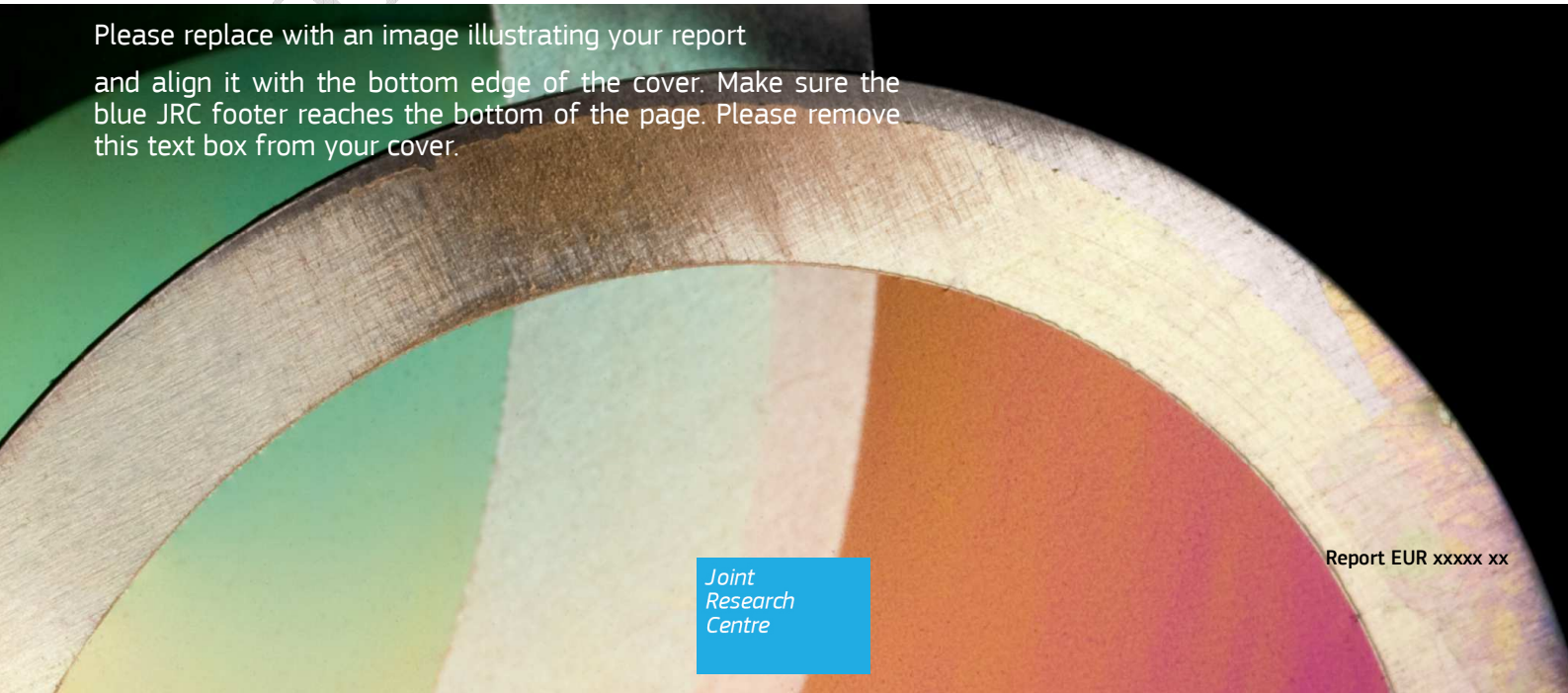
European Commission

JRC SCIENCE AND POLICY REPORTS

Technical guidelines for compliance testing – Annexes

In the framework of Regulation (EU) No 10/2011 on plastic food contact materials

DRAFT FOR CONSULTATION



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European Commission
Joint Research Centre
Institute for xxx / Directorate for xxx *(remove the irrelevant)*

Contact information
Forename Surname
Address: Joint Research Centre, xxx *(relevant complete address)*
E-mail: forename.surname@ec.europa.eu
Tel.: +xx xxxx xx xxxx

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Abstract

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Annex 1 Example of sampling protocol

Sampling protocol in accordance with Regulation (EU) No 10/2011		
Competent authority and/ or competent inspection unit {Address}	Date of sampling	Time of sampling
Manufacturer of sample {Address}	Place of sampling {Address}	Best before date: Used by:
Identification of sample ☐ Lot number: ☐ Batch number: ☐ Item number: ☐ EU control number: ☐ Custom number: ☐ Other:	Type of sample ☐ Article ☐ Material ☐ Starting substances ☐ Product from intermediate stages of manufacturing	Type of business ☐ Producer ☐ Manufacture and Converter ☐ Importer ☐ Wholesale trade ☐ Retailer ☐ Service company
Quantity of sample Quantity of sample for dispute and/or reference Quantity of inventory Approximately:	Amount and/or size of sample Amount and/or size of sample for dispute and/or reference Amount and/or size of inventory Approximately:	Detailed description of sample: ☐ Set-off?
Sampling temperature During sampling: °C Surrounding temperature: °C	Transport of sample ☐ Not cooled ☐ Cooled at: °C ☐ Freezed at: °C ☐ Sealed Sealed sample arrived undamaged: ☐ Yes ☐ No Transport of sample via ☐ Courier ☐ Inspectors ☐ Authorized and instructed person:	Spot of Sampling ☐ Production / -plant: Details: ☐ Storage ☐ Sale ☐ Self-Service ☐ Cooling unit ☐ Freezer Possibility of Set-Off ☐ Yes ☐ No Details:

Photo taken at the spot of sampling ☐ Yes, see annex ☐ No	Reason of sampling ☐ Routine sample ☐ Suspicion sample ☐ Appeal sample ☐ Import sample ☐ Import suspicion sample ☐ Traceability sample ☐ Sample in the context of Declaration of compliance	Request for Declaration of Compliance ☐ Yes ☐ No Address:
Enforcement laboratory {Address}	Date of receipt	Sample code or sample ID
Name and signature of responsible person {Place of sampling}	Name and signature of sampler {Competent authority}	Name and signature confirmation of sample receipt {Enforcement laboratory}

2

3

Annex 2 Abstract Fraunhofer IVV Report 'FPE Functional Barrier Project', 09.12.2011. Publication in preparation

From a food regulatory standpoint it is of high interest to define under which conditions a barrier layer acts as a functional barrier, i.e. prevents migration from layers behind, such as printing ink layers or other, higher than 10 ppb.

The intention of this project was to investigate into this question by carrying out a series of permeation tests on flexible films with and without barrier layers using a selection of test permeants. Permeation tests were preferred over (experimentally much more complicated migration tests) because the experimental set up used in this study allows to test much more combinations of test substances, test films and test parameters compared to migration tests.

Altogether 24 different flexible packaging films of practical relevance and 12 different test permeants (organic chemicals of representative chemical properties) were selected in agreement with the customer. Critera for the selection of substances were chemical representativeness and relevance to finished packaging films. i.e. occurring for instance in printing inks or other layers behind the food contact layer. Target test scenarios were selected such that they cover typical and worst case food-packaging contact conditions used in compliance testing according to Regulation (EU) No 10/2011, i.e. temperatures in the range between 20 °C and 60 °C. In practical terms and with already existing knowledge of permeation this means predominantly tests at 40°C and 60 °C for time periods of up to 40 days and 14 days, respectively, because of too many expectable non-detectable permeation results at room temperature.

Based on the test results proposal are made how to apply the measured permeation behaviour can be applied to evaluate or predict the barrier behaviour of packaging films against migration from outside layers into the food. A list of general functional barriers which would always be efficient and a list of relative functional barriers which would generally work at room temperature was established.

Annex 3 Cleaning procedures for food simulant E

Gas chromatograms obtained from extracts of new commercial poly(2,6-diphenyl-p-phenylene oxide) (PPPO) have shown that unacceptably high levels of impurities may be present. Therefore, prior to its first use in this test procedure, the PPPO shall be purified by soxhlet extraction. There are two methods described.

Method 1 using acetone (Beldì et al., 2012)

1. Put PPPO into the Soxhlet thimble;
2. Add acetone into the boiling flask (250 mL of acetone for 20 g of PPPO);
3. Turn on the heater and clean (extract continuously) the PPPO for 6 h;
4. After 6 h turn off the heating system take out the PPPO from the cartridge into the beaker
5. or big Petri dish;
6. Place the covered Petri dish under the fume hood to evaporate the solvent while mixing;
7. Put Petri dish into the oven at 160°C for 6 h;
8. After heating, store the PPPO into closed Erlenmeyer flask.

Method 2 using diethylether.

The extraction is carried as follows:

1. place the PPPO in a soxhlet cartridge and extract for 6 h with diethylether.
2. Spread the PPPO in a Petri dish of a suitable diameter and place the dish in a fume hood.
3. AlIow the diethylether to evaporate while frequently mixing with a glass rod.
4. Then place the dish in an oven al 160°C for 6 h.
5. After heating store the PPPO, if not needed immediately, in a closed conical flask.

NOTE 1 Heating of PPPO saturated with diethylether can be explosive. Therefore, it should be ensured that diethylether is completely evaporated before drying at 160°C.

NOTE 2 PPPO cleaned in this way can be used repeatedly.

NOTE 3 PPPO is powdery and lightweight and is readily blown about by air currents. When drying PPPO or carrying out the contact in a forced air oven, the oven should be set to low and cover dishes to prevent the MPPO from blowing about.

Annex 4 Background Table 5, 7, 9

Scientific reasoning for the calculation of (1) maximum layer thickness to be used for worst case calculation of migration, (2) minimum layer thickness at which functional barrier properties occur, (3) area considerations for full immersion testing.

Introduction

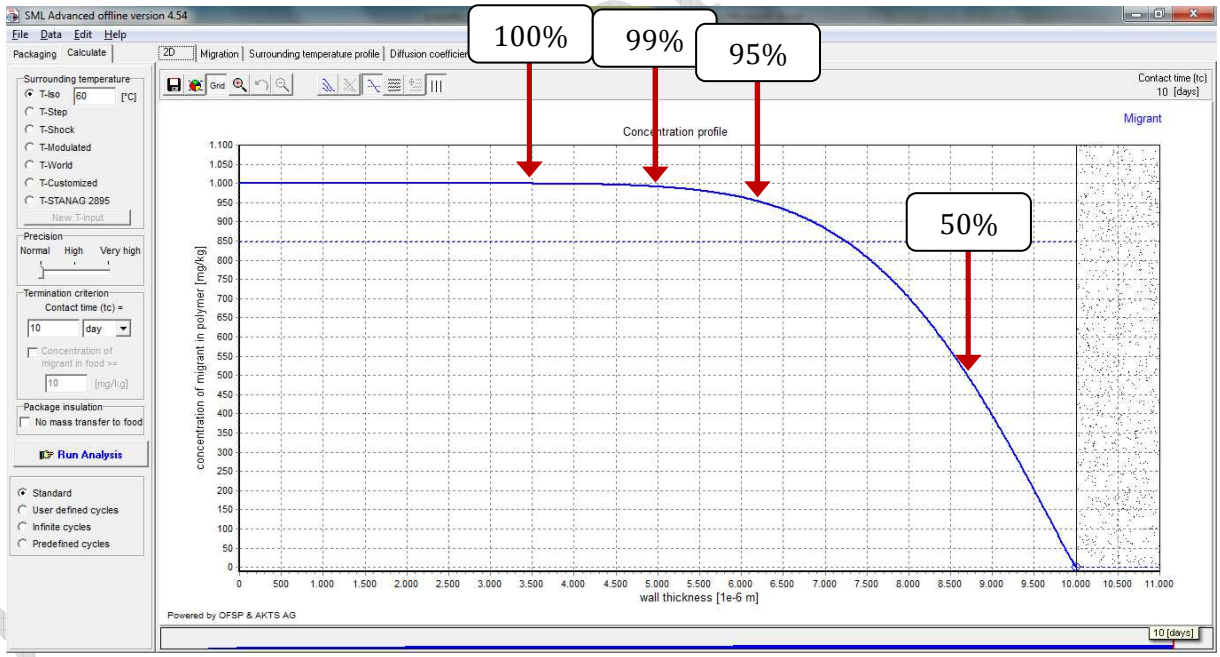
The maximum layer thickness (L_{WC} in µm) for worst case migration calculation is 1/2 of the 99% layer thickness.

The minimum layer thickness at which it can be considered that the layer acts as an absolute barrier is equal to the 100% layer thickness.

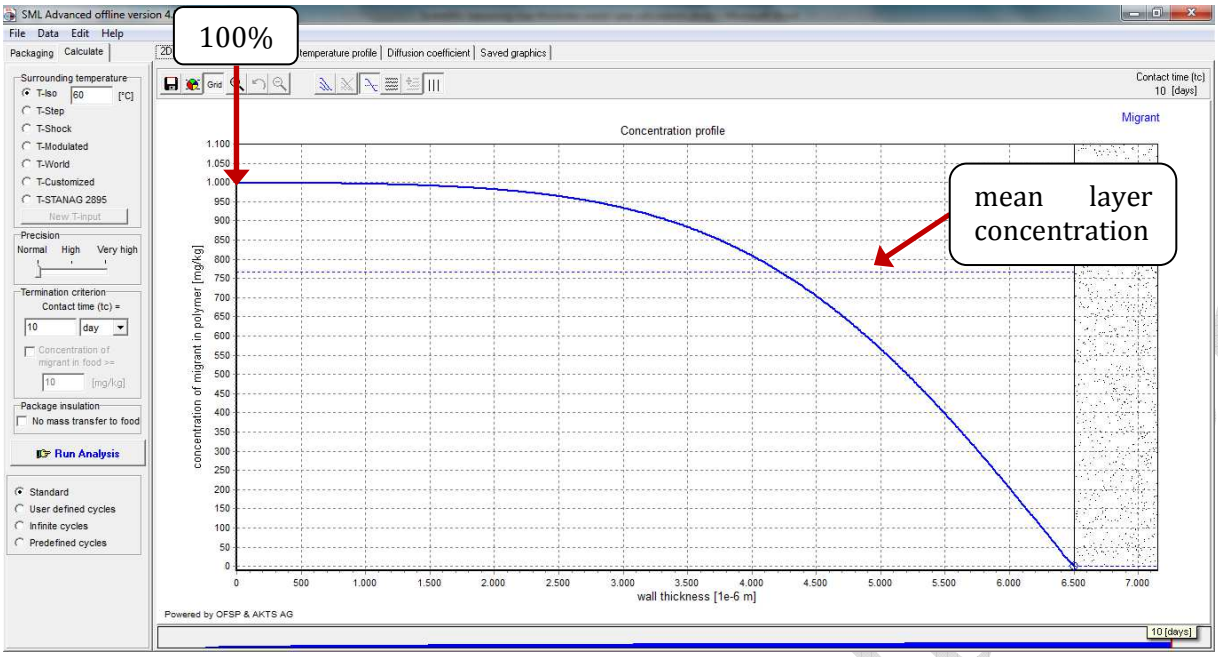
The minimum layer thickness at which for full immersion testing both sides of the sample can be considered for calculation of the migration result is 2 times the 99% layer thickness.

To explain the calculation procedure a LDPE monolayer with material thickness 10000 µm (= 10 cm) and "upper limit" polymer specific constant A_P' = 11.5; tau = 0 was selected. The loss of migrant (specific migration to an infinite contact medium) with molecular mass of 500 g/mol (initial concentration 1000 mg/kg plastic) after 10 days at 60°C was calculated. The concentration profile is shown in the figure below.

The 100% layer thickness is where the concentration profile is not affected by one sided loss of migrant (in this example molecular mass = 500 g/mol) under the time temperature conditions considered (in this example 10 days at 60°C), i.e. the concentration is still equal with the initial concentration.



For LDPE and under assumption of "upper limit" diffusion behavior for a migrant with molecular mass of 500 g/mol a 100% layer thickness of 6500 µm results under the time temperature conditions 10 days at 60°C.



For a material with 100% layer thickness (in this example 6500 µm for LDPE) approximate 23.3% = (initial concentration 1000 ppm - mean layer concentration 767 / initial concentration 1000 x 100) of the initial amount of the substance (in this example 500 g/mol molecular mass of migrant) left the material under the time temperature conditions considered (in this example 10 days at 60°C).

The mean concentration in the layer is shown in the graph above by the blue dashed line.

LDPE; PP, rubbery

► molecular mass 100 - 250 g/mol

10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied

10d @ 40°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied

10d @ 20°C

=> 100% layer thickness = 7000 µm
no absolute barrier at thicknesses below 7000 µm

=> 99% layer thickness = 5000 µm

=> 1/2 x 99% layer thickness = 2500 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 10000 µm
above 10000 µm two sides to be considered for calculation of migration if full immersion testing applied

2h @ 100°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied

► **molecular mass 251 - 500 g/mol**

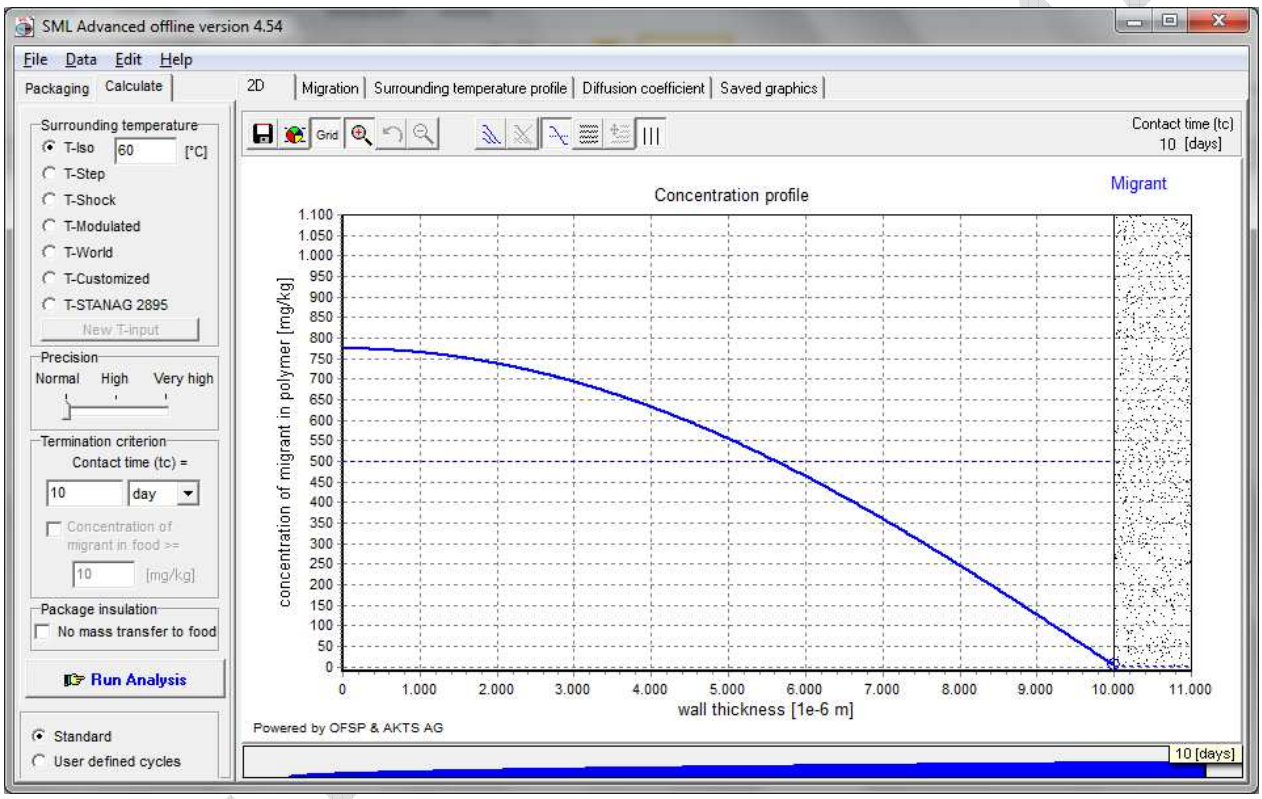
10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing applied



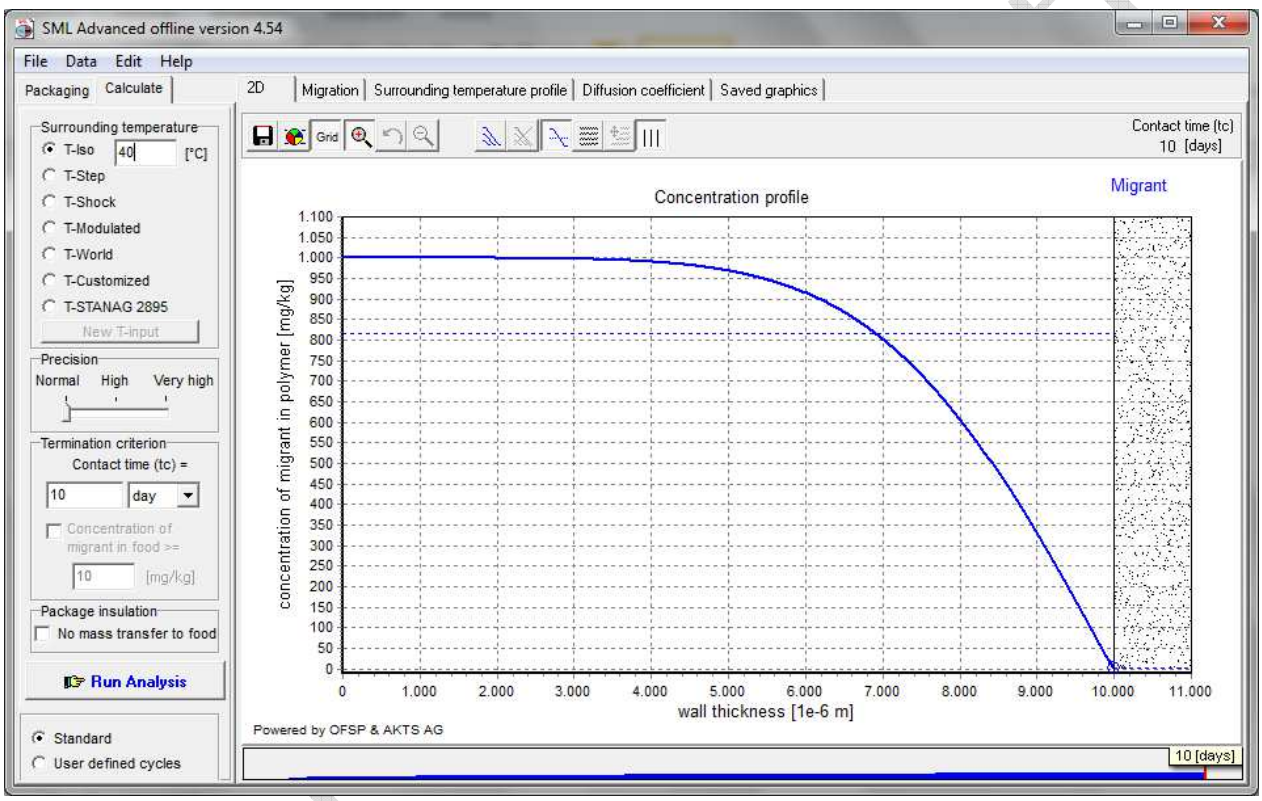
10d @ 40°C

=> 100% layer thickness = 8800
no absolute barrier at thicknesses below 8800 µm

=> 99% layer thickness = 6000 µm

=> 1/2 x 99% layer thickness = 3000 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 12000
above 12000 µm two sides to be considered for calculation of migration if full immersion testing applied



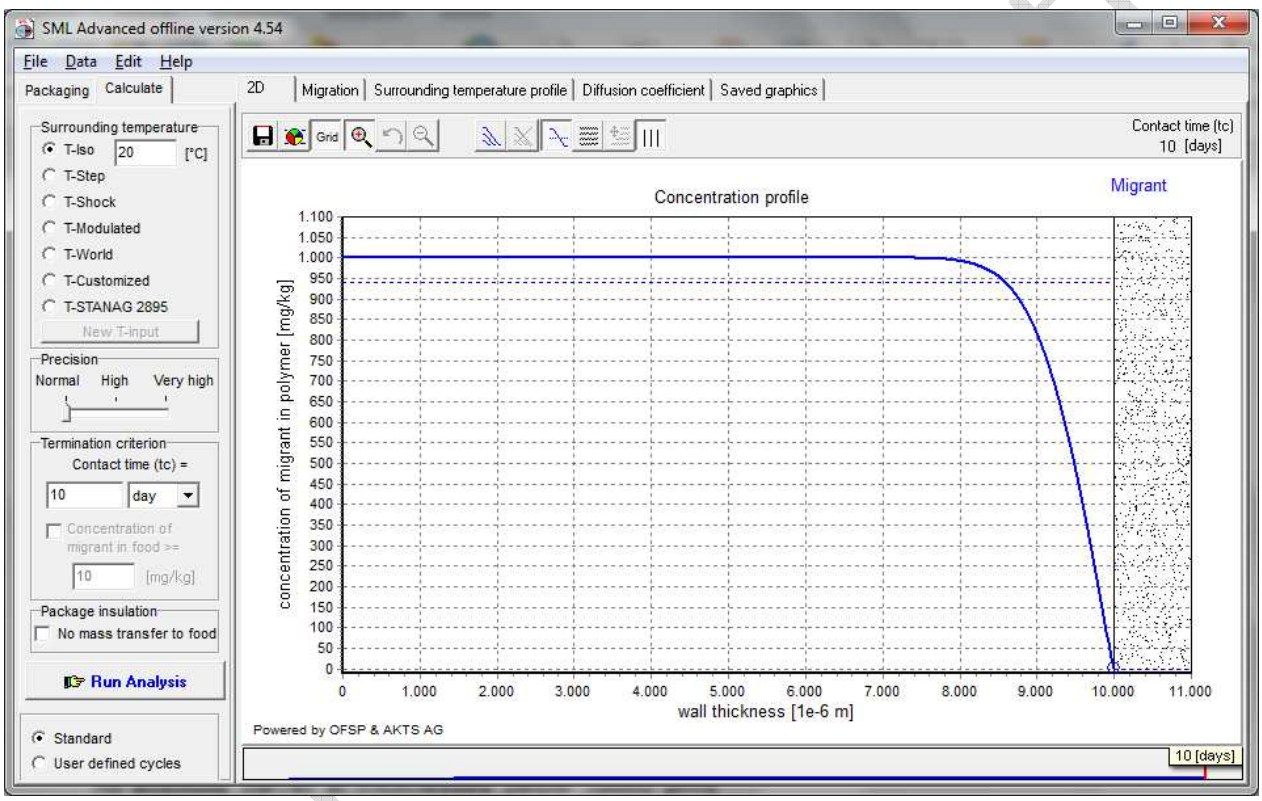
10d @ 20°C

=> 100% layer thickness = 3000 µm
no absolute barrier at thicknesses below 3000 µm

=> 99% layer thickness = 1760 µm

=> 1/2 x 99% layer thickness = 880 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3520 µm
above 3520 µm two sides to be considered for calculation of migration if full immersion testing applied



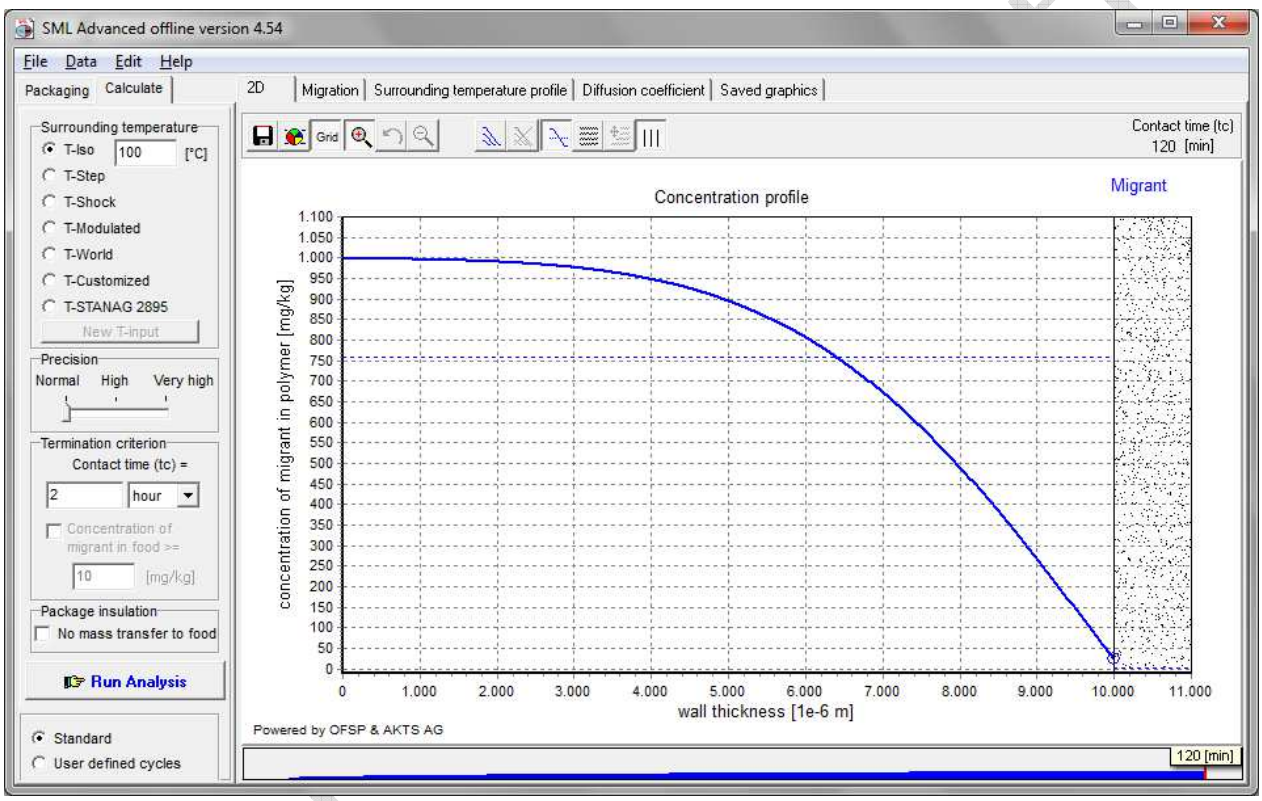
2h @ 100°C

=> 100% layer thickness = 10000
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = 8000

=> 1/2 x 99% layer thickness = 4000
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 16000
above 16000 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

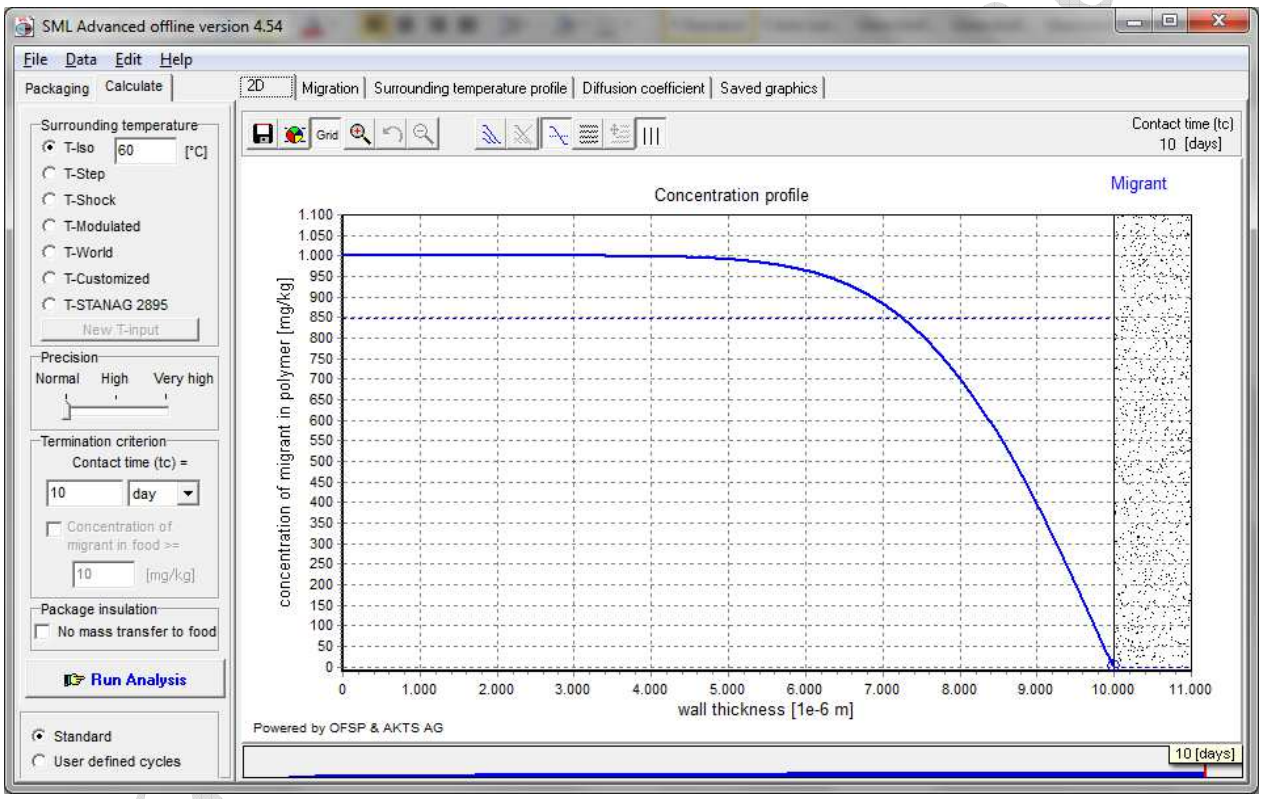
10d @ 60°C

=> 100% layer thickness = 7000 µm
no absolute barrier at thicknesses below 7000 µm

=> 99% layer thickness = 4800

=> 1/2 x 99% layer thickness = 2400 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 9600
above 9600 µm two sides to be considered for calculation of migration if full immersion testing applied



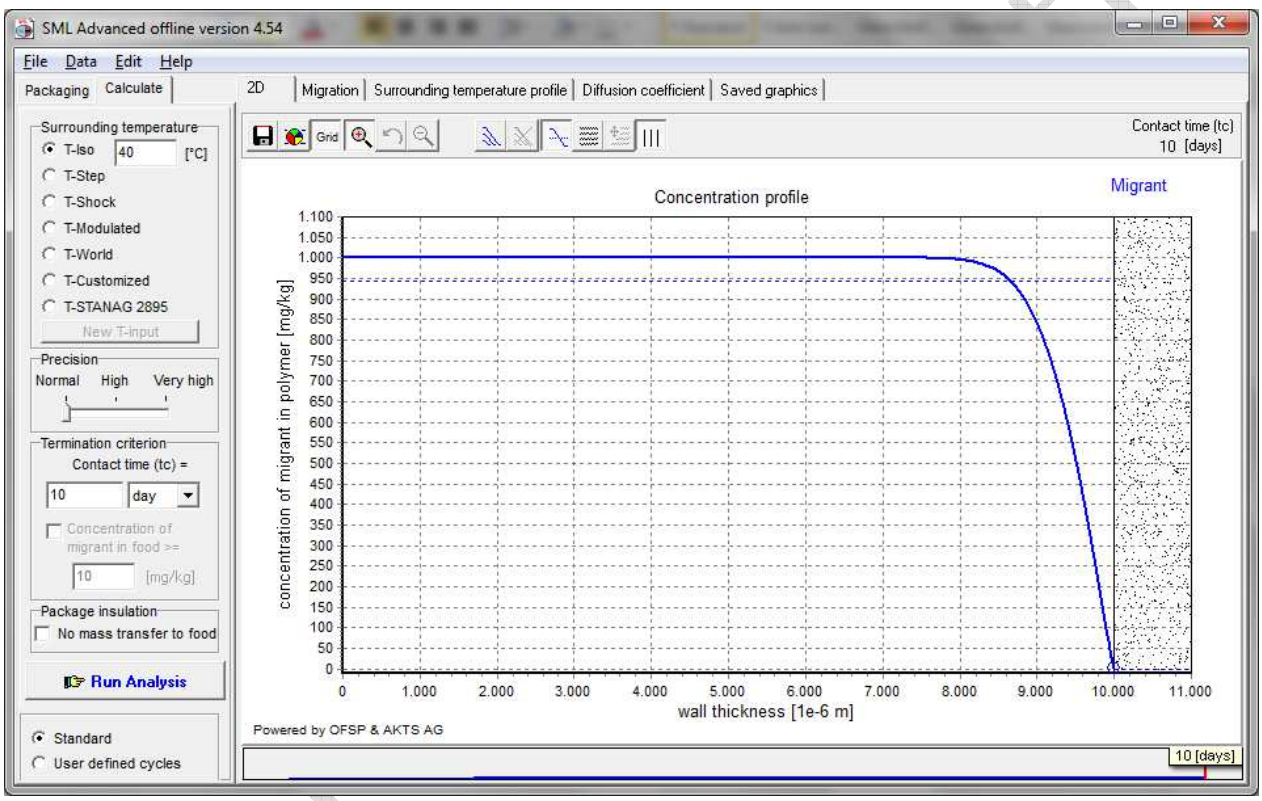
10d @ 40°C

=> 100% layer thickness = 2640
no absolute barrier at thicknesses below 2640 µm

=> 99% layer thickness = 1840 µm

=> 1/2 x 99% layer thickness = 920 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3680
above 3680 µm two sides to be considered for calculation of migration if full immersion testing applied



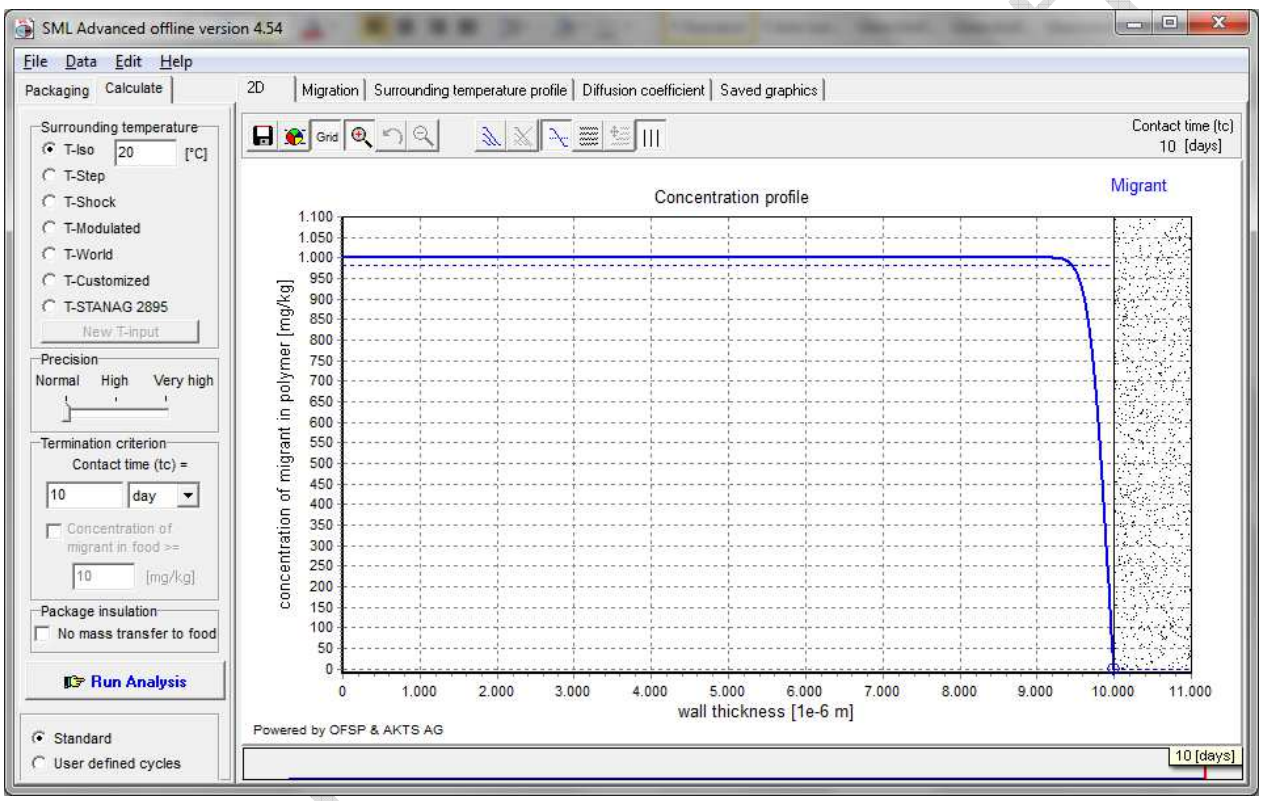
10d @ 20°C

=> 100% layer thickness = 800 µm
no absolute barrier at thicknesses below 800 µm

=> 99% layer thickness = 600 µm

=> 1/2 x 99% layer thickness = 300 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1200 µm
above 1200 µm two sides to be considered for calculation of migration if full immersion testing applied



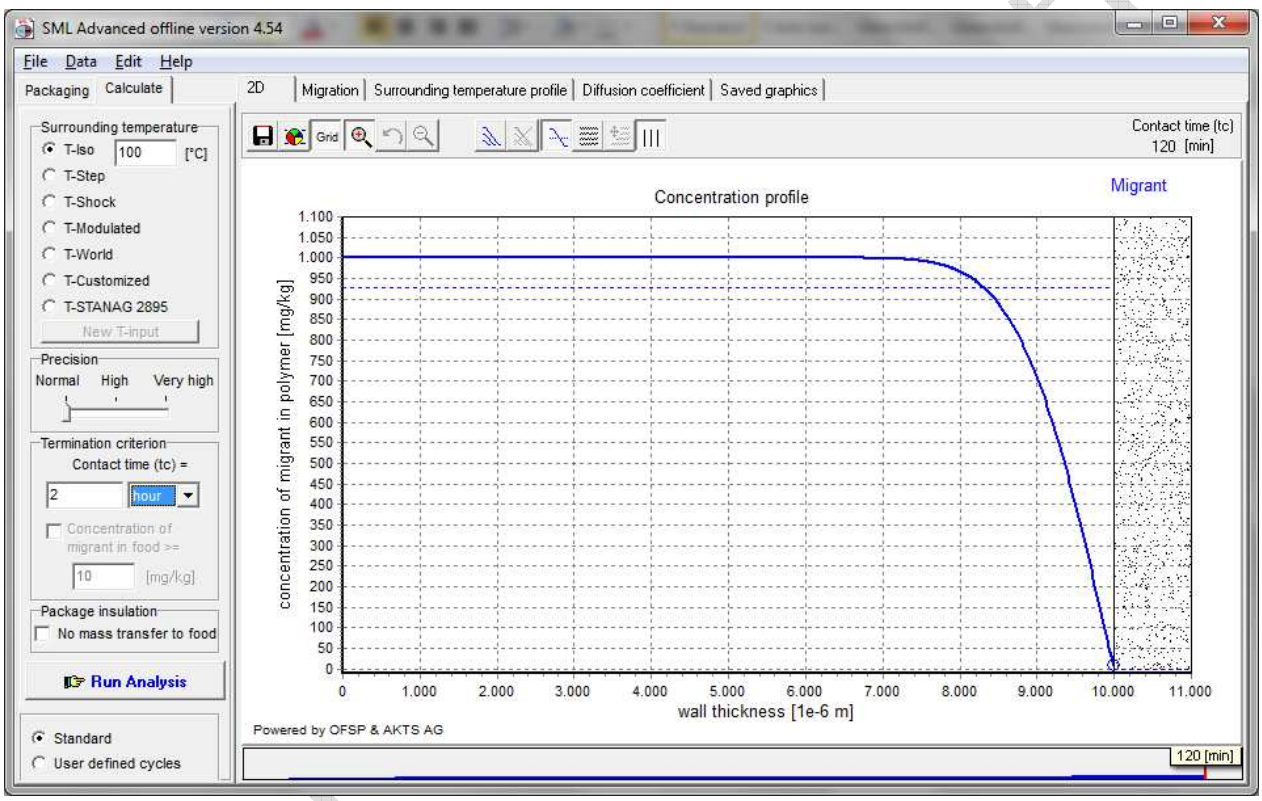
2h @ 100°C

=> 100% layer thickness = 3240
no absolute barrier at thicknesses below 3240 µm

=> 99% layer thickness = 2440 µm

=> 1/2 x 99% layer thickness = 1220 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4880
above 4880 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

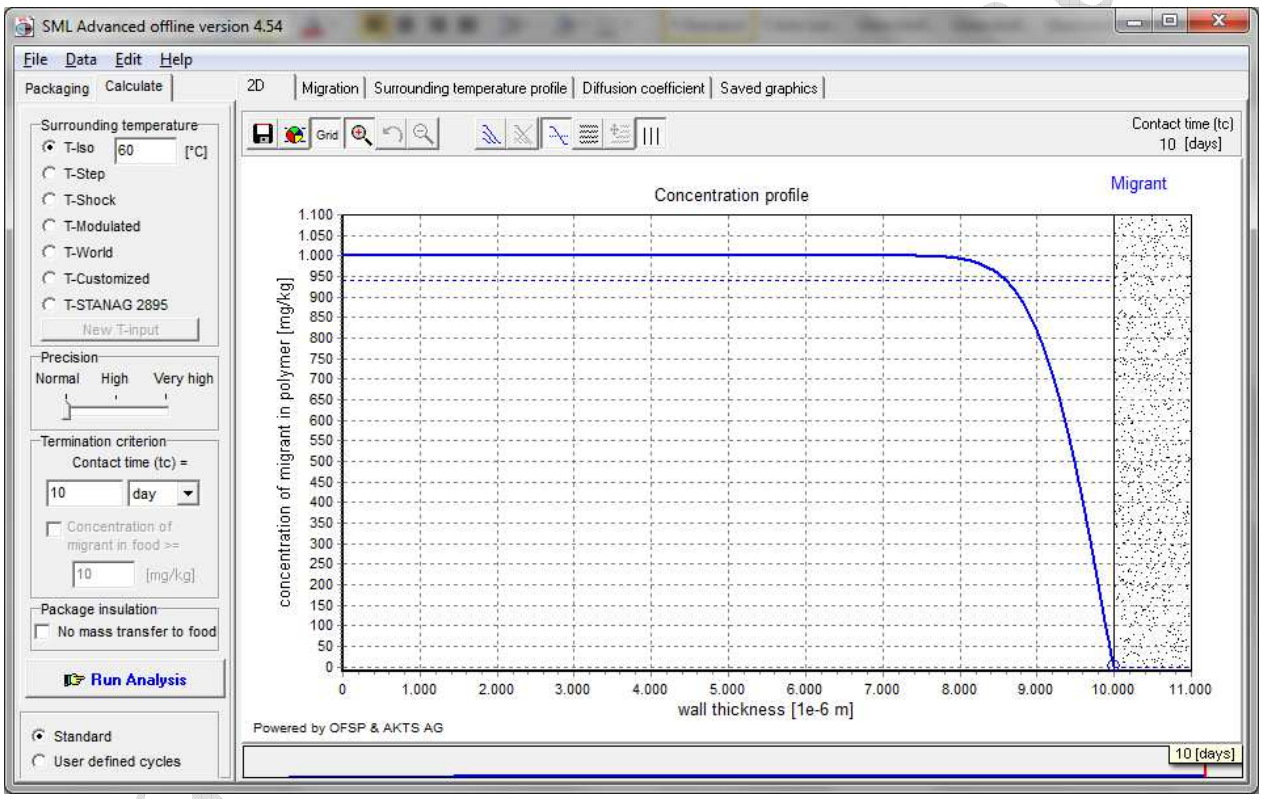
10d @ 60°C

=> 100% layer thickness = 2600 µm
no absolute barrier at thicknesses below 2600 µm

=> 99% layer thickness = 1920

=> 1/2 x 99% layer thickness = 960 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2840
above 2840 µm two sides to be considered for calculation of migration if full immersion testing applied



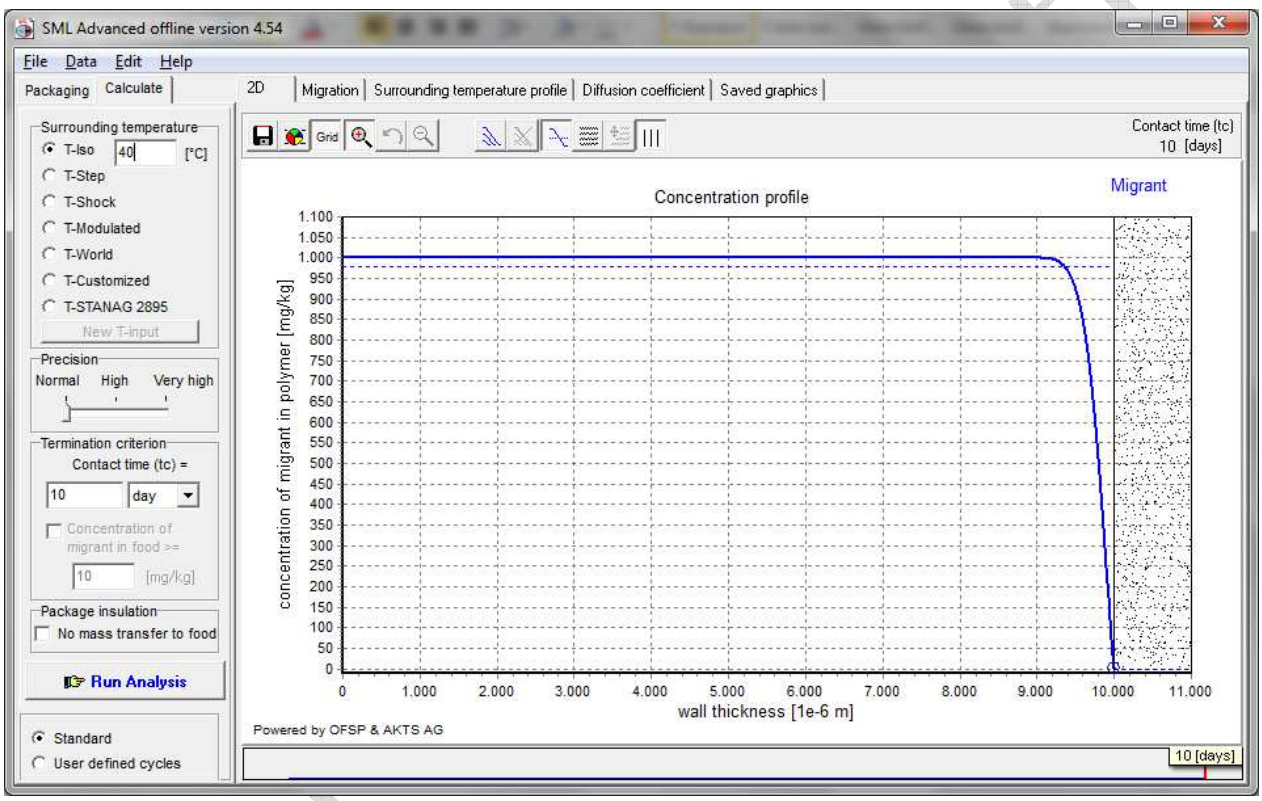
10d @ 40°C

=> 100% layer thickness = 1000
no absolute barrier at thicknesses below 1000 µm

=> 99% layer thickness = 720 µm

=> 1/2 x 99% layer thickness = 360 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1440
above 1440 µm two sides to be considered for calculation of migration if full immersion testing applied



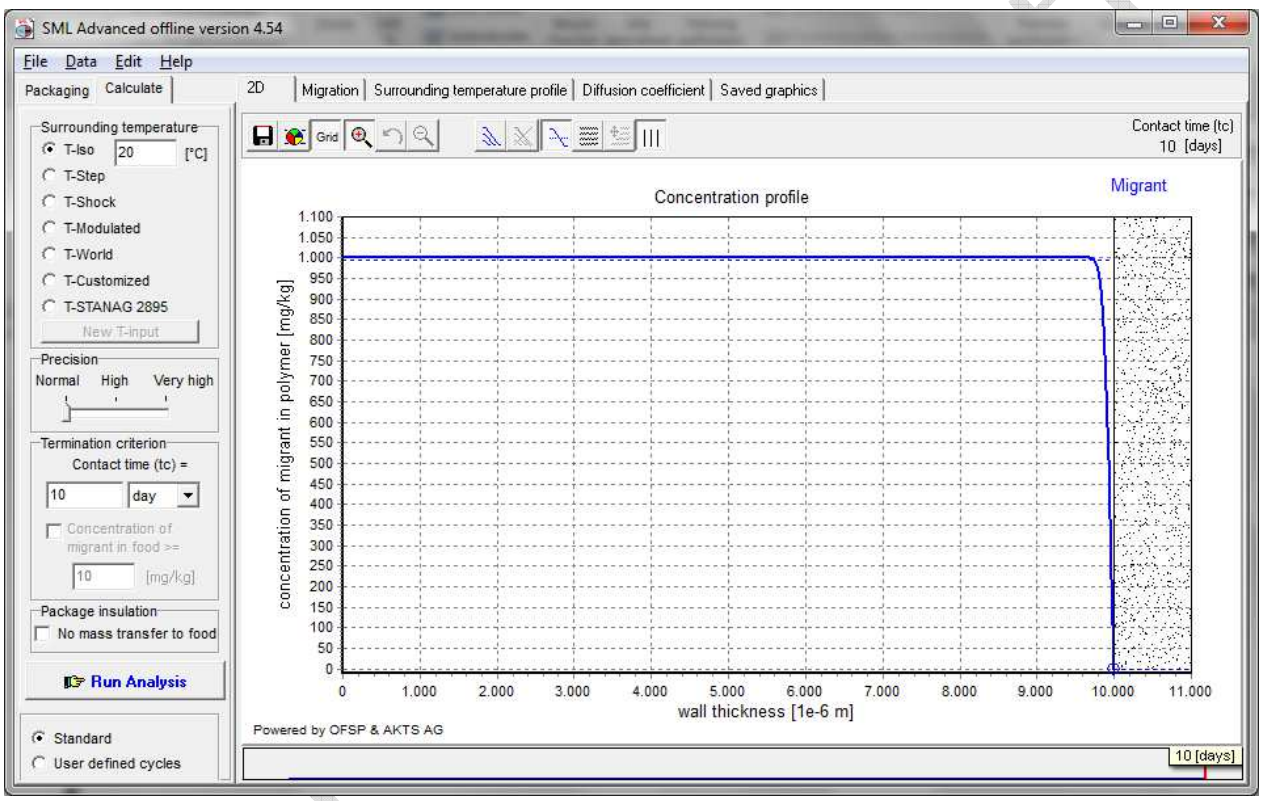
10d @ 20°C

=> 100% layer thickness = 340 µm
no absolute barrier at thicknesses below 340 µm

=> 99% layer thickness = 240 µm

=> 1/2 x 99% layer thickness = 120 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 480 µm
above 480 µm two sides to be considered for calculation of migration if full immersion testing applied



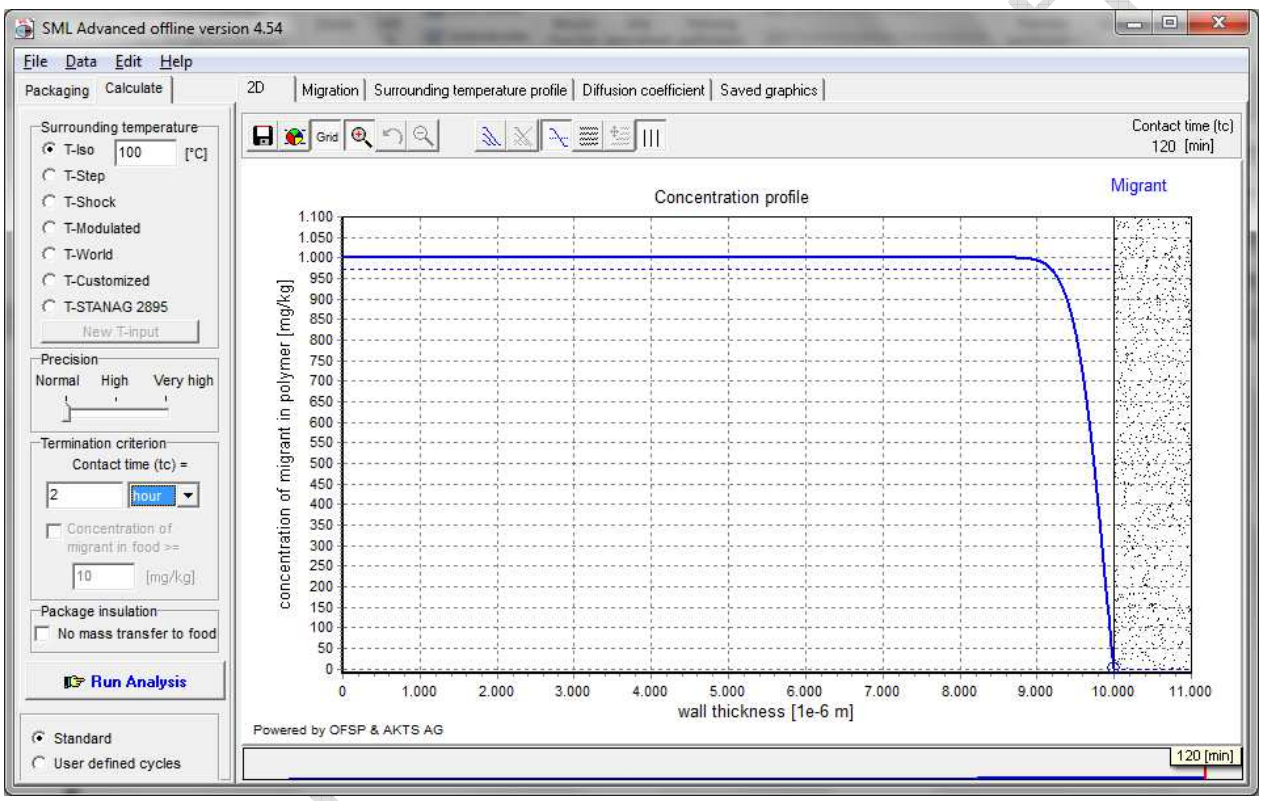
2h @ 100°C

=> 100% layer thickness = 1360
no absolute barrier at thicknesses below 1360 µm

=> 99% layer thickness = 960 µm

=> 1/2 x 99% layer thickness = 480 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1920
above 1920 µm two sides to be considered for calculation of migration if full immersion testing applied



HDPE

► molecular mass 100 - 250 g/mol

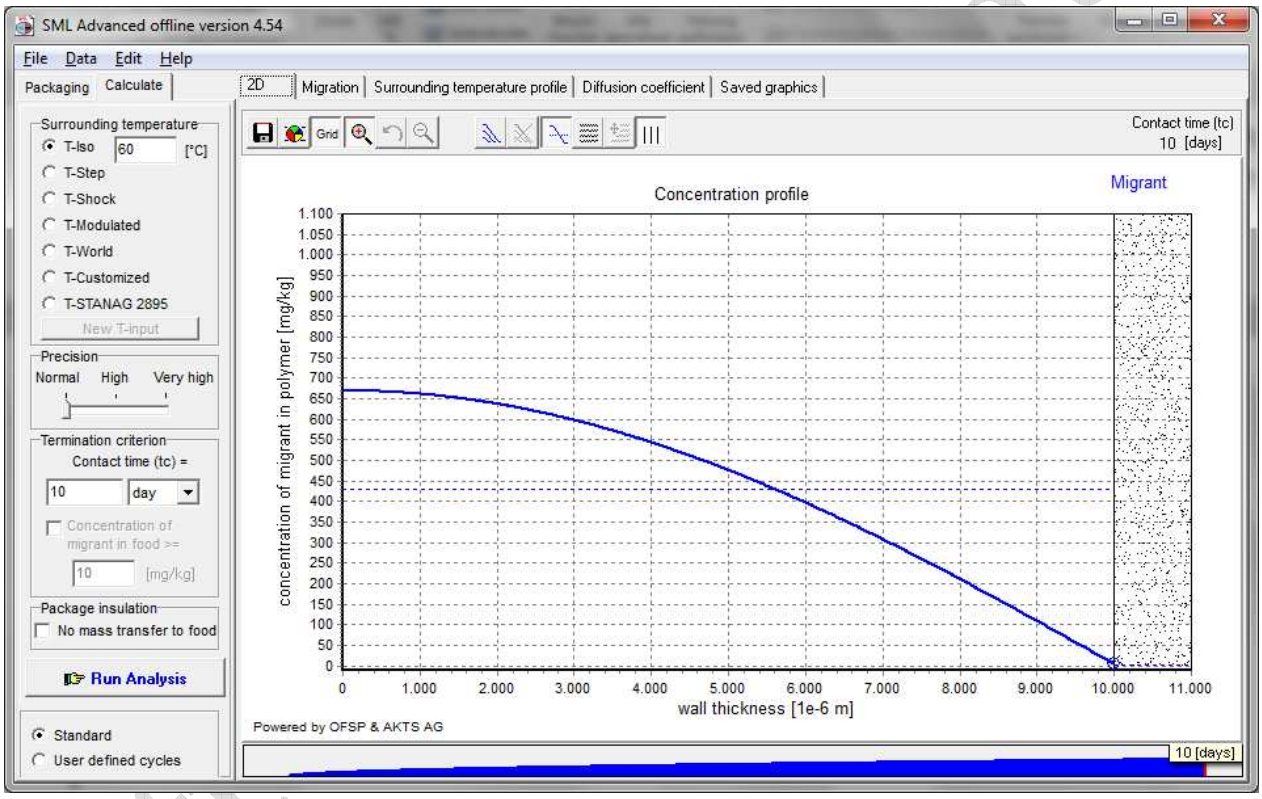
10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier

=> 99% layer thickness = full length

=> 1/2 x 99% layer thickness = full length
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
one side to be considered for calculation of migration if full immersion testing applied



