HS 1002 Overall Migration Test by Article Filling 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 test method is used when the test sample is in the form of a container.

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 – see Section 3 of HS 1001

4. Reagents – see Section 4 of HS 1001

5. Food simulants – see Section 5 of HS 1001

6. Migration test conditions – see Section 6 of HS 1001

7. Preparation of test specimens

7.1 General – see Section 7.1 of HS 1001

7.2 Single surface testing by filling

For articles in container form, it is often most convenient to test them by filling with food simulant.

7.3 Number of test specimen for aqueous food simulants

Determine and record the volume of food simulant required to fill an article. Also determine and record the surface area of the test specimen exposed to food simulant when it is filled to its nominal volume using the Schlegel Method (see Annex A of HS 1001) or other suitable method.

- For samples with a nominal volume of more than 200 mL, five articles are required to provide five test specimens. Three test specimens are utilised for migration test and two test specimens are utilised for determination of surface area.
- For samples with a nominal volume of less than 200 mL, the number of articles required to provide a test specimen is dependent on their volume. A test specimen shall be made up of sufficient articles to contain 200 mL of food simulant. Three test specimens are required for migration test and two specimens are required for determination of surface area. Record the number of articles used to provide the test specimen.
- For samples with a capacity of not less than 500 mL and not more than 10 L, it is not necessary to determine the surface area of these articles, the migration result must be expressed in mg/kg of food simulant and three test specimens are required for migration test.
- For samples with a capacity of more than 10 L, it is recommended to perform total immersion test as described in HS 1001.

8. Operating procedures

8.1 Exposure to food simulant

- Mark each of the articles making up each test specimen with an identification code.
- Place, in a beaker, a sufficient volume of the food simulant to fill the three test specimens and two blanks in the thermostatically controlled oven or incubator. Set the oven or incubator at the test temperature and leave the simulants inside the oven or incubator until the test temperature has been attained.
- Remove the beaker containing the food simulant from the thermostatically controlled oven or incubator. Measure the food simulant by measuring cylinder and fill the three test specimens with simulant to within 0.5 cm from the top. Mark the volume of food simulant that has been used.
- Cover the test specimens and the remaining simulant with an inert material to prevent evaporation, e.g. glass. This part of the operation should be carried out in the minimum time to prevent undue heat loss from simulant.
- Place the test specimens and food simulant in the thermostatically controlled oven or incubator set at the selected test temperature (40 °C or 70 °C or the other selected temperature from Section 6 of HS 1001), leave the test specimens and the remaining simulant in the oven or incubator for the required test period (Section 6) after the air bath of the thermostatically controlled oven or incubator has reached a temperature within 1 °C of the set temperature.

  Note: If the exposed surface of simulant is large, a check should be made to ensure that excessive loss of simulant by evaporation does not occur.

9. Determination of migrating substances

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 and record their masses.
- Use evaporation method or distillation method to obtain the mass of the residues.

9.2 Evaporation method

- Take the test specimens and transfer 200 mL of the simulant by using a pipette in separate 250 mL beakers, make sure the simulants is mixed.
- Measure aliquots of 200 mL of the food simulant into two more beakers to provide blanks, which are handled in the same way as simulants but have not been in contact with the test specimens. 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 into separate dishes. Wash out each of the beakers 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 dessicator and allow it to cool 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 200 ml of the simulants by using a pipette to individual round bottom flasks (250ml are suitable).

- Measure two more portions of unused food simulant into an individual round bottom flasks to provide blanks.
- Place the flasks in an electric heating mantle and connect to a side arm distillation arrangement; or place them in a rotary evaporator instead. Distill 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.

