drying) processes to the filling and sealing of packages.

4.1.5 Work zones of different cleanliness levels shall be effectively separated. Independent air cleaning systems with filters shall be installed at the clean work zones. Furthermore, positive pressure in the areas shall be maintained so as to prevent the air that is not purified from going into the clean work areas and causing cross contamination.

4.1.6 There shall be proper limitation and control over the behaviors of entering and exiting the clean work areas so as to avoid or reduce microorganism pollution. There shall be relevant measures to prevent cross contamination due to the personnel, raw materials, packaging materials, waste and equipment that enter or exist the clean work area, such as the work clothes, shoes or shoe covers replaced in the changing rooms, special logistics channels and waste channels. Proper air filtration systems shall be designed and installed for powder materials or products which enter the clean work zones via pipelines.

4.1.7 The purification levels in every work zone shall meet the requirements of formulated foods for special medical purposes for air purification when being processed. The air cleanliness in clean work zones and sub-clean work zones for solid and liquid products shall be in accordance with Table 1 and Table 2, and be inspected on a regular basis.

Table 1 Requirements for control over the air cleanliness in clean and sub-clean work areas for solid products

Items		Requirements		Test method
		Sub-clean work areas	Clean work areas	
Dust count/m ³	≥0.5μm	—	≤7,000,000	To be tested in accordance with GB/T 16292. The test state is static state.
	≥5μm	—	≤60,000	
Ventilation rate ^a (per hour)		—	10~15	—
Total bacterial count (CFU/皿)		≤30	≤15	To be tested with the natural sinking method in accordance with GB/T 18204.1.
^a Ventilation rate is applicable to clean work zones with floors lower than 4.0m.				

Table 2 Requirements for control over the air cleanliness in clean work areas for liquid products

Items		Requirements	Test method
		Clean work areas	
Dust count/m ³	≥0.5μm	≤3,500,000	To be tested in accordance with GB/T 16292. The test state is static state.
	≥5μm	≤20,000	
Ventilation rate ^a (per hour)		10~15	—
Total bacterial count (CFU/皿)		≤10	To be tested with the natural sinking method in accordance with GB/T 18204.1.
^a Ventilation rate is applicable to clean work zones with floors lower than 4.0m.			

4.1.8 Clean work areas shall be dry. Water facilities and systems shall be prevented as much as possible there. If unable to avoid them, they shall not pass through the upper space of the production work area in case secondary pollution occurs. There shall also be preventive measures.

4.1.9 There shall be facilities to keep animals such as insects and rats away from plants, workshops and warehouses.

4.2 Interior structures and materials of the buildings

4.2.1 Ceilings

4.2.1.1 They shall be in accordance with GB 14881.

4.2.1.2 Internal ceilings and top corners in work sites such as workshops shall be easy to clean so as to avoid dust accumulation, dew formation, mold growth or exfoliation and other conditions. Smooth and cleanable ceilings shall be added where, in clean work zones, sub-clean work zones and other sites where foods are exposed, ceilings are apt to harbor dusts. Ceilings made of reinforced concrete shall be flat and gap-free.

4.2.1.3 Flat-topped ceilings in workshops shall be made of white or light-colored, non-toxic, odorless waterproof materials. Mold-proof, durable and cleanable coating shall be used when necessary.

4.2.2 Walls

They shall be in accordance with GB 14881.

4.2.3 Doors and windows

They shall be in accordance with GB 14881. Self-closing doors (with auto sensors or door closers) and/or air curtains shall be installed at the entrances and exits of clean work zones and sub-clean work zones.

4.2.4 Grounds

They shall be in accordance with GB 14881. Grounds, where drainage or waste water flows and which are often damp or washed with water during work, shall be acid- and alkali- resistant and slope at some angle for drainage.

4.3 Facilities

4.3.1 Water facilities

4.3.1.1 They shall be in accordance with GB 14881.

4.3.1.2 Water facilities and equipment shall be in accordance with relevant national administrative regulations.

4.3.1.3 Safety and health facilities shall be installed at the entrances of water facilities so as to prevent animals and other substances from entering and causing food pollution.

4.3.1.4 Secondary water supply facilities shall be in accordance with GB 17051-- Hygienic Regulations for Secondary Water Supply Facilities.

4.3.2 Drainage facilities

4.3.2.1 They shall be in accordance with GB 14881.

4.3.2.2 The drainage system shall slope at some angle and keep unobstructed so as to be cleaned and maintained. There shall be some curve at the junction of flanks and bottoms of the drainage ditches.

4.3.2.3 There shall not be water supply pipes for production water within the drainage systems and underneath.

4.3.3 Facilities for cleansing and disinfection.

They shall be in accordance with GB 14881.

4.3.4 Private sanitary facilities

4.3.4.1 They shall be in accordance with GB 14881.

4.3.4.2 Secondary dressing rooms shall be installed at the entrances of clean work areas. Hand disinfection facilities shall be installed at the entrances of clean work areas.

4.3.5 Ventilation equipment

4.3.5.1 They shall be in accordance with GB 14881. Environment temperature and, when necessary, air humidity shall be controlled at clean work areas where powder products are manufactured.

4.3.5.2 Air conditioning facilities shall be installed at the clean work areas so as to avoid devaporation and keep the indoor air fresh. Proper facilities for removal, collection or control shall be installed at the areas where odors, gas (steam, poisonous and hazardous gas) or powder may contaminate foods.

