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Methods of Test for Food Utensils, Containers and Packages - Test of Polypropylene Plastic Products

1. Scope

These methods are applicable to the inspection of polypropylene plastic food utensils, containers and packages.

2. Material identification

The test is according to the material identification in the “Methods of Test for Food Utensils, Containers and Packages - Test of Plastic Products”.

3. Material test

3.1. Lead (Pb) test

3.1.1. Method

After ashing, lead is determined by atomic absorption spectrophotometry (AAS).

3.1.1.1. Equipment

3.1.1.1.1. Atomic absorption spectrophotometer: with a lead hollow cathode lamp at a wavelength of 283.3 nm.

3.1.1.1.2. Furnace: with an automatic temperature controller, capable of controlling temperature at ±1.5°C.

3.1.1.1.3. Hot plate.

3.1.1.2. Chemicals

Sulfuric acid, reagent grade;
Nitric acid, reagent grade;
Deionized water, resistivity ≥ 18 MΩ•cm (at 25°C);
Lead (1000 μg/mL), reference standard, atomic absorption analysis grade.

3.1.1.3. Apparatus

3.1.1.3.1. Crucible(note): 50 mL, porcelain or platinum with cover.

3.1.1.3.2. Volumetric flask(note): 10 mL, 50 mL and 100 mL, Pyrex.

3.1.1.3.3. Storage bottle: 50 mL, PP.

Note: Soak the apparatus in nitric acid：water (1:1, v/v) overnight after cleaning. Take the apparatus out, wash away the residual nitric acid with water, rinse with deionized water and dry.

3.1.1.4. 0.1 N Nitric acid

Add 7 mL of nitric acid into 600 mL of deionized water slowly, and then dilute with deionized water to 1000 mL.

3.1.1.5. Standard solution preparation

Accurately transfer 1 mL of lead reference standard to a 50-mL volumetric flask, make up to volume with 0.1 N nitric acid, and transfer to a storage bottle as the standard stock solution. When to use, transfer appropriate amount of the standard stock solution, and dilute with 0.1 N nitric acid to 0.5-10 μg/mL as the standard solutions.

3.1.1.6. Sample solution preparation

Finely cut the sample into pieces smaller than 5 mm. Transfer about 1 g of the sample accurately weighed into a crucible, and add 10 drops of sulfuric acid. Evaporate most of sulfuric acid on a hot plate slowly, and heat continuously until white smoke disappear. Transfer the crucible into the furnace, and ash at 450°C. When ashing is incomplete, rinse with little amount of sulfuric acid, and continuously ash after drying. Repeat above rinse procedures until ashing is complete. Dissolve the residue with 0.1 N nitric acid, and make up to 10 mL as the sample solution. Take a blank crucible, add 10 drops of sulfuric acid, and perform the same procedure as the blank solution.

3.1.1.7. Quantification

Inject the sample solution, blank solution and standard solutions into the atomic absorption spectrophotometer separately, and detect at 283.3 nm. Calculate the amount of lead in the sample by the following formula based on the absorbance of the sample solution and the blank solution:

$$\text{The amount of lead in the sample (ppm)} = \frac{(C - C_0) \times V}{M}$$

Where,

C: the concentration of lead in the sample solution calculated by the standard curve (μg/mL)

C_0: the concentration of lead in the blank solution calculated by the standard curve (μg/mL)

V: the final make-up volume of the sample (mL)

M: the weight of the sample (g)

3.2. Cadmium (Cd) test

3.2.1. Method

After ashing, cadmium is determined by atomic absorption spectrophotometry (AAS).

3.2.1.1. Equipment

3.2.1.1.1. Atomic absorption spectrophotometer: with a cadmium hollow cathode lamp at a wavelength of 228.8 nm.

3.2.1.1.2. Furnace: with an automatic temperature controller, capable of controlling temperature at ±1.5°C.

3.2.1.1.3. Hot plate.

3.2.1.2. Chemicals

Sulfuric acid, reagent grade;

Nitric acid, reagent grade;

Deionized water, resistivity ≥ 18 MΩ•cm (at 25°C);

Cadmium (1000 μg/mL), reference standard, atomic absorption analysis grade.