  Note: The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

10. Expression of results

10.1 Method of calculation for article filling by aqueous food simulants

10.1.1 For test article whose surface area in contact with foodstuffs can be estimated, the overall migration shall be expressed in milligrams of residue per square decimetre of the surface of the sample which is intended to come into contact with foodstuffs. The overall migration shall be calculated for each test specimen using the following formula:

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

- For the test articles with a volume of more than 200 mL but less than 500 mL; the calculation formula will be:

$$M = \frac{(m_a - m_b)}{S \times 200} \times 1000 \times v \qquad (1.1)$$

- For the test articles with a volume of less than 200 mL, the calculation formula will be:

$$M = \frac{(m_a - m_b)}{A \times N} \times 1000 \qquad (1.2)$$

where:

M is the overall migration into the simulant, in milligrams per square decimetre of food simulant;

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 test specimen intended to come into contact with food, in square decimetre;

v is the volume of the food simulant which had filled the article, in millilitres;

A is the surface area of one test article in square decimetres;

N is the number of articles exposed to the simulant.

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. See Section 11 for the directions to determine whether the results are valid. Compile tables (listed in Annex A) containing the measured and calculated data on the test material and the blanks.

10.1.2 For the test articles whose surface area in contact with foodstuffs cannot be estimated, or for test articles with volume of not less than 500 mL and not more than 10 L, the overall migration shall be expressed in milligrams lost per kilogram of foodstuff (mg/kg). The overall migration shall be calculated for each test specimen using the following formula:

$$ML = \frac{(m_a - m_b)}{V} \times 1000 \qquad (2)$$

- For the test articles with a volume of not less than 500 mL and not more than 10 L, the calculation formula will be (0.2L of food simulant is used):

$$ML = \frac{(m_a - m_b)}{0.2} \times 1000$$

that is:

$$ML = 5000 \times (m_a - m_b) \qquad (2.1)$$

where:

ML is the overall migration into the simulant, in milligrams per kilogram of food simulant;

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;

V is the volume of the test specimen intended to come into contact with foodstuff in litres (that is 200 ml, i.e. 0.2 L), since the specific gravity of the food simulants is conventionally assumed to be 1.

Calculate the result for each test specimen to the nearest 1 mg/kg and the mean of the individual test results, to the nearest 1 mg/kg. See Section 11 for the directions to determine whether the results are valid. Compile tables (listed in Annex A) containing the measured and calculated data on the test material and the blanks.

11. Validity of results – see Section 11 of HS 1001

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.
6. Commission Directive 93/8/EEC, Amending 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 – 15 March 1993, The Commission of the European Communities.
7. Commission Directive 93/9/EEC, Amending Directive 90/128/EEC - Plastics Materials and Articles Intended to come into Contact with Foodstuffs – 15 March 1993, The Commission of the European Communities.
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.
9. Commission Directive 97/48/EC, Amending 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 – 29 July 1997, The Commission of the European Communities.
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.
11. Commission Directive 93/10/EEC, Relating to materials and articles of regenerated cellulose film intended to come into contact with foodstuffs – 15 March 1993 (replaces 83/229/EEC), The Commission of the European Communities.
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 1186-9, Materials and articles in contact with foodstuffs – Plastics – Part 3: Test methods for overall migration into aqueous simulants by article filling, CEN, European Committee for Standardization, Brussels.

Annex A – 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 value of mass of residue in the blank (m_b): _____g

Volume of food simulant which had filled the article (v): _____ ml

Surface area of individual article (A): _____ dm^2

No. of articles exposed to the simulant (N): _____

	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 from food simulant/g (m_a)			
Overall migration, M or ML			

Calculate the overall migration into the simulant and expressed in milligrams of residue per square decimetre of the surface of the sample:

$$M = \frac{(m_a - m_b)}{A \times N \times 200} \times 1000 \times v \qquad (1)$$

Or express the overall migration in milligrams lost per kilograms of foodstuffs:

$$ML = 5000 \times (m_a - m_b) \qquad (2)$$

where:

M is the overall migration into the simulant, in milligram per square decimetres.

ML is the overall migration into the simulant, in milligrams per kilogram of food simulant;

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;

v is the volume of food simulant which had filled the article, in milliltres;

S is the surface area of test specimen intended to come into contact with food, in square decimetre;

A Surface area of individual article, in square decimetre.

N Number of articles exposed to the simulant