4.3.5.3 The air inlet shall be 2m above the ground or roof. It shall be away from the pollution sources and air outlets. Air filtering facilities shall be installed.

4.3.5.4 Compressed air or other inert gases that are used to transport or package foods, clean the food contact surfaces or facilities shall be filtered and purified.

4.3.6 Lighting facility

It shall be in accordance with GB 14881. The workshop daylight factor shall reach Standard IV or higher. Mixing illumination in the quality control working plane shall reach 540 lx or higher. And the working plane in the processing sites shall reach 220 lx or higher and that in other sites shall reach 110 lx or higher. Beam-focusing sensitivity test zones are exceptions.

4.3.7 Warehousing facilities

4.3.7.1 They shall be in accordance with GB 14881.

4.3.7.2 Storage sites shall be divided according to the different characteristics of raw materials, semi-finished products, finished products and packing materials. Refrigerating (freezing) chambers shall be used when necessary. Different substances shall be properly separated or divided (classified or stored in different shelves or zones) and obviously marked when stored in the same warehouse.

4.3.7.3 Temperature monitoring facilities, such as thermometers, temperature measuring devices or automatic temperature recorders which can indicate the right temperatures in the refrigerating (freezing) chambers, shall be installed to carry out real time monitoring and keep records.

5. Facilities

5.1 Production equipment

5.1.1 General requirements

5.1.1.1 They shall be in accordance with GB 14881.

5.1.1.2 Operation specifications of special equipment (such as pressure vessels and pressure pipes) shall be formulated during the productive process.

5.1.2 Materials

Materials for production equipment shall be in accordance with GB 14881.

5.1.3 Design

5.1.3.1 It shall be in accordance with GB 14881.

5.1.3.2 The food contact surfaces shall be smooth, indentation- or crack-free so as to reduce the accumulation of food scraps, dirt or organic matters.

5.1.3.3 The facilities that are in contact with ingredients shall have smooth, complete, anti-corrosion, dead-zone-free interiors that are easy to clean. These interiors shall be made of materials that neither react with ingredients nor release particles nor absorb ingredients.

5.1.3.4 Storage, transportation and processing systems (including gravity, pneumatic, airtight and automatic systems) shall be designed and manufactured to facilitate the maintenance of their good sanitary conditions.

5.1.3.5 There shall be special zones for the storage of spare parts so as to provide necessary parts for equipment maintenance. Storage zones for spare parts shall be kept clean and dry.

5.1.3.6 Production equipment shall bear obvious labels for running status and be kept, maintained and verified on a regular basis. The product quality shall not be impaired during their installation and maintenance. Facilities shall be verified or determined to ensure that all their performance meets the requirements of the technology. Disqualified facilities shall be removed away from the production area. They shall have obvious labels before being removed.

5.1.3.7 Measuring instruments and key gauges for production shall be examined on a regular basis. Facilities for dry blending can ensure the mixing uniformity of products.

5.2 Monitoring equipment

5.2.1 It shall be in accordance with GB 14881.

5.2.2 See the related functions of computer systems and their network techniques in Appendix A when carrying out collection of key control points monitoring data and managing various records with these computer systems and their network techniques.

5.3 Maintenance and repair of facilities

5.3.1 They shall be in accordance with GB 14881.

5.3.2 Every time before production, facilities shall be checked to find if they are under normal conditions so as to ensure that the hygienic quality of products is not impaired. Failures shall be timely eliminated once they occur. Time and causes of failures, as well as the product groups that may be affected, shall be recorded.

6. Sanitary management

6.1 Sanitation management systems

They shall be in accordance with GB 14881.

6.2 Sanitary management of plants and facilities

6.2.1 It shall be in accordance with GB 14881.

6.2.2 Removable devices and tools which have been cleaned and sterilized shall be placed in a proper site where food contact surfaces will not be contaminated again so as to keep them applicable.

6.3 Cleaning and sterilization

6.3.1 Effective plans and procedures for cleaning and sterilization shall be formulated to ensure the sanitation and hygiene of food processing sites, equipment and facilities and to avoid food contamination.

6.3.2 In clean work zones where dry operation is needed (such as dry blend, powder product filling, etc), to perform the effective dry-clean process on the production equipment and processing environments is an effective way to prevent microorganisms from breeding. Wet-clean processes shall be avoided as much as possible. Wet-clean processes shall be limited to equipment components that can be carried to special rooms or conditions where dry-clean processes are not available. When dry-clean processes are not available, wet-clean processes under control shall be performed only if equipment and environments can be timely, thoroughly recovered to dryness in case the zone is contaminated.

6.3.3 Effective supervisory processes shall be formulated so as to ensure that key processes (such as manual cleaning, cleaning in place and facility maintenance) are in accordance with relevant regulations and standard requirements. In particular, applicability of cleaning and disinfection solutions shall be ensured. Concentration of disinfectants and detergents shall be appropriate. CIP System shall meet the relevant requirements for temperature and time and the equipment shall be properly rinsed when necessary.

6.3.4 The periodic chart to clean and sterilize all the workshops shall be formulated so as to ensure all the zones are cleaned and special cleaning are carried out on important zones, equipment and tools. The cycle and effectiveness to clean the equipment shall be verified or justified.