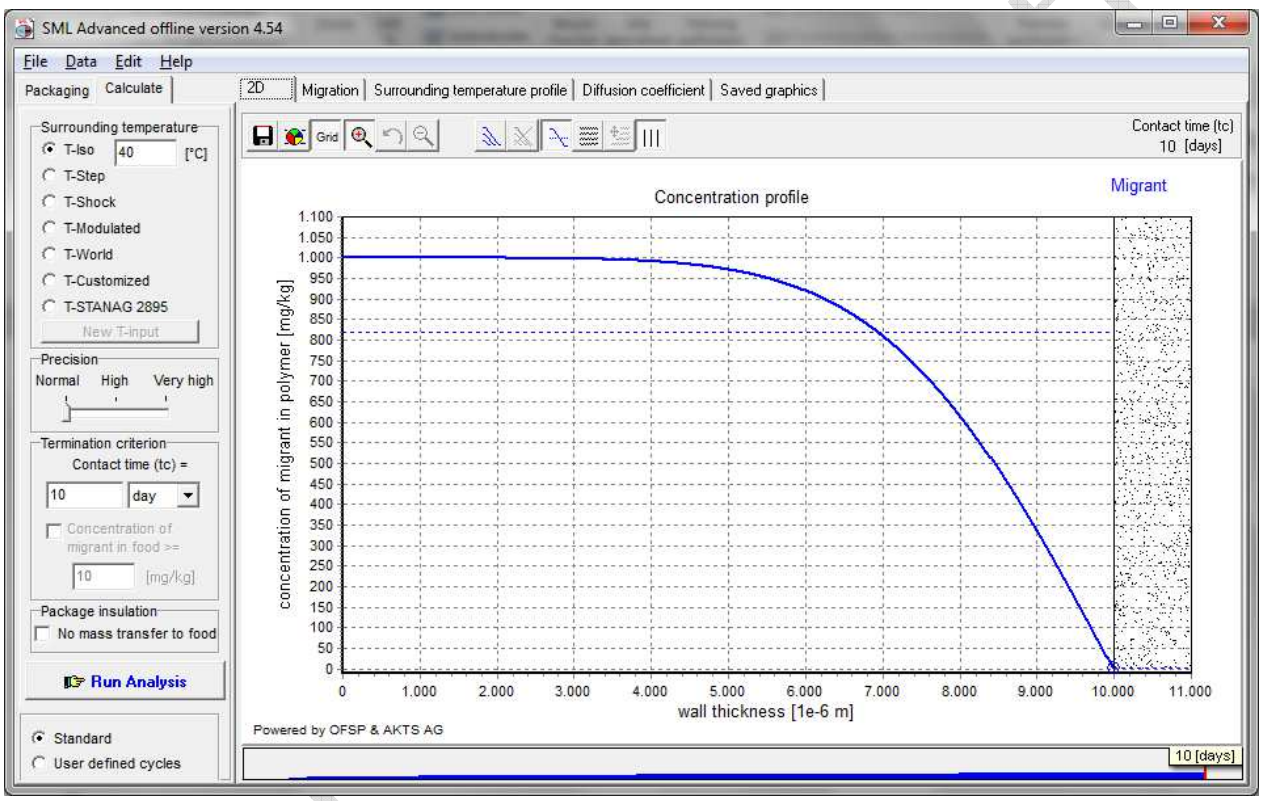
10d @ 40°C

=> 100% layer thickness = 8500
no absolute barrier at thicknesses below 8500 µm

=> 99% layer thickness = 5900 µm

=> 1/2 x 99% layer thickness = 2950 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 11800
above 11800 µm two sides to be considered for calculation of migration if full immersion testing applied



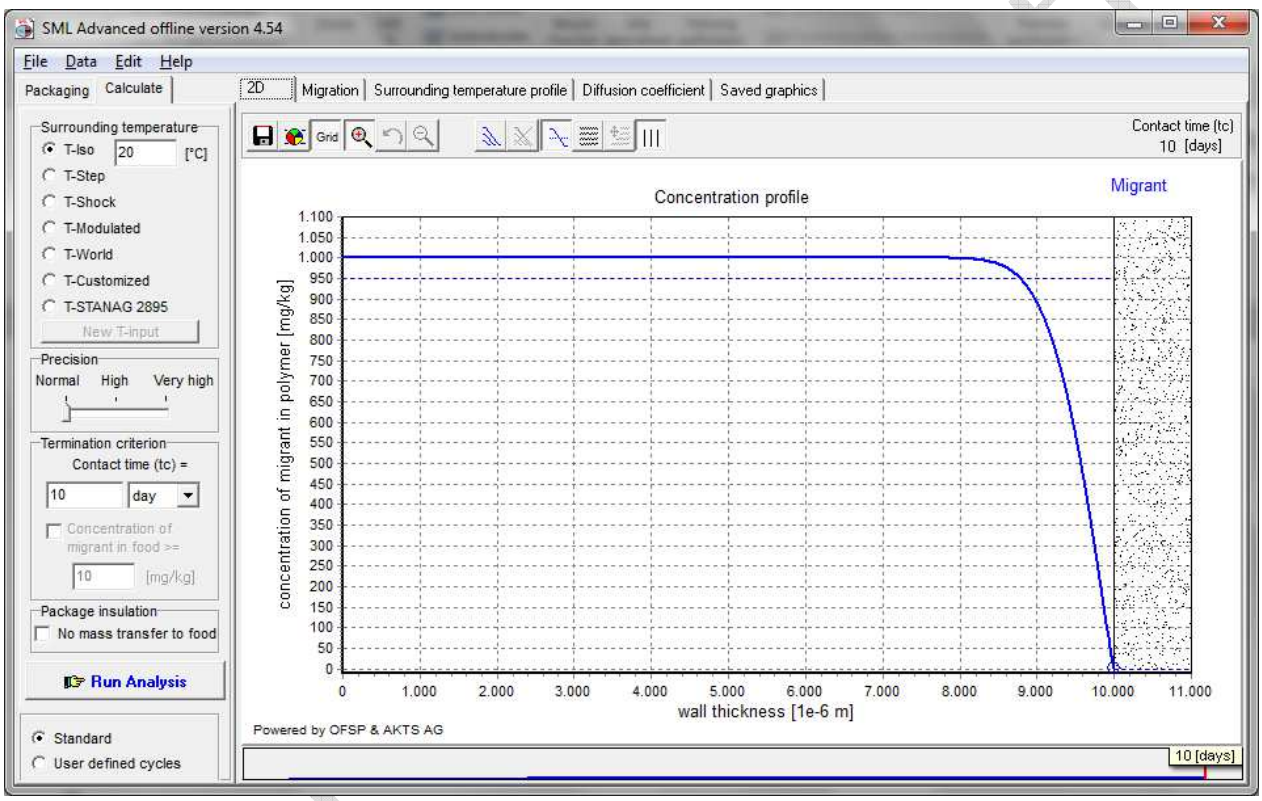
10d @ 20°C

=> 100% layer thickness = 2280 µm
no absolute barrier at thicknesses below 2280 µm

=> 99% layer thickness = 1600 µm

=> 1/2 x 99% layer thickness = 800 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3200 µm
above 3200 µm two sides to be considered for calculation of migration if full immersion testing applied



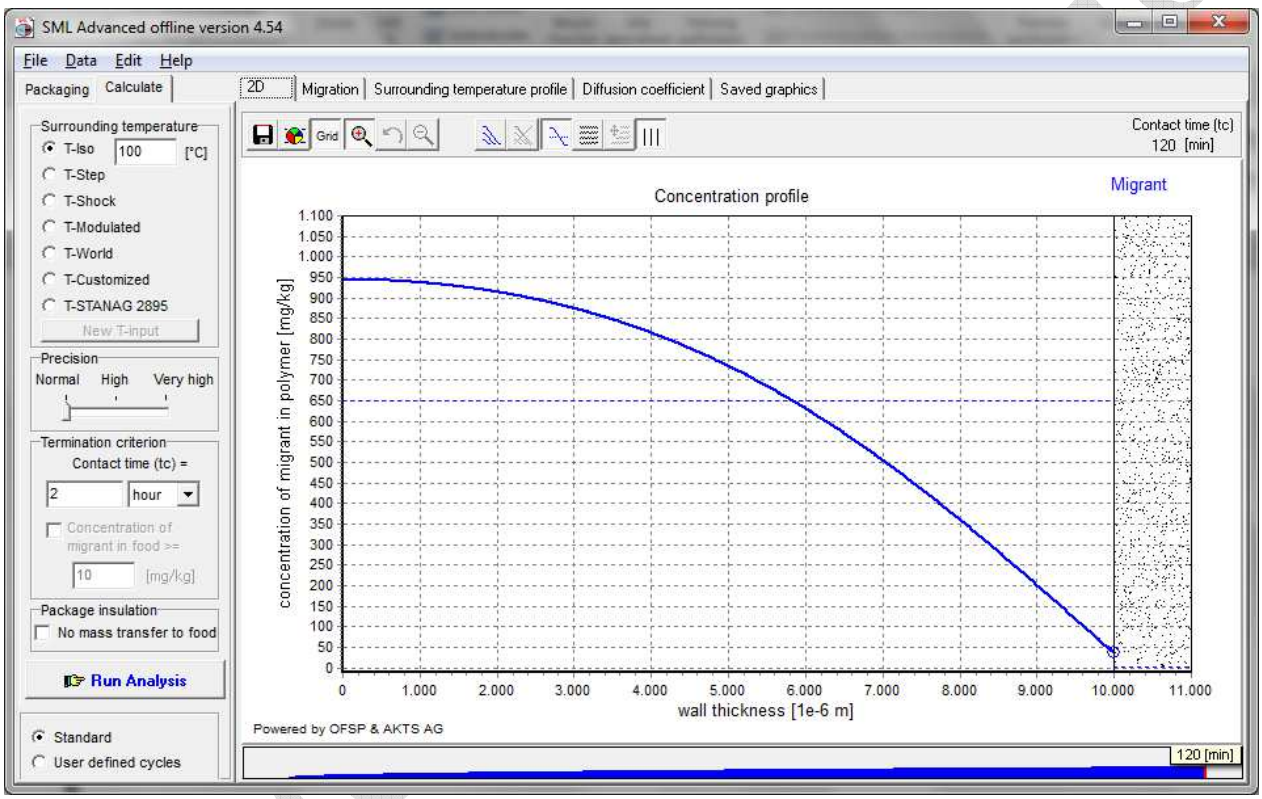
2h @ 100°C

=> 100% layer thickness = full length
no absolute barrier

=> 99% layer thickness = full length

=> 1/2 x 99% layer thickness = none
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
one side to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

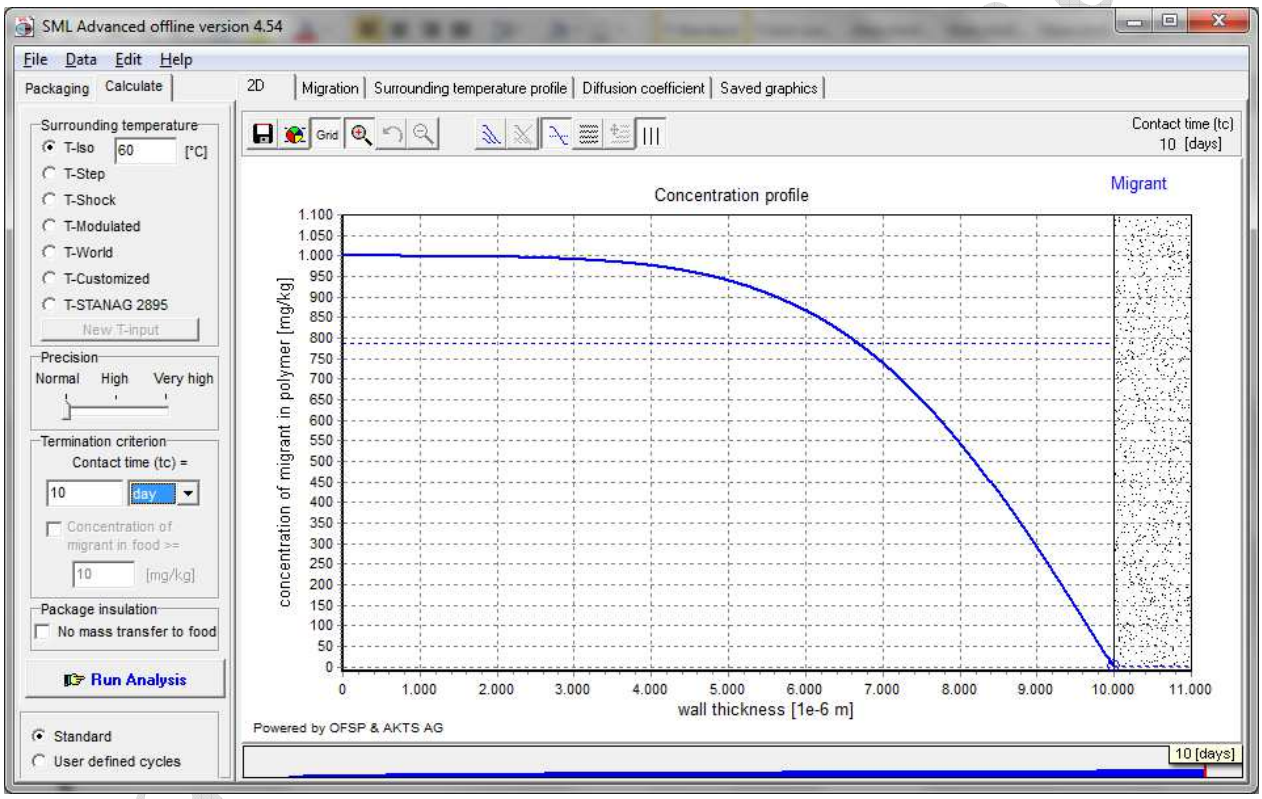
10d @ 60°C

=> 100% layer thickness = 9000 µm
no absolute barrier at thicknesses below 9000 µm

=> 99% layer thickness = 6850 µm

=> 1/2 x 99% layer thickness = 3425 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 13700 µm
above 13700 µm two sides to be considered for calculation of migration if full immersion testing applied



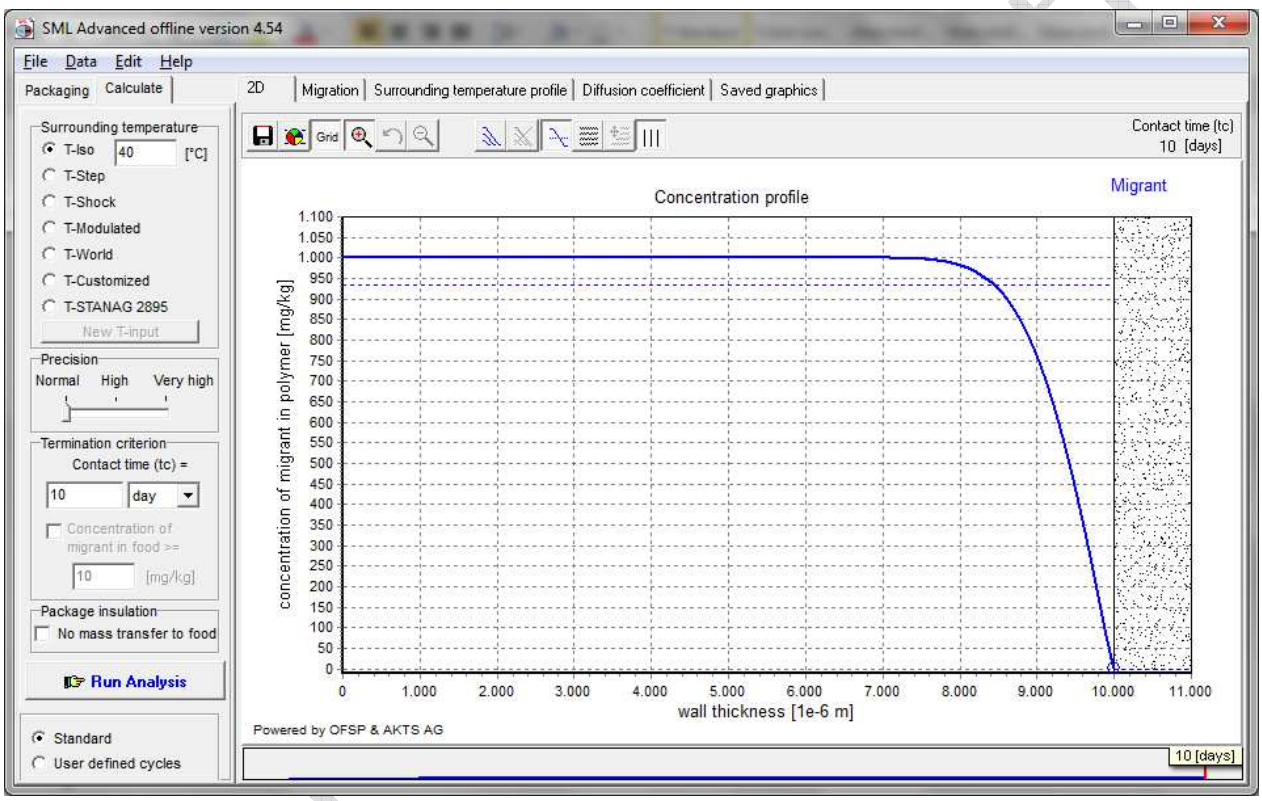
10d @ 40°C

=> 100% layer thickness = 3000
no absolute barrier at thicknesses below 3000 µm

=> 99% layer thickness = 2200 µm

=> 1/2 x 99% layer thickness = 1100 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4800
above 4800 µm two sides to be considered for calculation of migration if full immersion testing applied



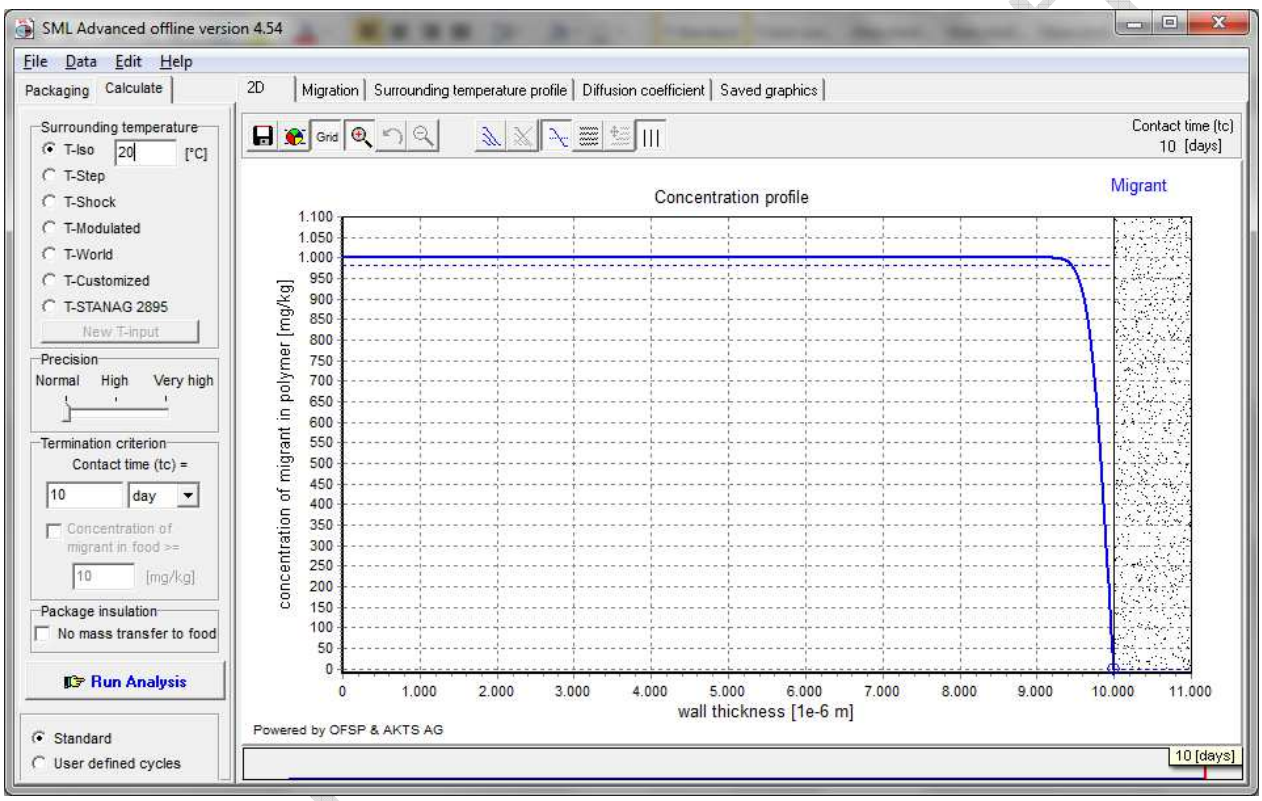
10d @ 20°C

=> 100% layer thickness = 800 µm
no absolute barrier at thicknesses below 800 µm

=> 99% layer thickness = 600 µm

=> 1/2 x 99% layer thickness = 300 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1200 µm
above 1200 µm two sides to be considered for calculation of migration if full immersion testing applied



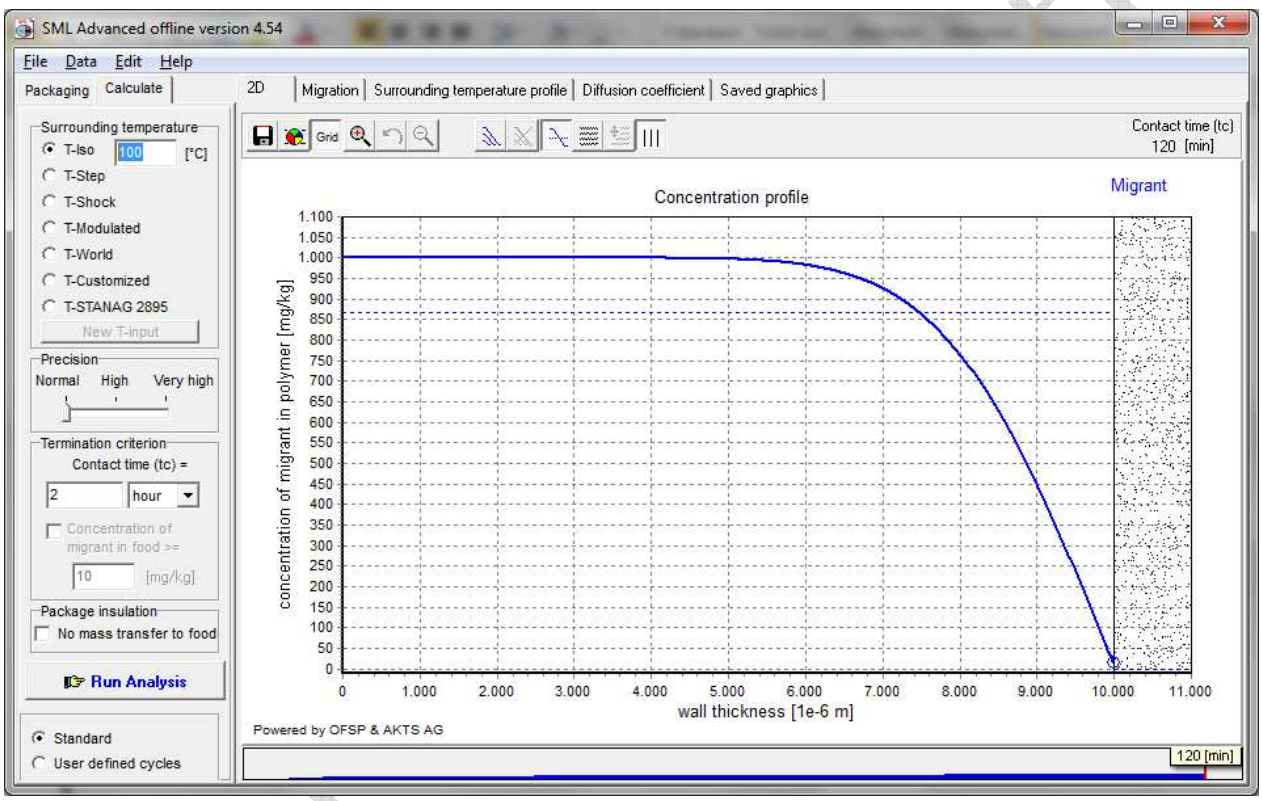
2h @ 100°C

=> 100% layer thickness = 6400 µm
no absolute barrier at thicknesses below 6400 µm

=> 99% layer thickness = 4300 µm

=> 1/2 x 99% layer thickness = 2150 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 8400 µm
above 8400 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

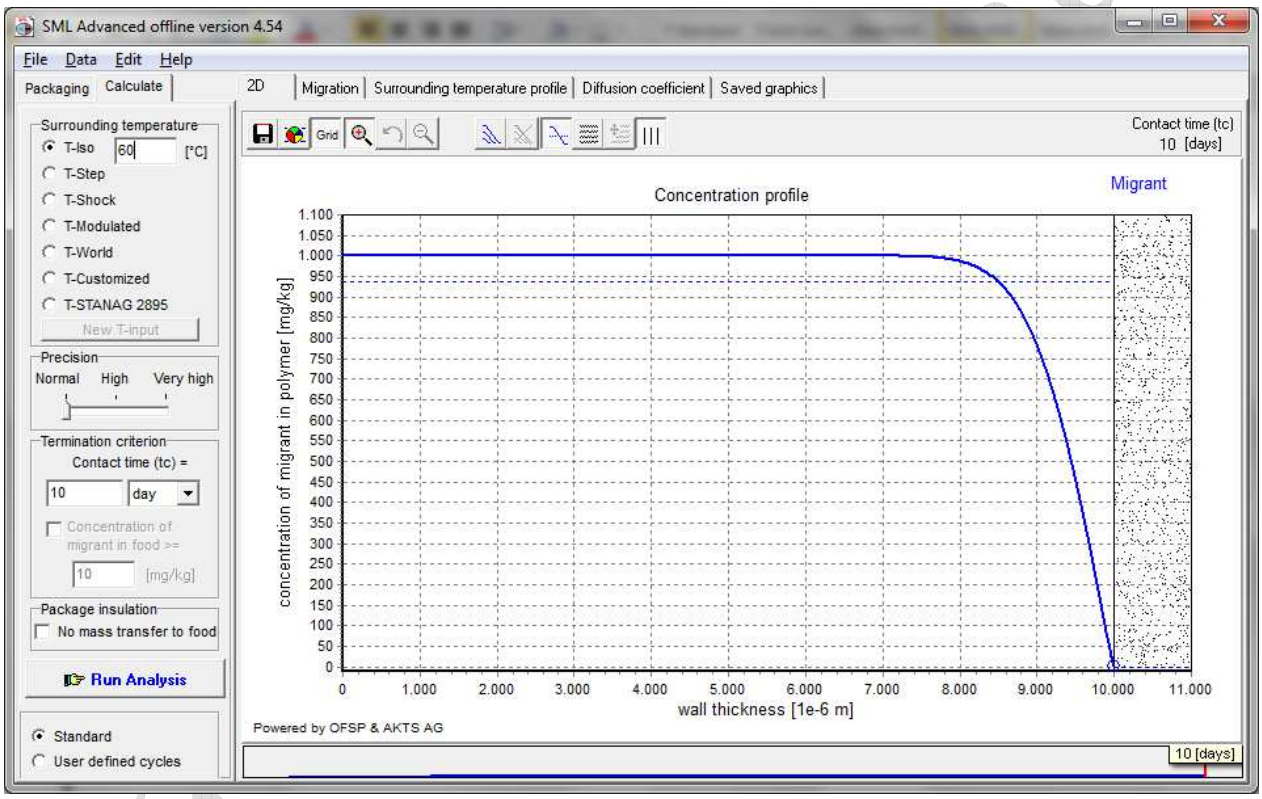
10d @ 60°C

=> 100% layer thickness = 3300 µm
no absolute barrier at thicknesses below 3300 µm

=> 99% layer thickness = 2100 µm

=> 1/2 x 99% layer thickness = 1050 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4200 µm
above 4200 µm two sides to be considered for calculation of migration if full immersion testing applied



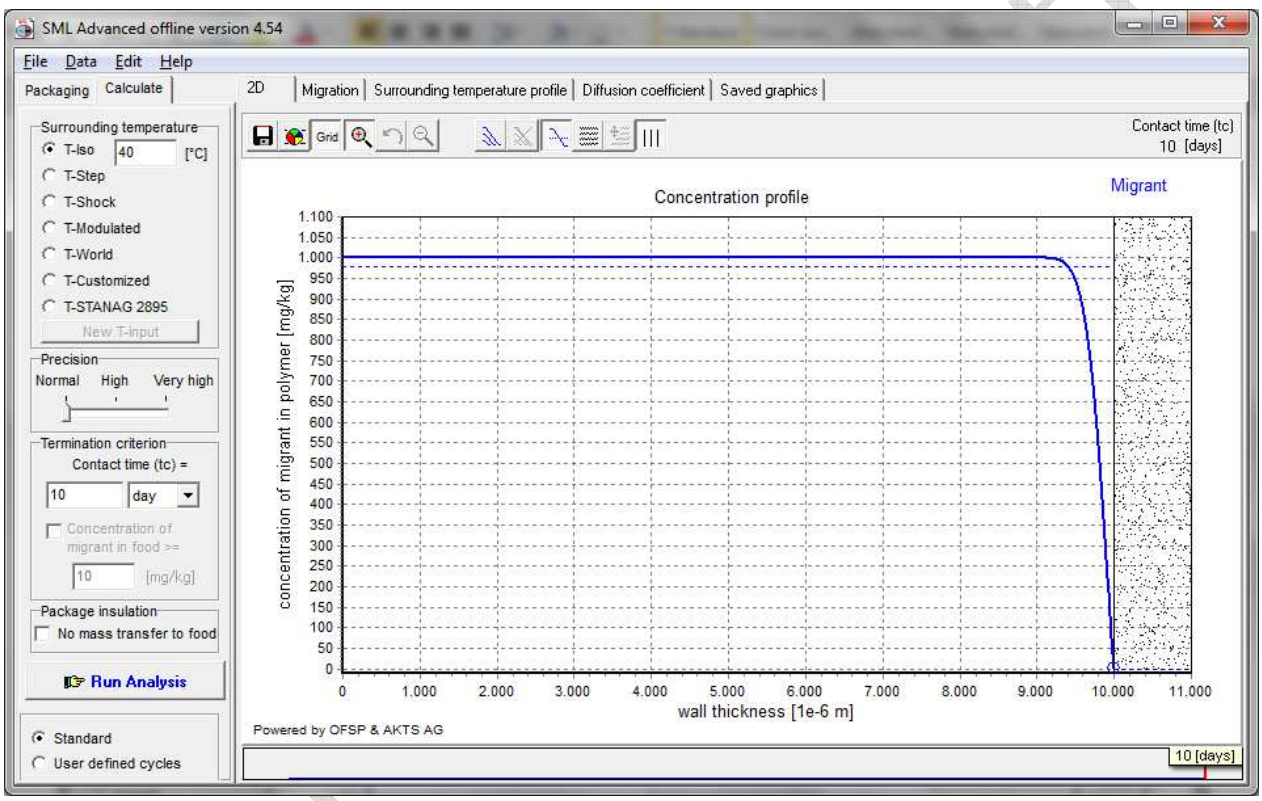
10d @ 40°C

=> 100% layer thickness = 960
no absolute barrier at thicknesses below 960 µm

=> 99% layer thickness = 660 µm

=> 1/2 x 99% layer thickness = 330 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1320
above 1320 µm two sides to be considered for calculation of migration if full immersion testing applied



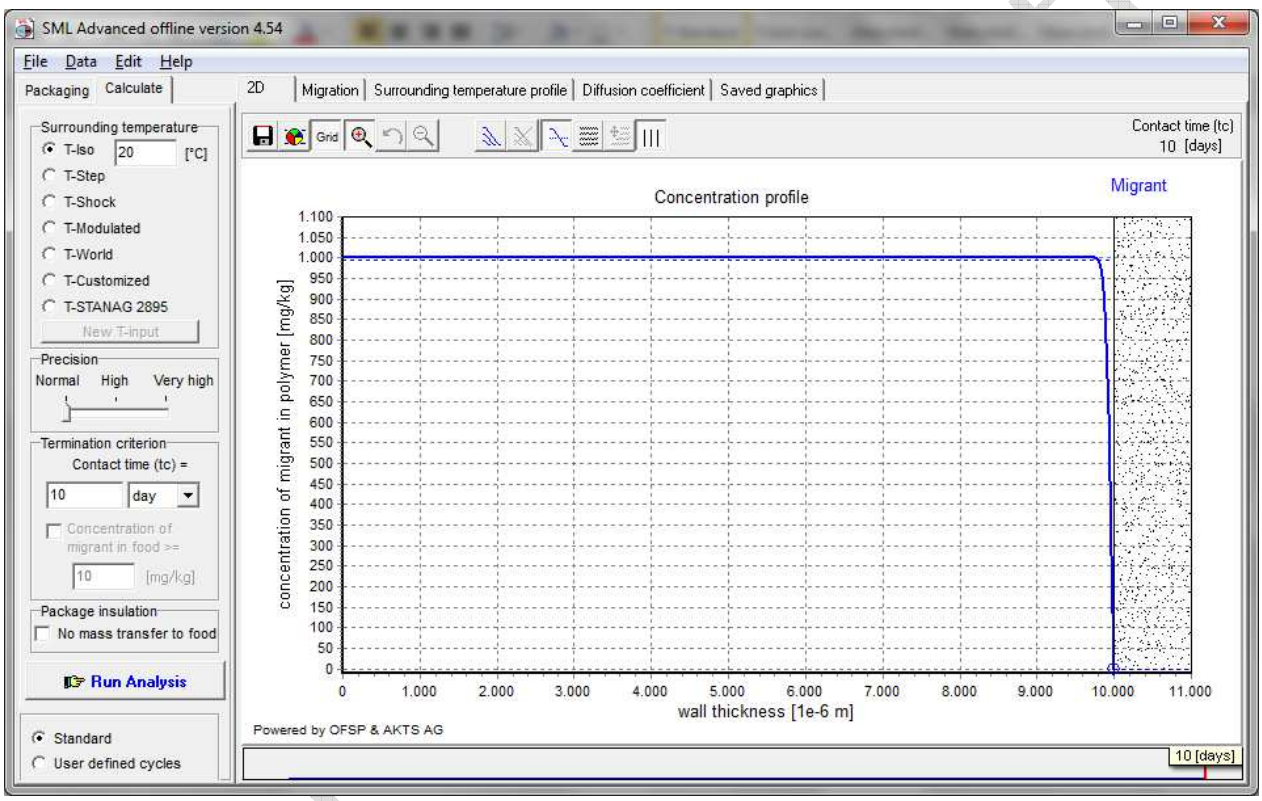
10d @ 20°C

=> 100% layer thickness = 280 µm
no absolute barrier at thicknesses below 280 µm

=> 99% layer thickness = 200 µm

=> 1/2 x 99% layer thickness = 100 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 400 µm
above 400 µm two sides to be considered for calculation of migration if full immersion testing applied



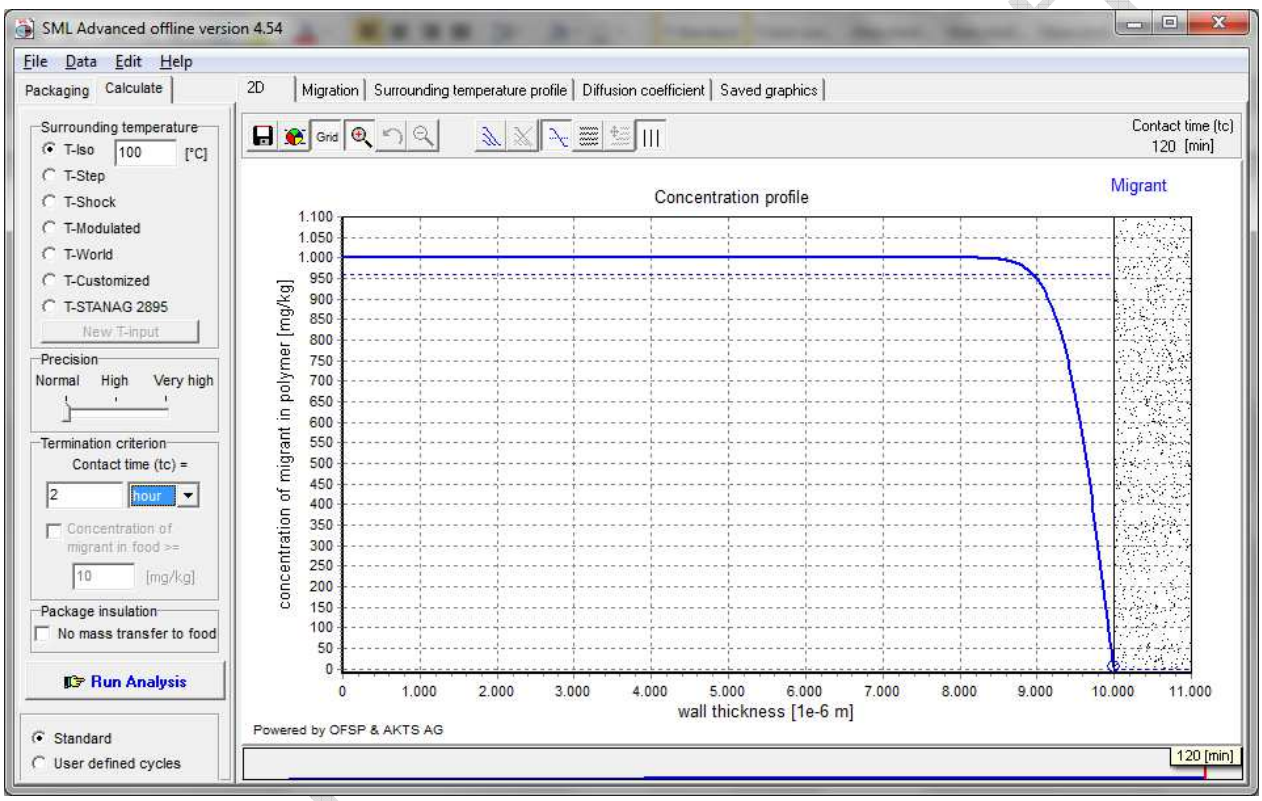
2h @ 100°C

=> 100% layer thickness = 1800 µm
no absolute barrier at thicknesses below 1800 µm

=> 99% layer thickness = 1320 µm

=> 1/2 x 99% layer thickness = 660 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2640 µm
above 2640 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 750 - 1000 g/mol**

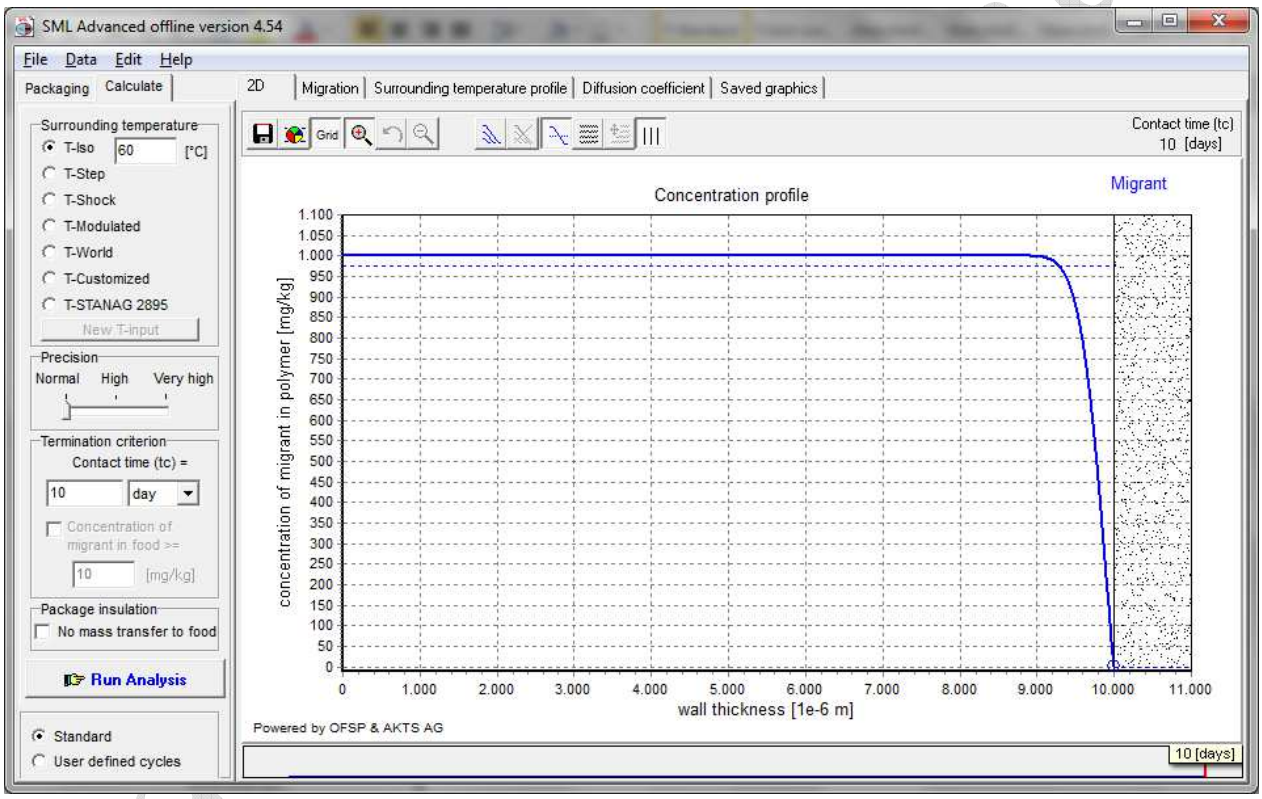
10d @ 60°C

=> 100% layer thickness = 1080 µm
no absolute barrier at thicknesses below 1080 µm

=> 99% layer thickness = 840 µm

=> 1/2 x 99% layer thickness = 420 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1680 µm
above 1680 µm two sides to be considered for calculation of migration if full immersion testing applied



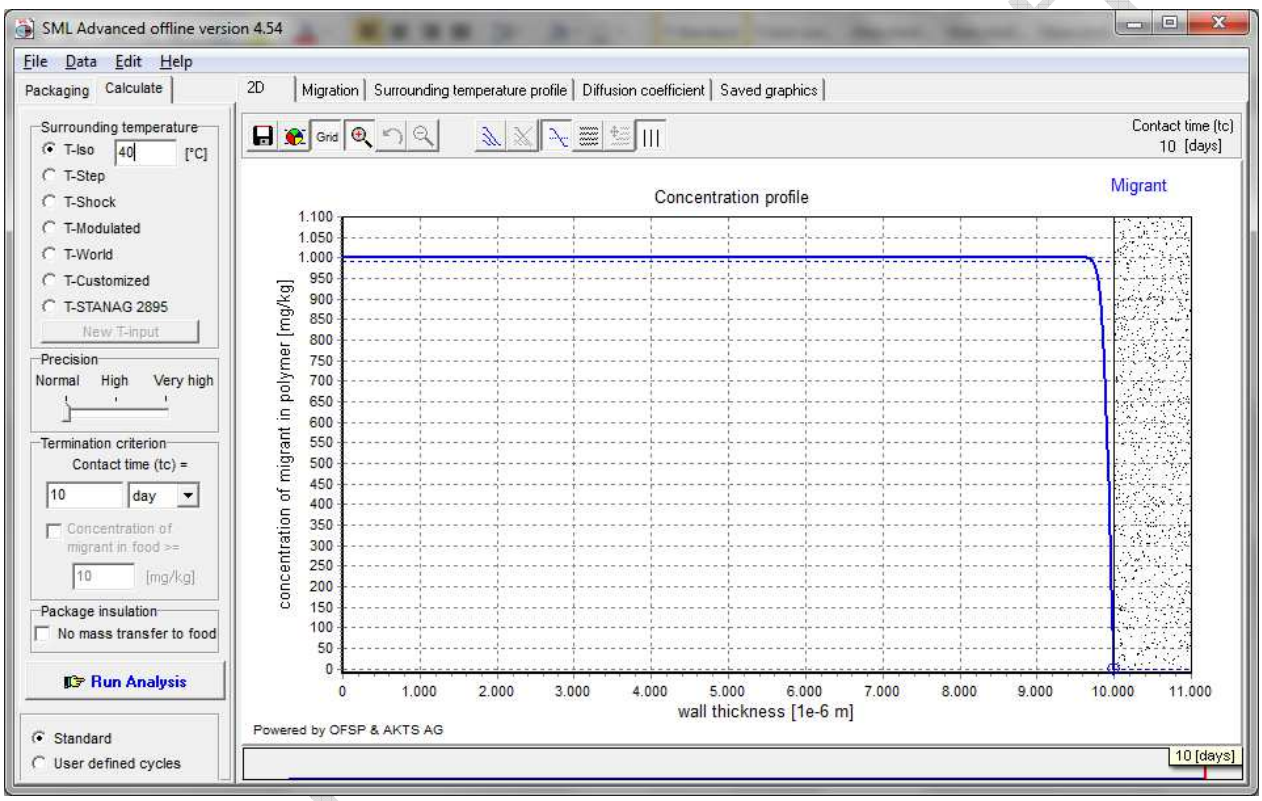
10d @ 40°C

=> 100% layer thickness = 400
no absolute barrier at thicknesses below 400 µm

=> 99% layer thickness = 270 µm

=> 1/2 x 99% layer thickness = 135 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 540
above 540 µm two sides to be considered for calculation of migration if full immersion testing applied



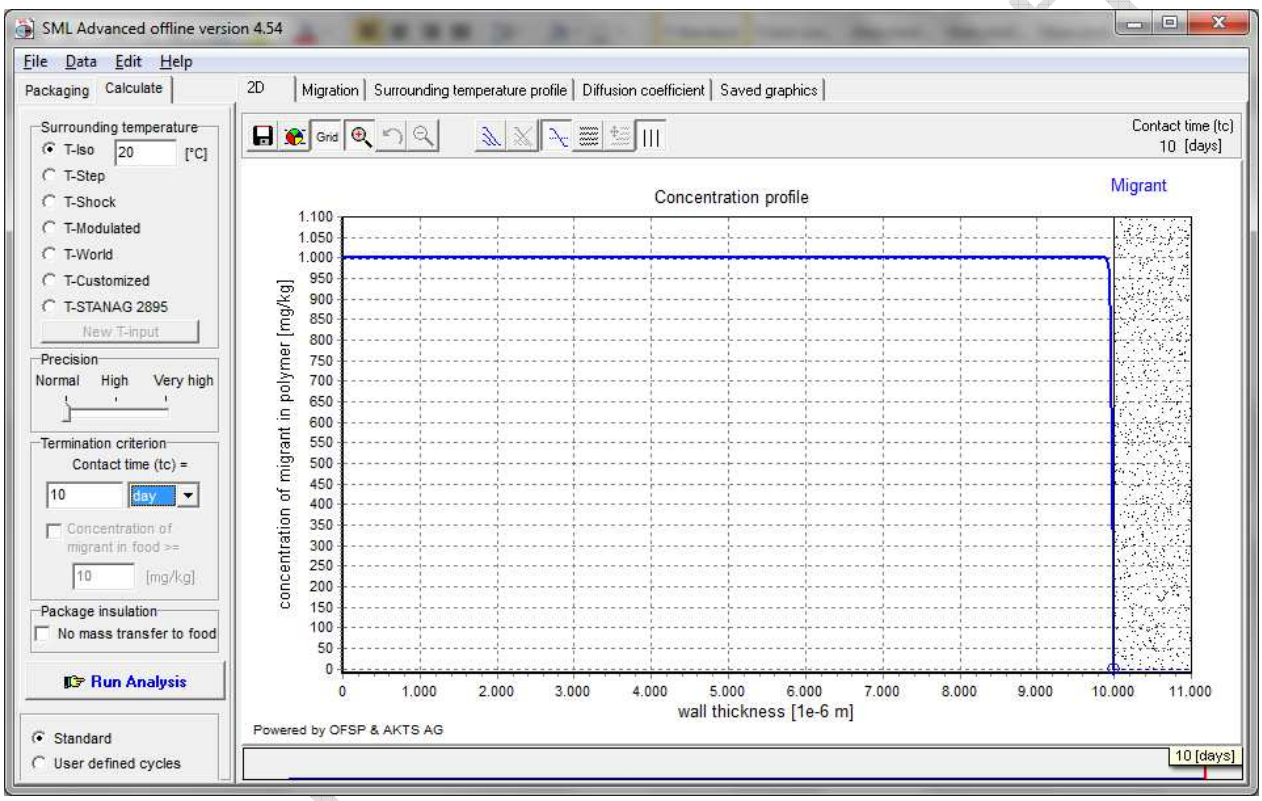
10d @ 20°C

=> 100% layer thickness = 130 µm
no absolute barrier at thicknesses below 130 µm

=> 99% layer thickness = 84 µm

=> 1/2 x 99% layer thickness = 42 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 168 µm
above 168 µm two sides to be considered for calculation of migration if full immersion testing applied



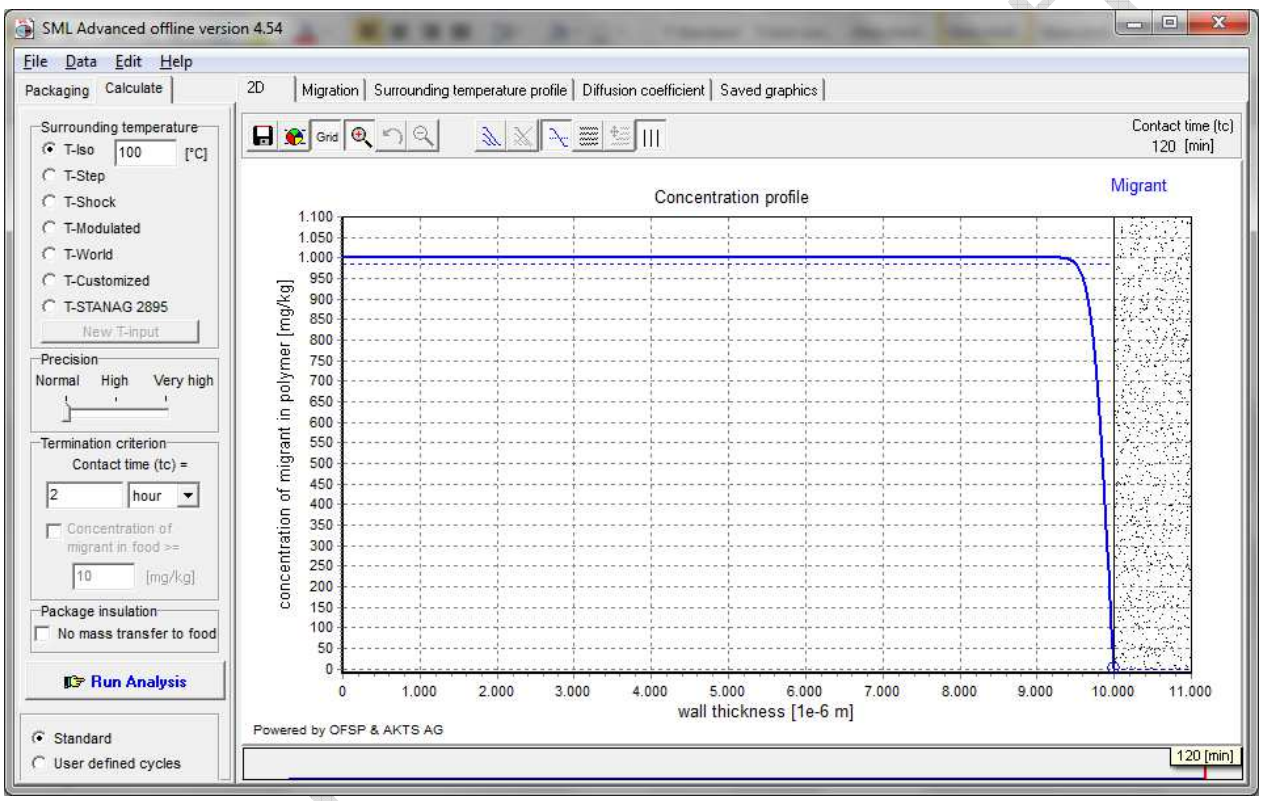
2h @ 100°C

=> 100% layer thickness = 700 µm
no absolute barrier at thicknesses below 700 µm

=> 99% layer thickness = 520 µm

=> 1/2 x 99% layer thickness = 260 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1040 µm
above 1040 µm two sides to be considered for calculation of migration if full immersion testing applied



PP, homo; PP random

► molecular mass 100 - 250 g/mol

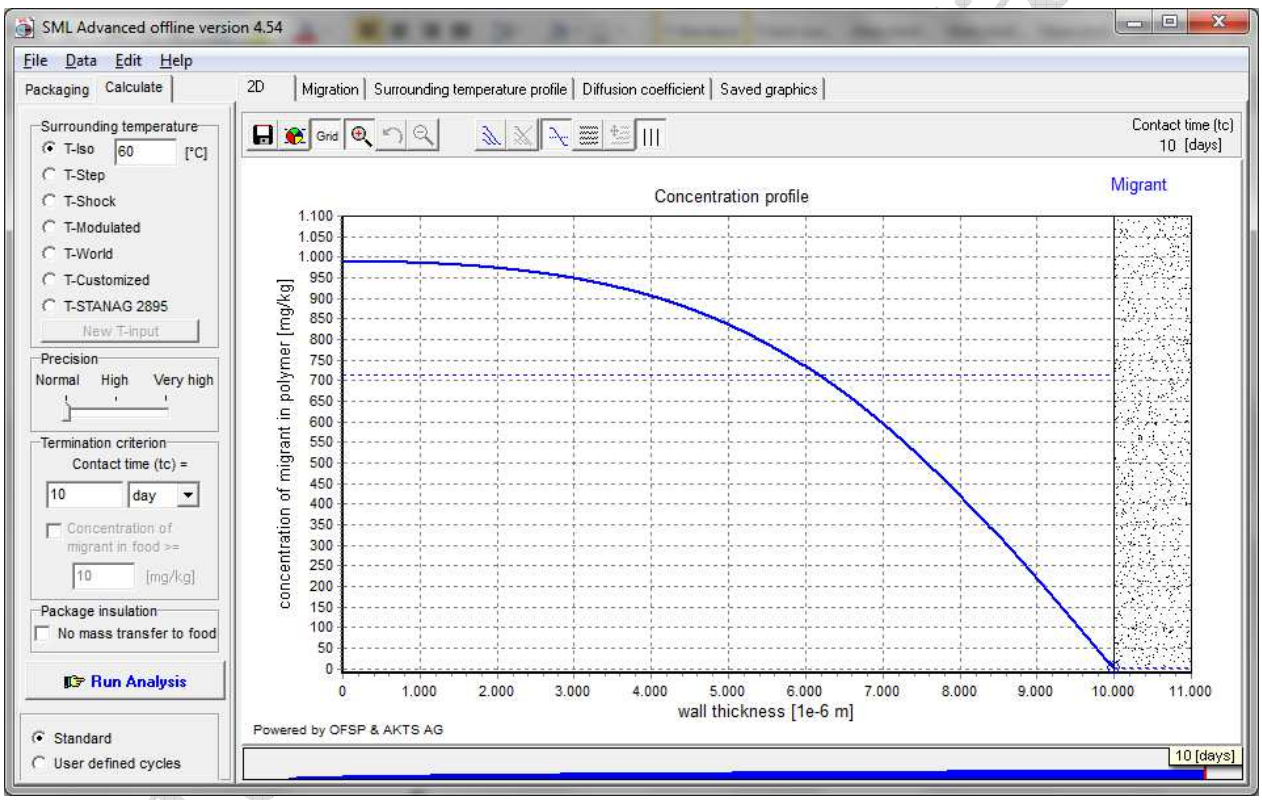
10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier

=> 99% layer thickness = 10000 µm

=> 1/2 x 99% layer thickness = 5000 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 20000 µm
above 20000 µm two sides to be considered for calculation of migration if full immersion testing applied



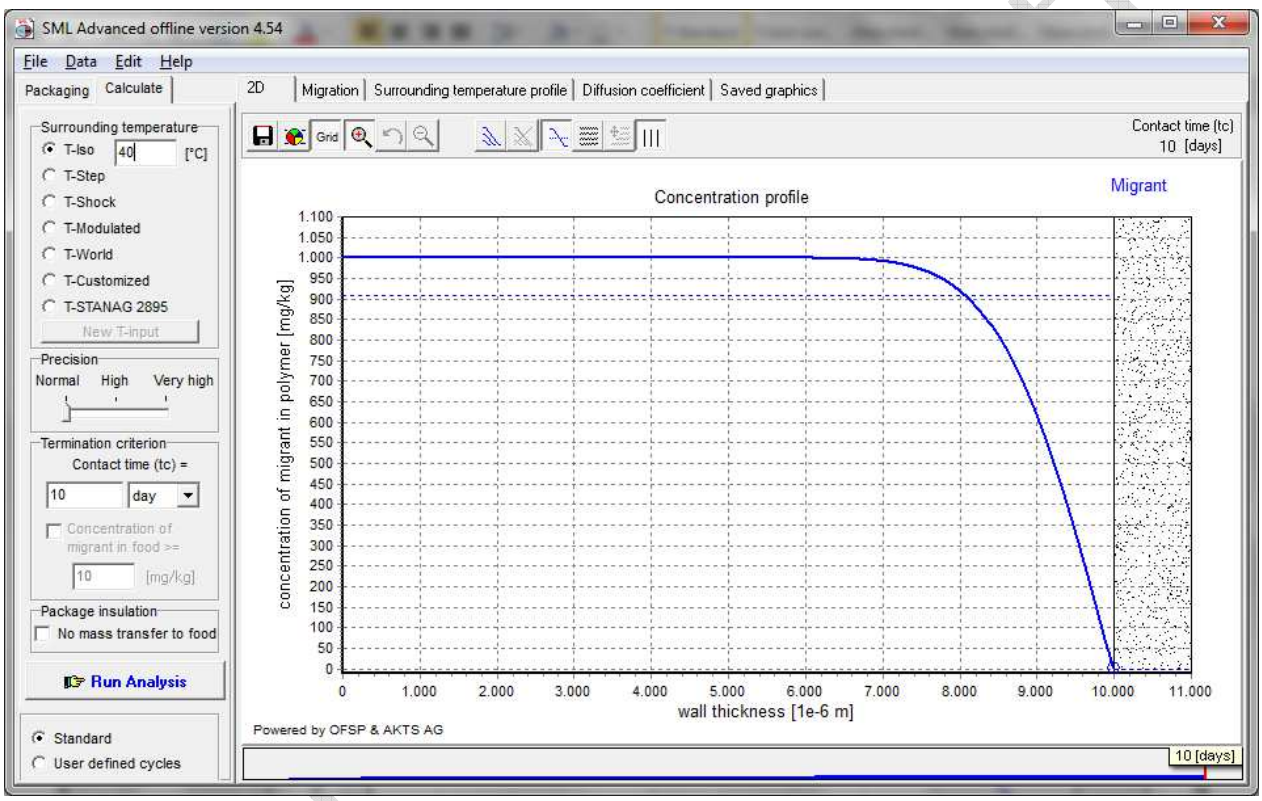
10d @ 40°C

=> 100% layer thickness = 3900
no absolute barrier at thicknesses below 3900 µm

=> 99% layer thickness = 2920 µm

=> 1/2 x 99% layer thickness = 1460 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 5840
above 5840 µm two sides to be considered for calculation of migration if full immersion testing applied



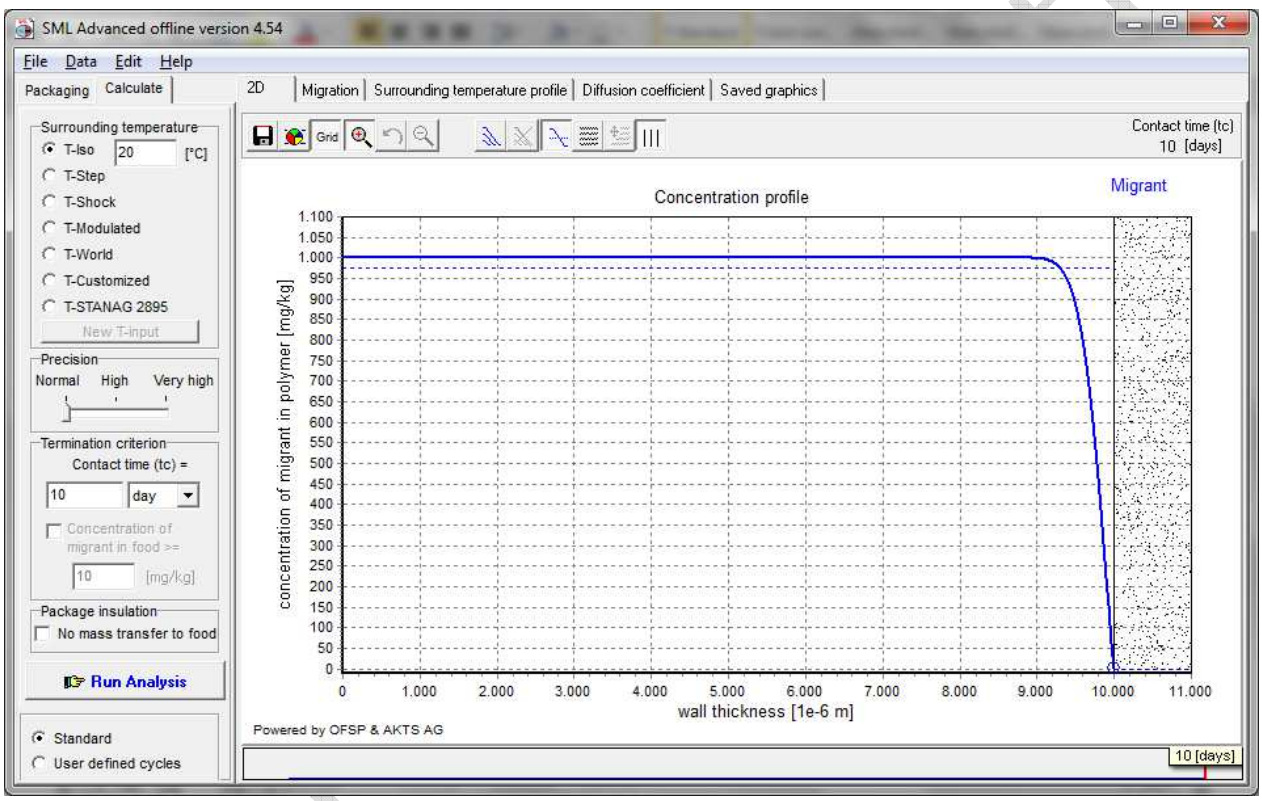
10d @ 20°C

=> 100% layer thickness = 1080 µm
no absolute barrier at thicknesses below 1080 µm

=> 99% layer thickness = 800 µm

=> 1/2 x 99% layer thickness = 400 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1600 µm
above 1600 µm two sides to be considered for calculation of migration if full immersion testing applied



