식품의약품안전처
MINISTRY OF FOOD AND DRUG SAFETY

Standards and Specifications for Food Utensils, Containers and Packages

Ministry of Food and Drug Safety
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Note to reader : If there should be any differences between the original Korean texts and English translation, the original Korean texts shall prevail. For further information, please contact Food Additive Standards Division, Ministry of Food and Drug Safety.

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I. General Rules

1. Purpose of the standards and specifications

The Standards and Specifications have been established to promote the distribution of safe utensils, containers and packages, and to contribute to securing consumer safety through the prevention of harm on public welfare, by establishing specifications etc. for suspected hazardous materials that can be migrated to food from the utensils, containers and packages that has direct contact with food or food additives.

2. Scope of the standards and specifications

「Standards for the Manufacture of Utensils, Containers and Packages」 and 「Specifications for Utensils, Containers and Packages and their Raw Materials」 in accordance with Article 9 (1) of the Food Sanitation Act.

3. Structure of the standards and specifications

a. The Standards and Specifications shall be composed of the General Rules, Common Standards and Specifications, Specifications for Individual Material, and Test Method.

b. The Standards and Specifications for Individual material shall be classified into synthetic resin products, cellophane products, rubber products, paper or processed paper products, metal products, wood products, glass products, ceramic products, porcelain enamel and pottery products, and starch products, and shall be composed of the definition, residue specifications, migrant specifications, and test method.

1) Definition shall be composed of substance of the raw material, its content, and process method, etc. that are used in manufacture, in order to regulate the scope of relevant materials.

2) Residue specifications and migrant specifications shall provide the specifications etc. regarding hazardous substances that may remain as residue within the

material which has been used as raw material during the course of manufacturing the utensils, containers or packages, or that may have migrated from the utensils, containers or packages to food.

3) Test method provides for testing methods for each item which have determined standards in the form of common standards and specifications, residue specifications and migrant specifications.

c. Test Method shall be composed of general principles and test method by item.

II. Common Standards and Specifications

1. Common Manufacturing Standards

a. Machinery, apparatus, and subsidiary facilities that are used for the manufacturing and processing of the utensils, containers and packages shall be sanitarily maintained and administered.

b. Raw materials that are used for the utensils, containers and packages shall be of good quality and not contaminated with poisonous or hazardous substances, and thus confirmed of their safety and integrity.

c. Di-(2-ethylhexyl)phthalate(DEHP) shall not be used in the manufacture of utensils, containers and packages, except there is no possibility that di-(2-ethylhexyl)phthalate migrates into foods.

d. Tin plating used in food contact surface of utensils, containers and packages shall not contain more than 0.1% of lead and the test method shall conform to IV. 2. 2-1 Lead Test Method a. Residue Test

e. Metals for manufacture or repair in food contact surface of utensils, containers and packages shall not contain more than 0.1% of lead or 5% of antimony and the test method shall conform to IV. 2. 2-1 Lead Test Method a. Residue Test and 2-10 Antimony Test Method a. Residue Test.

f. Solder for manufacture or repair of utensils, containers and packages shall not contain more than 0.1% of lead and the test method shall conform to IV. 2. 2-1 Lead Test Method a. Residue Test.

g. Electrode of utensils applied the current to food directly shall not make of metals other than iron, aluminum, platinum, titanium, and stainless steel.

h. Utensils, containers and packages of synthetic resin products, cellophane products, rubber products, paper products, and starch products shall not contain not more than 100 mg/kg(as sum) of lead, cadmium, mercury and hexavalent chromium and the test method shall conform to IV. 2. 2-1 Lead Test Method a. Residue Test, 2-2 Cadmium Test Method a. Residue Test, 2-3 Mercury Test Method, 2-4 Hexavalent Cromium Test Method a. Residue Test.

i. Food contact surface of utensils, containers and packages which are made of copper or copper alloy must be properly treated with tin coating, silver coating or other methods to ensure hygiene safety. However, exempt materials having specific its own gloss and plate concerned to be peeled using at high temperature.

j. Colorant for manufacturing of utensils, containers and packages shall not use other than permitted as food additives, except the case that colorant are melted glaze, glass, enamel, or there is no possibility that colorant migrates into foods.

k. Food contact surface shall not be printed in the manufacture of utensils, containers and packages. Printing inks applied to the non food-contact side must be sufficiently dried and in this case the benzophenone as ink compounds shall not migrate more than 0.6 mg/L, and the test method shall conform to IV. 2. 2-13 Benzophenone Test Method. In case of flexible packages, among synthetic polymer packages with printing non food-contact side, residual toluene as ink compounds shall not be more than 2 mg/m^2, and the test method shall conform to IV. 2. 2-14 Toluene Test Method.

l. The Head of the Ministry of Food and Drug Safety may review the usability of the raw material in case a new fact related to the safety of the raw material has been discovered or presented.

2. Common specifications

a. Utensils, containers and packages shall not have a structure, in which their contents are easily contaminated physically or chemically.

b. When recollecting and reusing food containers or packages, containers or packages shall be used only after confirming that there are no residue impurities etc. after cleaning with water conforming to the water quality standard as provided in the Drinking Water Management Act.

3. Usage Specifications

a. Di-(2-ethylhexyl)adipate(DEHA) shall not be used in the manufacture of cling wraps, except there is no possibility that di-(2-ethylhexyl)adipate migrates into foods.

b. Di-n-butylphthalate(DBP), benzyl-n-butylphthalate(BBP) and bisphenol A(BPA) shall not be used in the manufacture of feeding bottles(including nipples).

c. The interval of marking indicating volume on feeding bottle(except glass and metal) should be 10 mL, except the lowest mark of the scale is difficult. The highest mark of scale on the feeding bottle and their error tolerance is as the following table(However, if the highest mark of scale is not showed in the following table, the nearest value specified in the table shall be applied and higher value in the middle).

[Table] The highest mark of scale of the feeding bottle and error tolerance(unit : mL)

Highest mark of scale		50	100	120	150	200	240	250	300
Error tolerance	Glass	±4	±6.5	±7	±8	±9	±10	±10	±12
	Synthetic polymer	±3	±4	±4	±4	±4	±5	±5	±6

d. The usage and thermal shock strength(endurable temperature difference) of heat-cooking glassware is as follows in the following table, and when testing according to IV. 2. 2-5 Heat Impact Strength Test Method, there shall be no brokenness or fissures.

[Table] The usage and thermal shock strength(endurable temperature difference) of glassware used for cooking purposes

Usage		Thermal shock strength (Endurable temperature difference)
For direct heating	Used by direct exposure to flame for such purpose as cooking with heat, able to withstand sudden heating and cooling	not less than 400℃

	Used by direct exposure to flame for such purpose as cooking with heat	not ness than 150℃
For oven	Used without direct exposure to flame for such purpose as cooking with heat	not less than 120℃
For microwave oven	Used by microwave for such purpose as cooking with heat	not less than 120℃
For boiling water	Used for purpose other than the above, able to sufficiently withstand a thermal impact of the degree caused by boiling water	not less than 120℃

4. Application of the Standards and Specifications for Utensils, Containers and Packages

a. The specifications for utensils, containers and packages shall, in principle, apply together with “II. Common Standards and Specifications” and “III. Specifications for Utensils, Containers and Packages for Individual Material”. Provided that, when there is very little need or effectiveness in doing so, considering the feature of the utensils, containers or packages, the applicable standards and specifications shall be selectively chosen according to their level of importance.

b. As for the container or package where food-grade substances such as starch, glycerin, wax etc. has contact with the food-contact surface, the specifications on non-volatile residues may not apply.

c. As for the utensils, containers or packages that are not hazardous since the material that has contact with food or food additives are made of rock or untinged glass (except for heat-cooking glassware and leaded crystal glassware) and other natural raw materials, the specifications may not apply.

d. When a product contains 50% each of basic polymer that composes synthetic resin products, and thus belongs to two types of definitions of material which is categorized within the Specifications, relevant residue specifications shall all be applied. In case the specifications overlap, the more stringent specifications shall apply.

e. When the utensils, containers and packages are composed of more than two types of material, and accordingly difficult to respectively apply the relevant b. residue specifications by categorizing per type of material, all specifications for the materials contained in the product shall be applied, and in case the specifications overlap, the more stringent specifications shall apply.

f. The standards and specifications for the utensils, containers and packages that are not listed in this Standard and Specifications shall be determined in accordance with the 「Approval Standard of Temporary Standards and Specifications for Food etc.」

5. (In)Conformity Determination of Standards and Specifications

a. Utensils, containers and packages shall be determined of its conformity or inconformity (hereinafter referred to as "(in)conformity") in accordance with the standards and specifications that are established by the Head of the Ministry of Food and Drug Safety.

b. The determination of the standards and specifications shall, in principle, be conducted by the test methods as regulated within this Standards and Specifications. However, in case a new method is accepted as being more precise and accurate than the test method, the new method may be used.

c. The determination of (in)conformity in accordance with the test method shall be concluded after comparing the test value (value obtained from the test), which is obtained by rounding off the value that has been obtained by calculating to the (n+1) digit more than the standard value (value set by the Standards and Specifications; n digits), with the standard value.

6. Sample Extraction and Treatment Method

a. Significance of sample extraction

Sample extraction shall be conducted by the food sanitation inspector by obtaining a specimen from the inspection object in accordance with the Food Sanitation Act, and conducting inspections regarding conformity to the Standards and Specifications and safety from contaminants etc., which shall esult in administrative measures in accordance with the inspection results. Therefore, the selection of the inspection object, the extraction, treatment, transportation, and test inspection of the inspection object shall be conducted in an efficient and scientific manner. Accordingly, since requesting for inspection of the extracted sample to the food sanitation inspection agency is significantly important, the food sanitation inspector shall perform one's duties with sufficient knowledge on the method of sample extraction and treatment.

b. Definition of terms

1) Sample: Specimen extracted from the inspection object.

2) Inspection object: A subject of sample extraction from the utensils, containers or packages that is produced, manufactured or processed under the same conditions.

3) Large-sized packaged sample: A subject of inspection that is packaged in bulk, not in the form of distribution and sales to end-consumers.

c. General rules on sample extraction

1) Extraction of samples shall be conducted by the food sanitation inspector in accordance with Article 32 of the Food Sanitation Act and Article 16 of the Enforcement Decree of the same Act.

2) The sample shall be extracted in a minimum amount which can represent the population of the inspection object in consideration of the purpose of inspection and inspection items.

3) When extracting the sample, the sample shall have representativeness of the

inspection object, by using the table of random sampling numbers in [Annex Table 1]. Provided that, however, when there is a reason for not being able to use the table, the collector may select and extract the inspection object.

4) When extracting the sample, the sample shall be extracted only in necessary amounts for conducting the specification inspection per type of material.

5) The extracted sample shall be cautiously treated so as not to damage the inspection object, and the extracted sample shall be sealed so that without breaking the seal, it cannot be opened.

6) When the utensils, containers or packages is of the same type of material and ground color, but different in terms of simply usage, shape and size or name of product, the representative sample may serve as the inspection object. Provided that, however, in the case of set products containing different type of material and ground color, the sample to be extracted may be the set product which is the unit of sale.

d. Method of extraction and treatment of samples

When extracting the sample, due consideration shall be made of the physical and chemical state of the sample, including the purpose of inspection, and the type and amount of the object utensils, containers and packages.

1) Method of sample extraction

a) Extraction of packaged samples

(1) Utensils, containers and packages that are put in boxes for distribution shall be, as much as possible, extracted in its intact form, without being released.

(2) Utentils, containers and packages that are put in large-sized containers and packages may be extracted in its partial form that can represent the entire inspection object.

b) Extraction of container samples

(1) When multiple containers are a single inspection object, the container shall be released in accordance with the following Table in order for the sample

to be extracted. Provided that, however, in consideration of the number of locations for sample extraction as regulated in this Method of Extraction and Treatment of Samples, the number of releases may be increased or reduced.

Number of containers	1-3	4-6	7-10	11-20	21-30	31-50	Not less than 51
Number of releases	1	Not less than 2	Not less than 3	Not less than 4	Not less than 6	Not less than 8	Not less than 10

(2) When it is not possible to extract samples that can represent the population in the form loaded on the container, one-third (1/3) of the relevant utensils, containers or packages may be taken out in order to make inspection possible, prior to sample extraction.

2) Filling in the sample extraction statement

The food sanitation inspector shall, when extracting the sample, attach the sample extraction statement as provided in [Annex Table 2] together with the relevant sample. However, this regulation may not apply when it is recognized that there is no problem with the standards and specifications inspection by omitting the sample extraction statement.

3) Attachment of the identification table

When conducting inspection on imported food (except for inspection by collection from distribution), the identification table as provided in [Annex Table 3], that demonstrates the collection of the sample after sample extraction, shall be attached to the relevant product in the bonded warehouse etc.

4) Method of transportation of sample

a) The extracted sample shall be transported cautiously to the inspection room, without contaminating, destroying, damaging or transforming, etc. of the sample.

b) When in case the sample is transported long-distance or using mass transportation means, special care shall be taken not to damage the sample at the packaging stage.

7. Storage and Distribution Standards

a. Utensils, containers and packages shall be treated and sold in a sanitary manner, and they shall not be located in unsanitary storage and sales locations.

b. The location for treating utensils, containers and packages shall be where protection from snow, rain, etc. can be provided, and shall not be stored together with hazardous chemicals, agricultural chemicals, and toxic substances etc.

c. Products shall be stored and distributed in cool locations so as not to cause physical transformations or rust.

III. Specifications for Individual Materials

1. Synthetic Resin

1-1 Poly(vinyl chloride)(PVC)

a. Definition

Poly(vinyl chloride) is containing above 50% of vinyl chloride among the base polymers.

b. Residue Specification(mg/kg)

1) Vinyl chloride : not more than 1
2) Dibutyltin compound : not more than 50(as dibutyltin dichloride)
3) Cresol esters of phosphoric acid : not more than 1,000

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 150 if n-heptane is used as the food simulant)
4) Di-butylphthalate : not more than 0.3
5) Benzyl-n-butylphthalate : not more than 30
6) Di-(2-ethylhexyl)phthalate : not more than 1.5
7) Di-n-octylphthalate : not more than 5
8) Diisononylphthalate and Diisodecylphthalate : not more than 9(as sum)
9) Di-(2-ethylhexyl)adipate : not more than 18

d. Test Method

1) Vinyl chloride : IV. 2. 2-16 Vinyl chloride Test Method a. Residue Test
2) Dibutyltin compound : IV. 2. 2-17 Dibutyltin compound Test Method

3) Cresol esters of phosphoric acid : IV. 2. 2-18 Cresol esters of phosphoric acid Test Method
4) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
5) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
6) Non-volatile Residue : IV. 2. 2-8 Non-volatile residue Test Method
7) Di-n-butylphthalate, Benzyl-n-butylphthalate, Di-(2-ethylhexyl)phthalate, Di-n-octylphthalate, Diisononylphthalate, Diisodecylphthalate and Di-(2-ethyl hexyl)adipate : IV. 2. 2-19 Di-n-butylphthalate, Benzyl-n-butylphthalate, Di-(2-ethylhexyl)phthalate, Di- n-octylphthalate, Diisononylphthalate, Diisodecylphthalate, and Di-(2-ethyl hexyl)adipate Test Method

1-2 Polyethylene(PE) and Polypropylene(PP)

a. Definition

Polyethylene is containing above 50% of ethylene among the base polymers.

Polypropylene is containing above 50% of propylene among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 150 if n-heptane used as the food simulant and using temperature is not more than 100℃)
4) 1-Hexene : not more than 3(in compliance with only the polyethylene)
5) 1-Octene : not more than 15(in compliance with only the polyethylene)

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 1-Hexene and 1-Octene : IV. 2. 2-20 1-Hexene and 1-Octene Test Method

1-3 Polystyrene(PS)

a. Definition

Polystyrene is containing above 50% of styrene or α-methyl styrene among the base polymers and including foamed polystyrene.

b. Residue Specification(mg/kg)

1) Volatile compound(as sum of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene) : not more than 5,000(However, in case of formed polystyrene used to boiling water, total amount of volatile organic compounds shall be not more than 2,000 and amount of styrene and ethyl benzene among total volatile organic compounds are not more than 1,000 respectively)

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residues : not more than 30(However, not more than 240 if n-heptane is used as the food simulant)

d. Test Method

1) Volatile compound : IV. 2. 2-21 Volatile compound Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-4 Poly(vinylidene chloride)(PVDC)

a. Definition

Poly(vinylidene chloride) is containing above 50% of vinylidene chloride among the base polymers.

b. Residue Specification(mg/kg)

1) Vinylidene chloride : not more than 6

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Barium : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

d. Test Method

1) Vinylidene chloride : IV. 2. 2-22 Vinylidene chloride Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Barium : IV. 2. 2-23 Barium Test Method
4) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
5) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-5 Poly(ethyleneterephthalate)(PET)

a. Definition

Poly(ethyleneterephthalate) is containing above 50% of polymers that terephthalic acid or dimethyl terephthalate react with ethyleneglycol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Antimony : not more than 0.04
5) Germanium : not more than 0.1
6) Terephthalic acid : not more than 7.5
7) Isophthalic acid : not more than 5

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Antimony : IV. 2. 2-10 Antimony Test Method b. Migrant Test Method
5) Germanium : IV. 2. 2-24 Germanium Test Method
6) Terephthalic acid and Isophthalic acid : IV. 2. 2-25 Terephthalic acid and Isophthalic acid Test Method

1-6 Phenol-formaldehyde resin(PF)

a. Definition

Phenol-formaldehyde resin is containing above 50% of polymers consisting of phenol and formaldehyde among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Non-volatile residue : not more than 30
3) Phenol : not more than 5
4) Formaldehyde : not more than 4

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
3) Phenol : IV. 2. 2-26 Phenol Test Method
4) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method

1-7 Melamine-formaldehyde resin(MF)

a. Definition

Melamine-formaldehyde resin is containing above 50% of polymers consisting of melamine and formaldehyde among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Non-volatile residue : not more than 30
3) Phenol : not more than 5
4) Formaldehyde : not more than 4
5) Melamine : not more than 30

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
3) Phenol : IV. 2. 2-26 Phenol Test Method
4) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method
5) Melamine : IV. 2. 2-26 Melamine Test Method

1-8 Urea-formaldehyde resin(UF)

a. Definition

Urea-formaldehyde resin is containing above 50% of polymers consisting of urea and formaldehyde among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Non-volatile residue : not more than 30
3) Phenol : not more than 5
4) Formaldehyde : not more than 4

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
3) Phenol : IV. 2. 2-26 Phenol Test Method
4) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method

1-9 Polyacetal(polyoxymethylene(POM))

a. Definition

Polyacetal is containing above 50% of polymers consisting of formaldehyde and trioxymethylene among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residues : not more than 30
4) Formaldehyde : not more than 4

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method

1-10 Acrylic resin

a. Definition

Acrylic resin is containing above 50% of acrylic acid, methacrylic acid, acrylate or methyl methacrylate etc. among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residues : not more than 30
4) Methylmethacrylate : not more than 6(in compliance with only the synthetic polymer containing above 50% of methylmethacrylate among the base polymers)

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Methyl methacrylate : IV. 2. 2-29 Methyl methacrylate Test Method

1-11 Polyamide(PA)

a. Definition

Polyamide is containing above 50% of polymers that lactam, amino carboxylate or dibasic acid react with diamine among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Caprolactam : not more than 15
5) Primary aromatic amine(sum as aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) : not more than 0.01
6) Ethylenediamine : not more than 12
7) Hexamethylenediamine : not more than 2.4
8) Laurolactam : not more than 5

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Caprolactam and Laurolactam : IV. 2. 2-30 Caprolactam and Laurolactam Test Method
5) Primary aromatic amine(sum as aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) : IV. 2. 2-31 Primary aromatic amine(in compliance with only the aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) Test Method
6) Ethylendiamine and Hexamethylendiamine : IV. 2. 2-32 Ethylendiamine and Hexamethylendiamine Test Method

1-12 Polymethylpentene(PMP)

a. Definition

Polymethylpentene is containing above 50% of methylpentene among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 120 if n-heptane is used as the food simulant)
4) 4-methyl-1-pentene : not more than 0.05

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 4-methyl-1-pentene : IV. 2. 2-33 4-methyl-1-pentene Test Method

1-13 Polycarbonate(PC)

a. Definition

Polycarbonate is containing above 50% of polymers that bisphenol A react with diphenyl carbonate or carbonyl chloride among the base polymers.

b. Residue Specification(mg/kg)

1) Amine(as sum of triethylamine and tributylamine) : not more than 1

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : not more than 2.5(However, migrant specification of bisphenol A is not more than 0.6)
5) Diphenylcarbonate : not more than 0.05

d. Test Method

1) Amine(as sum of triethylamine and tributylamine) : IV. 2. 2-34 Amine(in compliance with only the triethylamine and tributylamine)
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
5) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : IV. 2. 2-35 Bisphenol A(including phenol and p-tert-butylphenol) Test Method
6) Diphenylcarbonate : IV. 2. 2-36 Diphenylcarbonate Test Method

1-14 Poly(vinylalcohol)(PVA)

a. Definition

Poly(vinylalcohol) is containing above 50% of vinyl alcohol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Vinyl acetate : not more than 12

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Vinyl acetate : IV. 2. 2-37 Vinyl acetate Test Method

1-15 Polyurethane(PU)

a. Definition

Polyurethane is containing above 50% of polymers consisting of isocyanate and polyol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residues : not more than 30
4) Isocyanate : not more than 0.1
5) 4,4'-methylenedianiline : not less than 0.01

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Isocyanate : IV. 2. 2-38 Isocyanate Test Method
5) 4,4'-Methylenedianiline : IV. 2. 2-31 Primary aromatic amine(in compliance with only the aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) Test Method

1-16 Polybutene-1(PB-1)

a. Definition

Polybutene-1 is containing above 50% of butene-1 among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 150 if n-heptane used as the food simulant and using temperature is below 100℃, and not more than 120 if n-heptane used as the food simulant and using temperature is above 100℃)

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-17 Acrylonitrile-butadiene-styrene copolymer(ABS) and Acrylonitrile-styrene copolymer(AS)

a. Definition

Acrylonitrile-butadiene-styrene copolymer is containing above 60% of copolymers consisting of styrene(including α-methylstyrene) and acrylonitrile, and butadiene rubber among the base polymers. Acrylonitrile-styrene copolymer is containing above 50% of styrene(including α-methylstyrene) polymer, plus acrylonitrile polymer among the base polymers.

b. Residue Specification(mg/kg)

1) Volatile compound(as sum of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene) : not more than 5,000
2) 1,3-butadiene : not more than 1(in compliance with only the acrylonitrile-butadiene-styrene copolymer)

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 240 if n-heptane is used as the food simulant)
4) Acrylonitrile : not more than 0.02

d. Test Method

1) Volatile compound : IV. 2. 2-21 Volatile compound Test Method
2) 1,3-butadiene : IV. 2. 2-39 1,3-butadiene Test Method
3) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
4) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
5) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
6) Acrylonitrile : IV. 2. 2-40 Acrylonitrile Test Method

1-18 Polymethacrylstyrene(MS)

a. Definition

Polymethacrylstyrene is containing above 20% of methyl methacrylate and styrene(including α-methylstyrene), respectively, among the base polymers and the sum of 2 chemicals is above 60%.

b. Residue Specification(mg/kg)

1) Volatile compound(as sum of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene) : not more than 5,000

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30(However, not more than 240 if n-heptane is used as the food simulant)
4) Methylmethacrylate : not more than 6(in compliance with only the synthetic polymer containing above 50% of methylmethacrylate among the base polymers)

d. Test Method

1) Volatile compound : IV. 2. 2-21 Volatile compound Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
5) Methylmethacrylate : IV. 2. 2-29 Methylmethacrylate Test Method

1-19 Poly(butyleneterephthalate)(PBT)

a. Definition

Poly(butyleneterephthalate) is containing above 50% of polymers that terephthalic acid or dimethyl terephthalate react with buthylene glycol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Terephthalic acid : not more than 7.5
5) Isophthalic acid : not more than 5
6) 1,4-butanediol : not more than 5

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Terephthalic acid and Isophthalic acid : IV. 2. 2-25 Terephthalic acid and Isophthalic acid Test Method
5) 1,4-butanediol : IV. 2. 2-41 1,4-butanediol Test Method

1-20 Polyarylsulfone(PASF)

a. Definition

Polyarylsulfone is containing above 50% of polymers consisting of 4,4'-dichlorodiphenylsulfone and bisphenol A among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : not more than 2.5(However, migrant specification of bisphenol A is not more than 0.6)
5) 4,4'-Dichlorodiphenylsulfone : not more than 0.05

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : IV. 2. 2-35 Bisphenol A(including phenol and p-tert-butylphenol) Test Method
5) 4,4'-Dichlorodiphenyl sulfone : IV. 2. 2-42 4,4'-Dichlorodiphenyl sulfone Test Method

1-21 Polyarylate(PAR)

a. Definition

Polyarylate is containing above 50% of polymers that terephthalic acid or isophthalic acid react with bisphenol A among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Terephthalic acid : not more than 7.5
5) Isophthalic acid : not more than 5
6) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : not more than 2.5(However, migrantion specification of bisphenol A is not more than 0.6)

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Terephthalic acid and Isophthalic acid : IV. 2. 2-25 Terephthalic acid and Isophthalic acid Test Method
5) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : IV. 2. 2-35 Bisphenol A(including phenol and p-tert-butylphenol) Test Method

1-22 Hydroxybutyl polyester(HBP)

a. Definition

Hydroxybutyl polyester is containing above 50% of copolymers consisting of hydroxybenzoic acid, aromatic dicarbonate, and aromatic diol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-23 Polyacrylonitrile(PAN)

a. Definition

Polyarylonitrile is containing above 50% of acrylonitrile among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Acrylonitrile : not more than 0.02

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Acrylonitrile : IV. 2. 2-40 Acrylonitrile Test Method

1-24 Fluorocarbon resin(FR)

a. Definition

Fluorocarbon resin is containing above 50% of fluoro-containing monomers among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-25 Poly(phenylene ether)(PPE)

a. Definition

Poly(phenylene ether) is containing above 60% of 2,6-dimethylphenol and styrene among the base polymers.

b. Residue Specification(mg/kg)

1) Volatile compound(as sum of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene) : not more than 5,000

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

D. Test Method

1) Volatile compound : IV. 2. 2-21 Volatile compound Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-26 Ionomer resin

a. Definition

Ionomer is a copolymer of ethylene and methyl acrylic acid, in which zinc, sodium, potassium, calcium and ammonium etc. are cross-linked to carboxyl group.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-27 Ethylene-vinylacetate copolymer(EVA)

a. Definition

Ethylene-vinylacetate is a copolymer consisting of ethylene and vinylacetate.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residues : not more than 30
4) Vinyl acetate : not more than 12

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Vinyl acetate : IV. 2. 2-37 Vinyl acetate Test Method

1-28 Methylmethacrylate-acrylonitrile-butadiene-styrene copolymer(MABS)

a. Definition

Methylmethacrylate-acrylonitrile-butadiene-styrene copolymer is containing above 60% of copolymers consisting of methacrylate, acrylonitrile, butadiene, and styrene(including α-methylstyrene) among the base polymers.

b. Residue Specification(mg/kg)

1) Volatile compound(as sum of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene) : not more than 5,000
2) 1,3-butadiene : not more than 1