3.2.1.3. Apparatus

3.2.1.3.1. Crucible(note): 50 mL, porcelain or platinum with cover.

3.2.1.3.2. Volumetric flask(note): 10 mL, 50 mL and 100 mL, Pyrex.

3.2.1.3.3. Storage bottle: 50 mL, PP.

Note: Soak the apparatus in nitric acid: water (1:1, v/v) overnight after cleaning. Take the apparatus out, wash away the residual nitric acid with water, rinse with deionized water and dry.

3.2.1.4. 0.1 N Nitric acid

Add 7 mL of nitric acid into 600 mL of deionized water slowly, and then dilute with deionized water to 1000 mL.

3.2.1.5. Standard solution preparation

Accurately transfer 1 mL of cadmium reference standard to a 50-mL volumetric flask, make up to volume with 0.1 N

nitric acid, and transfer to a storage bottle as the standard stock solution. When to use, transfer appropriate amount of the standard stock solution, and dilute with 0.1 N nitric acid to 0.05-1 μg/mL as the standard solutions.

3.2.1.6. Sample solution preparation

Finely cut the sample into pieces smaller than 5 mm. Transfer about 1 g of the sample accurately weighed into a crucible, and add 10 drops of sulfuric acid. Evaporate most of sulfuric acid on a hot plate slowly, and heat continuously until white smoke disappear. Transfer the crucible into the furnace, and ash at 450°C. When ashing is incomplete, rinse with little amount of sulfuric acid, and continuously ash after drying. Repeat above rinse procedures until ashing is complete. Dissolve the residue with 0.1 N nitric acid, and make up to 10 mL as the sample solution. Take a blank crucible, add 10 drops of sulfuric acid, and perform the same procedure as the blank solution.

3.2.1.7. Quantification

Inject the sample solution, blank solution and standard solutions into the atomic absorption spectrophotometer separately, and detect at 228.8 nm. Calculate the amount of cadmium in the sample by the following formula based on the absorbance of the sample solution and the blank solution:

$$\text{The amount of cadmium in the sample (ppm)} = \frac{(C - C_0) \times V}{M}$$

Where,

C: the concentration of cadmium in the sample solution calculated by the standard curve (μg/mL)

C_0: the concentration of cadmium in the blank solution calculated by the standard curve (μg/mL)

V: the final make-up volume of the sample (mL)

M: the weight of the sample (g)

4. Migration test

4.1. Potassium permanganate ($KMnO_4$) consumption test

4.1.1. Method

After migration of the sample, the migration solution is analyzed by titration.

4.1.1.1. Equipment

4.1.1.1.1. Water bath: capable of controlling temperature at ±1°C.

4.1.1.1.2. Oven: with an automatic temperature controller, capable of controlling temperature at ±1°C.

4.1.1.2. Chemicals

Potassium permanganate, reagent grade;
Sodium oxalate, reagent grade;
Sulfuric acid, reagent grade.

4.1.1.3. Apparatus

4.1.1.3.1. Single-sided migration apparatus

Each part of apparatus is shown in **Figure 1**.

A: Migration slot, made of glass. Internal diameter is 9 cm (surface area is 63.62 cm^2), outer diameter is 11.5 cm, and bottle height is 7 cm.

B: Ring, with rubber gaskets, made of teflon or stainless steel. Inner diameter is 9 cm, outer diameter is 15 cm and height is 1.8 cm.

C: Disc, with rubber gaskets, made of teflon or stainless steel. Diameter is 15 cm and height is 1.8 cm.

D: Fixing bolts.

