HS 1001 Overall Migration Test by Total Immersion Method

1. Purpose

The Overall Migration Test is to determine the quantity of substance that may migrate from the sample to the foodstuffs. However, it is not always possible to use real foodstuffs for testing, food simulants are therefore introduced. Aqueous food is simulated by distilled water, acetic acid and ethanol.

This method is designed for food contacting articles, which are in the form of bag/ container of volume larger than 10 L. For containers with a volume less than 10 L, the Article Filling Test (HS 1002) is more appropriate.

2. Terminology

2.1 Food simulant

Medium intended to simulate a foodstuff

2.2 Overall migration

Mass of material transferred to the food simulant as determined by the relevant test method

3. Apparatus

3.1 Cutting slab, clean smooth glass, metal or plastic slab of sufficient area to prepare test specimen, 250 mm × 250 mm is suitable.

3.2 Lint-free cloth or brush.

3.3 Tweezers, stainless steel, blunt nosed.

3.4 Cutting implement, scalpel, scissors, sharp knife or other suitable device.

3.5 Metal templates (100 mm ± 0.2 mm) × (100 mm ± 0.2 mm) (square).

3.6 Ruler, 25 mm ± 1 mm wide.

3.7 Ruler, graduated in mm, and with an accuracy of 0.1 mm.

3.8 Analytical balance capable of determining a change in mass of 0.1 mg.

3.9 Cruciform specimen supports, or other supports that are capable of holding and keeping the test pieces apart and ensuring complete contact with the simulant. The supports are constructed of stainless steel with cross arms attached by welding or silver soldering. The steel supports should be thoroughly cleaned before initial use. The use of a degreasing solvent and then dilute nitric acid has been found to be suitable.

Note: For aqueous acetic acid food simulant, use supports constructed out of glass as there is a tendency for acetic acid to corrode stainless steel supports, particularly if the joints are silver soldered. However, stainless steel supports may

be used for acetic acid if it can be demonstrated that they do not contribute to the residue. Check all supports to ensure that they do not contribute significantly to the residue.

3.10 Gauze, pieces of fine stainless steel gauze with a mesh size of 1mm have been found to be suitable, approximately 25 mm × 100 mm for insertion between the test pieces on the supports. Thoroughly clean the gauze before initial use, first with a degreasing solvent and then with dilute nitric acid.

3.11 Glass tubes, ground neck and stoppers, for retaining the food simulant and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the ground neck have been found to be satisfactory.

3.12 Thermostatically controlled oven or incubator capable of maintaining a temperature of 40 °C ± 1 °C and 70 °C ± 2 °C.

3.13 Steam bath or hot plate, distillation apparatus or other appropriate equipment such as rotary evaporator for evaporation of food simulant.

3.14 Glass beads, 2 mm to 3 mm in diameter or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long.

3.15 Dessicator containing self-indicating silica gel or anhydrous calcium chloride.

3.16 Dishes, platinum, platinum alloy with 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of aqueous food simulants and weighing the residues. Glass, glass ceramic or glazed ceramic dishes may be used provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified food simulants followed by conditioning in the dessicator used can remain constant within ± 0.5 mg.

3.17 Beakers, 2 L, 250 mL.

3.18 Measuring cylinder, 100 mL.

4. Reagents

4.1 Distilled water.

4.2 Acetic acid 3 % (w/v) in aqueous solution (simulant B).

4.3 Ethanol 15 % (v/v) in aqueous solution (simulant C).

Note: In the case of food/drink containers intended to come into contact with liquids or beverages of alcoholic strength exceeding 15 % (v/v). Test may be carried out with aqueous solutions of ethanol of a similar strength.

5. Food Simulants

The food simulants should have the following quality:

- Simulant A, distilled water or water of equivalent quality;
- Simulant B, acetic acid in aqueous solution, 3 % w/v aqueous acetic acid.

- Simulant C, ethanol 15 % (v/v) in aqueous solution.

  Note: Alcoholic simulants for liquids or beverages with alcoholic strength exceeding 15 % (v/v) - test may be carried out with aqueous solutions of ethanol of a similar strength.