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Methylmethacrylate : not more than 6(in compliance with only the synthetic polymer containing above 50% of methylmethacrylate among the base polymers)
5) Acrylonitrile : not more than 0.02

d. Test Method

1) Volatile compound : IV. 2. 2-21 Volatile compound Test Method
2) 1,3-butadiene : IV. 2. 2-39 1,3-butadiene Test Method
3) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
4) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
5) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Methylmethacrylate : IV. 2. 2-29 Methyl methacrylate Test Method
7) Acrylonitirile : IV. 2. 2-40 Acrylonitirile Test Method

1-29 Poly(ethylenenaphthalate)(PEN)

a. Definition

Poly(etylrenenaphthalate) is containing above 50% of polymers consisting of 2,6-dimethylnaphthalene dicarboxylate and ethylene glycol among the base polymer.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) 2,6-dimethylnaphthalene dicarboxylate : not more than 0.05

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 2,6-dimethylnaphthalene dicarboxylate : IV. 2. 2-43 2,6-dimethylnaphthalene dicarboxylate Test Method

1-30 Epoxy resin

a. Definition

Epoxy Resin is a polymer consisting of mainly bisphenol A and epichlorohydrine.

b. Residue Specification(mg/kg)

1) Amine(as sum of triethylamine and tributylamine) : not more than 1

c. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : not more than 2.5(However, migrant specification of bisphenol A is not more than 0.6)
5) Bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) : not more than 1
6) Bisphenol F diglycidyl ether(including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) : not more than 1
7) Epichlorohydrin : not more than 0.5
8) 4,4-methylenedianiline : not more than 0.01

d. Test Method

1) Amine(as sum of triethylamine and tributylamine) : IV. 2. 2-34 Amine(in compliance with only the triethylamine and tributylamine)
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
5) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : IV. 2. 2-35 Bisphenol A(including phenol and p-tert-butylphenol) Test Method

6) Bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) and bisphenol F diglycidyl ether (including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) : IV. 2. 2-44 Bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) and bisphenol F diglycidyl ether (including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) Test Method
7) Epichlorohydrine : IV. 2. 2-45 Epichlorohydrine Test Method
8) 4,4'-Methylenedianiline : IV. 2. 2-31 Primary aromatic amine(in compliance with only the aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) Test Method

1-31 Poly(phenylenesulfide)(PPS)

a. Definition

Poly(phenylenesulfide) is containing above 50% of polymers consisting of 1,4-dichlorobenzene and sodium sulfide among the base polymer.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) 1,4-Dichlororbenzene : not more than 12

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 1,4-Dichlororbenzene : IV. 2. 2-46 1,4-Dichlororbenzene Test Method

1-32 Poly(ethersulfone)(PES)

a. Definition

Poly(ethersulfone) is containing above 50% of polymers that 4,4'-dihydroxy diphenylsulfone or 4,4'-dihydroxybiphenyl react with 4,4'-dichlorodiphenylsulfone among the base polymers, and not using bisphenol A.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) 4,4'-dichlorodiphenylsulfone : not more than 0.05
5) 4,4'-dihydroxydiphenylsulfone : not more than 0.05

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 4,4'-dichlorodiphenylsulfone : IV. 2. 2-42 4,4'-dichlorodiphenylsulfone Test Method
5) 4,4'-dihydroxydiphenylsulfone : IV. 2. 2-47 4,4'-dihydroxydiphenylsulfone Test Method

1-33 Poly(cyclohexane-1,4-dimethylene terephthalate)(PCT)

a. Definition

Poly(cyclohexane-1,4-dimethylene terephthalate) is containing above 50% of polymers that terephthalic acid or dimthyl terephthalate react with 1,4-cyclohexane dimethanol among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Antimony : not more than 0.04
5) Terephthalic acid : not more than 7.5
6) Isophthalic acid : not more than 5

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Antimony : IV. 2. 2-10 Antimony Test Method b. Migrant Test Method
5) Terephthalic acid and Isophthalic acid : IV. 2. 2-25 Terephthalic acid and Isophthalic acid Test Method

1-34 Polyimide(PI)

a. Definition

Polyimide is containing above 50% of polymers that aromatic or aliphatic dianhydride react with aromatic or aliphatic diamine among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

1-35 Polyetheretherketone(PEEK)

a. Definition

Polyetheretherketone is containing above 50% of polymers consisting of 4,4'-dihalogenated diphenylketone and hydroquinone among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Hydroquinone : not more than 0.6

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Hydroquinone : IV. 2. 2-48 Hydroquinone Test Method

1-36 Polylactide(poly(lactic acid), PLA)

a. Definition

Polylactide is containing above 50% of lactic acid among the base polymers.

b. Migrant Specification(mg/L)

1) In case starch is not included
   a) Lead : not more than 1
   b) Consumption of potassium permanganate : not more than 10
   c) Non-volatile residues : not more than 30
2) In case starch is included
   a) Arsenic : not more than 0.1 (as As_2O_3)
   b) Lead : not more than 1
   c) Consumption of potassium permanganate : not more than 10
   d) Formaldehyde : not more than 4
   e) Fluorescence whitening agent : shall not be detected

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
3) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method
4) Arsenic : IV. 2. 2-9 Arsenic Test Method
5) Fluorescence whitening agent : IV. 2. 2-53 Fluorescence whitening agent Test Method

1-37 Butylenesuccinate-adipate copolymer(PBSA)

a. Definition

Butylenesuccinate-adipate is containing above 60% of copolymers consisting of succinic acid, adipic acid, and 1,4-butanediol among the base polymers.

b. Migrant Specification(mg/L)

1) In case starch is not included
   a) Lead : not more than 1
   b) Consumption of potassium permanganate : not more than 10
   c) Non-volatile residue : not more than 30
   d) 1,4-Butanediol : not more than 5
2) In case starch is included
   a) Arsenic : not more than 0.1 (as As_2O_3)
   b) Lead : not more than 1
   c) Consumption of potassium permanganate : not more than 10
   d) Formaldehyde : not more than 4
   e) Fluorescence whitening agent : shall not be detected
   f) 1,4-Butanediol : not more than 5

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) 1,4-Butanediol : IV. 2. 2-41 1,4-Butanediol Test Method
5) Arsenic : IV. 2. 2-9 Arsenic Test Method
6) Formaldehyde : IV. 2. 2-26 Formaldehyde Test Method
7) Fluorescence whitening agent : IV. 2. 2-53 Fluorescence whitening agent Test Method

1-38 Cross-linked polyester resin

a. Definition

Crosses-linked polyester resin is containing above 50% of polymers that polyol or epoxide react with unsaturated dibasic acid among the base polymers.

b. Migrant Specification(mg/L)

1) Lead : not more than 1
2) Consumption of potassium permanganate : not more than 10
3) Non-volatile residue : not more than 30
4) Terephthalic acid : not more than 7.5
5) Isophthalic acid : not more than 5.0

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
4) Terephthalic acid and Isophthalic acid : IV. 2. 2-25 Terephthalic acid and Isophthalic acid Test Method

2. Cellophane

a. Definition

Cellophane is a regenerated cellulose film which is viscosified pulp and then coagulated, and includes cellophane acquired by adding proper substances to raw materials or treating surface such as a coating in order to achieve technical goals.

b. Migrant specification(mg/L)

1) Arsenic : not more than 0.1(As As_2O_3)
2) Lead : not more than 1
3) Non-volatile residue: not more than 30

c. Test method

1) Arsenic : IV. 2. 2-9 Arsenic Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

3. Rubber

a. Definition

Rubber is containing above 50% of natural rubber, synthetic rubber(including silicon rubber and butadiene rubber), their latex or thermoplastic elastomer(TPE) among the base polymers.

b. Residue specification(mg/kg)

1) Lead
   a) Rubber except rubber nipple : not more than 100
   b) Rubber nipple : not more than 10
2) Cadmium
   a) Rubber except rubber nipple : not more than 100
   b) Rubber nipple : not more than 10
3) 2-Mercaptoimidazoline (in compliance with only the rubber containing chlorine) : Not detected
4) 1,3-butadiene : not more than 1(in compliance with only the rubber material that contain 50% or more of 1,3-butadiene in base polymer)

c. Migrant specification

1) Lead : not more than 1 mg/L
2) Non-volatile residue
   a) Rubber except rubber nipple : not more than 60 mg/L
   b) Rubber nipple : not more than 40 mg/L
3) Phenol : not more than 5 mg/L
4) Formaldehyde : not more than 4 mg/L
5) Zinc
   a) Rubber except rubber nipple : not more than 15 mg/L
   b) Rubber nipple : not more than 1 mg/L
6) N-Nitrosamines(as sum of N-nitrosodimethylamine, N-nitrosodiethylamine, N-

nitrosodi-n-propyl amine, N-nitrosodi-n-buthylamine, N-nitrosopiperidine, N-nitrosopyrrolidine, N-nitroso morpholine) : not more than 0.01 mg/kg(in compliance with only the rubber nipple)

7) N-Nitrosatable substances(as sum of N-nitrosodimethylamine, N-nitrosodiethylamine, N- nitrosodi-n- propyl amine, N-nitrosodi-n-buthylamine, N-nitrosopiperidine, N-nitroso pyrrolidine, N-nitrosomorpholine) : not more than 0.1 mg/kg(in compliance with only the rubber nipple)

d. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method a. Residue Test and b. Migrant Test
2) Cadmium : IV. 2. 2-2 Cadmium Test Method a. Residue Test
3) 2-Mercaptoimidasoline(in compliance with only the rubber containing chlorine) : IV. 2. 2-49 2-Mercaptoimidasoline Test Method
4) 1,3-Butadiene : IV. 2. 2-39 1,3-Butadiene Test Method
5) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method
6) Phenol : IV. 2. 2-26 Phenol Test Method
7) Formaldehyde : IV. 2. 2-27 Formaldehyde Test Method
8) Zinc : IV. 2. 2-50 Zinc Test Method
9) N-Nitrosamines and N-Nitrosatable substances : IV. 2. 2-51 N-Nitrosamines and N-Nitrosatable substances Test Method

4. Paper or Processed paper

a. Definition

Paper is made from pulp as the main material through the manufacturing process and processed paper is made by coating paper with food wax, synthetic resins, rubber, etc.

b. Residue specification(mg/kg)

Following specifications are not applicable to processed paper.

1) PCBs : not more than 5

c. Migrant specification(mg/L)

Migrant specification for Individual Materials must be applied for processed paper whose food-contact surfaces is synthetic resins, etc.

1) Arsenic : not more than 0.1(as AS_2O_3)
2) Lead : not more than 1
3) Formaldehyde : not more than 4
4) Fluorescence whitening agent : shall not be detected

d. Test Method

1) PCBs : IV. 2. 2-52 PCBs Test Method
2) Arsenic : IV. 2. 2-9 Arsenic Test Method
3) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
4) Formaldehyde : IV. 2. 2-27 Formaldehyde Test Method
5) Fluorescence whitening agent : IV. 2. 2-53 Fluorescence whitening agent Test Method

5. Metal

a. Definition

Metal is the material that is composed of metal.

b. Migrant specification(mg/L)

Metal contained dry food(except oil·fat and fatty food) could not be applied following specifications. If a side that is not directly contacting food is not coated with synthetic resin or rubber, specifications 6) to 14) listed below are not applicable.

1) Lead : not more than 0.4
2) Cadmium : not more than 0.1
3) Nickel : not more than 0.1
4) Hexavalent chromium : not more than 0.1
5) Arsenic : not more than 0.2(as As_2O_3)
6) Non-volatile residue : not more than 30(In case of using n-heptane as food simulant, non-volatile residues of the coating layer containing natural oil as main material and exceeding 3% of zinc oxide, shall not be more than 90. Even if it exceeds 30 using water as food simulant, the chloroform soluble substance is not more than 30, it is acceptable for the specification)
7) Formaldehyde : not more than 4
8) Vinyl chloride : not more than 0.05
9) Epichlorohydrine : not more than 0.5
10) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : not more than 2.5(However, migration specification of bisphenol A is not more than 0.6)
11) Bisphenol A diglycidyl ether (including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) : not more than 1
12) Bisphenol F diglycidyl ether (including Bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) : not more than 1

13) 4,4'-methylenedianiline : not more than 0.01

14) Zinc : not more than 15

c. Test Method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test

2) Cadmium : IV. 2. 2-2 Cadmium Test Method b. Migrant Test

3) Nickel : IV. 2. 2-1 Nickel Test Method

4) Hexavalent chromium : IV. 2. 2-4 Hexavalent chromium Test Method b. Migrant Test

5) Arsenic : IV. 2. 2-9 Arsenic Test Method

6) Non-volatile residue : IV. 2. 2-8 Non-volatile residue Test Method

7) Formaldehyde : IV. 2. 2-27 Formaldehyde Test Method

8) Vinyl chloride : IV. 2. 2-16 Vinyl Chloride Test Method b. Migrant Test

9) Epichlorohydrine : IV. 2. 2-45 Epichlorohydrine Test Method

10) Bisphenol A(as sum of phenol, bisphenol A and p-tert-butylphenol) : IV. 2. 2-35 Bisphenol A(including phenol and p-tert-butylphenol) Test Method

11) Bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) and bisphenol F diglycidyl ether (including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) : IV. 2. 2-44 Bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) and bisphenol F diglycidyl ether (including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) Test Method

12) 4,4'-Methylenedianiline : IV. 2. 2-31 Primary aromatic amine(in compliance with only the aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) Test Method

13) Zinc : IV. 2. 2-50 Zinc Test Method

6. Wood

a. Definition

Wood refers to one that consists of tree or bamboo tree or one that is made by coating tree or bamboo tree with latices of Rhus Vermicifera.

b. Migrant specification(mg/L)

1) Arsenic : not more than 0.1(as As_2O_3)
2) Lead : not more than 1
3) Sulfur dioxide : not more than 12.8
4) Ortho-phenylphenol : not more than 7.3
5) Thiabendazole : not more than 1.8
6) Biphenyl : not more than 0.9
7) lmazalil : not more than 0.6

c. Test Method

1) Arsenic : IV. 2. 2-9 Arsenic Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Sulfur dioxide : IV. 2. 2-55 Sulfur dioxide Test Method
4) Ortho-phenylphenol, thiabendazole, biphenyl and imazalil : IV. 2. 2-56 Ortho-phenylphenol, thiabendazole, biphenyl and imazalil Test Method

7. Glass, Ceramic, Porcelain enamel and Pottery

a. Migrant Specifications for sample whose depth is over 2.5cm when liquid is filled (excluding porcelain enamel ware whose volume is larger than 3L)

1) Utensils, containers and packages of Glass

Capacity		Lead(μg/mL)	Cadmium(μg/mL)
Other than heat-cooking ware	less than 600 mL	not more than 1.5	not more than 0.5
	600 mL and more, and less than 3L	not more than 0.75	not more than 0.25
	3L and more	not more than 0.5	not more than 0.25
Heat-cooking ware		not more than 0.5	not more than 0.05

2) Utensils, containers and packages of ceramic and pottery

Capacity		Lead(μg/mL)	Cadmium(μg/mL)	Arsenic(μg/mL)
Other than heat-cooking ware	less than 1.1L	not more than 2	not more than 0.5	not more than 0.05(as As_2O_3) (only limited to pottery)
	1.1L and more, and less than 3L	not more than 1	not more than 0.25	
	3L and more	not more than 0.5	not more than 0.25	
Heat-cooking ware		not more than 0.5	not more than 0.05	

3) Utensils, containers and packages of porcelain enamel

Capacity	Lead(μg/mL)	Camium(μg/mL)	Antimony(μg/mL)
Other than heat-cooking ware whose capacity is less than 3L	not more than 0.8	not more than 0.07	not more than 0.1
Heat-cooking ware whose capacity is less than 3L	not more than 0.4	not more than 0.07	not more than 0.1

b. Migrant Specifications for sample that can not contain water or sample whose depth is less than 2.5cm when liquid is filled or porcelain enamel whose volume is larger than 3L

			Lead(μg/cm^2)	Cadmium (μg/cm^2)	Antimony (μg/mL)
(1) Utensils, containers and packages of glass			not more than 8	not more than 0.7	-
(2) Utensils, containers and packages of ceramic and pottery			not more than 8	not more than 0.7	-
(3) Utensils, containers and packages of porcelain enamel	When fill with liquid, the depth is 2.5 cm or deeper, capacity is 3L or over		not more than 1	not more than 0.5	not more than 1
	Cannot be filled, or less than 2.5 cm depth	Other than heat -cooking ware	not more than 8	not more than 0.7	not more than 1
		Heat-cooking ware	not more than 1	not more than 0.5	not more than 1

c. Test method

1) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
2) Cadmium : IV. 2. 2-2 Cadmium Test Method b. Migrant Test
3) Arsenic : IV. 2. 2-9 Arsenic Test Method
4) Antimony : IV. 2. 2-10 Antimony Test Method b. Migrant Test

8. Starch

a. Definition

Starch is processed properly using 70% or more starch.

b. Migrant specification(mg/L)

1) Arsenic : not more than 0.1(as As_2O_3)
2) Lead : not more than 1
3) Consumption of potassium permanganate : not more than 10(however, non-water resistance containers is excluded)
4) Formaldehyde : not more than 4
5) Fluorescence whitening agent : shall not be detected

c. Test method

1) Arsenic : IV. 2. 2-9 Arsenic Test Method
2) Lead : IV. 2. 2-1 Lead Test Method b. Migrant Test
3) Consumption of potassium permanganate : IV. 2. 2-7 Consumption of potassium permanganate Test Method
4) Formaldehyde : IV. 2. 2-27 Formaldehyde Test Method
5) Fluorescence whitening agent : IV. 2. 2-53 Fluorescence whitening agent Test Method

IV. Test Method

1. General Principles

a. Weights and measures shall be applied by the metric system and are indicated in following standards and specifications.

1) Length : m, cm, mm, μm, nm

2) Volume : L, mL, μL

3) Weight : kg, g, mg, μg, ng, pg

4) Area : cm^2, m^2, mm^2

5) Calorie : kcal, kj

6) Temperature : ℃

b. Atomic weight is based on the latest Table of Standard Atomic Weights.

c. Weight percentage is indicated with the symbol of %. However, material content(g) in 100 mL solution is indicated with w/v% and material content(mL) in 100 mL solution with v/v%. Weight parts per million may be indicated with the symbol of mg/kg or mg/L.

d. Standard temperature is defined as 20℃, and ordinary temperature as 15~25℃, and room temperature as 1~35℃, and slightly warm temperature as 30~40℃.

e. Cold place, except as otherwise specified, means a place of 0~15℃ without light.

f. Test shall, except as otherwise specified, be performed at the normal temperature and observed within 30 seconds after operation. However, test, which is affected by temperature, shall be performed at a standard temperature.

g. Water used in the test is, except as otherwise specified, distilled water or purified water.

h. pH is, except as otherwise specified, determined as acidic, alkaline or neutral by Litmus test. Detailed pH is described as pH. Strongly acidic is defined as about pH 3.0 or lower, weakly acidic as about pH 3.0~5.0, slightly acidic as about pH 5.0~6.5, neutral as about pH 6.5~7.5, slightly alkaline as about pH 7.5~9.0, weakly alkaline as about pH 9.0~11.0, and strongly alkaline as about pH 11.0

or higher.

i. Indication of mixture such as (1:1), (4:2:1) etc. means the mixed volume ratio of liquid reagent or the mixed weight ratio of solid reagent. Also indication of solution concentration such as (1→5), (1→10), (1→100) etc. means that solid reagent of 1 g or liquid reagent of 1 mL is dissolved in solvent to produce each 5 mL, 10 mL, 100 mL etc.

j. "Precisely measuring" weight is said to measure weight to 0.1 mg, 0.01 mg, or 0.001 mg in consideration of minimum weight unit. "Correctly measuring" weight is said to measure the weight of specified value to 2 decimals.

k. Indication of sampling quantity with "about" is said to take the sample of recorded quantity in a range of 90~110%, except as otherwise specified.

l. Record of "constant weight" in drying or heating means that weight difference between weight firstly measured in drying or heating and weight secondly measured in one more drying or heating for one hour shall be within 0.1% of firstly measured weight. However, if the weight difference is not more than 0.5 mg in measuring by chemical balance or not more than 0.01 mg by micro-chemical balance, it is regarded as "constant weight".

m. Desiccator's drying agent is silica gel, except as otherwise specified.

n. Decompression means, except as otherwise specified, the pressure of 15 mmHg or lower.

o. Glass container for the Reagent, solution, and standard solution preserving must be low solubility and alkaline, must not contain lead and cadmium as possible

p. Solvent used in test, except as otherwise specified, must be "HPLC grade".

q. Reagent and standard solution used in test, except as otherwise specified, must be extra pure grade.

r. Except as otherwise specified, cold water is defined as water of 15℃ or lower, hot water as water of 60~70℃, boiling water as water of about 100℃, and heating temperature "in/under water bath" is, except as otherwise specified, defined as about 100℃ and water bath can be replaced with steam bath of about 100℃.

s. When 20 drops of distilled water are loaded at 20℃ in measuring the number of water drop, the weight of 20 drops shall be in the range of 0.90~1.10 g.

t. Nessler's tube is a flat bottom tube, which is made with colorless glass of inner diameter(ID) 20 mm, outer diameter(OD) 24 mm, and length 20 cm from its bottom to the bottom of stopper, and its volume shall be 50 mL. The difference between scales in each tube shall be not more than 2 mm.

u. When all-in-one utensils, containers or packages consists of a variety of materials and ground colors, the sampling may be by material.

v. When the utensils, containers or packages is of the same ground color, but different in terms of shape and size, the sampling may be by material. However, glass, ceramic, porcelain enamel and pottery are excluded

2. Test Method by Item

2-1. Lead Test Method

a. Residue Test

1) Analysis principle

Measure lead residue in sample by atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer.

2) Apparatus

Atomic absorption spectrophotometry or Inductively coupled plasma atomic emission epectrometry

3) Standard solution

Accurately weigh 159.8 mg of lead nitrate and dissolve in 10 mL of 10% nitric acid. Dilute to 100 mL with water. Take 1 mL into a 200 mL volumetric flask and add 0.1M nitric acid to make 200 mL. This solution is used as standard solution(5 μg/mL).

4) Preparation of test solution

a) Synthetic resin, cellophane, rubber, paper and processed paper, and starch

Accurately weigh 1.0 g of sample and put into platinum, quartz, or heat-resistant glass crucible. Add 2 mL of sulfuric acid and slowly heat the crucible until not produce white smoke of sulfuric acid and most of the sample is carbonized. In an about 450℃ electric furnace, the sample is heated and incinerated to be ash. This operation, which cool it down, moisten with sulfuric acid, and heat again, is repeated until the sample is completely incinerated. After cooling, add 5 mL of hydrochloric acid(1→2) to the residue, mixed, and evaporated in a water bath. After cooling, dissolve it with 0.1 M nitric acid. If insoluble matters exist, filter to be 20 mL. This solution is used as test solution.

b) Metal

The sample is prepared that scraped out several parts with its own gloss in food contact surface of metal apparatus, containers and packages. Plating part of food contact surface in metal apparatus, containers and packages itself is cautiously scratched and it is used as sample(However, the place which it solders on the food contact surface is scratched and it is used as sample). 0.1g of sample is taken to platinum dish or crucible and dissolved in the small amount of nitric acid(diluted hydrochloric acid for aluminum). In necessity, it is filtered and diluted to 20 mL with water. This solution is used as test solution.

5) Test procedure

For test solution and standard solution, proceed directly under 2-11 Atomic absorption spectrophotometry Test Method(wavelength 283.3 nm) or 2-12 Inductively coupled plasma atomic emission epectrometry Test Method(wavelength 220.4 nm) and calculate the concentration of lead in test solution. Calaulate the content of lead in sample under following equation.

a) Synthetic resin, cellophane, rubber, paper and processed paper, starch

$$\text{Lead(mg/kg)} = \frac{\text{Concentration of lead in test solution(μg/mL)}}{\text{Amount of sample(g)}} \times 20\text{(mL)}$$

b) Metal product

$$\text{Lead(\%)} = \frac{\text{Concentration of lead in test solution(μg/mL)} \times 20\text{(mL)}}{\text{Amount of sample(g)} \times 10^6} \times 100$$

b. Migrant Test

1) Synthetic resin, cellophane, rubber, paper and processed paper, wood, and starch

a) Analysis principle

Measure Lead migrated from synthetic resin, cellophane, rubber, paper and processed paper, wood, and starch with atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer

c) Standard solution

Accurately weigh 159.8 mg of lead nitrate and dissolve in 0.1M nitric acid to make 100 mL. Take 1 mL of this solution into a 1,000 mL volumetric flask and dilute 1000 mL with food simulant. This solution is used as standard solution(1 μg/mL).

d) Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. In case of wood prepare the test solution using water as food simulant under 2-6 Preparation of migration test solution for each material. 50 mL of test solution is taken into platinum or quartz crucible and evaporated to be dried in water bath. 10 drops of sulfuric acid are added and slowly heated to evaporate most of sulfuric acid and dried on direct flame. By being strengthened heating power continuously, the sample is incinerated at 450℃. This operation is repeated until the sample color almost becomes white. After it is cooled, 20 mL of 4% acetic acid is added to residue and heated to dissolve it. 4% acetic acid is added to be 50 mL.

e) Test Procedure

Test for standard solution and test solution is performed according to 2-11 Atomic absorption spectrophotometry Test Method(wavelength : 283.3 nm) or

2-12 Inductively coupled plasma atomic emission spectrometry Test Method(wavelength : 220.4 nm) and measure the concentration of lead in test solution.

2) Metal

a) Analysis principle

Measure lead migrated from metal by Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer

c) Reagent and Solution

(1) 0.5% citric acid solution

Dissolve 5 g of citric acid monohydrate into water to make 1,000 mL. Adjust the pH to 3.5 with sodium hydroxide solution.

(2) Sodium hydroxide solution

Dissolve 4.3 g of sodium hydroxide into water to make 100 mL.

d) Standard solution

Accurately weigh 159.8 mg of lead nitrate and dissolve in 10 mL of 10% nitric acid and add water to make 100 mL. Take 1 mL of this solution into a 200 mL volumetric flask and dilute 200 mL with food simulant. Again, take 8 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with food simulant. This solution is used as lead standard solution(0.4 μg/mL). When the food simulant is water, add 5 drops of nitric acid to lead standard solution.

e) Preparation of test solution

Utensils, containers and packages for food, which is listed in the column 1 of the following table, is tested using each solvent of column 2 as food simulant under 2-6 Preparation of migration test solution for each material. When the food simulant is water, add 5 drops of nitric acid to 100 mL of test solution.

Column 1	Column2
Food of not more than pH 5	Water
Food of more than pH 5	0.5% Citric acid

f) Test procedure

Test for standard solution and test solution is performed according to 2-11 Atomic absorption spectrophotometry Test Method(wavelength : 283.3 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry Test Method(wavelength : 220.4 nm) and measure the concentration of lead in test solution.