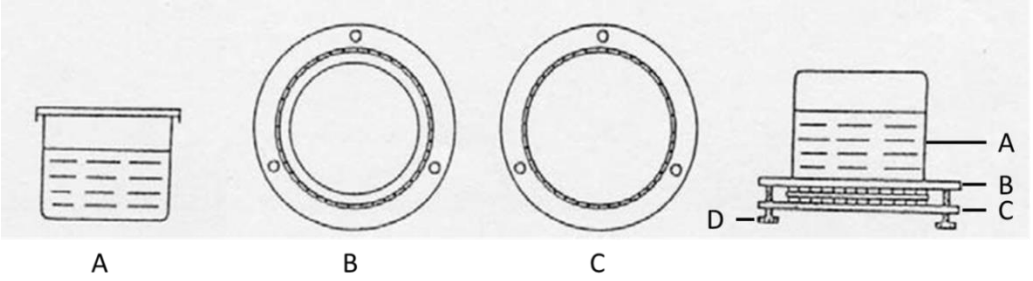

Figure 1. Single- sided migration apparatus

4.1.1.3.2. Erlenmeyer flask: 250 mL.

4.1.1.3.3. Burette: 25 mL, the minimum scale is 0.05 mL, brown.

4.1.1.3.4. Volumetric flask: 1000 mL, Pyrex.

4.1.1.4. Reagents

4.1.1.4.1. Sulfuric acid：water (1:2, v/v)

Mix sulfuric acid and water at the ratio of 1:2 (v/v).

4.1.1.4.2. 0.01 N Potassium permanganate

Transfer about 0.33 g of potassium permanganate into a 1000-mL volumetric flask, dissolve and make up to volume with water. When to use, calibrate the titer with 0.01 N sodium oxalate.

4.1.1.4.3. 0.01 N Sodium oxalate

Transfer 0.67 g of sodium oxalate into a 1000-mL volumetric flask, dissolve and make up to volume with water.

4.1.1.5. Sample solution preparation

4.1.1.5.1. Liquid container

Wash the sample with water and then air-dry. According to the migration condition listed in **Table 1**, add water preheated to the specified temperature whose volume is about 80% of the capacity of the sample, or add 2 mL of water preheated to the specified temperature per cm^2 surface area of the sample. Cover with aluminum foil, and place in an oven preadjusted to the specified temperature. After 30 min, take the migration solution out as the sample solution.

4.1.1.5.2. Monolayer films and sheets

For the sample made of the same material on the surface and interior, sum the areas of the surface and the inside of the sample as the area of the sample. Add 2 mL of water preheated to the specified temperature per cm^2 area of the sample. The following operation is the same as section 4.1.1.5.1. For the sample made of different materials on the surface and interior, the sample solution is prepared using a single-sided migration apparatus in the area which actually contacts with food. According to the migration condition listed in

Table 1, spread the sample on a migration slot which contains 127 mL of water preheated to the specified temperature. The surface which contacts with food is toward the bottom of the migration slot. Put the migration slot into the ring, add the disc on it, and clamp it with the fixing bolt. Turn the single-sided migration apparatus upside down so that the sample is in contact with water, and place in an oven preadjusted to the specified temperature. After 30 min, take the migration solution out as the sample solution.

Table 1. Migration conditions for potassium permanganate consumption test

Migration condition	Remark
60°C, 30 min	The condition used for those products which are heated to below 100°C during food processing or cooking
95°C, 30 min	The condition used for those products which are heated to higher than 100°C during food processing or cooking

4.1.1.6. Determination

Transfer 100 mL of water into an Erlenmeyer flask. Add 5 mL of sulfuric acid：water (1:2, v/v) and 10 mL of 0.01 N potassium permanganate, and heat to boil for 5 min. Remove the solution, and wash the Erlenmeyer flask with water. Accurately transfer 100 mL of the sample solution to the Erlenmeyer flask, add 5 mL of sulfuric acid：water (1:2, v/v), and add 10 mL of 0.01 N potassium permanganate using a brown burette. Heat to boil and last for 5 min, or heat in a boiling water bath for 15 min. After stop heating, immediately decolorize with 10 mL of 0.01 N sodium oxalate using another burette and titrate with 0.01 N potassium permanganate dropwise to the reddish color that doesn’t disappear. Take another 100 mL of water, and

perform the same procedure as the blank solution. Calculate the amount of potassium permanganate consumption in the migration solution by the following formula:

The amount of potassium permanganate consumption in the migration solution (ppm) = $\frac{(a - b) \times f \times 1000 \times 0.316 \times V}{100 \times 2 \times A}$

Where,

a: the titration amount of 0.01 N potassium permanganate for the sample solution (mL)

b: the titration amount of 0.01 N potassium permanganate for the blank solution (mL)

f: the titer of 0.01 N potassium permanganate

V: the volume of the migration solution (mL)

A: the area of the sample in contact with water (cm^2)

4.2. Heavy metal test

4.2.1. Method

After migration of the sample, the migration solution is analyzed by colorimetry.