Each of the above food simulants should give a non-volatile residue of less than 5 mg/L, when evaporated to dryness and dried to constant weight at 105 °C to 110 °C.

If an article is intended for use in contact with all types of food, it should be tested with simulants A, B and C.

6. Migration Test Conditions (time and temperature)

6.1 Contact conditions in actual use are known:

Select the corresponding test conditions (exposure time and temperature) from Table 1 which is applicable to the sample when it is in actual use. If the sample is intended for a food contact application under two or more conditions, the migration test shall be carried out at the most stringent test conditions (i.e. the longest test time and/or the highest test temperature).

Table 1: Migration Test Conditions.

Conditions of contact in actual use	**Test Condition**
Contact time t	**Test time**
0.5 hr ≥ t	0.5 hr
1 hr ≥ t > 0.5 hr	1hr
2 hr ≥ t > 1 hr	120 min
24 hr ≥ t > 2 hr	24 hr
t > 24 hr	240 hr
Contact temperature T	**Test temperature**
5 °C ≥ T	5 °C
20 °C ≥ T > 5 °C	20 °C
40 °C ≥ T > 20 °C	40 °C
70 °C ≥ T > 40 °C	70 °C
T > 70 °C	100 °C or reflux

6.2 Contact conditions in actual use are not known:

In case the contact conditions (temperature and time) are not known, only 10-day test at 40 °C and 2-hour test at 70 °C are to be carried out for food simulants (A, B and C).

7. Preparation of test specimens

7.1 General

The sample taken for testing should be in its ready-for-use state. The sample should be clean and free from surface contamination. Dust may be removed by wiping the sample with a lint-free cloth or brushing with a soft brush. Under no circumstances should the sample be washed by water or solvent.

For very large samples, testing by Article Filling Method (i.e. HS 1002) may not be practicable. It may then be necessary to cut them into smaller test specimens for testing. Testing can also be carried out on cut test specimens by total immersion.

As many articles have irregular shape or dimensions, e.g. in thickness, the test specimens selected must be representative of the whole of those parts of the article that are intended to come into contact with food. Also, the dimensions of the replicate test specimens should also be similar to allow valid comparison of results.

7.2 The single surface versus double surface testing by total immersion

The surface to volume ratio in the total immersion test is conventionally 1 dm^2 of food contact area to 100 mL of food simulant.

- The surface area of test specimens should be approximately 1 dm^2. Calculate the surface area of each test specimen to the nearest 0.01 dm^2.
- For articles with individual areas less than 1 dm^2, use a number of articles to provide each test specimen.
- Cut each test specimen into four test pieces 25 mm x 100 mm. Assemble one test specimen onto the support by piercing suitable holes in the test pieces and placing two test pieces on each side of the cross arms of the support (Section 3.9). Repeat this procedure for all remaining test specimens.
- For those samples of irregular shape or thickness, the test specimens should have a surface area of approximately 2 dm^2. Calculate the surface area of each test specimen to the nearest 0.05 dm^2 using the Schlegel Method (see Annex A), or other suitable method.
- Measure the dimensions of each test specimen to the nearest 1 mm using the ruler.

For the Total Immersion Test, both the inside and outside surfaces of the test specimen are in contact with food simulant. Although the total surface exposed to food simulant is 2 dm^2, only 1 dm^2 (i.e. the food contact surface) is taken into account in the calculation. No allowance is made for this in the calculation of migration per unit surface area. This test is more stringent than testing by filling.

Samples of test specimen with cut edges tend to give a higher result. In normal use, the containers should not have cut edges in contact with foodstuff. Therefore, in calculating the overall migration result, the area of cut edge should not be taken into account. The test specimens prepared by cutting sections from the food/drink containers for total immersion in the food simulants can be regarded as a more stringent test.

7.3 Number of test specimen for aqueous food simulants

Three test specimens are required for testing, either in the form of thin film, sheet, containers or similar articles. However, five test specimens with similar dimensions are required for articles with irregular shape.

These test specimens are utilised as follows:

- Three test specimens for the migration test;
- Two test specimens for the determination of surface area, in the case of samples with irregular shape.