6.3.5 The amount of cleaning staff shall be guaranteed and their individual responsibilities shall be specified. All the cleaning staff shall be well trained and acquainted with the hazard of pollution and the importance to prevent it. Records on cleaning and sterilization shall be well made.

6.3.6 Cleaning tools for different clean zones shall be definitely marked and cannot be mixed.

6.4 Personnel health and sanitary requirements

6.4.1 General requirements

Management of food processing personnel health shall be in accordance with GB 14881.

6.4.2 Sanitary requirements for food processing personnel

6.4.2.1 It shall be in accordance with GB14881.

6.4.2.2 Personnel at sub-clean and ordinary work areas shall wear work clothes which are in accordance with the sanitary requirements of relevant areas. They shall also be equipped with caps and work shoes. Personnel at clean work areas shall wear work clothes (or disposable work clothes) that meet the sanitary requirements of the area. They shall also be equipped with caps (or hoods), masks and work shoes (or shoe covers).

6.4.2.3 Personnel shall enter the clean work areas after procedures such as secondary dressing and hand cleaning and sterilizing so as to ensure their hand sanitation. They shall wear work clothes, hoods or caps, work shoes or shoe covers. Work clothes and shoes designated for clean and sub-clean work areas shall not be worn outside designated areas.

6.4.3 Visitors

They shall be in accordance with GB 14881.

6.5 Insect pest control

It shall be in accordance with GB 14881.

6.6 Waste disposal

6.6.1 It shall be in accordance with GB 14881.

6.6.2 Containers for waste, by-products and inedible or hazardous substances shall be well-textured and waterproof with special marks on. When necessary, they shall be sealed so as to avoid food contamination.

6.6.3 Temporary storage facilities for waste shall be installed in appropriate places, where waste is classified and stored according to its characteristics. Corruptible waste shall be timely removed.

6.7 Management of poisonous and hazardous substances

Management of cleaning agents, disinfectants, pesticides and other poisonous and hazardous substances shall be in accordance with GB 14881.

6.8 Management of waste water

Waste water shall be properly dealt with before discharge so as to meet the national requirements for sewage discharge.

6.9 Management of work clothes

It shall be in accordance with GB 14881.

7. Requirements for raw and packaging materials

7.1 General requirements

It shall be in accordance with GB 14881.

7.2 Requirements for purchase and acceptance inspection of raw and packaging materials

7.2.1 Purchase of raw and packaging materials shall be in accordance with GB 14881.

7.2.2 Companies shall formulate regulations to manage suppliers and specify the procedures to select, examine and evaluate them.

7.2.3 Once found, food safety problems on raw and packaging materials shall be reported to the local food safety supervision department.

7.2.4 As to raw materials which directly go into the dry-mixing procedure, integrity of their packages shall be ensured and no traces of insect pests or other pollution shall be spotted.

7.2.5 As to raw materials which directly go into the dry-mixing procedure, the company shall take measures to ensure that the microbiological indicator meet the requirements for finished products. It shall be ensured that the urease activity of soybean raw materials is negative.

7.2.6 Procedures and safety precautions related to suppliers shall be evaluated. Site assessment or procedures shall be monitored on a regular basis when necessary.

7.3 Requirements for transportation and storage of raw and packaging materials

7.3.1 Companies shall transport and store the raw and packaging materials while meeting the requirements for quality safety.

7.3.2 During transportation and storage, raw and packaging materials shall avoid direct sunshine, rain, intensive temperature, humidity change and intensive impact. They shall not be transported together with poisonous, hazardous substances.

7.3.3 During transportation and storage, raw and packaging materials shall not be polluted and damaged, thus reducing quality deterioration to the minimum level. Materials and packaging materials which have special requirements for temperature, humidity or others shall be delivered and stored as specified.

7.3.4 During storage, different raw and packaging materials shall be separately stored according to their characteristics and labels shall be established to show relevant information and quality conditions.

7.3.5 Stored raw and packaging materials shall be checked on a regular basis. Those raw and packaging materials which have been stored for a long time and whose quality may have changed must be sampled to determine their quality. Spoiled or out-of-date raw and packaging materials shall be timely disposed of.

7.3.6 Qualified raw and packaging materials shall be used in accordance with the principle of first-in first-out (FIFO) or First Expiration First Out (FEFO) so as to be organized in a proper way.

7.3.7 Food additives and food nutritive fortifiers shall be managed by specially-assigned persons. Special warehouses or zones shall be used for storage and exclusive registers (or warehouse management software) shall be used to record the names, purchase time, purchase volume and usage amount of additives and nutrition enhancers. Furthermore, term of validity shall be paid attention to.

7.3.8 Raw material verification shall be done to ensure the qualification of vitamins and mineral substances which may change during the storage. When necessary, tests shall be done so as to ensure they meet the requirements for raw materials.

7.3.9 Raw materials which contain allergen shall be separately stored and well marked so as to avoid cross contamination.

7.4 Others

Relevant records on the purchase, acceptance inspection, storage and transportation of raw and packaging materials shall be kept.

8. Food safety control during the production

8.1 Risk control over product pollution

It shall be in accordance with GB 14881.

8.2 Control over microbial contamination

8.2.1 Temperature and time

8.2.1.1 The way to kill microorganisms or inhibit microorganism growth and breeding shall be specified according to the product characteristics, such as heat treatment, freezing or refrigerating storage. Effective monitoring shall be carried out.