4.2.1.1. Equipment

4.2.1.1.1. Oven: with an automatic temperature controller, capable of controlling temperature at ±1°C.

4.2.1.2. Chemicals

Glacial acetic acid, reagent grade;

Nitric acid, reagent grade;

Sodium sulfide, reagent grade;

Glycerol, reagent grade;

Deionized water, resistivity ≥ 18 MΩ•cm (at 25°C);

Lead (1000 μg/mL), reference standard, atomic absorption analysis grade.

4.2.1.3. Apparatus.

4.2.1.3.1. Single-sided migration apparatus: same as section 4.1.1.3.1.

4.2.1.3.2. Nessler tube: 50 mL, 20 mm i.d., with scale.

4.2.1.3.3. Volumetric flask: 10 mL, Pyrex.

4.2.1.4. Reagents

4.2.1.4.1. 0.1 N Nitric acid

Add 0.7 mL of nitric acid into 60 mL of deionized water slowly, and dilute with deionized water to 100 mL.

4.2.1.4.2. Sodium sulfide solution

Weigh 5 g of sodium sulfide, dissolve in 10 mL of deionized water, and mix with 30 mL of glycerol. Seal and store in a dark place. The use period of the solution is 3 months.

4.2.1.4.3. 4% Acetic acid

Take 40 mL of glacial acetic acid, and dilute with deionized water to 1000 mL.

4.2.1.5. Standard solution preparation

Accurately take appropriate amount of lead reference standard, and dilute with 0.1 N nitric acid to 10 μg/mL as the standard solution.

4.2.1.6. Sample solution preparation

4.2.1.6.1. Liquid container

Wash the sample with water and then air-dry. According to the migration condition listed in **Table 2**, add 4% acetic acid preheated to the specified temperature whose volume is about 80% of the capacity of the sample, or add 2 mL of 4% acetic acid preheated to the specified temperature per cm^2 surface area of the sample. Cover with a watch glass, and place in an oven preadjusted to the specified temperature. After 30 min, take the migration solution out, and adjust the volume by adding 2 mL of 4% acetic acid per cm^2 surface area of the sample as the sample solution.

4.2.1.6.2. Monolayer films and sheets

For the sample made of the same material on the surface and interior, sum the areas of the surface and the inside of the sample as the area of the sample. Add

2 mL of 4% acetic acid preheated to the specified temperature per cm^2 area of the sample. The following operation is the same as section 4.2.1.6.1. For the sample made of different materials on the surface and interior, the sample solution is prepared using a single-sided migration apparatus in the area which actually contacts with food. According to the migration condition listed in **Table 2**, spread the sample on a migration slot which contains 127 mL of 4% acetic acid preheated to the specified temperature. The surface which contacts with food is toward the bottom of the migration slot. Put the migration slot into the ring, add the disc on it, and clamp it with the fixing bolt. Turn the single-sided migration apparatus upside down so that the sample is in contact with 4% acetic acid and place in an oven preadjusted to the specified temperature. After 30 min, take the migration solution out as the sample solution.

Table 2. Migration conditions for heavy metal test

Migration condition	Remark
60°C, 30 min	The condition used for those products which are heated to below 100°C during food processing or cooking
95°C, 30 min	The condition used for those products which are heated to higher than 100°C during food processing or cooking

4.2.1.7. Determination

Transfer the specified amount of the sample solution into a Nessler colorimetric tube, and dilute with deionized water to 50 mL. Accurately transfer 2 mL of the standard solution into another Nessler colorimetric tube, add 20 mL of 4% acetic acid, and dilute with deionized water to 50 mL. Add 2 drops of the sodium sulfide solution to each tube and mix thoroughly. After standing for 2 min, examine the tubes

from above against a white background to compare the colors of the two tubes. The color of the sample solution should not be darker than that of the standard solution.

4.3. Evaporation residue test

4.3.1. Method

After migration of the sample, the migration solution is analyzed by weighing.

4.3.1.1. Equipment

4.3.1.1.1. Water bath: capable of controlling temperature at ±1°C.

4.3.1.1.2. Oven: with an automatic temperature controller, capable of controlling temperature at ±1°C.

4.3.1.2. Chemicals

Ethanol (95%), reagent grade;
Glacial acetic acid, reagent grade;
n-Heptane, reagent grade.