8. Operating procedures

8.1 Exposure to food simulant

- Take three of the glass tubes, measure by measuring cylinder 100 mL ± 2 mL of food simulant into each tube and stopper the tube.
- If the evaporation method is to be used, measure into two additional tubes 120 mL ± 2 mL of food simulant to provide blanks.
- If the distillation method is to be used, measure into two additional tubes 100 mL ± 2 mL of food simulant to provide blanks.
- Place the five tubes that contained food simulant in the thermostatically controlled oven or incubator, set at the test temperature (40 °C or 70 °C or the selected temperature from Section 6), and leave until the test temperature has been attained.
- Take the tubes out of the thermostatically controlled oven or incubator. Place a test specimen into each of the three tubes containing 100 mL of simulant and stopper the tubes.
- Mark the tubes for identification.
- Ensure the test specimens are totally immersed in the simulant. If they are not, add either glass beads or rods to raise the level of the simulant until total immersion is achieved. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the simulant.
- Mark the liquid level on the outside of each tube with a suitable marker.
- Place all tubes in the thermostatically controlled oven or incubator, set at the test temperature and observe the temperature, leave the tubes there for a selected test period (see Section 6) after the air bath of the thermostatically controlled oven or

incubator has reached a temperature within 1 °C of the set temperature.

- Take the tubes from the oven or incubator and check the level of simulant in each, if this has dropped to more than 10 mm below the mark, or has been exposed to any part of the test pieces, repeat the test using fresh samples.
- If the level of simulant in a tube is less than 10 mm below the mark, remove the test specimen from the tube, and allow the simulant adhering to the test specimen and support to drain back into the tube. Recover at least 90 % of the original volume of simulant or simply repeat the test.
- Determine the amount of residues in the simulant, using the procedure described in Section 9.

9. Determination of migrating substances

The analytical determination of total quantity of substances released by the sample can be undertaken by evaporation method or distillation method as described below:

9.1 Preparation of dishes

- Take five dishes, mark for identification and place in an oven that has been maintained at 105 °C to 110 °C for a period of 30 min ± 5 min to dry.
- Remove the dishes from the oven, place in a dessicator and allow cooling to ambient temperature. Weigh and record the individual masses of each dish.
- Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0.5 mg, weigh their masses.
- Use evaporation method or distillation method to obtain the mass of the residues.

9.2 Evaporation method

- Take the tube containing the simulant and pour 40 to 50 mL from each into separate dishes. Using a steam bath, hot plate or other form of heating to evaporate the simulant to a lower volume (taking care to avoid loss, in particular, by sputtering or overheating of the residues).

  Note: The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.
- When most of simulant has evaporated, pour the remaining simulant from each the tubes into respective dishes and continue the evaporation. Wash out each of the tubes which had contained test specimens with two lots of 10 mL ± 1 mL of unused simulant and pour these washings into the respective dishes. Continue the evaporation.

  Note: A stream of nitrogen may facilitate evaporation.

- When the simulant has almost completely evaporated, place the dish in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to complete the evaporation and dry the residue.
- Remove the dishes from the oven, place in a desiccator and allow it cooling to ambient temperature. Weigh and record the individual masses of a dish and residue.
- Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0.5 mg.
- Determine the mass of the residue by subtracting the original mass of the dish from the stable mass of the dish and residue.

9.3 Distillation method

- Transfer the simulants to individual round bottom flasks (250mL are suitable). Rinse each tube twice, including the blank tubes, with 20 mL ± 2 mL of fresh simulant, add these rinses to the respective flasks.
- Place the flasks in an electric heating mantle and connect to a side arm distillation arrangement; or place them in a rotary evaporator instead. Distil off the simulants until approximately 30 mL to 50 mL remains in the flask.
- Transfer the remaining simulant to an evaporating dish. Rinse the flask with 10 mL ± 1 mL of fresh simulant and add the rinses to the appropriate dishes. Continue the evaporation of the simulant by using a steam bath, hot plate or other form of heating described in evaporation method.

10. Expression of Results

Compile tables (listed in Annex B) containing the measured and calculated data on the test material and the blanks.