3) Glass, ceramic, porcelain enamel, and pottery

a) Analysis principle

Measure lead migrated from Glass, Ceramic, porcelain enamel, and pottery utensils, containers and packages using 4% acetic acid as food simulant by Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer

c) Standard solution

Accurately weigh 159.8 mg of lead nitrate and dissolve in 10 mL of 10% N volumetric flask and dilute 200 mL with 4% acetic acid. Again, take 0.2 mL, 2 mL, 4 mL, 6 mL, and 8 mL of this solution into a 10 mL volumetric flask respectively and add 4% acetic acid to make 10 mL. This solution is used as lead standard solution(each of the solution is 0.2 μg/mL, 2 μg/mL, 4 μg/mL, 6 μg/mL, and 8 μg/mL).

d) Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material.

e) Test procedure

(1) Preparation of calibration curve

Proceed directly under 2-11 Atomic absorption spectrophotometry(wavelength : 283.3 nm) or 12. Inductively coupled plasma atomic emission Spectrometry(wavelength : 220.4nm) and obtain absorption. Plot the absorption on each concentration and prepare calibration curve of each of lead.

(2) Test

Measure absorbance for test solution in the same manner as (1) Preparation of calibration curve and absorption is obtained. Using the absorbance, calculate the content of lead or cadmium in test solution from previously prepared calibration curve. However, the sample which cannot be filled or depth is less than 2.5 cm with filling liquid or enameled sample whose capacity is 3 L or over, calculate the content of lead or cadmium per unit area under following equation.

$$\text{Migration per unit area}(\mu g/cm^2) = \frac{C \times V}{S}$$

C : Concentration of lead in test solution by calibration curve(μg/mL)

V : Total amount of food simulant(mL)

S : Surface area contacted with food simulant(cm^2)

2-2. Cadmium Test Method

a. Residue Test

1) Analysis principle

Measure cadmium residue in sample by Atomic absorption Spectrophotometer or Inductively coupled plasma atomic emission spectrometer.

2) Apparatus

Atomic absorption spectrophotometer or Inductively coupled plasma atomic emission spectrometer

3) Standard solution

Accurately weigh 100 mg of cadmium and dissolve in 50 mL of 10% nitric acid. Evaporate it in a water bath and dissolve the residue in 0.1M nitric acid to make 1,000mL. Take 10 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 0.1M nitric acid. Again, take 5 mL of this solution into 100 mL volumetric flask and dilute to 100 mL with 0.1M nitric acid. This solution is used as standard solution(0.5 μg/mL).

4) Preparation of test solution

Prepare test solution following a. Residue Test 4) Preparation of test solution a) Synthetic resin, cellophane, rubber, paper and processed paper, and starch in 2-1 Lead test method. Take 2 mL of this test solution and dilute to 20 mL with 0.1M nitric acid.

5) Test Procedure

For test solution and standard solution, proceed directly under 2-11 Atomic absorption apectrophotometry Test Method(wavelength : 228.8nm) or 2-12 Inductively coupled plasma atomic emission spectrometry Test Method(wavelength : 228.8nm) and calculate the concentration of cadmium in test solution. Calculate the content of cadmium in sample under following equation.

$$\text{Cadmium(mg/kg)} = \frac{\text{Concentration of cadmium in test solution(μg/mL)}}{\text{Amount of sample(g)}} \times 200\text{(mL)}$$

b. Migrant Test

1) Metal

a) Analysis principle

Measure cadmium migrated from metal by Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer

c) Reagent and Solution

(1) 0.5% citric acid solution

Dissolve 5 g of citric acid monohydrate into water to make 1,000 mL. Adjust the pH to 3.5 with sodium hydroxide solution.

(2) Sodium hydroxide solution

Dissolve 4.3 g of sodium hydroxide into water to make 100 mL.

d) Standard solution

Accurately weigh 100 mg of cadmium and dissolve in 50 mL of 10% nitric acid. Evaporate it in a water bath and dissolve the residue in 0.1M nitric acid to make 1,000 mL. Take 1 mL of this solution into a 200 mL volumetric flask and dilute to 200 mL with food simulant specified in each test method. Again, take 2 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with food simulant specified in each test method. This solution is used as cadmium standard solution(0.1 μg/mL). When the food simulant is water, add 5 drops of nitric acid to cadmium standard solution.

e) Preparation of test solution

Utensils, containers and packages for food, which is listed in the column 1 of the following table, is tested using each solvent of column 2 as food simulant under 2-6 Preparation of migration test solution for each material. When the food simulant is water, add 5 drops of nitric acid to 100 mL of test solution.

Column 1	Column2

Food of not more than pH 5	Water
Food of more than pH 5	0.5% Citric acid

f) Test procedure

Test for standard solution and test solution is performed according to 2-11 Atomic absorption spectrophotometry Test Method(wavelength : 228.8 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry Test Method(wavelength : 228.8 nm) and measure the concentration of cadmium in test solution.

2) Glass, ceramic, porcelain enamel, and pottery

a) Analysis principle

Measure cadmium migrated from glass, ceramic, porcelain enamel, and pottery using 4% acetic acid as food simulant by Atomic absorption spectrophotometer or Inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption spectrophotometer or Inductively coupled plasma atomic emission spectrometer

c) Standard solution

Accurately weigh 10 mg of cadmium and dissolve in 50 mL of 10% nitric acid. Evaporate it in a water bath and dissolve the residue in 0.1M nitric acid to make 100 mL. Take 0.2 mL of this solution into a 200 mL volumetric flask and add 4% acetic acid to make 200 mL. Again, take 0.2 mL, 2 mL, 4 mL, 6 mL, and 8 mL of this solution into a 10 mL volumetric flask respectively and add 4% acetic acid to make 10 mL. This solution is used as cadmium standard solution(Each of the solution is 0.02 μg/mL, 0.2 μg/mL, 0.4 μg/mL, 0.6 μg/mL, and 0.8 μg/mL).

d) Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material.

e) Test procedure

(1) Preparation of calibration curve

Proceed directly under 2-11 Atomic absorption spectrophotometry(wavelength : 228.8 nm) or 12. Inductively coupled plasma atomic emission Spectrometry(wavelength : 228.8 nm) and obtain absorption. Plot the absorption on each concentration and prepare calibration curve of each of lead.

(2) Test

Measure absorbance for test solution in the same manner as (1) Preparation of calibration curve and absorption is obtained. Using the absorbance, calculate the content of lead or cadmium in test solution from previously prepared calibration curve. However, the sample which cannot be filled or depth is less than 2.5 cm with filling liquid or enameled sample whose capacity is 3 L or over, calculate the content of lead or cadmium per unit area under following equation.

$$\text{Migration per unit area}(\mu g/cm^2) = \frac{C \times V}{S}$$

C : Concentration of lead in test solution by calibration curve(μg/mL)

V : Total amount of food simulant(mL)

S : Surface area contacted with food simulant(cm^2)

2-3. Mercury Test Method

a. Analysis principle

Measure mercury residue in sample by Cold vapor atomic absorption spectrophotometer or Gold amalgam atomic absorption spectrophotometer.

b. Cold vapor atomic absorption spectrophotometer Test Method

1) Apparatus

a) Atomic absorption spectrometer : quartz absorption cell is attached

b) Lamp : Hollow cathode mercury lamp

c) Mercury vapor apparatus

2) Reagent and Solution

a) Tin chloride solution : 10 g of tin chloride is dissolved in 0.5N sulfuric acid to make 1,000 mL.

3) Standard solution

Accurately weigh 135.4 mg of mercury dichloride into 100 mL of 10% nitric acid and dilute to 1,000 mL with water. When using, take 0.1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 1% nitric acid. This solution is used as standard solution(0.1 μg/mL).

4) Preparation of test solution

Unless otherwise specified, transfer 5 ~ 10 g of sample into a decomposition flask. Add 10 mL of water and 20 mL of nitric acid, shake slowly and 20 mL of sulfuric acid is slowly added. Attach a reflex condenser to the flask, which is heat until brown smoke is not generated. When the decomposed solution doesn't become colorless ~ light yellow transparent solution, add 5 mL of nitric acid after cooling, and heat. After cooling, add 50 mL of water and 10 mL of 10% urea solution and boil for 10 min. Cool it down, add 1 g of potassium permanganate, and occasionally shake for 10 min, when purple pink color disappear, add 1 g of potassium permaganate and occasionally

shake. Repeat this until purple-pink color remains. After boiling for 20 min, purple-pink color disappears, then cool it down. Add 1 g of potassium permanganate and heat for 20 min again. When purple-pink color of the solution disappears, repeat 2 times adding of potassium permanganate and heating, and cool. Add 20 % Hydroxylamine Hydrochloride carefully until the solution becomes colorless and transparent. After cooling, transfer the decomposed solution to another flask and inside, connecting part of a reflex condenser and the decomposition flask for washed with sulfuric acid(1→100) 20 mL. Add washing water to this, become a certain amount with water. This solution is used as test solution.

5) Test procedure

100 mL each of test solution and blank test solution whose concentration of sulfuric acid is previously adjusted to 20%(v/v) is taken to test solution bottle. After being connected to vapor apparatus, 10 mL of tin chloride solution is added, and immediately, stopper is placed. Absorbance is measured at a 253.7 nm wavelength by circulating air in absorption cell using diaphram pump. Separately, water is added to 1, 5, 10, 15, 20 mL each of mercury standard solution to make 100 mL respectively. The standard solution proceed in the same manner as test solution and calibration curve is prepared by measuring absorbance. Absorbance of test solution is substituted to the calibration curve and calculate the content of mercury.

c. Gold amalgam atomic absorption spectrophotometer Test Method

1) Apparatus

Use mercury measurement apparatus, which automatizes combustion of sample, collection by gold amalgam, and measurement by Cold atomic absorption spectrophotometry. Mercury measurement apparatus, which is set a special catalyst on the combustion part, can be used.

2) Reagent and Solution

a) Additives

When using (a) aluminum oxide and (b) calcium hydroxide · sodium carbonate(1:1), activate for 30 min at 950℃.

3) Standard solution

Accurately weigh 135.4 mg of mercury dichloride and dissolve in 0.001% L-cysteine Solution to make 1,000 mL. Dilute this solution to 0 ~ 200 ng/mL with 0.001% L-cysteine solution. This solution is used as standard solution.

4) Preparation of test solution

Proceed as directed under 4) Preparation of test solution in b. Cold vapor atomic absorption spectrophotometer.

5) Test procedure

About 1 g of additive(a) is uniformly spread on crucible and each 100 μL of merucry standard solution is instilled on additive(a). On that, about 0.5 g of additive(a) and 1 g of additive(b) are uniformly spread in turn to form the layer. In case of automatic mercury measurement apparatus, which a special catalyst is set on the combustion part, additive is not added to crucible and only add each 100 μL of mercury standard solution. Crucible is transferred into combustion furnace. Air or oxygen is flowed and heat about at not less than 700℃ and collect mercury in collection tube. Collection tube is boiled at not less than 600℃, mercury vapor is sent to atomic absorption spectrophotometer and each absorbance is measured, As. Separately, absorbance is measured in the same manner with only adding additive on crucible, Ab. Calculate the value of As - Ab and prepare calibration curve. Separately, using 20 mg of finely cut sample, prepare in the same manner as mercury standard solution and measure absorbance, At. Using value of At - Ab, calculate the content of mercury in sample from calibration curve.

2-4. Hexavalent Chromium Test Method

a. Residue Test

1) Analysis principle

Measure hexavalent chromium residue in sample by spectrophotometer.

2) Apparatus

Spectrophotometer

3) Solution

a) Diphenylcarbazide Solution

Dissolve 0.25 g of 1,5-diphenylcarbazide in 50 mL of acetone. Preserve in amber glass bottle and use in 7 days after preparation.

b) Alkali Decomposition Solution

Dissolve 20 g of sodium hydroxide and 30 g of sodium carbonate in water to make 1,000 mL.

c) 0.1M phosphate buffer solution

Accurately weigh 531.3 mg of dipotassium phosphate(K_2HPO_4) and 265.4 mg of potassium phosphate(KH_2PO_4) and dissolve in water to make 100 mL.

4) Standard solution

Accurately weigh 311.5 mg of sodium chromate(Na_2CrO_4) and dissolve in water to make 1,000 mL. Take 2 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with water. This solution is used as standard solution(2 μg/mL).

5) Preparation of test solution

1 g of sample is finely cut and taken in 250 mL erlenmeyer flask and add 50 mL of alkali decomposed solution. Add 400 mg of magnesium chloride and 0.5 mL of 0.1M phosphate buffer solution and mix well for 5 min. While stirring

this solution for 60 min on the heater of 90～95℃, heat, cool it down to room temperature and filter with 0.45 μm filter. To the filtrate, add 5M nitric acid drop-wise to adjust the pH to 7.5±0.5 and dilute to 100 mL with water. This solution is used as test solution. Separately, take 50 mL of alkali decomposition solution and proceed in the same manner as sample. This solution is used as blank test solution.

6) Test procedure

a) Preparation of calibration curve

Take 1 mL, 10 mL, and 25 mL(containing 2 μg, 20 μg, and 50 μg of hexavalent chromium) of standard solution into a 100 mL volumetric flask respectively and add water to be the half of volumetric flask. To each solution, add 2 mL of diphenylcarbazide solution and adjust the pH 2±0.5 with 10% sulfuric acid and add water to make 100 mL. Allow to stand each solution for 10 min, measure absorbance at wavelength 540 nm and prepare calibration curve.

b) Test

Take 50 mL of test solution into a 100 mL volumetric flask. Add 2 mL of diphenylcarbazide solution and adjust the pH to 2±0.5 with 10% sulfuric acid. Add water to make 100 mL. Allow to stand each solution for 10 min, measure absorbance at wavelength 540 nm, and calculate the content of hexavalent chromium in sample from previously prepared calibration curve. Separately, using 50 mL of blank test solution, proceed in the same manner as test solution, measure absorbance at wavelength 540 nm, and correct it.

b. Migrant Test

1) Analysis principle

Measure hexavalent chromium migrated from metal by spectrophotometer.

2) Apparatus

Spectrophotometer

3) Solution

Diphenylcarbazide solution : Dissolve 0.25 g of 1,5-diphenylcarbazide in 50ml of acetone. Preserve in amber glass bottle and use in 7 days after preparation.

4) Standard solution

Accurately weigh 311.5 mg of sodium chromate(Na_2CrO_4) and dissolve in water to make 1,000 mL. Take 2 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with water. This solution is used as standard solution(2 μg/mL).

5) Preparation of test solution

Utensils, containers and packages for food, which is listed in the column 1 of the following table, is tested using each solvent of column 2 as food simulant under 2-6 Preparation of migration test solution for each material. When the food simulant is water, add 5 drops of nitric acid to 100 mL of test solution.

Column 1	Column2
Food of not more than pH 5	Water
Food of more than pH 5	0.5% Citric acid

6) Test procedure

For test solution and standard solution, proceed as directly under a. Residue Test 6) Test procedure and calculate the content of hexavalent chromium in test solution. When 0.5% citric acid solution is used as food simulant, use 0.5% citric acid instead of water.

2-5 Thermal shock strength Test Method

a. Analysis principle

Measure durability of heat-cooking glassware by sudden temperature changes.

b. Apparatus

Constant-temperature oven

c. Test procedure

Put heat-cooking glassware into constant-temperature oven adjusted with a temperature, satisfying temperature difference in the following table. It taken out after 30 minutes immersed into cold water immediately. Examine whether brokenness or fissures or not in the heat-cooking glassware after 1 minute.

Usage and Thermal shock strength		Test temperature difference	Error tolerance
For direct heating	not less than 400℃	400℃	not more than +20℃
	not less than 150℃	150℃	not more than +10℃
For oven	not less than 120℃	120℃	not more than +8℃
For microwave oven	not less than 120℃	120℃	not more than +8℃
For boiling water	not less than 120℃	120℃	not more than +8℃

2-6 Preparation of migration test solution for each material

Unless otherwise specified, well wash utensils, containers and packages for food contacted in the column 1 of the following table and then prepare migration test solution by using each solvent of column 2 as food simulant to the food contact surface, like the following a to h for each material.

Column 1		Column 2
Oil·fat and fatty food		n-heptane
Alcoholic food	Food of not more than alcohol content of 20%	20% alcohol
	Food of more than alcohol contents of 20%	50% alcohol
Food of other than oil·fat and fatty food, and alcoholic food	Food of not more than pH 5	4% acetic acid
	Food of more than pH 5	water

a. Preparation of synthetic resin migration test solution

1) Sample that can be filled with liquid(means the utensils, containers of a type that can be filled with liquid but general packages are excluded)

Sample is fully filled with food simulant, which is heated at 60℃, and covered by watch glass and maintained at 60℃. It is sometimes shaken and allowed to stand for 30 min. This solution is used as test solution. However, when sample is used more than 100℃ and food simulant is water or 4% acetic acid, allow to stand at 95℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.

2) Sample that cannot be filled with liquid(means the utensils, containers and packages of a type that cannot be filled with liquid)

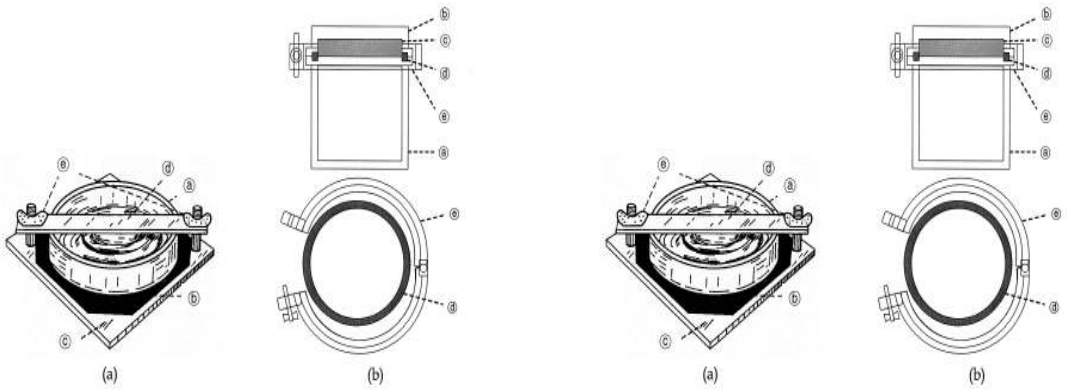
a) When inside and outside of the sample are same material

Dip the sample in the food simulant, which is heated at 60℃ at the rate of 2

mL/cm^2 surface area(calculated by combining surface area of both sides) and then covered with watch glass. Hold the temperature to 60℃ and allow to stand for 30 min. This solution is used as test solution. However, when sample is used more than 100℃ and food simulant is water or 4% acetic acid, allow to stand at 95℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.

b) When inside and outside of the sample are different

After food simulant which is heated at 60℃ at the rate of 2 mL/cm^2 surface area is contacted with food contact surface, hold the temperature to 60℃ and allow to stand for 30 min. Transfer this solution to a beaker. This is used as test solution. If necessary, prepare test solution using Fig 1. (a) or (b) migration single-side cell migration apparatus. When using (a) migration apparatus, sample is put on the large rubber plate. In this case, the food contact surface is upward. Stainless steel or glass cylinder is put on the plate and firmly fixed by metal screw. After put the food simulant heated at 60℃ at the rate of 2 mL/cm^2 surface area into stainless steel or glass cylinder, covered with watch glass. Hold the temperature to 60℃ and allow to stand for 30 min. This solution is used as test solution. When using (b) migration apparatus, put food simulant heated at 60℃ at the rate of 2 mL/cm^2 surface area into the cell, and the food contact surface is downward. On that, put teflon gasket and cover with a cap. Tighten it up firmly with clamp for solvent not to be leaked. Place the cell upside down, contact food simulant and sample, and hold the temperature to 60℃ and allow to stand for 30 min. This solution is used as test solution. However, when sample is used more than 100℃ and food simulant is water or 4% acetic acid, allow to stand at 95℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.



(a) (b)

ⓐ : hard glass and stainless steel ring, of which migration surface area is accurately 100 cm^2(diameter 112.8 ±0.1mm, height 35 mm)

ⓑ : silicon rubber mat

ⓒ : large stainless steel plate(about 150 x 150 mm)

ⓓ : rod(stainless steel) to fix the stainless steel or glass cylinder

ⓔ : fixed metal screw(width about 30 mm)

ⓐ : Stainless steel cylindrical cell, of which migration surface area is accurately 100 cm^2(diameter 112.8 ±0.1 mm, height not less than 50 mm)

ⓑ : Stainless steel stopper

ⓒ : Teflon mat

ⓓ : Teflon gasket

ⓔ : Stainless steel clamp

Fig 1. Migration apparatus for single-side cell migration method

c) In case of gasket

Wash well the container in which gasket is used with water and fill it with food simulant heated at 60℃ to the volume of the container. Seal it tightly with gasket and upside it down. Hold the temperature to 60℃ and allow to stand for 30 min. This solution is used as test solution. However, Wash well the container in which gasket is used with water and fill it with food simulant heated at 60℃ to the volume of the container. Seal it tightly with watch glass and upside it down. Hold the temperature to 60℃ and allow to stand for 30 min. This solution is used as blank test solution. However, when sample is used more than 100℃ and food simulant is water or 4% acetic acid, allow to stand at 95℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.

b. Preparation of cellophane migration test solution

Proceed as directed under a. Preparation of synthetic resin migration test solution

c. Preparation of rubber migration test solution

1) Rubber product except rubber nipple

Proceed as directed under A. Preparation of synthetic polymer migration test solution.

2) Rubber nipple

After washing sample well with water, immerse the sample into food simulant heated at 40℃, corresponding 20 mL per 1g of sample. Hold the temperature to 40℃ and allow to stand for 24 hours.

d. Preparation of paper or processed paper migration test solution

1) Sample that can be filled with liquid(means the utensils, containers of a type that can be filled with liquid but general packages are excluded)

Sample is fully filled with food simulant, covered by watch glass and maintained at 25℃ and allow to stand for 10 min. This solution is used as test solution.

2) Sample that cannot be filled with liquid(means the utensils, containers and packages of a type that cannot be filled with liquid)

a) When inside and outside of the sample are same

Sample is dipped in the food simulant heated at the rate of 2 mL/cm^2 surface area(calculated by combining surface area of both sides) and then covered with watch glass. Hold the temperature to 25℃ and allow to stand for 10 min. This solution is used as test solution.

However, in case of tea bags and coffee filter paper, immerse sample in food simulant heated to 95℃ and cover with watch glass. Hold the temperature to 95℃ and allow to stand for 5 min. This solution is used as test solution.

b) When inside and outside of the sample are different

After food simulant at the rate of 2 mL/cm^2 surface area is contacted with food contact surface, hold the temperature to 25℃ and allow to stand for 10 min. This solution is used as test solution. If necessary, prepare test solution using Fig 1. (a) or (b) migration apparatus. However, in case of tea bags and coffee filter paper, immerse sample in food simulant heated to 95℃ and cover with watch glass. Hold the temperature to 95℃ and allow to stand for 5 min. This solution is uses as test solution.

e. Preparation of metal migration test solution

1) Sample that can be filled with liquid

Sample is fully filled with food simulant, which is heated at 60℃, and covered with watch glass and maintained at 60℃ and allowed to stand for 30 min. This solution is used as test solution. However, when sample is used more than 10 0℃ and food simulant is water, 4% acetic acid or 0.5% citric acid, allow to stand at 95℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.

2) Sample that cannot be filled with liquid

After food simulant which is heated at 60℃ at the rate of 2 mL/cm^2 surface area is contacted with food contact surface, hold the temperature to 60℃ and allow to stand for 30 min. However, when sample is used more than 100℃ and food simulant is water, 4% acetic acid or 0.5% citric acid, allow to stand at 9 5℃ for 30 min. In case of using n-heptane as food simulant, sample is left alone at 25℃ for 1 hour. Each solution is test solution.

f. Preparation of wood migration test solution

1) Sample that can be filled with liquid

Sample is fully filled with food simulant, which is heated at 60℃, and covered with watch glass and maintained at 60℃ and allowed to stand for 30 min. However, When sample is used more than 100℃, allow to stand at 95℃ for 30 min. Each solution is used as test solution.

2) Sample that cannot be filled with liquid

After food simulant which is heated at 60℃ at the rate of 2 mL/cm^2 surface area is contacted with food contact surface, hold the temperature to 60℃ and allow to stand for 30 min. However, When sample is used more than 100℃, allow to stand at 95℃ for 30 min. Each solution is used as test solution.

g. Preparation of glass, ceramic, enameled and pottery migration test solution

1) Sample whose depth is 2.5 cm or deeper when filled with liquid(However, enameled utensils, containers and packages whose capacity is 3L or over are excluded)

Food simulant is filled to 5 mm under the surface on which liquid is overflowed. It is covered with watch glass and left alone in dark place being held at 25℃ for 24 hours. This solution is used as test solution.

2) Sample that cannot be filled with liquid or those whose depth is less than 2.5cm when filled with liquid or enameled utensils, containers and packages whose capacity is 3L or over

a) Sample whose depth is less than 2.5 cm when filled with liquid

Food simulant is filled to 5 mm under the surface on which liquid is overflowed. It is covered with clock dish and left alone in dark place being held at 25℃ for 24 hours. This solution is used as test solution.

b) ample that cannot be filled with liquid

After food simulant at the rate 2 mL/cm^2 surface area is contacted with food contact surface, hold the temperature to 25℃ for 24 hour in dark place. This solution is used as test solution.

h. Preparation of Starch migration test solution

1) Sample that can be filled with liquid(means the utensils, containers of a type that can be filled with liquid but general packages are excluded)

Put the food simulant into sample fully and covered with watch glass and left alone at 25℃ and allow to stand for 10 min. However, in case of sample

used for hot water, put the food simulant heated at 95℃ into sample fully, and covered with watch glass and left alone at 95℃ and allow to stand for 5 min. Each solution is used as test solution.

2) Sample that cannot be filled with liquid(means the utensils, containers and packages of a type that cannot be filled with liquid)

Sample is contacted in the food simulant, of which rate is 2 mL/cm^2 surface area(the sum of both side surface area) and then covered with watch glass. Hold the temperature to 25℃ and allow to stand for 10 min. This solution is used as test solution.

2-7 Consumption of potassium permanganate Test Method

a. Analysis principle

Measure the content organic compounds oxidized by potassium permanganate among migrated substances from sample using water as food simulant.

b. Reagent and Solution

1) Diluted sulfuric acid (1→3)

Slowly add 100 mL of sulfuric acid to 200 mL of water while stirring.

2) 0.005 M sodium oxalate

Dissolve 0.6700g of sodium oxalate, which is dried at 150 ~ 200℃ for 1～1.5 hours and cooled in desiccator in water to make 1,000 mL. Preserve in a brown bottle. Use in 1 month after preparation.

3) 0.002M Potassium permanganate solution

Dissolve 0.31g of potassium permanganate into water to be 1,000 mL. Preserve it in a brown bottle. When using, standardize using 0.005 M sodium oxalate solution.