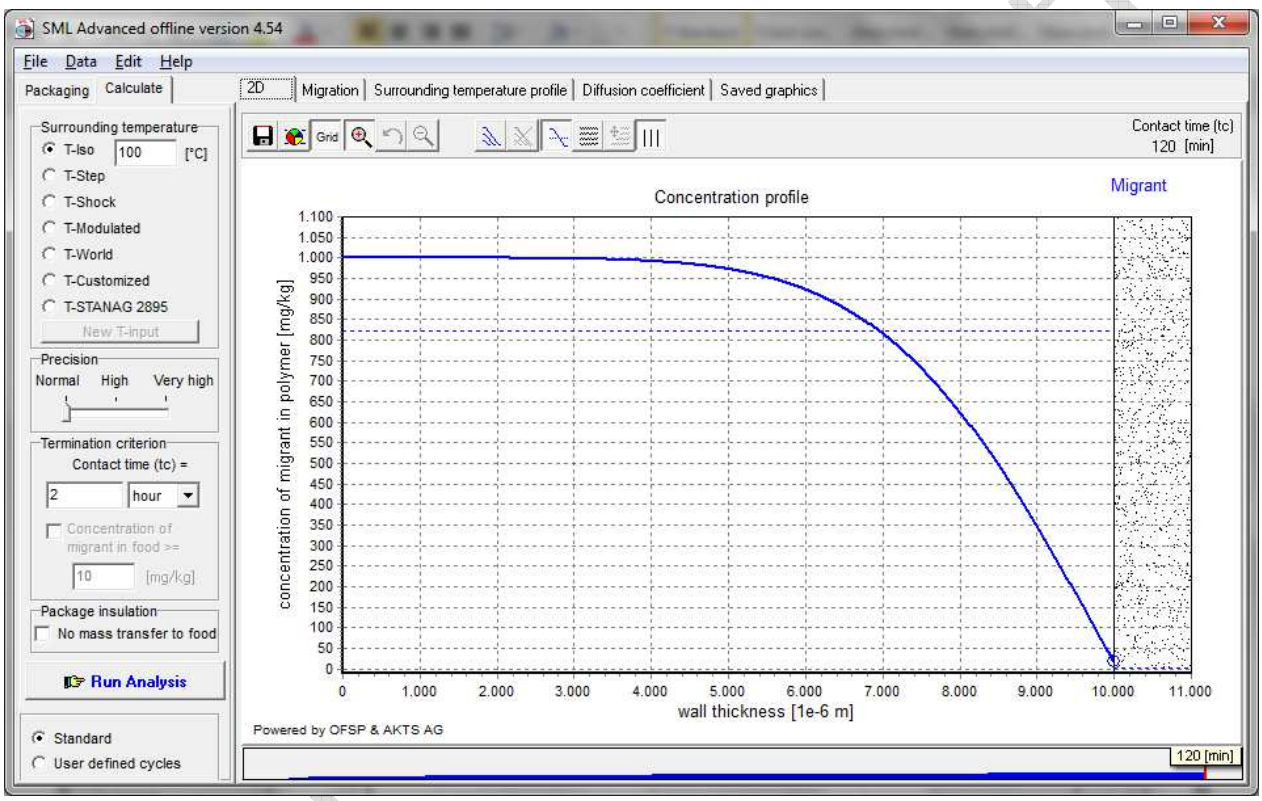
2h @ 100°C

=> 100% layer thickness = 8000 µm
no absolute barrier at thicknesses below 8000 µm

=> 99% layer thickness = 5850 µm

=> 1/2 x 99% layer thickness = 2925 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 11700 µm
above 11700 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

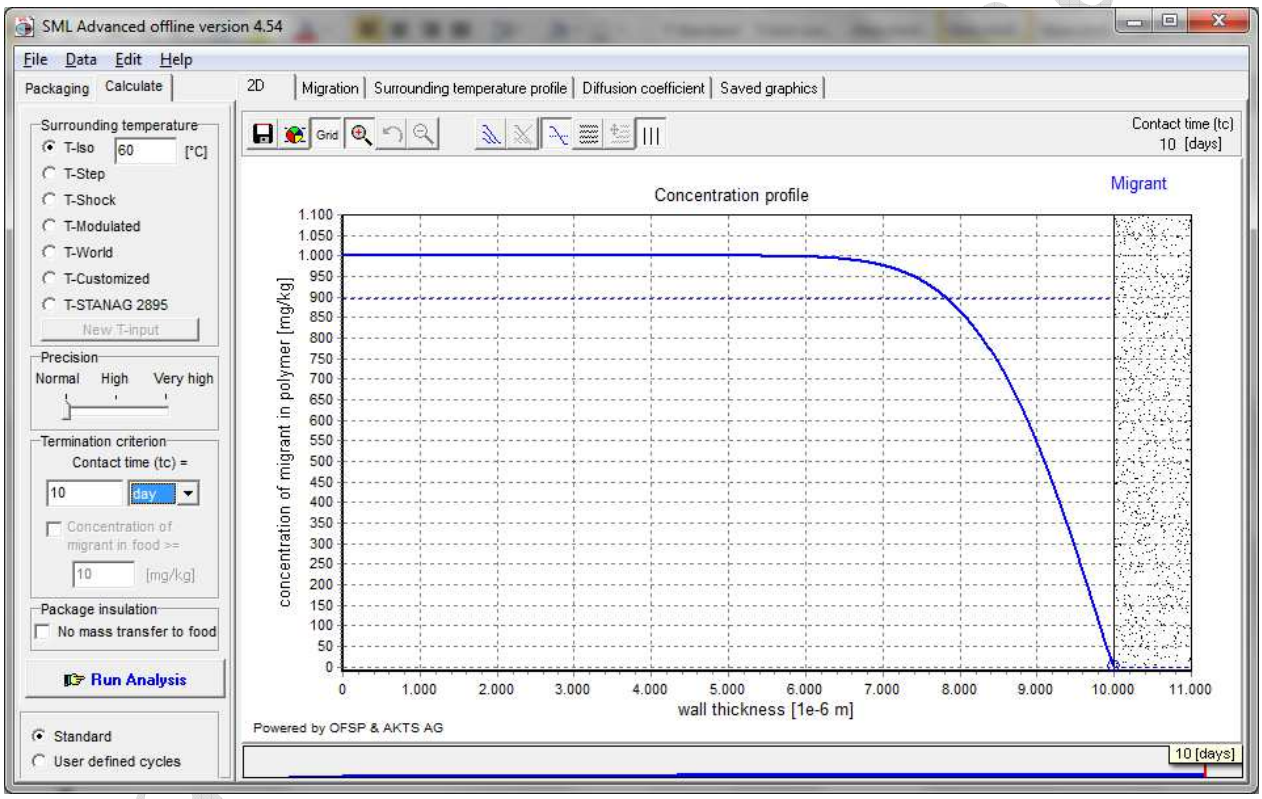
10d @ 60°C

=> 100% layer thickness = 4600 µm
no absolute barrier at thicknesses below 4600 µm

=> 99% layer thickness = 3400 µm

=> 1/2 x 99% layer thickness = 1700 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 6800 µm
above 6800 µm two sides to be considered for calculation of migration if full immersion testing applied



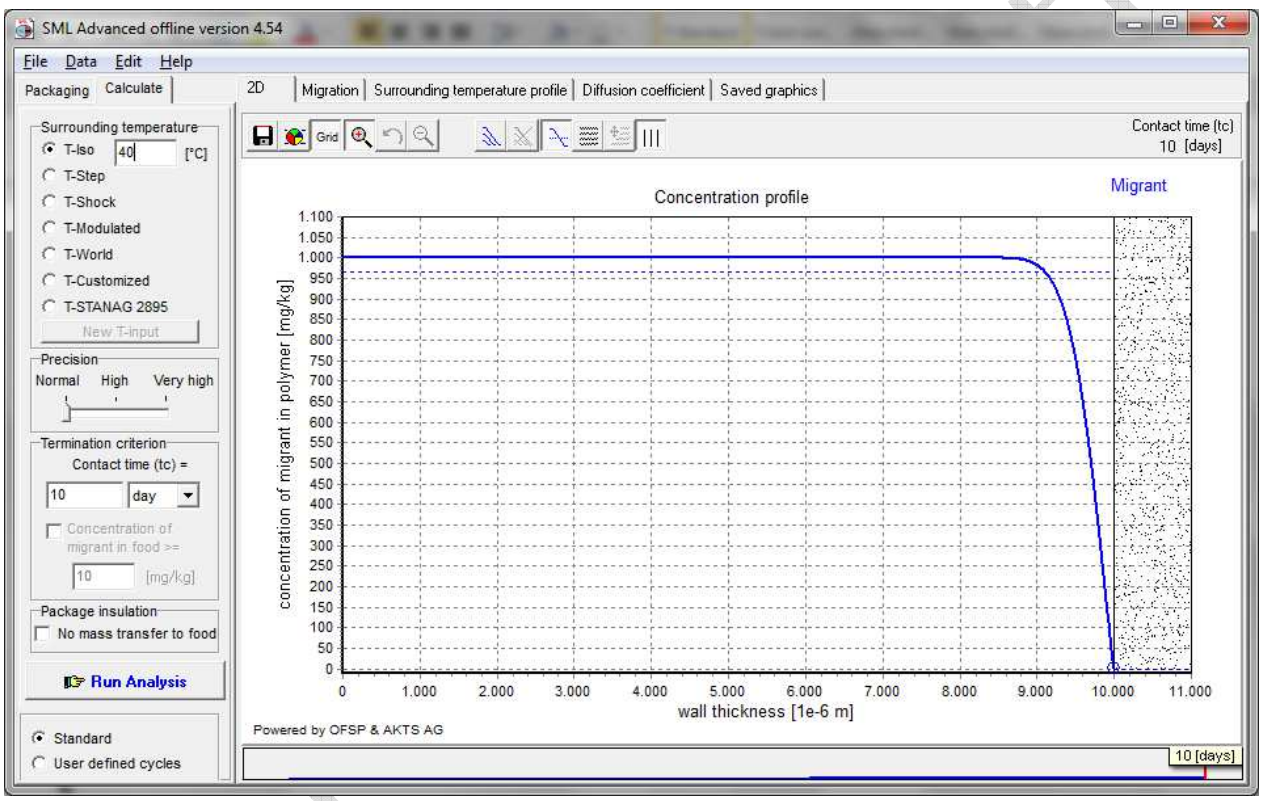
10d @ 40°C

=> 100% layer thickness = 1480 µm
no absolute barrier at thicknesses below 1480 µm

=> 99% layer thickness = 1100 µm

=> 1/2 x 99% layer thickness = 550 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2200 µm
above 2200 µm two sides to be considered for calculation of migration if full immersion testing applied



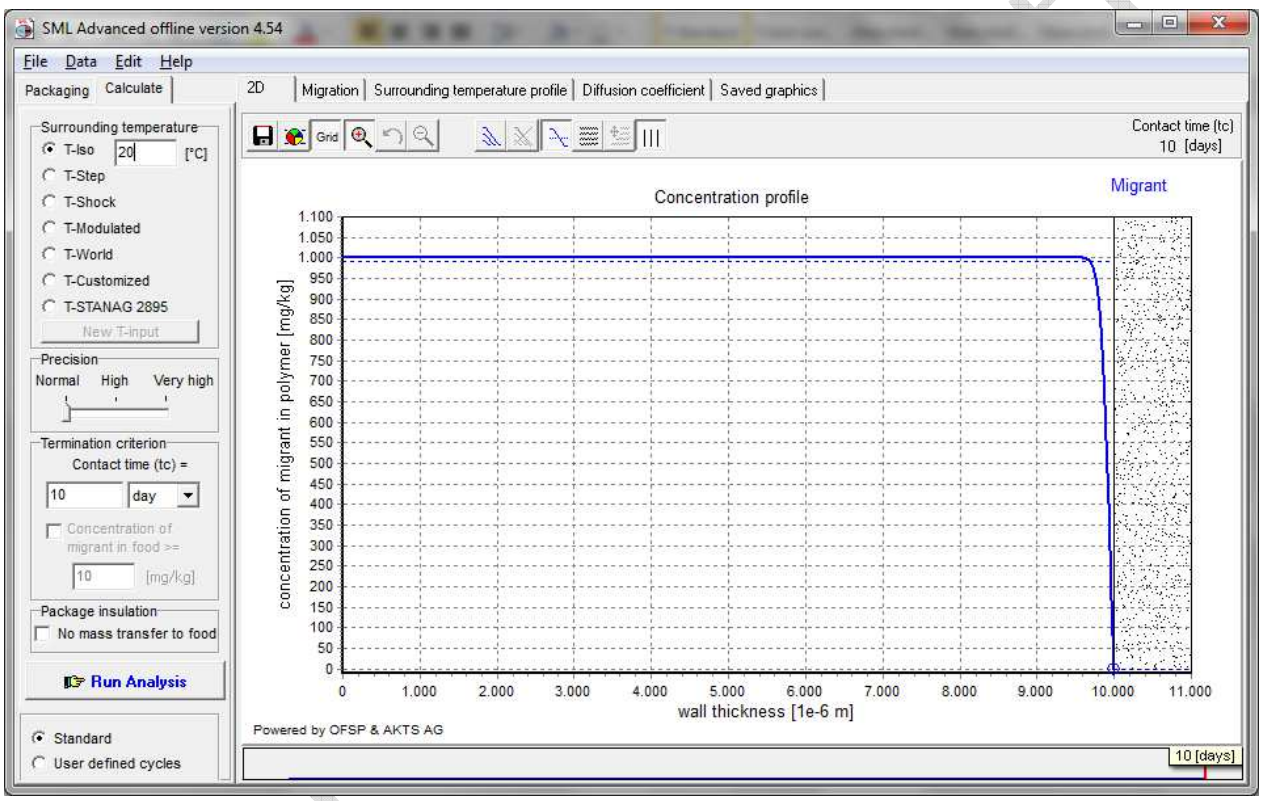
10d @ 20°C

=> 100% layer thickness = 440 µm
no absolute barrier at thicknesses below 440 µm

=> 99% layer thickness = 310 µm

=> 1/2 x 99% layer thickness = 155 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 620 µm
above 620 µm two sides to be considered for calculation of migration if full immersion testing applied



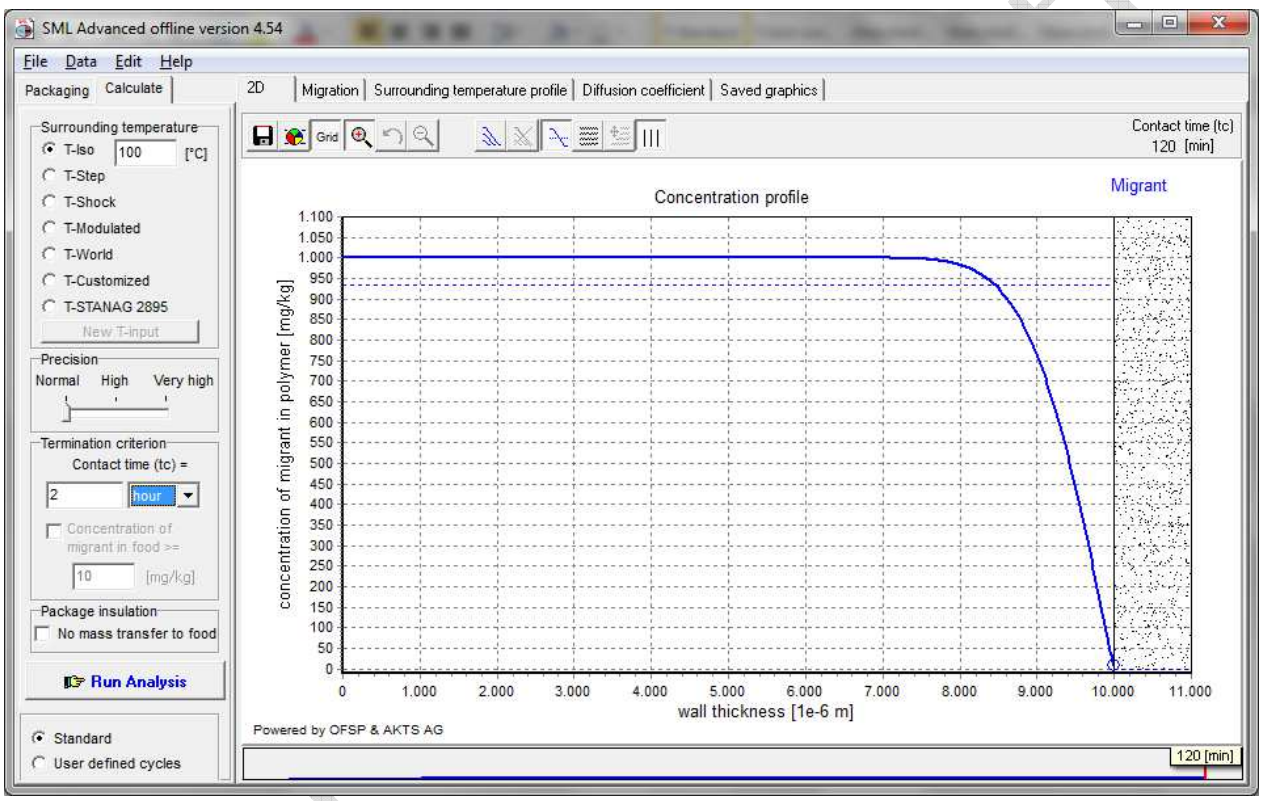
2h @ 100°C

=> 100% layer thickness = 3000 µm
no absolute barrier at thicknesses below 3000 µm

=> 99% layer thickness = 2160 µm

=> 1/2 x 99% layer thickness = 1080 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4320 µm
above 4320 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

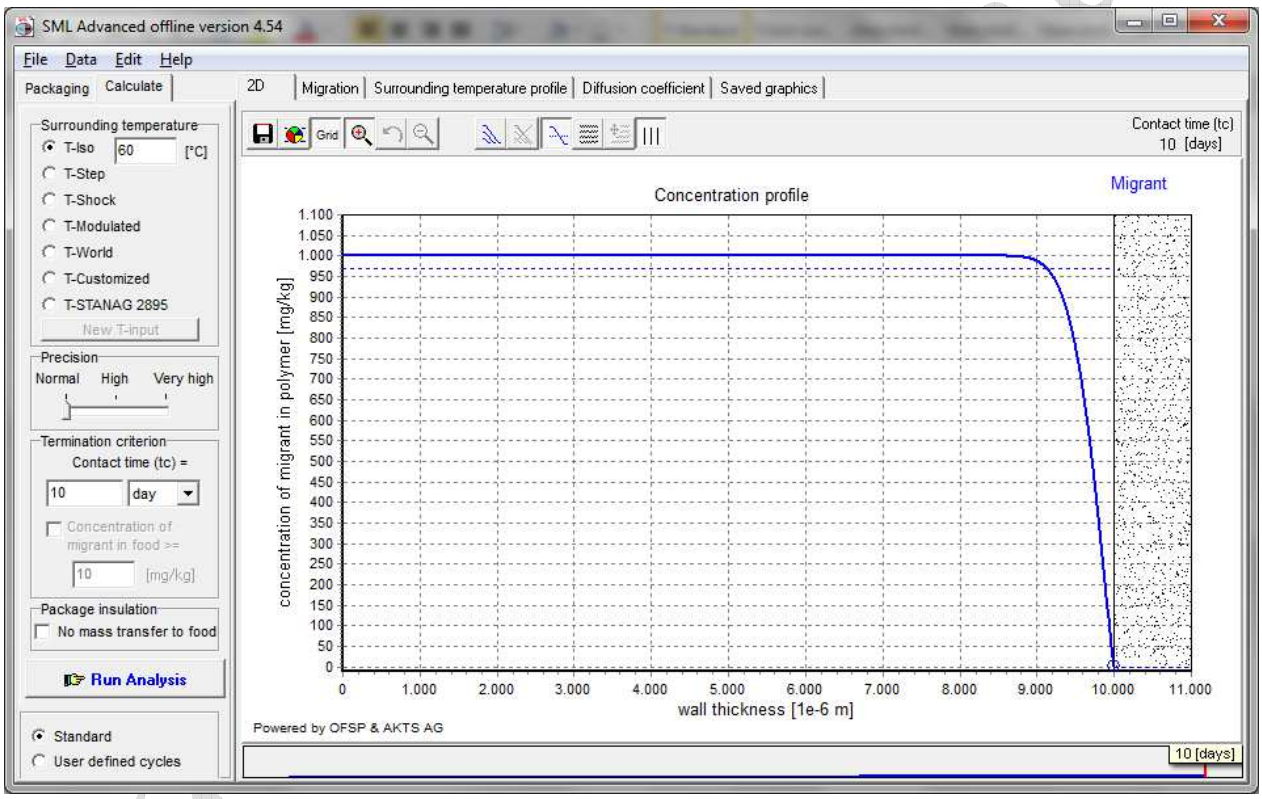
10d @ 60°C

=> 100% layer thickness = 1400 µm
no absolute barrier at thicknesses below 1400 µm

=> 99% layer thickness = 1040 µm

=> 1/2 x 99% layer thickness = 520 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2080 µm
above 2080 µm two sides to be considered for calculation of migration if full immersion testing applied



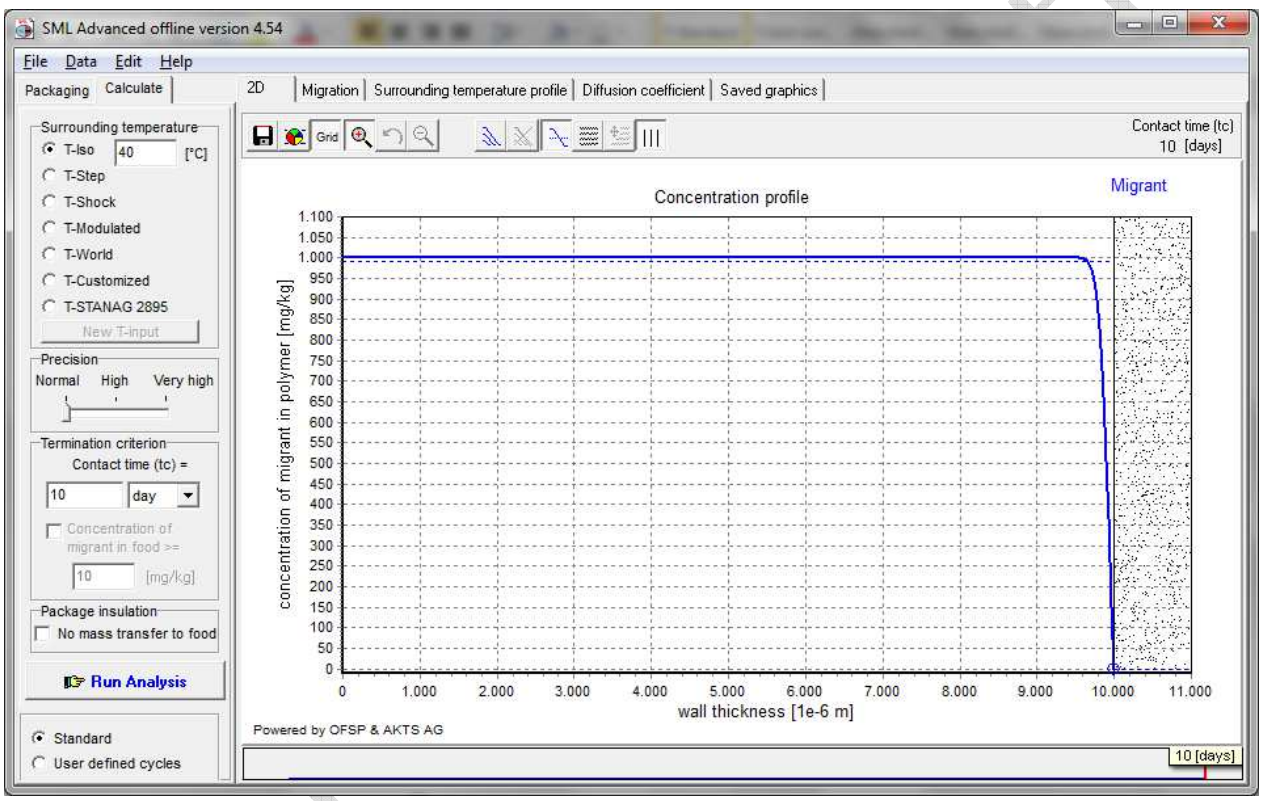
10d @ 40°C

=> 100% layer thickness = 500 µm
no absolute barrier at thicknesses below 500 µm

=> 99% layer thickness = 340 µm

=> 1/2 x 99% layer thickness = 170 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 680 µm
above 680 µm two sides to be considered for calculation of migration if full immersion testing applied



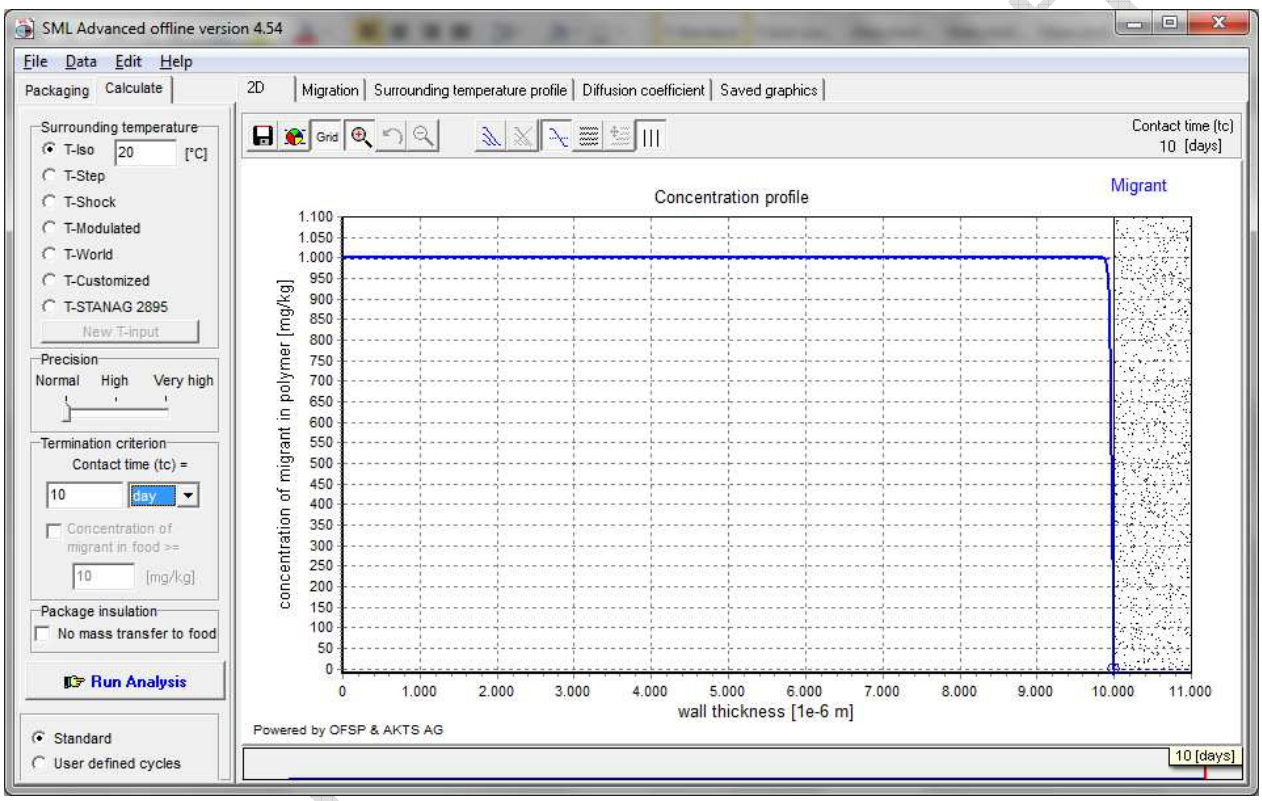
10d @ 20°C

=> 100% layer thickness = 160 µm
no absolute barrier at thicknesses below 160 µm

=> 99% layer thickness = 110 µm

=> 1/2 x 99% layer thickness = 55 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 220 µm
above 220 µm two sides to be considered for calculation of migration if full immersion testing applied



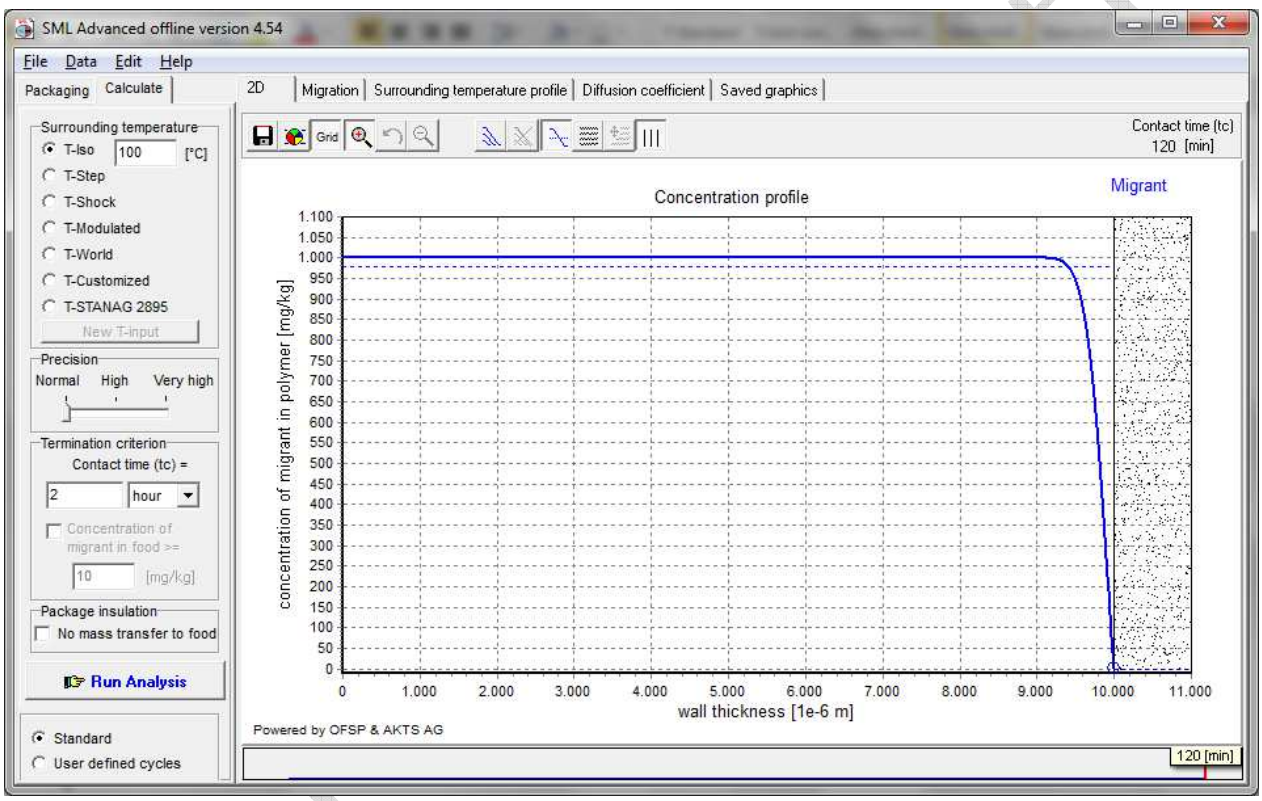
2h @ 100°C

=> 100% layer thickness = 900 µm
no absolute barrier at thicknesses below 900 µm

=> 99% layer thickness = 660 µm

=> 1/2 x 99% layer thickness = 330 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1320 µm
above 1320 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

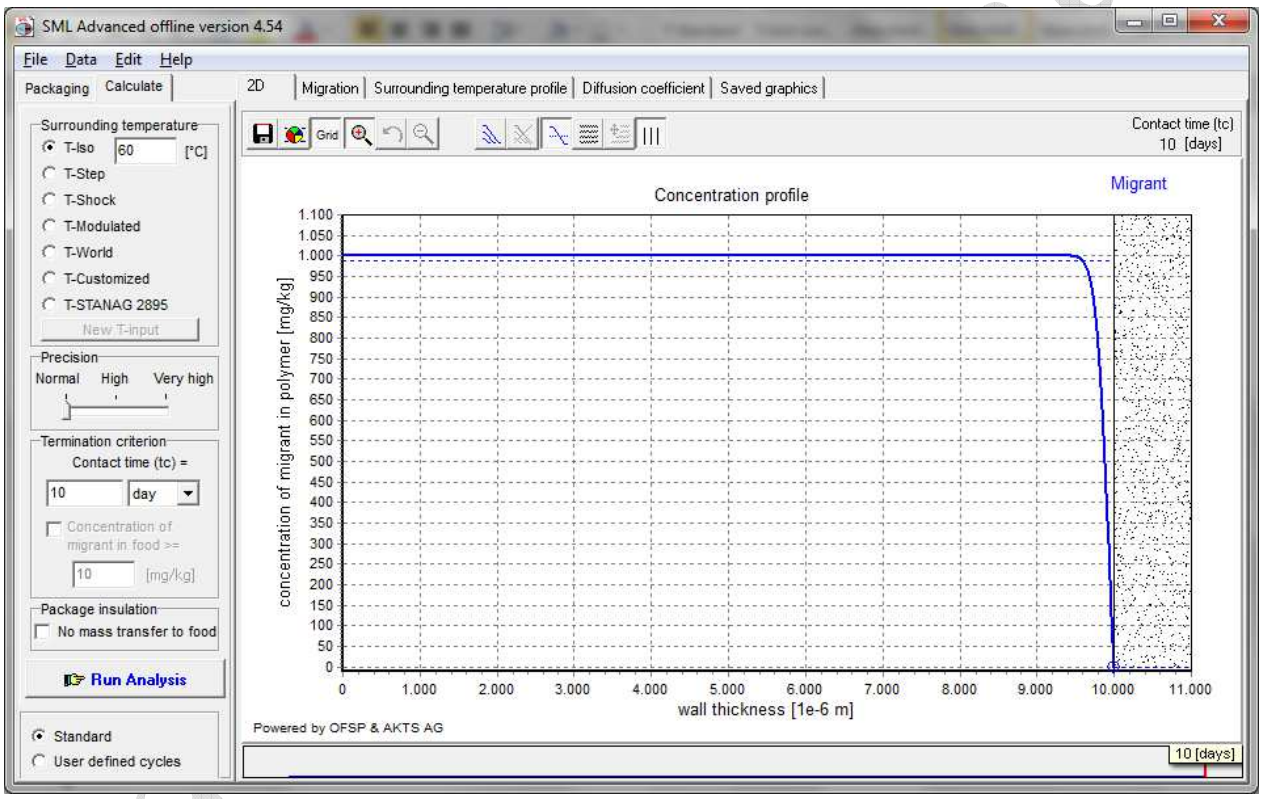
10d @ 60°C

=> 100% layer thickness = 580 µm
no absolute barrier at thicknesses below 580 µm

=> 99% layer thickness = 420 µm

=> 1/2 x 99% layer thickness = 210 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 840 µm
above 840 µm two sides to be considered for calculation of migration if full immersion testing applied



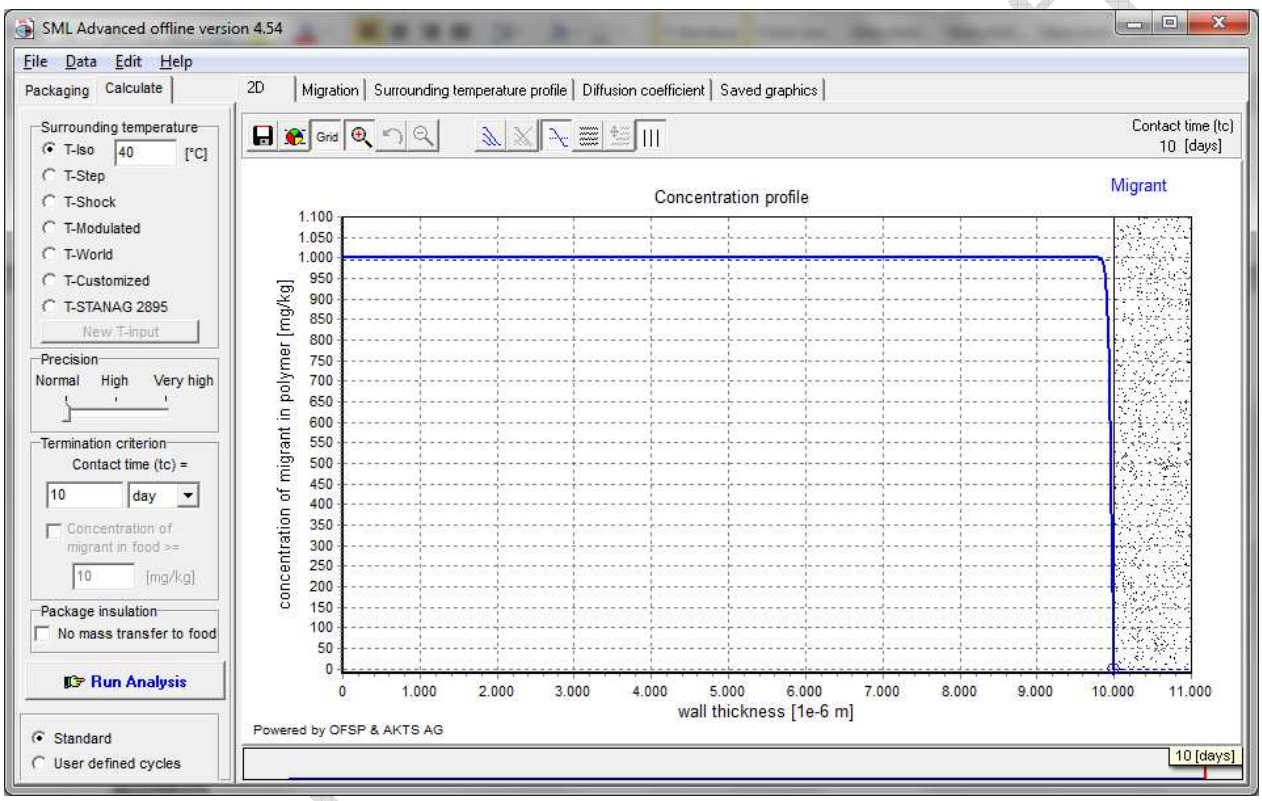
10d @ 40°C

=> 100% layer thickness = 220 µm
no absolute barrier at thicknesses below 220 µm

=> 99% layer thickness = 144 µm

=> 1/2 x 99% layer thickness = 72 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 288 µm
above 288 µm two sides to be considered for calculation of migration if full immersion testing applied



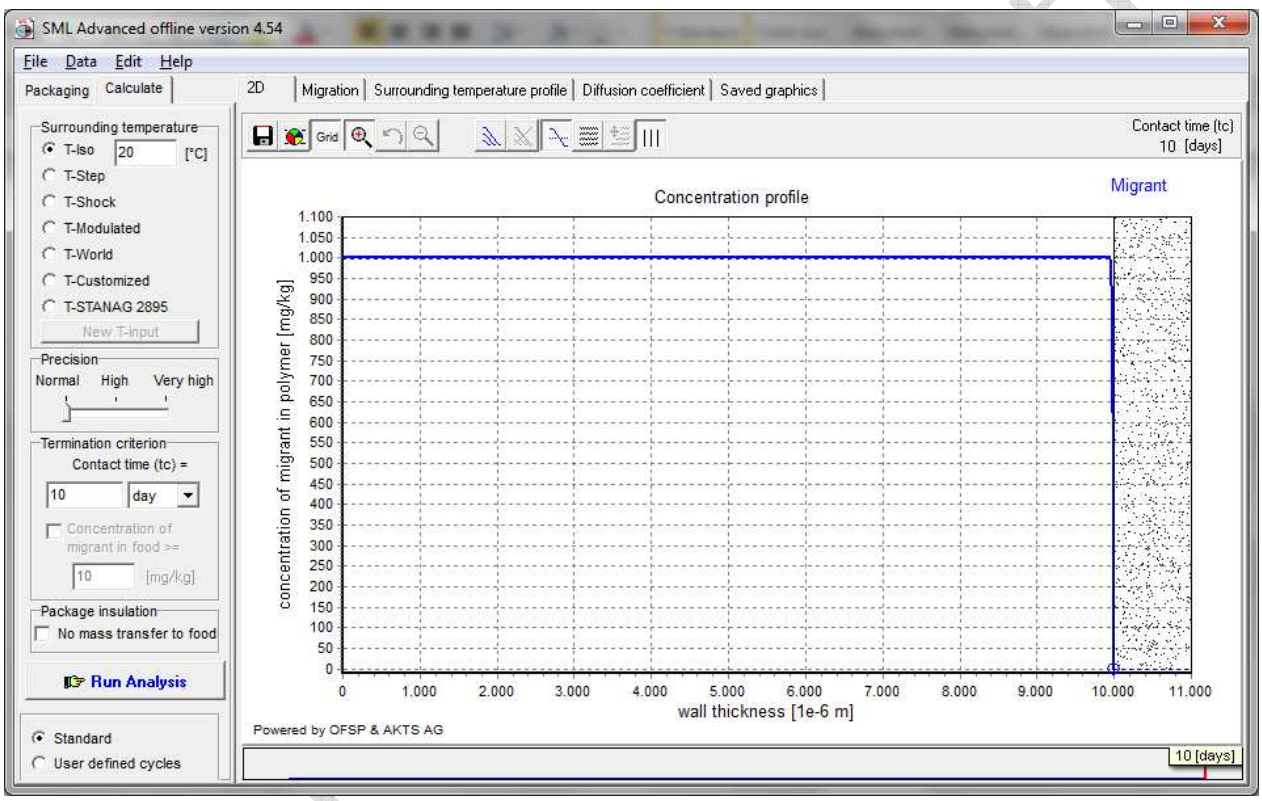
10d @ 20°C

=> 100% layer thickness = 70 µm
no absolute barrier at thicknesses below 70 µm

=> 99% layer thickness = 40 µm

=> 1/2 x 99% layer thickness = 20 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 80 µm
above 80 µm two sides to be considered for calculation of migration if full immersion testing applied



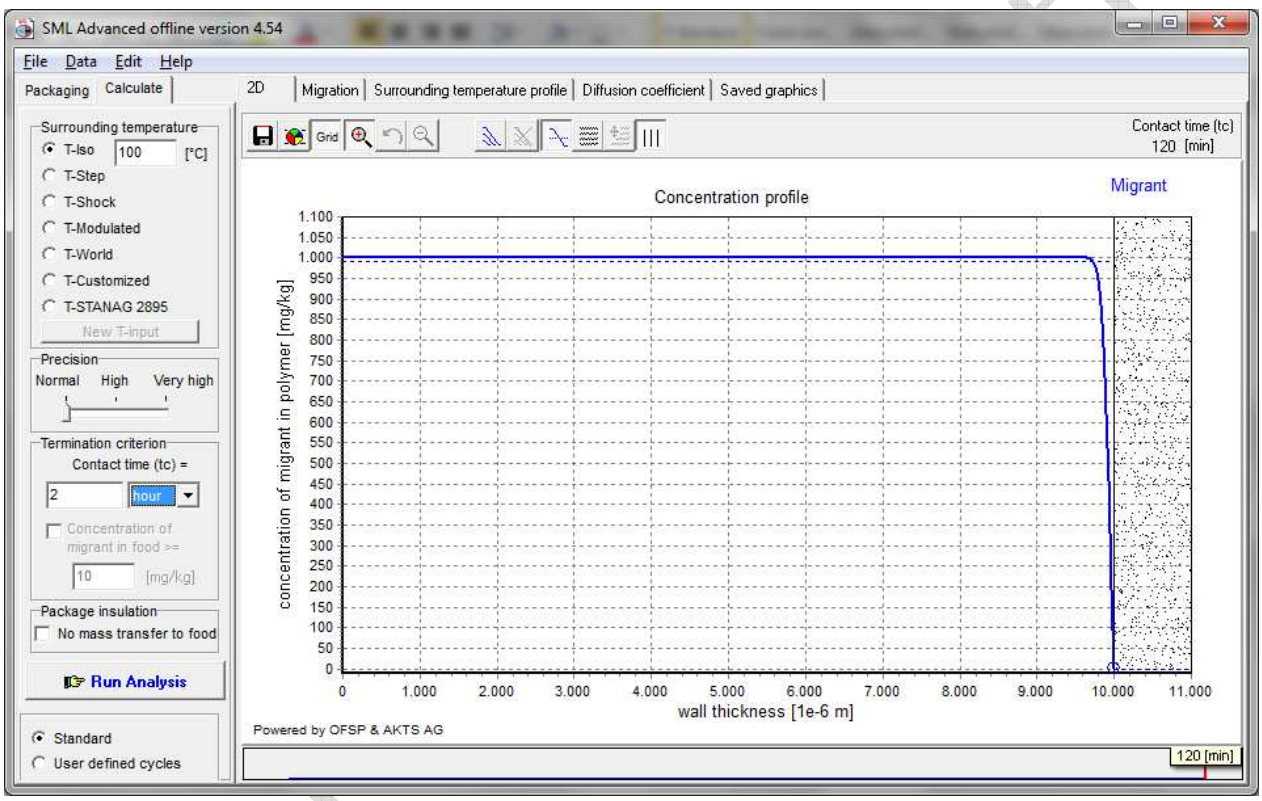
2h @ 100°C

=> 100% layer thickness = 380 µm
no absolute barrier at thicknesses below 380 µm

=> 99% layer thickness = 270 µm

=> 1/2 x 99% layer thickness = 135 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 540 µm
above 540 µm two sides to be considered for calculation of migration if full immersion testing applied



PET, PBT, PEN

► molecular mass 100 - 250 g/mol

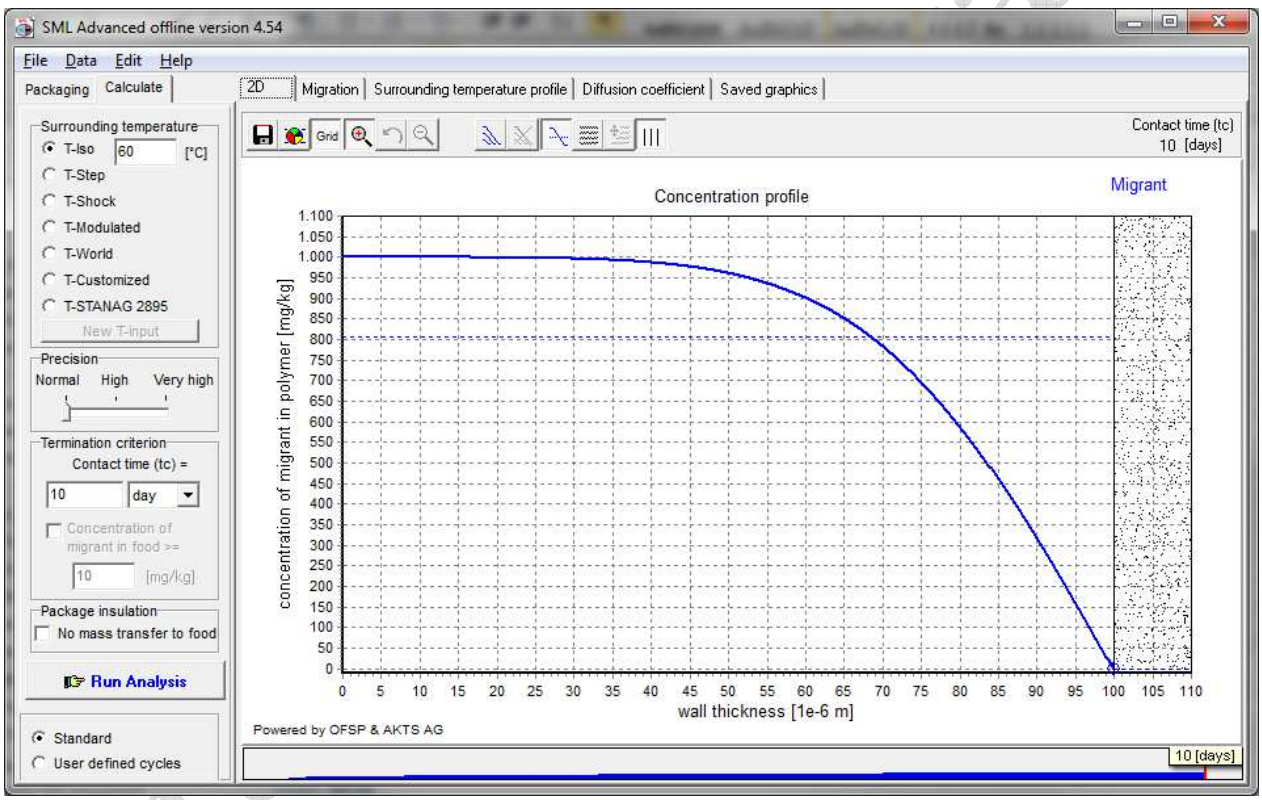
10d @ 60°C

=> 100% layer thickness = 91
no absolute barrier at thicknesses below 91 µm

=> 99% layer thickness = 80 µm

=> 1/2 x 99% layer thickness = 40 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 160 µm
above 160 µm two sides to be considered for calculation of migration if full immersion testing applied



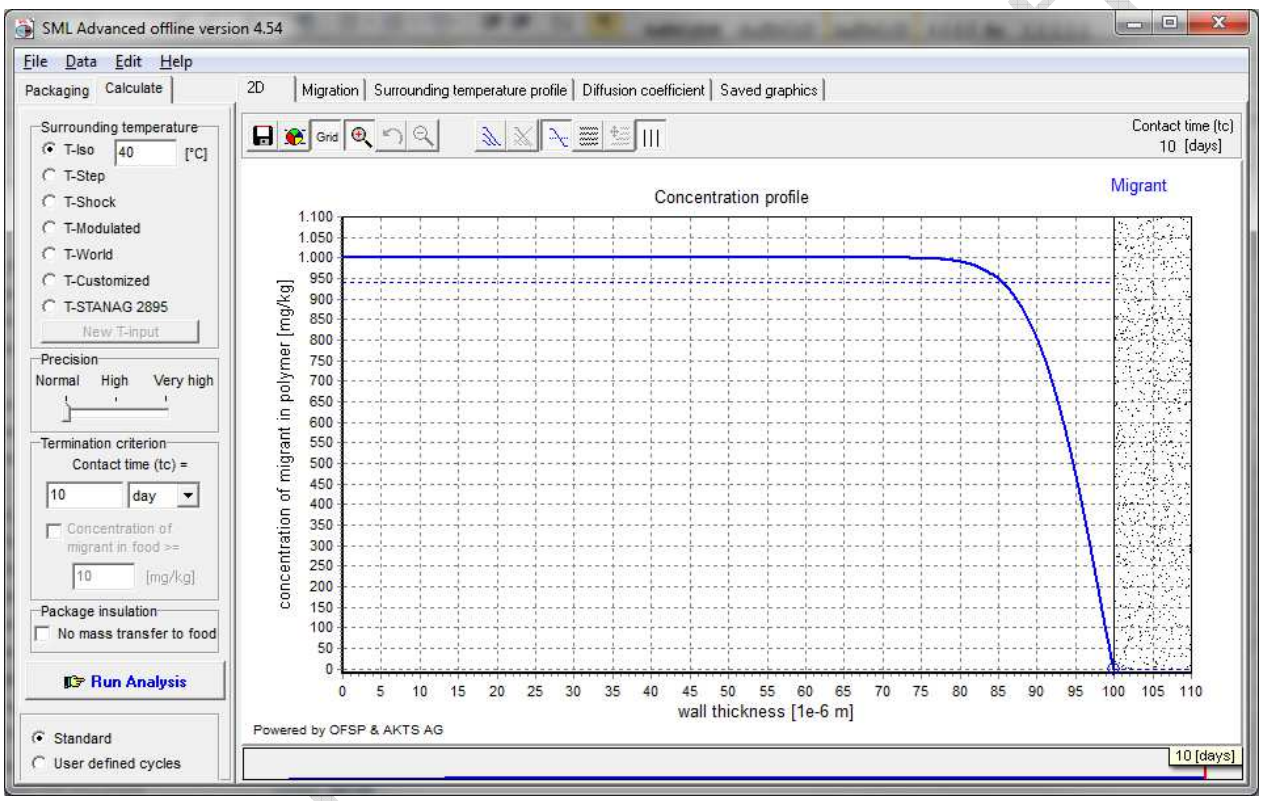
10d @ 40°C

=> 100% layer thickness = 31
no absolute barrier at thicknesses below 31 µm

=> 99% layer thickness = 26 µm

=> 1/2 x 99% layer thickness = 13 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 52
above 52 µm two sides to be considered for calculation of migration if full immersion testing applied



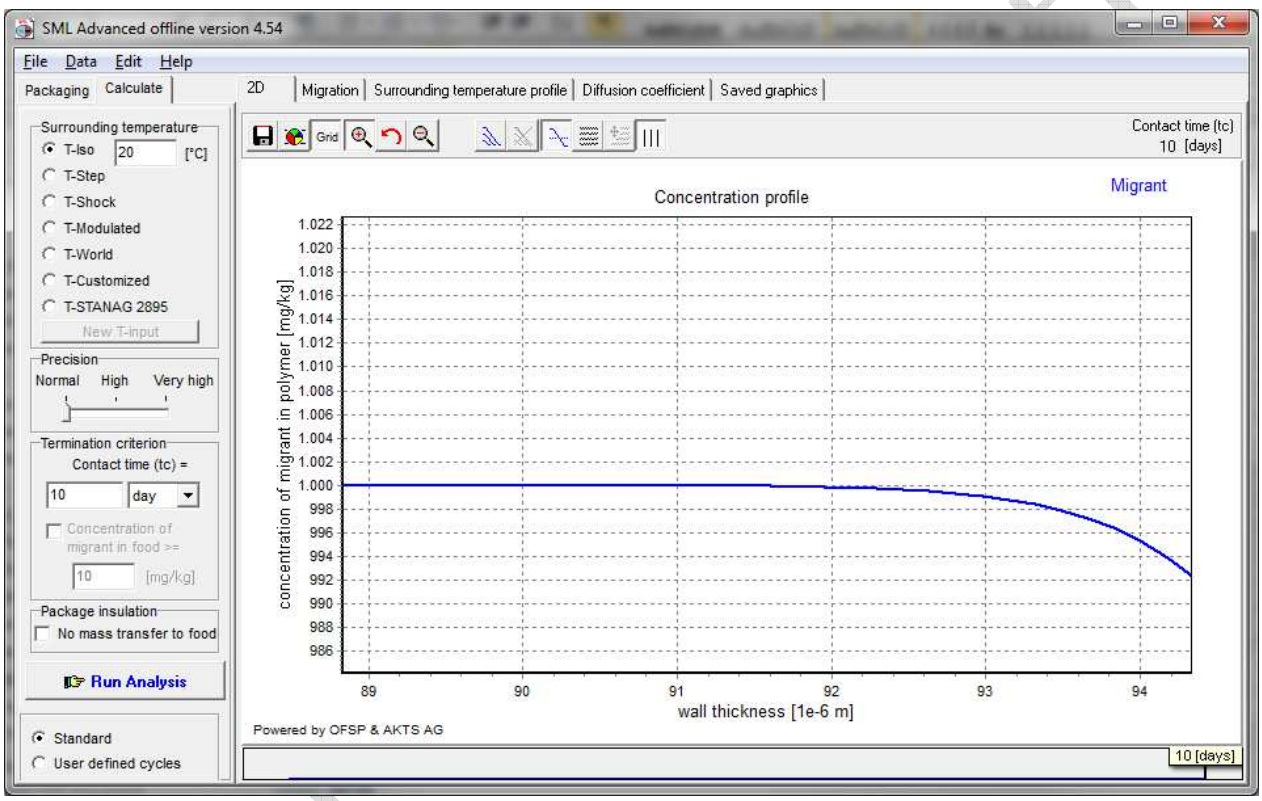
10d @ 20°C

=> 100% layer thickness = 9 µm
no absolute barrier at thicknesses below 9 µm

=> 99% layer thickness = 7 µm

=> 1/2 x 99% layer thickness = 4 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 14 µm
above 14 µm two sides to be considered for calculation of migration if full immersion testing applied



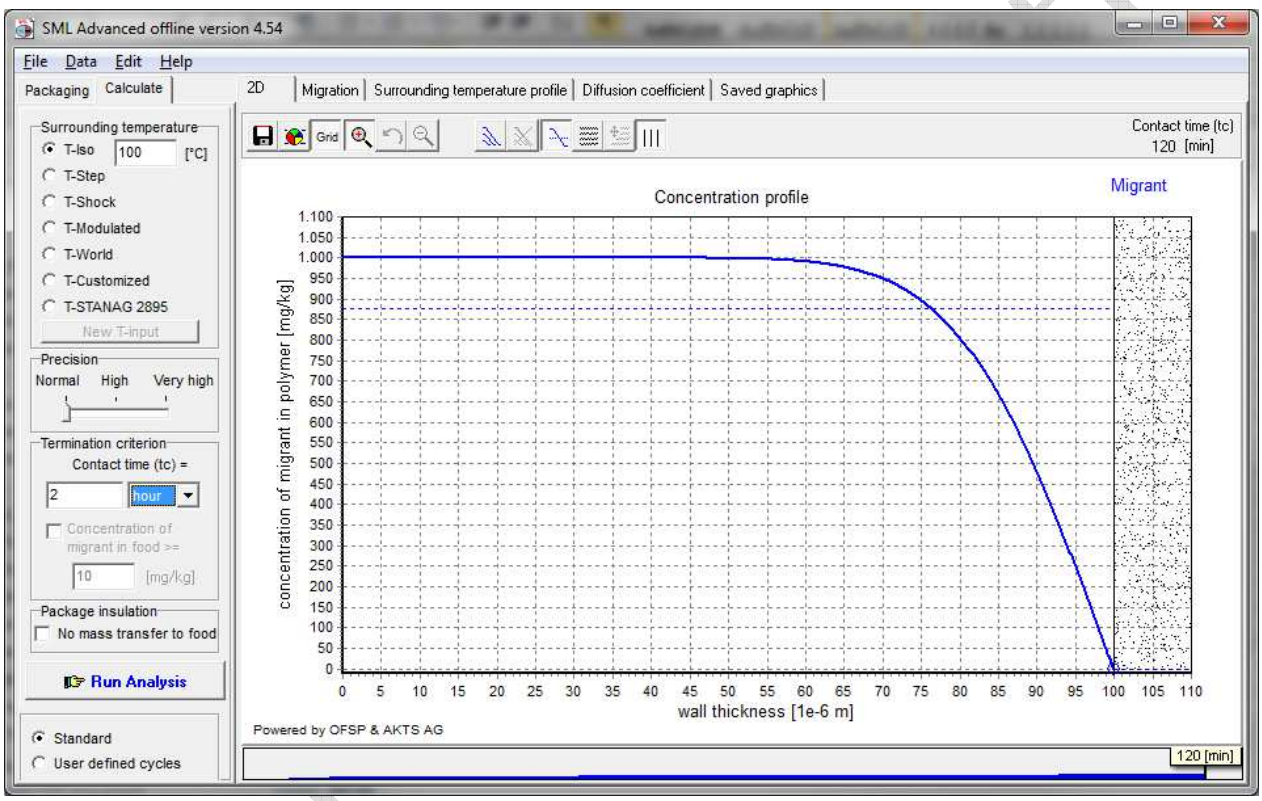
2h @ 100°C

=> 100% layer thickness = 61 µm
no absolute barrier at thicknesses below 61 µm

=> 99% layer thickness = 50 µm

=> 1/2 x 99% layer thickness = 25 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 100 µm
above 100 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

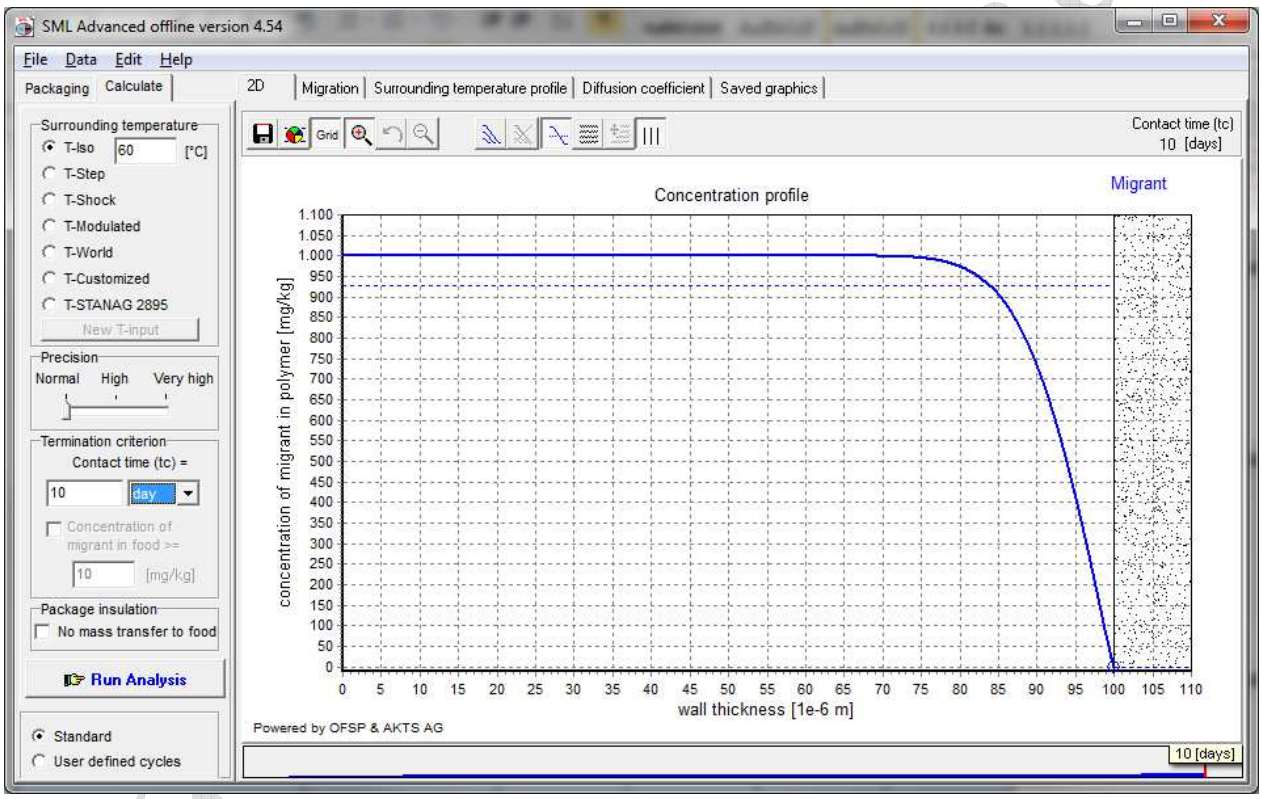
10d @ 60°C

=> 100% layer thickness = 35 µm
no absolute barrier at thicknesses below 35 µm

=> 99% layer thickness = 29 µm

=> 1/2 x 99% layer thickness = 15 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 58 µm
above 58 µm two sides to be considered for calculation of migration if full immersion testing applied