8.2.1.2 Measures to control the temperature and time and to correct errors shall be formulated and verified on a regular basis.

8.2.1.3 Real time monitoring measures shall be formulated for processing procedures which require strict temperature and time control. Monitoring records shall be kept.

8.2.2 Humidity

8.2.2.1 Control over air humidity in places where it is required shall be carried out according to the product and technique characteristics so as to reduce the breeding of harmful microbe. Critical limit of air humidity shall be formulated and put into effective operation.

8.2.2.2 Real time control over air humidity and monitoring measures shall be formulated to make verification and keep records on a regular basis.

8.2.3 To avoid microbial contamination

8.2.3.1 Necessary measures shall be taken during the whole procedure from the inbound raw and packaging materials to the outbound finished products so as to avoid microbial contamination.

8.2.3.2 The operation, usage and maintenance of equipment, containers and tools which are used to deliver, carry or store the raw materials, semi-finished products and finished products shall avoid the contamination to the processed or stored foods.

8.2.4 Monitoring over the microorganisms during the manufacturing process

8.2.4.1 It shall be in accordance with GB14881.

8.2.4.2 Monitoring plans on microorganisms shall be formulated during the manufacturing process to carry out effective supervisory control in accordance with GB 14881-2013--Appendix A in combination with manufacturing techniques and requirements of National Standard for Food Safety--General Rule on Formulated Foods for Special Medical Purposes and GB 25596--National Standard for Food Safety--General Rule on Infant Formula Food for Special Medical Purposes. Total bacterial count and the

coli group shall be regarded as the indicator microorganisms for health standard. When the monitored results show deviations, proper corrective actions shall be taken to the control measures.

8.2.4.3 Powder foods for special medical purposes shall be in accordance with Appendix B. Environmental monitoring plans shall be formulated against the salmonella, enterobacter sakazakii and other enterobacterium in clean work zones. When the monitored results show deviations, proper corrective actions shall be taken to the control measures.

8.3 Control over chemical pollution

8.3.1 It shall be in accordance with GB 14881.

8.3.2 Chemical substances shall be separately stored with foods, definitely labeled and kept by specially-assigned persons.

8.4 Control over physical pollution

8.4.1 It shall be in accordance with GB 14881.

8.4.2 Work such as electric soldering, incision and polish cannot be done during the manufacture in case off-flavor and chippings appear.

8.5 Food additives and food nutritive fortifiers

8.5.1 Food additives and food nutritive fortifiers shall be rationally used according to the types, scope and dosage specified by National Standard for Food Safety.

8.5.2 When being used, food additives and food nutritive fortifiers shall be accurately weighed and recorded.

8.6 Packages

8.6.1 It shall be in accordance with GB 14881.

8.6.2 Packing materials shall be clean, non-toxic and in accordance with relevant national regulations.

8.6.3 Packaging materials or gas shall be non-toxic and shall not affect the food safety and product features under special storage and usage conditions.

8.6.4 Reusable packaging materials such as glass bottle and stainless steel containers shall be thoroughly rinsed and sterilized before being used.

8.7 Specific processing steps

8.7.1 General requirements

All treatment procedures in the manufacturing techniques of formulated foods for special medical purposes shall meet the requirements of corresponding specific processing steps and be in accordance with 8.7.2 to 8.7.9.

8.7.2 Heat treatment

The heat treatment procedure shall be the critical control point to ensure the safety of formulated foods for special medical purposes. As to the heat treatment temperature and time, the impact of factors such as product attributes (such as fat content, total solid content) on heat resistance of target microorganisms shall be considered. So relevant procedures shall be formulated to see if the temperature and time is deviated and proper corrective actions shall be taken.

When the purchased soybean raw materials fail to go through heated enzyme deactivation treatment (or the enzyme deactivation is incomplete), such soybean-based products shall go through the heat treatment while achieving the desired effect of pathogen killing and thorough enzyme deactivation (the urease shall be negative) and be monitored as a critical control point.

Records on critical process parameters such as the time, temperature and enzyme deactivation time in the heat treatment shall be kept.

8.7.3 Intermediate storage

During the production of formulated foods for special medical purposes, relevant measures shall be taken in the intermediate storage of liquid semi-finished products so as to prevent the growth of microorganisms. Raw material powder that is exposed during the dry production of powder formulated foods for special medical purposes or powder semi-finished products which are exposed during wet production shall be stored in the clean work zones.

8.7.4 Commercial sterile operation of liquid formulated foods for special medical purposes

It shall be in accordance with the operation guide in Appendix C.

8.7.5 Processing steps of powder formulated foods for special medical purposes from heat treatment to dryness

During the production of powder formulated foods for special medical purposes, the running pipes and equipment shall be airtight from heat treatment to dryness and be thoroughly cleaned and sterilized on a regular basis.

8.7.6 Cooling

The dry, exposed powder semi-finished products shall be cooled down in the clean work zone.

8.7.7 Key factor control over the dry blending in dry process of powder formulated foods for special medical purposes and dry-wet compound technology

8.7.7.1 The exposed powder process that is in contact with the air (such as premixing, sub-packaging and batch charging) shall be carried out in the clean work zone. The temperature and relative humidity in the clean work zones shall be in accordance with the manufacturing techniques of powder formulated foods for special medical purposes. When there is no special requirement, the temperature shall be 25°C or lower and the relative humidity shall be below 65%.