<Standardization of 0.002 M potassium permanganate solution>

Take 100 mL of water, 5 mL of diluted sulfuric acid(1→3) and 5 mL of 0.002M potassium permanganate solution into erlenmeyer flask. Boil for 5 min. Stop heating, immediately add 10 mL of 0.005M hydroxide oxalate solution to decolorize, and add 0.002M potassium permanganate solution continuously until the pale red is no more disappeared but remained. Again, add 5 mL of diluted sulfuric acid(1→3) and 5 mL of 0.002M potassium permanganate solution. After boil it for 5 min, add 10 mL of 0.005 sodium oxalate and immediately titrate with 0.002M Potassium permanganate solution. Calculate the activity(f) from the consumption of 0.002M Potassium permanganate solution on titration, by following equation.

$$f = \frac{10}{5 + \text{Titrated amount of 0.002M potassium permanganate solution(mL)}}$$

c. Preparation of test solution

Prepare test solution using water as food simulant under 5. Preparation of migration test solution for each material.

d. Test procedure

Take 100 mL of water, 5 mL of diluted sulfuric acid(1→3) and 5 mL of 0.002M potassium permanganate solution into erlenmeyer flask. After boiling for 5 min, discard the solution and wash it. To this erlenmeyer flask, take 100 mL of test solution and add 5 mL of diluted sulfuric acid(1→3). Again add 10 mL of 0.002M potassium permanganate solution and boil for 5 min. Stop heating, immediately add 10 mL of 0.005M hydroxide oxalate solution to decolorize, and add 0.002M potassium permanganate solution continuously until the pale red is no more disappeared but remained. Separately, perform blank test in the same manner and calculate the potassium permanganate consumption under following equation.

$$\text{Consumption of potassium permanganate(mg/L)} = \frac{(a-b) \times f \times 1{,}000}{100} \times 0.316$$

a : Titrated amount of 0.002M Potassium permanganate solution in test(mL)

b : Titrated amount of 0.002M Potassium permanganate solution in blank test(mL)

f : Activity of 0.002M potassium permanganate solution

2-8 Non-volatile residue Test Method

a. Analysis principle

Using food simulant corresponding the food for use, prepare test solution. After evaporating test solution, heat at 105℃ for 2 hours and weigh the residue.

b. Preparation of test solution

1) Synthetic Resin

Unless otherwise specified, well wash utensils, containers and packages for food contacted in the column 1 of the following table and then prepare migration test solution by using each solvent of column 2 as food simulant to the food contact surface, according to 2-6 Preparation of migration test solution for each material.

Column 1		Column 2
Oil·fat and fatty food		n-heptane
Alcoholic food	Food of not more than alcohol content of 20%	20% alcohol
	Food of more than alcohol contents of 20%	50% alcohol
Food of other than oil·fat and fatty food, and alcoholic food	Food of not more than pH 5	4% acetic acid
	Food of more than pH 5	water

2) Cellophane

Prepare the test solution using 4% acetic acid as food simulant according to 2-6 Preparation of migration test solution for each material. Test solution use supernatant, which is precipitated well for floating matter such as paper fiber not

to be contained.

3) Rubber

a) For rubber except rubber nipple

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, water for utensils, and 20% ethanol for containers and packages contacted with oil·fat and fatty food are used as food simulant.

b. Rubber nipple

Prepare the test solution using water as food simulant according to 2-6 Preparation of migration test solution for each material.

4) Metal

Prepare the test solution under 2-6 Preparation of migration test solution for each material.

c. Test Procedure

200 ~ 300 mL of test solution(in case of use n-heptane as food simulant, 2 ~ 6 mL concentrates, which 200 ~ 300 mL of test solution is taken in branch flask and concentrated at vacuum, and washed solution, which the flask is doubly washed by each about 5 mL of n-heptane) is taken in an platinum, quartz or glass evaporating dish, which was dried at 105℃ and weighed constantly, and evaporated and dried in water bath. After being dried at 105℃ for 2 hour, it is cooled in desiccator. After being cooled, obtain changed weight of evaporating dish, a(mg) and then the quantity of non-volatile residues is obtained by the following equation.

$$\text{Non-volatile residue(mg/L)} = \frac{(a-b) \times 1{,}000}{\text{Quantity of test solution(mL)}}$$

b : Blank test value(mg) acquired from simulant, which is the same amount as test solution

When the amount of non- volatile residue using water as food simulant is more than 30 mg/L, it should be appropriate to following test for chloroform soluble. To non-volatile residues obtained by using water as food simulant, add 30 mL of chloroform, heat, and filter. Put filtrate into platinum or quartz evaporation dish which is previously dried at 105℃ and weighed. Again, wash the non volatile residues with 10 mL of chloroform 2 times, heat, and filter. Add the filtrate to evaporation dish and evaporate it in a water bath. After being dried at 105℃ for 2 hours, it is cooled at desiccator and weighed. Calculate the weight difference of evaporation dish between before and after the test, a(mg). When measure the content of chloroform soluble substance under following equation and the content should not be more than 30 mg/L.

$$\text{Chloroform soluble substance(mg/L)} = \frac{(a-b) \times 1{,}000}{\text{Quantity of test solution(mL)}}$$

b: Blank test data obtained by using same amount of food simulant as the test solution(mg)

Determine whether the content of zinc oxide is more than 3% as varnish in which natural oil is used as main material as follows.

When 1% sodium hydroxide of 50% amount of net contents in sample is put into sample and the sample is left alone at 60℃ for 15 min, varnish film contains more than 3% of zinc oxide content if varnish film loses its gloss and shows thick color.

2-9 Arsenic Test

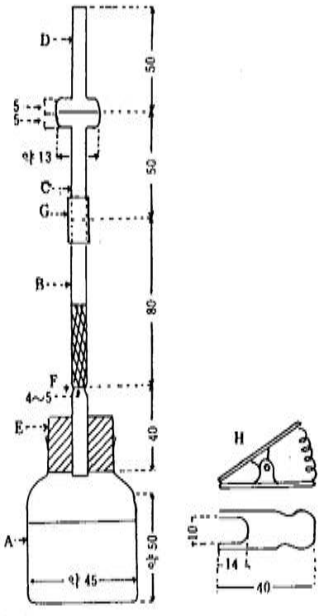
a. Analysis principle

Measure the allowable limit of migrated arsenic from polylactide, butylenesuccinate-adipate copolymer, cellophane, paper and processed paper, metal, wood, pottery, and starch using water or 4% acetic acid as food simulant, The lmit is expressed in terms of arsenic trioxide.

b. Gutzeit method

1) Apparatus

The apparatus is depicted in the figure below (unit : mm).



A : Generator bottle (Capacity: about 60 ml, with a marked line indicating 40 ml)

B : Glass tube with 6.5 mm internal diameter

C and D : Tube with a ground joint of 6.5 mm in internal diameter and about 18 mm in external diameter; both circles have the same center.

E : Rubber stopper

F : Dent at the lower part of glass tube B for supporting a plug of glass fiber

G : Rubber tube

H : Clip

Stuff a plug of glass fiber into glass tube B about 30 mm in height from dent F, moisten the fiber uniformly with a mixture of an equal volume of lead

acetate TS and water, then remove the excess of the solution from the glass fiber and the wall of the tube by gentle suction from the lower end of the tube.

Just before use, place a sheet of mercuric bromide test paper between the joint of glass tubes C and D, and fix both tubes with clip H.

2) Reagent and Solution

a) Acidic stannous chloride solution

Accurately weigh 4 g of tin chloride($SnCl_2 \cdot 2H_2O$) and dissolve in 125 mL of hydrochloric acid. Add water to make 250 mL. Seal tightly to preserve and use in a month after preparation.

b) Arsenic-free zinc

Use zinc (Arsenic-free, about 1,000 ~ 1,410 μm).

c) Potassium Iodide solution

Dissolve 16.5 g of potassium iodide(KI) into water to make 100 mL. Preserve in a brown bottle.

d) Lead acetate solution

Dissolve 11.8 g of lead acetate into water to make 100 mL. Add 2 drops of acetic acid. Seal tightly to preserve.

e) Mercury bromide test paper

Cut the filter paper for chromatography(width : about 3 cm, length : about 10 cm). Immerse this paper into the solution, which 5 g of mercury bromide is dissolved in 100 mL of ethanol, and occasionally shake. Allow to stand for more than 1 hour in a dark place, take it out and dry it while maintaining level in a dark place. Cut by circle of which diameter is about 18 mm and preserve in a brown bottle. The parts which is colored should not be touched by hands.

3) Standard solution

Grind arsenic trioxide(As_2O_3) into fine powder, dry in a desiccator(sulfuric acid), accurately weigh 0.10 g, and dissolve in 5 mL of 20% sodium hydroxide solution. Neutralize this solution with diluted sulfuric acid(1→20) and again, add 10 mL of diluted sulfuric acid(1→20). Dilute to 1,000 mL with boiled and cooled water. Again, to 10 mL of this solution, add 10 mL of diluted sulfuric acid(1→20) and dilute to 1,000 mL with boiled and cooled water. Prepare when using. Seal tightly to preserve.

Standard solution 1mL = 1.0 μg As_2O_3

4) Preparation of test solution

a) Synthetic resin, Wood, Starch

Prepare 10 mL test solution using water as food simulant according to 2-6 Preparation of migration test solution for each material. However, for the test according to d. Inductively coupled plasma atomic emission spectrometer 2-9 Arsenic Test Method, concentrate the test solution under a reduced pressure to 10-fold enrichment.

b) Cellophane, Paper and processed paper

Prepare 10 mL test solution using 4% acetic acid as food simulant according to 2-6 Preparation of migration test solution for each material. However, for the test according to d. Inductively coupled plasma atomic emission spectrometer 2-9 Arsenic Test Method, concentrate the test solution under a reduced pressure to 10-fold enrichment.

c) Metal

Prepare 5 mL test solution according to b. Migrant test 2) Metal e) Preparation of migration test solution in 2-1 Lead Test Method. However, for the test according to d. Inductively coupled plasma atomic emission spectrometer of 2-9 Arsenic Test Method, concentrate the test solution under a reduced pressure to 10-fold enrichment.

d) Glass, ceramic, enameled and pottery

Prepare 20 mL test solution using 4% acetic acid as food simulant according to 2-6 Preparation of migration test solution for each material. However, for the test according to d. Inductively coupled plasma atomic emission spectrometer 2-9 Arsenic Test Method, concentrate the test solution under a reduced pressure to 20-fold enrichment.

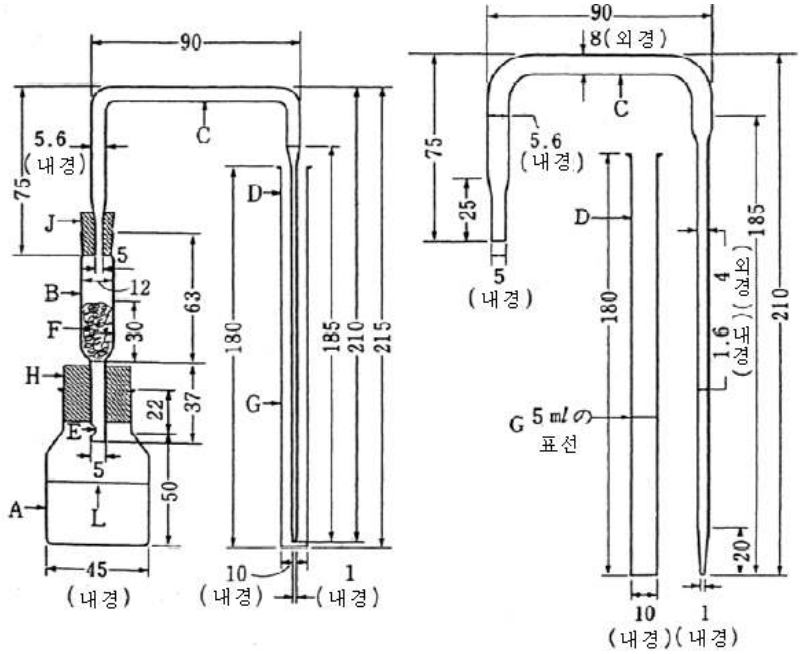
5) Test procedure

The test solution is transferred into the generator bottle, add 5 mL of diluted hydrochloric acid(1→2) and 5 mL of potassium iodide solution, and allow to stand for 2 ~ 3 min. To this solution, add 5 mL of acidic stannous chloride solution, and allow to stand for 10 min. Then add water to make 40 mL, add 2 g of arsenic-free zinc, immediately place rubber stopper E attached to glass tubes B, C, and D, then immerse the generator bottle up to the shoulder in water at a temperature of 25℃, and allow to stand for 1 hour. Separately, add 1 mL of arsenic standard solution into the generator bottle and proceed in the same manner as the test solution above. Immediately observe the color of the mercuric bromide test paper. The color of the paper is not deeper than the following standard color.

c. Silver N,N-Dimethyl dithiocarbamate method

1) Apparatus

The apparatus is depicted in the figure below (unit : mm).



A : Generator bottle (capacity up to the shoulder: approximately 70 ml)

B : Exit tube

C : Glass tube (internal diameter: 5.6 mm, the tip of the part to be inserted in the absorber tube D is stretched out to 1 mm in diameter)

D : Absorber tube (internal diameter: 10 mm

E : Small perforation

F : glass fiber (about 0.2g)

G : mark of 5 mL

H and J : rubber stopper

L : mark of 40 mL

Stuff glass fiber F in exit tube B up to about 30 mm in height, moisten the glass fiber uniformly with a mixture of equal volumes of lead acetate TS and water, and gentle suction to the lower end to remove the excess of the mixture. Insert the tube vertically into the center of rubber stopper H, and attach the tube to generator bottle A so that small perforation E in the lower end of B extends slightly below. To the upper end of B, attach rubber stopper J to which

tube C is vertically fitted. Make the lower end to the exit tube of C level with the lower end of rubber stopper J.

2) Reagent and Solution

a) Bromophenolblue solution

Dissolve 0,1g of bromophenolblue in 100 mL of 50% ethanol. Filter if necessary.

b) Ammonia solution

To 400 mL of ammonia water, add water to make 1,000 mL.

c) Acidic stannous chloride solution

Accurately weigh 4 g of tin chloride($SnCl_2 \cdot 2H_2O$) and dissolve in 125 mL of hydrochloric acid. Add water to make 250 mL. Seal tightly to preserve and use in a month after preparation.

d) Silver N,N-dimethyl dithiocarbamate solution

Accurately weigh 0.50 g of silver N,N-dimethyl dithiocarbamate and dissolve in pyridin to make 100 mL. This is Silver N,N-Dimethyl dithiocarbamate solution. Put it in the light shielded glass bottle and keep in a refrigerator.

3) Standard solution

Prepare arsenic standard solution according to 3) Standard solution in b. Gutzeit method.

4) Preparation of test solution

Prepare the test solution following 4) Preparation of test solution stated in b. Gutzeit method.

5) Test procedure

Add test solution of which amount is specified in the specification for each material into generator bottle. Add 1 drop of bromophenolblue solution and neutralize with ammonia water or ammonia solution. However, using water as food simulant, can be skip neutralization. To this solution, add 5 mL of

hydrochloric acid (1→2) and 5 mL of potassium iodide solution and allow to stand for 2 ~ 3 min. Add 5 mL of acidic stannous chloride solution and allow to stand for 10 min at room temperature. Add water to make 40 mL and add 2 g of zinc(for arsenic test), immediately connect the rubber stopper H, fitted with B and C, with generator bottle A. Insert the tip of C to the bottom of absorber tube D containing 5 mL of the absorbing solution for arsenic hydride, then immerse the generator bottle A up to the shoulder in water maintained at 25℃, and allow to stand for 1 hour. Disconnect the absorber tube, add pyridine to make 5 mL, if necessary, and observe the color of the absorbing solution. The color produced is not deeper than the standard color. Preparation of Standard Color is carried out at the same time as the test of test solution. Transfer test solution, same amount of food simulant, and 2 mL of standard solution into a generator bottle and proceed in the same manner as the test solution, use the color produced by the absorbing solution as the standard color.

d. Inductively coupled plasma atomic emission spectrometry

1) Apparatus

Inductively coupled plasma atomic emission spectrometry

2) Standard solution

Proceed as directed under 3) Standard solution in b. Gutzeit method.

3) Preparation of test solution

Prepare the test solution following 4) Preparation of test solution stated in b. Gutzeit method.

4) Test procedure

When test is performed according to 2-12 Inductively coupled plasma atomic emission spectrometry(wavelength : 193.7 nm), the atomic emission intensity of the test solution should not more than the atomic emission intensity of the standard solution.

2-10 Antimony Test Method

a. Residue Test

1) Analysis principle

Measure antimony residue in metal product by Atomic absorption spectrophotometer or Inductively coupled plasma atomic emission spectrometer.

2) Apparatus

Atomic Absorption Spectrophotometer or Inductively Coupled Plasma Atomic Emission Spectrometer

3) Standard solution

Accurately weigh 1.874 g of antimony trichloride and dissolve into small amount of diluted hydrochloric acid(1→2). Add diluted hydrochloric acid(1→10) to make 1,000 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 0.1M nitric acid. Again, take 1 mL of this solution into 10 mL volumetric flask and dilute to 10 mL with 0.1M nitric acid. This solution is used as standard solution(1.0μg/mL).

4) Preparation of test solution

Prepare test solution following a. Residue Test 4) Preparation of test solution b) Metal in 2-1 Lead Test Method. Take 1 mL of this test solution and dilute to 250 mL with 0.1M nitric acid.

5) Test Procedure

For test solution and standard solution, proceed directly under 2-11. Atomic absorption spectrophotometry(wavelength : 217.6nm) or Inductively coupled plasma emission spectroscope(wavelength : 206.8nm) and calculate the concentration of antimony in test solution. Calculate the content of antimony in sample under following equation.

$$\text{Antimony}(\%) = \frac{\text{Concentration of antimony in test solution}(\mu g/mL) \times 5{,}000(mL)}{\text{Amount of sample}(g) \times 10^6} \times 100$$

b. Migrant Test

1) Synthetic resin

a) Analysis principle

Measure antimony migrated from Poly(ethyleneterephthalate), Poly(cyclohexane-1,4-dimethylene terephthalate) using 4% acetic acid as food simulant by atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer

c) Standard solution

Accurately weigh 1.874 g of antimony trichloride and dissolve into small amount of hydrochloric acid. Add diluted hydrochloric acid(3→10) to make 1,000 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. Again, take 4 mL of this solution into 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. This solution is used as standard solution(0.4μg/mL).

d) Preparation of test solution

Transfer 200 mL solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material into a beaker and evaporate to 20 mL. This solution is used as test solution.

e) Test Procedure

For test solution and standard solution proceed as directed under 2-11 Atomic absorption spectrophotometry(wavelength : 217.6 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry(wavelength: 206.8 nm). Calculate

the concentration of antimony in test solution(adjust with concentration factor 10).

2) Porcelain enamel

a) Analysis principle

Measure antimony migrated from prcelain enamel using 4% acetic acid as food simulant by Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer.

b) Apparatus

Atomic absorption apectrophotometer or Inductively coupled plasma atomic emission spectrometer

c) Standard solution

Accurately weigh 1.874 mg of antimony trichloride and dissolve into small amount of hydrochloric acid. Add diluted hydrochloric acid(3→10) to make 1,000 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. Again, take 0.5 mL, 1 mL, 2 mL, 5 mL, and 10 mL of this solution into 100 mL volumetric flask respectively and dilute to 100 mL with 4% acetic acid. This solution is used as lead standard solution(each of the solution is 0.05 μg/mL, 0.1 μg/mL, 0.2 μg/mL, 0.5 μg/mL, and 1 μg/mL).

d) Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material.

e) Test procedure

(1) Preparation of calibration curve

For standard solution proceed directly under 2-11 Atomic absorption spectrophotometry(wavelength : 217.6 nm) or 12. Inductively coupled plasma atomic emission Spectrometry(wavelength : 206.8 nm) and obtain absorption. Plot the absorption on each concentration and prepare calibration curve of each of antimony.

(2) Test

Measure absorbance for test solution in the same manner as (1) Preparation of calibration curve and absorption is obtained. Using the absorbance, calculate the content of lead or cadmium in test solution from previously prepared calibration curve. However, the sample which cannot be filled or depth is less than 2.5 cm with filling liquid or enameled sample whose capacity is 3 L or over, calculate the content of lead or cadmium per unit area under following equation.

$$\text{Migration per unit area}(\mu g/cm^2) = \frac{C \times V}{S}$$

C : Concentration of lead in test solution by calibration curve(μg/mL)

V : Total amount of food simulant(mL)

S : Surface area contacted with food simulant(cm^2)

2-11 Atomic absorption spectrophotometry

a. Analysis principle

Determine the concentration of the object element in test solution, utilizing the phenomenon that the atoms in the ground state absorb the light of characteristic wavelength passing through an atomic vapor layer of the element.

b. Apparatus

Usually the apparatus consists of a light source, a sample-atomizer, a spectroscope, and a photometer, and a recording system. The light source is used a hollow cathode lamp. In atomization part, Flame type(direct vaporizer) of atomizer is composed of a burner and a gas flow regulator. And, electrothermal type is composed of an electric furnace and a power source. For the spectroscope, a grating for light diffraction or an interference filter prism is used. The photometer mainly consists of a detector and a signal treatment system. A recording system is composed of a display and a recording device.

c. Standard solution

Unless otherwise specified, use standard solution corresponding to element.

d. Test procedure

Unless otherwise specified, proceed under one of following.

1) Flame Type (direct vaporizer)

Fit the specific light source lamp(a hollow cathode lamp corresponding to element) to the lamp housing, and switch on the instrument. Light the source lamp, adjust the wavelength dial of the spectroscope to the wavelength of the analytical line specified, and set at an appropriate current value and slit-width. Using the supporting gas and combustible gas specified, ignite the mixture of

these gases, adjust the gas flow rate and pressure, and make the zero adjustment after nebulizing the solvent into the flame. Nebulize the test solution or the standard solution in the flame, and measure the absorbance.

2) Electrothermal type

Fit the specific light source(a hollow cathode lamp corresponding to element) to the lamp housing and switch on the instrument. After lighting the lamp and selecting the analytical wavelength specified in the individual monograph, set an appropriate electric current and slit-width. A suitable amount of sample solution, standard solution, prepared as specified in the individual monograph, is injected to the furnace and an appropriate stream of inert gas is made to flow through the furnace. The specimen is dried and ashed, and the element included is atomized, on heating at appropriate temperature for an appropriate time in appropriate mode. The atomic absorption specified is observed and the intensity of absorption is measured.

2-12 Inductively coupled plasma atomic emission spectrometry

a. Analysis principle

Determine the concentration of the object element in test solution, by atomizing and exciting the element by inductively coupled plasma, and determining the intensity of atomic emission spectrum.

b. Apparatus

Usually the apparatus consists of excitation source part, sample introduction part, light emission part, spectroscope part and photometry part and indication and recording part. The excitation source part is composed of an electric power source, a control system, and circuit to supply and control the electric energy which excites and emits an element in sample. This part also includes a gas source and cooling system. The sample introduction part is composed of a nebulizer and a spray chamber. The light emission part is composed of a torch and an high- frequency induction coil. The spectroscope part is composed of a light-converging system and a spectroscope such as a diffracting grating. The photometry part is composed of a detector and a signal processing system. The indication and recording part is composed of a display and a recording system. The ICP-atomic emission spectrometry includes single-element-sequential-type- and multiple-element-sequential-type-measuring methods using a wavelength scanning spectroscope, and a simultaneously measuring method using a wavelength-fixed-type polychrometer.

c. Standard solution

Unless otherwise specified,, use standard solution corresponding to element.

d. Procedure

Confirm that all live parts are normal. Switch on the excitation source part and the control system. When a vacuum-type spectroscope is used to measure the emission line in vacuum-ultraviolet region, purge sufficiently the light-path between the light emission part and the spectroscope with argon or nitrogen gas for 10 min. Set the flow rate for argon or nitrogen gas to the specified rate, switch on the high frequency power, and generate the plasma. Correct the wavelength of spectroscope with the emission spectral line of a mercury lamp. Introduce the test solution and the standard solution prepared as specified in the individual monograph and measure the emission intensity of an appropriate emission line of the object element.

Compare the intensity of emission of test solution with the intensity of emission of standard solution obtained by same procedure with test solution, and calculate the concentration of object element in test solution.

2-13 Benzophenone Test Method

a. Analysis principle

For utensils, containers and packages on which printed non-food contact surface, measure benzophenone residue on food contact surface by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 10 mg of benzophenone and dissolve in methanol to be 100 mL. Take 0.6 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with methanol. This solution is used as standard solution(0.6 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared by using n-heptane as food simulant, take 10 mL of test solution, concentrate under reduced pressure, and dissolve the residue in methanol to make 10 mL. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorption detector(wavelength : 254 nm)

- Mobile phase : A : water, B : acetonitrile
- Gradient : Perform linear gradient from A:B(90:10) to A:B(40:60) for 30 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the liquid chromatography with each 20 μL of test solution and standard solution under 1) operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of peak in chromatogram of standards solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of benzophenone in chromatogram for test solution and standard solution. Calculate the content of benzophenone in test solution.

2-14 Toluene Test Method

a. Analysis principle

Measure toluene residue in synthetic polymer packages that is transformed when contents are input by gas chromatography

b. Apparatus

Gas chromatograph

c. Pre-treatment

Sample of width 40 cm and length 50 cm(0.2㎡) is properly cut by about 2 cm x 3 cm and put into 500 mL erlenmeyer flask and sealed by silicone rubber stopper. Tetrahydrofurane(THF) 1 μL as internal standards material is injected to the flask through silicone rubber stopper and allow to stand for 30 min in the f 80℃ drier.

d. Test procedure

1) Operation condition of gas chromatograph

- Column : DB-1 Capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 5 min and temperature is raised to 80℃ at a rate of 10℃/min. Hold at 80℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 230℃
- Injection mode : Split(10:1)
- Detector : Flame ionization detector

- Detector temperature : 250℃

- Carrier gas : Nitrogen or Helium (Flow rate : 1 mL/min)

2) Preparation of calibration curve

To each 500 mL erlenmeyer flask sealed with silicon rubber stopper, add 0.5 μL, 1 μL, 1.5 μL, 2.0 μL of toluene and 1μL of tetrahydrofurane as internal standard through silicon rubber stopper and allow to stand for 30 min in the 80℃ drier. Right after taking out erlenmeyer flask, using 1 mL of gas in headspace, which is taken thourgh silicon rubber stopper with gastight syringe, run gas chromatography under 1) operation condition of gas chromatograph. From the obtained chromatogram, measure the ratio of peak area of toluene against peak area of tetrahydrofurane. Then plot this on concentration of toluene and prepare calibration curve.

3) Test

According as C. Pre-treatment, right after taking out erlenmeyer flask, which is allowed to stand for 30 min in the 80℃ drier, using 1 mL of gas in headspace, which is taken through silicon rubber stopper with gastight syringe, run gas chromatography in the same manner as 2) Preparation of calibration curve. Measure the ratio of peak area of toluene against peak area of tetrahydrofurane Calculate the content of toluene(μL) using previously prepared calibration curve and calculate the content of toluene in sample under following equation.

$$\text{Toluene}(mg/m^2) = \frac{\text{amount of toluene from calibration curve}(\mu L) \times 0.866}{0.2\ m^2}$$

(0.866 :specific gravity of toluene)

2-15 Coloring agent Test Method

a. Analysis principle

Analyze coloring agent migrated from sample using water as food simulant by thin layer chromatograph or paper chromatograph.

b. Apparatus

1) Silicagel or polyamide thin layer

2) Paper(filter paper) : for paper chromatography

3) Developing bath

c. Reagent and Solution

1) Degreasing wool

Method 1 : Immerse 100 g of white wool into the solution which 1 ~ 4 mL of strong ammonia water is diluted with water. Sometimes stir, allow to stand for 30 ~ 60 min at 45℃. Then allow to stand in diluted ammonia water for a while. Scoop it, squeeze lightly, put into ammonia water(1→100) for a while, wash with hot water first, and then cool water, squeeze lightly again, and dry in the wind.