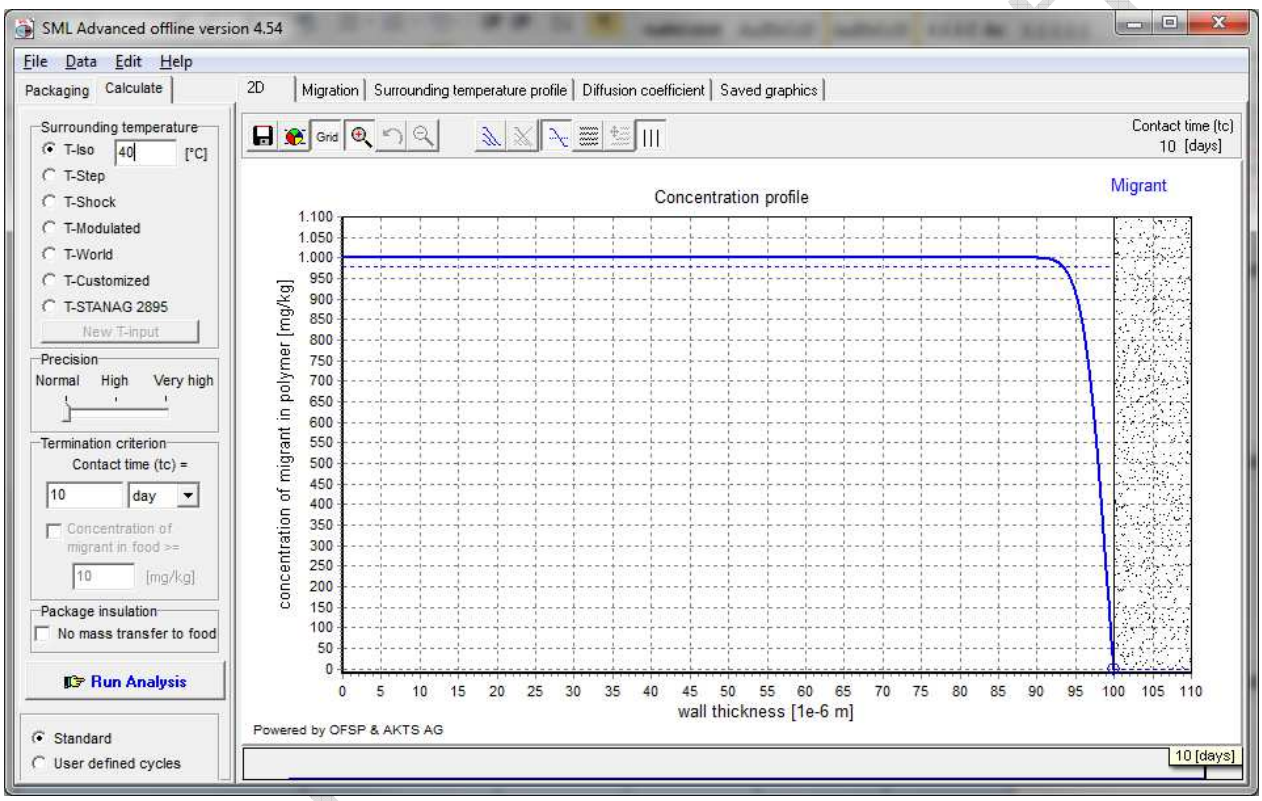
10d @ 40°C

=> 100% layer thickness = 14 µm
no absolute barrier at thicknesses below 14 µm

=> 99% layer thickness = 9.4 µm

=> 1/2 x 99% layer thickness = 5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 19 µm
above 19 µm two sides to be considered for calculation of migration if full immersion testing applied



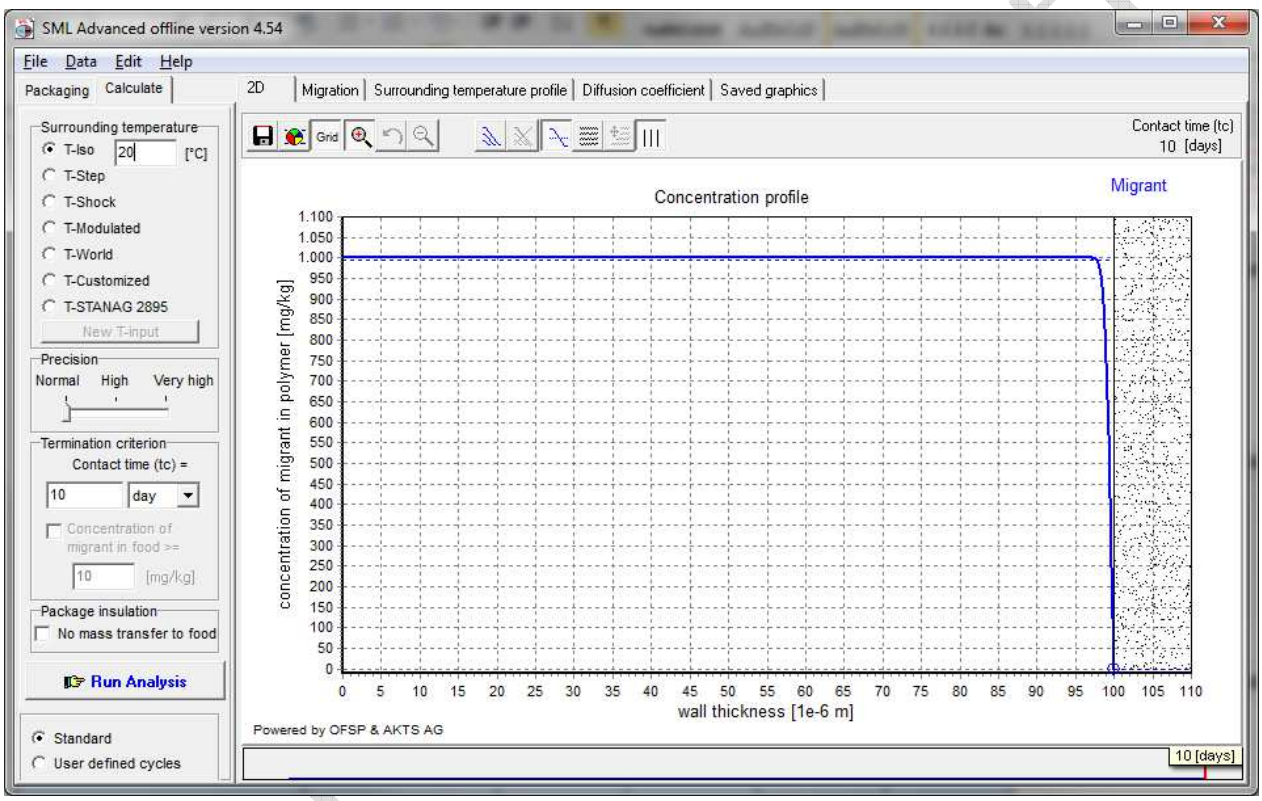
10d @ 20°C

=> 100% layer thickness = 4 µm
no absolute barrier at thicknesses below 4 µm

=> 99% layer thickness = 3 µm

=> 1/2 x 99% layer thickness = 1.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 6 µm
above 6 µm two sides to be considered for calculation of migration if full immersion testing applied



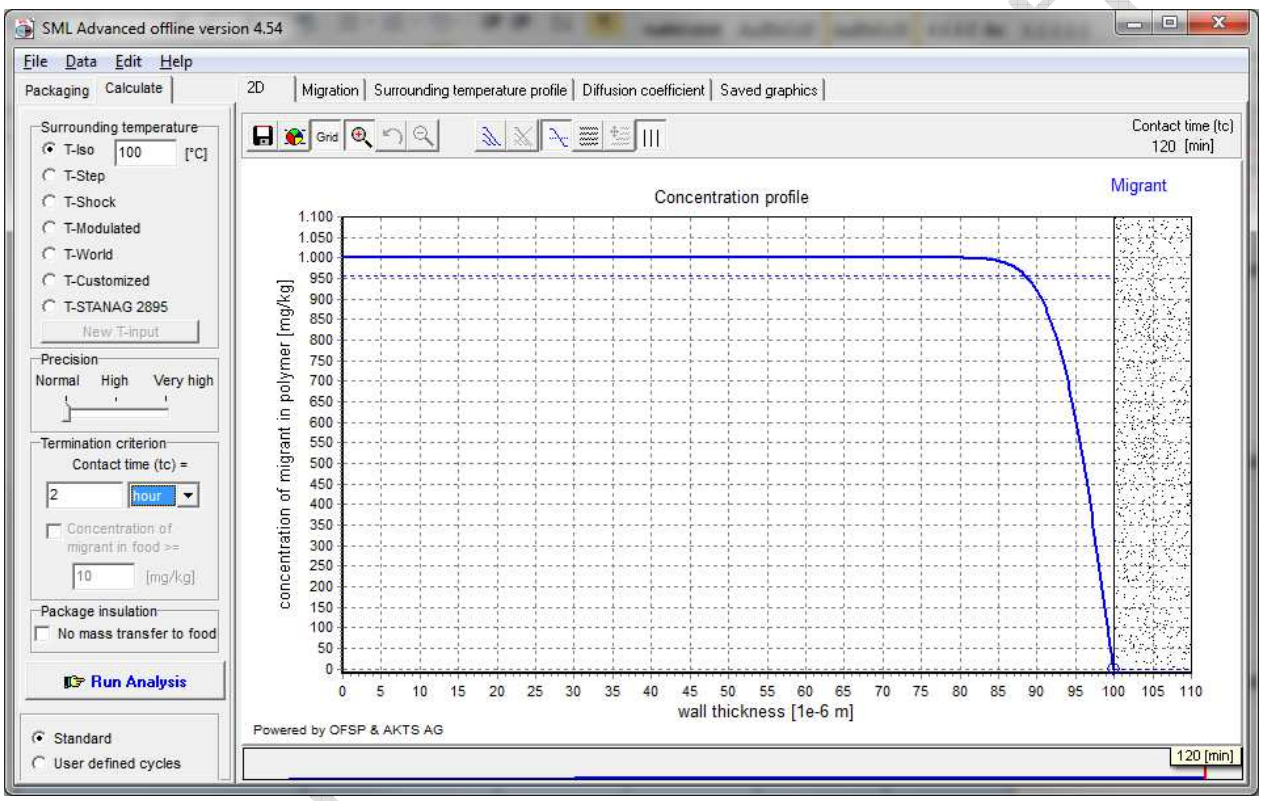
2h @ 100°C

=> 100% layer thickness = 23 µm
no absolute barrier at thicknesses below 23 µm

=> 99% layer thickness = 19 µm

=> 1/2 x 99% layer thickness = 10 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 38 µm
above 38 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

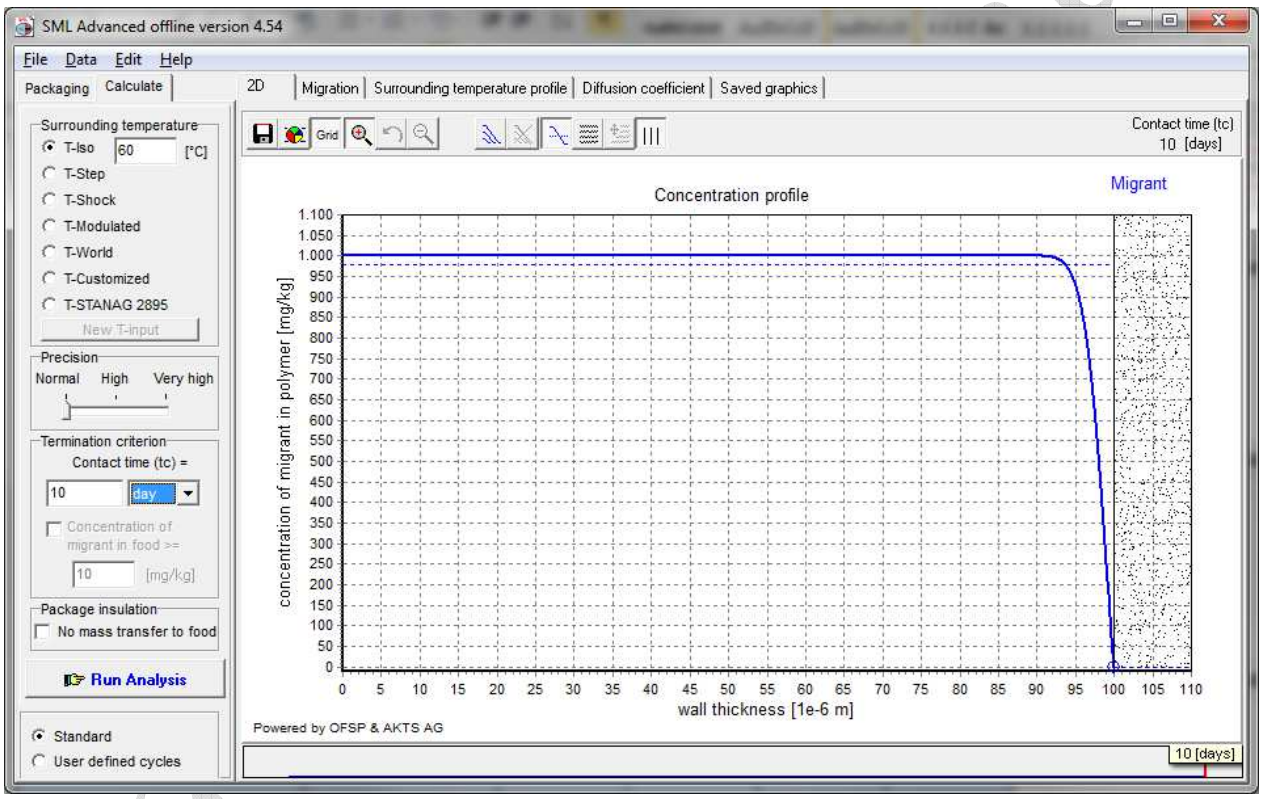
10d @ 60°C

=> 100% layer thickness = 12 µm
no absolute barrier at thicknesses below 12 µm

=> 99% layer thickness = 9 µm

=> 1/2 x 99% layer thickness = 5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 18 µm
above 18 µm two sides to be considered for calculation of migration if full immersion testing applied



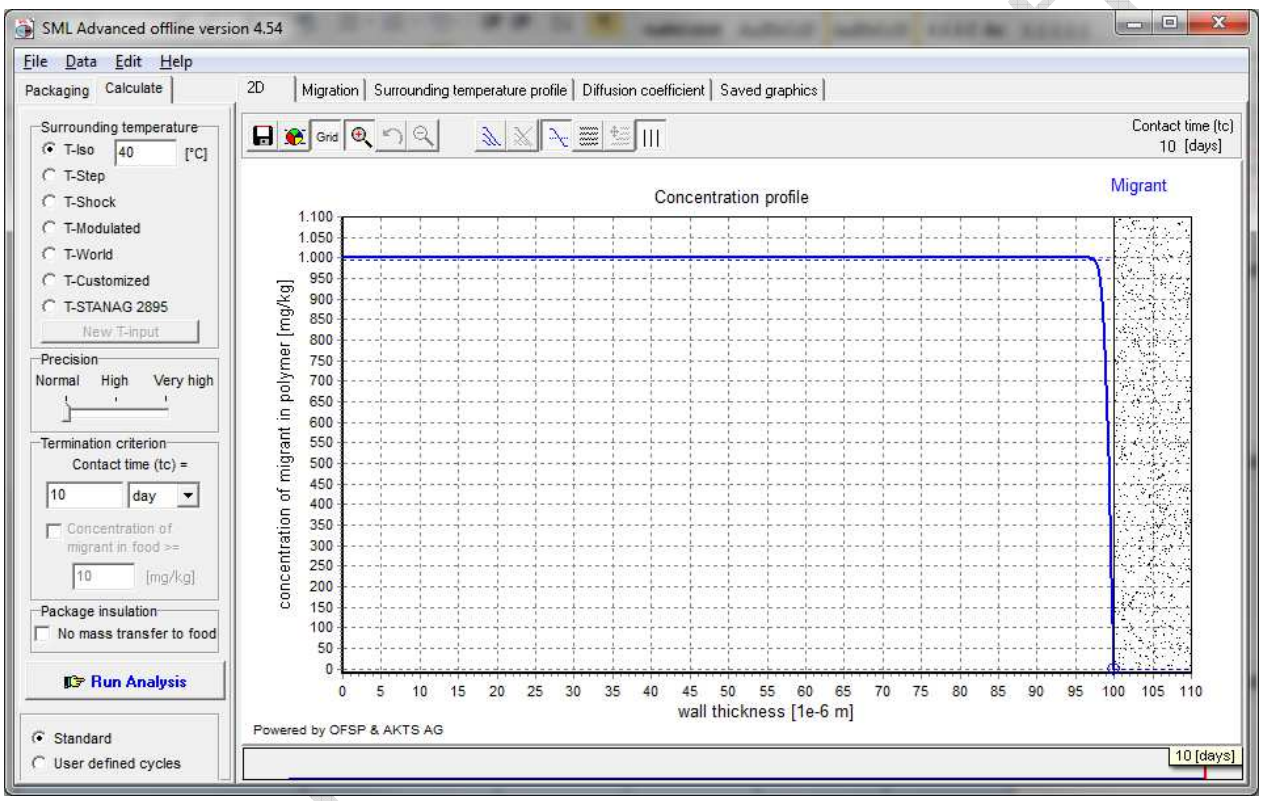
10d @ 40°C

=> 100% layer thickness = 4 µm
no absolute barrier at thicknesses below 4 µm

=> 99% layer thickness = 3 µm

=> 1/2 x 99% layer thickness = 1.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 6 µm
above 6 µm two sides to be considered for calculation of migration if full immersion testing applied



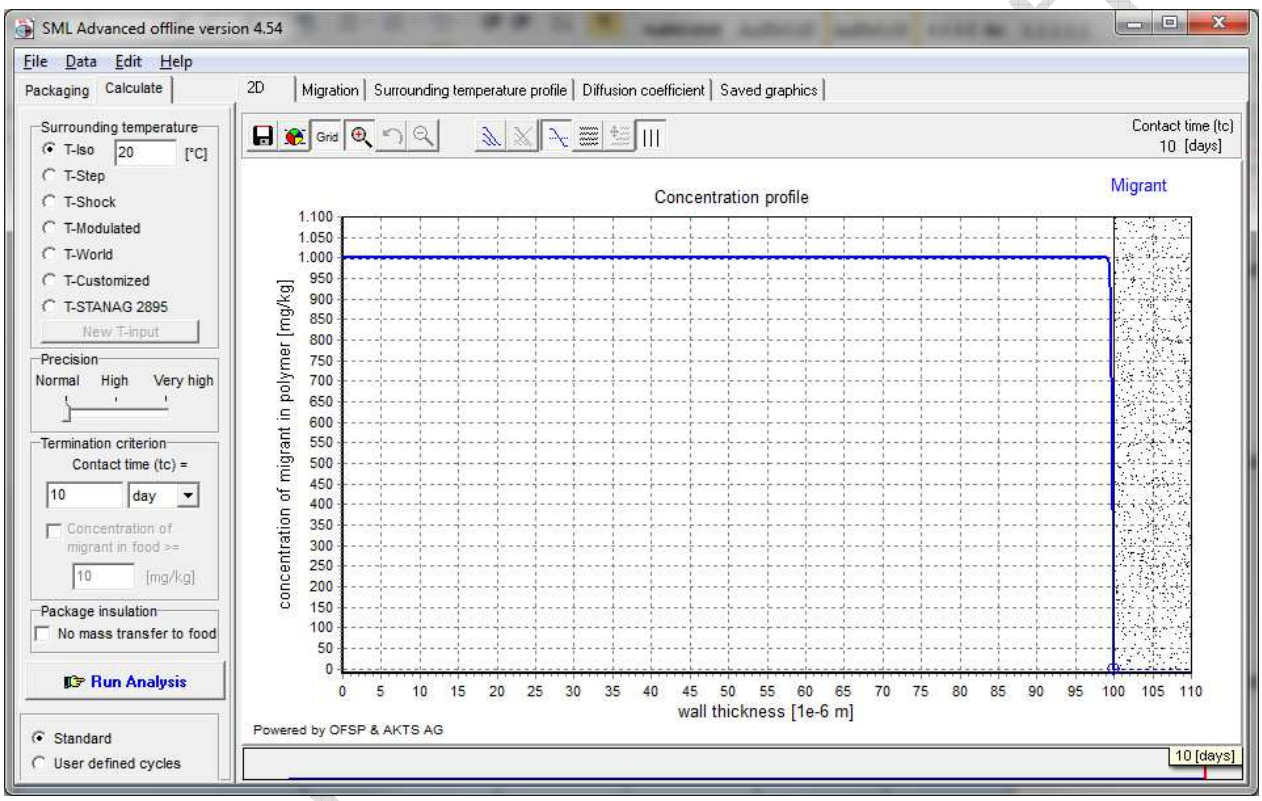
10d @ 20°C

=> 100% layer thickness = 1.3 µm
no absolute barrier at thicknesses below 1.3 µm

=> 99% layer thickness = 1 µm

=> 1/2 x 99% layer thickness = 0.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2 µm
above 2 µm two sides to be considered for calculation of migration if full immersion testing applied



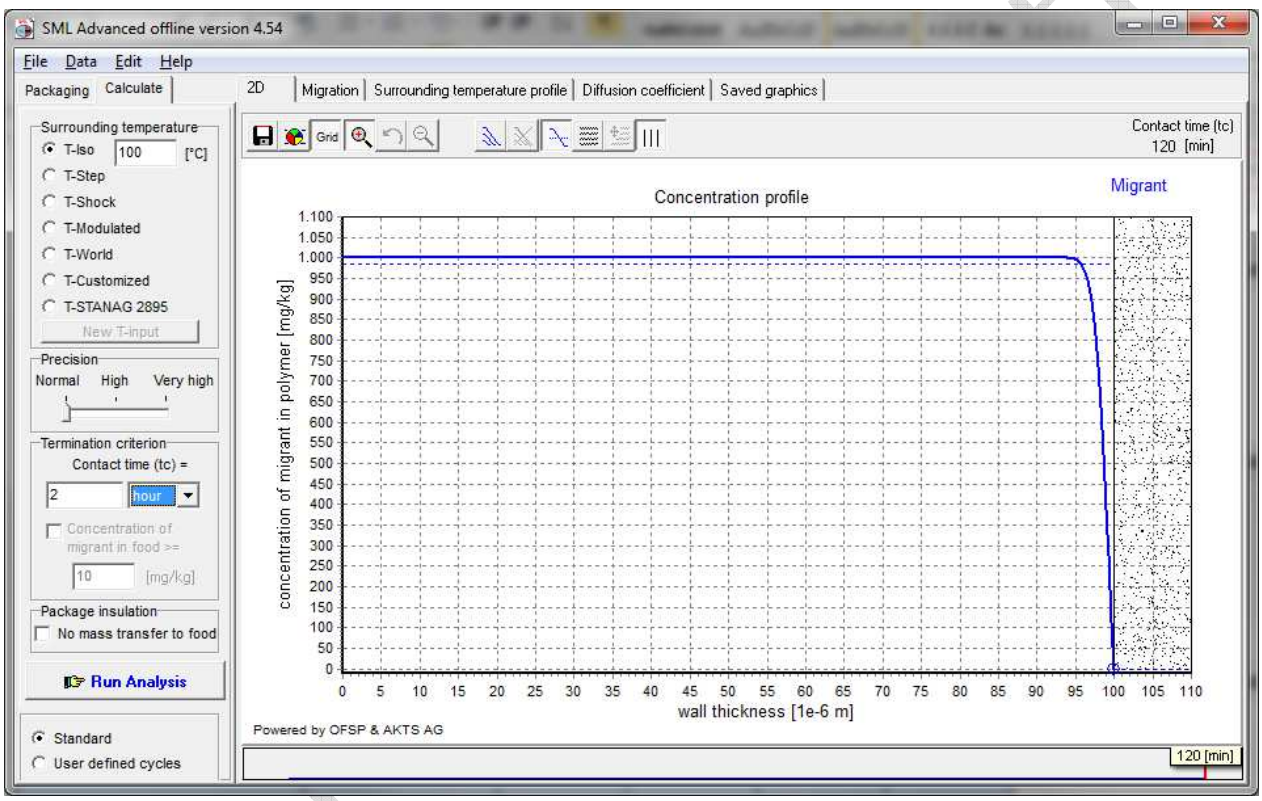
2h @ 100°C

=> 100% layer thickness = 7 µm
no absolute barrier at thicknesses below 7 µm

=> 99% layer thickness = 6 µm

=> 1/2 x 99% layer thickness = 3 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 12 µm
above 12 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

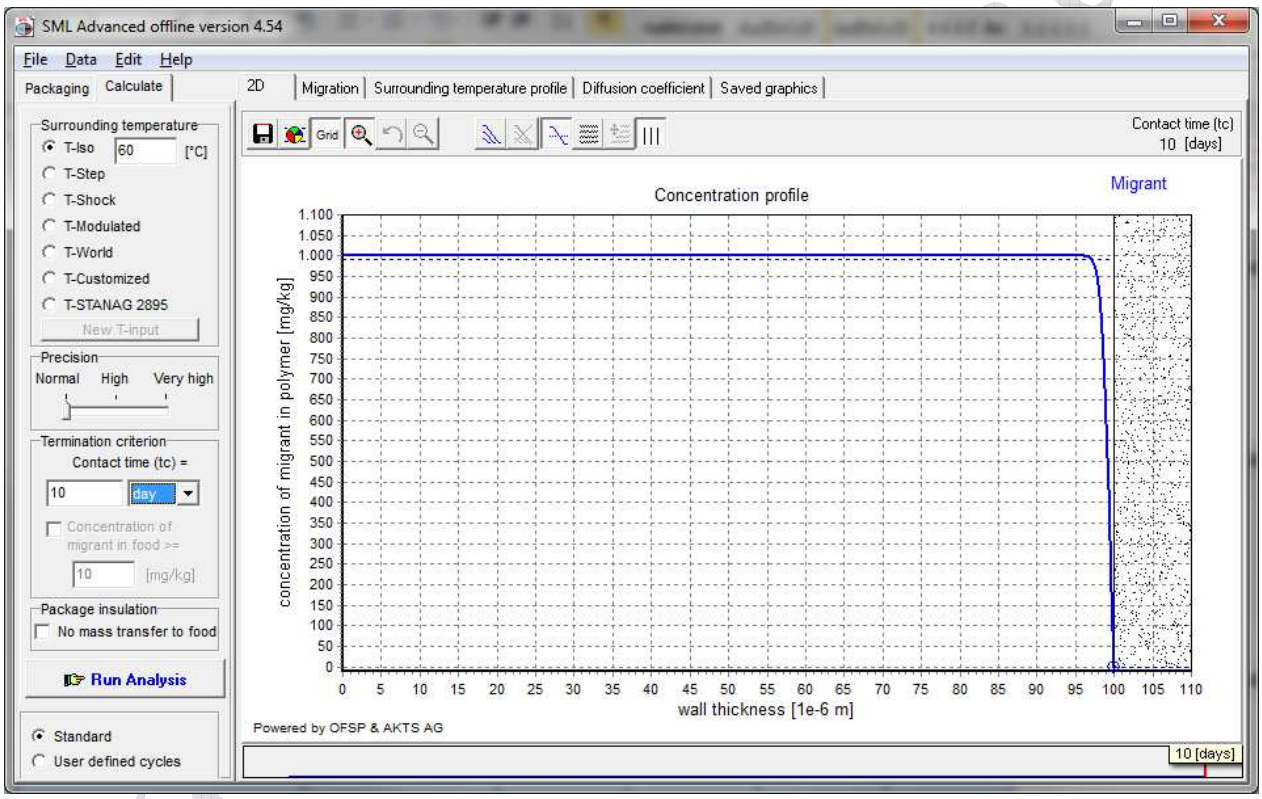
10d @ 60°C

=> 100% layer thickness = 4.4 µm
no absolute barrier at thicknesses below 4.4 µm

=> 99% layer thickness = 3.6 µm

=> 1/2 x 99% layer thickness = 2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 7 µm
above 7 µm two sides to be considered for calculation of migration if full immersion testing applied



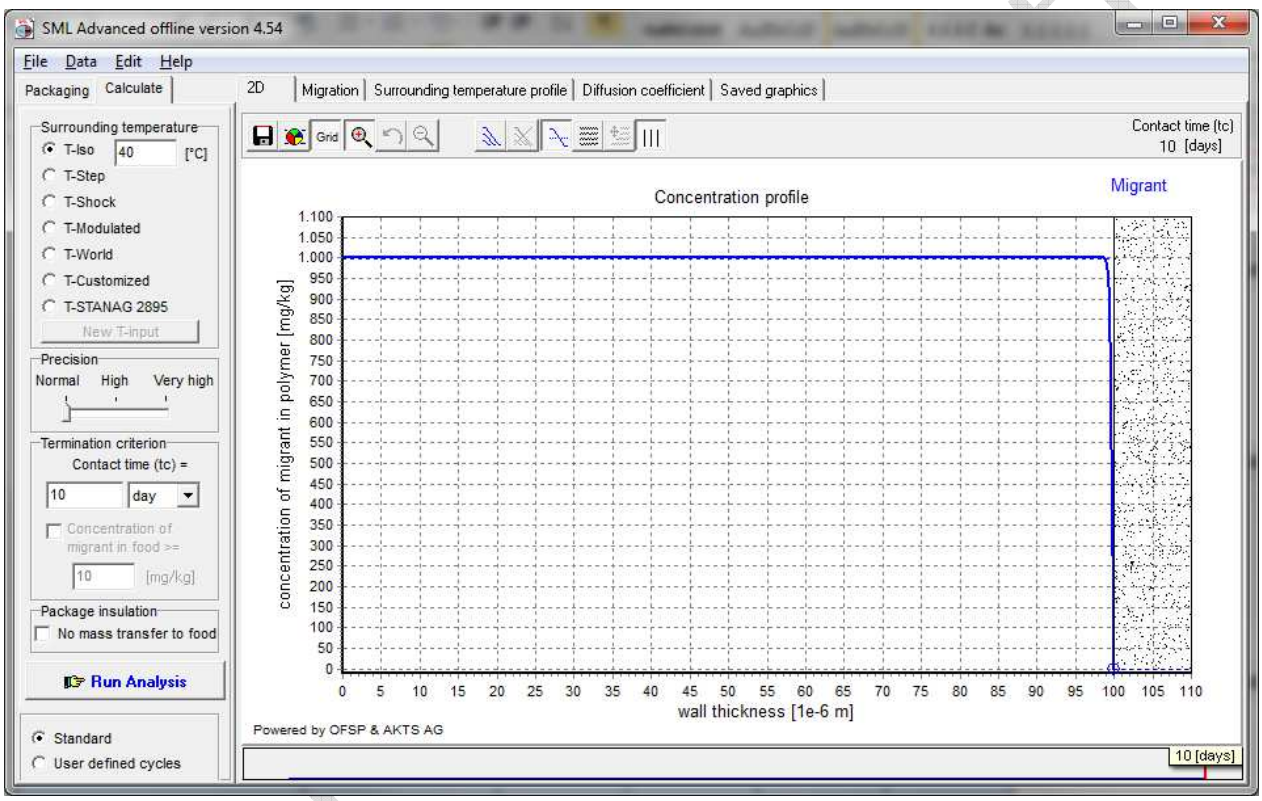
10d @ 40°C

=> 100% layer thickness = 1.7 µm
no absolute barrier at thicknesses below 1.7 µm

=> 99% layer thickness = 1.3 µm

=> 1/2 x 99% layer thickness = 0.7 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2.6 µm
above 2.6 µm two sides to be considered for calculation of migration if full immersion testing applied



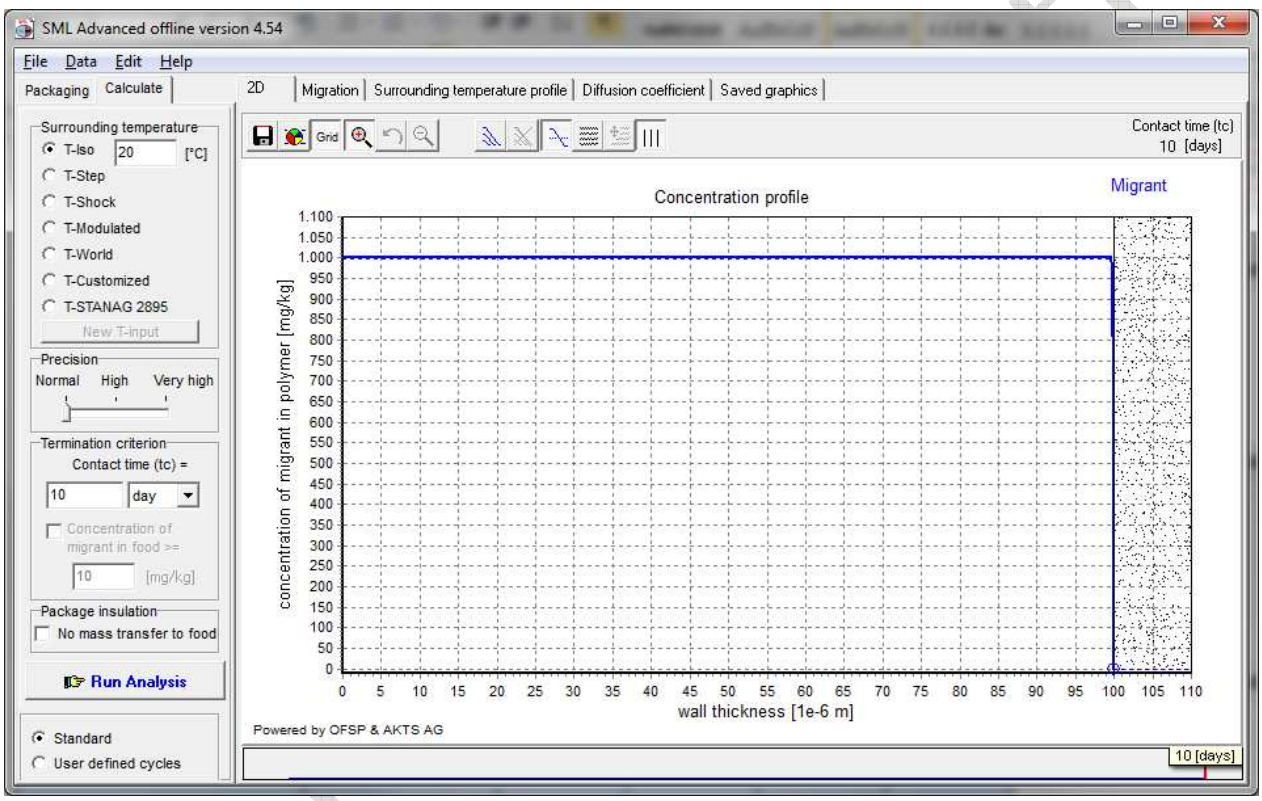
10d @ 20°C

=> 100% layer thickness = 0.6 µm
no absolute barrier at thicknesses below 0.6 µm

=> 99% layer thickness = 0.4 µm

=> 1/2 x 99% layer thickness = 0.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 0.8 µm
above 0.8 µm two sides to be considered for calculation of migration if full immersion testing applied



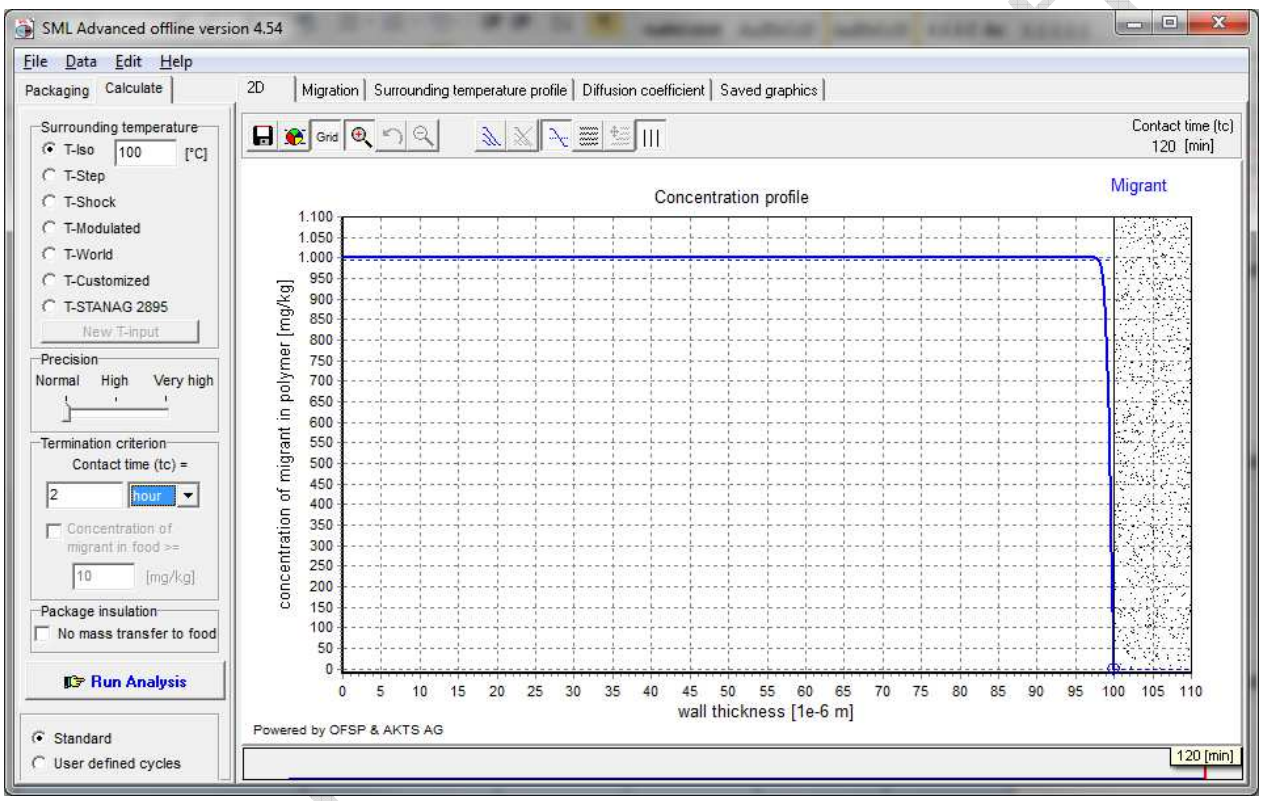
2h @ 100°C

=> 100% layer thickness = 3 µm
no absolute barrier at thicknesses below 3 µm

=> 99% layer thickness = 2.4 µm

=> 1/2 x 99% layer thickness = 1.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 5 µm
above 5 µm two sides to be considered for calculation of migration if full immersion testing applied



PS

► molecular mass 100 - 250 g/mol

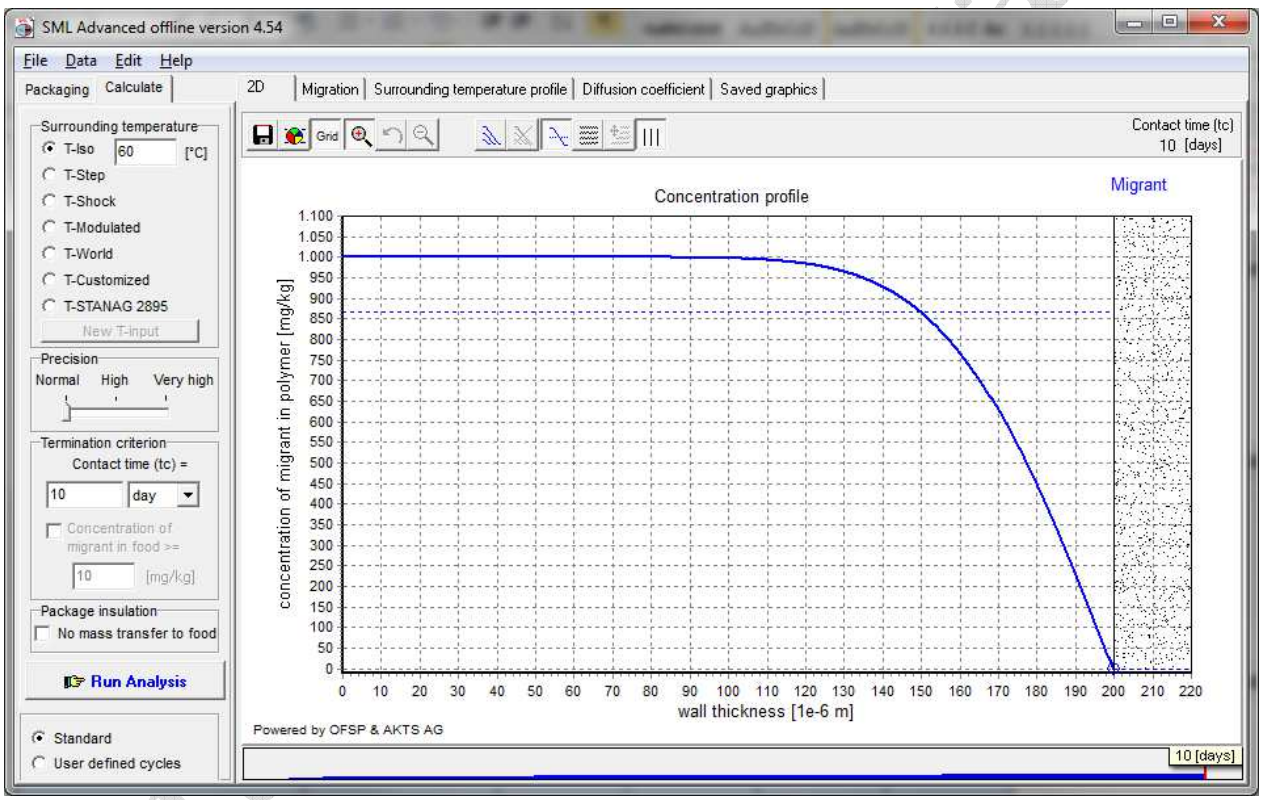
10d @ 60°C

=> 100% layer thickness = 127
no absolute barrier at thicknesses below 127 µm

=> 99% layer thickness = 110 µm

=> 1/2 x 99% layer thickness = 55 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 220 µm
above 220 µm two sides to be considered for calculation of migration if full immersion testing applied



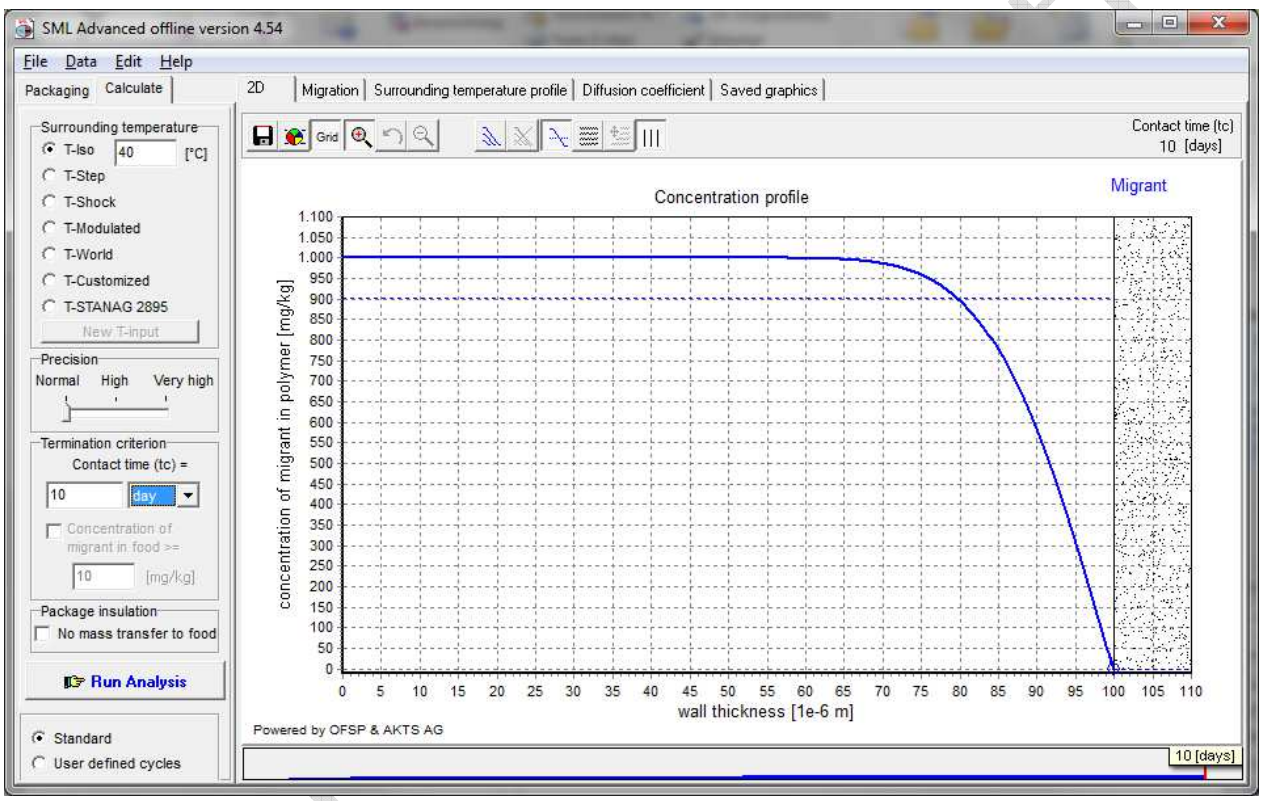
10d @ 40°C

=> 100% layer thickness = 46
no absolute barrier at thicknesses below 46 µm

=> 99% layer thickness = 40 µm

=> 1/2 x 99% layer thickness = 20 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 80
above 80 µm two sides to be considered for calculation of migration if full immersion testing applied



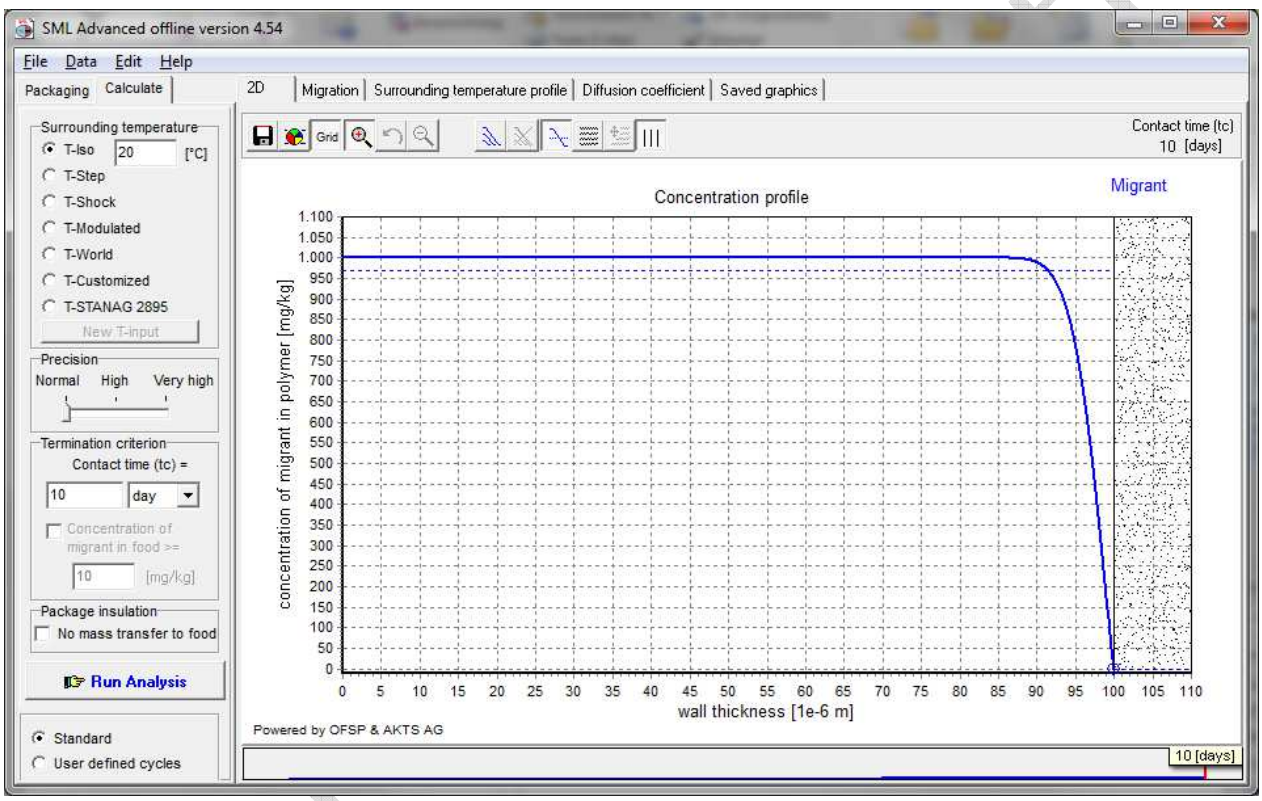
10d @ 20°C

=> 100% layer thickness = 17 µm
no absolute barrier at thicknesses below 17 µm

=> 99% layer thickness = 13 µm

=> 1/2 x 99% layer thickness =7 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 26 µm
above 26 µm two sides to be considered for calculation of migration if full immersion testing applied



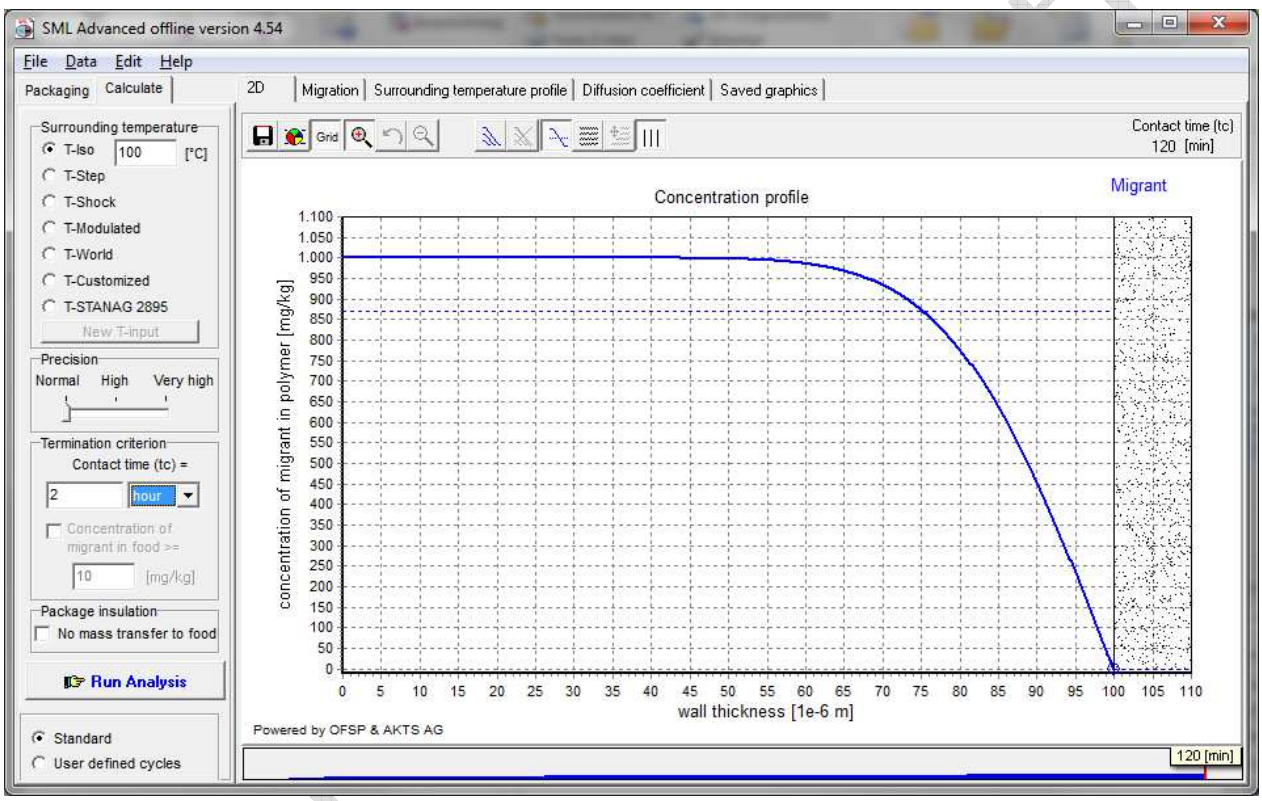
2h @ 100°C

=> 100% layer thickness = 65 µm
no absolute barrier at thicknesses below 65 µm

=> 99% layer thickness = 54 µm

=> 1/2 x 99% layer thickness = 27 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 108 µm
above 108 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

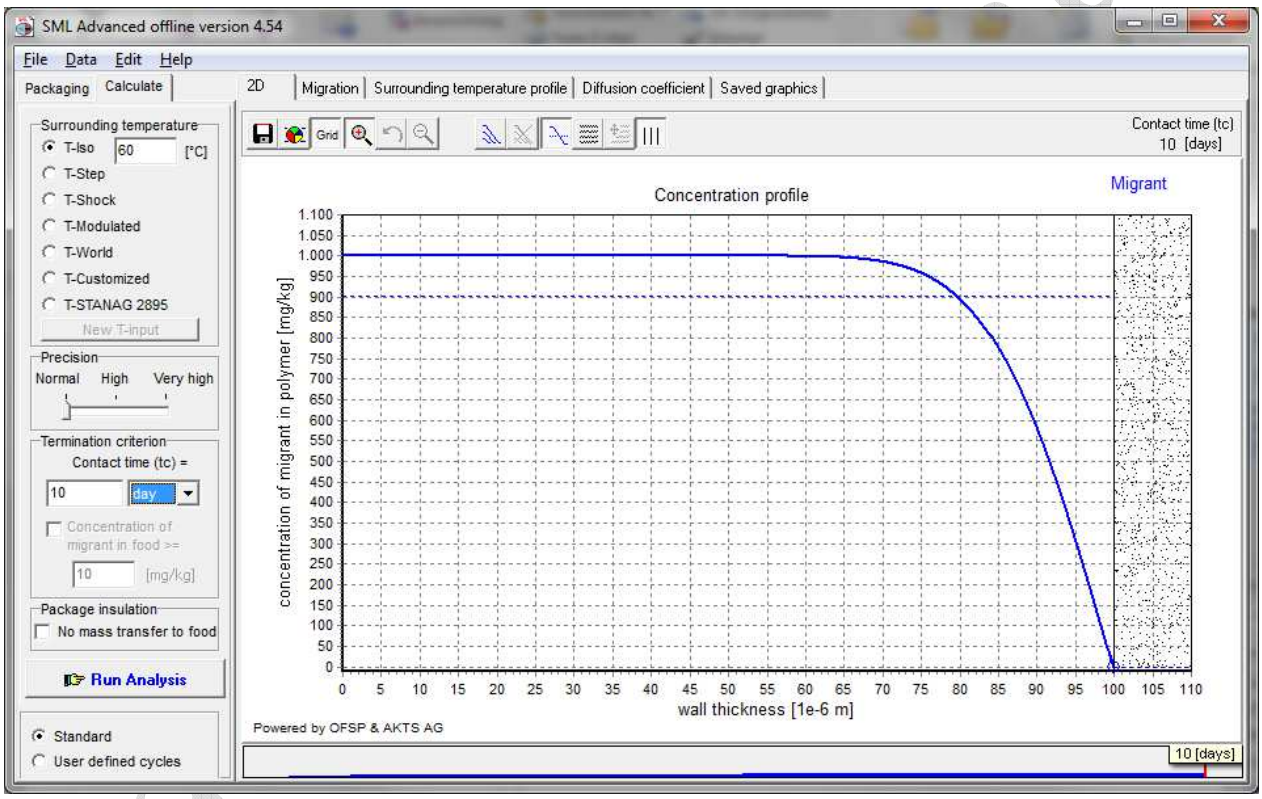
10d @ 60°C

=> 100% layer thickness = 49 µm
no absolute barrier at thicknesses below 49 µm

=> 99% layer thickness = 41 µm

=> 1/2 x 99% layer thickness = 20.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 82 µm
above 82 µm two sides to be considered for calculation of migration if full immersion testing applied



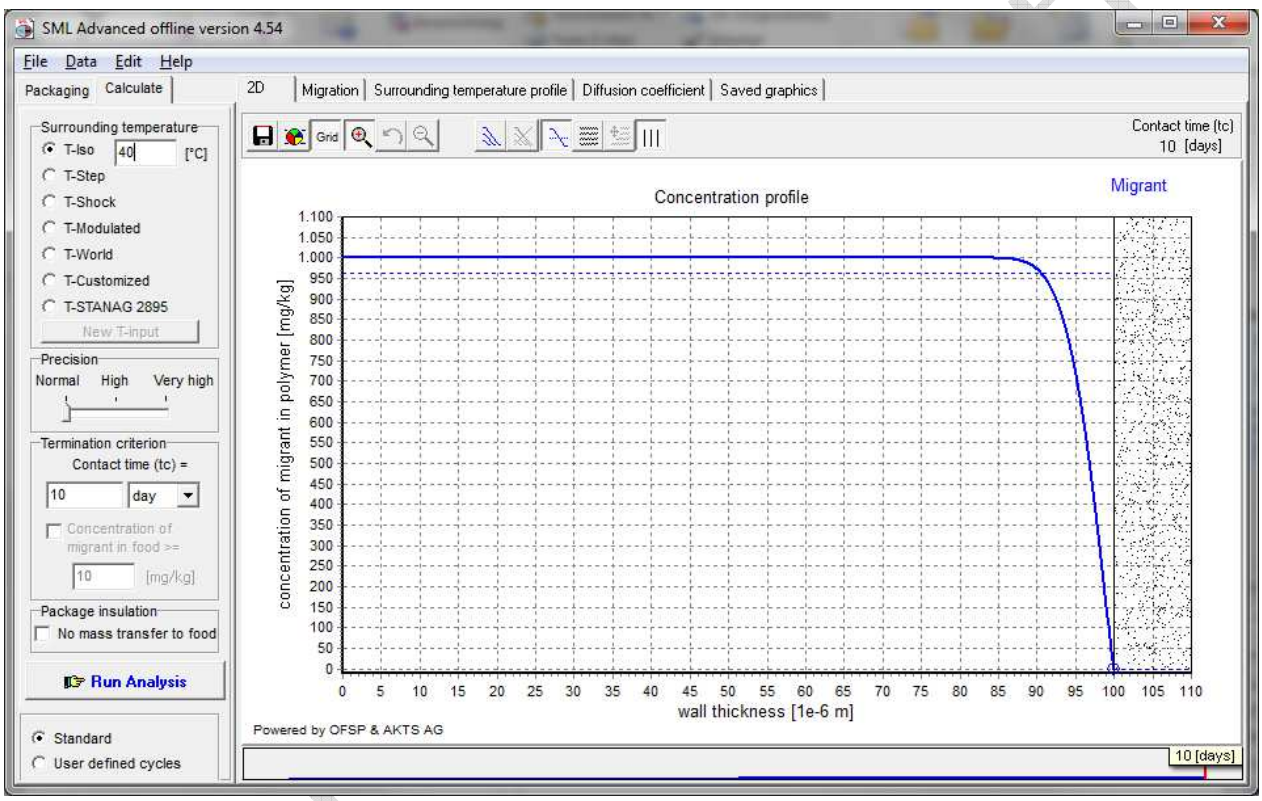
10d @ 40°C

=> 100% layer thickness = 18 µm
no absolute barrier at thicknesses below 18 µm

=> 99% layer thickness = 15 µm

=> 1/2 x 99% layer thickness = 7.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 30 µm
above 30 µm two sides to be considered for calculation of migration if full immersion testing applied



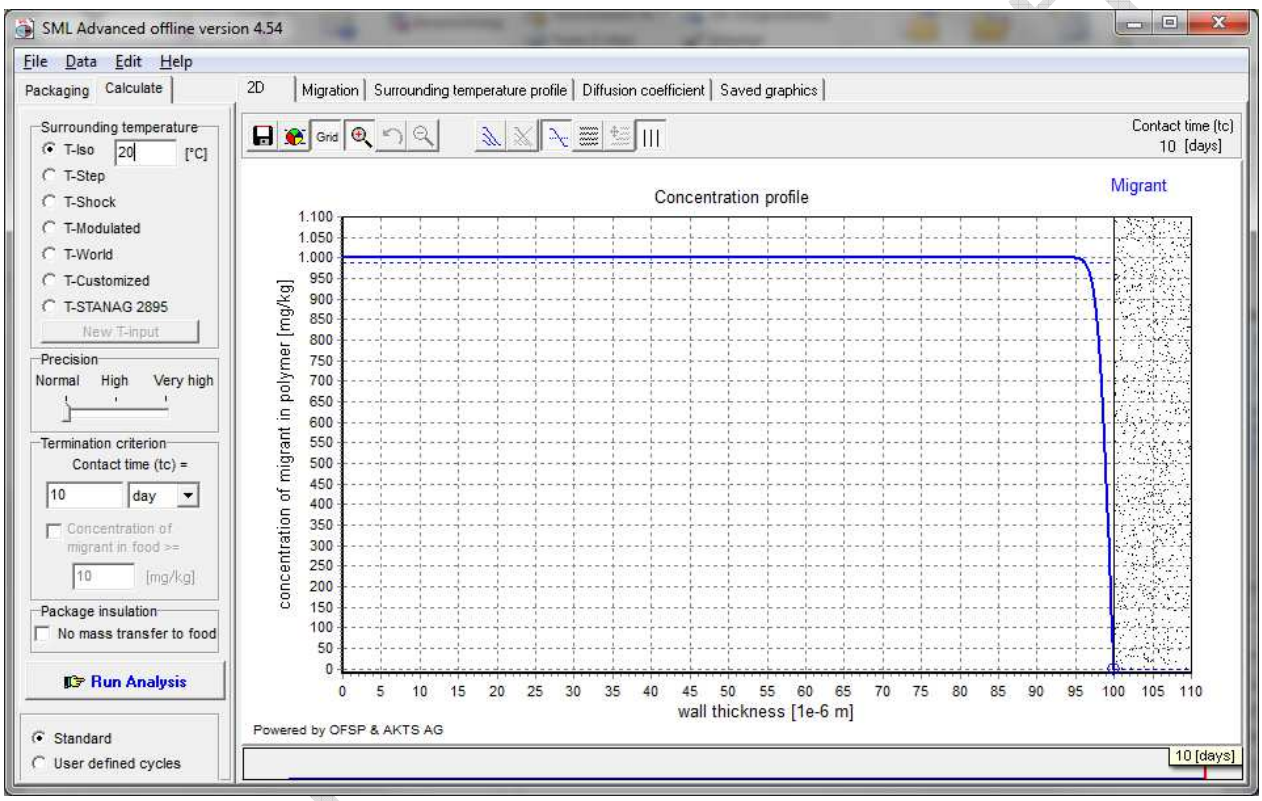
10d @ 20°C

=> 100% layer thickness = 6.2 µm
no absolute barrier at thicknesses below 6.2 µm

=> 99% layer thickness = 5 µm

=> 1/2 x 99% layer thickness = 2.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 10 µm
above 10 µm two sides to be considered for calculation of migration if full immersion testing applied