8.7.7.2 The batching shall be accurately calculated. There shall be review processes during the calculation of food additives and food nutritive fortifiers.

8.7.7.3 Critical process parameters (such as mixing time) related to mixing uniformity shall be verified. The mixing uniformity shall also be determined.

8.7.7.4 Compressed air which is needed to transfer materials with positive pressure shall only be used after the oil and water removal, cleaning, filtering and sterilization.

8.7.7.5 Sanitary control requirements for raw materials, packaging materials and personnel shall be strictly formulated. The raw materials shall enter the work zones via the necessary cleaning procedures and material channels. They shall be in accordance with the treatment procedure of outer package removal or outer package sterilization.

8.7.8 Key factor control over the inner packing of powder formulated foods for special medical purposes

8.7.8.1 Inner packing shall be carried out in clean work areas.

8.7.8.2 The wrapping room shall only be accessible to relevant personnel. Requirements for raw and packaging materials and the personnel shall be in accordance with 8.7.7.5 and 6.4.2.

8.7.8.3 The outer packages of the packaging materials shall be examined before use to see if they are intact so as to ensure that these materials are not contaminated.

8.7.8.4 Manufacturers shall take effective measures to control, prevent and inspect the extraneous substances. For example, screens, strong magnets and metal detectors shall be installed. Supervisory control or validation verification shall be carried out on the procedures of these measures.

8.7.8.5 When different types of products are produced on the same assembly line, they shall be cleaned in an effective way and site-clearing records shall be kept so as to ensure that product switch doesn't affect the next group of products.

8.7.9 Control over production water supply

8.7.9.1 Production water supply, equipment cleaning water, ice and steam which is in direct contact with foods shall be in accordance with GB 5749--Hygienic Standard of Drinking Water.

8.7.9.2 Recycle-water from the steam or dry process in the food processing or water in cycle use can be reused, but they shall not jeopardize the food safety and product features. Water treatment and effective monitoring shall be carried out when necessary.

8.7.9.3 When liquid products are produced, production water in direct contact with the products shall be manufactured with the de-ionization method or ion exchange method, reverse osmosis method and other proper methods according to the characteristic of the products so as to ensure that the requirements for product quality and technology can be met.

9. Verification

9.1 The productive process shall be verified so as to ensure the reproducibility of the whole process and the controllability of the product quality. The production verification shall include the installation determination, operation determination, performance determination and product verification of plants, facilities and equipment.

9.2 Verification projects shall be put forward according to the verified objects. Verification schemes shall be formulated and implemented.

9.3 Manufacturing techniques and key facilities and equipment of the products shall be verified according to the verification schemes. When key factors which affect the product quality (including the nutrient content)

change, such as the technologies, quality control methods, main raw materials, main production equipment, or when the production has undergone a period, re-validation shall be carried out.

9.4 After the verification is done, test reports shall be written and then examined and approved by verifiers. Data and analytical content during the verification shall be recorded in files. Verifying files include verification schemes, verification reports, comments and Suggestions, approvers, etc.

10. Test

10.1 It shall be in accordance with GB 14881.

10.2 Representative samples shall be taken from finished products group by group. The samples shall be tested and kept in accordance with the relevant national regulations and standards.

10.3 Quality control over the labs shall be strengthened so as to ensure the accuracy and authenticity of verification results.

11. Storage and transportation of products

11.1 It shall be in accordance with GB 14881.

11.2 Storage and transportation of products shall be in accordance with the storage condition on their labels.

11.3 Products in the warehouses shall be checked on a regular basis. Temperature and/or humidity records shall be kept when necessary. Abnormal situations shall be immediately processed when occurring.

11.4 Quality conditions shall be marked on verified products.

11.5 Relevant records shall be made on the storage and transportation of products. Outbound products shall bear delivery records so that they can be recalled immediately when problems occur.

12. Product tracing and recall

12.1 Product tracing systems shall be formulated to ensure that products can be effectively traced during the whole process from raw material purchase to product distribution.

12.2 Product recall systems shall be formulated. When a group or a type of products are found to or be likely to jeopardize the consumers' health, product recall systems shall be launched according to the relevant national regulations. Relevant departments shall be immediately notified and relevant records shall be made.

12.3 Measures such as non-hazardous treatment and destruction shall be taken after the foods are recalled. Relevant departments shall be notified of the food recall and processing.

12.4 Customer complaint and processing systems shall be formulated. Relevant company management departments shall keep records of and find the reasons to the customers' written or oral advice and complaints before appropriately solving them.

13. Training

13.1 It shall be in accordance with GB 14881.

13.2 Annual training plans shall be formulated according to the different requirements for duties to carry out corresponding training. Special types of work require relevant work licenses.

14. Management system and personnel

14.1 It shall be in accordance with GB 14881.

14.2 Perfect food safety management systems shall be formulated and relevant management measures shall be taken to carry out safety quality control over the formulated foods for special medical purposes during the whole process from the inbound raw materials to outbound finished products so as to ensure that products are in accordance with laws and regulations and relevant standards.

14.3 Supervision organs of food safety shall be established to manage food safety.

14.4 Head of the above organ shall be the legal representative or a person-in-charge assigned by the legal representative.