Method 2 : In soxhlet extraction apparatus, defat white wool fully with petroleum ether. Evaporate ether at room temperature, wash with water, squeeze lightly, and dry in the wind.

2) Developing solvent

a) Thin layer chromatography (Silicagel thin layer)

(1) Ethyl acetate : methanol : 28% ammonia water (4.5:1:1 or 3:1:1)

(2) Amyl alcohol : ethanol : 28% ammonia water (10:10:1)

(3) Methyl ethyl ketone : ethylene glycol monomethyl ether : ethanol : 28% ammonia water (20:15:12:1)

b) Thin layer chromatography(polyamide thin layer)

(1) Methanol : ethanol : isoamyl alcohol : 28% ammonia water (15:10:5:3)

(2) Pyridine : methanol : 28% ammonia water : water (5:6:1:16)

c) Paper chromatography

(1) Acetone : isoamyl alcohol : water (6:5:5)

(2) n-Butanol : anhydrous ethanol : 1% ammonia water (6:2:3)

(3) 25% Ethanol solution : 5% ammonia water (1:1)

d. Preparation of test solution

Prepare the test solution using water as food simulant under 5. Preparation of migration test solution for each material. To 20 mL of this solution, add 1mL of 1% acetic acid and 0.1 g of Degreasing wool . Shake well, mix, heat for 30 min in a water bath and scoop wool. If wool is not be colored, no detection. If wool is colored, put this colored wool into 5 mL of 1% ammonia solution. Heat for 30 min, scoop wool, neutralize with acetic acid and concentrate. This solution is used as test solution.

e. Test procedure

1) Thin layer chromatography

Designate a line about 20 mm distant from one end of the thin-layer plate as the starting line. At least 10 mm distant from both edges, spot the specified volumes of the test solution and the standard solution with about 3 mm in diameter. After drying, immersing 0.5 ~ 1 cm of the bottom of thin layer plate into developing solvent. develop this up to 8 ~ 15 cm. After finishing developing, observe and compare the location and color of each spots obtained

from test solution and standard solution under natural sunlight and then UV light(about 365nm).

2) Paper chromatography

With a pencil, a straight line is drawn at approximately 40 mm from the bottom edge of the chromatography paper. On this line, a specified amount of the test solution and the standard solution are spotted with a micro take or a capillary tube and dried. The spots should be approximately 20 mm apart from each other and the diameter of circle is about 5 mm. The filter paper is hung vertically on the stopper in a bath that contains a specified developing solvent without touching the wall of the bath. The filter paper is immersed approximately up to 10 mm from bottom edge in the solvent. The bath is sealed and set aside. When the solvent front reaches 13 ~ 25 cm from the spots, the paper is removed from the bath and dried. The positions and colors of the developed spots from the test and standard solutions are observed under natural sunlight and then UV light(about 365nm).

2-16 Vinyl chloride Test Method

a. Residue Test

1) Analysis principle

Extract vinyl chloride residues in poly(vinyl chloride) with N,N-dimethylacetamide and measure by gas chromatograph/mass spectrometer.

2) Apparatus

Gas chromatograph/mass spectrometer

3) Standard stock solution

Dissolve vinyl chloride in N,N-dimethyacetamide to make 5 ug/mL. This solution is used as standard stock solution of vinyl chloride.

4) Internal standard solution

Accurately weigh 50 mg of 1-chloropropane and dissolve in N,N-dimethyacetamide to make 100 mL. Take 1 mL of this solution into 100 mL volumetric flask and add N,N-dimethyacetamide to make 100 mL. This solution is used as internal standard solution(5 ug/mL).

5) Test procedure

a) Operation condition of gas chromatograph/mass spectrometer

- Column : Plot Q capillary column(0.32 mm I.D. × 30 m, 20 μm), DB-624(0.25 mm I.D. × 30 m, 1.4 μm) or its equivalent is used.
- Column temperature : Hold at 70℃ for 2 min and temperature is raised to 250℃ at a rate of 10℃/min. Hold at 250℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃

- Injection mode : Split(10:1)
- Detector : Mass spectrometer(monitor ion : 62(quantifier ion), 61, 64 (qualifier ion), internal standard : 42)
- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1 mL/min)

b) Qualitative test

Sample is finely cut by not more than 5×5 mm and accurately weigh 0.5 g of sample and put the sample into a 20 mL of glass vial for headspace. Add 5.1 mL of N,N-dimethyacetamide, 0.2 mL of internal standard solution and magnetic bar and seal it tightly. Hold sealed vial at 90℃, stir for 30 min at a constant rate so that the inside is stabilized. This solution is used as test solution. Seperately, prepare standard solution in the same manner as test solution by adding 5 mL of N,N-dimethyacetamide, 0.1 mL of standard stock solution and 0.2 mL of internal standard solution to 20 mL glass vial for headspace and seal tightly. This solution is used as standard solution. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography/mass spectrometry under (1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of vinyl chloride peak in chromatogram for standard solution are compared if they're identical.

c) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of vinyl chloride peak in chromatogram for standard solution in b) Qualitative test, the following test is performed.

Based on test result in b) Qualitative test, measure ratio of peak area of vinyl chloride against peak area of 1-chloropropane in chromatogram for test solution and standard solution. Then calculate the content of vinyl chloride in sample

under following equation.

$$\text{Content(mg/kg)} = w \times \frac{Rt}{Rs} \times \frac{1}{\text{Weight of sample(g)}}$$

w : Contents of vinyl chloride in standard solution (μg)

Rt : Ratio of peak area of vinyl chloride against peak area of 1- chloropropane in chromatogram for test solution

Rs : Ratio of peak area of vinyl chloride against peak area of 1- chloropropane in chromatogram for standard solution

b. Migrant Test

1) Analysis principle

Measure vinyl chloride migrated from poly(vinyl chloride) by gas chromatograph/mass spectrometry.

2) Apparatus

Gas chromatograph/mass spectrometry

3) Standard solution

Dissolve vinyl chloride in ethanol to make 0.05 ug/mL. This solution is used as standard solution.

4) Internal standard solution

Proceed directly under a. Residue Test 4) Internal standard solution.

5) Preparation of test solution

For sample that can be filled with liquid is filled with ethanol, which is cooled at 5℃ or lower, sealed and allow to stand at 5℃ or lower for 24 hours. For sample that cannot be filled with liquid is used with ethanol, which is cooled at 5℃ or lower, at the rate 2 mL/cm^2 surface area, and allow to stand in sealed

containers at 5℃ or lower for 24 hours. These solutions are used as test solution respectively.

6) Test procedure

a) Operation condition of gas chromatograph/mass spectrometer

Proceed directly under a. Residue Test 5) Test procedure.

b) Qualitative test

Take each 10 mL of test solution and standard solution into 20 mL glass vial respectively. Add 0.2 mL of internal standard solution and magnetic bar into each solution and seal it tightly. Hold each sealed vial at 50℃, stir for 30 min at a constant rate so that the inside is stabilized. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography under a) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of vinyl chloride peak in chromatogram for standard solution are compared if they're identical.

c) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of vinyl chloride peak in chromatogram for standard solution in b) Qualitative Test, the following test is performed.

Based on test result in b) Qualitative Test, measure ratio of peak area of vinyl chloride against peak area of 1-chloropropane in chromatogram for test solution and standard solution. Then calculate contents of vinyl chloride in test solution.

2-17 Dibutyltin compound Test Method

a. Analysis principle

Extract dibutyltin compound residues in poly(vinyl chloride) with mixture of acetone · hexane[3:7(v/v)] in acidic condition. Derivatize with sodium tetraethylborate and measure by gas chromatograph/mass spectrometer.

b. Apparatus

Gas chromatograph/mass spectrometer

c. Reagent and solution

1) Sodium tetraethylborate solution

Dissolve sodium tetraethyborate in water to make 20 mg/mL. Prepare before use.

2) Acetic acid · sodium acetate buffer solution

Dissolve 12 g of acetic acid in water to make 100 mL and dissolve 16.4 g of sodium acetate in water to make 100 mL. Mix both solutions at the rate of 3 : 7(v/v).

d. Standard solution

Accurately weigh 100 mg of dibutyltin dichloride and dissolve in 50 mL of acetone and 2~3 drops of hydrochloric acid. Add acetone to make 100 mL. Take 1 mL of this solution into a 1000 mL volumetric flask and add 100 mL of n-hexane and 2~3 drops of hydrochloric acid and mix. Again, add n-hexane to make 1,000 mL. This solution is used as standard solution(1 μg/mL).

e. Preparation of test solution

Sample is finely cut by not more than 5×5 mm and accurately weigh 0.5 g of sample and put the sample into a flask. Add 20 mL of mixture of acetone · hexane solution[3:7(v/v)] and 1 drop of hydrochloric acid and place the stopper. Hold at 40℃, shake occasionally and allow to stand for a night. After cooling down the solution, filter this solution. Concentrate to about 1 mL under 40℃ with a rotary evaporator. Transfer the entire solution into a 25 mL volumetric

flask with hexane to make 25 mL. If necessary, the supernatant centrifuged at 2500 rpm for 10 min can be used as test solution.

f. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : DB-5 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 70℃ for 3 min and temperature is raised to 280℃ at a rate of 10℃/min. Hold at 280℃ for 10 min. Adjust the temperature properly if necessary.
- Injection Temperature : 240℃
- Injection Mode : Split(10 : 1)
- Detector : Mass spectrometer(monitor ion : 263(quantifier ion), 207, 235 (qualifier ion))
- Ionization Mode : EI mode
- Ionization Energy : 70 eV
- Carrier Gas : Helium(Flow rate : 1 mL/min)

2) Qualitative test

Take each 2 mL of test solution and standard solution into 20 mL glass vial respectively. Add 5 mL of acetic acid · sodium acetate buffer solution and 1 mL of sodium tetraethyborate solution respectively and seal tightly. Shake each sealed vial vigorously for 20 min and mix. Allow the solution to stand for 1 hour at room temperature and take hexane phase. Run the gas chromatography/mass spectrometry with each 1 μL of solution under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of dibutyl tin compound derivative peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with retention time of dibutyl tin compound derivative peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of dibutyl tin compound derivative in chromatogram for test solution and standard solution. Then calculate the content of dibutyl tin compound in sample under following equation.

$$\text{Content(mg/kg)} = c \times \frac{At}{As} \times \frac{25(mL)}{\text{Weight of sample(g)}}$$

c : Concentration of dibutyl tin compound in standard solution(μg/mL)

At : Peak area of dibutyl tin compound derivative in chromatogram for test solution

As : Peak area of dibutyl tin compound derivative in chromatogram for standard solution

2-18 Cresol esters of phosphoric acid Test Method

a. Analysis principle

Extract cresol esters of phosphoric acid residues in poly(vinyl chloride) with acetonitrile and measure by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 100 mg of tricresyl phosphate and dissolve in acetonitrile to make 100 mL. Take 1 mL of this solution into a 100 mL flask and add mixture of acetonitrle · water[2:1(v/v)] to make 100 mL. This solution is used as standard solution(10 μg/mL).

d. Preparation of test solution

Sample is finely cut by not more than 5×5 mm and accurately weigh 0.5 g of sample and put the sample into a flask. Add 15 mL of acetonitrile and place the stopper and hold at 40℃. Shake occasionally and allow to stand for a night. After cooling down the solution, filter this solution. Add acetonitrile to make 25 mL. This solution is acetonitrile extract. Slowly inject the mixture of 5 mL of acetonitrile extract and 5 mL of water into C_{18} cartridge, which is previously activated by passing 5 mL of acetonitrile and 5 mL of mixture of acetonitrile · water [1:1(v/v)]. Take 10 mL of the solution which is extracted with acetonitrile·water mixture[2:1(v/v)]. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector (wavelength : 264 nm)
- Mobile phase : A : water, B : 95% acetonitrile

- Gradient : Perform linear gradient from A:B(80:20) to A:B(20:80) for 20 min. Properly control if necessary.
- Flow Rate : 1 mL/min

2) Qualitative test

Run the liquid chromatography with each 20 μL of test solution and standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of tricresyl phosphate peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with retention time of tricresyl phosphate peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure peak area of tricresyl phosphate in chromatogram for test solution and standard solution. Then calculate the content of tricresyl phosphate in sample under following equation.

$$\text{Content(mg/kg)} = c \times \frac{At}{As} \times \frac{25(\text{mL}) \times 2}{\text{Weight of sample(g)}}$$

c : Concentration of tricresyl phosphate in standard solution (μg/mL)

At : Peak area of tricresyl phosphate in chromatogram for test solution

As : Peak area of tricresyl phosphate in chromatogram for standard solution

2-19 Di-n-butylphthalate, Benzyl-n-butylphthalate, Di-(2-ethylhexyl)phthalate, Di-n-octylphthalate, Diisononylphthalate, Diisodecylphthalate, and Di-(2-ethyl hexyl)adipate Test Method

a. Analysis principle

Measure di-n-butylphthalate, benzyl-n-butylphthalate, di-(2-ethylhexyl) phthalate, di-n-octylphthalate, diisononylphthalate, diisodecylphthalate and di-(2-ethylhexyl)adipate, which are migrated from poly(vinyl chloride), by gas chromatograph/mass spectrometer.

b. Apparatus

Gas chromatograph/mass spectrometer

c. Standard solution

1) Standard stock solution

Accurately weigh 30 mg of di-n-butylphthalate(DBP), 3,000 mg of benzyl-n-butylphthalate(BBP), 150 mg of di-(2-ethylhexyl)phthalate(DEHP), 500 mg of di-n-octylphthalate(DNOP), 900 mg of diisononylphthalate(DINP), 900 mg of diisodecylphthalate(DIDP), and 1,800 mg of di-(2-ethylhexyl) adipate(DEHA) and dissolve in acetone to make 100 mL respectively. These solutions are used as each standard stock solution.

2) Mixed standard solution

Take 5 mL each of standard stock solution(di-n-butylphthalate, benzyl -n-butylphthalate, di-(2-ethylhexyl)phthalate, di-n-octylphthalate and di-(2-ethylhexyl)adipate) into 100 mL volumetric flask and diluted to 100 mL with acetone. Again, take 2 mL of this solution into a 100 mL volumetric flask and diluted to 100 mL with acetone. This solution is used as mixed standard solution(0.3 μg/mL of di-n-butylphthalate, 30 μg/mL of benzyl-n-butylphthalate, 1.5 μg/mL of di-(2-ethylhexyl)phthalate, 5.0 μg/mL of di-n-octylphthalate, and 18 μg/mL of di-(2-ethylhexyl)adipate).

3) Standard solution of diisononylphthalate

Take 5 mL of standard stock solution of diisononylphthalate into a 100 mL volumetric flask and dilute 100 mL with acetone. Take 2 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with acetone. This solution is used as standard solution of diisononylphthalate(9 μg/mL).

4) Standard solution of diisodecylphthalate

Take 5 mL of standard stock solution of diisodecylphthalate into a 100 mL volumetric flask and dilute 100 mL with acetone. Take 2 mL of this solution into a 100 mL volumetric flask and dilute 100 mL with acetone. This solution is used as standard solution of diisodecylphthalate(9 μg/mL).

d. Preparation of test solution

1) Utensils, containers and packages contacting with food containing oil·fat or fatty food

Prepare the test solution using n-heptane as food simulant under 2-6 Preparation of migration test solution for each material Separately, prepare blank test solution in the same manner by using n-heptane instead of sample. Glass apparatus for migration test should be washed with food simulant, n-heptane or heated for a few hours at 180~200℃ before use. Food simulant, n-heptane could be contaminated by di-(2-ethylhexyl)phthalate, etc. so, check if it is contaminated before use.

2) Utensils, containers and packages contacting with the food other than food containing oil·fat or fatty food

Utensils, containers and packages contacting with food, which is listed in the column 1 of the following table, are prepared test solution using each solvent of column 2 as food simulant, according to 2-6 Preparation of Migration test solution for each material. Transfer 25 mL of the solution to separatory funnel and add 50 mL of acetone · hexane mixture [1:1(v/v)] and vigorously shake for 5 min and allow to stand. Transfer acetone·hexane phase into a 250 mL

flask. Add 50 mL of acetone·hexane mixture to the filtrate and prepare twice in the same manner as above. Add acetone·hexane phase to the flask above, concentrate under reduced pressure. Dissolve the residues in acetone to make 25 mL. This solution is used as test solution. Separately, prepare blank test solution in the same manner by using food simulant instead of sample. Glass apparatus for migration test should be washed with food simulant, n-heptane or heated for a few hours at 180~200℃ before use. Solvent used as food simulant should be check if it is contaminated by di-(2-ethylhexyl)phthalate etc. before use.

Column 1		Column 2
Alcoholic food	Food of not more than alcohol content of 20%	20% alcohol
	Food of more than alcohol contents of 20%	50% alcohol
Food of other than oil·fat and fatty food, and alcoholic food	Food of not more than pH 5	4% acetic acid
	Food of more than pH 5	water

e. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : DB-1701 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 120℃ for 2 min and temperature is raised to 280℃ at a rate of 10℃/min. Hold at 280℃ for 10 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Split(10 : 1)
- Detector : Mass spectrometer(monitor ion : 129, 149)

- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium (Flow rate : 1 mL/min)

2) Qualitative test

Run the gas chromatography/mass spectrometry with each 1 μL of test solution, mixed standard solution, diisononylphthalate standard solution, and diisodecylphthalate standard solution under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of each substance peak in chromatogram for mixed standard solution, diisononylphthalate standard solution or diisodecylphthalate standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of each substance peak in chromatogram for mixed standard solution, diisononylphthalate standard solution or diisodecylphthalate standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure peak area of each substance in chromatogram for test solution, mixed standard solution, diisononylphthalate standard solution or diisodecylphthalate standard solution. Then calculate each content of each substance in test solution. Separately, measure each content of each substance in blank test solution and adjust the each content of substance.

2-20 1-Hexene and 1-Octene Test Method

a. Analysis principle

Measure 1-hexene and 1-octene migrated from polyethylene by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

1) Standard stock solution

Accurately weigh 30 mg of 1-hexene and 150 mg of 1-octene and dissolve in dimethylacetamide to make 100 mL, respectively. This solution is used as each standard stock solution.

2) Mixed standard solution

Take 1 mL of each standard stock solution into 100 mL volumetric flask and add food simulant to make 100 mL. This solution is used as mixed standard solution(3 μg/mL of 1-hexene and 15 μg/mL of 1-octene).

3) Internal standard solution

Accurately weigh 50 mg of isooctane(2,2,4-trimethylpentane) and dissolve in dimethylacetamide to make 100 mL. This solution is used as internal standard solution(500 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material, This solution is used as test solution.

e. Test procedure

1) Operation condition of gas chromatograph

- Column : HP-1 capillary column(0.25 mm I.D. × 60 m, 1 μm) or its equivalent is used.

- Column temperature : Hold at 60℃ for 5 min and temperature is raised to 80℃ at a rate of 5℃/min. Hold at 80℃ for 3 min. Again, the temperature is raised to 180℃ at a rate of 10℃/min. Hold at 180℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 150℃
- Injection mode : Split(5 : 1)
- Detector : Flame ionization detector
- Detector temperature : 250℃
- Carrier gas : Nitrogen or Helium (Flow rate : 1 mL/min)

2) Qualitative test

a) When n-heptane is used as food simulant

Run the gas chromatography/mass spectrometry with each 1 μL of test solution and mixed standard solution under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of 1-hexene or 1-octene peak in chromatogram for mixed standard solution are compared if they're identical.

b) When water, 4% acetic acid or 20% ethanol is used as food simulant

Take each 5 mL of test solution and mixed standard solution into 20 mL glass vial respectively. Add 50 μL of internal standard solution and magnetic bar into each solution and seal tightly. Hold each sealed vial at 70℃, stir for 40 min at a constant rate so that the inside is stabilized. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of 1-hexene or 1-octene peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 1-hexene or 1-octene peak in chromatogram for mixed standard solution in 2) Qualitative Test, the following test is performed.

a) When n-heptane is used as food simulant

Based on test result in 2) Qualitative Test, measure peak area of 1-hexene or 1-octene in chromatogram for test solution and mixed standard solution. Then calculate the content of 1-hexene or 1-octene in test solution.

b) When water, 4% acetic acid or 20% ethanol or 50% ethanol is used as food simulant

Based on test result in 2) Qualitative Test, measure ratio of peak area of 1-hexene or 1-octene against peak area of isooctane in chromatogram for test solution and mixed standard solution. Then calculate content of 1-hexene or 1-octene in test solution.

2-21 Volatile organic compounds Test Method

a. Analysis principle

Extract styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene with tetrahydrofuran and measure by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

1) Mixed standard stock solution

Accurately weigh each 50 mg of styrene, toluene, ethyl benzene, isopropyl benzene and n-propyl benzene and dissolve in tetrahydtofuran to make 100 mL. This solution is used as mixed standard stock solution.

2) Mixed standard solution

Take 1 mL, 2 mL, 3 mL, 4 mL, and 5 mL of mixed standard stock solution into each of 20 mL volumetric flask. Add 1 mL of internal standard solution into each the solution and add tetrahydrofuran to make 20 mL. Each of the solution is mixed standard solution(25 μg/mL of styrene, 50 μg/mL of toluene, 75 μg/mL of ethyl benzene, 100 μg/mL of isopropyl benzene, 125 μg/mL of n-propyl benzene)

3) Internal standard solution

Take 1 mL of cyclopentanol into a 100 mL volumetric flask and add tetrahydrofuran to make 100 mL. Take 10 mL of this solution and add tetrahydrofuran to make 100 mL. This solution is used as internal standard solution.

d. Preparation of test solution

Sample is finely cut by not more than 5×5 mm and accuretely weigh 0.5 g of sample and put the sample into a flask. Add a suitable amount of

tetrahydtofuran. After dissolving the sample, add 1 mL of internal standard solution and add tetrahydrofuran to make 20 mL. This solution is used as test solution. If necessary, centrifuged supernatant can be used as test solution.

e. Test procedure

1) Operation condition of gas chromatograph

- Column : DB-Wax capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 3 min and temperature is raised to 150℃ at a rate of 5℃/min. Hold at 150℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Splitless
- Detector : Flame ionization detector
- Detector temperature : 280℃
- Carrier gas : Nitrogen or Helium (Flow rate : 0.7 mL/min)

2) Preparation of calibration curve

Run gas chromatography under 1) Operation condition of gas chromatograph using 1 μL each of mixed standard solution. From the obtained chromatogram, measure ratio of each peak area of styrene, toluene, ethyl benzene, isopropyl benzene or n-propyl benzene against peak area of cyclopentanol. Then plot this on concentration of each substance and prepare calibration curve.

3) Test

Run gas chromatography under same manner as 2) Preparation of calibration curve using 1 μL of test solution. From the obtained chromatogram, measure ratio of each peak area of styrene, toluene, ethyl benzene, isopropyl benzene or n-propyl benzene against peak area of cyclopentanol. Measure the concentration of styrene, toluene, ethyl benzene, isopropyl benzene or n-propyl benzene in test

solution using previously prepared calibration curve. Then calculate the content of each substance in sample under following equation.

$$\text{Content (mg/kg)} = \frac{\text{Concentration of each substance}(\mu g/mL) \times 20(mL)}{\text{Weight of sample}(g)}$$

2-22 Vinylidene chloride Test Method

a. Analysis Principle

Extract vinylidene chloride residues in poly(vinylidene chloride) with N,N-dimethylacetamide and measure by gas chromatograph/mass spectrometry.

b. Apparatus

Gas chromatograph/mass spectrometer

c. Standard stock solution

Dissolve vinylidene chloride in N,N-dimethyacetamide to make 6 ug/mL. This solution is used as standard stock solution of vinylidene chloride.

d. Internal standard solution

Accurately weigh 50 mg of 1-chloropropane and dissolve in N,N-dimethyacetamide to make 100 mL. Take 1 mL of this solution into 100 mL volumetric flask and add N,N-dimethyacetamide to make 100 mL. This solution is used as internal standard solution(5 ug/mL).

e. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : Plot Q capillary column(0.32 mm I.D. × 30 m, 20 μm), DB-624(0.25 mm I.D. × 30 mm, 1.4 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to 200℃ at a rate of 10℃/min. Hold at 200℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Split(10:1)
- Detector : Mass spectrometer(monitor ion : 61(quantifier ion), 96, 98 (qualifier

ion), internal standard : 42)

- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1 mL/min)

2) Qualitative test

Sample is finely cut by not more than 5×5 mm and acurately weigh 0.5 g of sample and put the sample into 20 mL of glass vial for headspace. Add 5.5 mL of N,N-dimethyacetamide, 0.2 mL of internal standard solution and magnetic bar and seal tightly. Hold at 90℃, stir for 30 min at a constant rate so that the inside is stabilized. This solution is used as test solution. Seperately, prepare standard solution in the same manner as test solution by adding 5 mL of N,N-dimethyacetamide, 0.5 mL of standard stock solution and 0.2 mL of internal standard solution to 20 mL glass vial for headspace and seal tightly. This solution is used as standard solution. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography/mass spectrometry under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of vinylidene chloride peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of vinylidene chloride peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure ratio of peak area of vinylidene chloride against peak area of 1-chloropropane in chromatogram for test solution and standard solution. Then calculate the content of vinylidene chloride in sample under following equation.

$$\text{Content}(mg/kg) = w \times \frac{Rt}{Rs} \times \frac{1}{\text{Weight of sample}(g)}$$

w : Contents of vinylidene chloride in standard solution(μg)

Rt : Ratio of peak area of vinylidene chloride against peak area of 1-chloropropane in chromatogram for test solution

Rs : Ratio of peak area of vinylidene chloride against peak area of 1-chloropropane in chromatogram for standard solution

2-23 Barium Test Method

a. Analysis principle

Measure barium migrated from Poly(vinylidene chloride), using 4% acetic acid as food simulant by atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer.

b. Apparatus

Atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer

c. Standard solution

Accurately weigh 190.3 mg of barium nitrate and dissolve in 0.1M nitric acid to make 100 mL. Take 1mL of this solution into a 1,000 mL volumetric flask, add 0.1M nitric acid to make 1,000 mL. This solution is used as standard solution(1 μg/mL).

d. Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material.

e. Test Procedure

For test solution and standard solution proceed as directed under 2-11 Atomic absorption spectrophotometry(wavelength : 553.6 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry(wavelength: 455.4 nm). Calculate the concentration of barium in test solution.

2-24 Germanium Test Method

a. Analysis principle

Measure germanium migrated from Poly(ethyleneterephthalate), using 4% acetic acid as food simulant, by atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer.

b. Apparatus

Atomic absorption spectrophotometer or inductively coupled plasma atomic emission spectrometer

c. Standard solution

Transfer 144 mg of germanium dioxide into platinum crucible and add 1 g of sodium carbonate. Mix throughly, heat, dissolve, cool, and dissolve in water. Neutralize the solution with hydrochloric acid, add above 1 mL of hydrochloric acid, and add water to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. Again, take 10 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. This solution is used as standard solution(1.0 μg/mL).

d. Preparation of test solution

Prepare the solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. Transfer 200 mL of this solution into a beaker and evaporate to 20 mL. This solution is used as test solution.

e. Test procedure

For test solution and standard solution proceed as directed under 2-11. Atomic absorption spectrophotometry(wavelength : 265.2 nm) or 2-12. Inductively coupled plasma atomic emission spectrometry(wavelength : 265.1 nm). Calculate the concentration of germanium in test solution(adjust with concentration factor 10).