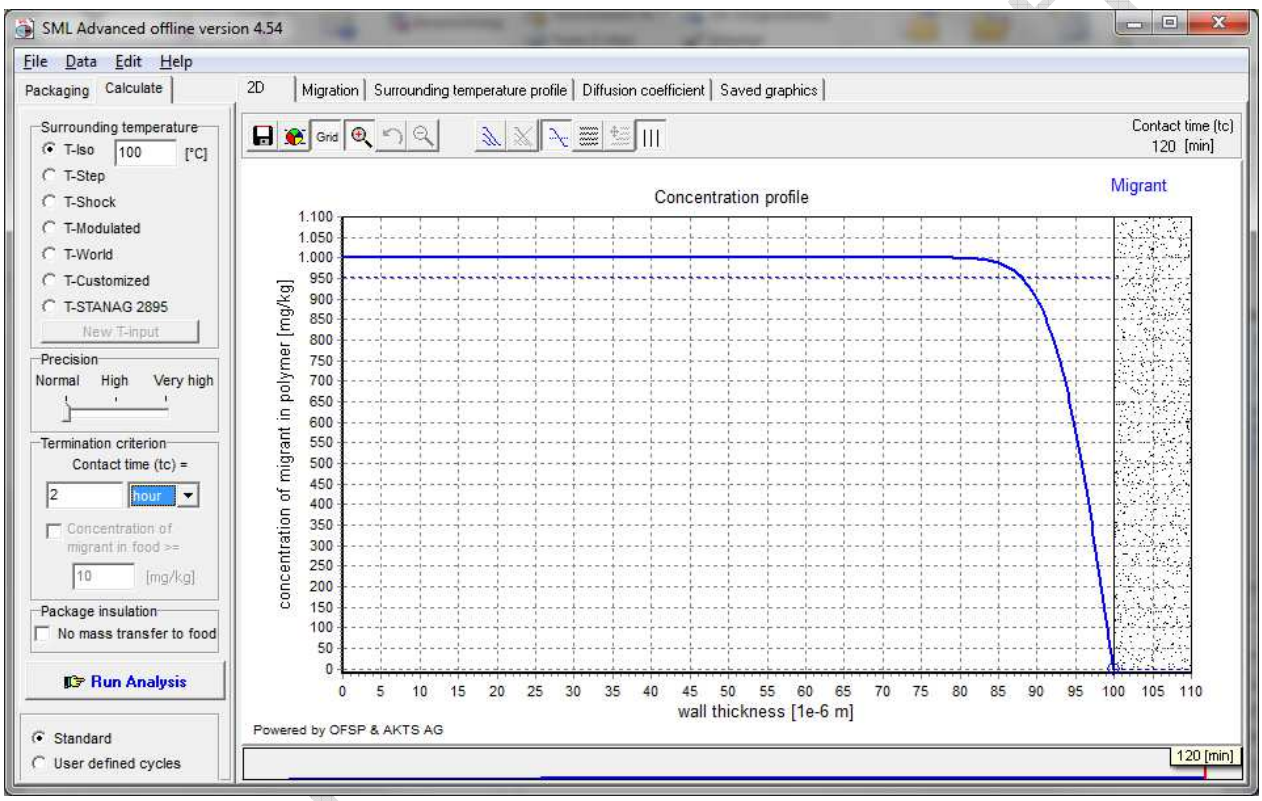
2h @ 100°C

=> 100% layer thickness = 26 µm
no absolute barrier at thicknesses below 26 µm

=> 99% layer thickness = 20 µm

=> 1/2 x 99% layer thickness = 10 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 40 µm
above 40 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

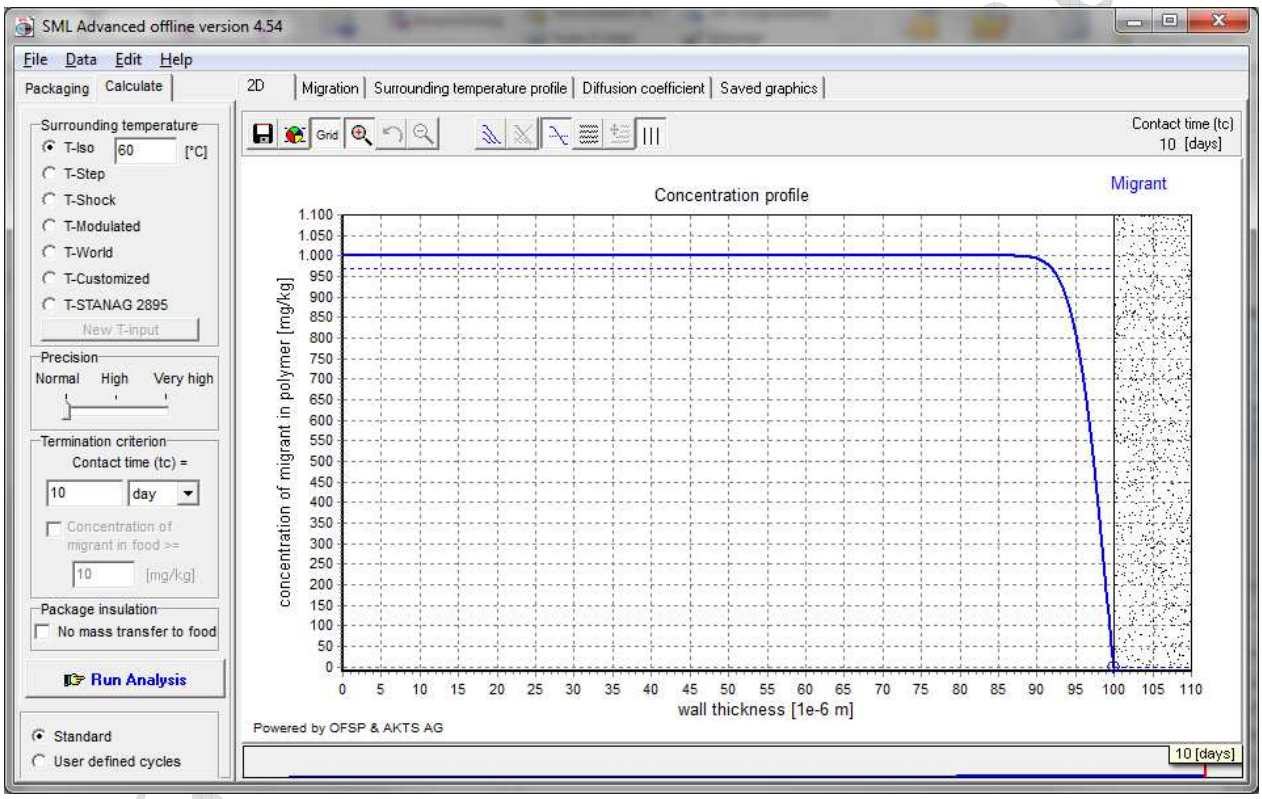
10d @ 60°C

=> 100% layer thickness = 15.2 µm
no absolute barrier at thicknesses below 15.2 µm

=> 99% layer thickness = 12.4 µm

=> 1/2 x 99% layer thickness = 6.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 24.8 µm
above 24.8 µm two sides to be considered for calculation of migration if full immersion testing applied



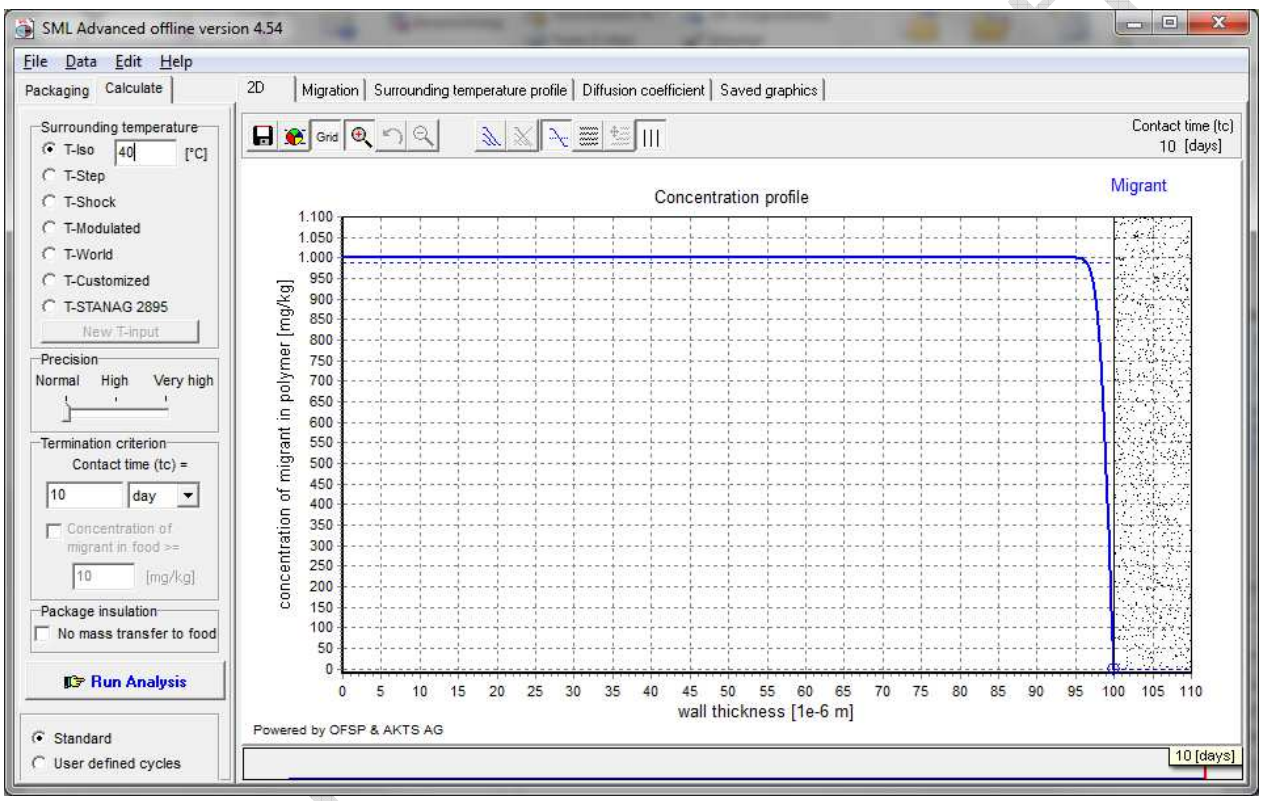
10d @ 40°C

=> 100% layer thickness = 6 µm
no absolute barrier at thicknesses below 6 µm

=> 99% layer thickness = 5 µm

=> 1/2 x 99% layer thickness = 2.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 10 µm
above 10 µm two sides to be considered for calculation of migration if full immersion testing applied



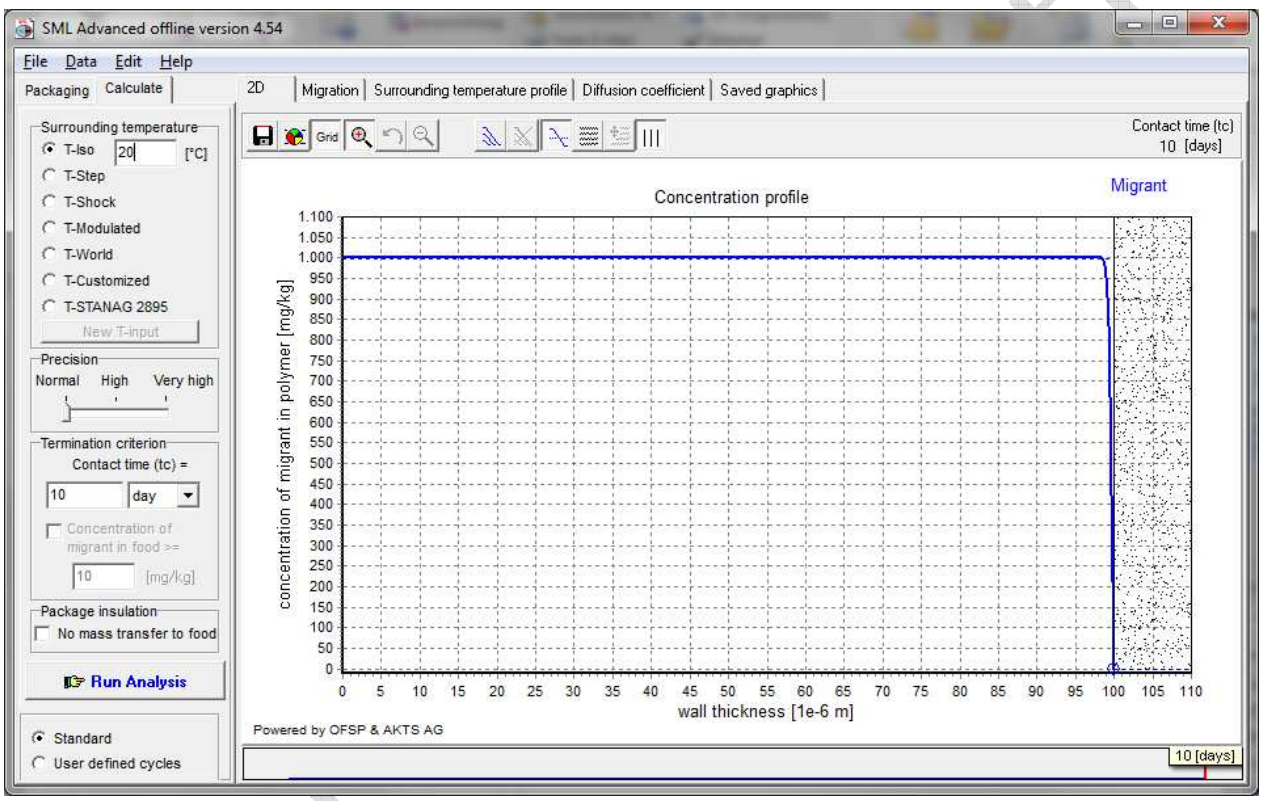
10d @ 20°C

=> 100% layer thickness = 2.2 µm
no absolute barrier at thicknesses below 2.2 µm

=> 99% layer thickness = 1.6 µm

=> 1/2 x 99% layer thickness = 0.8 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3.2 µm
above 3.2 µm two sides to be considered for calculation of migration if full immersion testing applied



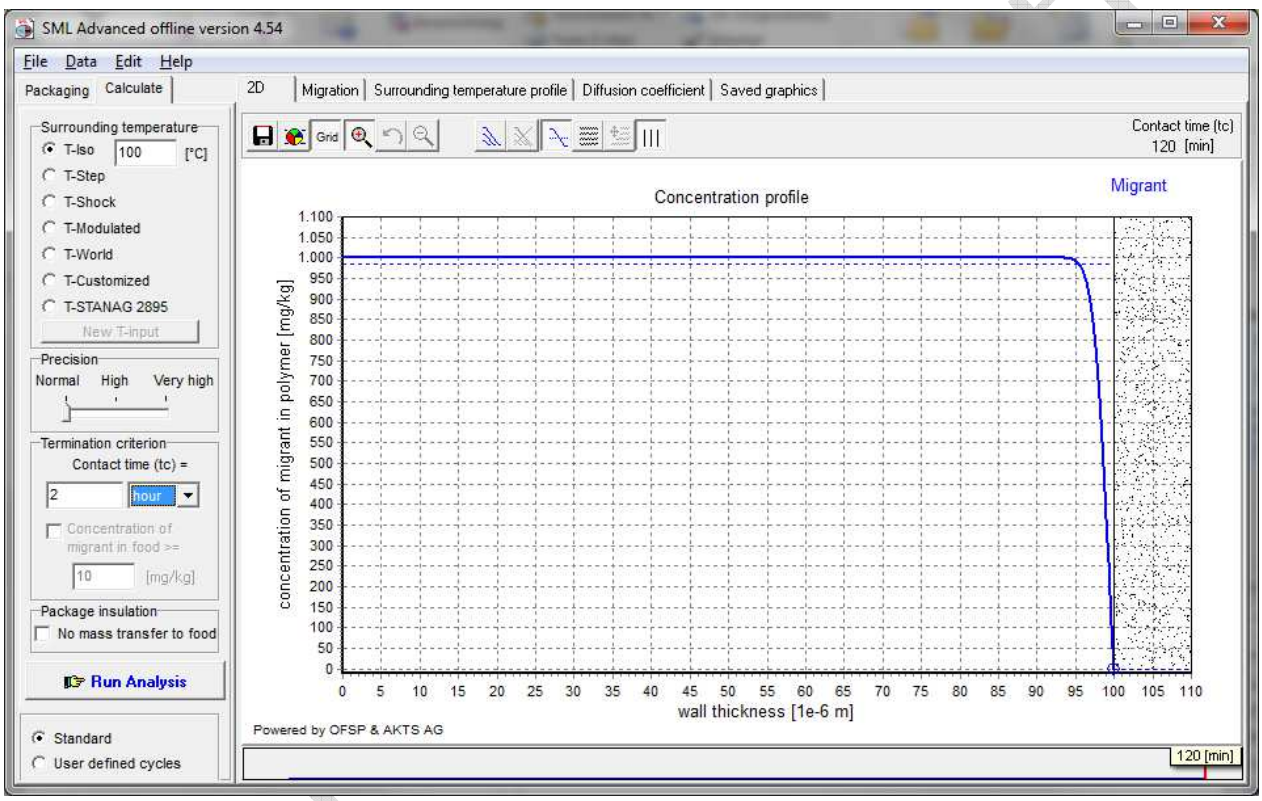
2h @ 100°C

=> 100% layer thickness = 8 µm
no absolute barrier at thicknesses below 8 µm

=> 99% layer thickness = 6.2 µm

=> 1/2 x 99% layer thickness = 3.1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 12.4 µm
above 12.4 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

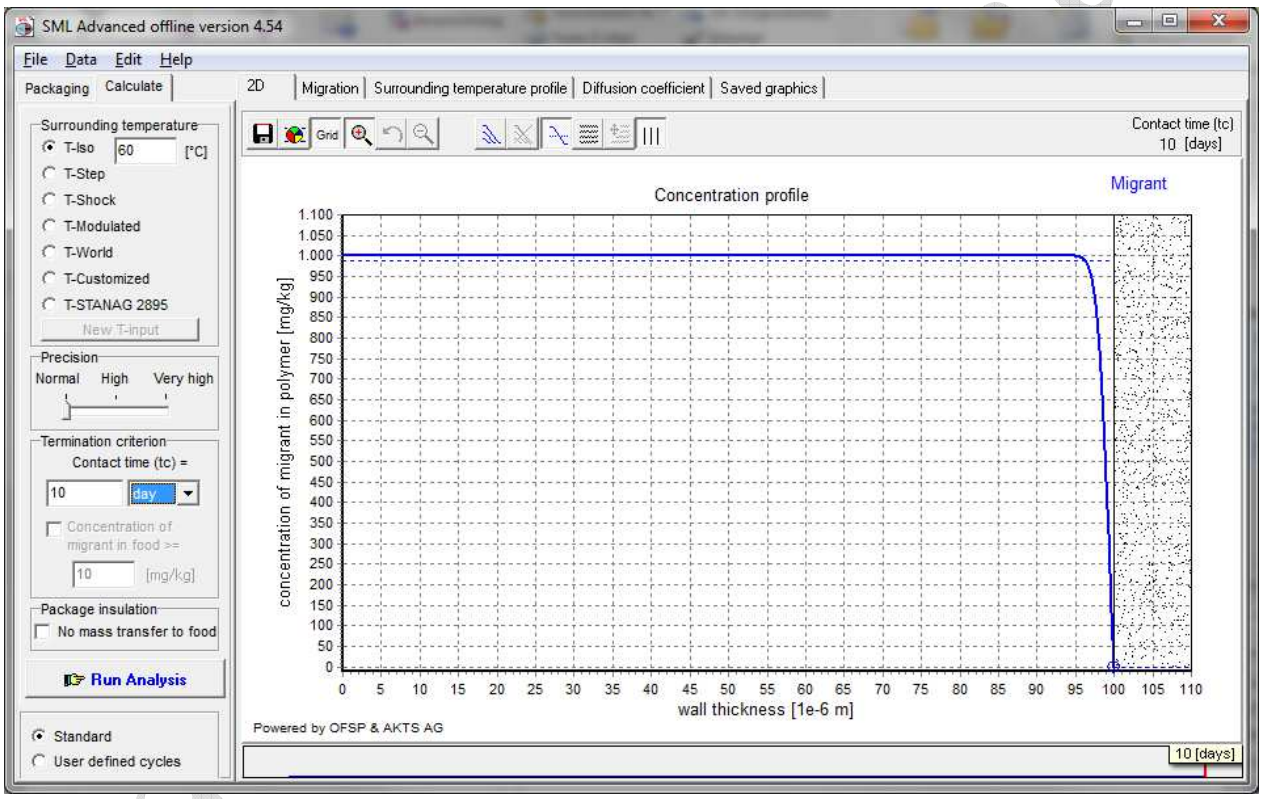
10d @ 60°C

=> 100% layer thickness = 6 µm
no absolute barrier at thicknesses below 6 µm

=> 99% layer thickness = 4.8 µm

=> 1/2 x 99% layer thickness = 2.4 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 9.2 µm
above 9.2 µm two sides to be considered for calculation of migration if full immersion testing applied



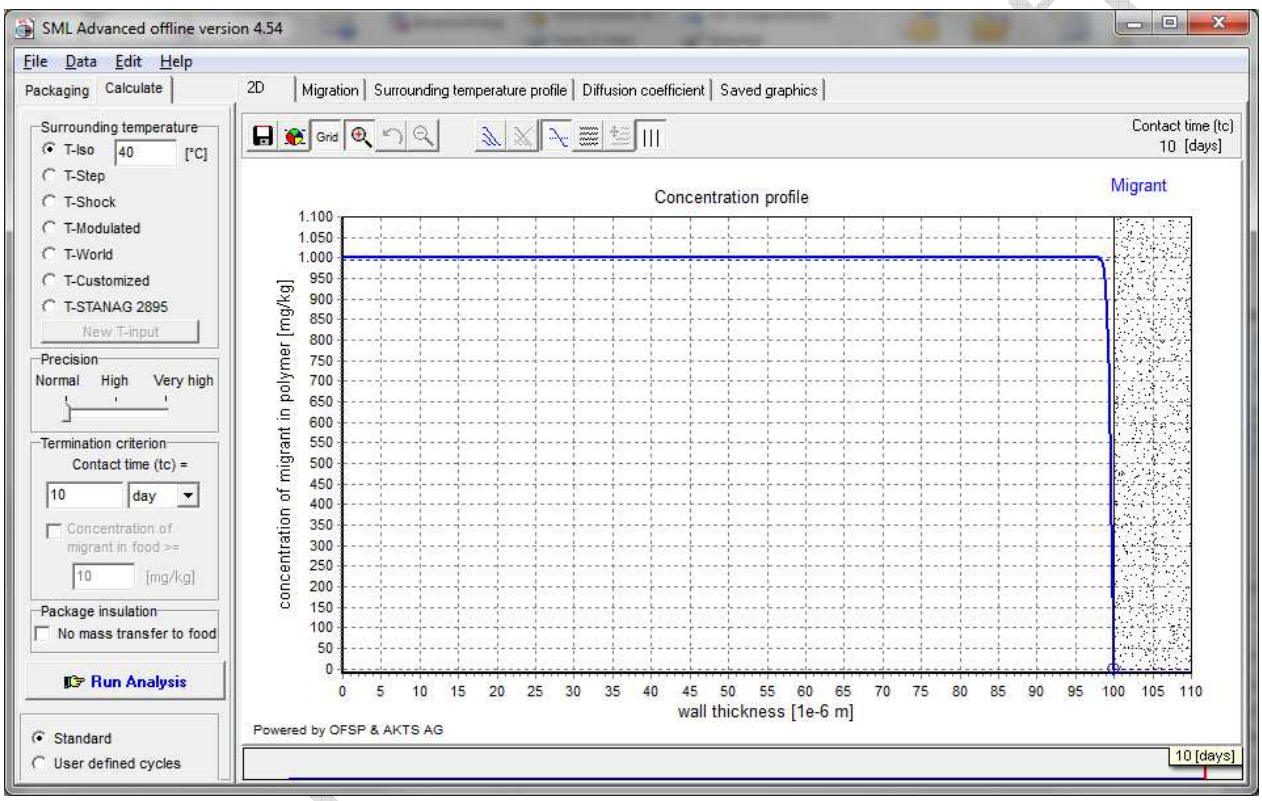
10d @ 40°C

=> 100% layer thickness = 2.8 µm
no absolute barrier at thicknesses below 2.8 µm

=> 99% layer thickness = 2 µm

=> 1/2 x 99% layer thickness = 1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4 µm
above 4 µm two sides to be considered for calculation of migration if full immersion testing applied



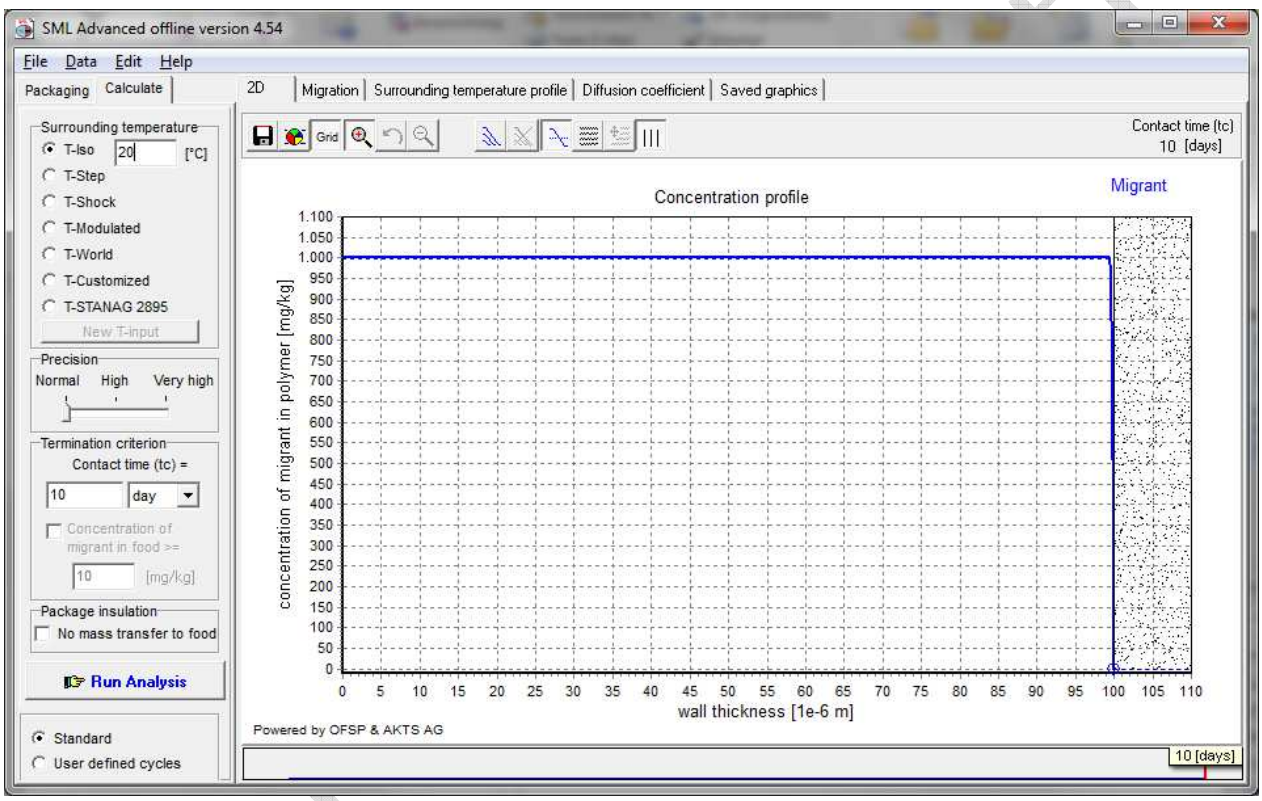
10d @ 20°C

=> 100% layer thickness = 0.8 µm
no absolute barrier at thicknesses below 0.8 µm

=> 99% layer thickness = 0.6 µm

=> 1/2 x 99% layer thickness = 0.3 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1.2 µm
above 1.2 µm two sides to be considered for calculation of migration if full immersion testing applied



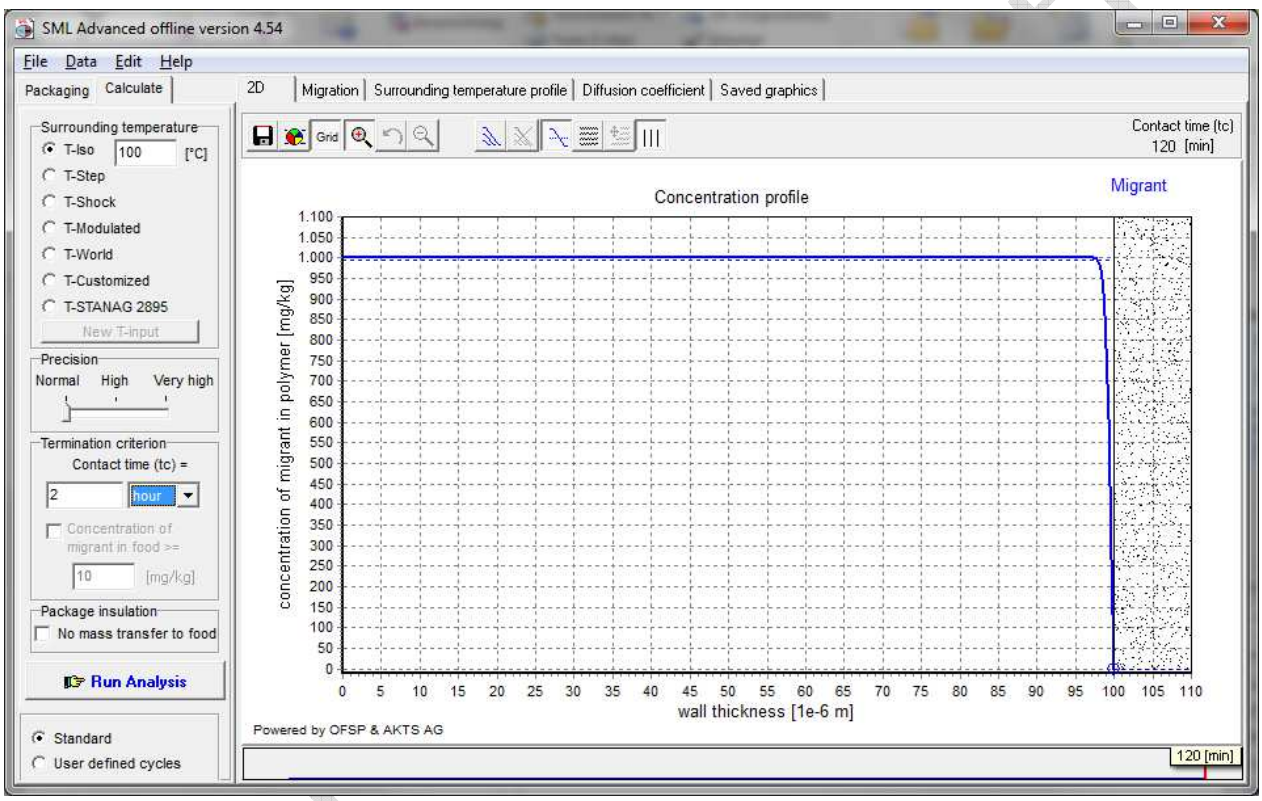
2h @ 100°C

=> 100% layer thickness = 3.3 µm
no absolute barrier at thicknesses below 3.3 µm

=> 99% layer thickness = 2.6 µm

=> 1/2 x 99% layer thickness = 1.3 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 5.2 µm
above 5.2 µm two sides to be considered for calculation of migration if full immersion testing applied



SBS

► molecular mass 100 - 250 g/mol

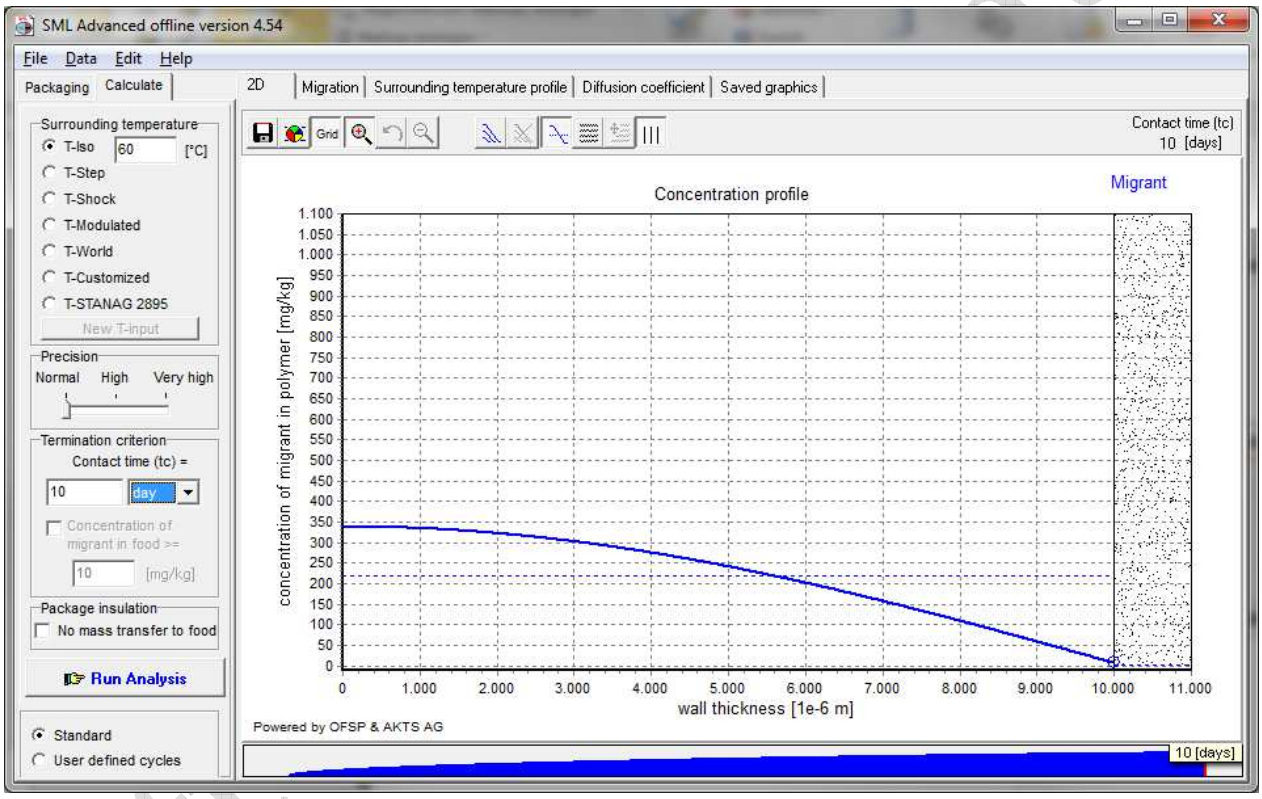
10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied



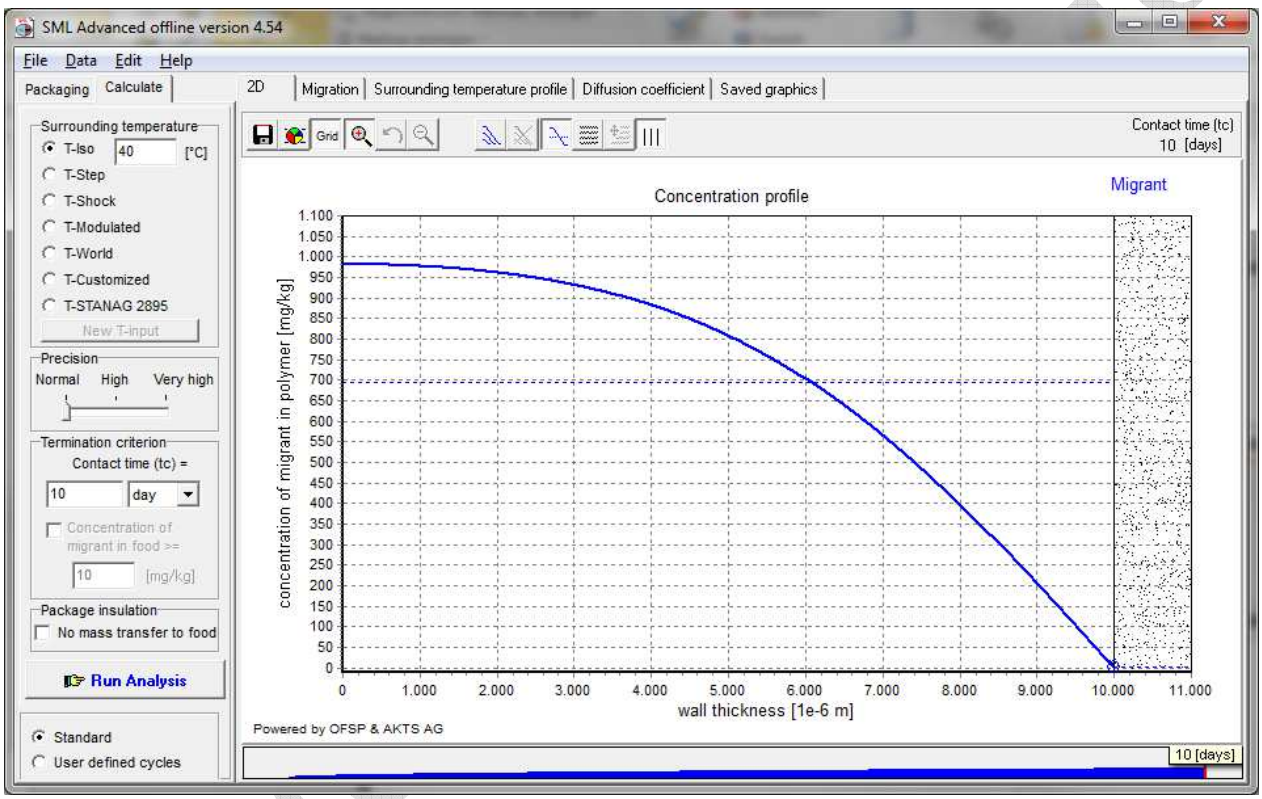
10d @ 40°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied



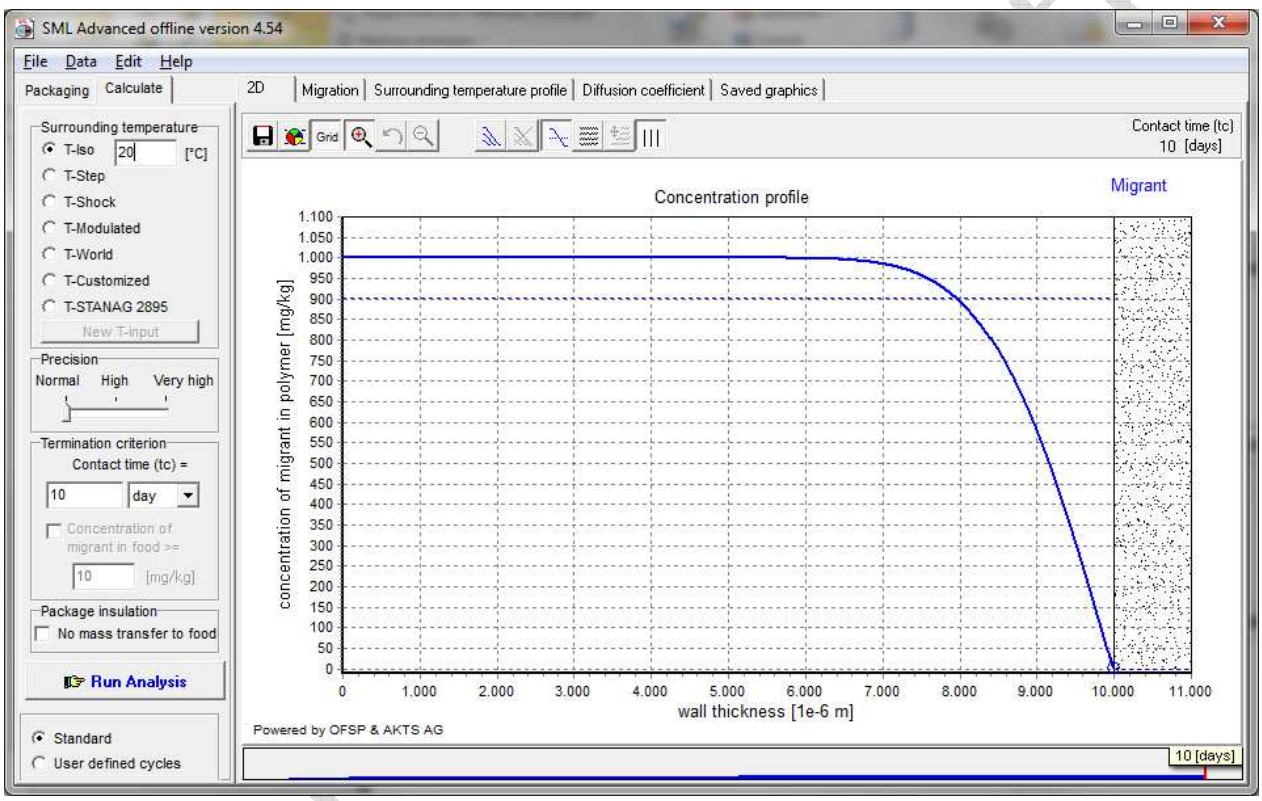
10d @ 20°C

=> 100% layer thickness = 5000 µm
no absolute barrier at thicknesses below 5000 µm

=> 99% layer thickness = 4200 µm

=> 1/2 x 99% layer thickness =2100 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 8400 µm
above 8400 µm two sides to be considered for calculation of migration if full immersion testing applied



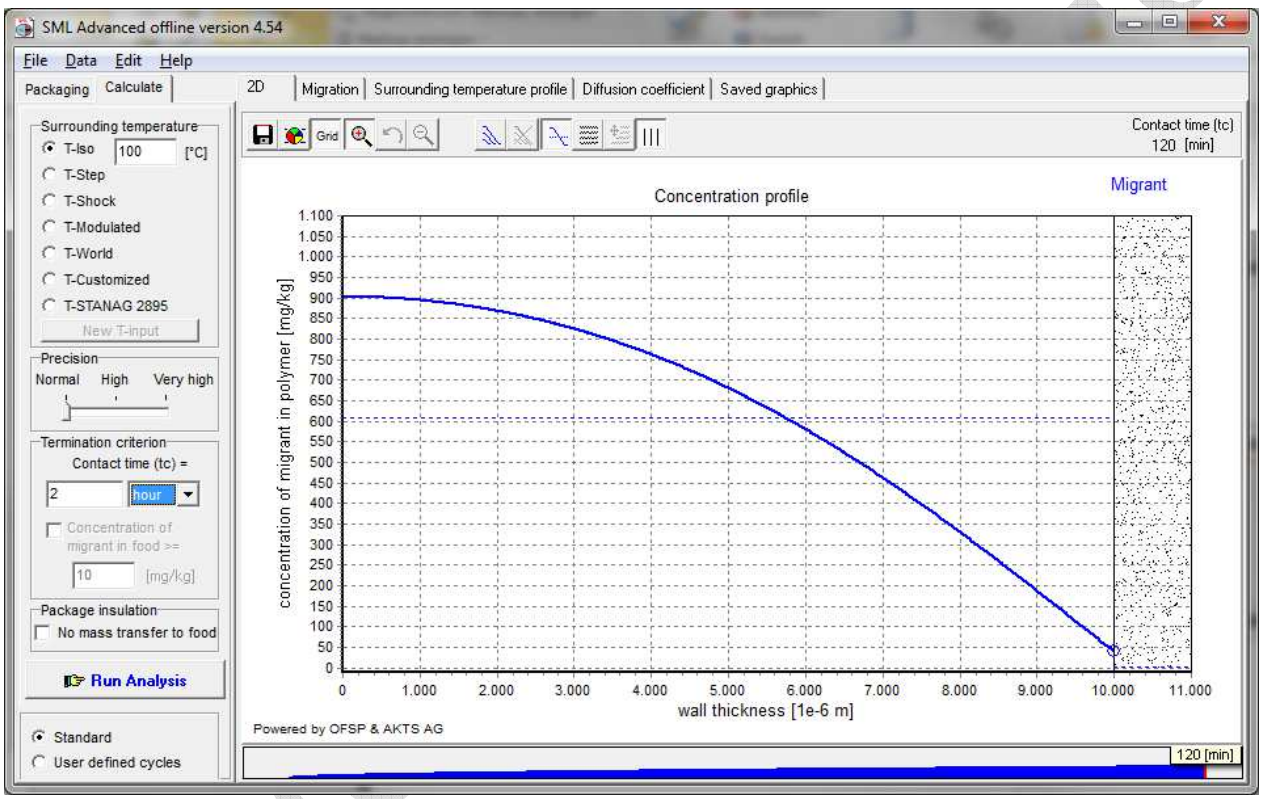
2h @ 100°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied



► **molecular mass 251 - 500 g/mol**

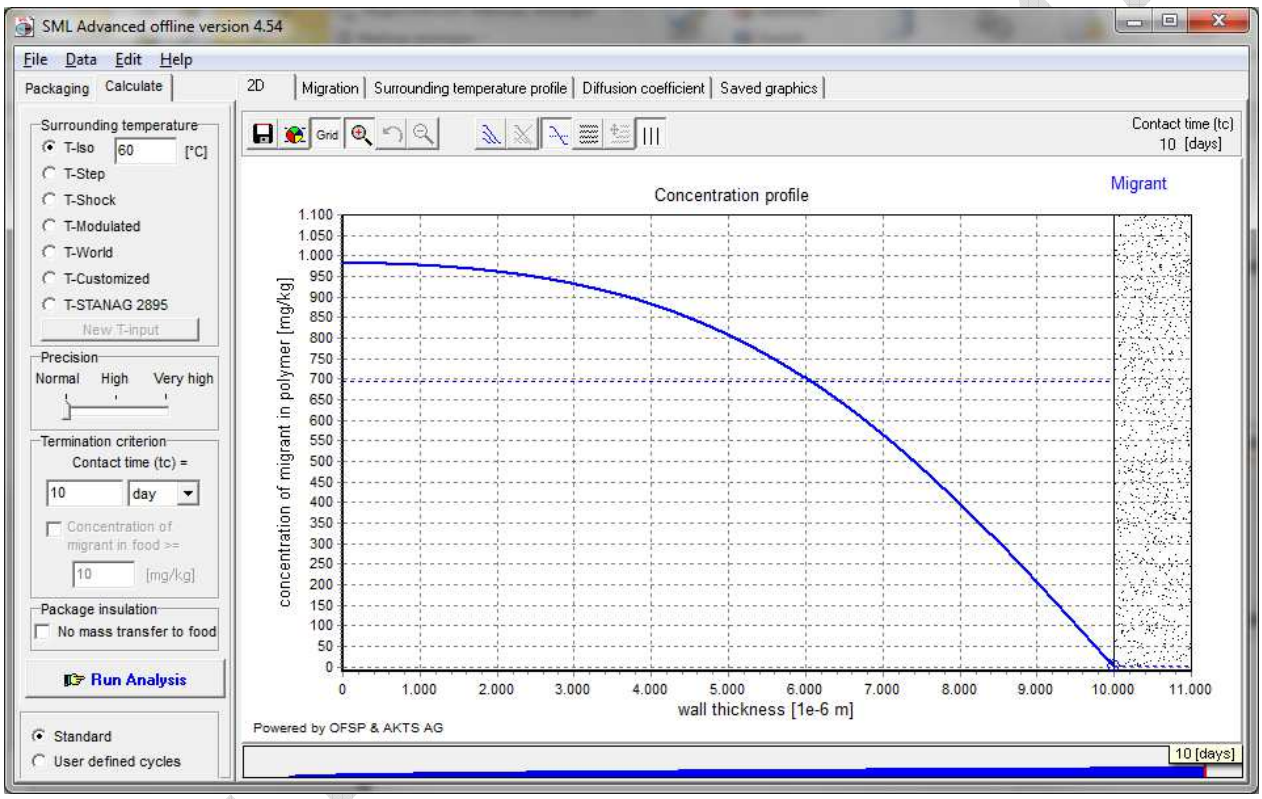
10d @ 60°C

=> 100% layer thickness = full length
no absolute barrier at thicknesses below 10000 µm

=> 99% layer thickness = full length

=> full length to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = none
only one side to be considered for calculation of migration if full immersion testing is applied



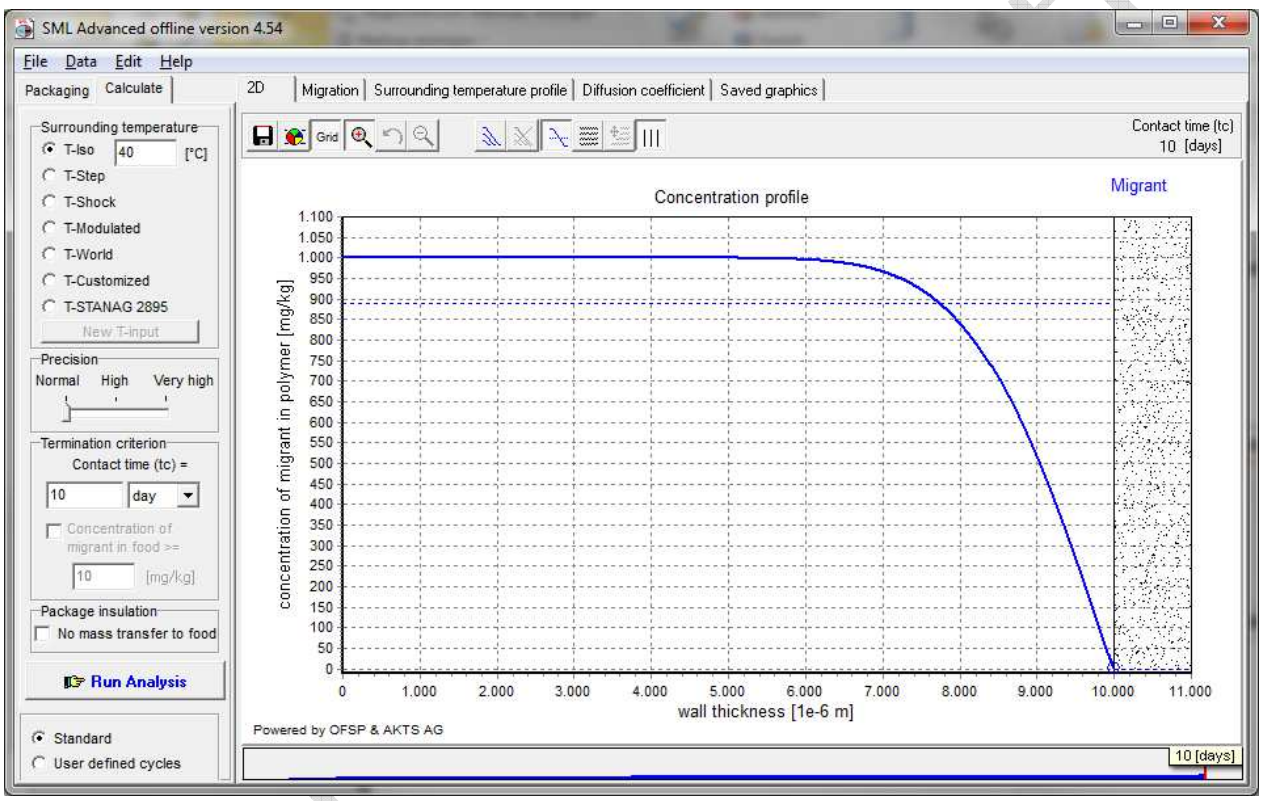
10d @ 40°C

=> 100% layer thickness = 5800 µm
no absolute barrier at thicknesses below 5800 µm

=> 99% layer thickness = 4600 µm

=> 1/2 x 99% layer thickness = 2300 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 9200 µm
above 9200 µm two sides to be considered for calculation of migration if full immersion testing applied



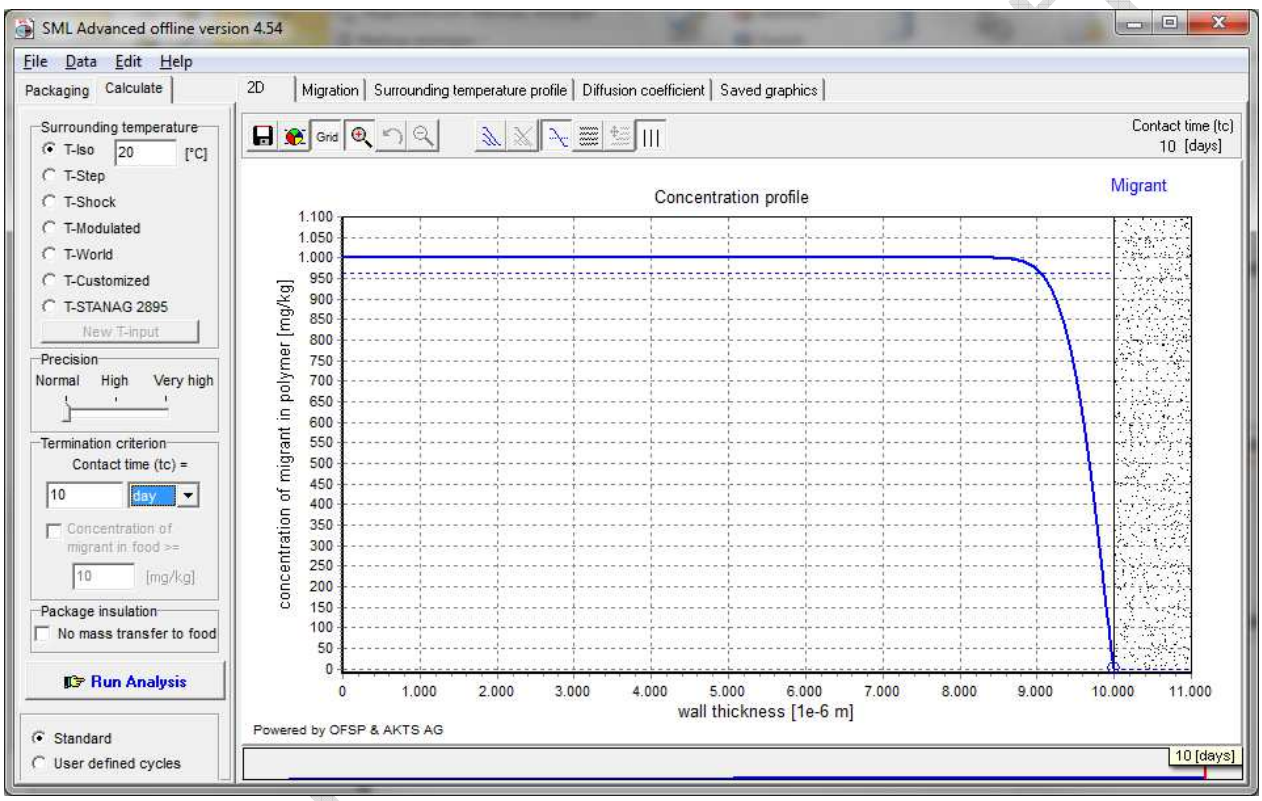
10d @ 20°C

=> 100% layer thickness = 1900 µm
no absolute barrier at thicknesses below 1900 µm

=> 99% layer thickness = 1500 µm

=> 1/2 x 99% layer thickness = 750 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3000 µm
above 3000 µm two sides to be considered for calculation of migration if full immersion testing applied



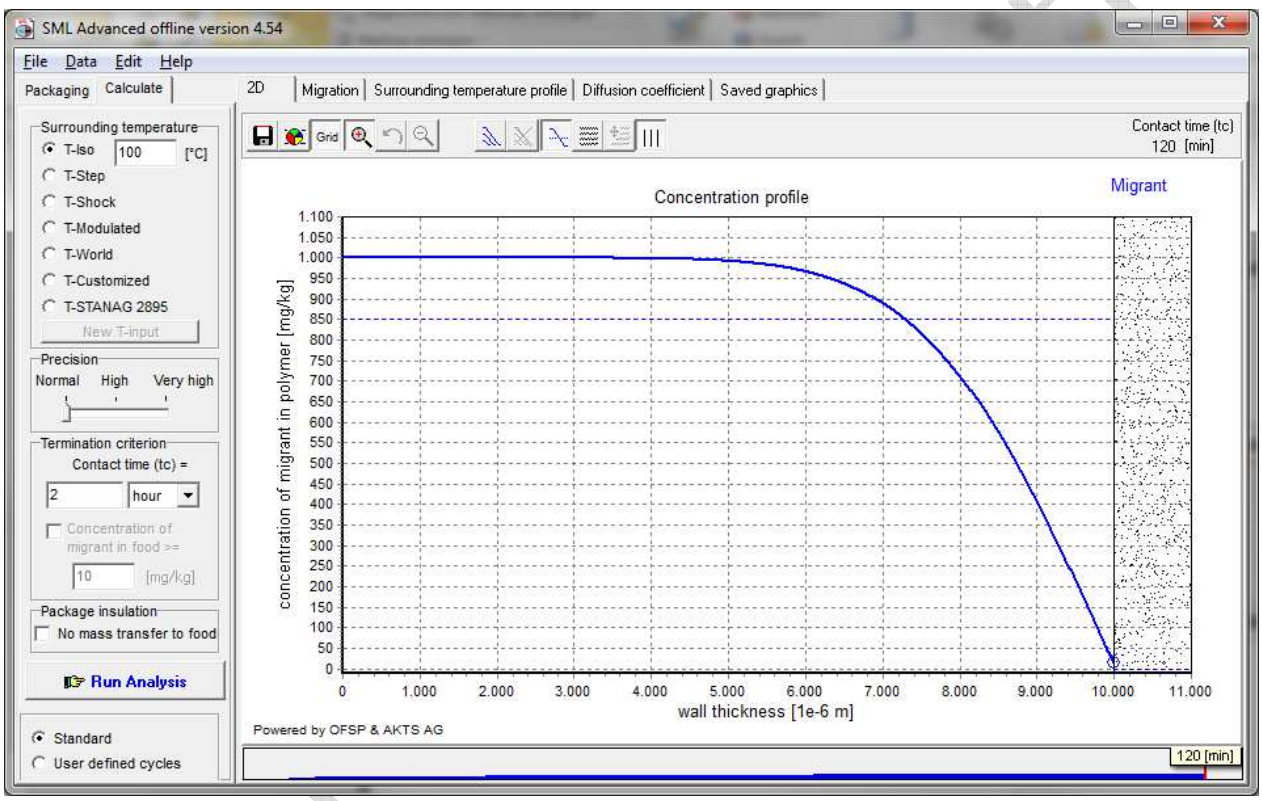
2h @ 100°C

=> 100% layer thickness = 7600 µm
no absolute barrier at thicknesses below 7600 µm

=> 99% layer thickness = 6200 µm

=> 1/2 x 99% layer thickness = 3100 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 12400 µm
above 12400 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

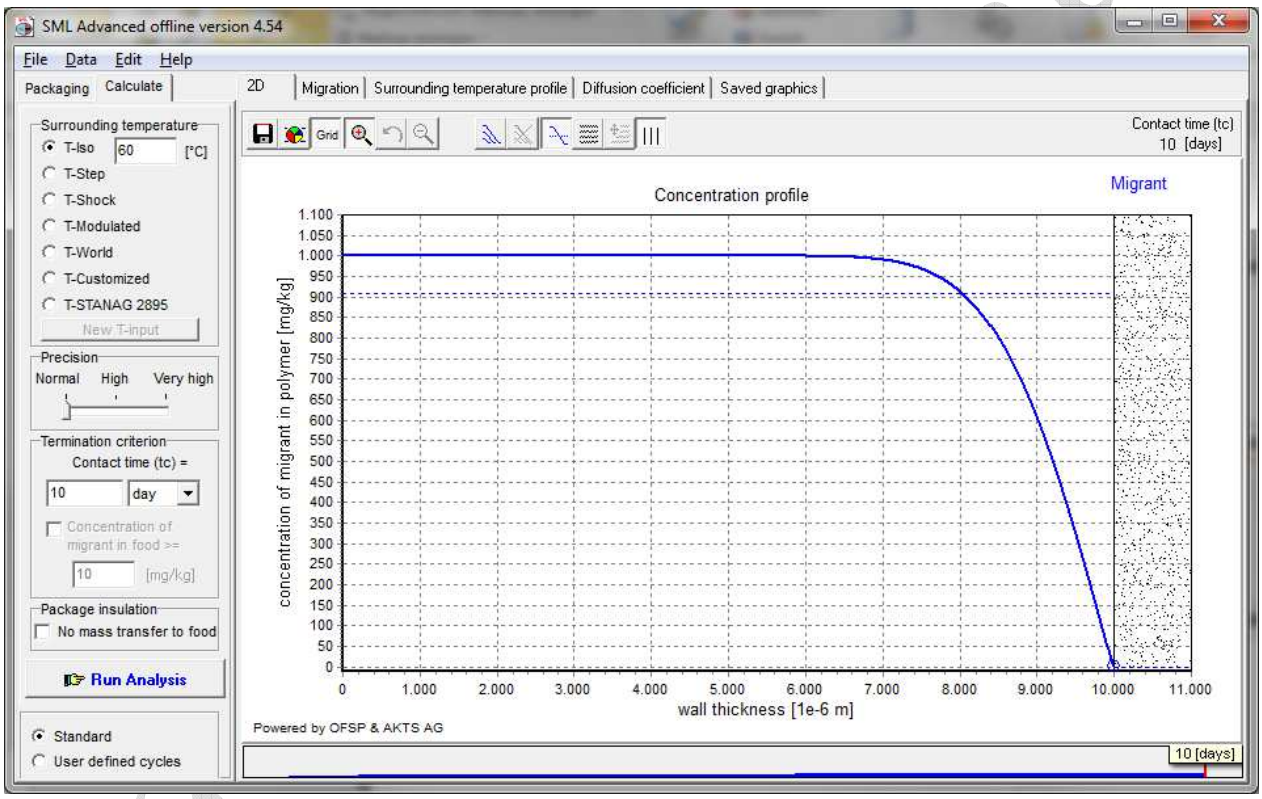
10d @ 60°C

=> 100% layer thickness = 4600 µm
no absolute barrier at thicknesses below 4600 µm

=> 99% layer thickness = 3800 µm

=> 1/2 x 99% layer thickness = 1900 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 7200 µm
above 7200 µm two sides to be considered for calculation of migration if full immersion testing applied