14.5 Different departments within the organ shall have their definite management responsibilities to ensure that management responsibilities related to quality and safety are put in place. Every department shall have its effective division mechanism to avoid responsibility overlapping, repetition or absence. Corresponding management systems shall be formulated for the maintenance and management of the inside and outside plant environments, plant facilities and equipment, the quality safety management during the production, sanitary control and quality tracking. The managers and their responsibilities shall be clear and definite.

14.6 Supervision organs of food safety shall be equipped with especially trained food safety managers who are responsible for publicizing and implementing food safety regulations and relevant rules, as well as supervising the implementation of examination and keeping relevant records.

15. Management of records and files

15.1 Record management

15.1.1 It shall be in accordance with GB 14881.

15.1.2 All records shall be signed or stamped by executives or relevant supervisors for review. Once altered, the records shall be clear enough to show their original appearance. Modifiers shall sign or stamp the area near the words.

15.1.3 All production and quality management records shall be examined and verified by relevant departments so as to make sure all the processing is in accordance with regulation. When occurring, abnormal events shall be dealt with immediately.

15.2 File management

File management systems shall be formulated in accordance with GB 14881 and intact quality management files shall be established. Files shall be classified and stored. Files for distribution and use shall be approved current texts. The abolished or invalid files shall be only stored for inspection. They shall not appear at the working sites.

16. Monitoring and evaluation of the effectiveness of food safety control measures

Monitoring and evaluation measures in Appendix C shall be taken to ensure the effectiveness of food safety control measures for powder formulated foods for special medical purposes.

Appendix A

Computer system application guide for manufacturers of formulated foods for special medical purposes

A.1 Computer systems for manufacturers of formulated foods for special medical purposes shall be in accordance with Food Safety Law and relevant laws, regulations and standards on food safety. An integrated information link which supports the traceability, tracing and location of food safety during all processes from inbound raw materials to outbound products. Relevant data shall be submitted or reported from a long distant away according to the requirements of supervision departments. This computer system shall be in accordance with but not limited to the requirements of A.2 to A.11.

A.2 The system shall include the purchase and acceptance check of raw materials, storage and use of raw materials, supervisory control over the critical control links of production and processing, outbound product inspection, storage and transportation of products, distribution and other links, as well as the collection of data related to food safety and record retention.

A.3 The system shall be able to evaluate and send pre-warning about related raw materials, processing techniques and risks of food safety.

A.4 Perfect authorization management mechanisms shall be formulated in the system and the matched database so as to ensure the mandatory usage of workers' accounts/passwords. Bugs which allow unauthorized access shall not be allowed in the systems and database on the security architecture.

A.5 The system shall achieve perfect safety strategies based on the authorization management mechanism and ensure that specific role users only have their corresponding authorities by setting up strategy groups according the different workers. All data which the system is in contact with and produce shall be saved in the corresponding database. They shall not be saved as files so as to ensure access to all data is subject to the authority management control of the system and database.

A.6 Special security strategies taken to the confidential information ensure that only information owners have the right to read, write and cancel the information. If it is required that confidential information be saved and transmitted outside the safety control scope of the systems and database, the following shall be guaranteed:

a) To encrypt and save confidential information in case the information is read by unauthorized persons.

b) Check codes shall be generated before the transmission of confidential information; Check codes and information (encrypted) shall be separately transmitted; Check codes shall be used at the receiving end so as to ensure the information is not falsified.

A.7 If the system requires to collect data generated by automation detecting instruments, the system shall provide safe, reliable data interfaces and guarantee their accuracy and high availability, thus ensuring the data generated by instruments can be timely and accurately collected by the system.

A.8 Log management functions of complete, exhaustive systems and database shall be achieved, which includes:

- a) Every user log-in on the system's logging system and database (users, time, locations of log-in computers);
- b) Every alteration of the logging data (including the altering users, altering time, altered content, original content, etc);
- c) Conversation strategies shall be in the system log and operation log. They shall not be canceled or altered by any user (except for the administrators) within the set time limit so as to guarantee some time traceability.

A.9 Detailed usage and management system shall be formulated and contain at least the following:

- a) To formulate the real-time record system to record the original data, intermediate data, generated data and process flow during the work flow so as to reproduce the whole working process;
- b) To formulate detailed backup management systems to ensure that the whole system and corresponding data can be recovered as soon as possible after failures occur;
- c) The machine rooms shall be equipped with smart UPS to be linked with work systems and ensure that UPS can supply power and warn the work system to carry out data storage and log operation when power failures occur outside. (UPS can supply the power to guarantee the operation time of system emergency saving);
- d) Perfect data access management systems shall be formulated and restricted data shall not be saved on shared device. Authority management systems shall be applied to the internal data sharing to achieve authorized visit;
- e) Matched system maintenance mechanisms including regular storage arrangement and system detection shall be formulated to ensure the long-term stable operation of the system;
- f) Safety management systems shall be formulated. User passwords for the systems shall be replaced on a regular basis. Log-in sites of some users shall be restricted and unnecessary accounts shall be canceled.
- g) It shall be specified that users who log in on outer nets shall not start and use the user/password memory function provided by the operating systems of outer computers so as to prevent the information from being embezzled.

A.10 When the data from the real-time monitoring of critical control points are inconsistent with the set standard values, the system can record the deviation dates, batches, the way to rectify the deviation and operator names.