2-25 Terephthalic acid and Isophthalic acid Test Method

a. Analysis principle

Measure terephthalic acid and isophthalic acid, which is migrated from Poly(ethyleneterephthalate) with liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Mixed standard solution

Accurately weigh 75 mg of terephthalic acid and 50 mg of isophthalic acid and dissolve in 2 mL of 1N sodium hydroxide. Again, add 50% acetonitrile to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 50% acetonitrile. This solution is used as mixed standard solution(terephthalic acid 7.5 μg/mL and isophthalic acid 5.0 μg/mL).

d. Preparation of test solution

Prepare the solution under 2-6 Preparation of migration test solution for each material This solution is used as test solution. However, for the test solution prepared using n-heptane as food simulant, take 10 mL of test solution and concentrate under reduced pressure. Dissolve the residue in 200 μL of 1N sodium hydroxide solution and add 50% acetonitrile to make 10 mL. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 230 nm)
- Mobile phase : A : Add 2.5 mL of phosphoric acid to 1 L of water

B : Add 2.5 mL of phosphoric acid to 1 L of 95% acetonitrile

- Gradient : Perform linear gradient from A:B(100:0) to A:B(0:100) for 20 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the liquid chromatography with each 10 uL of test solution and standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of terephthalic acid and isophthalic acid peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of terephthalic acid and isophthalic acid peak in chromatogram for mixed standards solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of terephthalic acid or isophthalic acid in chromatogram of test solution and mixed standard solution. Then calculate the content of terephthalic acid and isophthalic acid in test solution.

2-26 Phenol Test Method

a. Analysis principle

Measure phenol migrated from phenol-formaldehyde resin, using 4% acetic acid as food simulant by liquid chromatograph.

b Apparatus

Liquid chromatograph

c Standard solution

Accurately weigh 50 mg of phenol and dissolved in 4% acetic acid to make 100 mL. Take 1mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 4% acetic acid. This solution is used as standard solution(5 μg/mL).

d Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution.

e Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : Fluorescence detector(excitation wavelength : 275 nm, emission wavelength : 300 nm)
- Mobile phase : A : water, B : acetonitrile
- Gradient : Perform linear gradient from A:B(100:0) to A:B(0:100) for 30 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative pest

Run the liquid chromatography with each 10 uL of test solution and standard solution under 1) Operation condions of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of phenol peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of phenol peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of phenol in chromatogram for test solution and standard solution. Then calculate the content of phenol in test solution.

2-27 Formaldehyde Test Method

a. Analysis principle

Measure formaldehyde migrated from phenol-formaldehyde resin using 4% acetic acid as food simulant, by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Reagent and solution

1) 2,4-dinitrophenylhydrazine solution

Accurately weigh 300 mg of 2,4-dinitrophenylhydrazine and dissolve in acetonitrile to make 100 mL. This solution is used as 2,4-dinitrophenylhydrazine solution.

2) Citric acid buffer solution

Dissolve 21.0 g of citric acid monohydrate in water to make 100 mL and dissolve 25.8 g of trisodium citrate in water to make 100 mL. Mix both solutions at the rate of 8:2(v/v).

d. Standard solution

Dissolve formaldehyde in 4% acetic acid to make 4 ug/mL. This solution is used as standard solution of formaldehyde.

e. Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution. However, use water as food simulant in case of the test solution for paper or processed paper.

f. Derivatization

Take 25 mL of each test solution and standard solution into 50 mL volumetric

flask. Add 4 mL of citric acid buffer solution and 2 mL of 2,4-dinitrophenylhydrazine solution, respectively and seal tightly. Allow to stand at 40℃ for 1 hour while occasionally shaking. After cooling, add water to make 50 mL.

g. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 354 nm)
- Mobile phase : 55% acetonitrile
- Flow rate : 1 mL/min

2) Qualitative test

Run the liquid chromatography with each 10 uL of test solution and standard solution, which are processed by f. Derivatization under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of formaldehyde derivative peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of formaldehyde derivative peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure peak area of formaldehyde derivatives in chromatogram for test solution and standard solution. Then calculate the content of formaldehyde derivative in test solution.

2-28 Melamine Test Method

a. Analysis principle

Measure melamine migrated from melamine-formaldehyde resin using 4% acetic acid as food simulant, by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Reagent and Solution

1) 0.1M phosphate buffer solution

Add phosphoric acid to 0.1M potassium dihydrogen phosphate solution so that pH adjusts 3.0.

d. Standard solution

Accurately weigh 100 mg of melamine[(2,4,6-triamino-1,3,5-triazine)] and dissolve in water to make 100 mL. Take 3 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 0.1M phosphate buffer solution. This solution is used as standard solution(30 μg/mL).

e Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution.

f Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 235 nm)
- Mobile phase : 0.1M phosphate buffer solution(pH 3.0)

- Flow rate : 0.8 mL/min

2) Qualitative test

Run the liquid chromatography with each 10 uL of test solution and standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of melamine peak in chromatogram for standard solution are compared if they're identical

3) Quantitative test

When the retention time of chromatogram peak for test solution coincides with the retention time of melamine peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure peak area of melamine in chromatogram for test solution and standard solution. Then calculate the content of melamine in test solution.

2-29 Methyl methacrylate Test Method

a. Analysis principle

Measure methyl methacrylate migrated from acrylic resin using 20% ethanol as a food simulant, by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

Accurately weigh 60 mg of methyl methacrylate and dissolve in 20% ethanol to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask, dilute to 100 mL with 20% ethanol. This solution is used as standard solution(6 μg/mL).

d. Preparation of test solution

Prepare the test solution using 20% ethanol as a food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : DB-1 capillary column(0.25 mm I.D. × 60 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 10 min and temperature is raised to 180℃ at a rate of 10℃/min. Hold at 180℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 200℃
- Injection mode : Splitless
- Detector : Flame ionization detector

- Detector temperature : 250℃

- Carrier gas : Nitrogen or Helium(Flow rate : 1 mL/min)

2) Qualitative test

Run the gas chromatography with each 2 uL of test solution and standard solution under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of methy methacrylate peak in chromatogram for standard solution are compared if they're identical

3) Quantitative test

When the retention time of chromatogram peak for test solution coincides with the retention time of methyl metacrylate peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of methyl methacrylate in chromatogram for test solution and standard solution. Then calculate the content of methyl methacrylate in test solution.

2-30 Caprolactam and Laurolactam Test Method

a. Analysis principle

Measure caprolactam and laurolactam migrated from polyamide using 20% ethanol as food simulant, by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard stock solution

Accurately weigh 150 mg of caprolactam 50 mg of laurolactam and dissolve in ether to make 100 mL, respectively. This solution is used as standard stock solution.

d. Mixed standard solution

Take 1 mL each of standard stock solution into 100 mL volumetric flask and diluted to 100 mL with ether. This solution is used as mixed standard solution(15 μg/mL of caprolactam, 5 μg/mL of laurolactam).

e. Preparation of test solution

Prepare the test solution using 20% ethanol as food simulant under 2-6 Preparation of migration test solution for each material, This solution is used as test solution. Transfer 50 mL of the solution to separatory funnel and add 20 mL of ether and vigorously shake and allow to stand. Transfer ether phase into a 50 mL flask. Add 20 mL of ether to the filtrate and prepare twice in the same manner as above. Add ether phase to the flask above to make 50 mL. This solution is used as test solution.

f. Test procedure

1) Operation condition of gas chromatograph

- Column : DB-5 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its

equivalent is used.

- Column temperature : Hold at 80℃ for 2 min and temperature is raised to 240℃ at a rate of 10℃/min. Hold at 240℃ for 2 min. Adjust the temperature properly if necessary.
- Injection temperature : 220℃
- Injection mode : Split(10:1)
- Detector : Flame ionization detector
- Detector Temperature : 240℃
- Carrier Gas : Nitrogen or Helium(Flow rate : 1 mL/min)

2) Qualitative test

Run the gas chromatography with each 1 uL of test solution and mixed standard solution under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of caprolactame and laurolactam peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of chromatogram peak for test solution coincides with the retention time of caprolactame and laurolactam peak in chromatogram for mixed standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of caprolactam and laurolactam in chromatogram for test solution and mixed standard solution. Then calculate the content of caprolactam in test solution.

2-31 Primary aromatic amine(in compliance with only the aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) Test Method

a. Analysis principle

Measure primary aromatic amine(aniline, 4,4'-methylenedianiline and 2,4-toluenediamine) migrated from polyamide using 4% acetic acid as food simulant, by liquid chromatograph/mass spectrometer/ mass spectrometer.

b. Apparatus

Liquid chromatograph/mass spectrometer/mass spectrometer

c. Standard solution

1) Standard stock solution

Accurately weigh each 10 mg of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine and dissolve in 4% acetic acid and make to 100 mL respectively. These solutions are used as each standard stock solution.

2) Mixed standard stock solution

Take 1 mL of each standard stock solution into a 100 mL volumetric flask and add 4% acetic acid and make to 100 mL. This solution is used as mixed standard stock solution(each of the solution is 1 μg/mL).

3) Mixed standard solution

Take 1 mL of mixed standard stock solution into a 100 mL volumetric flask and add 4% acetic acid and make to 100 mL. This solution is used as mixed standard solution(each of the solution is 0.01 μg/mL).

d. Preparation of test solution

Prepare the test solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph/mass spectrometer/mass spectrometer

- Column : C_{18}(2 mm I.D. × 150 mm, 3 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : mass spectrometer
  · Ionization : ESI(positive)
  · Capillary temperature : 330℃
  · Collision gas : Ar
  · Collision voltage : 23 V
  · Specific ion

	Precursor ion (m/z)	Fragment ion (m/z)
Aniline	94	51, 77
4,4'-Methylenedianiline	199	106, 182, 165
2,4-Toluenediamine	123	106, 108

Properly control if necessary.

- Mobile phase : A : Solution containing 4.7 mM pentafluoropropionic acid (75:25)
  B : Methanol solution containing 4.7 mM pentafluoropropionic acid (75:25)
- Gradient : Perform linear concentration gradient from A : B(100 : 0) to A : B(0 : 100) for 15 min, and hold for 5 min with A : B(0 : 100). Properly control if necessary.
- Flow Rate : 0.2 mL/min

2) Qualitative test

Run the liquid chromatography/mass spectrometry with each 20 μL of test solution, mixed standard solution under 1) Operation condition of liquid

chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of each substance peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine peak in chromatogram for mixed standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine. Using the content of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine in test solution from previously prepared calibration curve

<Preparation of calibration curve>

Run liquid chromatography under 1) Operation condition of liquid chromatograph using 20 μL of each mixed standard stock solution, diluted with step-by-step. From the obtained chromatogram, measure each peak area of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine. Then plot this on concentration of each substance and prepare calibration curve.

2-32 Ethylendiamine and Hexamethylendiamine

a. Analysis principle

Measure ethylendiamine and hexamethylendiamine, which is migrated from polyamide by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

1) Standard stock solution

Accurately weigh 120 mg of ethylenediamine and 24 mg of hexamethylenediamine and dissolve in water to make 100 mL respectively. These solutions are used as each standard stock solution.

2) Mixed standard solution

Take 1 mL of standard solution into 100 mL volumetric flask and add water to make 100 mL. This solution is used as mixed standard solution(12 μg/mL of ethylendiamine and 2.4 μg/mL of hexamethylendiamine).

3) Internal standard solution

Accurately weigh 25 mg of 1,3-propylenediamine and dissolve in water to make 100 mL. This solution is used as internal standard solution(250 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. This solution is used as test solution. However, for the test solution prepared using n-heptane as food simulant, transfer 50 mL of test solution into a 100 mL separatory funnel. Add 25 mL of 3% acetic acid, shake vigorously for 10 min, and allow to stand. Transfer 3% acetic acid phase into a 250 mL separatory funnel. Add 25 mL of 3% acetic acid to the remaining solution and proceed 2 times in the same manner above. Add the phase of 3% acetic acid to the 250 mL separatory funnel above. Then add 100 mL of diethyl ether, shake,

and take the 3% acetic acid phase. Proceed this procedure 2 times, and the phase of 3% acetic acid is diluted to 100 mL to be final test solution.

e. Test procedure

1) Operation condition of gas chromatograph

- Column : HP-1 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 100℃ for 3 min and temperature is raised to 270℃ at a rate of 25℃/min. Hold at 270℃ for 4 min. Adjust the temperature properly if necessary.
- Injection temperature : 250℃
- Injection Mmode : Split(10 : 1)
- Detector : Flame ionization detector
- Detector temperature : 300℃
- Carrier gas : Nitrogen or Helium(Flow rate : 1mL/min)

2) Qualitative test

Take each 2.5 mL of test solution and mixed standard solution in flask. Add 0.1 mL of internal standard solution, 2.5 mL of 3% ammonia water, 7.5 mL of 5M sodium hydroxide solution and 5 mL of toluene and mix. To this mixture, add 0.25 mL of ethylchloroformate drop-wise, stir for 15 min(about 1000 rpm) and allow to stand. Take 1 mL of supernatant and dry using nitrogen gas and dissolve in 0.5 mL of toluene. Run gas chromatography under 1) Operation condition of gas chromatograph using 1 μL of this solution. From the obtained chromatogram, retention time of peak in chromatogram for test solution and retention time of peak in chromatogram for mixed standard solution are compared if they're identical. If necessary, proceed gas chromatography/mass spectrometer under following <Operation condition>. Identify the detected peak is ethylendiamine or hexamethylendiamine from obtained mass spectrum. For ethylendiamine, ion of derivative, m/z 87, 102, and 115 should be identified. For

hexamethylendiamine, ion of derivative, m/z 74, 102, and 130 should be identified. For propylenediamine derivatives, which is internal standard, ion m/z 56, 102, and 116 should be identified.

<Operation condition>

- Column : HP-1 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 100℃ for 3 min and temperature is raised to 270℃ at a rate of 25℃/min. Hold at 270℃ for 4 min. Adjust the temperature properly if necessary.
- Injection temperature : 250℃
- Injection mode : Split(10 : 1)
- Detector : Mass spectrometer(scan mode)
- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1mL/min)

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of ethylendiamine and hexamethylendiamine peak in chromatogram for mixed standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure ratio of peak area of ethylenediamine and hexamethylendiamine against peak area of 1,3-propylenediamine in chromatogram for test solution and mixed standard solution. Then calculate contents of ethylenediamine or hexamethylendiamine in test solution. However, calculate the concentration of test solution, which is prepared using n-heptane as food simulant, by adjusting dilution factor

2-33 4-Methyl-1-pentene Test Method

a. Analysis principle

Measure 4-methyl-1-pentene migrated from polymethylpentene by gas chromatograph/mass spectrometer.

b. Apparatus

Gas chromatograph/mass spectrometer.

c. Standard solution

1) Standard stock solution

Dissolve 4-methyl-1-pentene in N,N-dimethyacetamide to make 5 ug/mL. This solution is used as standard stock solution.

2) Standard solution

Take 1 mL of 4-methyl-1-pentene into 100 mL volumetric flask and add food simulant to make 100 mL. This solution is used as 4-methyl-1-pentene standard solution(0.05 μg/mL).

d. Internal standard solution

Dissolve 1-chloropropane in N,N-dimethyacetamide to make 5 ug/mL. This solution is used as standard stock solution.

e. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material.

f. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : PLOT Q capillary column(0.32 mm I.D. × 30 m, 20 μm), DB-624(0.25 mm I.D. × 30 m, 1.4 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to

200℃ at a rate of 10℃/min. Hold at 200℃ for 5 min. Adjust the temperature properly if necessary.

- Injection temperature : 240℃
- Injection mode : Split(10:1)(Use splitless mode if n-heptane is used as the food simulant)
- Detector : Mass spectrometer(monitor ion : 56(quantifier ion), 69, 84(qualifier ion), internal standard : 42)
- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1 mL/min)

2) Qualitative test

a) When n-heptane is used as food simulant

Take each 5 mL of test solution and standard solution into 20 mL glass vial, respectively. Add 0.2 mL of internal standard solution into each solution and mix. Use 2 uL of each solution and run the gas chromatography/mass spectrometry under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of 4-methyl-1-pentene peak in chromatogram for standard solution are compared if they're identical.

b) When water, 4% acetic acid or 20% ethanol is used as food simulant

Take each 5 mL of test solution and standard solution into 20 mL glass vial respectively. Add 0.2 mL of internal standard solution and magnetic bar into each solution and seal them tightly. Hold each sealed vial at 90℃, stir for 30 min at a constant rate so that the inside is stabilized. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography/mass spectrometry under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of 4-methyl-1-pentene peak

in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 4-methyl-1-pentene peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure ratio of peak area of 4-methyl-1-pentene against peak area of 1-chloropropane in chromatogram for test solution and standard solution. Then calculate contents of 4-methyl-1-pentene in test solution.

2-34 Amine(in compliance with only the triethylamine and tributylamine)

a. Analysis principle

Extract triethylamine residue and tributylamine residue in polycarbonate, epoxy resin and measure by gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

1) Standard stock solution

Accurately weigh each 10 mg of triethylamine and tributylamine and dissolve in dichloromethane to make 100 mL respectively. These solutions are used as each standard stock solution.

2) Mixed standard solution

Take 0.5 mL of each standard stock solution into 100 mL volumetric flask and add dichloromethane to make 100 mL. This solution is used as mixed standard solution(each of the solution is 0.5 μg/mL).

d. Preparation of test solution

Sample is finely cut by not more than 5 x 5 mm and accurately weigh 1.0 g of the sample and put into 200 mL erlenmeyer flask. Add 20 mL of dichloromethane. After dissolving sample, precipitate polymer by slowly dropping 100 mL of acetone and stirring. After centrifuge at 3,000 rpm for about 10 min, the supernatant is concentrated to be 1 mL by using rotary concentrator. Then, dilute to 2 mL with dichloromethane. This solution is used as test solution.

e. Test procedure

1) Operation condition of gas chromatograph

- Column : DB-5 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 5 min and temperature is raised to

250℃ at a rate of 20℃/min. Hold at 250℃ for 5 min. Adjust the temperature properly if necessary.

- Injection temperature : 200℃
- Injection mode : Split(10:1)
- Detector : Nitrogen phosphorus detector
- Detector temperature : 250℃
- Carrier gas : Helium(Flow rate : 1mL/min)

2) Qualitative test

Run the gas chromatography with each 1 uL of test solution and mixed standard solution under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of triethylamine or tributylamine peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of triethylamine or tributylamine peak in chromatogram for mixed standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of triethylamine and tributylamine. Using the content of triethylamine and tributylamine in test solution from previously prepared calibration curve.

<Preparation of calibration curve>

Run liquid chromatography under 1) Operation condition of liquid chromatograph using 20 μL of each mixed standard stock solution, diluted with step-by-step. From the obtained chromatogram, measure each peak area of aniline, 4,4'-methylenedianiline and 2,4-toluenediamine Then plot this on concentration of each substance and prepare calibration curve.

$$\text{Content(mg/kg)} = \frac{\text{Concentration of each substance(μg/mL)} \times \text{2(mL)}}{\text{Weight of sample(g)}}$$

<Preparation of calibration curve>

Run gas chromatography under 1) Operation condition of gas chromatograph using 1 μL of each mixed stock solution, diluted with step-by-step. From the obtained chromatogram, measure each peak area of triethylamine and tributylamine. Then plot this on concentration of each substance and prepare calibration curve.

2-35 Bisphenol A(including Phenol and p-tert-Butylphenol) Test Method

a. Analysis principle

Measure bisphenol A, phenol and p-tert-butylphenol, which is migrated from polycarbonate, polyarylsulfone, polyarylate, epoxy resin and metal by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

1) Mixed standard stock solution

Accurately weigh each 10 mg of bisphenol A, phenol and p-tert-butylphenol and dissolve in methanol to make 100 mL. This solution used as mixed standard stock solution.

2) Mixed standard solution

Take 4 mL of mixed standard stock solution into a 100 mL volumetric flask and dilute to 100 mL with water. Take 0.5 mL, 5 mL, 10 mL, 15 mL, and 20 mL each of this solution into 100 mL volumetric flask respectively and dilute to 100 mL with water. This solution is used as mixed standard solution(0.02 μg/mL, 0.2 μg/mL, 0.4 μg/mL, 0.6 μg/mL, and 0.8 μg/mL for each of bisphenol A, phenol and p-tert-butylphenol).

d. Preparation of test solution

1) Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, transfer 25 mL of test solution into a separatory funnel. Add 10 mL of acetonitrile, vigorously shake for 5 min and allow to stand. Transfer the acetonitrile phase into a 25 mL volumetric flask. Again, add 10 mL of acetonitrile to the n-heptane phase, shaking vigorously and allow to stand.

Transfer acetonitrile phase into a 25 mL volumetric flask above, mix, and dilute to 25 mL with acetonitrile. This is final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : fluorescence detector(excitation wavelength : 275 nm, emission wavelength : 300 nm)
- Mobile phase : A : water, B : acetonitrile
- Gradient : Perform linear gradient from A:B(70:30) to A:B(0:100) for 35 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Preparation of calibration curve

Run liquid chromatography under 1) Operation condition of liquid chromatograph using 20 μL of each mixed standard solution. From the obtained chromatogram, measure each peak area of bisphenol A, phenol and p-tert-butylphenol. Then plot this on concentration of each substance and prepare calibration curve.

3) Test

Run gas chromatography under same manner as 2) Preparation of calibration curve using 20 μL of test solution. Measure peak area of bisphenol A, phenol or p-tert-butylphenol from obtained chromatogram. Then calculate content of bisphenol A, phenol or p-tert-butylphenol in test solution.

2-36 Diphenylcarbonate Test Method

a. Analysis principle

Measure diphenylcarbonate migrated from polycarbonate with liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 50 mg of diphenylcarbonate and dissolve in 50% acetonitrile to make 100 mL. Take 0.1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with 50% acetonitrile. This solution is used as standard solution(0.5 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. This solution is used as test solution. Transfer 50 mL of the solution to separatory funnel and add 30 mL of n-heptane and vigorously shake and allow to stand. Transfer n-heptane phase into a 250 mL flask. Add 30 mL of n-heptane to the filtrate and prepare twice in the same manner as above, concentrate under reduced pressure. Dissolve the residues in acetonitrile to make 5 mL. This solution is used as test solution. However, for test solution prepared by n-heptane as food simulant, take 50 mL of test solution, concentrate under reduced pressure. Dissolve the residues in acetonitrile to make 5 mL. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃

- Detector : UV absorbance detector(wavelength : 217 nm)
- Mobile phase : 70% acetonitrile
- Gradient : Perform linear gradient from A:B(70:30) to A:B(0:100) for 35 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the gas chromatography with 20 uL of test solution and standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of diphenylcarbonate peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of diphenylcarbonate peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure the peak area of diphenylcarbonate in chromatogram for test solution and standard solution. Then calculate the content of diphenylcarbonate in test solution.

2-37 Vinyl acetate Test Method

a. Analysis Principle

Measure vinyl acetate migrated from poly(vinylalcohol), ethylene-vinylacetate copolymer with gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

Dissolve vinyl acetate in water to make 12 ug/mL. This solution is used as standard solution of vinyl acetate.

d. Internal standard Solution

Dissolve methyl propionate in water to make 500 ug/mL. This solution is used as internal standard solution.

e. Preparation of test solution

Prepare the test solution, using warer as food simulant, under 2-6 Preparation of migration test solution for each material.

f. Test Procedure

1) Operation Condition of Gas Chromatograph

- Column : Plot Q capillary column(0.32 mm I.D. × 30 m, 20 μm), DB-624(0.25 mm I.D. × 30 m, 1.4 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to 200℃ at a rate of 10℃/min. Hold at 200℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 140℃
- Injection mode : Splitless
- Detector : Flame ionization detector

- Detector temperature : 200℃

- Carrier gas : nitrogen(Flow rate : 1 mL/min)

2) Qualitative test

Take each 5 mL of test solution and standard solution into 20 mL glass vial respectively. Add 100 μL of internal standard solution and magnetic bar into each solution and seal tightly. Hold each sealed vial at 80℃, stir for 30 min at a constant rate so that the inside is stabilized. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 1 mL of gas and run the gas chromatography under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of vinyl acetate peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of vinyl acetate peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure ratio of peak area of vinyl chloride against peak area of methyl propionate in chromatogram for test solution and standard solution. Then calculate the content of vinyl acetate in test solution.

2-38 Isocyanate Test Method

a. Analysis principle

Measure isocyanate migrated from polyurethane by color developing with naphthylethyldiamine.

b. Apparatus

Spectrophotometer

c. Reagent and Solution

1) Hydrochloric acid·Glacial acetic acid mixed solution

35 mL of hydrochloric acid(35%) and 22 mL of glacial acetic acid are mixed and add water to make 1,000 mL.

2) Diazotization solution

3 g of sodium nitrite and 5 g of sodium bromide are dissolved in water to make 100 mL.

3) Sulfamic acid solution

10 g of sulfamic acid is dissolved in water to make 100 mL.

4) Naphthylethyldiamine solution

50 mg of N-(1-naphthyl)-ethyldiamine dihydrochloride is dissolved in 25 mL of water and 1 mL of hydrochloric acid(35%) and add water to make 50 mL.

5) Sodium carbonate solution

160 g of anhydrous sodium carbonate is dissolved in water to make 1,000 mL.

d. Standard solution

Accurately weigh 100 mg of diphenylmethane-4,4'-diisocyanate into a 100 mL volumetric flask. Dissolve by adding 60 mL of glacial acetic acid and add water to make 100 mL. Take 5 mL of this solution into a 1,000 mL volumetric flask and dilute to 1,000 mL with 19 mL of glacial acetic acid, 35 mL of

hydrochloric acid, and water. This is used as standard solution(5 μg/mL).

e. Preparation of test solution

Prepare the test solution, using warer as food simulant, under 2-6 Preparation of migration test solution for each material.

f. Preparation of calibration curve

Transfer standard solution, hydrochloric acid·glacial acetic acid mixed solution and acetone into a 25 mL volumetric flask as table below. Add 0.5 mL of diazotization solution respectively and allow to stand for 3 min. Add 1 mL of sulfamic acid solution to each solution, shake and allow to stand for 3 min. Add 1 mL of naphthylethyldiamine solution and 1 mL of sodium carbonate solution, mix, and allow to stand for 15 min. Add water to each solution to make 25 mL and mix. Measure the absorbance of the solution at wavelength 550 nm by being compared with blank test. Plot the obtained absorbance on the content of each diphenylmethane -4,4'-diisocyanate and prepare the calibration curve.

Content of diphenylmethane -4,4'-diisocyanate(μg) / Test solution	Blank test	5	10	20	30
Standard solution(mL)	0	1	2	4	6
Hydrochloric acid · glacial acetic acid (mL)	15	14	13	11	9
Acetone	1	1	1	1	1

g. Test procedure

Take 50 mL of test solution into a 100 mL evaporating flask. Add 1 mL of hydrochloric acid(35%) and vacuum dry at 65~70℃. Dissolve in 15 mL of hydrochloric acid·glacial acetic acid mixed solution and 1 mL of acetone and transfer to a 25 mL volumetric flask. Add 0.5 mL of diazotization solution to this solution, mix, and allow to stand for 3 min. Add 1 mL of sulfamic acid solution to each solution, mix, and allow to stand for 3 min. Add 1 mL of naphthylethyldiamine solution and 1 mL of sodium carbonate solution, mix, and allow to stand for 15 min. Again, add water to each solution to make 25 mL and mix. Measure absorbance at wavelength 550 nm by being compared with

blank test. Calculate the content of isocyanate monomer in test solution using previously prepared calibration curve.