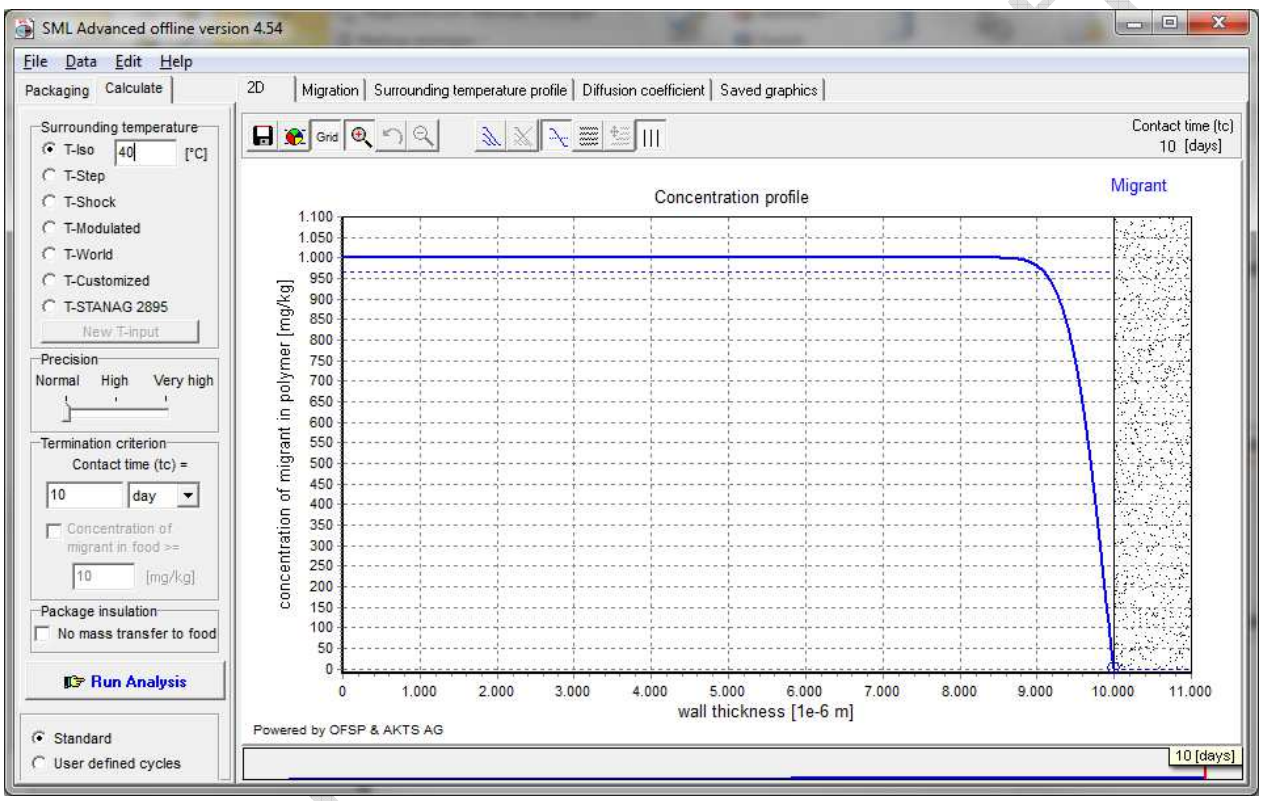
10d @ 40°C

=> 100% layer thickness = 1750 µm
no absolute barrier at thicknesses below 1750 µm

=> 99% layer thickness = 1400 µm

=> 1/2 x 99% layer thickness = 700 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2800 µm
above 2800 µm two sides to be considered for calculation of migration if full immersion testing applied



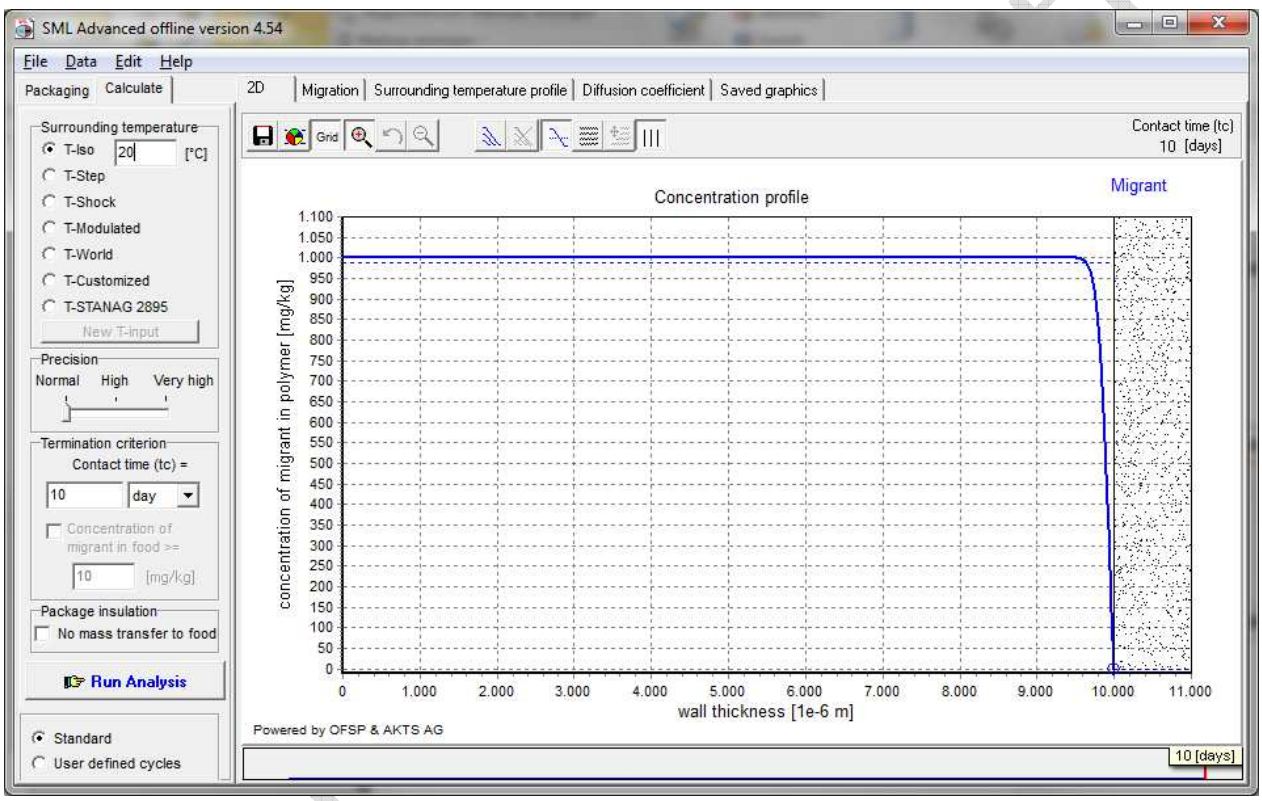
10d @ 20°C

=> 100% layer thickness = 600 µm
no absolute barrier at thicknesses below 600 µm

=> 99% layer thickness = 470 µm

=> 1/2 x 99% layer thickness = 235 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 940 µm
above 940 µm two sides to be considered for calculation of migration if full immersion testing applied



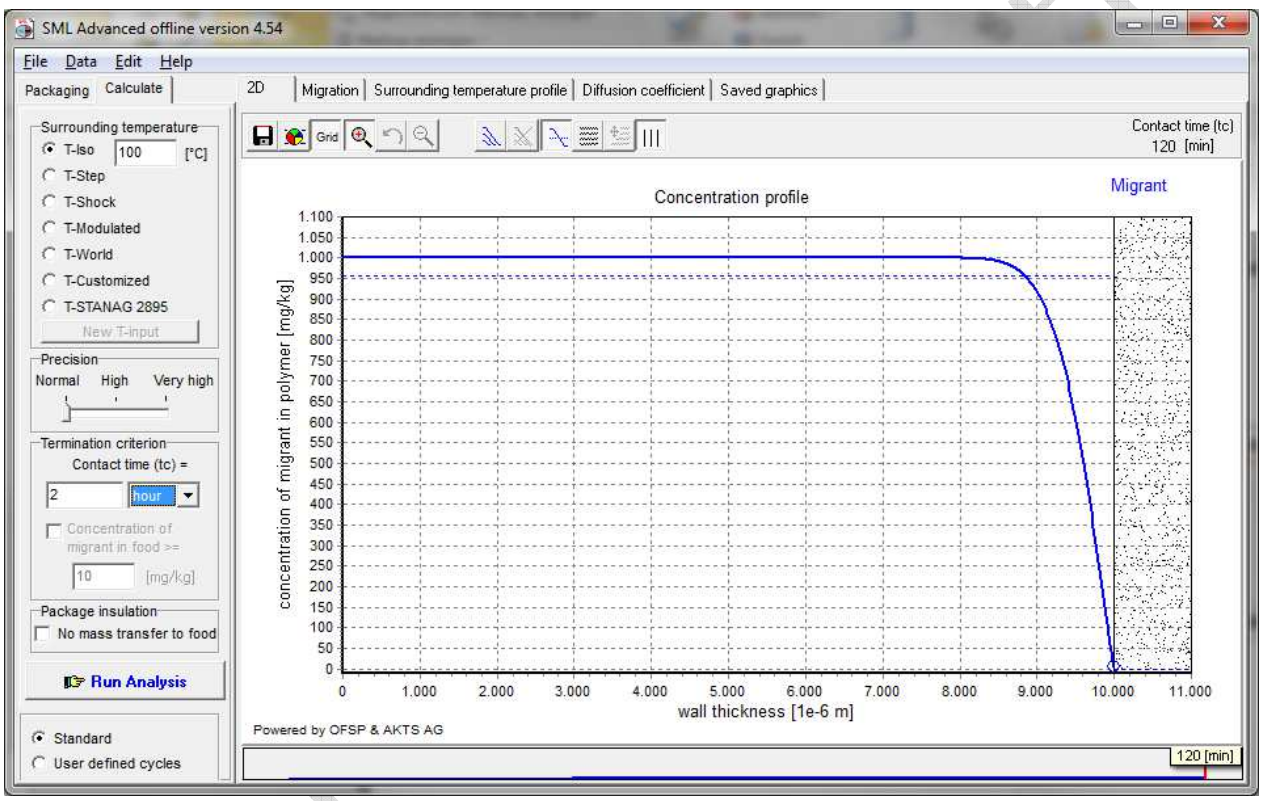
2h @ 100°C

=> 100% layer thickness = 3300 µm
no absolute barrier at thicknesses below 3300 µm

=> 99% layer thickness = 1900 µm

=> 1/2 x 99% layer thickness = 950 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3800 µm
above 3800 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

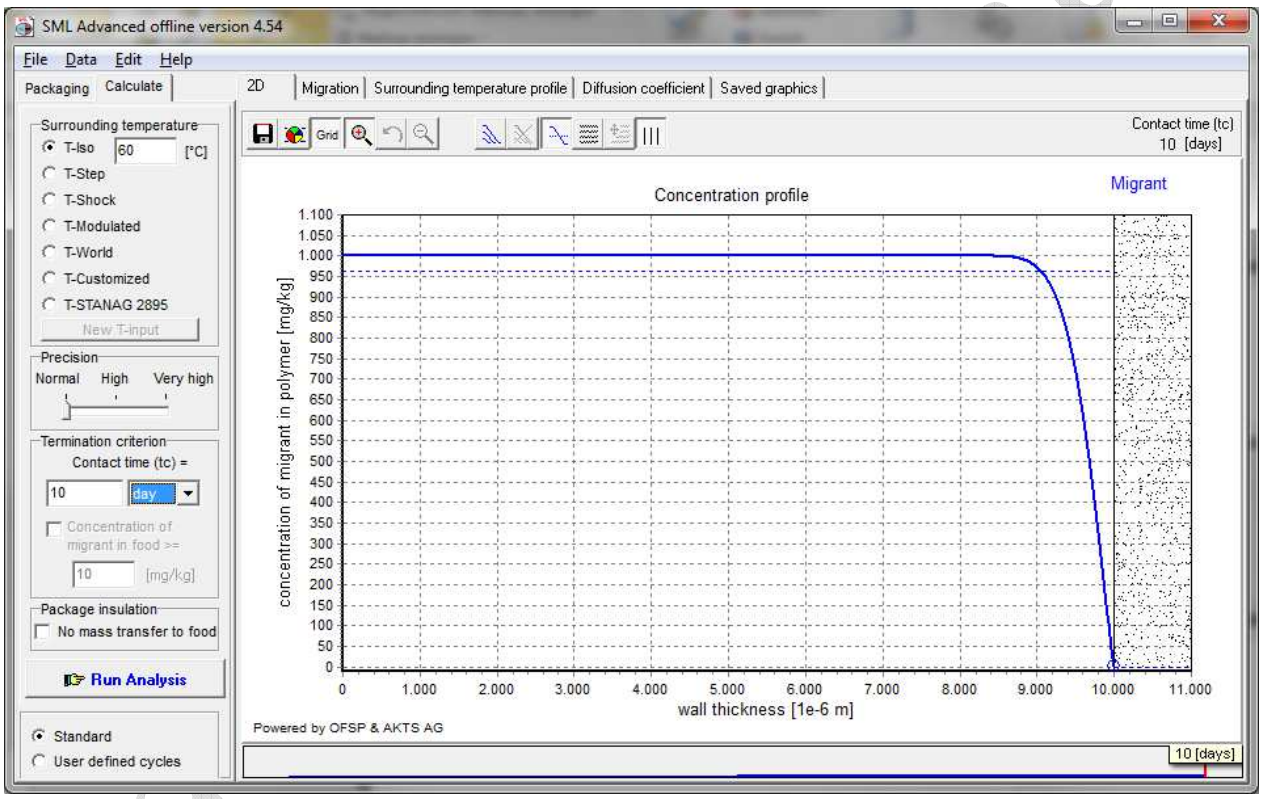
10d @ 60°C

=> 100% layer thickness = 1900 µm
no absolute barrier at thicknesses below 1900 µm

=> 99% layer thickness = 1500 µm

=> 1/2 x 99% layer thickness = 750 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3000 µm
above 3000 µm two sides to be considered for calculation of migration if full immersion testing applied



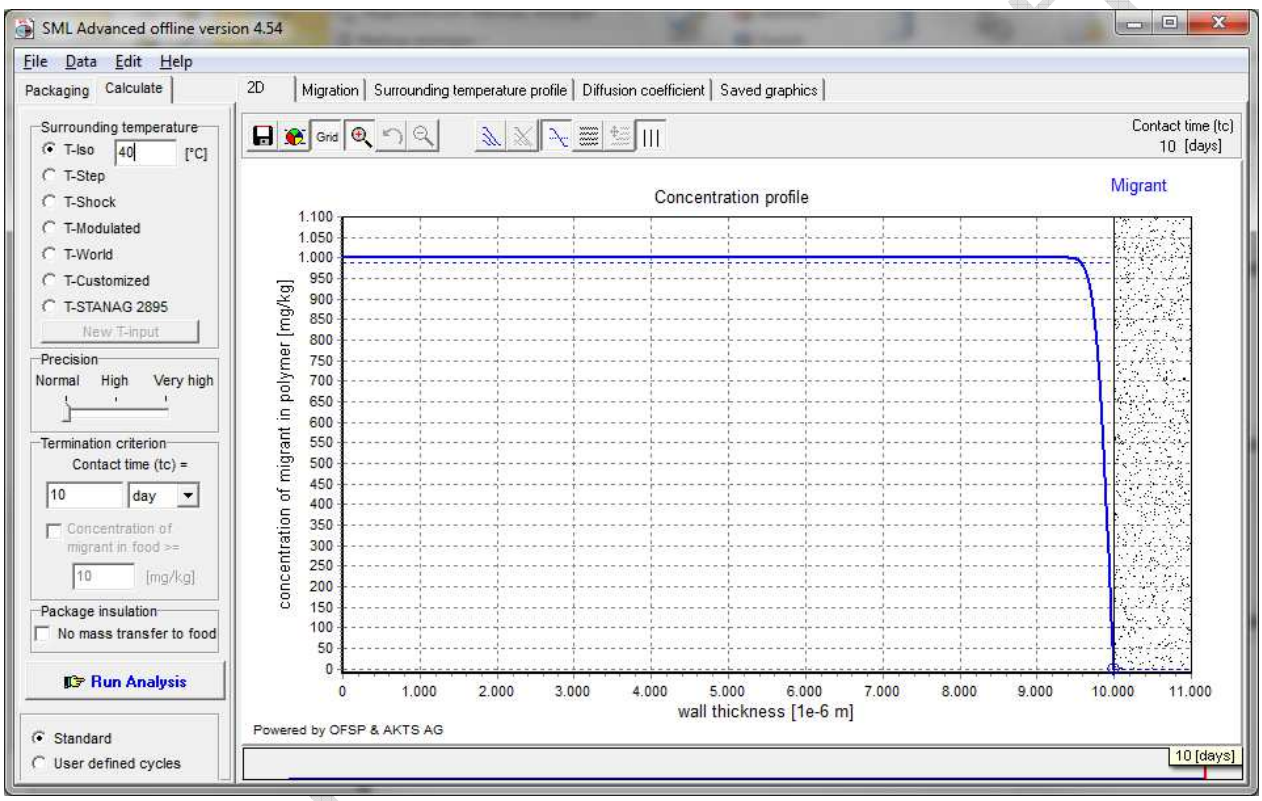
10d @ 40°C

=> 100% layer thickness = 700 µm
no absolute barrier at thicknesses below 700 µm

=> 99% layer thickness = 570 µm

=> 1/2 x 99% layer thickness = 285 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1140 µm
above 1140 µm two sides to be considered for calculation of migration if full immersion testing applied



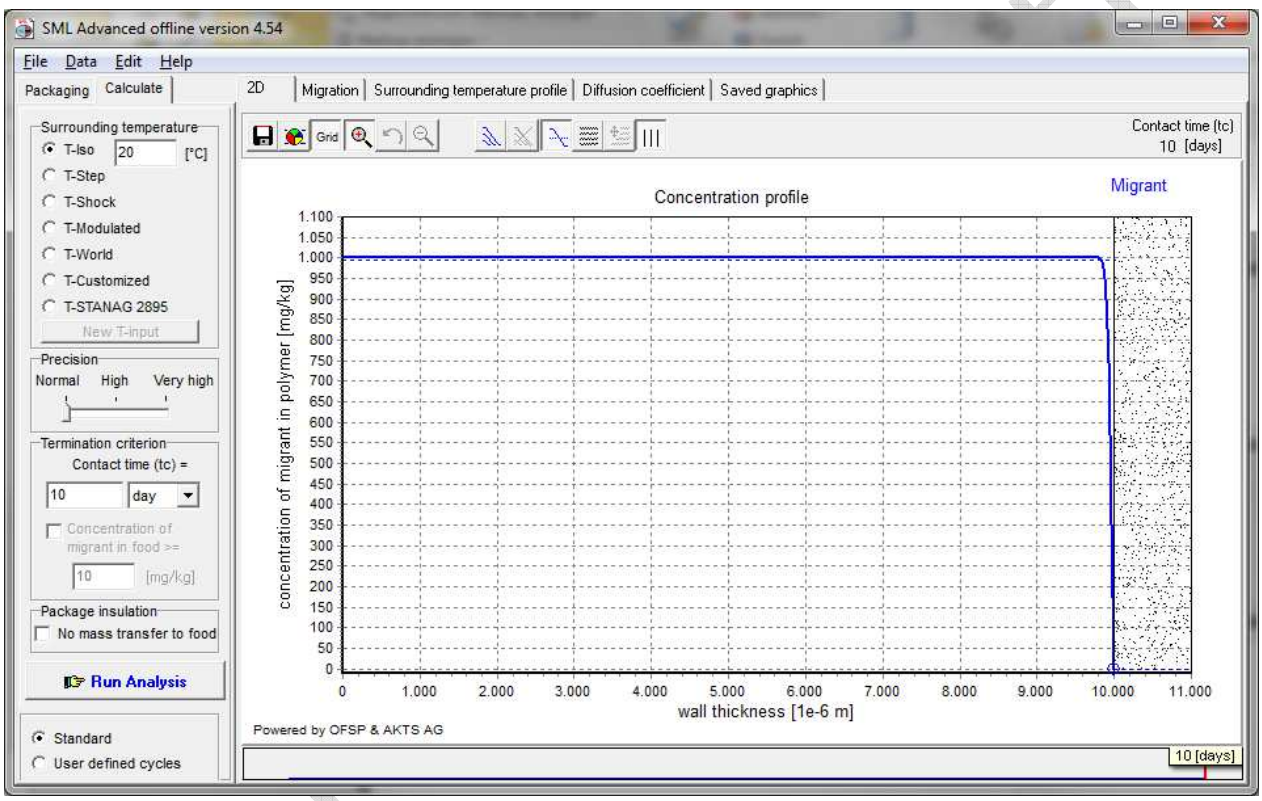
10d @ 20°C

=> 100% layer thickness = 280 µm
no absolute barrier at thicknesses below 280 µm

=> 99% layer thickness = 200 µm

=> 1/2 x 99% layer thickness = 100 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 400 µm
above 400 µm two sides to be considered for calculation of migration if full immersion testing applied



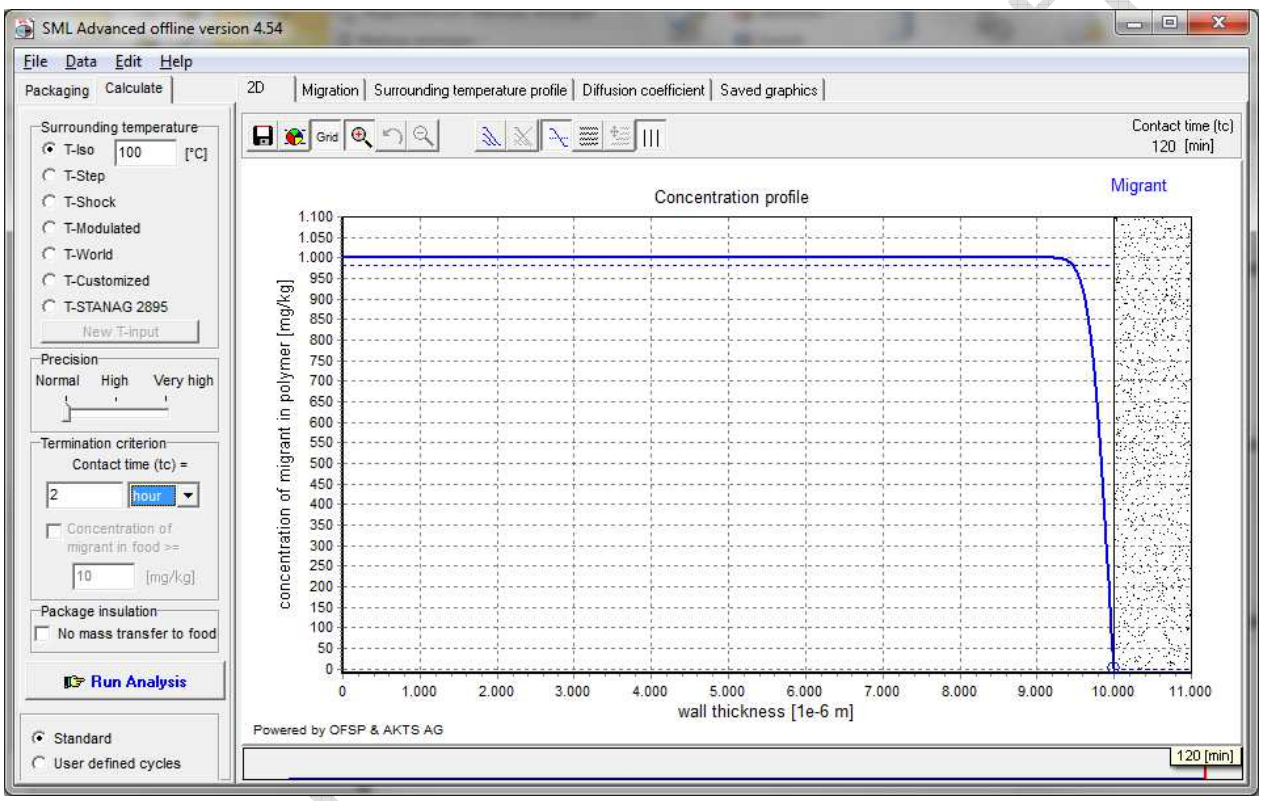
2h @ 100°C

=> 100% layer thickness = 1000 µm
no absolute barrier at thicknesses below 1000 µm

=> 99% layer thickness = 750 µm

=> 1/2 x 99% layer thickness = 375 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1500 µm
above 1500 µm two sides to be considered for calculation of migration if full immersion testing applied



PA6
(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct contact, e.g. plastic multilayer)

► molecular mass 100 - 250 g/mol

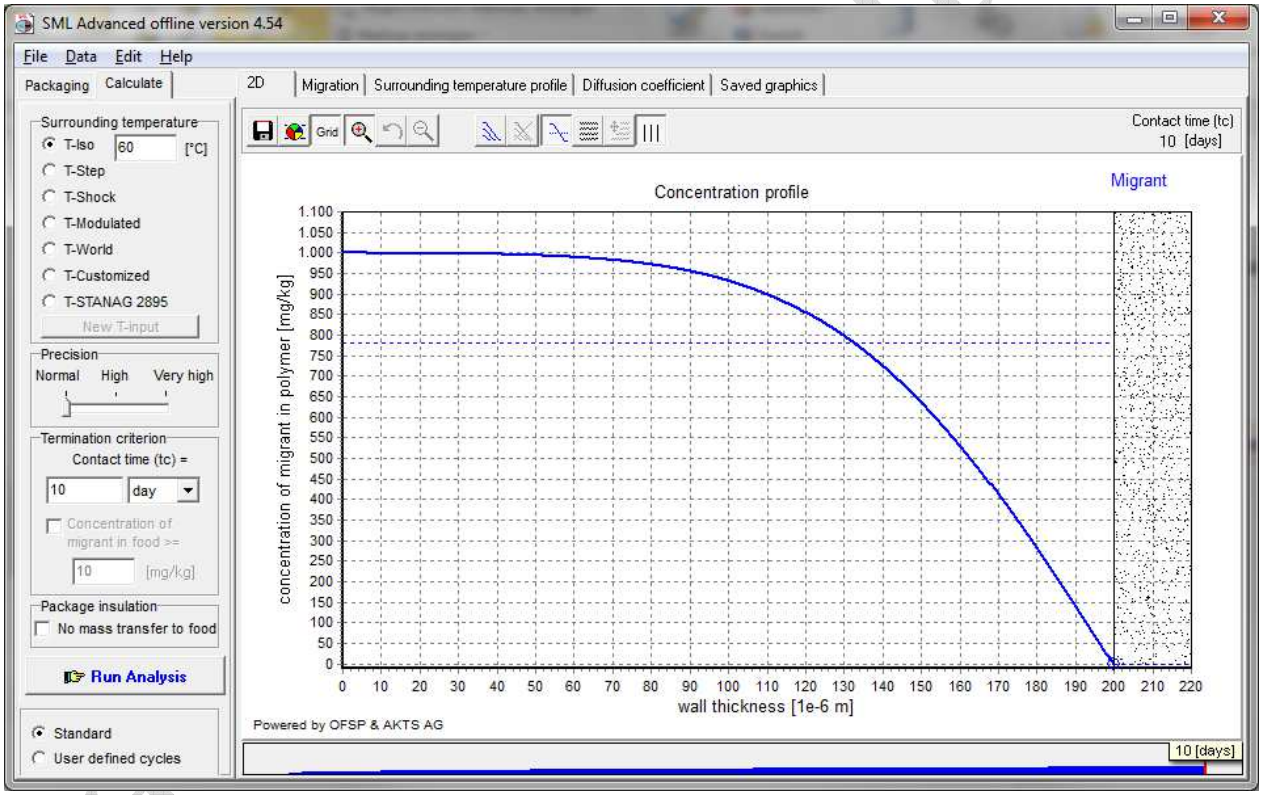
10d @ 60°C

=> 100% layer thickness = 210 µm
no absolute barrier at thicknesses below 210 µm

=> 99% layer thickness = 182 µm

=> 1/2 x 99% layer thickness = 91 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 364 µm
above 364 µm two sides to be considered for calculation of migration if full immersion testing applied



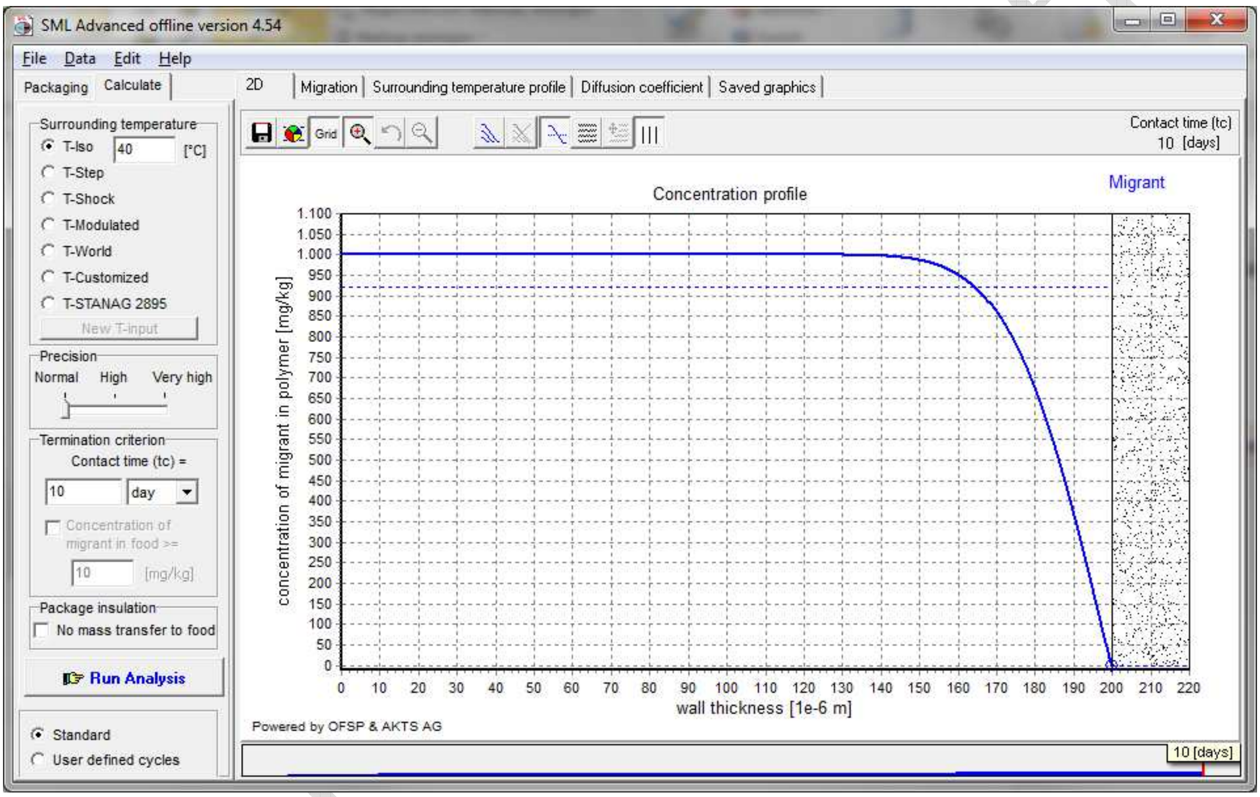
10d @ 40°C

=> 100% layer thickness = 80 µm
no absolute barrier at thicknesses below 80 µm

=> 99% layer thickness = 68 µm

=> 1/2 x 99% layer thickness =34 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 136 µm
above 136 µm two sides to be considered for calculation of migration if full immersion testing applied



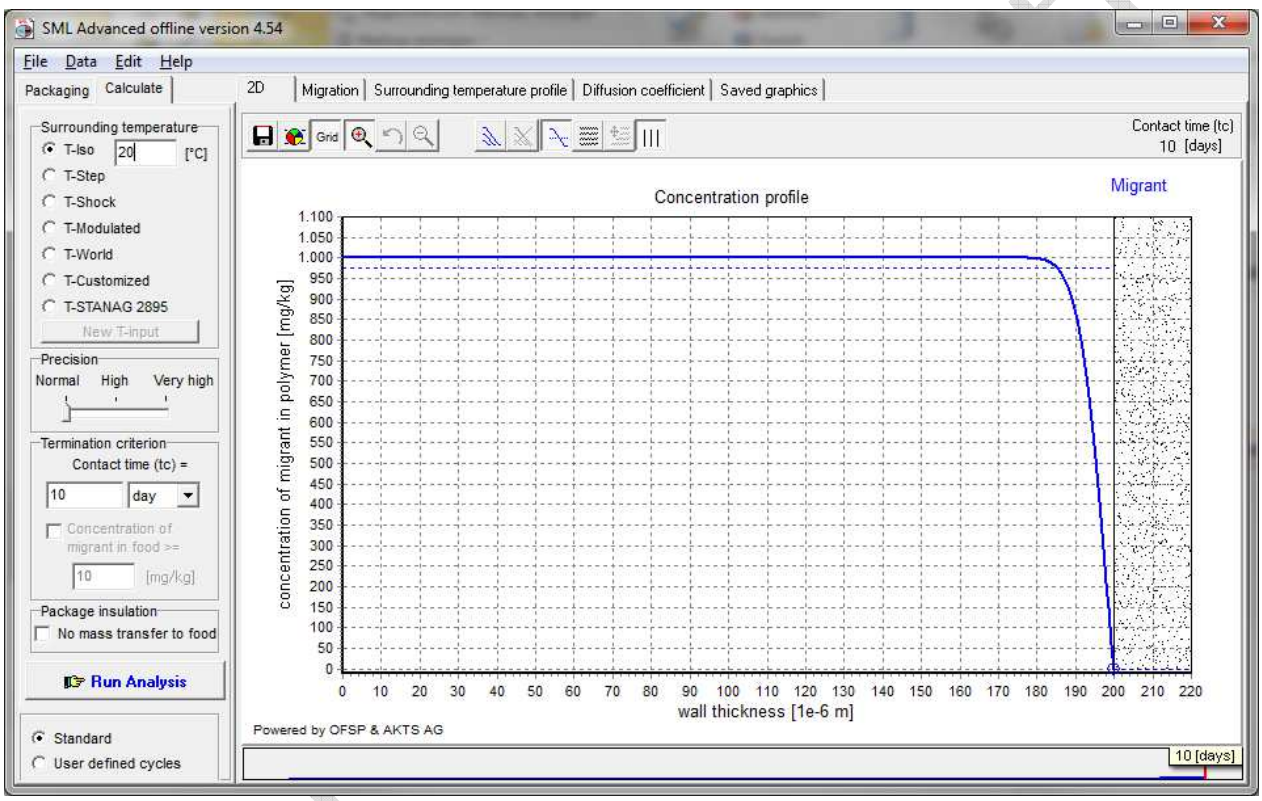
10d @ 20°C

=> 100% layer thickness = 26 µm
no absolute barrier at thicknesses below 26 µm

=> 99% layer thickness = 22 µm

=> 1/2 x 99% layer thickness =11 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 44 µm
above 44 µm two sides to be considered for calculation of migration if full immersion testing applied



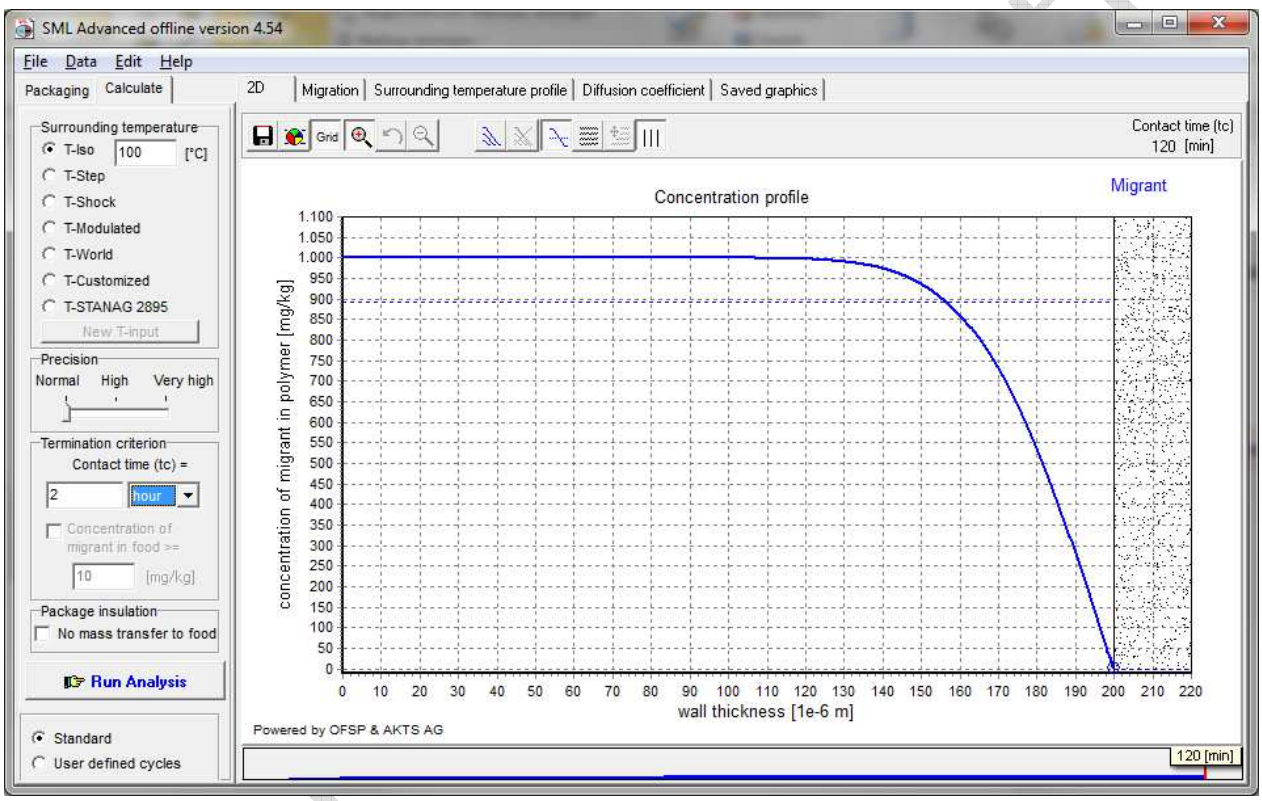
2h @ 100°C

=> 100% layer thickness = 105 µm
no absolute barrier at thicknesses below 105 µm

=> 99% layer thickness = 88 µm

=> 1/2 x 99% layer thickness =44 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 176 µm
above 176 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

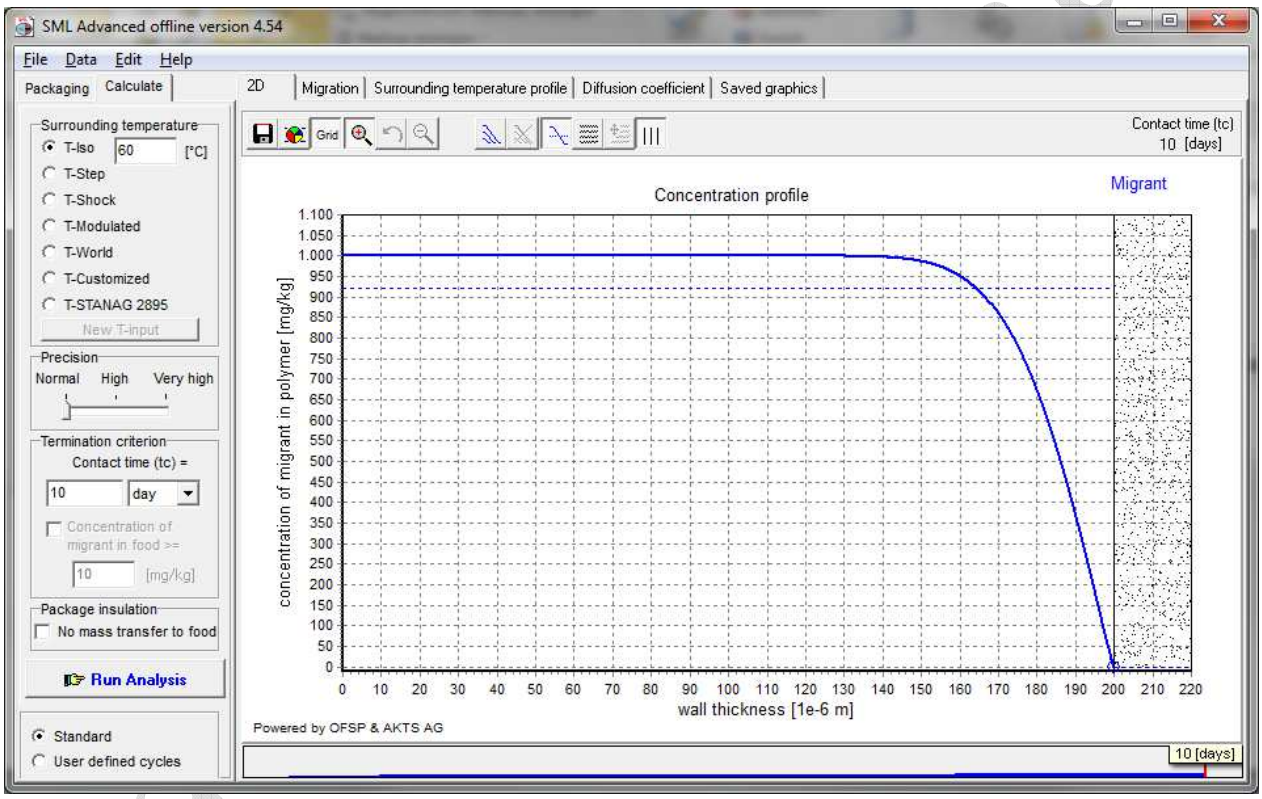
10d @ 60°C

=> 100% layer thickness = 82 µm
no absolute barrier at thicknesses below 82 µm

=> 99% layer thickness = 66 µm

=> 1/2 x 99% layer thickness =33 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 132 µm
above 132 µm two sides to be considered for calculation of migration if full immersion testing applied



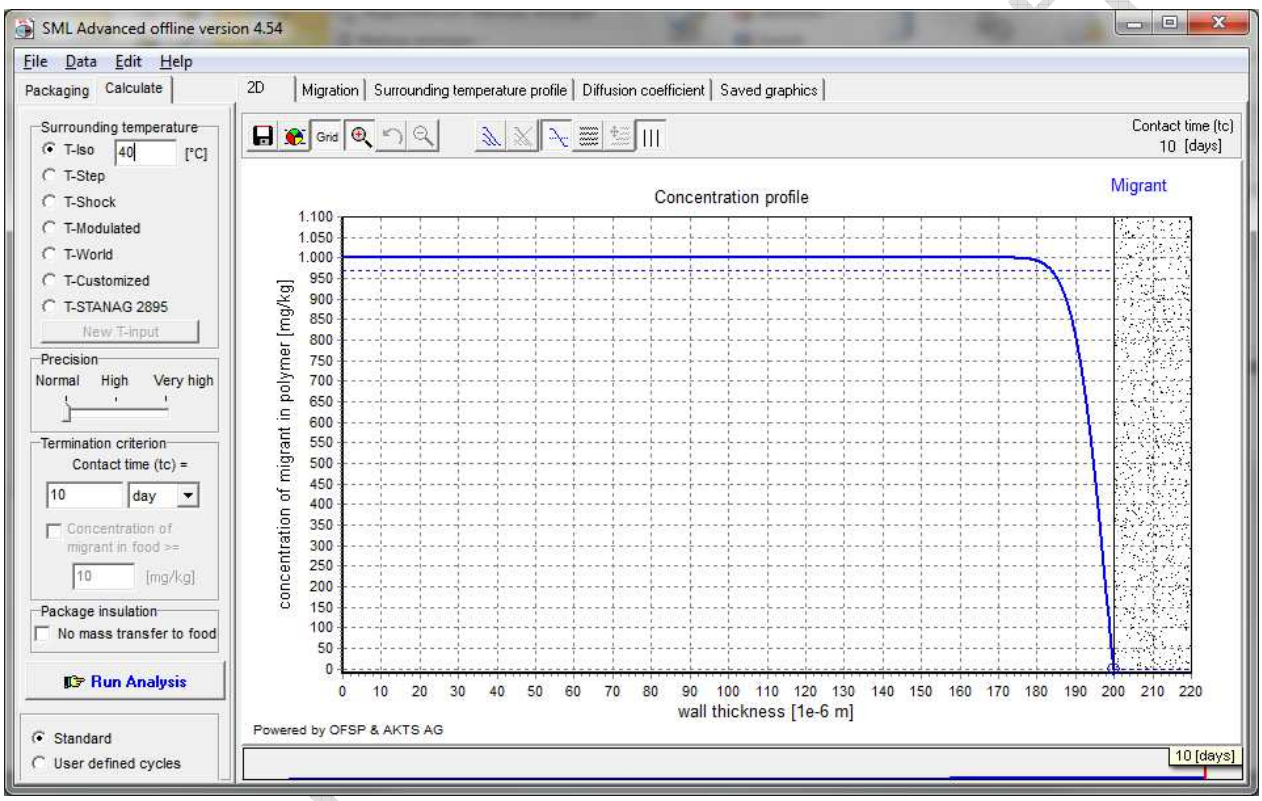
10d @ 40°C

=> 100% layer thickness = 32 µm
no absolute barrier at thicknesses below 32 µm

=> 99% layer thickness = 25 µm

=> 1/2 x 99% layer thickness = 12.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 50 µm
above 50 µm two sides to be considered for calculation of migration if full immersion testing applied



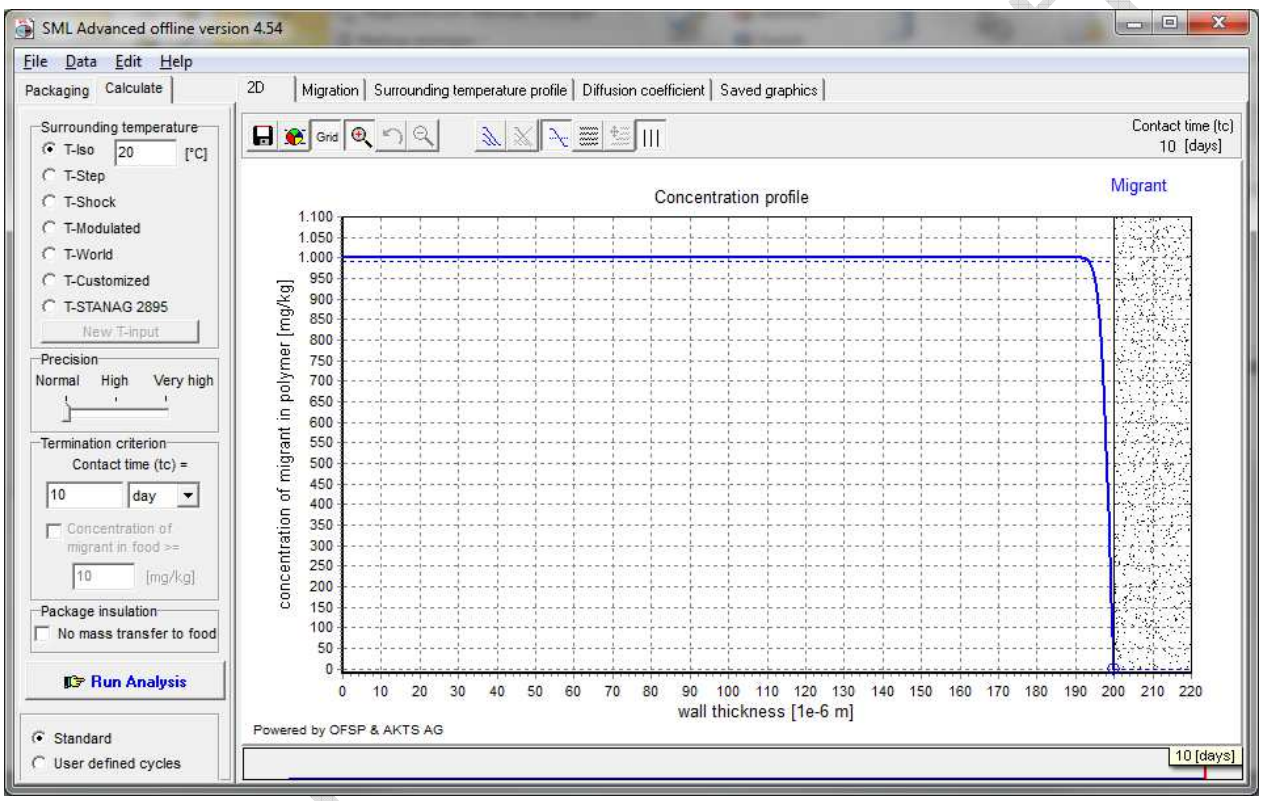
10d @ 20°C

=> 100% layer thickness = 11 µm
no absolute barrier at thicknesses below 11 µm

=> 99% layer thickness = 8.2 µm

=> 1/2 x 99% layer thickness = 4.1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 16.4 µm
above 16.4 µm two sides to be considered for calculation of migration if full immersion testing applied



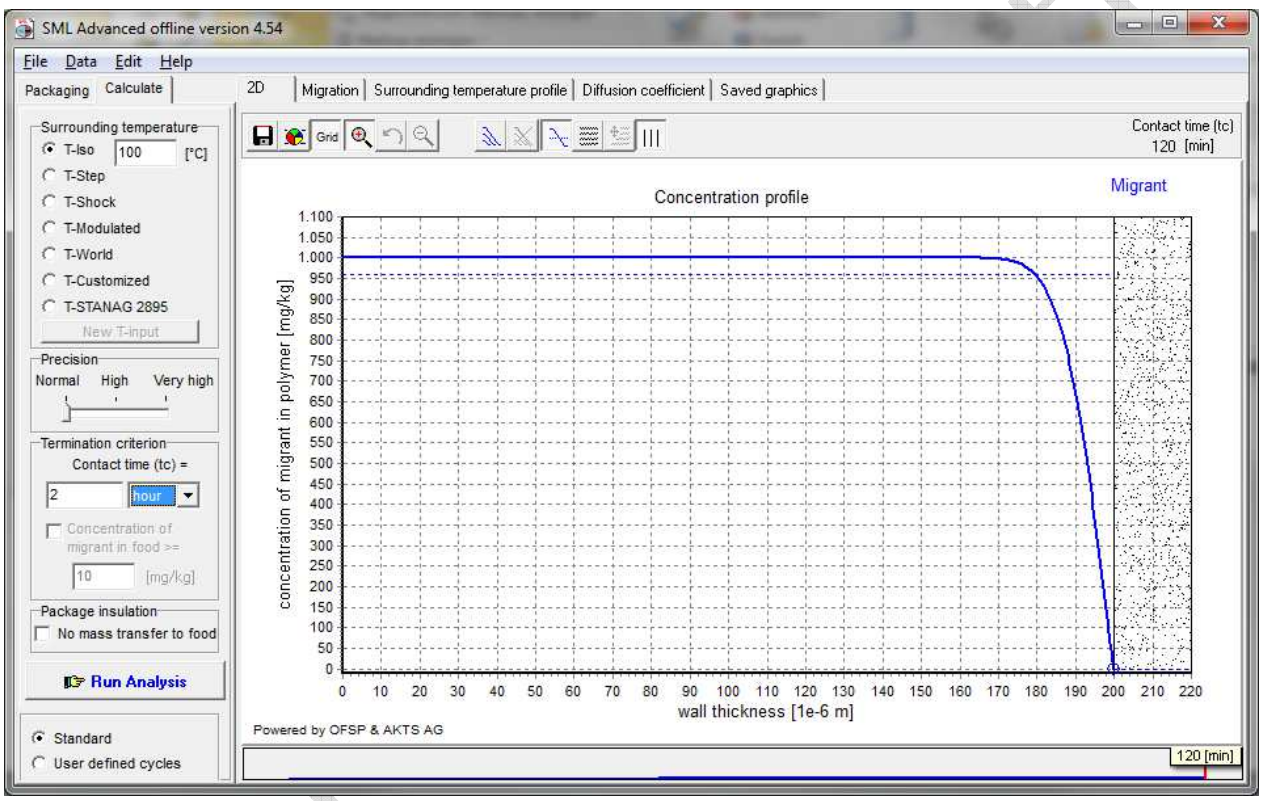
2h @ 100°C

=> 100% layer thickness = 40 µm
no absolute barrier at thicknesses below 40 µm

=> 99% layer thickness = 33 µm

=> 1/2 x 99% layer thickness = 16.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 66 µm
above 66 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

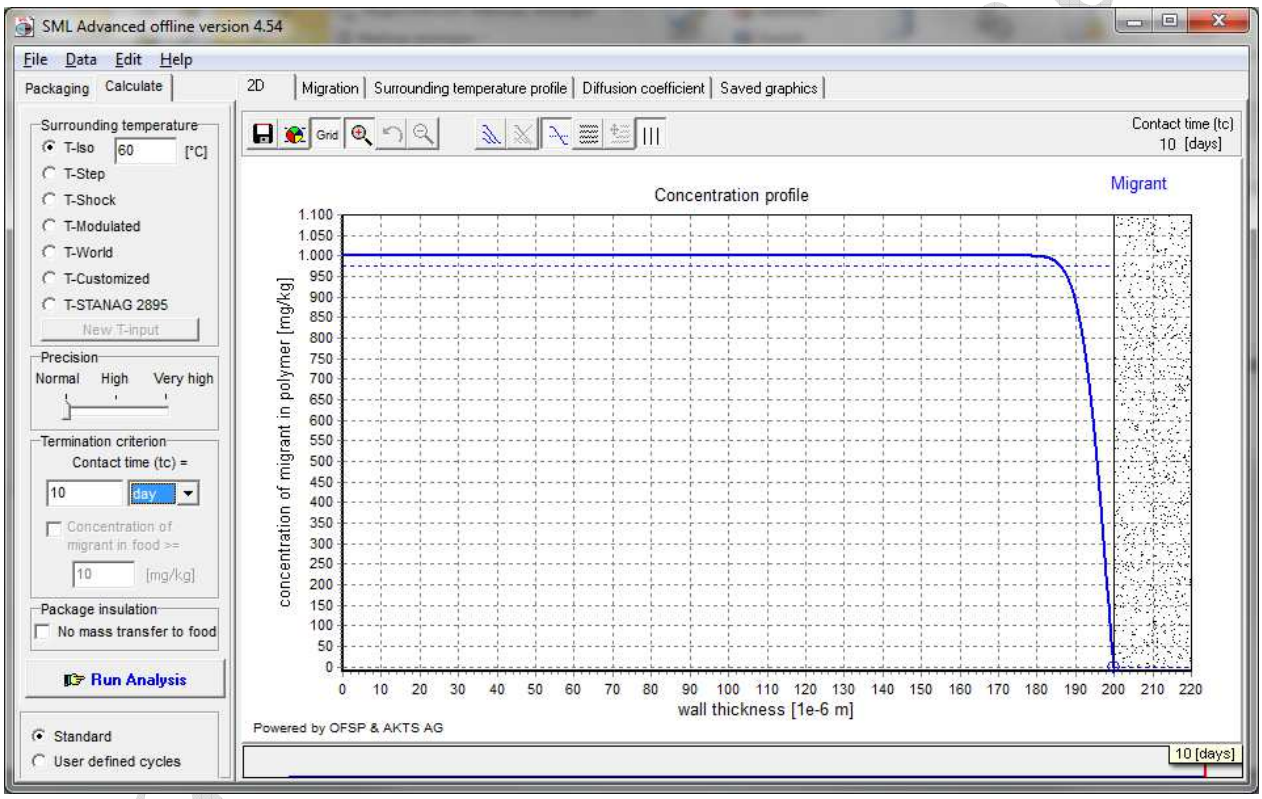
10d @ 60°C

=> 100% layer thickness = 25 µm
no absolute barrier at thicknesses below 25 µm

=> 99% layer thickness = 20 µm

=> 1/2 x 99% layer thickness = 10 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 40 µm
above 40 µm two sides to be considered for calculation of migration if full immersion testing applied



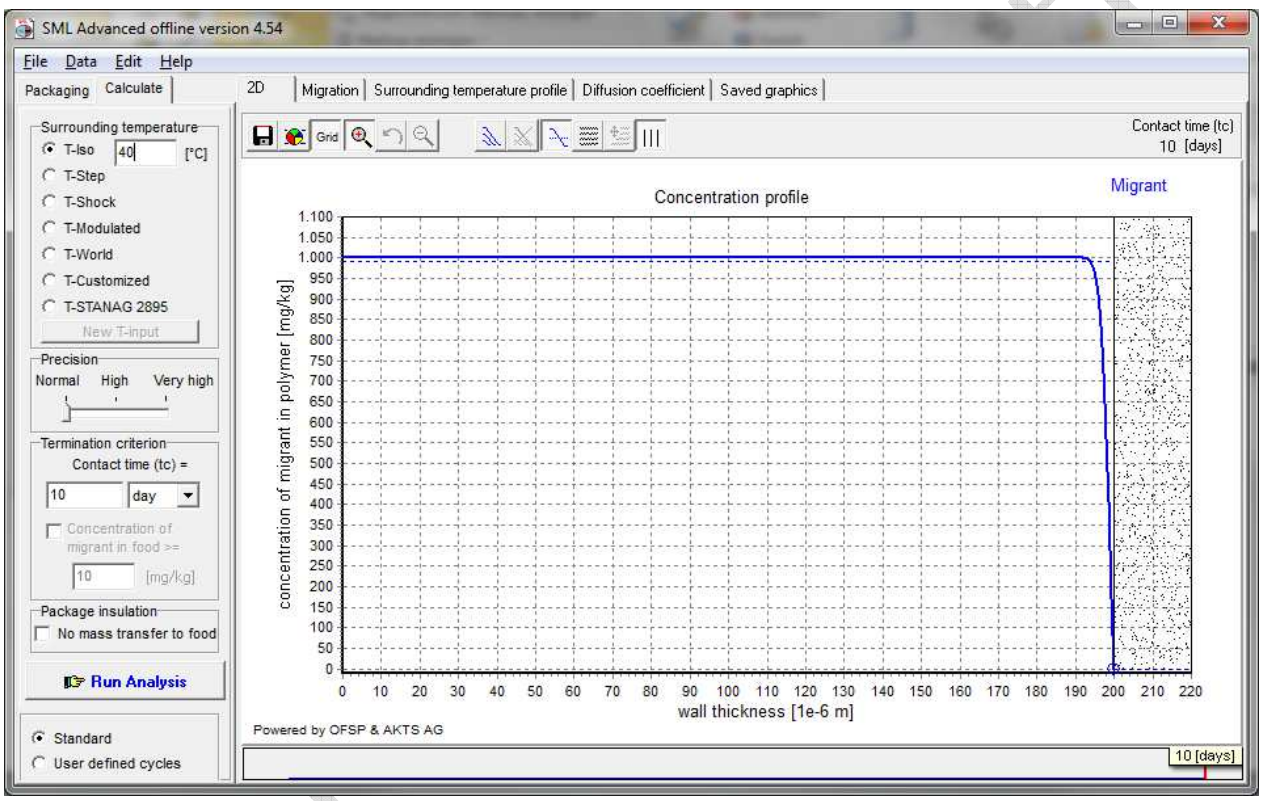
10d @ 40°C

=> 100% layer thickness = 10.3 µm
no absolute barrier at thicknesses below 10.3 µm

=> 99% layer thickness = 7.6 µm

=> 1/2 x 99% layer thickness = 3.8 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 15.2 µm
above 15.2 µm two sides to be considered for calculation of migration if full immersion testing applied



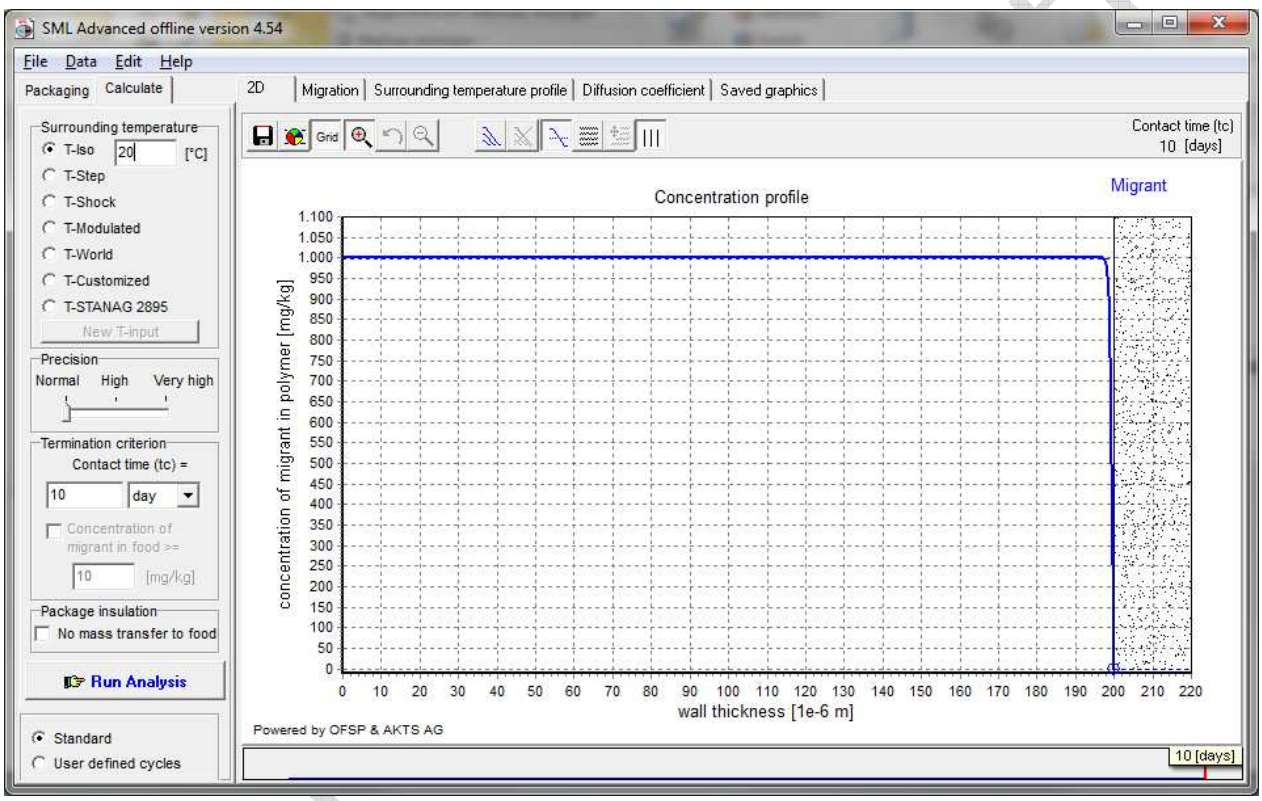
10d @ 20°C

=> 100% layer thickness = 4 µm
no absolute barrier at thicknesses below 4 µm

=> 99% layer thickness = 2.8 µm

=> 1/2 x 99% layer thickness = 1.4 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 5.6 µm
above 5.6 µm two sides to be considered for calculation of migration if full immersion testing applied