A.11 Data and relevant records in the system shall be reproducible so as to be inspected and analyzed by supervisors.

Appendix B

Environmental monitoring guide on salmonella, enterobacter sakazakii and other Enterobacteriums in the clean work areas of formulated foods for special medical purposes

B.1 Monitoring purposes

B.1.1 There may be a small amount of Enterobacteriaceae even in the sanitary production environments, including enterobacter sakazakii (belonging to Cronobacter). Pasteurized products may also be polluted by the environments, leading to the fact that a small amount of enterobacterium remains in the products. So enterobacterium in the production environments shall be monitored so as to determine whether the sanitation control procedure is effective. When deviation occurs, manufacturers shall take corrective actions and acquire the basic data of the hygiene status through continuous monitoring to trace the trend changes. According to relevant practices in the factory, reduction of the amount of enterobacterium in the environments can help decrease the enterobacterium in the final products (including enterobacter sakazakii and salmonella).

To prevent the contamination accidents and avoid the limitation of microbial sampling test in the final products, environmental monitoring plans shall be formulated as a food safety management tool and a basic procedure for Hazard Analysis and Critical Control Point (HACCP) to evaluate the sanitary conditions in clean work areas (dry areas).

B.1.2 Factors such as the ecological characteristics of salmonella, enterobacter sakazakii and other enterobacterium shall be considered when monitoring programs are formulated. Supervisory control over enterobacter sakazakii shall only be applicable to formulated infant foods for special medical purposes.

Salmonella can rarely be found in dry environments. Monitoring programs, however, shall be formulated to prevent its access. Effectiveness of hygiene control measures in the production environment shall be evaluated and relevant personnel shall be taught to prevent the salmonella from spreading when salmonella is found.

Enterobacter sakazakii is easier to be found in dry environments than salmonella and more likely to be detected with proper sampling and testing ways. Monitoring programs shall be formulated to appraise whether enterobacter sakazakii is increasing. Effective measures shall be taken to prevent its growth.

Enterobacterium, a common bacterium group in dry environments, is wide spread and easy to detect. Enterobacterium can be used as the indicator bacterium for environmental health conditions or during the production.

B.2 Factors which need considering in the design and sampling plans

B.2.1 Product category and technical process

The requirements and scope for sampling plans shall be determined according to the product feature, consumers' age and health conditions. In the standard, salmonella and enterobacter sakazakii are defined as pathogenic bacteria.

The emphasis of supervisory control shall be put on areas where microorganisms are likely to hide, such as the clean work area in the dry environments. Junctions between this area and the lower-level adjacent zones, as well as places that are close to the production line and equipment and are likely to be

contaminated, shall be paid more attention to, such as the opening of the enclosed equipment which is casually used for inspection. Areas that are known or may be contaminated shall be prioritized in supervisory control.

B.2.2 Two samples in the monitoring programs

B.2.2.1 Sampling from the surfaces which are in no touch with the food, such as equipment exteriors, ground around the production line, pipes and platforms. Under such circumstances, pollution risk degrees and pollutant content shall depend on the location and design of the production line and equipment.

B.2.2.2 Sampling from the surfaces which are in direct touch with the food, such as the equipment which may directly contaminate products from the spray tower to packaging. For example, microorganisms may breed at the sieve end due to the water absorption of clotting powdered formulas. Products are exposed to high contamination risks if indicator bacteria, enterobacter sakazakii or salmonella remain on the food contact surface.

B.2.3 Target microorganisms

Salmonella and enterobacter sakazakii are main target microorganisms. But the enterobacterium can be used as hygienic target bacteria. The enterobacterium content may show the possibility of salmonella to exist, as well as the conditions for salmonella and enterobacter sakazakii to grow.

B.2.4 Sampling location and sample amount

Sample amount shall be adjusted according to the complexity of techniques and the production line.

Sampling locations can be places where microorganisms may hide or go into so that contamination is caused. Sampling locations shall be determined according to relevant document literature or experience and expertise or historical data collected from factory pollution survey. Sampling locations shall be evaluated on a regular basis and necessary sampling locations shall be added to the monitoring programs according to special circumstances such as heavy maintenance, construction activities or worse sanitary conditions.

Sampling plans shall be comprehensive and representative. Scientific, rational sampling from various production shifts and different time frames in these shifts shall be considered. Sampling shall be carried out before production to verify the effect of cleaning measures.

B.2.5 Sampling frequency

The sampling frequency shall be determined according to B.2.1 and the data of existing microorganisms in different regions within the monitoring programs. If there are no such data, information shall be fully collected so as to determine the rational sampling frequency, including the long-term collection of the occurrence of salmonella or enterobacter sakazakii.

Implementation frequency of environmental monitoring plans shall be adjusted according to the testing results and severity of pollution risks. When pathogenic bacteria are detected in the final products or when the indicator bacteria increase, environmental sampling and investigation sampling shall be intensified so as to find the pollution source. When pollution risks increase (for example, after the maintenance, construction or wet cleaning), sampling frequency shall be properly increased.

B.2.6 Sampling tools and methods

Sampling tools and methods shall be selected according to the surface types and sampling locations. For

example, surface residues or powder in the dust collectors shall be directly chosen as samples. For larger surfaces, sponges or swabs shall be used for wiping sampling.