2-39 1,3-butadiene Test Method

a. Analysis Principle

Extract 1,3-butadiene residues in acrylonitrile-butadiene-styrene copolymer, methylmethacrylate-acrylonitrile-butadiene-styrene copolymer or rubber with N,N-dimethylacetamide and measure by gas chromatograph/mass spectrometer.

b. Apparatus

Gas chromatograph/mass spectrometer

c. Standard stock solution

Dissolve 1,3-butadiene in N,N-dimethyacetamide to make 5 ug/mL. This solution is used as standard stock solution of 1,3-butadiene.

d. Internal standard solution

Accurately weigh 50 mg of 1-chloropropane and dissolve in N,N-dimethyacetamide to make 100 mL. Take 1 mL of this solution into 100 mL volumetric flask and add N,N-dimethyacetamide to make 100 mL. This solution is used as internal standard solution(5 ug/mL).

e. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : Plot Q capillary column(0.32 mm I.D. × 30 m, 20 μm), DB-624(0.25 mm I.D. × 30 m, 1.4 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to 200℃ at a rate of 10℃/min. Hold at 200℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Split(10:1)
- Detector : Mass spectrometer(monitor ion : 54(quantifier ion), 39, 53 (qualifier

ion), internal standard : 42)

- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1 mL/min)

2) Qualitative test

Sample is finely cut by not more than 5×5 mm and accurately weigh 0.5 g of sample and put the sample into a 20 mL of glass vial for headspace. Add 5.1 mL of N,N-dimethyacetamide, 0.2 mL of internal standard solution and magnetic bar and seal tightly. Hold sealed vial at 90℃, stir for 30 min at a constant rate so that the inside is stabilized. This solution is used as test solution. Seperately, prepare standard solution in the same manner as test solution by adding 5 mL of N,N-dimethyacetamide, 0.1 mL of standard stock solution and 0.2 mL of internal standard solution to the glass vial for headspace and seal tightly. This solution is used as standard solution. Stick gastight syringe in headspace of each vial, test solution and standard solution. Take 0.5 mL of gas and run the gas chromatography/mass spectrometry under 1) Operation condition of gas chromatograph/mass spectrometer. Retention time of peak in chromatogram for test solution and retention time of 1,3-butadiene peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 1,3-butadiene peak in chromatogram for standard solution in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure ratio of peak area of 1,3-butadiene against peak area of 1-chloropropane in chromatogram for test solution and standard solution. Then calculate the content of 1,3-butadiene in sample under following equation.

$$\text{Content}(mg/kg) = w \times \frac{Rt}{Rs} \times \frac{1}{\text{Weight of sample}(g)}$$

w : Contents of 1,3-butadiene in standard solution(μg)

Rt : Ratio of peak area of 1,3-butadiene against peak area of 1-chloropropane in chromatogram for test solution

Rs : Ratio of peak area of 1,3-butadiene against peak area of 1-chloropropane in chromatogram for standard solution

2-40 Acrylonitrile Test Method

a. Analysis principle

Run gas chromatograph by absorbing acrylonitrile on solid phase microextraction fiber, which is migrated from acrylonitrile-butadiene-styrene copolymer, acrylonitrile-styrene copolymer, polyacrylonitrile or methylmethacrylate-acrylonitrile-butadiene-styrene copolymer using water as food simulant.

b. Apparatus

1) Gas chromatograph

2) Solid phase microextraction fiber

75 μm carboxen/polydimethylsiloxane or its equivalent(Expose fiber for 5 min to the injector of gas chromatograph before use to eliminate impurities)

c. Standard solution

After putting 20 mL of water into a 100 mL volumetric flask and place the stopper. Accurately weigh the flask with a stopper. Inject about 100 mg of acrylonitrile with syringe and accurately weigh the flask. Obtain the amount of added acrylonitrile. Inject water with syringe to make 100 mL. Take 1 mL of this solution into 100 mL volumetric flask and dilute 100 mL with water. Again, take 0.2 mL of the solution into a 100 mL volumetric flask and dilute 100 mL with water. This solution is used as standard solution(0.02 μg/mL).

d. Internal standard solution

After putting 20 mL of water into a 100 mL volumetric flask and place the stopper. Accurately weigh the flask with a stopper. Inject about 100 mg of propionitrile with syringe and accurately weigh the flask. Obtain the amount of added propionitrile. Inject water with syringe to make 100 mL. Again, take 0.1 mL of this solution into 100 mL volumetric flask and dilute to 100 mL with water. This solution is used as internal standard solution(1.0 μg/mL).

e. Preparation of test solution

Prepare the test solution, using warer as food simulant, under 2-6 Preparation of migration test solution for each material.

f. Test procedure

1) Operation condition of gas chromatograph

- Column : DB-5 capillary column(0.17 mm I.D. × 50 m, 0.32 μm) or its equivalent is used.
- Column temperature : 50℃. Adjust the temperature properly if necessary.
- Injection temperature : 260℃
- Injection mode : Splitless
- Detector : Nitrogen phosphorus detector
- Detector temperature : 270℃
- Carrier gas : Helium(Flow rate : 1mL/min)

2) Qualitative test

Take each 5 mL of test solution and standard solution into 10 mL glass vial respectively. Add 0.5 mL of internal standard solution, 1.75 g of sodium chloride and magnetic bar into each solution and seal tightly. Hold each sealed vial at 40℃, stir for 10 min at a constant rate so that the inside is stabilized. Stick solid phase microextraction fiber in headspace of vial. Absorb volatile compounds for 30 min. Inject absorbed solid phase microextraction fiber into gas chromatograph and run the gas chromatography under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of acrylonitrile peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with retention time of acrylonitrile peak in chromatogram for standard solution

in 2) Qualitative Test, the following test is performed.

Based on test result in 2) Qualitative Test, measure the ratio of peak area of acrylonitrile against peak area of propionitrile in chromatogram for test solution and standard solution. Then calculate content of acrylonitrile in test solution.

2-41 1,4-butanediol Test Method

a. Analysis Principle

Measure 1,4-butanediol migrated from poly(butyleneterephthalate), butylenesuccinate-adipate copolymer with gas chromatograph.

b. Apparatus

Gas chromatograph

c. Standard solution

Dissolve 1,4-butanediol in n-heptane to make 5 ug/mL. This solution is used as standard solution of 1,4-butanediol.

d. Internal standard solution

Dissolve 1,5-pentanediol in n-heptane to make 15 ug/mL. This solution is used as internal standard solution.

e. Preparation of test solution

Prepare the test solution, using n-heptane as food simulant, under 2-6 Preparation of migration test solution for each material.

f. Test Procedure

1) Operation Condition of Gas Chromatograph

- Column : DB-17 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to 250℃ at a rate of 10℃/min. Hold at 250℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Splitless
- Detector : Flame ionization detector

- Detector temperature : 250℃

- Carrier gas : Nitrogen(Flow rate : 1 mL/min)

2) Qualitative test

Take each 600 uL of test solution and standard solution into 2 mL glass vial respectively. Add 200 μL of ethyl acetate, 200 μL of internal standard solution and 200 μL of bis(trimethylsilyl)trifluoroacetamide(BSTFA) into each solution and seal tightly. Ultrasonicate each vial for 30 min, hold each sealed vial at 90℃, make 60 min reaction. Take 1 uL of liquid and run the gas chromatography under 1) Operation condition of gas chromatograph. Retention time of peak in chromatogram for test solution and retention time of 1,4-butanediol derivative peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 1,4-butanediol derivative peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure ratio of peak area of 1,4-butanediol derivative against peak area of 1,5-pentanediol derivative in chromatogram for test solution and standard solution. Then calculate the content of 1,4-butanediol in test solution.

2-42 4,4'-Dichlorodiphenyl sulfone Test Method

a. Analysis principle

Measure 4,4'-dichlorodiphenyl sulfone migrated from polyarylsulfone or polyethersulfone by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 10 mg of 4,4'-dichlorodiphenyl sulfone and dissolve in methanol to make 100 mL. Take 5 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with methanol. Again, take 1 mL of this solution into a 100 mL volumetric flask respectively and dilute to 100 mL with methanol. This solution is used as 4,4'-dichlorodiphenyl sulfone standard solution(0.05μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, transfer 50 mL of test solution into a separatory funnel. Add 20 mL of acetonitrile, shaking vigorously, and allow to stand. Transfer the acetonitrile phase into a 50 mL volumetric flask. Again, add 20 mL of acetonitrile to the n-heptane phase, shaking vigorously and allow to stand. Transfer acetonitrile phase into a 50 mL volumetric flask above, mix, and dilute to 50 mL with acetonitrile. This is final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 247 nm)

- Mobile phase : A : water, B : acetonitrile
- Gradient : Perform linear gradient from A:B(70:30) to A:B(20:80) for 20 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the gas chromatography with 50 uL of test solution and mixed standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of 4,4'-dichlorodiphenyl sulfone peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 4,4'-dichlorodiphenyl sulfone peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of 4,4'-dichlorodiphenyl sulfone in chromatogram for test solution and standard solution. Then calculate the content of diphenylcarbonate in test solution.

2-43 2,6-dimethylnaphthalene dicarboxylate Test Method

a. Analysis principle

Measure 2,6-dimethylnaphthalene dicarboxylate migrated from poly(ethylenenaphthalate) by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 10 mg of 2,6-dimethylnaphthalene dicarboxylate, put into 100 mL volumetric flask and add 30 mL of acetone. Dissolve the 2,6-dimethylnaphthalene dicarboxylate and dilute to 100 mL with acetonitrile. This solution is used as standard solution. Take 0.5 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with acetonitrile. This solution is used as standard solution(0.5 μg/mL)

d. Preparation of test solution

Slowly inject 20 mL of solution prepared using under 2-6 Preparation of migration test solution for each material into C_{18} cartridge, which is previously activated by passing 5 mL of acetonitrile and 5 mL of water. Take 2 mL of the solution which is slowly extracted with acetonitrile. This solution is used as test solution

e. Test Procedure

1) Operation condition of liquid chromatograph/mass spectrometer

- Column : C_{18}column(4.6 mm × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 244 nm)
- Mobile phase : A : Add 1.25 mL of phosphoric acid to 1 L of water

B : Add 1.25 mL of phosphoric acid to 1 L of acetonitrile

- Gradient : Perform linear gradient from A:B(100:0) to A:B(0:100) for 20 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the liquid chromatography with each 20 μL of test solution and standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of 2,6-dimethylnaphthalene dicarboxylate peak in chromatogram for standard solution are compared if they're identical(accordingly adjust with concentration factor 10).

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 2,6-dimethylnaphthalene dicarboxylate peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of 2,6-dimethylnaphthalene dicarboxylate in chromatogram for test solution and standard solution. Then calculate the content of 2,6-dimethylnaphthalene dicarboxylate in test solution.

2-44 Bisphenol A diglycidyl ether(including Bisphenol A diglycidyl ether dichloride and Bisphenol A diglycidyl ether dihydrate) and Bisphenol F diglycidyl ether (including Bisphenol F diglycidyl ether dichloride and Bisphenol F diglycidyl ether dihydrate) Test Method

a. Analysis principle

Measure bisphenol A diglycidyl ether(including bisphenol A diglycidyl ether dichloride and bisphenol A diglycidyl ether dihydrate) and bisphenol F diglycidyl ether(including bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate) migrated from epoxy resin by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

1) Mixed standard stock solution

Accurately weigh each 10 mg of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride and bisphenol F diglycidyl ether dihydrate and transfer into 100 mL volumetric flask. Dissolve in tetrahydrofuran to make 100 mL. This solution is used as mixed standard stock solution.

2) Mixed standard solution

Take 1 mL of mixed standard stock solution into a 100 mL volumetric flask and add methanol to make 100 mL. The solution is used as mixed standard solution(for each of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride, bisphenol F diglycidyl ether dihydrate, 1 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, take 25 mL of test solution and concentrate under a reduced pressure. Dissolve the residues in methanol to make 25 mL. This is final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : Fluorescence detector(excitation wavelength : 275 nm, emission wavelength : 300 nm)
- Mobile phase : A : water, B : methanol or acetonitrile
- Gradient : Perform linear gradient from A:B(60:40) to A:B(10:90) for 50 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run the gas chromatography with 20 uL of test solution and mixed standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride, bisphenol F diglycidyl ether dihydrate peak in chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride or bisphenol F diglycidyl ether

dihydrate. peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed. preparation of calibration cuve

Based on test result in 2) Qualitative test, measure the peak area of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride or bisphenol F diglycidyl ether dihydrate. Using the content of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride or bisphenol F diglycidyl ether dihydrate in test solution from previously prepared calibration curve.

<Preparation of Calibration curve>

Run liquid chromatography under 1) Operation condition of liquid chromatograph using 20 μL of each mixed standard solution, diluted with step-by-step. From the obtained chromatogram, measure each peak area of bisphenol A diglycidyl ether, bisphenol A diglycidyl ether dichloride, bisphenol A diglycidyl ether dihydrate, bisphenol F diglycidyl ether, bisphenol F diglycidyl ether dichloride or bisphenol F diglycidyl ether dihydrate Then plot this on concentration of each substance and prepare calibration curve.

2-45 Epichlorohydrine Test Method

a. Analysis principle

Measure epichlorohydrine migrated from epoxy resin using n-heptane as food simulant, by gas chromatograph/mass spectrometer.

b. Apparatus

Gas chromatograph/mass spectrometer

c. Standard solution

Accurately weigh 100 mg of epichlorohydrin and dissolve in n-heptane to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with n-heptane. Again, take 5 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with n-heptane. This solution is used as standard solution(0.5 μg/mL).

d. Preparation of test solution

Prepare the solution using n-heptane as food simulant under 2-6 Preparation of migration test solution for each material. This solution is used as test solution.

e. Test procedure

1) Operation condition of gas chromatograph/mass spectrometer

- Column : DB-Wax capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.
- Column temperature : Hold at 40℃ for 2 min and temperature is raised to 200℃ at a rate of 20℃/min. Hold at 200℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 240℃
- Injection mode : Split(10:1)
- Detector : mass spectrometer(monitor ion : 49(quantifier ion), 57, 62(qualifier

ion)

- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 0.6 mL/min)

2) Qualitative test

Run gas chromatography/mass spectrometry under 1) Operation condition of gas chromatograph/mass spectrometer by using each 1 uL of test solution and standard solution. Retention time of peak in chromatogram for test solution and retention time of epichlorohydrine in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of epichlorohydrine peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of epichlorohydrine in chromatogram for test solution and standard solution. Calculate the content of Epichlorohydrine in test solution.

2-46 1,4-Dichlororbenzene Test Method

a. Analysis principle

Measure 1,4-dichlorobenzene migrated from poly(phenylenesulfide) by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh 120 mg of 1,4-dichlorobenzene and dissolve in methanol to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with methanol(12 ug/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, transfer 25 mL of test solution into a separatory funnel. Add 10 mL of acetonitrile, shake vigorously for 5 min, and allow to stand. Transfer acetonitrile phase into a 25 mL volumetric flask. Add 10 mL of acetonitrile to the remaining solution and proceed in the same manner above. Add acetonitrile phase to the 25 mL volumetric flask above. Add acetonitrile to make 25 mL. This is a final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 225 nm)
- Mobile phase : 80% methanol

- Flow rate : 1 mL/min

2) Qualitative test

Run liquid chromatography under 1) Operation condition of liquid chromatograph by using 20 uL of each test solution and standard solution. Retention time of peak in chromatogram for test solution and retention time of 1,4-dichlorobenzene peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 1,4-dichlorobenzene peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of 1,4-dichlorobenzene in chromatogram for test solution and standard solution. Calculate the content of 1,4-dichlorobenzene in test solution.

2-47 4,4'-Dihydroxydiphenylsulfone Test Method

a. Analysis principle

Measure 4,4'-dihydroxydiphenylsulfone migrated from poly(ethersulfone) by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh each 100 mg of 4,4'-dihydroxydiphenylsulfone and dissolve in methanol to make 100 mL. Take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with methanol. Again, take 0.5 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with methanol(0.05 ug/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, transfer 50 mL of test solution into a separatory funnel. Add 20 mL of acetonitrile, shake vigorously for 5 min, and allow to stand. Transfer acetonitrile phase into a 50 mL volumetric flask. Add 20 mL of acetonitrile to the remaining solution and proceed in the same manner above. Add acetonitrile phase to the 50 mL volumetric flask above. Add acetonitrile to make 50 mL. This is a final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector(wavelength : 259 nm)

- Mobile phase : A : water, B : acetonitrile
- Gradient : Perform linear gradient from A:B(70:30) to A:B(20:80) for 20 min. Properly control if necessary.
- Flow rate : 1 mL/min

2) Qualitative test

Run liquid chromatography under 1) Operation condition of liquid chromatograph by using each 50 uL of test solution and standard solution. Retention time of peak in chromatogram for test solution and retention time of 4,4'-dihydroxydiphenylsulfone peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of 4,4'-dihydroxydiphenylsulfone peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of 4,4'-dihydroxydiphenylsulfone in chromatogram for test solution and standard solution. Calculate the content of 4,4'-dihydroxydiphenylsulfone in test solution.

2-48 Hydroquinone Test Method

a. Analysis principle

Measure hydroquinone migrated from Polyetheretherketone by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

Accurately weigh each 60 mg of hydroquinone and add the mixture of 10 mM potassium dihydrogen phosphate(KH_2PO_4) solution·acetonitrile[92:8(v/v)] to make 100 mL. Take 0.1 mL of the solution into a 100 mL volumetric flask and dilute to 100 mL with the mixture of 10 mM potassium dihydrogen phosphate(KH_2PO_4) solution·acetonitrile[92:8(v/v)]. This solution is used as standard solution(0.6 μg/mL).

d. Preparation of test solution

Prepare the test solution under 2-6 Preparation of migration test solution for each material. However, for the test solution prepared using n-heptane as food simulant, take 50 mL of test solution and concentrate under a reduced pressure. Dissolve the residues in the mixture of 10 mM potassium dihydrogen phosphate(KH_2PO_4) solution·acetonitrile[92:8(v/v)] to make 50 mL. This is final test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : UV absorbance detector (wavelength : 290 nm)
- Mobile phase : 10 mM potassium dihydrogen phosphate(KH_2PO_4) solution·

acetonitrile (92:8)

- Flow rate : 1 mL/min

2) Qualitative test

Run liquid chromatography under 1) Operation condition of liquid chromatograph by using each 10 uL of test solution and standard solution. Retention time of peak in chromatogram for test solution and retention time of hydroquinone peak in chromatogram for standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with the retention time of hydroquinone peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of hydroquinone in chromatogram for test solution and standard solution. Calculate the content of hydroquinone in test solution.

2-49 2-Mercaptoimidasoline Test Method

a. Analysis principle

Extract 2-mercaptoimidasoline residues in the rubber containing chlorine using methanol by sohxhlet extractor and then measure by thin-layer chromatograph.

b. Apparatus

1) Sohxhlet extractor

2) Thin-layer plate : Use a silica gel for thin-layer chromatography dried at 120℃ for 1 hour.

c. Reagent and Solution

1) 2,6-dichloroquinone · chloroimide solution

Dissolve 100 mg of 2,6-dichloroquinone · chloroimide in ethanol to make 10mL.

d. Standard solution

Accurately weigh 200 mg of 2-mercaptoimidasoline and dissolve in methanol to make 100 mL. Again take 1mL of this solution into a 100 mL volumetric flask and then dilute to 100 mL with methanol(20 μg/mL).

e. Preparation of test solution

Sample is finely cut by not more than 5×5 mm and 1.0 g of sample is put into thimble filter and extracted for 8 hours with 45 mL of methanol by soxhlet extractor. The extract is concentrated to be about 1 mL. This solution is used as test solution.

f. Test procedure

Each 10 uL of test solution and 2-mercaptoimidasoline standard solution is spotted to thin plate by using micropipette and dried. The thin plate was developed with the mixture of ethyl acetic acid·benzene[5:1(v/v)] and the mixture of ethyl acetic acid·methanol·ammonia water·water[30:2:1:1(v/v/v/v)], respectively. When developing solvent goes up to about 10 cm from the sample spot point, development is stopped. Dry the thin plate and spray 2,6-dichloroquinone · chloroimide solution and then heated at 120℃ for 10 min. In both developing solvents, for test solution, observe and compare the brown spot obtained from

2-mercaptoimidasoline standard solution.

- Discrimination method of utensils, containers and packages of rubber containing chlorine

  Copper net(net space 0.25 mm, net thickness 0.174 mm) is cut by width 1.5 cm x length 5 cm. Separately prepared one end of copper wire bound to the copper net. It is heated until green or blue of flame color is not shown in white color flame of Bunsen burner and then cooled. It is perfectly coated by thin film of copper oxide by repeating this procedure. After copper net is cooled, attach 1 mg of sample to the copper net and the sample is ignited and burned. After the treatment is repeated three times, put the copper net in the colorless flame and the color of flame is observed. In case of containing chlorine, it shows green flame. However, test can be performed by not winding copper net to end of copper wire but heating the end of wire thoroughly, putting on sample and attaching sample.

2-50 Zinc Test Method

a. Analysis principle

Measure zinc migrated from rubber, metal using water or 4% acetic acid as food simulant by Atomic Absorption Spectrophotometer or Inductively Coupled Plasma Atomic Emission Spectrometer.

b. Apparatus

Atomic Absorption Spectrophotometer or Inductively Coupled Plasma Atomic Emission Spectrometer

c. Standard solution

1) Standard stock solution

Accurately weigh 1.0 g of zinc and dissolve in 6M hydrochloric acid. Evaporate in a water bath and dissolve the residue in 1M hydrochloric acid to make 1,000 mL. Again, take 1 mL of this solution into a 50 mL volumetric flask and dilute to 50 mL with water. This solution is used as standard stock solution(20 μg/mL).

2) Standard solution

a) Standard solution for rubber except rubber nipple or metal

Take 1 mL of standard stock solution into a 20 mL volumetric flask and dilute to 20 mL with 4% acetic acid. This solution is used as Standard solution for testing rubber except rubber nipple or metal(1 μg/mL).

b) Standard solution for rubber nipple

Take 1 mL of standard stock solution into a 20 mL volumetric flask and dilute to 20 mL with water. Add 5 drops of acetic acid. This solution is used as standard solution for rubber nipple(1 μg/mL).

d. Preparation of test solution

1) For rubber except rubber nipple or metal

Prepare the solution using 4% acetic acid as food simulant under 2-6 Preparation of migration test solution for each material. Dilute 1 mL of this solution to 15 mL with 4% acetic acid.

2) For rubber nipple

Prepare the test solution under 2-6 Preparation of migration test solution for each material using water as food simulant. Add 5 drops of acetic acid to 20ml of this solution.

e. Test procedure

For test solution and standard solution proceed as directed under 2-11 Atomic absorption spectrophotometry(wavelength : 213.9 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry(wavelength: 206.2 nm). Calculate the concentration of zinc in test solution. However, for rubber except rubber nipple, adjust with dilution factor 15.

2-51 N-Nitrosamines and N-Nitrosatable substances Test Method

a. Analysis principle

Measure N-nitrosamines and N-nitrosatable substances migrated from rubber by liquid chromatograph/mass spectrometer/mass spectrometer.

b. Apparatus

Liquid chromatograph/mass spectrometer/mass spectrometer

c. Reagent and Solution

1) Artificial saliva

Dissolve 4.2 g of sodium hydrogen carbonate, 0.5 g of sodium chloride, 0.2 g of potassium carbonate and 30 mg of sodium nitrite in 900 mL of water. Adjust to pH 9.0 by adding 0.1N hydrochloric acid or 0.1N sodium hydroxide and add water to make 1 L.

d. Standard solution

1) Standard stock solution

Accurately weigh 20 mg of N-nitrosodimethylamine, N-nitrosodiethylamine, N-nitrosodi-n-propylamine, N-nitrosodi-n-buthylamine, N-nitrosopiperidine, N-nitrosopyrrolidine, N-nitrosomorpholine and dissolve in methanol respectively to make 100 mL.

2) Mixed standard solution

Take 5 mL each of standard stock solution into 100 mL volumetric flask and diluted to 100 mL with acetonitrile. Take 5 mL of this solution into a 100 mL volumetric flask and diluted to 100 mL with acetonitrile. And then mix with internal standard in the portion of 1:1(v/v). This solution is used as mixed standard solution(0.25 ug/mL for each nitrosamine). Shade the light to preserve at not more than 5℃.

3) Internal standard

Accurately weigh 20 mg of N-nitrosodi-n-propylamine-d_{14} or N-nitrosodiisopropylamine and dissolve in methanol to make 100 mL. Take 1

mL into 200 mL volumetric flask and diluted to 200 mL with acetonitrile. This solution is internal standard solution(1 ug/mL). Shade the light to preserve at not more than 5℃.

e. Test procedure

1) Operation condition of liquid chromatograph/mass spectrometer/mass spectrometer

- Column : C_{18}(3 mm I.D. × 250 mm, 3 μm) or its equivalent is used.
- Column temperature : 40℃
- Detector : mass spectrometer
  · Ionization : ESI(positive)
  · Capillary temperature : 330℃
  · Collision gas : Ar
  · Collision voltage : 10 ~ 40 V
  · Product ion

	Precursor ion (m/z)	Fragment ion (m/z)
N-nitrosodimethylamine	75.0	43.2
N-nitrosodiethylamine	103.0	43.3, 75.3
N-nitrosodi-n-propylamine	131.0	43.2, 89.2
N-nitrosodi-n-buthylamine,	159.1	57.2, 103.2
N-nitrosopiperidine	115.0	41.2, 69.0
N-nitrosopyrrolidine	101.1	55.2
N-nitrosomorpholine	117.1	86.2
N-nitrosodi-n-propylamine-d_{14}	145.0	50.8, 97.0
N-nitrosodiisopropylamine	131.0	43.2, 89.2

Adjust the temperature properly if necessary.

- Mobile phase : A : 0.1% formic acid, B : acetonitrile
- Gradient : Perform linear concentration gradient from A : B(80 : 20) to A : B(0 : 100) for 10 min, and hold for 5 min with A : B(0 : 100). Properly control if necessary.
- Flow rate : 0.2 mL/min

2) N-Nitrosamines

a) Preparation of test solution

Transfer the sample into beaker containing boiling water and boil for 10 min using minimum quantity of water necessary to cover the sample. Cool the sample and cut in two and dry. Accurately weigh 10 g ± 0.2 g of sample and transfer to flask. Add 40 mL of artificial saliva heated at 40℃ to cover the rubber nipple, and allow to stand at 40℃ for 24 hour. Transfer this solution into 50 mL of volumetic flask, wash the sample with 5 mL of artificial saliva. Add the washing solution and water to make 50 mL. This is solution A. Transfer 40 mL of solution A to separatory funnel. After adding 0.5 mL of internal standard and 1 mL of 0.1N sodium hydroxide, add 20 mL of dichloromethane and vigorously shake for 5 min and then allow to stand. Transfer dichloromethane phase into a kudernadanish flask. Add 20 mL of dichloromethane to the remaining solution and perform in the same manner as above. Add dichloromethane phase into the kudernadanish flask above, and add 1 mL of acetonitrile and mix and concentrate to 1 mL by nitrogen gas. This solution is used as test solution.

b) Qualitative test

Run the liquid chromatography/mass spectrometry under 1) Operation condition of liquid chromatograph/mass spectrometer/mass spectrometer by using each 5 uL of test solution and mixed standard solution. Retention time of peak in chromatogram for test solution and retention time of each N-nitrosamins peak in chromatogram for mixed standard solution are compared if they're identical.

c) Quantitative Test

When the retention time peak in chromatogram for test solution coincides with the retention time of each N-nitrosamines peak in chromatogram for mixed standard solution in b) Qualitative Test, the following test is performed.