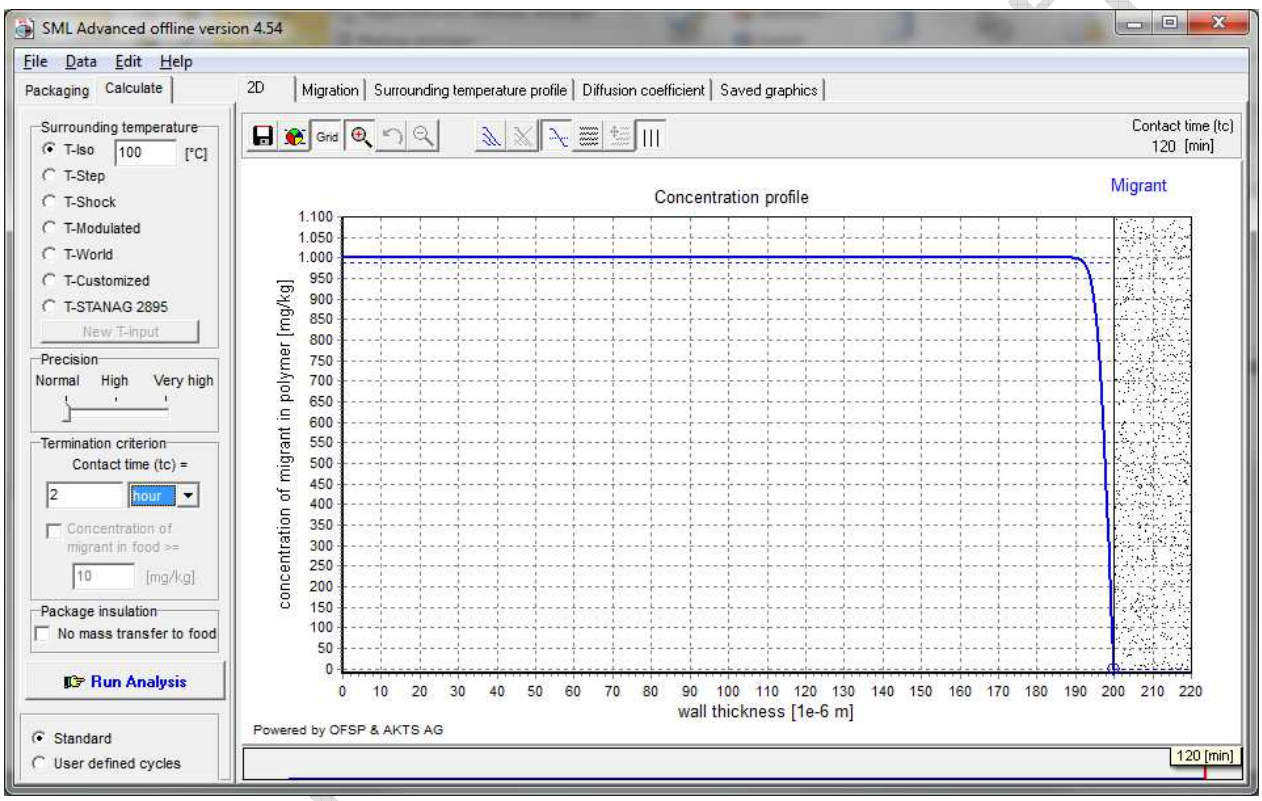
2h @ 100°C

=> 100% layer thickness = 14 µm
no absolute barrier at thicknesses below 14 µm

=> 99% layer thickness = 10.2 µm

=> 1/2 x 99% layer thickness = 5.1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 20.4 µm
above 20.4 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

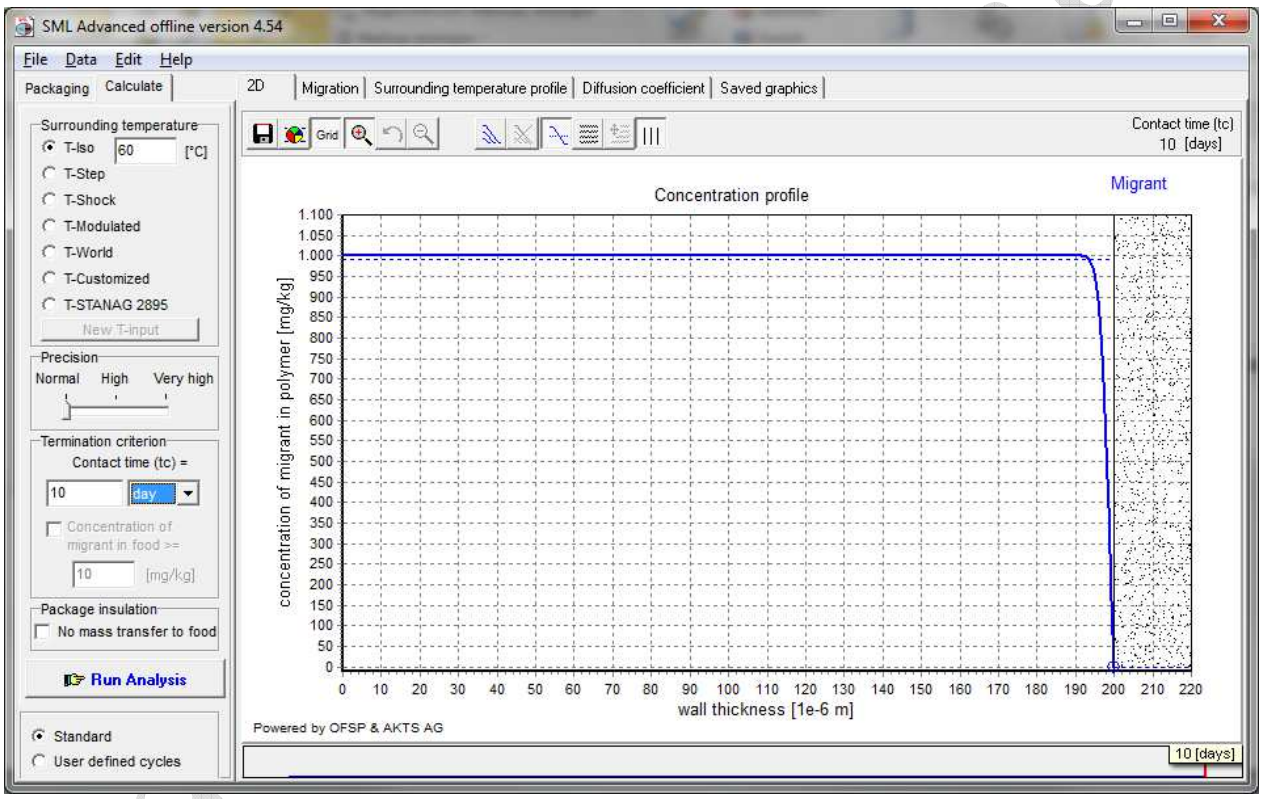
10d @ 60°C

=> 100% layer thickness = 10 µm
no absolute barrier at thicknesses below 10 µm

=> 99% layer thickness = 8 µm

=> 1/2 x 99% layer thickness = 4 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 16 µm
above 16 µm two sides to be considered for calculation of migration if full immersion testing applied



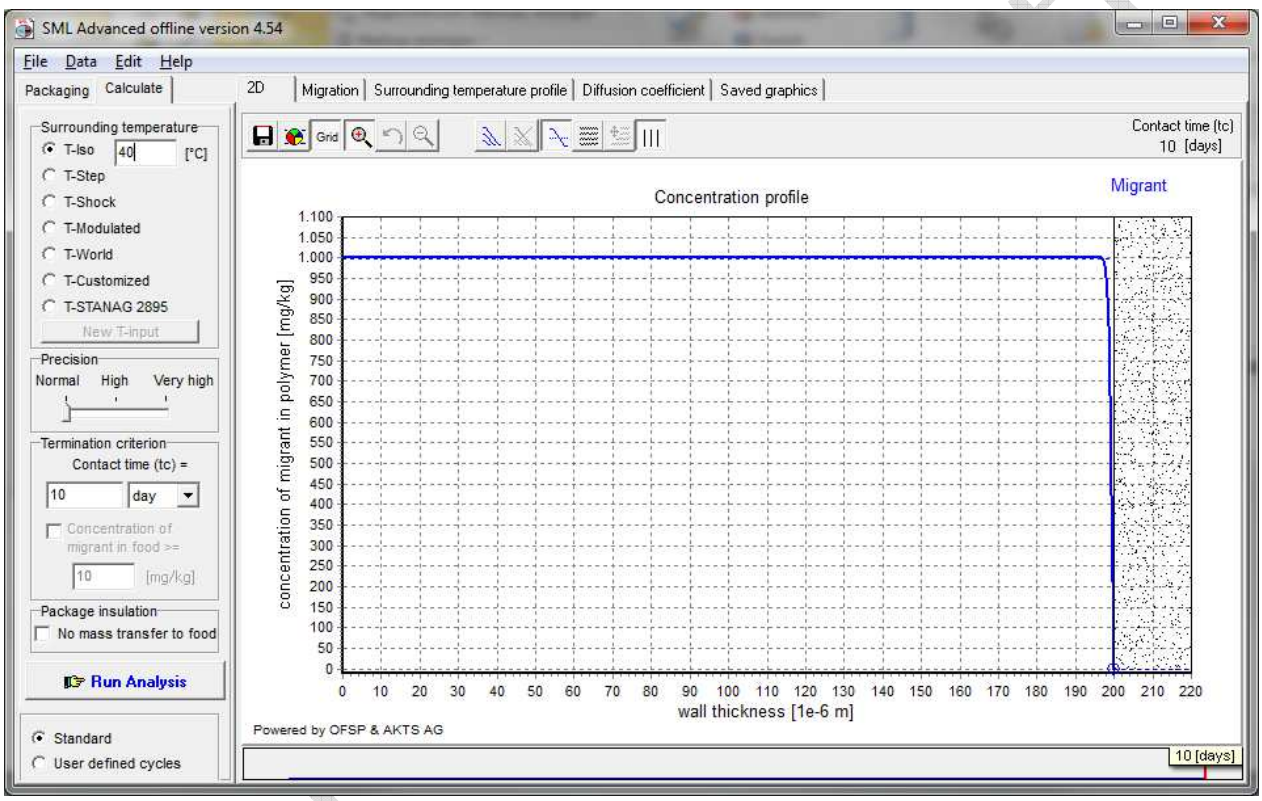
10d @ 40°C

=> 100% layer thickness = 4.2 µm
no absolute barrier at thicknesses below 4.2 µm

=> 99% layer thickness = 3.4 µm

=> 1/2 x 99% layer thickness = 1.7 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 6.8 µm
above 6.8 µm two sides to be considered for calculation of migration if full immersion testing applied



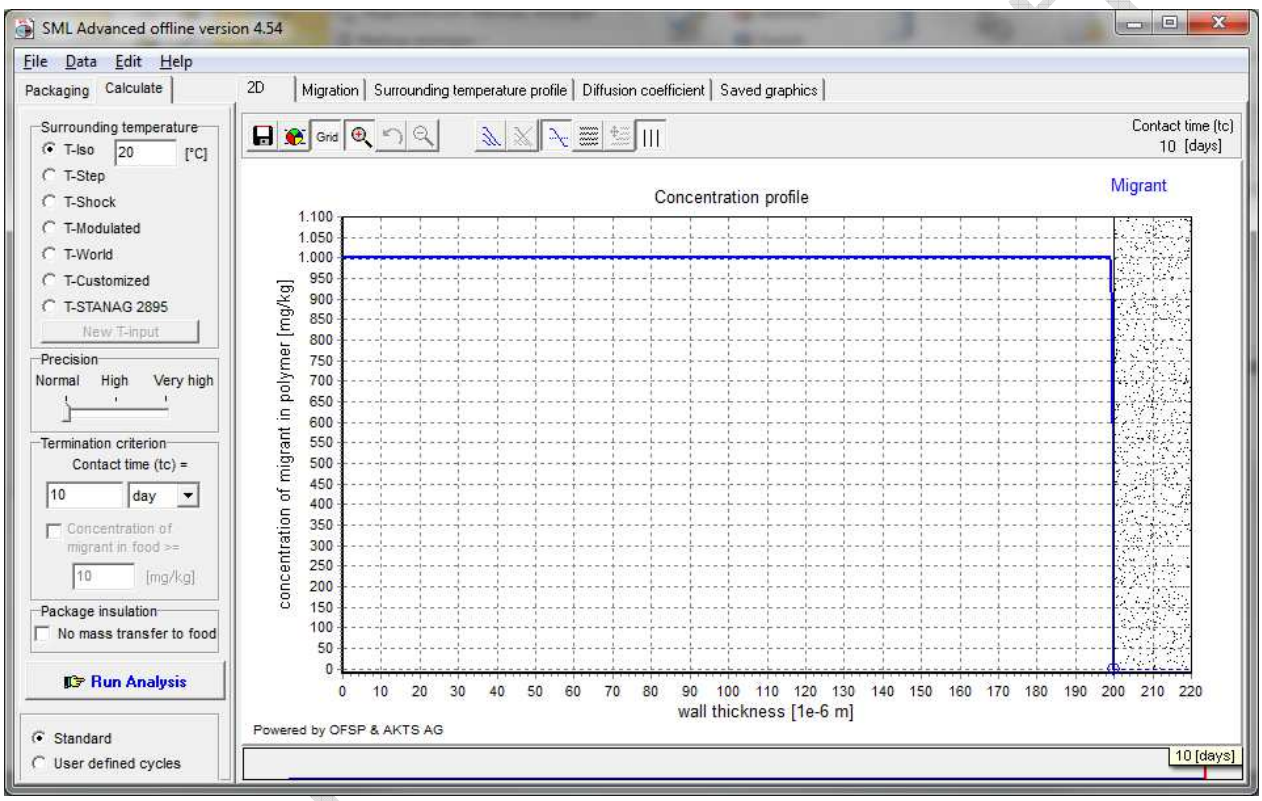
10d @ 20°C

=> 100% layer thickness = 1.8 µm
no absolute barrier at thicknesses below 1.8 µm

=> 99% layer thickness = 1.2 µm

=> 1/2 x 99% layer thickness = 0.6 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 2.4 µm
above 2.4 µm two sides to be considered for calculation of migration if full immersion testing applied



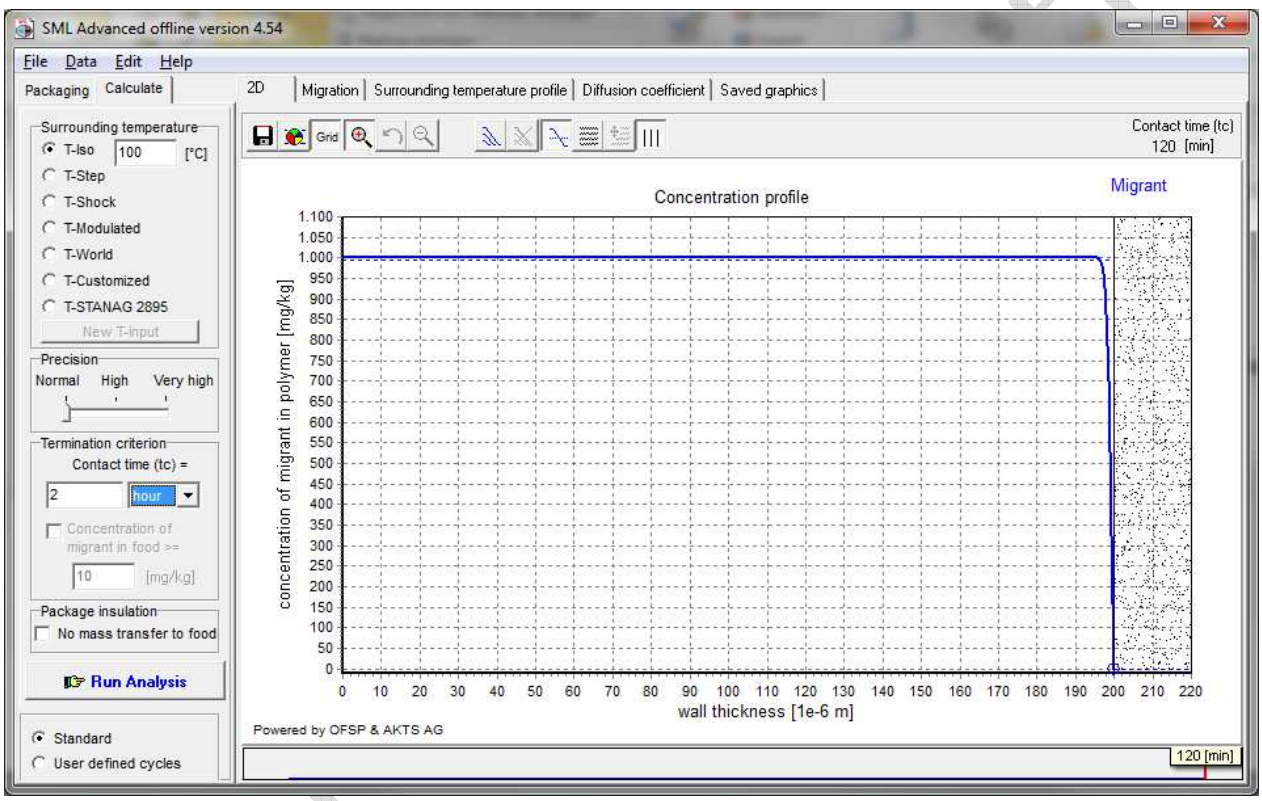
2h @ 100°C

=> 100% layer thickness = 5.5 µm
no absolute barrier at thicknesses below 5.5 µm

=> 99% layer thickness = 4.4 µm

=> 1/2 x 99% layer thickness = 2.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 8.8 µm
above 8.8 µm two sides to be considered for calculation of migration if full immersion testing applied



PA6,6
(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct contact, e.g. plastic multilayer)

► molecular mass 100 - 250 g/mol

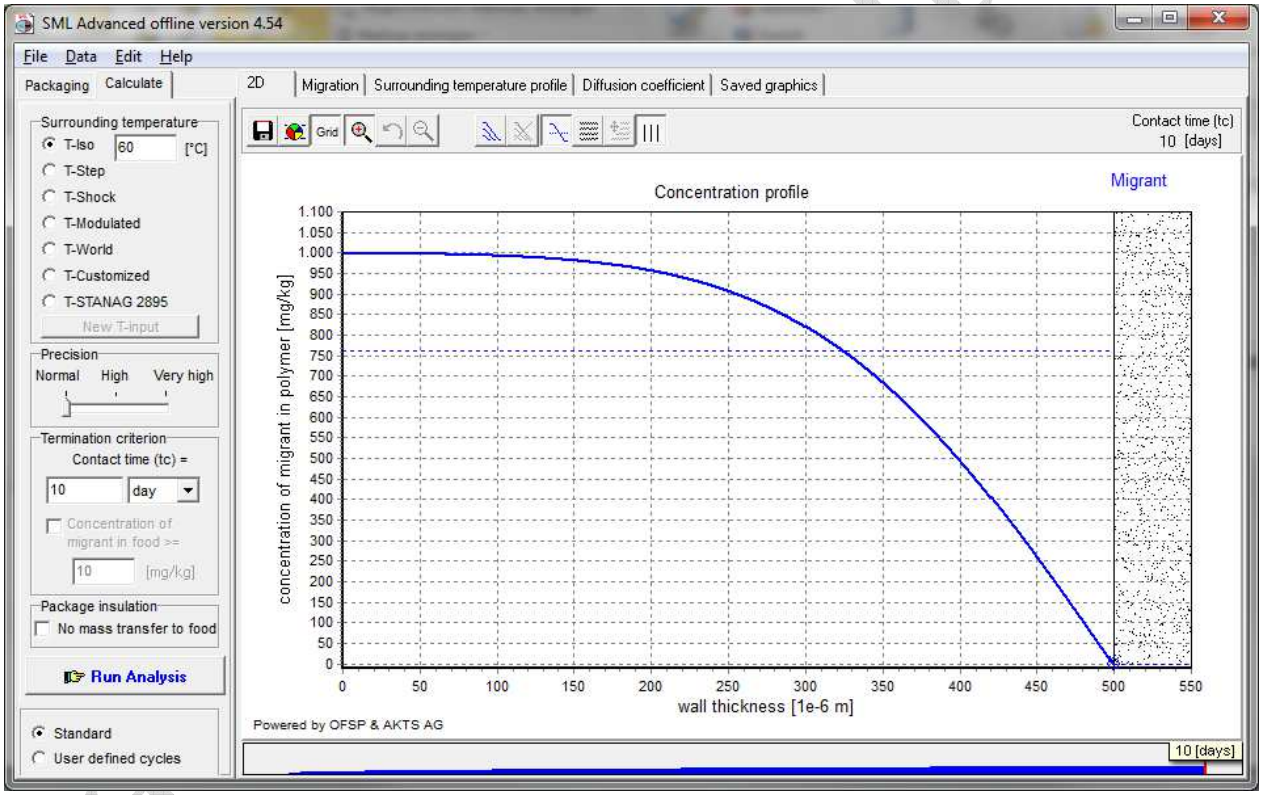
10d @ 60°C

=> 100% layer thickness = 565 µm
no absolute barrier at thicknesses below 565 µm

=> 99% layer thickness = 490 µm

=> 1/2 x 99% layer thickness = 245 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 980 µm
above 980 µm two sides to be considered for calculation of migration if full immersion testing applied



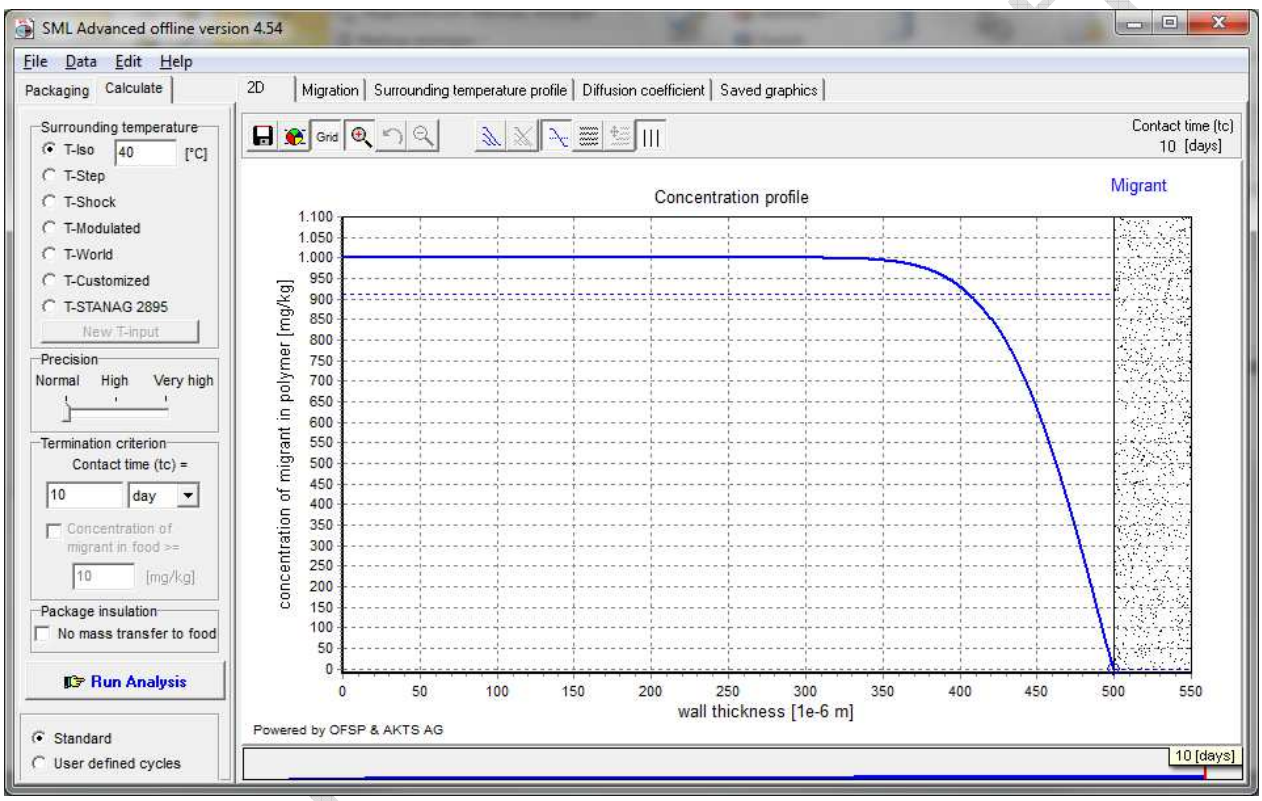
10d @ 40°C

=> 100% layer thickness = 220 µm
no absolute barrier at thicknesses below 220 µm

=> 99% layer thickness = 180 µm

=> 1/2 x 99% layer thickness =90 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 360 µm
above 360 µm two sides to be considered for calculation of migration if full immersion testing applied



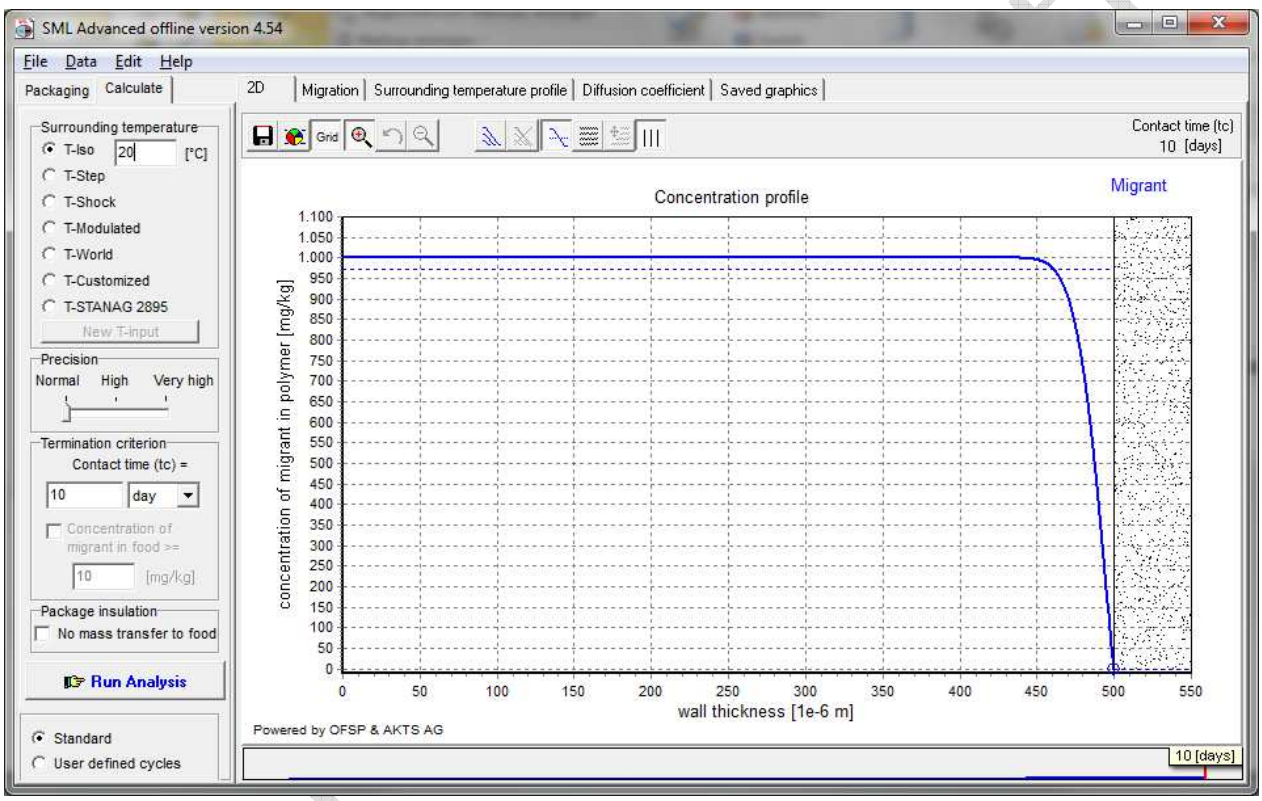
10d @ 20°C

=> 100% layer thickness = 76 µm
no absolute barrier at thicknesses below 76 µm

=> 99% layer thickness = 58 µm

=> 1/2 x 99% layer thickness = 29 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 116 µm
above 116 µm two sides to be considered for calculation of migration if full immersion testing applied



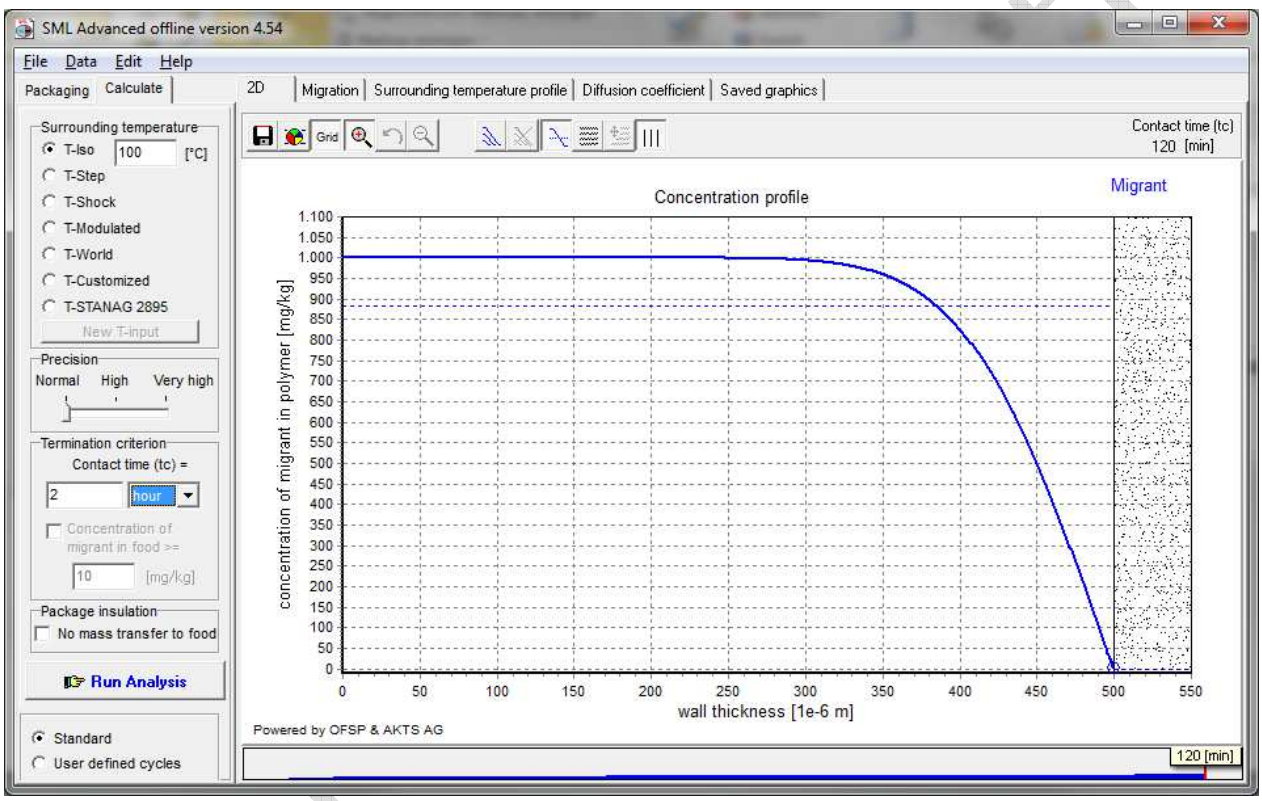
2h @ 100°C

=> 100% layer thickness = 300 µm
no absolute barrier at thicknesses below 300 µm

=> 99% layer thickness = 240 µm

=> 1/2 x 99% layer thickness = 120 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 480 µm
above 480 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

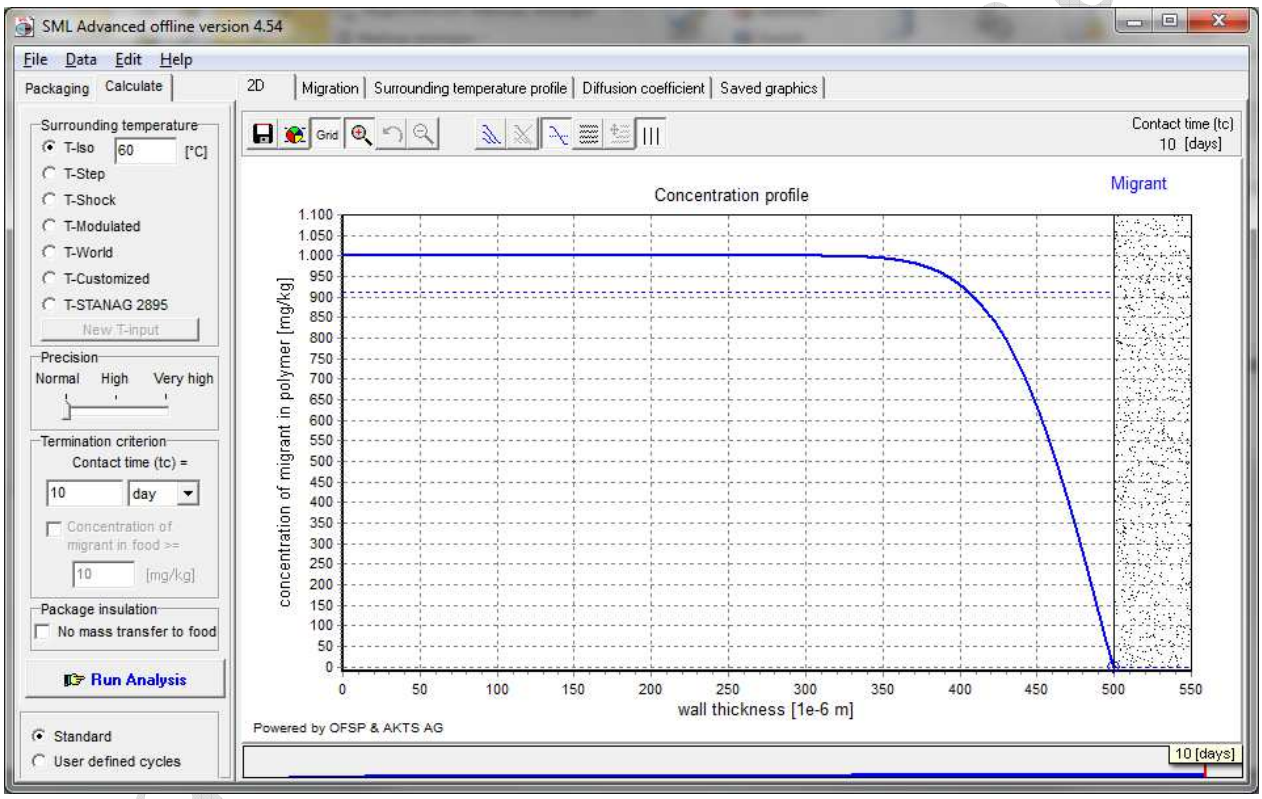
10d @ 60°C

=> 100% layer thickness = 225 µm
no absolute barrier at thicknesses below 225 µm

=> 99% layer thickness = 180 µm

=> 1/2 x 99% layer thickness =90 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 360 µm
above 360 µm two sides to be considered for calculation of migration if full immersion testing applied



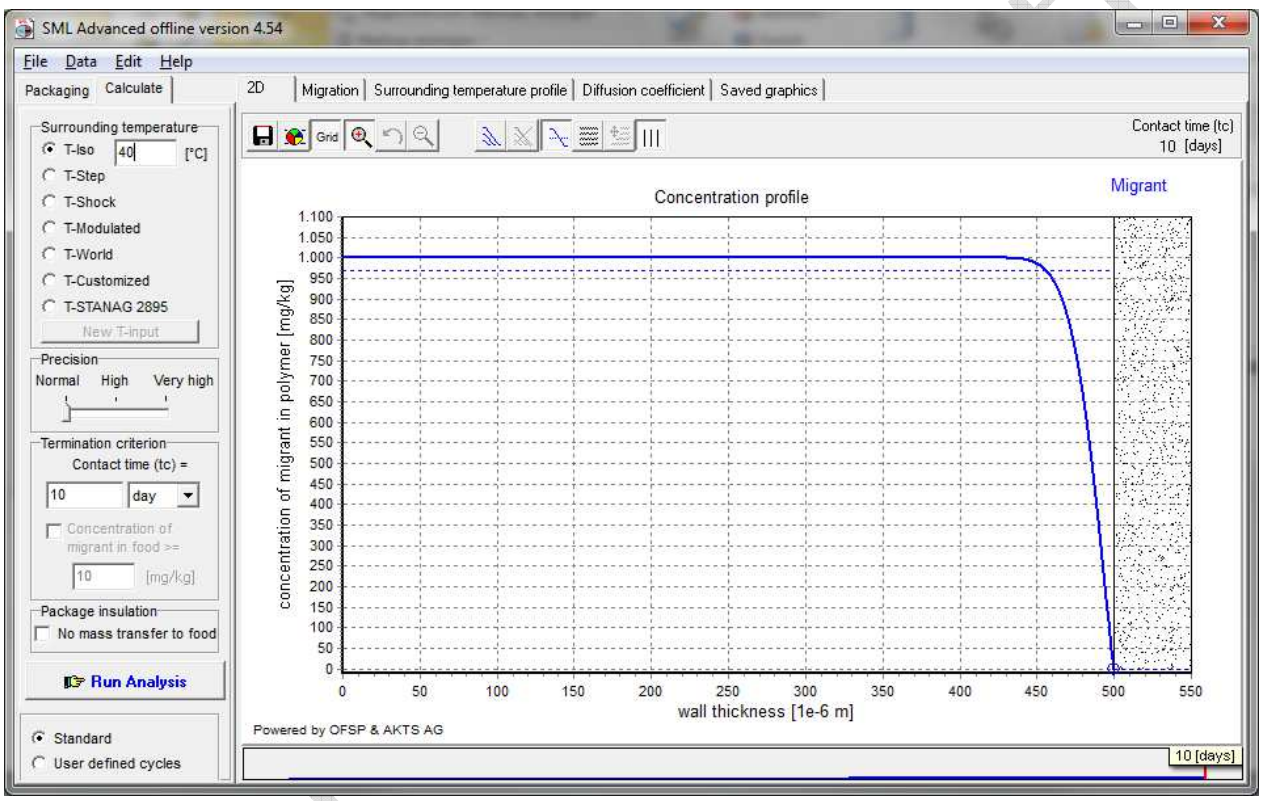
10d @ 40°C

=> 100% layer thickness = 85 µm
no absolute barrier at thicknesses below 85 µm

=> 99% layer thickness = 68 µm

=> 1/2 x 99% layer thickness = 34 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 136 µm
above 136 µm two sides to be considered for calculation of migration if full immersion testing applied



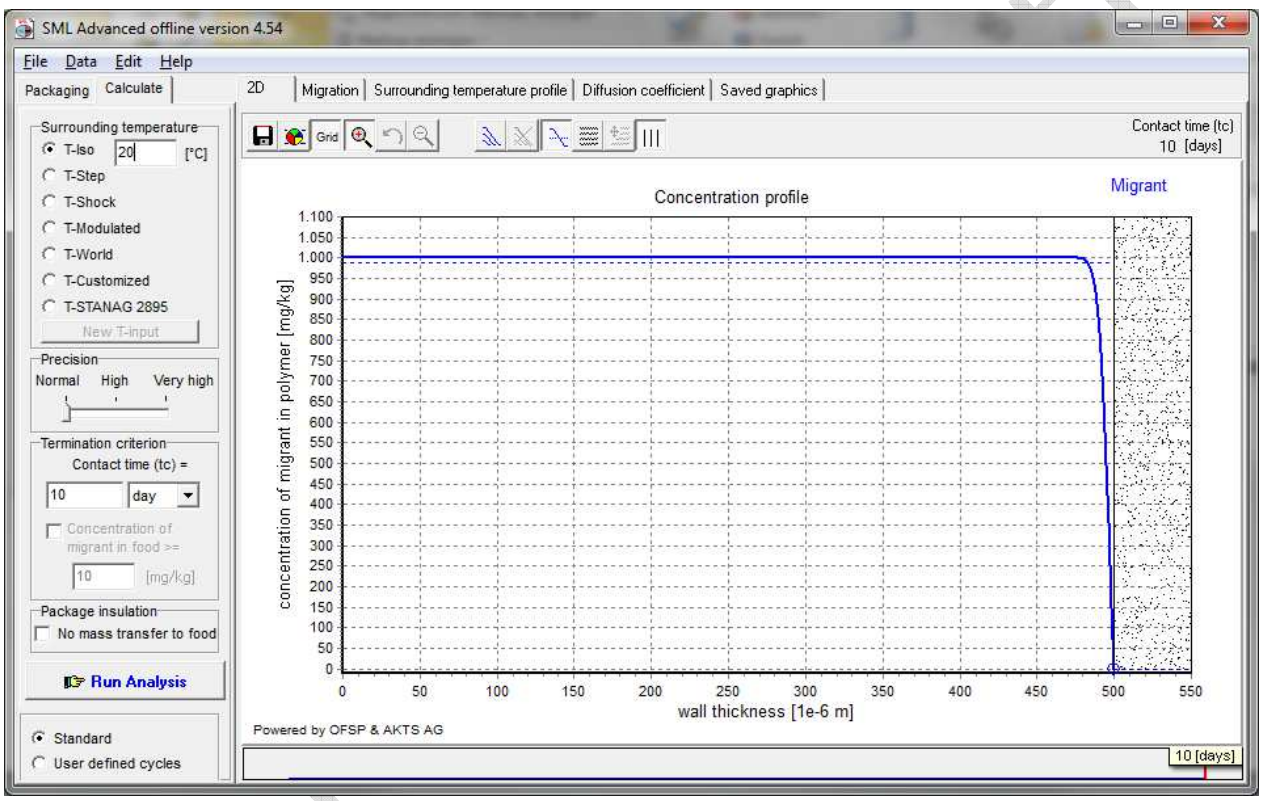
10d @ 20°C

=> 100% layer thickness = 28 µm
no absolute barrier at thicknesses below 28 µm

=> 99% layer thickness = 22 µm

=> 1/2 x 99% layer thickness = 11 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 44 µm
above 44 µm two sides to be considered for calculation of migration if full immersion testing applied



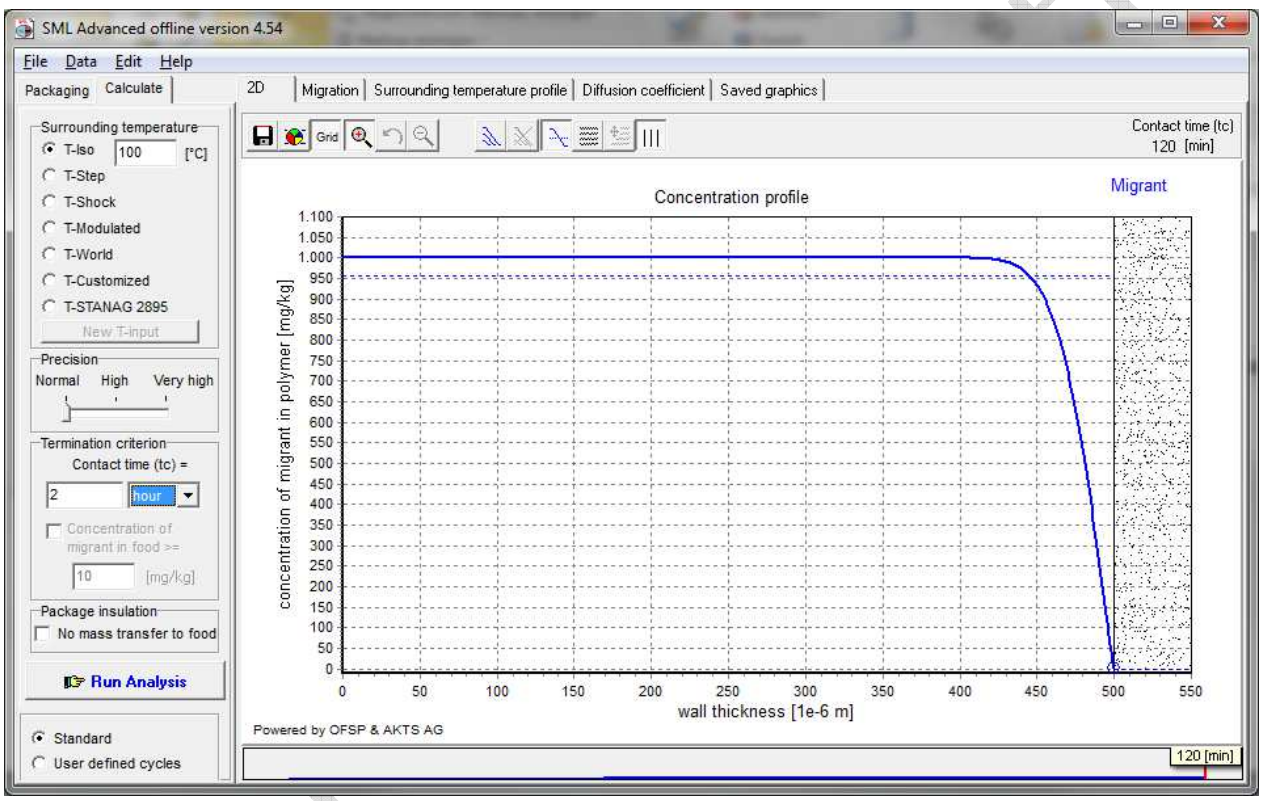
2h @ 100°C

=> 100% layer thickness = 120 µm
no absolute barrier at thicknesses below 120 µm

=> 99% layer thickness = 90 µm

=> 1/2 x 99% layer thickness =45 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 180 µm
above 180 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

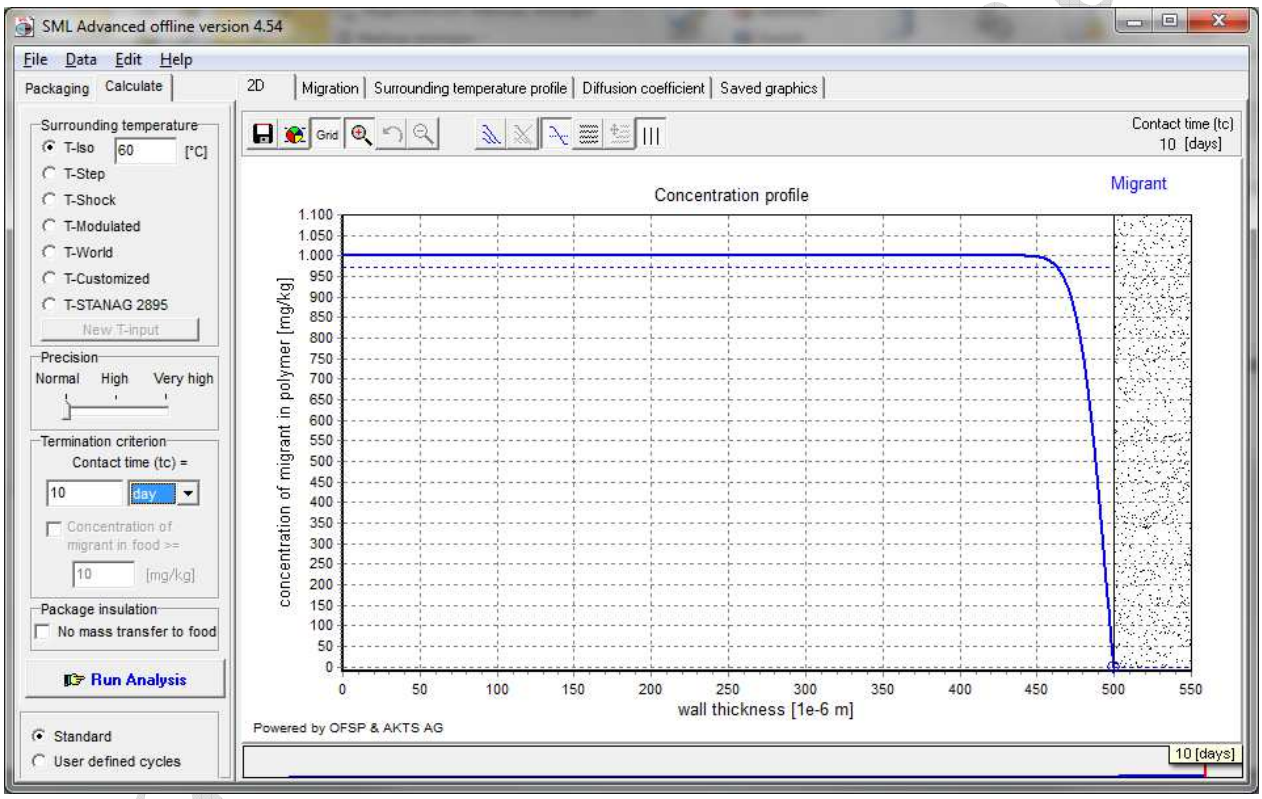
10d @ 60°C

=> 100% layer thickness = 70 µm
no absolute barrier at thicknesses below 70 µm

=> 99% layer thickness = 54 µm

=> 1/2 x 99% layer thickness = 27 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 108 µm
above 108 µm two sides to be considered for calculation of migration if full immersion testing applied



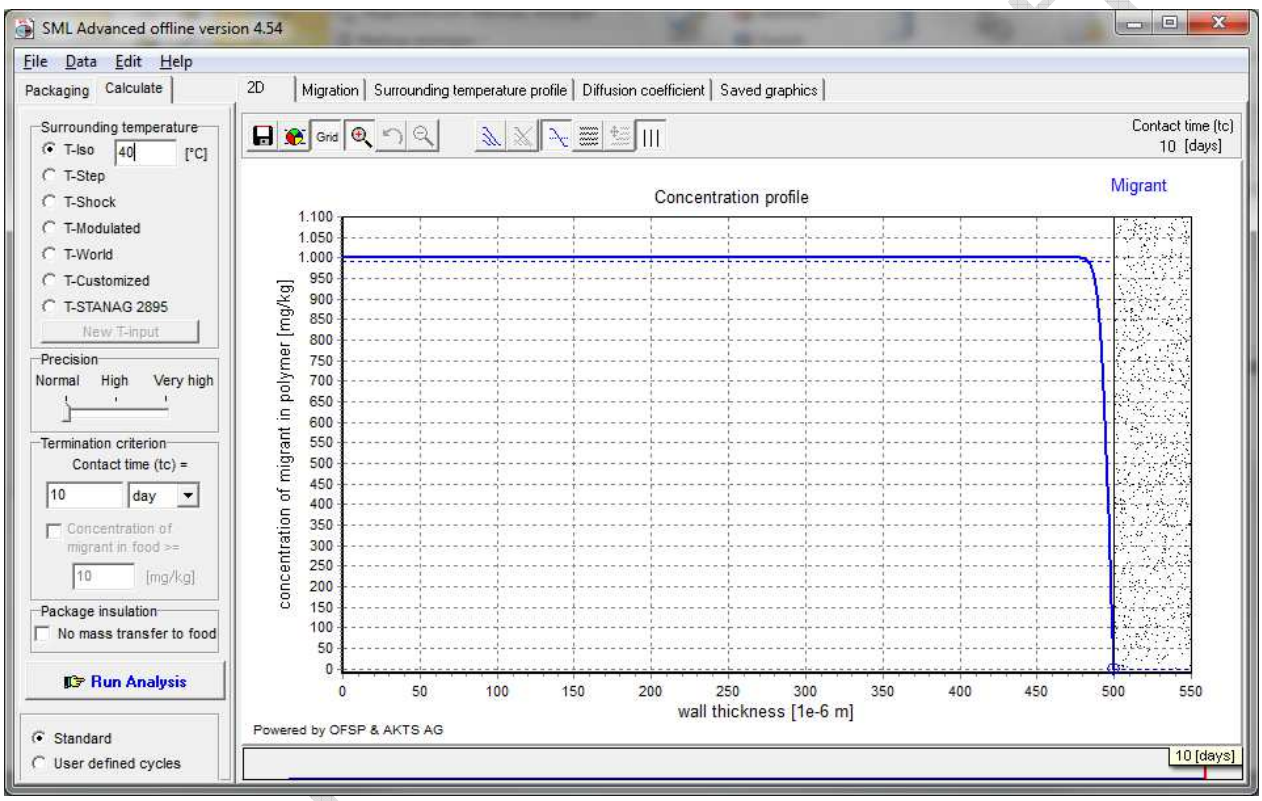
10d @ 40°C

=> 100% layer thickness = 26 µm
no absolute barrier at thicknesses below 26 µm

=> 99% layer thickness = 21 µm

=> 1/2 x 99% layer thickness = 10.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 42 µm
above 42 µm two sides to be considered for calculation of migration if full immersion testing applied



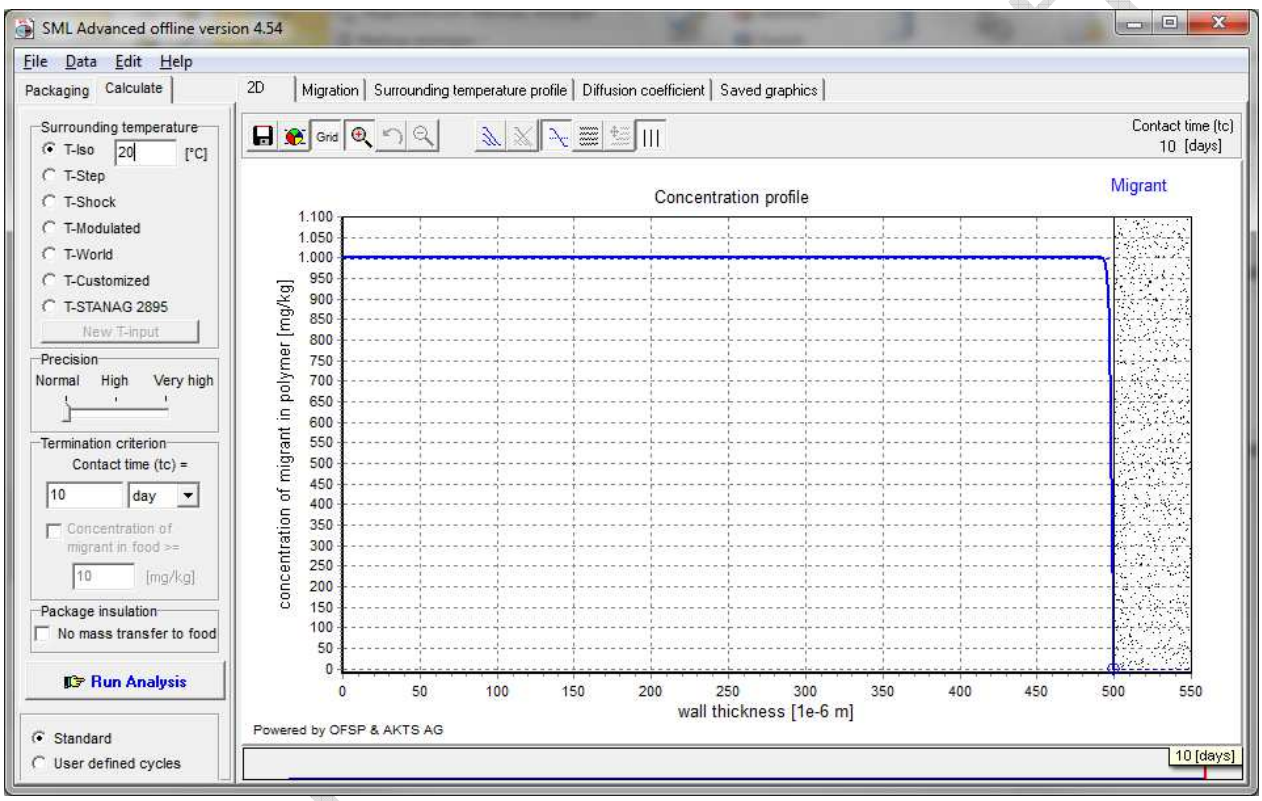
10d @ 20°C

=> 100% layer thickness = 10 µm
no absolute barrier at thicknesses below 10 µm

=> 99% layer thickness = 7.8 µm

=> 1/2 x 99% layer thickness = 3.9 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 15.6 µm
above 15.6 µm two sides to be considered for calculation of migration if full immersion testing applied



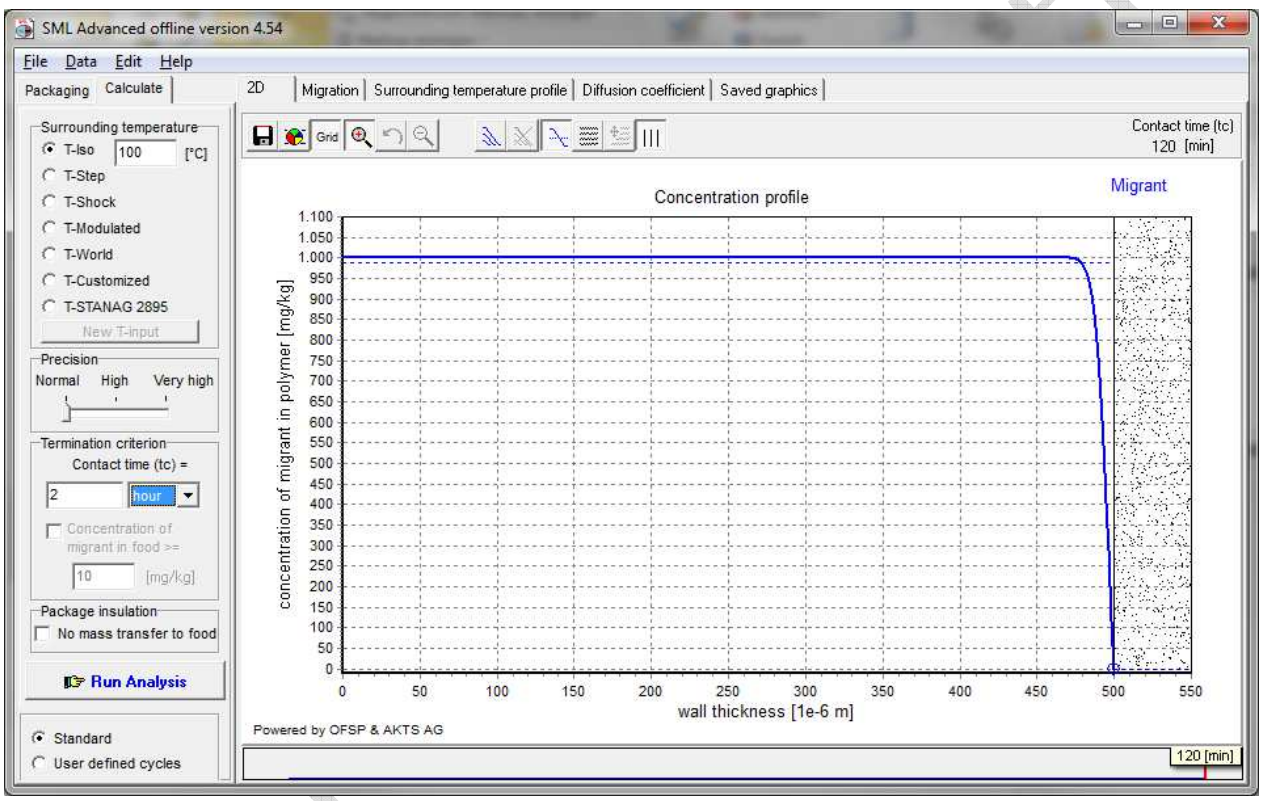
2h @ 100°C

=> 100% layer thickness = 36 µm
no absolute barrier at thicknesses below 36 µm

=> 99% layer thickness = 28 µm

=> 1/2 x 99% layer thickness = 14 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 56 µm
above 56 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

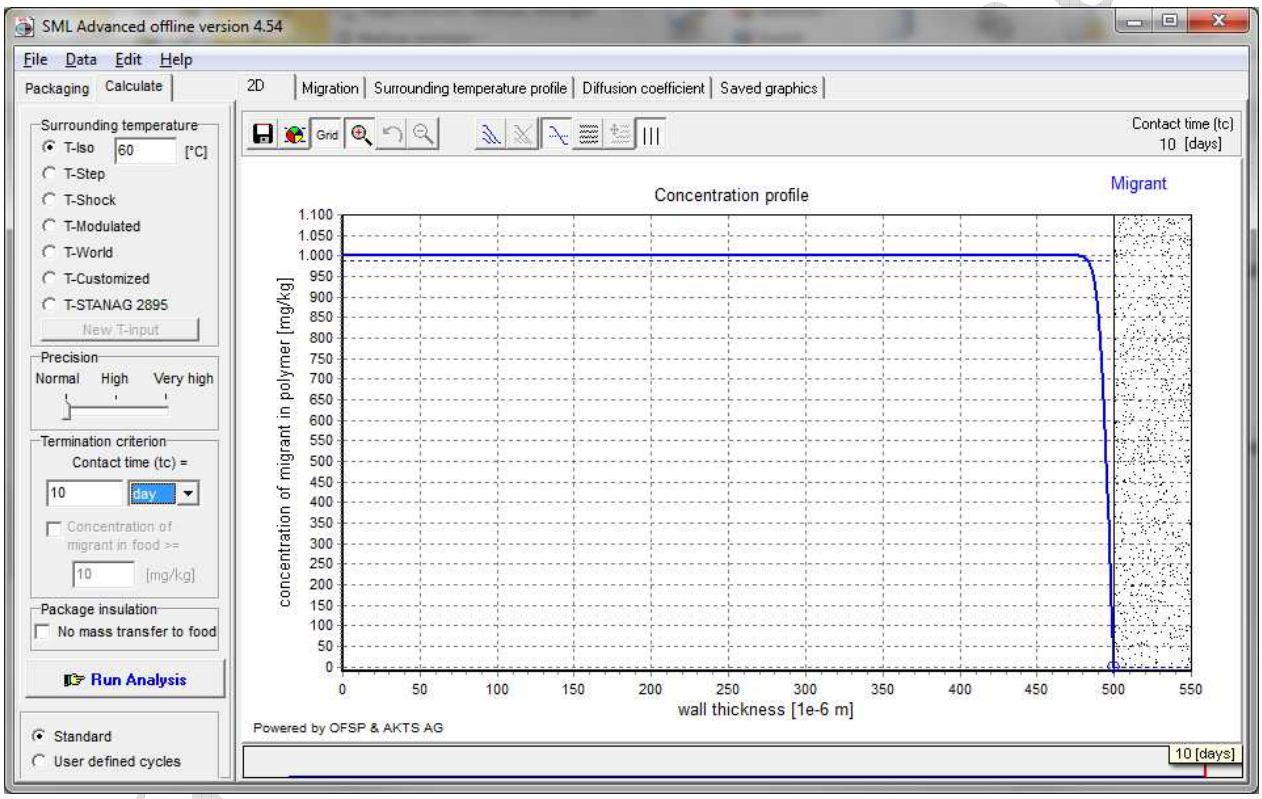
10d @ 60°C

=> 100% layer thickness = 28 µm
no absolute barrier at thicknesses below 28 µm

=> 99% layer thickness = 22 µm

=> 1/2 x 99% layer thickness = 11 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 44 µm
above 44 µm two sides to be considered for calculation of migration if full immersion testing applied