10.1 Method of calculation for total immersion with aqueous food simulants

Express the overall migration as milligrams of residue per decimetre of the surface of the sample which is intended to come into contact with foodstuffs, calculated for each test specimen using the following formula:

$$M = \frac{(m_a - m_b)}{S} \times 1000 \qquad (1)$$

where:

M is the overall migration into the simulant, in milligrams per square decimetre of the surface area of sample intended to come into contact with foodstuffs;

m_a is the mass of the residue from the test specimen after evaporation of the simulant in which it had been immersed, in grams;

m_b is the mass of residue from the food simulant only (blank), in grams;

S is the surface area of the test specimen intended to come into contact with foodstuffs,

in square decimetres.

Note: Only one surface is counted.

Calculate the result for each test specimen to the nearest 0.1 mg/dm^2 and the mean of the individual test results, to the nearest 0.1 mg/dm^2.

11. Validity of Results

The test results are considered to be valid only if the difference between the test results in the same test satisfy the following conditions:

Table 2: Analytical Tolerance (acceptable intra-test result difference) for different simulants.

Item	Analytical Tolerance
All aqueous food simulants	≤ 6 mg/kg or 1mg/dm^2

If more than two results are not within the analytical tolerance, then the test should be repeated using fresh test specimens from the sample.

12. References

1. Commission Directive 82/711/EEC, The basic rules necessary for testing migration of the constituents of plastic materials and articles intended to come into contact with foodstuffs of 18 October 1982, The Commission of the European Communities.
2. Commission Directive 85/572/EEC, Simulants for Plastics Migration Tests of 19 December 1985, The Commission of the European Communities.
3. Commission Directive 89/109/EEC, Approximation of the laws of the Member States relating to materials and articles intended to come into contact with foodstuffs of 21 December 1988, The Commission of the European Communities.
4. Commission Directive 90/128/EEC, Plastics Materials and Articles Intended to come into Contact with Foodstuffs of 23 February 1990, The Commission of the European Communities.
5. Commission Directive 92/39/EEC, Amending Directive 90/128/EEC - Plastics Materials and Articles Intended to come into Contact with Foodstuffs – 14 May 1992, The Commission of the European Communities.
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8. Commission Directive 95/3/EC, Amending Directive 90/128/EEC - Plastics Materials and Articles Intended to come into Contact with Foodstuffs – 14 February 1995, The Commission of the European Communities.
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10. Commission Directive 1999/91/EC, Amending Directive 90/128/EEC - Plastics Materials and Articles Intended to come into Contact with Foodstuffs – 23 November 1999, The Commission of the European Communities.
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12. Commission Directive 92/15/EEC, Amending Council Directive 83/229/EEC concerning regenerated cellulose film – 11 March 1992, The Commission of the European Communities.
13. ENV 1186-1, Materials and articles in contact with foodstuffs – Plastics – Part 1: Guide to the selection of conditions and test methods for overall migration, CEN, European Committee for Standardization, Brussels.
14. ENV 1186-3, Materials and articles in contact with foodstuffs – Plastics – Part 3: Test methods for overall migration into aqueous simulants by total immersion, CEN, European Committee for Standardization, Brussels.
15. ISO 8442-2: 1997, Materials and articles in contact with foodstuffs – Cutlery and table hollowware – Part 2: Requirements for stainless steel and silver-plated cutlery, International Organization for Standardization.

Annex A (normative) – Schlegel method of determining surface area

A.1 Principle

Under controlled conditions, the test item is coated with an adhesive and dipped in a fluidised bed of water-repellent or perfectly dry glass beads of uniform size. The mass of beads adhering to the item is proportional to the surface area of the test item. The relationship between the mass of beads and the surface area of the item is determined by applying the test to a standard specimen of regular shape and known area.

A.2 Apparatus and materials

A.2.1 Fluidised bed, bed of glass beads (see A.2.5) with a supply of fluidising air and means for heating the income air if water repellent beads are not used. This may be in the form of an electric heating element, near to the bottom of the bed, regulated by a voltage control, which is sufficient to raise the temperature of the fluidised beads to between 50 °C and 80 °C.

Note: it is not advisable to control bead temperature by means of a thermostatic device because, when the power supply is disconnected from the heating element, the beads can pick up moisture from air supply.

A.2.2 Laboratory balance capable of weighing to an accuracy of ± 2 mg.