B.2.7 Analytical methods

Analytical methods shall be able to effectively detect target microorganisms with acceptable sensitivity and relevant records. On the basis of sensitivity, many samples can be detected together. If the outcomes are positive, the locations of positive samples shall be further determined. When necessary, information on the source of enterobacter sakazakii and pollution path of powder formulated foods for special medical purposes shall be analyzed with the genetic technology.

B.2.8 Data administration

Monitoring programs shall include data records and evaluating systems, such as the trend analysis. Constant evaluation must be made on data so as to properly alter and adjust monitoring programs. Effective management shall be carried out on the data of enterobacterium and enterobacter sakazakii. Neglected slight or discontinuous pollution may be found.

B.2.9 Corrective actions of positive results

Monitoring programs aim to find whether target microorganisms exist in the environments. Before the monitoring programs are formulated, acceptance standards and countermeasures shall be formulated. The concrete action strategies and relevant reasons shall be specified in the monitoring programs. Related measures include no actions (no pollution risks), to strengthen the cleaning and pollution source tracing (to intensify the environmental testing), to evaluate sanitary measures, to seize and detect products.

Action strategies shall be formulated by manufacturers after enterobacterium and enterobacter sakazakii are detected so as to accurately respond to the abnormal situations. Sanitation procedures and control measures shall be evaluated. Corrective actions shall be taken immediately after salmonella is detected. Enterobacter sakazakii trend and changes of enterobacter amount shall be evaluated. Actions to be taken shall depend on the possibility of products being contaminated by salmonella and enterobacter sakazakii.

Appendix C

Commercial sterile operation guide on liquid formulated foods for special medical purposes

C.1 General requirements

Besides the regulations which are applicable to liquid formulated foods for special medical purposes in the standard, the commercial sterile operation of liquid products shall also be in accordance with C.2 to C.6.

C.2 Product techniques

C.2.1 Operation of all techniques shall be performed under the good conditions which are in accordance with the technique requirements.

C.2.2 Processes which are in contact with the air (such as weighing and batching), bottling rooms and auxiliary areas with special requirements for cleanness shall meet the requirements for liquid formulated foods for special medical purposes.

C.2.3 All the delivery pipes and equipment for the products shall be airtight.

C.2.4 Liquid products shall be filtered during the production. Filter materials with no fiber exfoliating and meeting the sanitary requirements shall be used. Asbestos shall not be used as filters.

C.2.5 Control measures shall be formulated to prevent extraneous matters from entering the products during the production.

C.3 Washing, sterilization and clean-keeping of packaging containers

C.3.1 Food containers, packing materials, detergents and disinfectants which are in accordance with the national food safety standard and the permission of health administrative departments shall be used.

C.3.2 The washed packaging materials, containers and equipment shall not be contaminated for the second time.

C.3.3 Packaging materials used in sterile filling systems shall be sterilized in a proper way. They shall be washed and dried when necessary. After sterilization, they shall be stored in clean work areas for use. Sterilization shall be performed again when storage period exceeds the set time limit.

C.4 Washing, sterilization and clean-keeping of product processing equipment for aseptic filling technology

C.4.1 Before production, water, filtered steam, fresh distilled water or other proper treating agents under high temperature pressing shall be used to keep the products under high temperatures or clean and sterilize all the pipes, valves, pumps, feeding hoppers and other product contact surfaces in the lower reaches of the pipeline. It shall be ensured that all surfaces that are in direct contact with the products shall meet the requirements of sterile filling and remain until the production is over.

C.4.2 Sterile warehouses for filling and packaging equipment shall be cleaned and sterilized, and meet the requirements of aseptic filling before the production. They shall remain until the production is over. When sterilization fails, sterile warehouses shall be sterilized again. Key indicators such as the time, temperature and disinfectant concentration shall be controlled and recorded when sterilization is carried out.

C.5 Product filling

C.5.1 Automatic mechanical devices shall be used for product filling. No manual operation shall be allowed.

C.5.2 As to all products which need to be sterilized after filling, the time from bottling to sterilization shall be limited within the time required by the technological procedures.

C.5.3 As to the sterilized products, monitoring standards for pollution levels of product microorganisms before sterilization shall be determined according to the effects of the used sterilization methods and monitored on a regular basis

C.6 Heat treatment of products

C.6.1 Proper heat treatment processes shall be formulated according to the quality of product heating and the dynamics to kill specific target microorganisms. Products shall be heated to the sterilizing temperature and remain at the temperature for some time to achieve commercial sterilization. All the heat treatment processes are verified so as to ensure the reproducibility and reliability of the techniques.

C.6.2 Liquid products shall be sterilized with thermal sterilization as much as possible. Thermal sterilization is usually divided into damp-heat sterilization and dry-heat sterilization. The products to be sterilized in the chambers of sterilizing equipment and the way to carry materials shall be determined via verification. Time-temperature curve of every sterilization process shall be recorded. There shall be definite ways to distinguish sterilized products from products that are to be sterilized. Sterilization records shall be the basis to permit the group of products.

C.6.3 Constant flowing products which adopt the aseptic filling technique shall remain at the sterilizing temperature in the high temperature sterilization places or within the pipe flowing time so as to achieve commercial sterilization. So, product types, as well as the size and design of flow rate, pipeline length and high temperature sterilization places of every product shall be accurately determined. When steam injection or other steam filling way is used, product size increase due to the water caused by vapor condensation shall be considered.