Based on test result in b) Qualitative Test, measure each the ratio of peak area of N-nitrosamine against peak area of internal standard in chromatogram for test solution and mixed standard solution. Calculate the content of each N-nitrosamine per sample weigh(M_A) under following equation. Sum obtained result, and then calculate the final content of N-nitrosamines.

$$\text{N-nitrosamine } (M_A,\ mg/kg) = \frac{5}{4} \times c \times \frac{Rt}{Rs} \times \frac{1}{\text{weigh of samople(g)}}$$

c : Concentrate of each N-nitrosamine in mixed standard solution(μg/mL)

Rt : Ratio of peak area of each N-nitrosamine against peak area of internal standard in chromatogram for test solution

Rs : Ratio of peak area of each N-nitrosamine against peak area of internal standard in chromatogram for mixed standard solution

3) N-nitrosatable substances

a) Preparation of test solution

Add 1 mL of 0.1N hydrochloric acid to 10 mL 2) a) of solution A. Seal tightly, and allow to stand for 30 min in the dark. Transfer this solution to separatory funnel. After add 0.5 mL of internal standard and 2 mL of 0.1N sodium hydroxide, add 20 mL of dichloromethane and vigorously shake for 5 min and then allow to stand. Transfer dichloromethane phase into a kudernadanish flask. Add 20 mL of dichloromethane to the filtrate and perform in the same manner as above. Add dichloromethane phase into the kudernadanish flask above, and add 1 mL of acetonitrile and mix and concentrate to 1 mL by nitrogen gas. This solution is used as test solution.

b) Qualitative Test

Liquid chromatography/mass spectrometry is performed according to (1) Operation condition of liquid chromatograph/mass spectrometer/mass spectrometer by using each 5 uL of test solution and mixed standard solution. Retention time of peak in chromatogram for test solution and retention time of each N-nitrosamine peak in chromatogram for mixed standard solution are compared if they're identical.

c) Quantitative Test

When the retention time of peak in chromatogram for test solution coincides with the retention time each N-nitrosamine peak in chromatogram for mixed standard solution in b) Qualitative Test, the following test is performed.

Based on test result in b) Qualitative Test, measure each the ratio of peak area of N-nitrosamine against peak area of internal standard in chromatogram for test solution and mixed standard solution. Calculate the content of each N-nitrosamines per sample weigh(M_B) under following equation. Calculate M_B-MA on detected each N-nitrosamines. Sum obtained result, and calculate the final content of N-nitrosatable substances.

$$\text{N-nitrosamines } (M_B, \text{ mg/kg}) = 5 \times c \times \frac{Rt}{Rs} \times \frac{1}{\text{weigh of sampl(g)}}$$

c : Concentrate of each N-nitrosamine in mixed standard solution(μg/mL)

Rt : Ratio of peak area of each N-nitrosamine against peak area of internal standard in chromatogram for test solution

Rs : Ratio of peak area of each N-nitrosamine against peak area of internal standard in chromatogram for mixed standard solution

2-52 PCBs Test Method

a. Analysis principle

Extract PCBs residues in paper or processed paper with 1M sodium hydroxide · ethanol solution(or 1M potassium hydroxide · ethanol solution) and refine. Measure by gas chromatograph peak pattern method.

b. Apparatus

Gas chromatograph

c. Reagent and Solution

1) 1M sodium hydroxide · ethanol solution

Accurately weigh 40 g of sodium hydroxide and dissolve in 100 mL of water. Dilute to 1,000 mL with ethanol.

2) 1M potassium hydroxide · ethanol solution

Accurately weigh 56 g of potassium hydroxide and dissolve in 100 mL of water. Dilute to 1,000 mL with ethanol.

3) Florisil

Use 60 ~ 120 mesh of florisil which is activated for a night at 130℃.

d. Standard solution

1) Mixed standard solution

Dissolve each PCB standards product below in n-hexane to make 1 μg/mL. Properly mix this solution if necessary.

- Reagent(standards) : Aroclor 1221(AC 1221, monochlorinated biphenyls)
  Aroclor 1232(AC 1232 dichlorinated biphenyls)
  Aroclor 1242(AC 1242, trichlorinated biphenyls)
  Aroclor 1248(AC 1248, tetrachlorinated biphenyls)
  Aroclor 1254(AC 1254, pentachlorinated biphenyls)
  Aroclor 1260(AC 1260, hexachlorinated biphenyls)

e. Preparation of test solution

Sample is finely cut and accurately weigh 2 g of sample and tranfer into 200 mL flask. Add 50 mL of 1M sodium hydroxide · ethanol(or 1M potassium

hydroxide · ethanol solution) to the solution. And the solution is refluxed for 30~60 min in a water bath attached condenser. After cooling the solution, filter with glass filter and the residues in flask and glass filter are sequentially washed by 20 mL of n-hexane, 20 mL of ethanol, and 20 mL of water. Add the washing solution to filtrate and transfer into 300 mL separatory funnel. Add 50 mL of n-hexane and shake for 1 min and allow to stand. n-Hexene phase is transferred to another separatory funnel and then 50 mL of n-hexane is added to lower phase and extracted two times. After all n-hexane phase are summed, wash two times by use 50 mL of water. Pass the solution through column filled with anhydrous sodium sulfate(5 cm height and inner diameter about 1 cm of column for column chromatography) and dehydrated and then concentrated to be 5 mL. In case that sample contains wax, Transfer obtained concentrate into 125 mL separatory funnel, wash the container by n-hexane and add the washing solution to make 15 mL. Extract this solution with each 30 mL of n-hexane saturated-acetonitrile 2 times. After all acetonitrile phase are summed, transfer into 1,000 mL funnel, in which 700 mL of 20% sodium chloride solution and 100 mL of n-hexane are summed. After shaking, allow to stand until 2 phases are separated. Transfer aqueous phase to another separatory funnel, add 100 mL of n-hexane, and extract in the same manner. All n-hexane extracts are summed and washed three times by respectively 10 mL of 20% sodium chloride solution. n-Hexane solution is filtered in sodium sulfate anhydrous column(length 6 cm) and dehydrated. It is concentrated to be 5 mL under reduced pressure. Concentrated n-hexane solution is injected to column(inner diameter 2 cm, length about 30 cm), in which 10 g of activated florisil is filled by using n-hexane, elute by 200 mL of n-hexane and eluted solution is concentrated to be 5 mL. The solution is used as test solution.

f. Test procedure

1) Operation Condition of Gas chromatograph

- Column : DB-5 capillary column(0.25 mm I.D. × 30 m, 0.25 μm) or its equivalent is used.

- Column temperature : Hold at 120℃ for 2 min and temperature is raised to 200℃ at a rate of 5℃/min. Hold at 200℃ for 7 min. Again, temperature is raised to 240℃ at a rate of 2℃ /min. Hold at 240℃ for 20 min. Adjust the temperature properly if necessary.
- Injection temperature : 200℃
- Injection mode : Split(10:1)
- Detector : Electron capture detector
- Detector temperature : 270℃
- Carrier gas : Nitrogen or Helium (Flow rate : 1 mL/min)

2) Qualitative test

Run the gas chromatography with each 1 μL of test solution and mixed standard solution under 1) Operation condition of gas chromatograph. Peak pattern in chromatogram for test solution and peak pattern in chromatogram for mixed standard solution are compared if they're identical. If necessary, prepare mixed standard solution which indicates the most similar peak pattern to the peak pattern of test solution(determine the kinds of PCBs in sample as the kinds and the component ratio of PCBs standard product, which composes of standard solution indicating the most similar peak pattern in gas chromatogram obtained from test solution).

3) Quantitative test

When the peak pattern in chromatogram for test solution coincides with the peak pattern in chromatogram for mixed standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure the peak area of PCBs in chromatogram for test solution and standard solution. Calculate the content of PCBs in test solution and content of PCBs in sample under following equation.

$$\text{Content of PCBs(mg/kg)} = c \times \frac{At}{As} \times \frac{5(mL)}{\text{Weight of sample(g)}}$$

c : Concentration of PCBs in mixed standard solution (μg/mL)

At : Sum of PCBs peak area in chromatogram for test solution

As : Sum of PCBs peak area in chromatogram for mixed standard solution

2-53 Fluorescent whitening agent Test Method

a. Analysis principle

Ultraviolet rays is exposed to polylactide, butylenesuccinate-adipate copolymer, paper or processed paper, starch and distinguished the presence of fluorescence. If the result is positive, qualify with a process of dyeing.

b. Apparatus

Ultraviolet lamp

c. Preliminary Test

Ultraviolet rays of dominant wavelength around 365 nm is exposed to food contact surface at dark place and distinguished the presence of fluorescent violet ~ white blue. If it shows the presence of fluorescence, perform following test.

d. Test

1) Preparation of test solution

a) Sample which cannot be filled liquid

Sample is cut by 5 cm x 5 cm = 25 cm^2. When the sample is less than 25cm^2, make it same area in total. Transfer 100 mL of water into a 200 mL beaker and a few drops of 0.1% ammonia water and then stir well. Determine if it is slightly alkaline(pH 7.5~9). To this solution, add sample, shake slowly and leach for 10 min. Filter it through glass wool. This solution is used as test solution.

b) Sample which can be filled liquid

Fill sample with slightly alkaline solution(pH 7.5~9) and sometimes stir at room temperature. Allow to stand for 10 min and filter it through glass wool. This solution is used as test solution.

2) Test procedure

To test solution, add 1~2 drops of diluted hydrochloric acid to be weak acidic(pH 3~3.5). Non-fluorescent gauze(2 x 4 cm) is put into the solution and heated in water bath for about 30 min. Take out gauze and wash by water and pressed out. Ultraviolet rays is exposed 30cm far from gauze in a dark room.

2-54 Nickel Test method

a. Analysis principle

Measure lead, cadmium, and nickel migrated from metal by Atomic Absorption Spectrophotometer or Inductively Coupled Plasma Atomic Emission Spectrometer.

b. Apparatus

Atomic Absorption Spectrophotometer or Inductively Coupled Plasma Atomic Emission Spectrometer

c. Reagent and solution

1) 0.5% citric acid solution

!Dissolve 5 g of citric acid monohydrate into water to make 1,000 mL. Adjust the pH to 3.5 with sodium hydroxide solution.

2) Sodium hydroxide solution

Dissolve 4.3 g of sodium hydroxide into water to make 100 mL.

d. Standard solution

1) Lead standard solution

Accurately weigh 159.8 mg of lead nitrate and dissolve in 10 mL of 10% nitric acid and add water to make 100 mL. Take 1 mL of this solution into a 200 mL volumetric flask and dilute 200 mL with food simulant. Again, take 8 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with food simulant. This solution is used as lead standard solution(0.4 μg/mL). When the food simulant is water, add 5 drops of nitric acid to lead standard solution.

2) Cadmium standard solution

Accurately weigh 100 mg of cadmium and dissolve in 50 mL of 10% nitric acid. Evaporate it in a water bath and dissolve the residue in 0.1M nitric acid to make 1,000 mL. Take 1 mL of this solution into a 200 mL volumetric flask and dilute to 200 mL with food simulant specified in each test method. Again, take 2 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with food simulant specified in each test method. This solution is used as cadmium standard solution(0.1 μg/mL). When the food simulant is water, add 5

drops of nitric acid to cadmium standard solution.

3) Nickel standard solution

Accurately weigh 673.0 mg of nickel-ammonium sulfate(hexahydrate) and dissolve in 10 mL of water and nitric acid. Dilute to 1,000ml with water. Take 10 mL of this solution into a 100 mL volumetric flask and dilute 100 mL with food simulant specified in each test method. Again, take 1 mL of this solution into a 100 mL volumetric flask and dilute to 100 mL with food simulant specified in each test method. This solution is used as nickel standard solution (0.1 μg/mL). When the food simulant is water, add 5 drops of nitric acid to nickel standard solution.

e. Preparation of test solution

Utensils, containers and packages for food, which is listed in the column 1 of the following table, is tested using each solvent of column 2 as food simulant under 2-6 Preparation of migration test solution for each material. When the food simulant is water, add 5 drops of nitric acid to 100 mL of test solution.

Column 1	Column2
Food of not more than pH 5	Water
Food of more than pH 5	0.5% Citric acid

f. Test procedure

For test solution and standard solution proceed as directed under 2-11 Atomic absorption spectrophotometry(wavelength : 232.0 nm) or 2-12 Inductively coupled plasma atomic emission spectrometry(wavelength: 231.6 nm). Calculate the concentration of nickel in test solution.

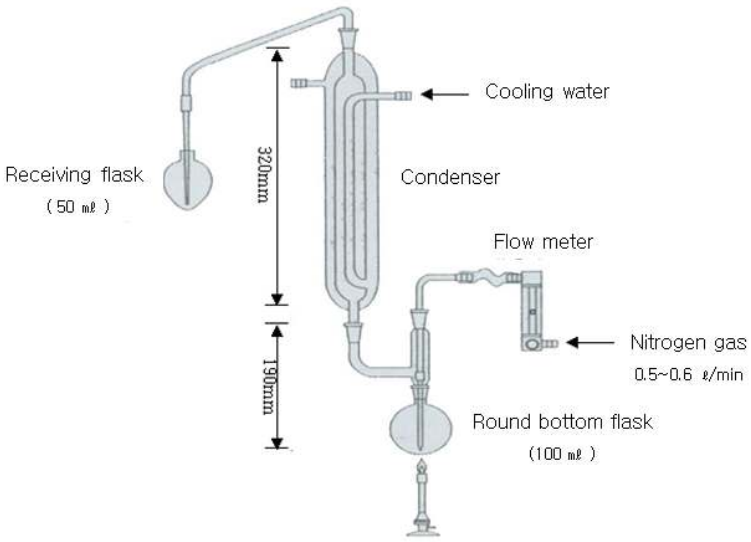
2-55 Sulfur dioxide Test Method

a. Analysis principle

Measure sulfur dioxide or sulphites as sulfur dioxide using water as food simulant by Distillation-alkali titration method.

b. Apparatus

Distillation apparatus



c. Reagent and Solution

1) Water

Distilled water for above 5 min by aerating nitrogen gas or helium or take deionized water when using.

2) 0.3% hydrogen peroxide

Dissolve 1 mL of 30% hydrogen peroxide in 100 mL of water. Prepare when using.

3) Methyl red·methylene blue solution

Dissolve 0.2 g of methyl red and 0.1 g of methylene blue in ethanol to make 100 mL. This solution is used as methyl red·methylene blue solution.

d. Preparation of test solution

Prepare the test solution using water as food simulant under 2-6 Preparation of

migration test solution for each material. However, migrate wooden chopsticks using test tube or nessler tube.

e. Test procedure

Transfer 10 mL of 0.3% hydrogen peroxide into a 50 mL of receiving flask and add 3 drops of methyl red·methylene solution (the color of the solution is violet). Add 1～2 drops of 0.001M sodium hydroxide solution so that the color of the solution turns olive green. Attach the receiving flask to the distillation apparatus. Accurately take 25 mL of test solution to the round bottom flask. Add 2 mL of ethanol, 2 drops of silicon oil and 10 mL of 25% phosphoric acid and attach quickly to the distillation apparatus. While aerating nitrogen gas through a flowmeter at the rate of 0.5~0.6L/min, heat the round bottom flask for 15 min. Detach the receiving flask and titrate with 0.01M sodium hydroxide solution until it turns olive green firstly appeared. Perform blank test using water instead of test solution(However, Not detected for less than 2 ppm).

$$\text{Sulfur dioxide}(\mu g/mL) \quad \frac{(a - b) \times f \times 320}{c}$$

a : Consumption of 0.01N sodium hydroxide solution in test solution(mL)

b : Consumption of 0.01N sodium hydroxide solution in blank test solution(mL)

c : Amount of test solution used for distillation(mL)

f : Activity of 0.01M sodium hydroxide solution

2-56 Ortho-phenylphenol, Thiabendazole, Biphenyl, and Imazalil Test Method

a. Analysis principle

Measure ortho-phenylphenol, thiabendazole, biphenyl, and imazalil migrated from woods using 20% ethanol as food simulant by liquid chromatograph.

b. Apparatus

Liquid chromatograph

c. Standard solution

1) Standard stock solution

Accurately weigh each 20mg of ortho-phenylphenol, thiabendazole, biphenyl, and imazalil and dissolve in ethanol to make 20 mL.

2) Mixed standard solution

Take 7.3 mL of ortho-phenylphenol standard stock solution, 1.8 mL of thiabendazole standard stock solution, 0.9 mL of biphenyl standard stock solution, and 0.6 mL of imazalil standard stock solution into 100 mL volumetric flask. Dilute to 100 mL with acetone. This solution is used as mixed standard solution(73 μg/mL of ortho-phenylphenol, 18 μg/mL of thiabendazole, 9 μg/mL of biphenyl, and 6 μg/mL of imazalil).

d. Preparation of test solution

Prepare the test solution using 20% ethanol as food simulant under 2-6 Preparation of migration test solution for each material. To 20 mL of this solution, add 20 mL of water and mix well. Use test tube or nessler tube for migration. Slowly inject the test solution into C_{18} cartridge, which is previously activated by passing 5 mL of methanol and 5 mL of water. Take 2 mL of the solution which is slowly extracted with methanol. This solution is used as test solution.

e. Test procedure

1) Operation condition of liquid chromatograph

- Column : C_{18}(4.6 mm I.D. × 250 mm, 5 μm) or its equivalent is used.
- Column Temperature : 40℃

- Detector : UV absorbance detector(wavelength : 230nm)
- Mobile phase : A : Add 2.5 mL of phosphoric acid to 1 L of water
  B : Add 2.5 mL of phosphoric acid to 1 L of 95% acetonitrile
- Flow rate : 1 mL/min
- Gradient : Perform linear gradient from A:B(100:0) to A:B(0:100) for 20 min. Properly control if necessary.

2) Qualitative test

Run the liquid chromatography with each 20 μL of test solution and mixed standard solution under 1) Operation condition of liquid chromatograph. Retention time of peak in chromatogram for test solution and retention time of ortho-phenyl phenol, thiabendazole, biphenyl, and imazalil peak n chromatogram for mixed standard solution are compared if they're identical.

3) Quantitative test

When the retention time of peak in chromatogram for test solution coincides with retention time of ortho-phenylphenol, thiabendazole, biphenyl, and imazalil peak in chromatogram for standard solution in 2) Qualitative test, the following test is performed.

Based on test result in 2) Qualitative test, measure peak area of o-phenylphenol, thiabendazole, biphenyl and imazalil in chromatogram for test solution and standard solution. Then calculate each content of each compound in test solution(accordingly adjust with concentration factor 10).

4) Identification test

When the retention time of peak in chroatogram for test solution coincides with retention time of ortho-phenylphenol, thiabendazole, biphenyl and imazalil peak in chromatogram for mixed standard solution, proceed as directly under gas chromatography/mass spectrometry under following <Operation condition>. Identify whether the detected peak is ortho-phenylphenol, thiabendazole, biphenyl or imazalil from obtained mass spectrum.

<Operation Condition>

- Column : DB-5 capillary column(0.25 mm I.D × 30 m, 0.25 μm) or its

equivalent is used.

- Column temperature : Hold at 40℃ for 1 min and temperature is raised to 300℃ at a rate of 10℃/min. Hold at 300℃ for 5 min. Adjust the temperature properly if necessary.
- Injection temperature : 220℃
- Injection mode : Split(10:1)
- Detector : mass spectrometer(scan mode)
- Ionization mode : EI mode
- Ionization energy : 70 eV
- Carrier gas : Helium(Flow rate : 1mL/min)

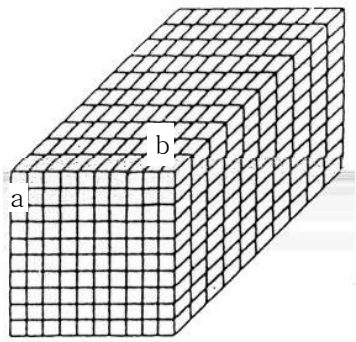
[Annex Table 1] Random sampling numbers

	1	2	3	4	5	6	7	8	9	10	
1	95767	26878	94971	75204	64775	52647	35672	97456	89815	16767	1
2	81934	99378	73901	46997	11173	80744	51957	50801	25785	40475	2
3	64959	10235	55441	87439	04966	96170	22582	13512	60227	81391	3
4	88488	81729	60733	84690	94315	10841	99296	47844	87998	01066	4
5	22306	61346	09026	66275	78204	11563	67457	69438	98753	23478	5
6	01436	13553	53487	19706	30740	83272	69602	99251	81599	79264	6
7	35962	90832	21604	29620	01151	81945	61894	91583	41133	83955	7
8	80319	01462	60338	69763	32812	84076	10555	88707	92309	34505	8
9	32141	01977	57874	25052	11567	35505	04797	21900	81703	11848	9
10	99197	78637	67636	35765	36871	22555	94013	15118	81620	76730	10
11	04030	88194	08580	86080	76119	62260	81248	51732	89078	21940	11
12	99835	54210	80413	82015	07459	93910	93694	41184	65698	31977	12
13	26400	63946	83204	36388	30497	24021	93962	25555	00357	93441	13
14	64363	11259	59066	92948	72291	31804	82029	15789	04683	34455	14
15	82878	87304	33618	19490	88779	38883	14481	77089	58544	22761	15
16	19485	82792	88195	31877	74902	13212	02514	47152	17292	62081	16
17	72762	47963	14380	96130	74633	21388	22096	24987	50056	26487	17
18	53747	17433	51614	97763	90426	47833	58245	14890	77303	35338	18
19	84760	29591	96142	02207	79847	89559	66803	15461	39639	23187	19
20	42365	42338	66105	03782	83727	14200	57263	85617	95577	88654	20
21	44336	05322	04540	75989	51385	52279	16661	91446	48811	25346	21
22	52930	61970	05808	22807	85460	22723	61318	23281	63426	69030	22
23	50436	54490	50650	59386	25572	23267	44039	08759	33702	34580	23
24	24980	54548	49428	91704	86913	92940	86923	22428	47877	20510	24
25	94186	95540	75071	10076	10263	27664	98534	81171	61507	74976	25

	1	2	3	4	5	6	7	8	9	10	
26	12359	46569	11050	90529	49289	94545	02258	20678	18898	50931	26
27	57132	49387	74792	50937	62339	43401	82825	03692	37836	68752	27
28	71239	49511	07466	15364	86050	31093	16730	73587	79875	43576	28
29	03935	83975	28893	66615	14015	22410	90938	35961	36373	84810	29
30	72801	73777	79101	26592	45424	00899	78462	74336	60478	13852	30
31	80252	11587	10862	11627	89857	07788	46817	55863	35713	44712	31
32	69388	92356	81282	33832	56676	35132	07119	27613	01304	70032	32
33	40225	60304	13526	47601	12751	19233	02057	28447	46676	29541	33
34	59925	62338	78784	63228	82603	31755	72733	80303	91066	52537	34
35	31172	89780	95384	51947	13841	74371	81016	39798	26690	00978	35
36	45587	42590	08443	20404	15100	74747	50642	34480	68080	23169	36
37	37819	60911	94740	05536	94042	17969	36747	92151	07501	67660	37
38	51402	74213	01105	15098	61470	23047	28521	58559	59305	08761	38
39	93873	99143	82254	53647	11010	68668	25888	36209	65412	00066	39
40	97645	51836	11835	99276	86466	67647	60834	30704	06711	11128	40
41	13123	15323	24885	74046	49145	99982	48231	26711	87670	22062	41
42	72110	70217	23475	48152	83457	01585	90915	69308	89090	77158	42
43	13751	40714	99330	05232	97056	40788	70005	36274	78649	50897	43
44	83809	98109	48292	06947	15924	08787	03919	72577	12906	80694	44
45	46264	57933	37607	58926	60772	62523	66260	95270	46469	45110	45
46	97426	78630	06115	44047	42350	88455	61426	08812	04255	87718	46
47	74715	09140	90315	03927	82118	00800	14314	06984	88951	10983	47
48	92481	55828	20144	77853	18100	13196	12279	20903	01252	00731	48
49	40176	70724	99476	34567	10214	97743	15770	78474	75691	59930	49
50	90176	19360	74015	89080	36337	40242	39621	71369	72350	73430	50

※ Use of random sampling numbers

When 1,000 agricultural box has horizontal, vertical, height of each box 10 as shown in the figure below, explanation how to select a box to extract the sample by using a table of random sampling numbers is as follows.



① Select a box randomly.

Ex: If you select the second box from the top of the far left side randomly, it is "a" in the above figure.

② Select a point in the Table of Random Numbers.

Ex: You randomly select 75071 located in column 3, row 25 in the random number table, then again randomly select the fourth number 7.

③ Read three numbers upward, downward, leftward, or rightward.

Ex: If you select to read rightward, then 7, 1, 1 are being read from the random number table.

④ A box that will be used for sampling is selected by counting boxes in three sequential directions based in the three selected number.

Ex: If you choose to count boxes rightward, upward, and inward sequence, count 7 rightward from position a, then 1 upward, and finally 1 inward to select the box for sampling, thus box b in the diagram is selected.

[Annex Table 2] Sample extraction statement

<table>
<tr><td rowspan="2">① Inspection Request No.</td><td rowspan="2"></td><td>② Product No.</td><td></td></tr>
<tr><td>③ Manufacturing No. (or Lot No.) or Sell-by-date</td><td></td></tr>
<tr><td>④ Extraction Date and Time</td><td>(YY-DD-DD, OO:OO)</td><td>⑤ Extracted Quantity</td><td></td></tr>
<tr><td rowspan="2">⑥ Reasons for Extraction</td><td rowspan="2">○ Surveillance
○ Research
○ Etc.( )</td><td>⑦ Product Type</td><td></td></tr>
<tr><td>⑧ Inspection Items</td><td></td></tr>
<tr><td rowspan="4">⑨ Storage and Transport Conditions in Extraction</td><td rowspan="4">○ Room Temperature
○ Chilled Temperature
○ Frozen Temperature
○ Etc.( )</td><td rowspan="2">⑩ Person Subjected to Extraction</td><td>Affiliation :</td></tr>
<tr><td>Name Sign</td></tr>
<tr><td rowspan="2">⑪ Person Subjected to Receipt</td><td>Affiliation :</td></tr>
<tr><td>Affiliation Sign</td></tr>
<tr><td colspan="4">※ Oher worth noting</td></tr>
</table>

[Annex Table 3] Identification table

① Extraction Date	(YY-DD-DD)	② Extracted Quantity	( ) mL ( ) kg ( ) 개 ( ) 기타
③ Reasons for Extraction	○ Inspection of Imported Foods	④ Person Subjected to Extraction	Food Safety Management Division/ Import Management Division O O O

O O Regional Korea Food & Drug Administration