A.2.3 Hoist for withdrawing the test item from the adhesive at 20 mm/min.

A.2.4 Adhesive composed of:

Alkyl resin	1 part by mass
Toluene (sulphur free)	1 part by mass

A.2.5 Glass beads, graded from 200 mm to 250 mm, preferably of water repellent type.

Note: Commercially available glass beads nominally graded to these limits may contain an undesirable proportion outside the limits; normally they should be regraded.

A.2.6 Specimens of known area, (at least two) in stainless steel, as follows:

a) a cylinder, of approximately 16 mm diameter and 110 mm in length, to indicate the mass of glass beads per square centimetre picked up by hollow handles;

b) a rectangle, of approximately 100 mm × 30 mm × 1 mm, to indicate the mass of glass beads per square centimetre picked up by areas other than hollow handles.

A.3 Procedure

A.3.1 Ensure that the glass beads (see A.2.5), not of the water-repellent type, are thoroughly dry so that they do not adhere to each other. These beads should be prevented from reabsorbing moisture from air supply by preheating them in the fluidised bed at between 50 °C and 80 °C until no beads will adhere to a clean, dry item of cutlery that is dipped into them. Usually a drying time of 1 hour is adequate.

Note: It has been found that once moisture has been eliminated, the beads will stay dry while the heating elements remain switched on.

Maintain the temperature of the fluidised bed of glass beads at between 50 °C and 80 °C until the procedure described in A.3.8 has been reached. If water-repellent beads are used, the bed may be used at ambient temperature for the procedure described in A.3.7.

A.3.2 Attach a thin wire hanger to the test specimen with a loop for suspension during weighing etc.

A.3.3 Thoroughly clean the test specimen with methylated spirits.

A.3.4 Dip the test specimen in the adhesive (see A.2.4) and withdraw at a rate of 20 mm/min using the hoist (see A.2.3). Do not allow the test specimen surface to come into contact with anything until stage A.3.7 is reached.

A.3.5 Allow the adhesive to dry for 60 min ± 5 min.

A.3.6 Weigh the test specimen to the nearest 2 mg.

A.3.7 Immerse and continuously agitate the test specimen in the fluidised bed of glass beads for 10 s ± 1 s. During immersion the air flow should be vigorous enough to raise mounds of beads to a height of at least 40 mm above the bed of fluidised beads. Do not immerse more suspension wire than necessary.

A.3.8 Reweigh the test specimen to the nearest 2 mg.

A.3.9 Carry out a duplicate test on each test specimen; include at least two specimens of known area (see A.2.6) in each batch of specimens tested.

A.4 Expression of results

A.4.1 Method of calculation

Calculate the area, A, in square centimetres, of the test specimen using the formula

$$A = \frac{m}{Q_A}$$

where

m is the mean mass, in grams, of beads adhering to the test specimen;

Q_A is the mean surface mass density, in grams per square centimetre, of beads corresponding to the mass adhering to the relevant specimen of known area.

Annex B – Overall Migration – Test Report for aqueous food simulant

Test Material: _____

Condition of temperature of exposure to simulant: _____°C

Condition of time of exposure to simulant: _____hour(s)

Average of mass of residue in the blank (m_b): _____g

	Test specimen 1	Test specimen 2	Test specimen 3
Initial mass of evaporating dish/g			
Final mass of evaporating dish + residue/g			
Mass of residue/g (m_a)			
The surface area of test specimen intended to come into contact with foodstuffs/dm^2 (S)			
Overall migration, M			

Calculate the overall migration into the simulant: $M = \frac{(m_a - m_b)}{S} \times 1000$

where:

M is the overall migration into the simulant, in milligrams per square decimetre of the surface area of sample intended to come into contact with foodstuffs;

m_a is the mass of the residue from the test specimen after evaporation of the simulant in which it had been immersed, in grams;

m_b is the mass of residue from the food simulant only (blank), in grams;

S is the surface area of the test specimen intended to come into contact with foodstuffs, in square decimetres.

Calculate the result for each test specimen to the nearest 0.1 mg/dm^2 and the mean of the individual test results, to the nearest 0.1 mg/dm^2.