4.3.1.3. Apparatus

4.3.1.3.1. Single-sided migration apparatus: same as section 4.1.1.3.1.

4.3.1.3.2. Evaporating dish: quartz or platinum.

4.3.1.4. Reagents

4.3.1.4.1. 4% Acetic acid

Dilute 40 mL of glacial acetic acid with water to 1000 mL.

4.3.1.4.2. 20% Ethanol

Dilute 210 mL of ethanol with water to 1000 mL.

4.3.1.5. Sample solution preparation

4.3.1.5.1. Liquid container

Wash the sample with water and then air-dry. According to the migration conditions listed in **Table 3**, add the food simulant preheated to the specified temperature whose volume is about 80% of the capacity of the sample, or add 2 mL of the food simulant preheated to the specified temperature per cm^2 surface area of the sample. Cover with aluminum foil or a watch glass when 4% acetic acid is used as the food simulant, and place in

an oven preadjusted to the specified temperature. After the specified time, take the migration solution out as the sample solution.

4.3.1.5.2. Monolayer films and sheets

For the sample made of the same material on the surface and interior, sum the areas of the surface and the inside of the sample as the area of the sample. According to the migration condition listed in **Table** 3, add 2 mL of the food simulant preheated to the specified temperature per cm^2 area of the sample. The following operation is the same as section 4.3.1.5.1. For the sample made of different materials on the surface and interior, the sample solution is prepared using a single-sided migration apparatus in the area which actually contacts with food. According to the migration condition listed in **Table 3**, spread the sample on a migration slot which contains 127 mL of food simulant preheated to the specified temperature. The surface which contacts with food is toward the bottom of the migration slot. Put the migration slot into the ring, add the disc on it, and clamp it with the fixing bolt. Turn the single-sided migration apparatus upside down so that the sample is in contact with the food simulant, and place in an oven preadjusted to the specified temperature. After the specified time, take the migration solution out as the sample solution.

Table 3. Migration conditions for evaporation residue test

Application	Food simulant	Migration condition
Utensils, containers and packages for foods with a pH > 5	Water	60°C, 30 min[a]
		95°C, 30 min[b]
Utensils, containers and	4% Acetic acid	60°C, 30 min[a]

packages for foods with a pH 5 or lower		95°C, 30 min[b]
Utensils, containers and packages for oils, fats and fatty foods	*n*-Heptane	25°C, 1 hr
Utensils, containers and packages for alcoholic products	20% Ethanol	60°C, 30 min

[a]The condition used for those products which are heated to below 100°C during food processing or cooking.

[b]The condition used for those products which are heated to higher than 100°C during food processing or cooking.

4.3.1.6. Determination

Accurately transfer 200-300 mL of the sample solution into an evaporating dish predried to constant weight at 105°C, and then evaporate to dryness in a boiling water bath. Place the evaporating dish in an oven, and dry for 2 hr at 105°C. Remove the evaporating dish from the oven, place in a desiccator to cool to room temperature, and weigh the evaporating dish. Take the same amount of the food simulant, and perform the same procedure as the blank solution. Calculate the amount of evaporation residue in the migration solution by the following formula:

The amount of evaporation residue in the migration solution (ppm) = $\frac{(a - b) \times 1000 \times V}{M \times 2 \times A}$

Where,

a: the weight of the sample solution after drying (mg)

b: the weight of the blank solution after drying (mg)

M: the volume of the sample solution (mL)

V: the volume of the migration solution (mL)

A: the area of the sample in contact with the food

simulant (cm^2)

Remark

1. Limits of quantification (LOQs) are 5 ppm for lead and 0.5 ppm for cadmium.
2. The amount of the analyte in the migration solution by the migration test is calculated based on adding 2 mL of the food simulant per cm^2 surface area of containers and packages.
3. When lead and cadmium are determined by other instruments, verification by the certified reference material (CRM) or the standard reference material (SRM), or validation of the method should be performed.

Reference

The Pharmaceutical Society of Japan. 2015. Method of Analysis in Health Science. Kanehara & Co., Ltd, Tokyo, Japan.