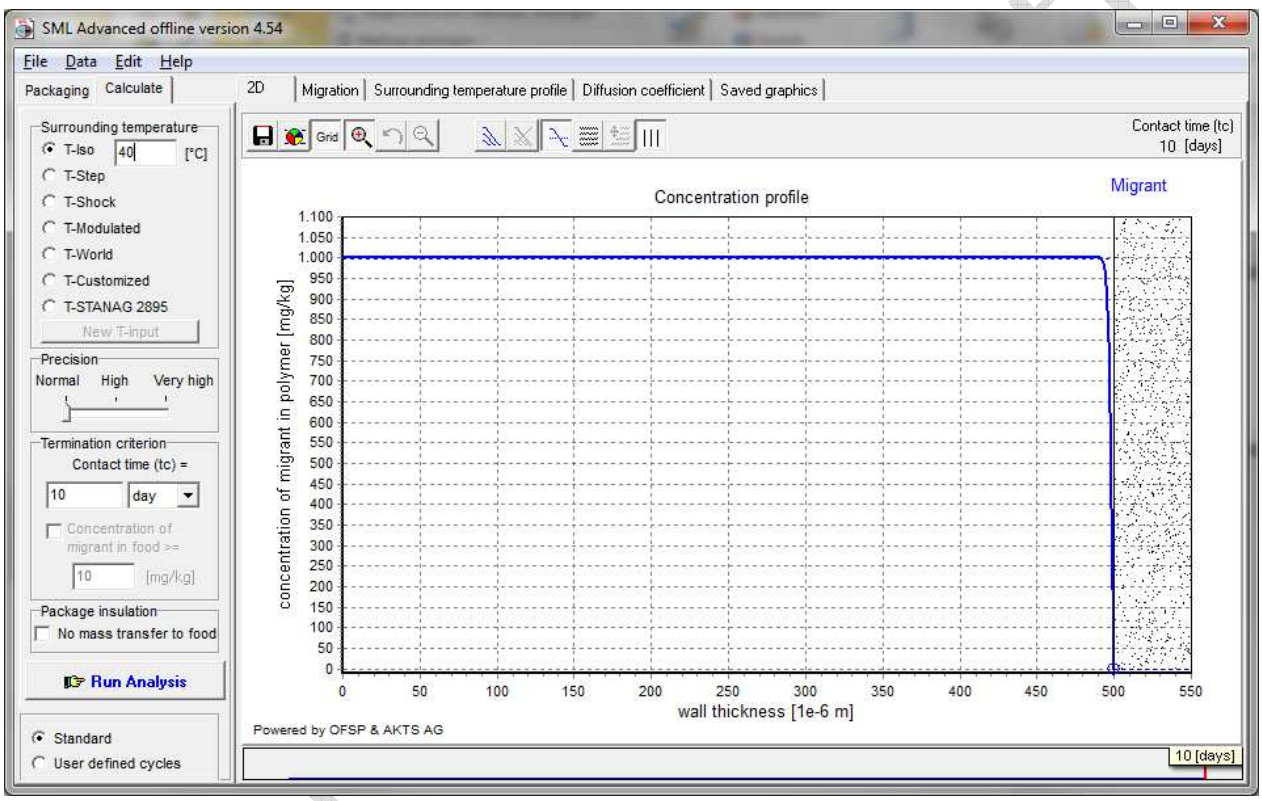
10d @ 40°C

=> 100% layer thickness = 13 µm
no absolute barrier at thicknesses below 13 µm

=> 99% layer thickness = 9.2 µm

=> 1/2 x 99% layer thickness = 4.6 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 18.4 µm
above 18.4 µm two sides to be considered for calculation of migration if full immersion testing applied



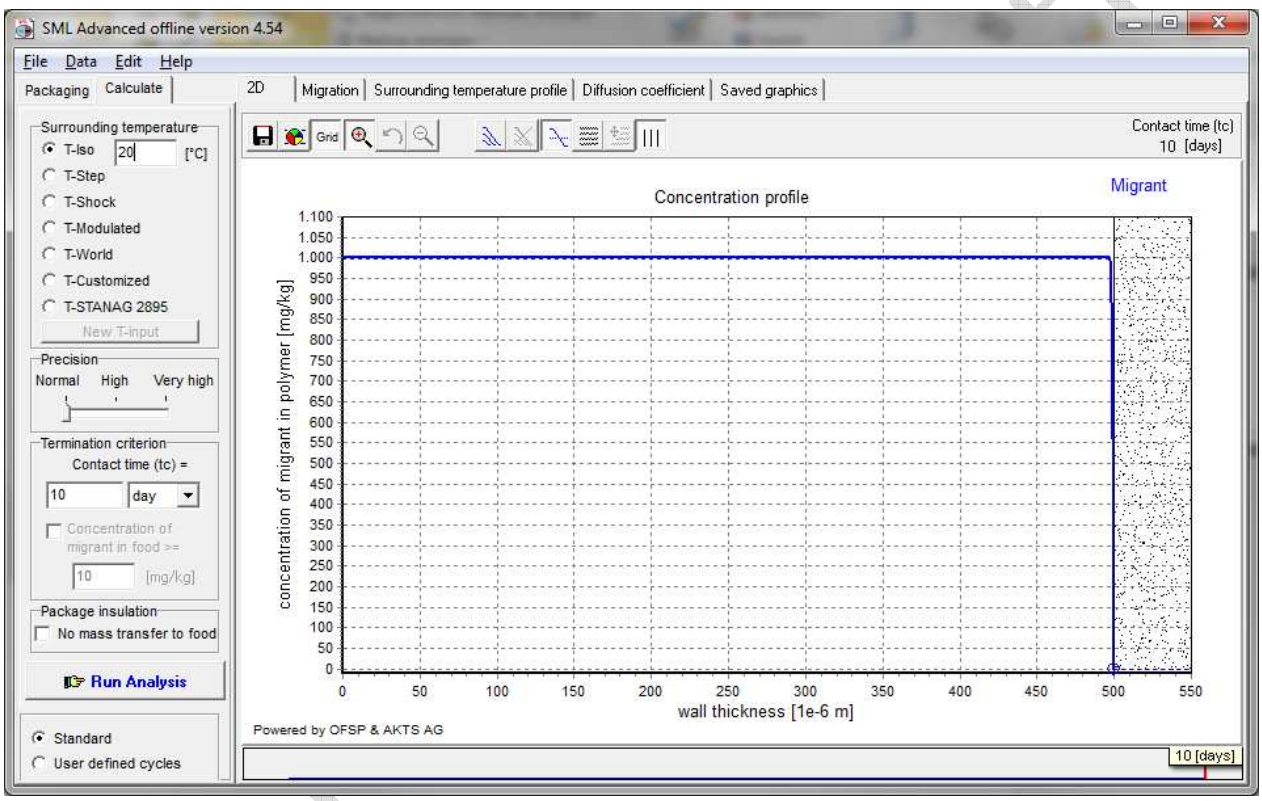
10d @ 20°C

=> 100% layer thickness = 4.4 µm
no absolute barrier at thicknesses below 4.4 µm

=> 99% layer thickness = 3.2 µm

=> 1/2 x 99% layer thickness = 1.6 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 6.4 µm
above 6.4 µm two sides to be considered for calculation of migration if full immersion testing applied



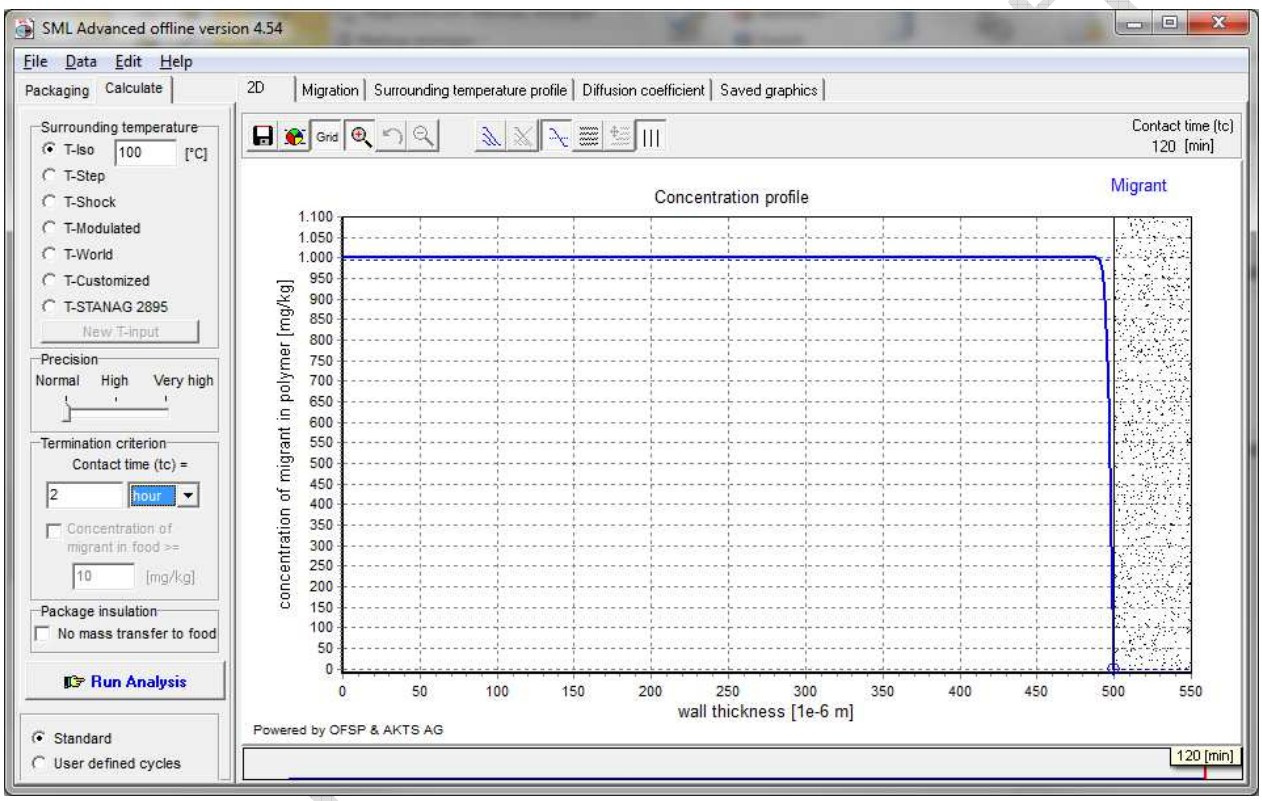
2h @ 100°C

=> 100% layer thickness = 15.6 µm
no absolute barrier at thicknesses below 15.6 µm

=> 99% layer thickness = 11.6 µm

=> 1/2 x 99% layer thickness = 5.8 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 23.2 µm
above 23.2 µm two sides to be considered for calculation of migration if full immersion testing applied



PA12
(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct contact, e.g. plastic multilayer)

► molecular mass 100 - 250 g/mol

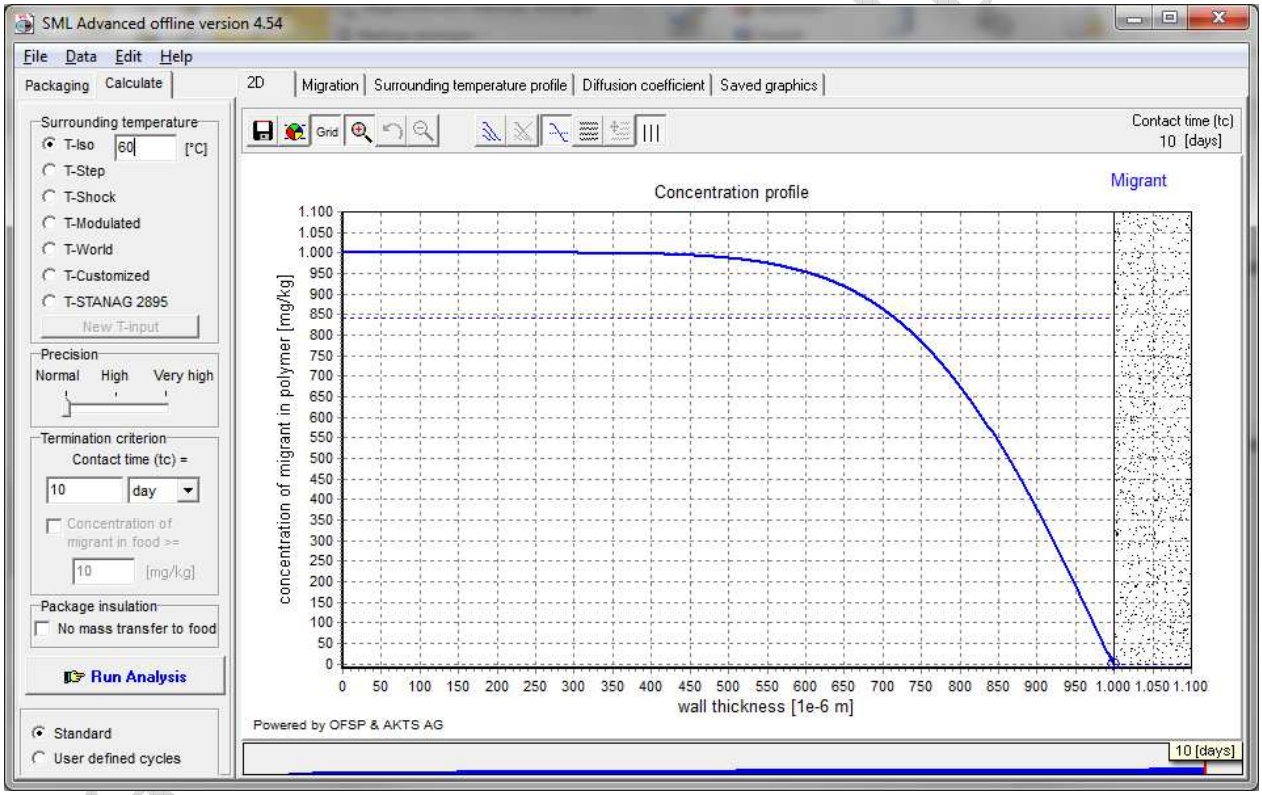
10d @ 60°C

=> 100% layer thickness = 810 µm
no absolute barrier at thicknesses below 810 µm

=> 99% layer thickness = 660 µm

=> 1/2 x 99% layer thickness = 330 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1320 µm
above 1320 µm two sides to be considered for calculation of migration if full immersion testing applied



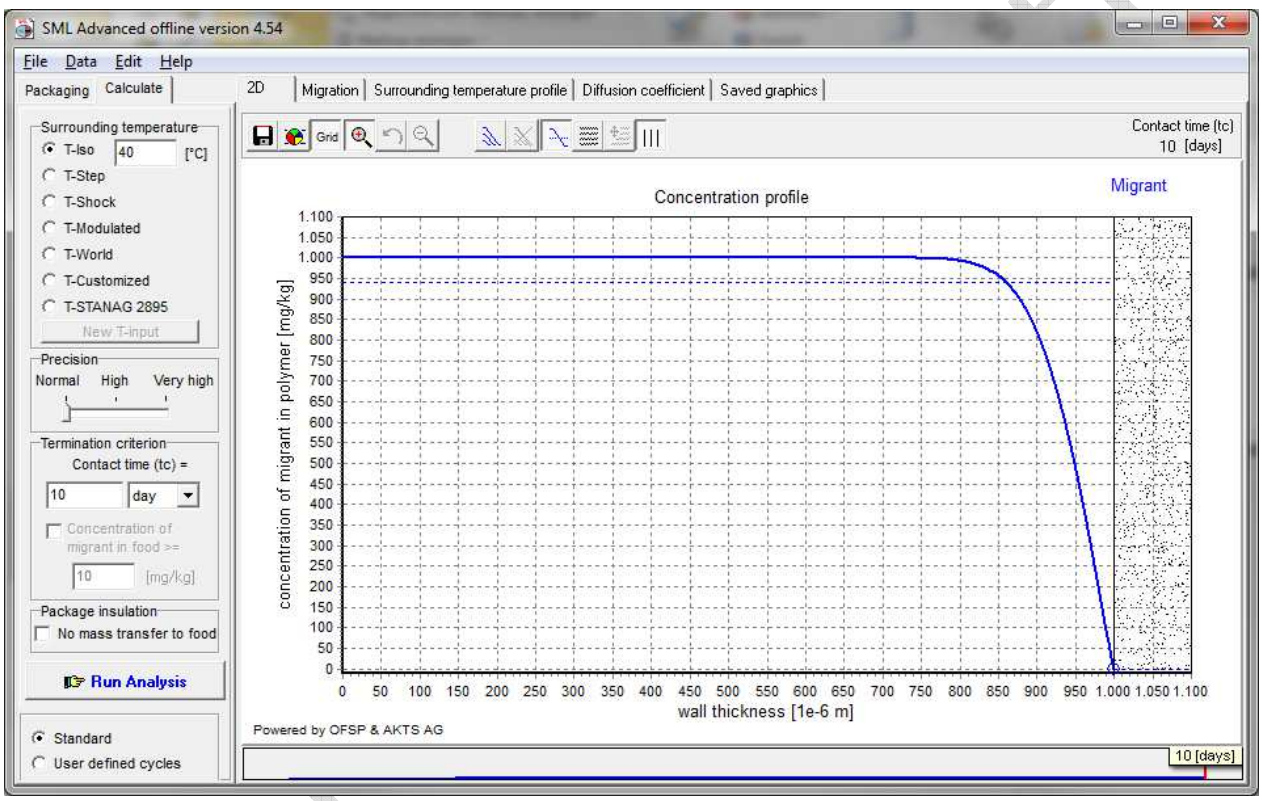
10d @ 40°C

=> 100% layer thickness = 420 µm
no absolute barrier at thicknesses below 420 µm

=> 99% layer thickness = 250 µm

=> 1/2 x 99% layer thickness =125 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 500 µm
above 500 µm two sides to be considered for calculation of migration if full immersion testing applied



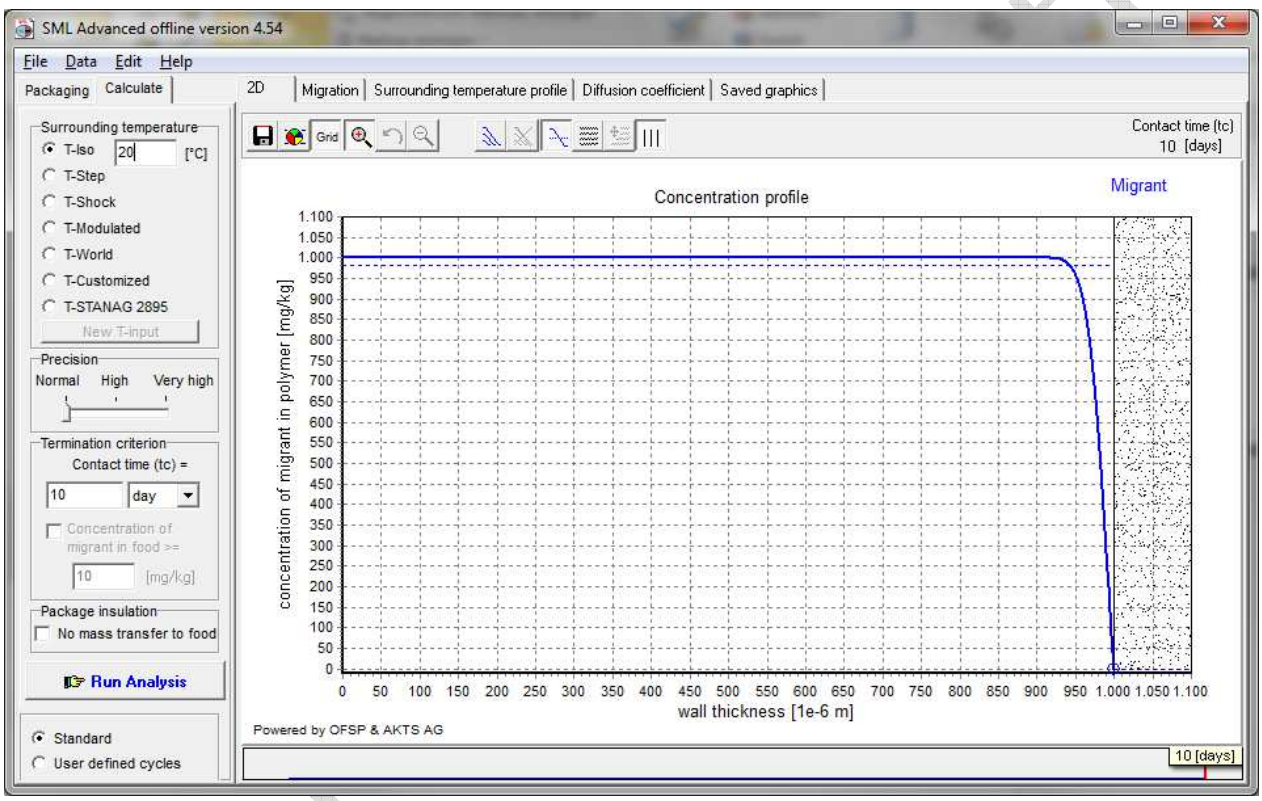
10d @ 20°C

=> 100% layer thickness = 100 µm
no absolute barrier at thicknesses below 100 µm

=> 99% layer thickness = 80 µm

=> 1/2 x 99% layer thickness = 40 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 160 µm
above 160 µm two sides to be considered for calculation of migration if full immersion testing applied



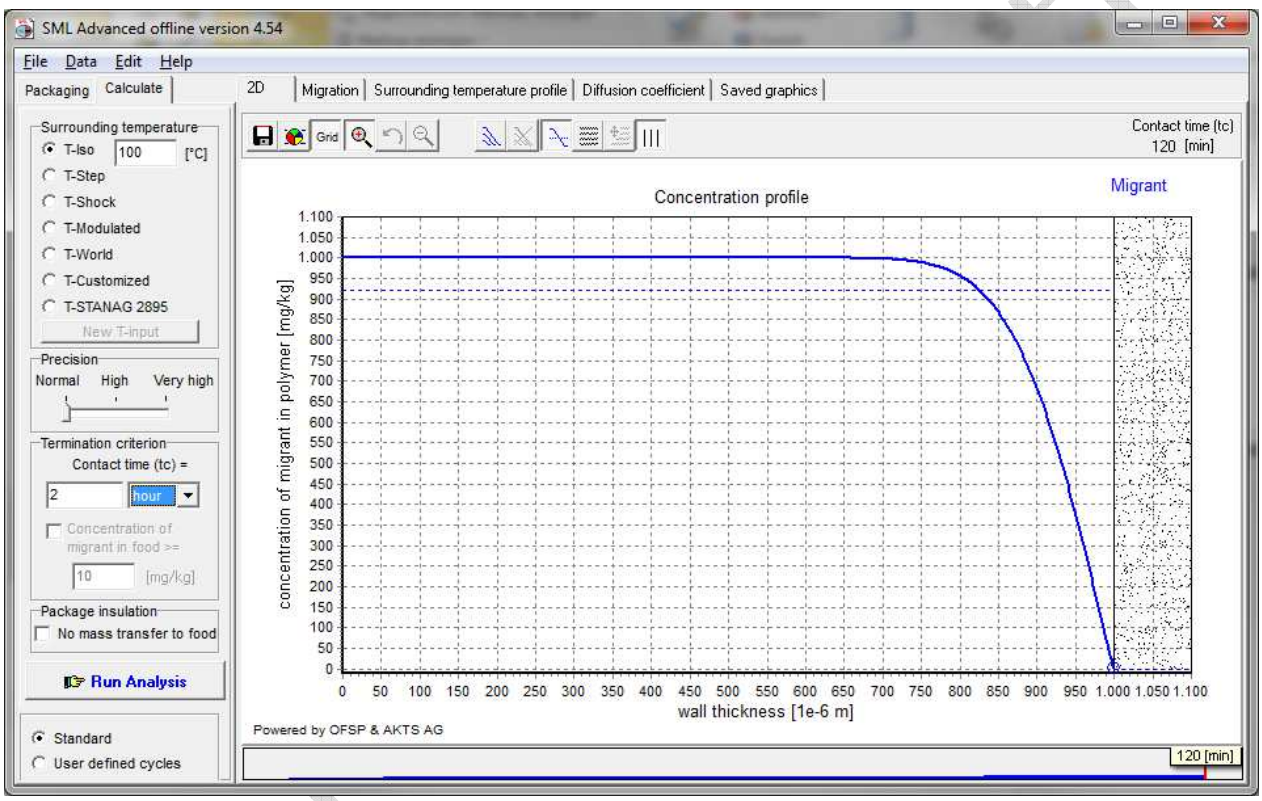
2h @ 100°C

=> 100% layer thickness = 400 µm
no absolute barrier at thicknesses below 400 µm

=> 99% layer thickness = 330 µm

=> 1/2 x 99% layer thickness = 165 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 660 µm
above 660 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

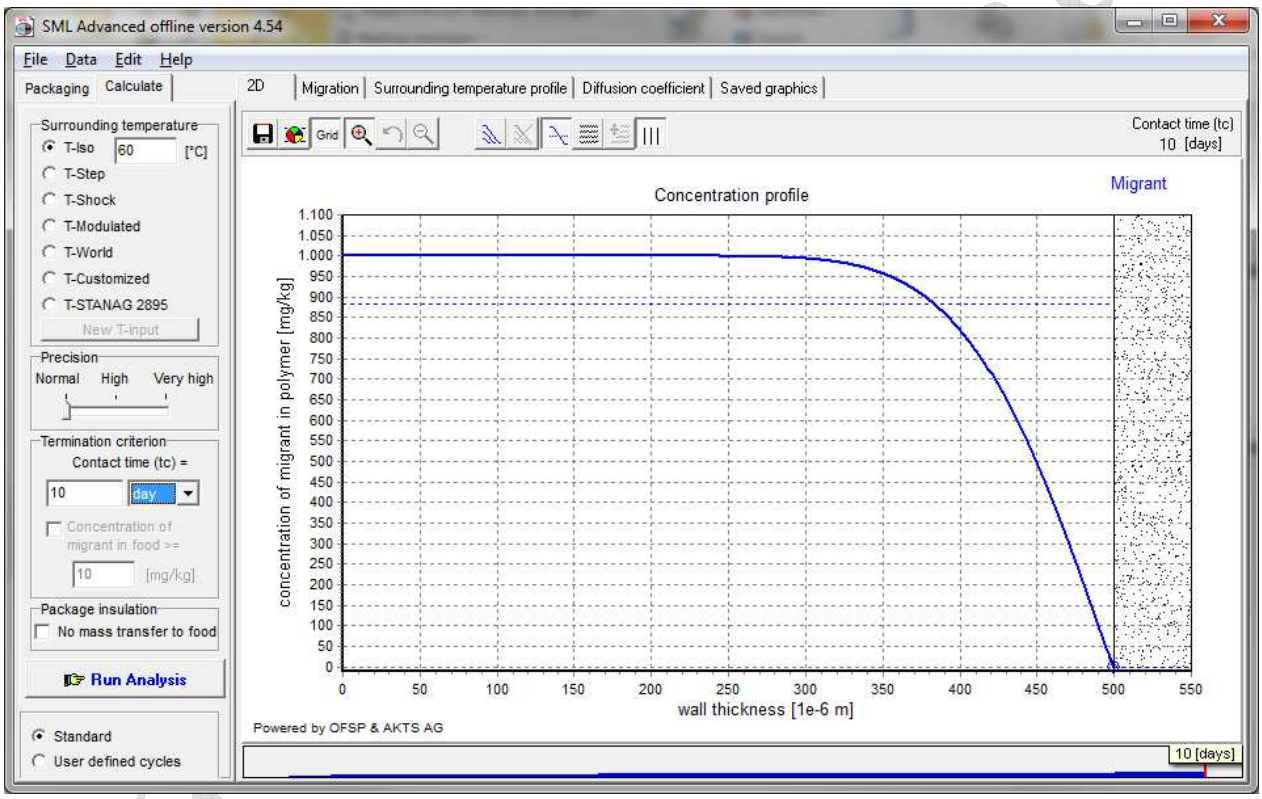
10d @ 60°C

=> 100% layer thickness = 300 µm
no absolute barrier at thicknesses below 300 µm

=> 99% layer thickness = 250 µm

=> 1/2 x 99% layer thickness = 125 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 500 µm
above 500 µm two sides to be considered for calculation of migration if full immersion testing applied



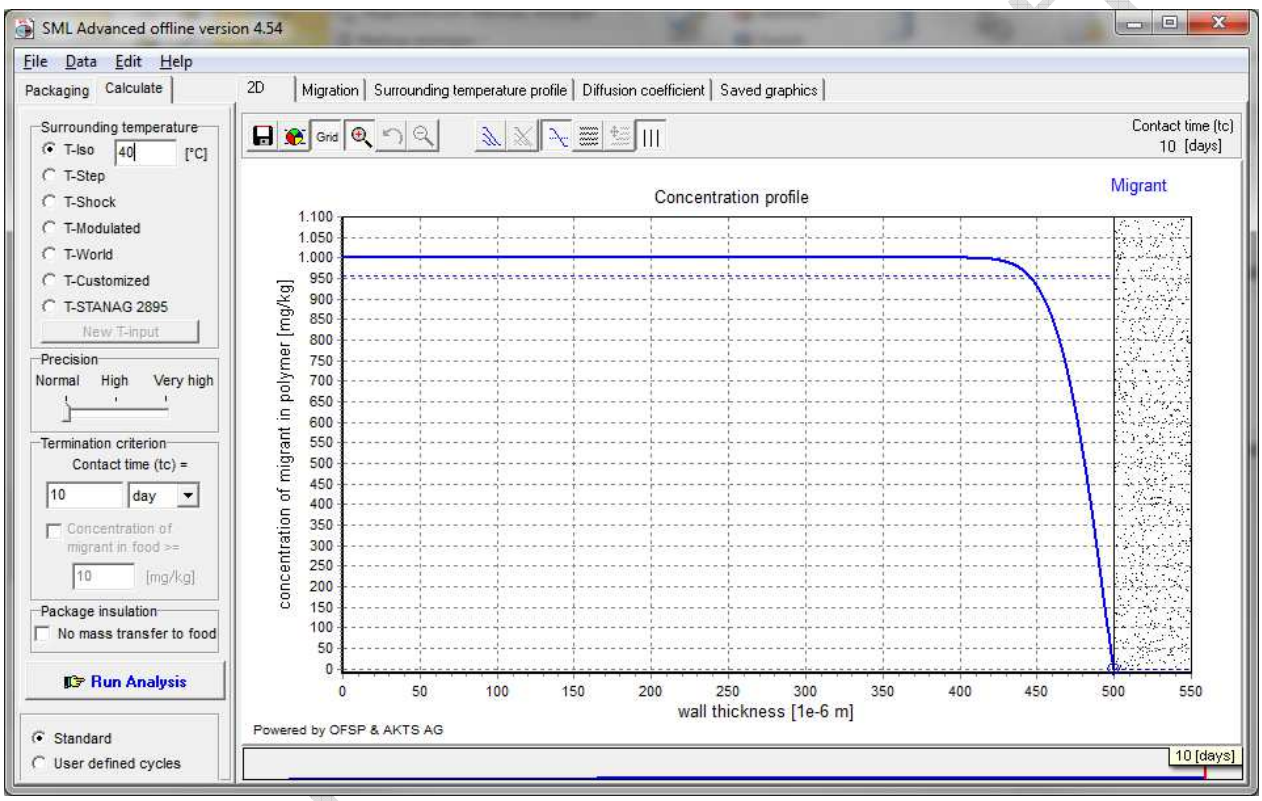
10d @ 40°C

=> 100% layer thickness = 114 µm
no absolute barrier at thicknesses below 114 µm

=> 99% layer thickness = 90 µm

=> 1/2 x 99% layer thickness = 45 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 180 µm
above 180 µm two sides to be considered for calculation of migration if full immersion testing applied



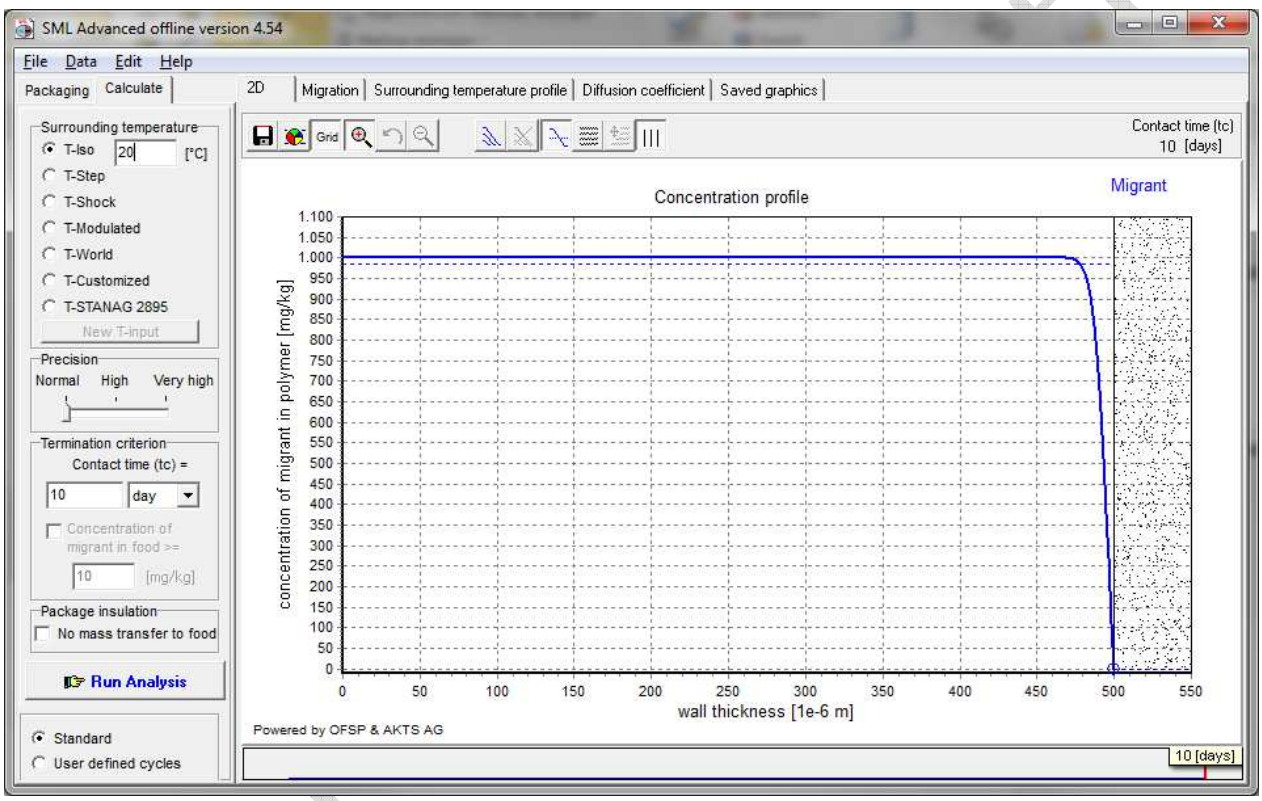
10d @ 20°C

=> 100% layer thickness = 44 µm
no absolute barrier at thicknesses below 44 µm

=> 99% layer thickness = 30 µm

=> 1/2 x 99% layer thickness = 15 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 60 µm
above 60 µm two sides to be considered for calculation of migration if full immersion testing applied



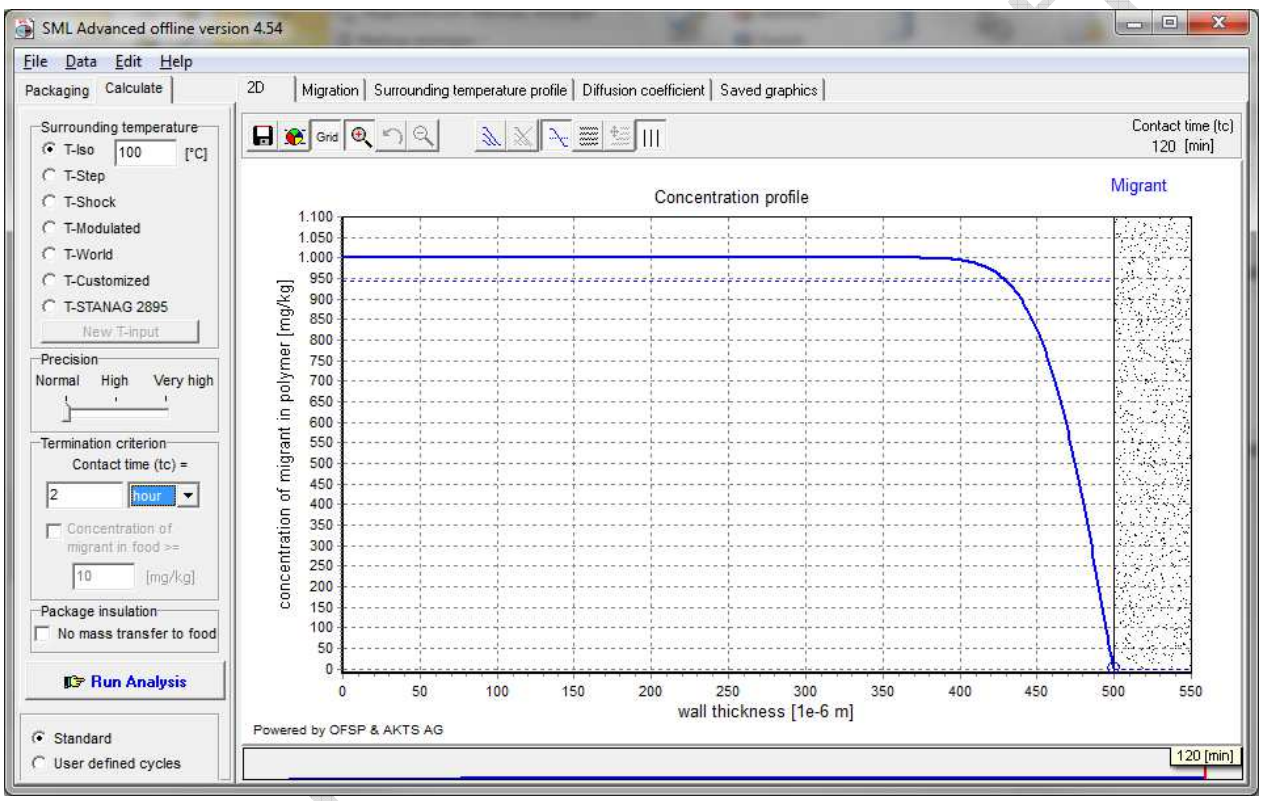
2h @ 100°C

=> 100% layer thickness = 147 µm
no absolute barrier at thicknesses below 147 µm

=> 99% layer thickness = 120 µm

=> 1/2 x 99% layer thickness =60 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 240 µm
above 240 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

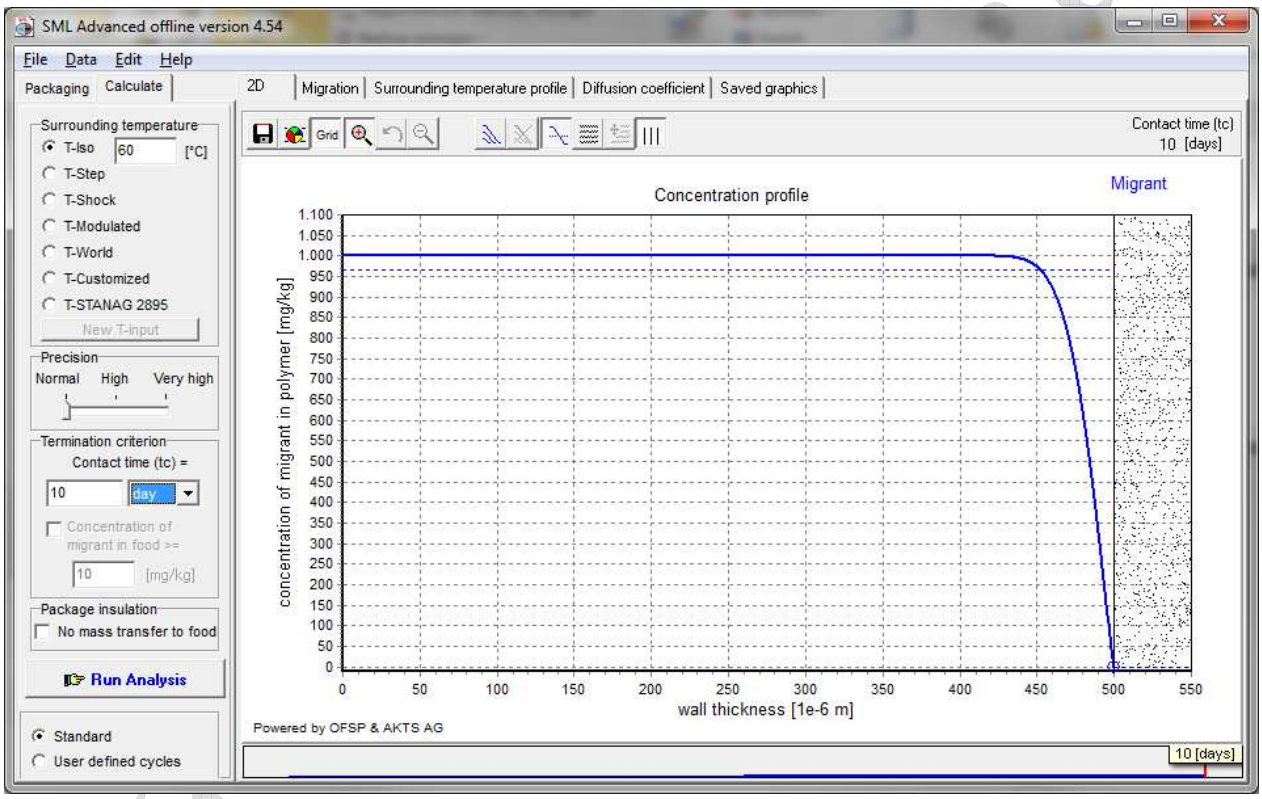
10d @ 60°C

=> 100% layer thickness = 91 µm
no absolute barrier at thicknesses below 91 µm

=> 99% layer thickness = 74 µm

=> 1/2 x 99% layer thickness = 37 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 148 µm
above 148 µm two sides to be considered for calculation of migration if full immersion testing applied



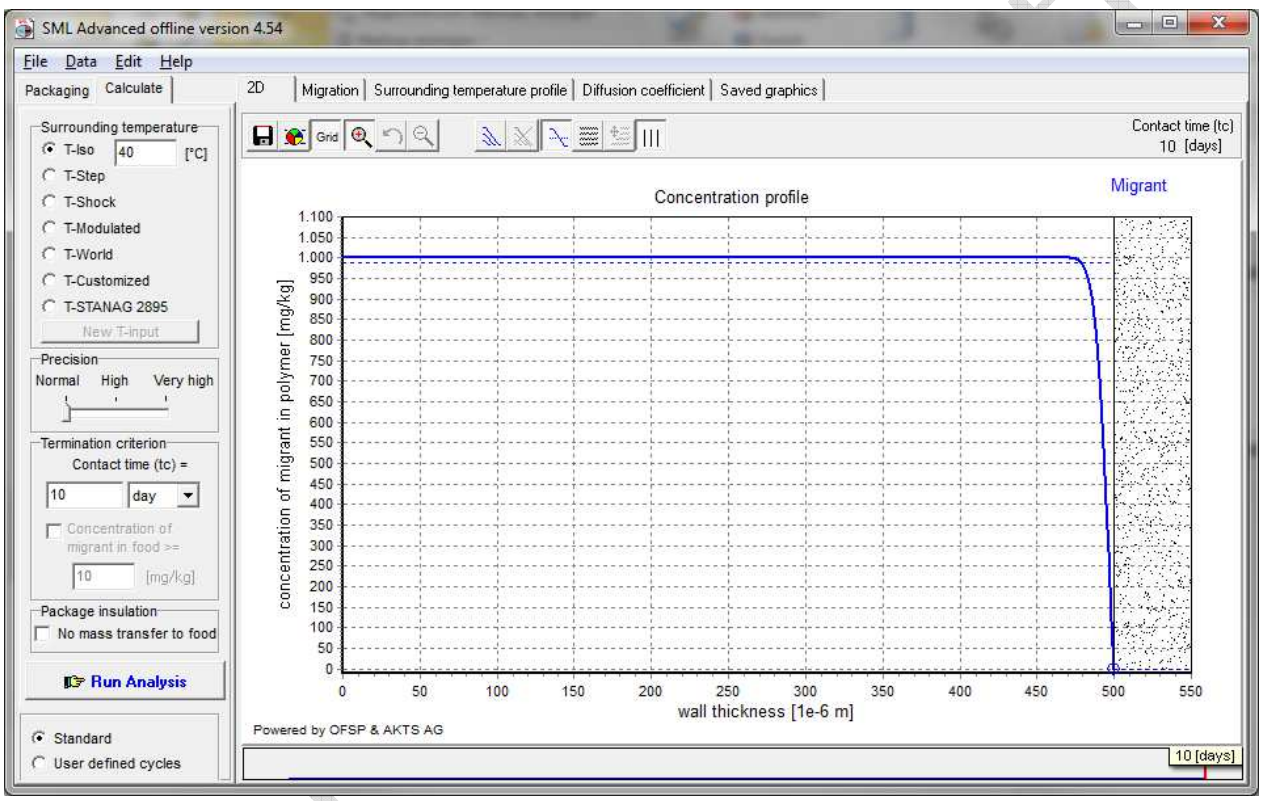
10d @ 40°C

=> 100% layer thickness = 34 µm
no absolute barrier at thicknesses below 34 µm

=> 99% layer thickness = 28 µm

=> 1/2 x 99% layer thickness = 14 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 56 µm
above 56 µm two sides to be considered for calculation of migration if full immersion testing applied



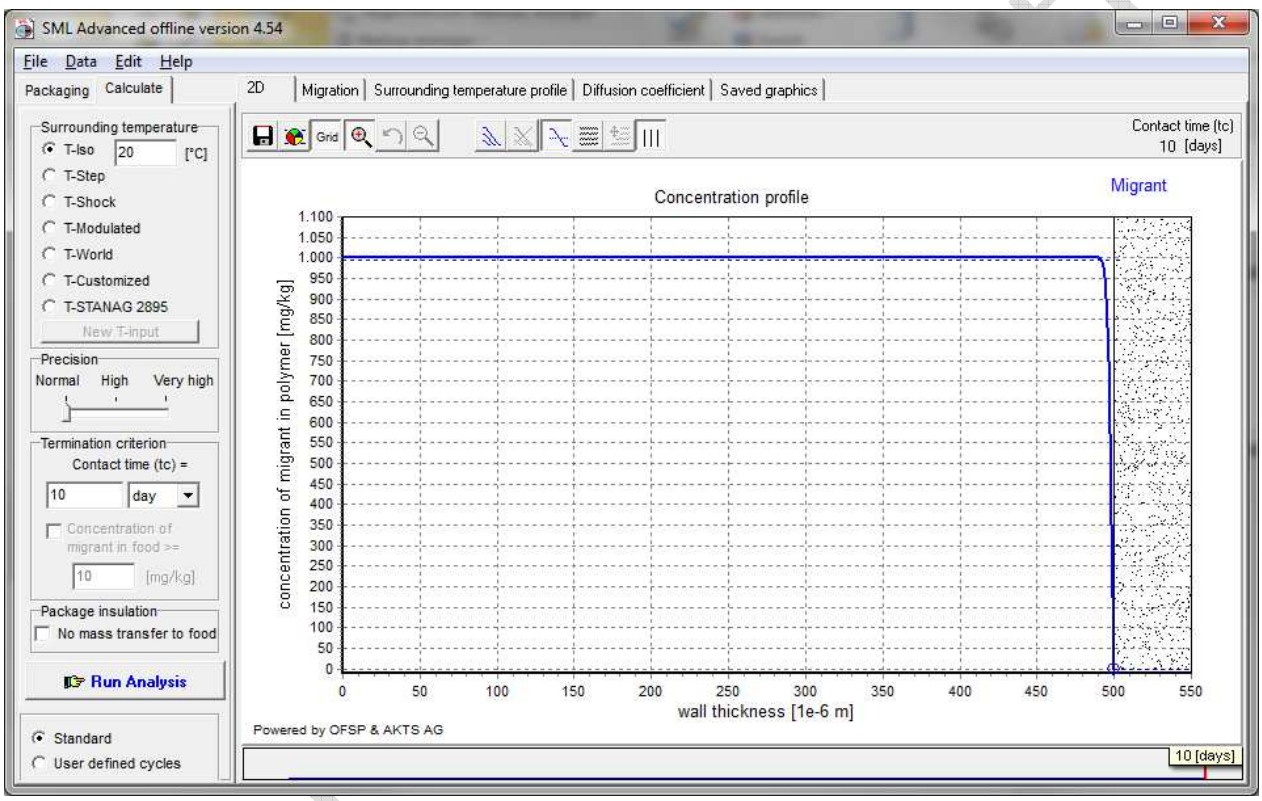
10d @ 20°C

=> 100% layer thickness = 13 µm
no absolute barrier at thicknesses below 13 µm

=> 99% layer thickness = 10 µm

=> 1/2 x 99% layer thickness = 5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 20 µm
above 20 µm two sides to be considered for calculation of migration if full immersion testing applied



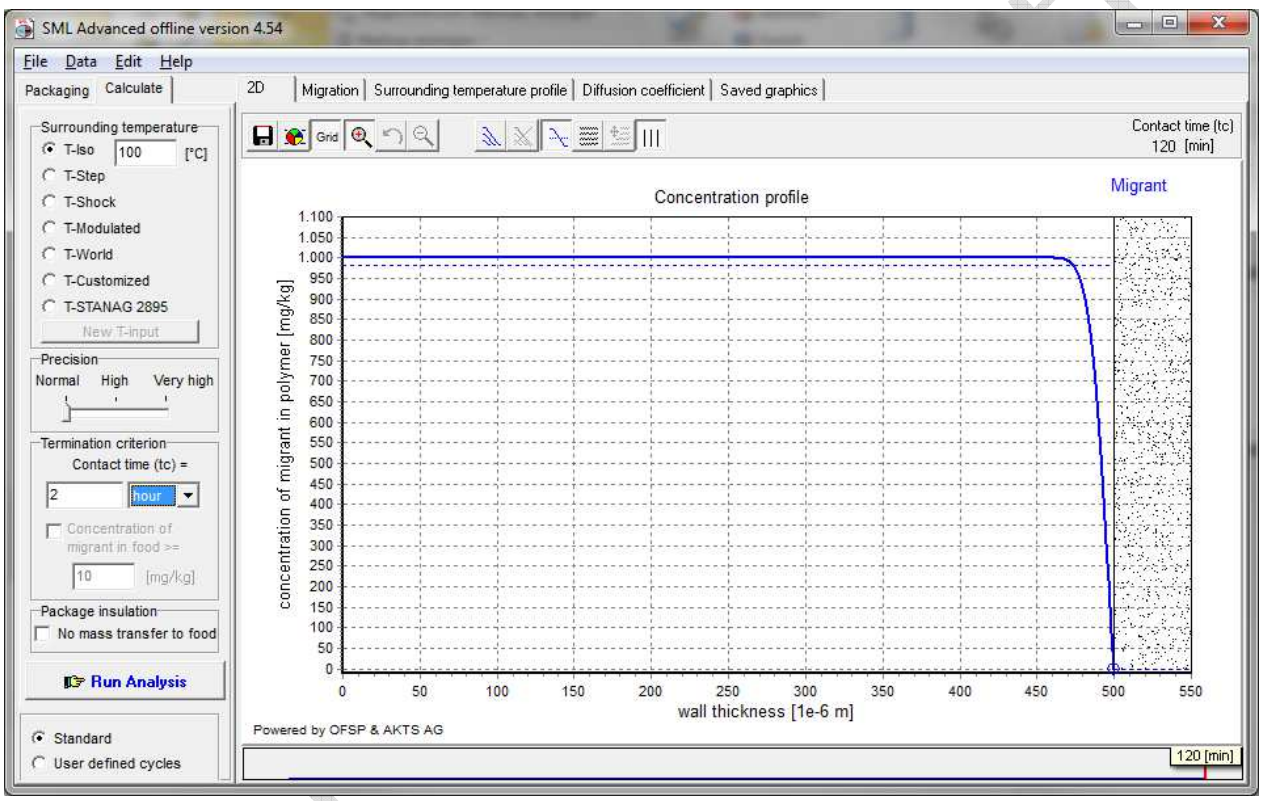
2h @ 100°C

=> 100% layer thickness = 46 µm
no absolute barrier at thicknesses below 46 µm

=> 99% layer thickness = 37 µm

=> 1/2 x 99% layer thickness = 18.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 74 µm
above 74 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

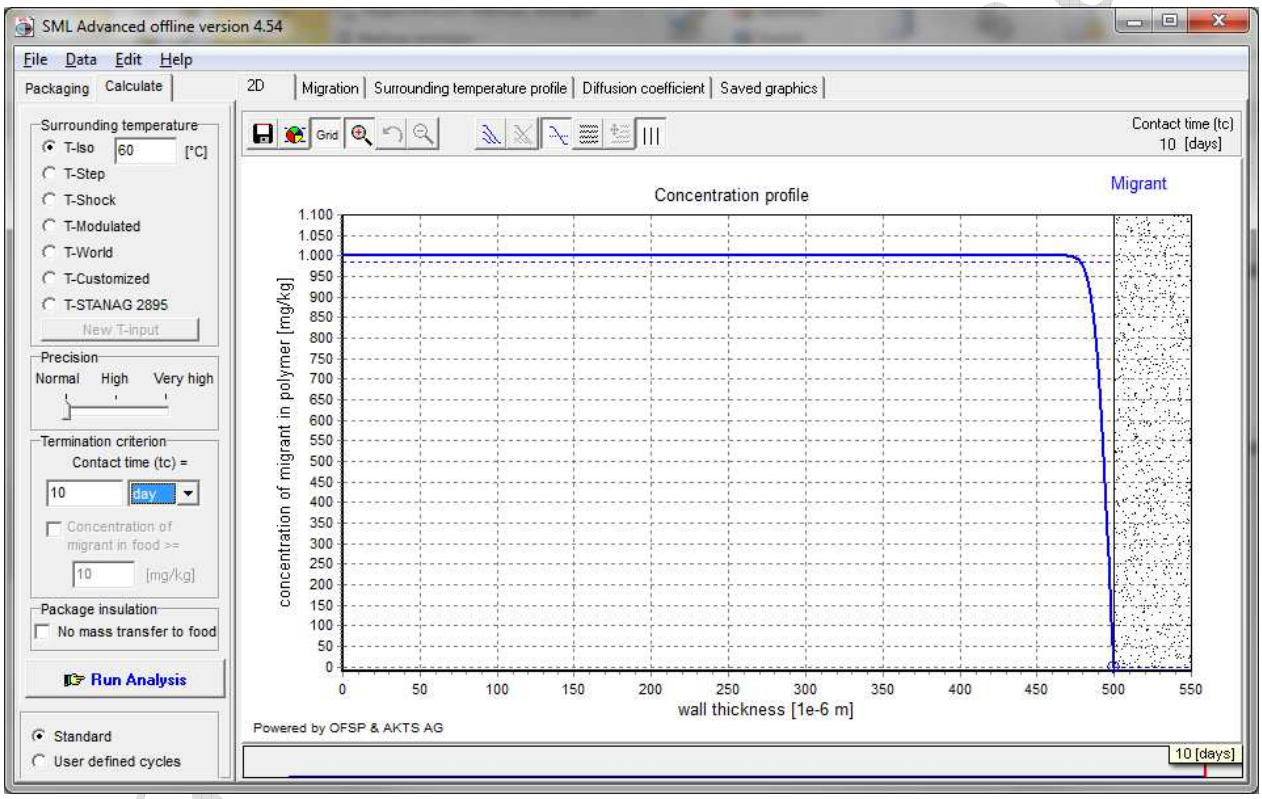
10d @ 60°C

=> 100% layer thickness = 37 µm
no absolute barrier at thicknesses below 28 µm

=> 99% layer thickness = 30 µm

=> 1/2 x 99% layer thickness = 15 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 60 µm
above 60 µm two sides to be considered for calculation of migration if full immersion testing applied



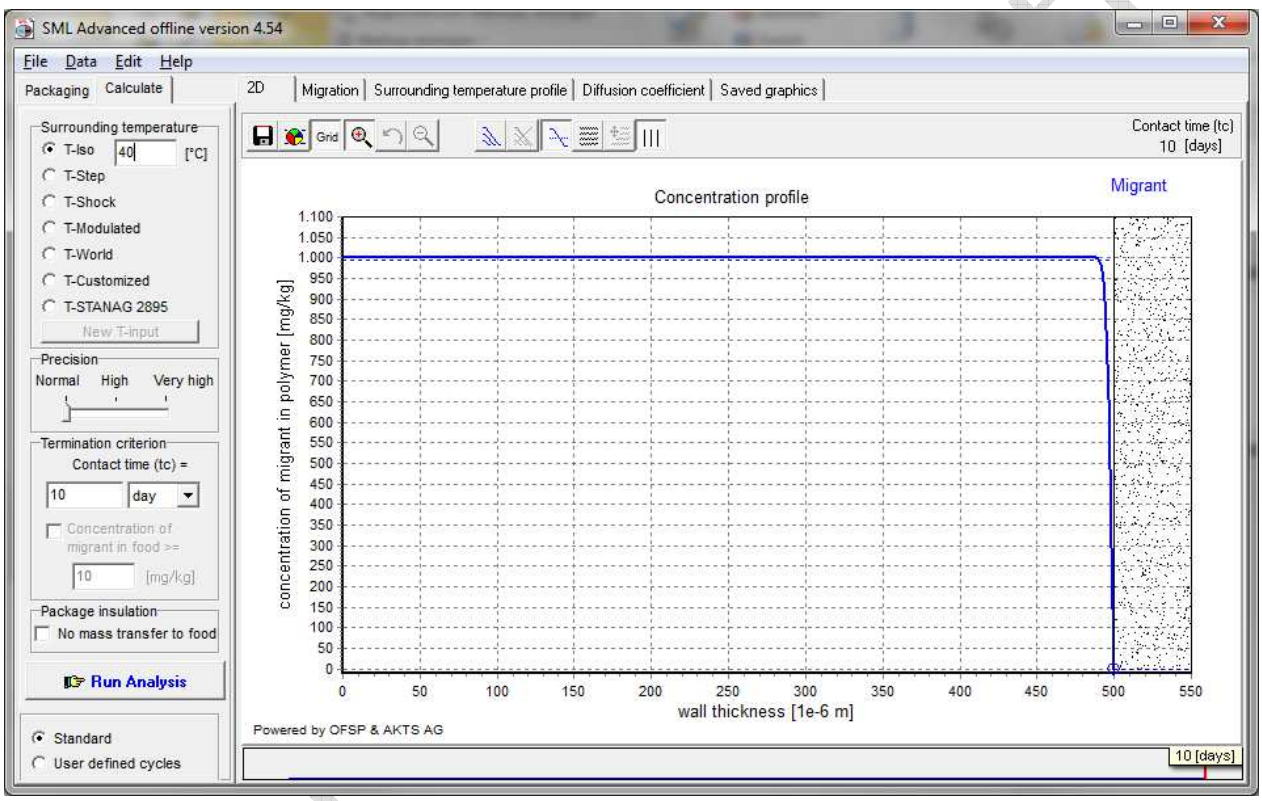
10d @ 40°C

=> 100% layer thickness = 15 µm
no absolute barrier at thicknesses below 15 µm

=> 99% layer thickness = 11.6 µm

=> 1/2 x 99% layer thickness = 4.6 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 18.4 µm
above 18.4 µm two sides to be considered for calculation of migration if full immersion testing applied



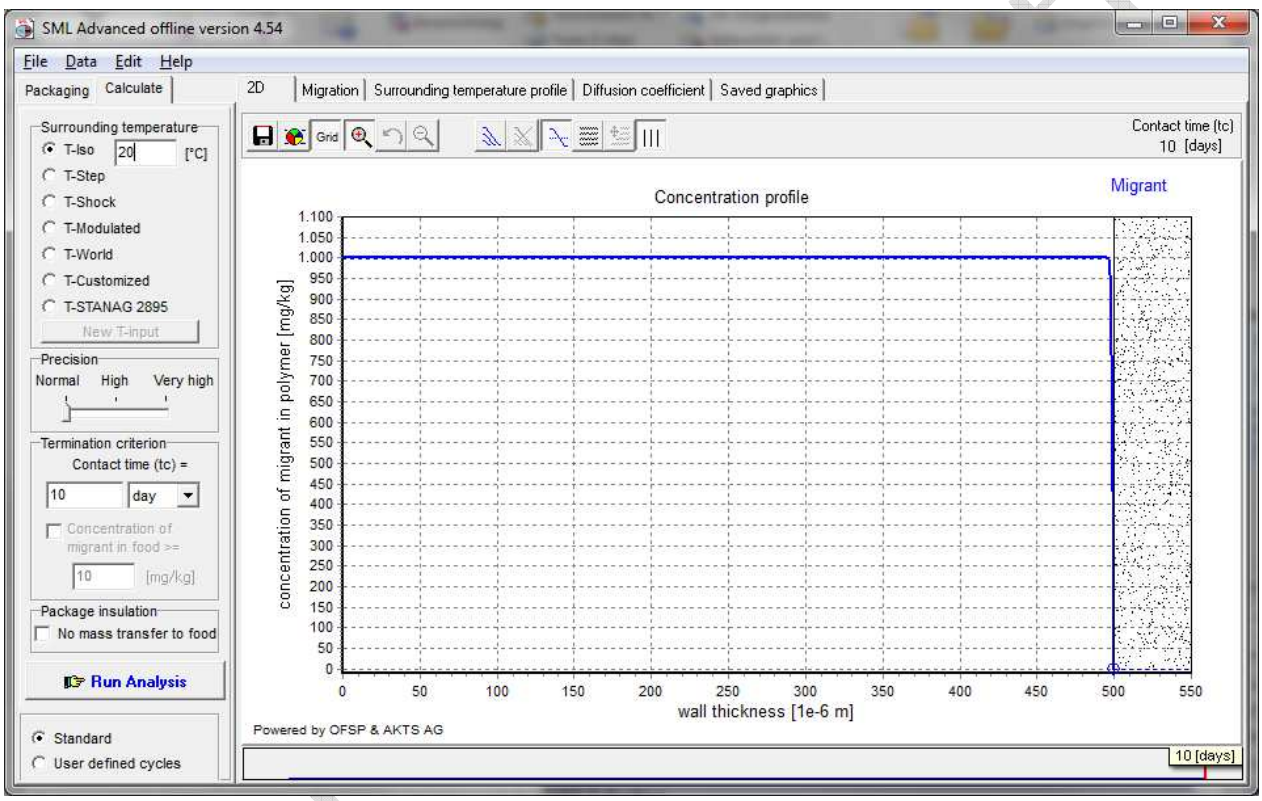
10d @ 20°C

=> 100% layer thickness = 6 µm
no absolute barrier at thicknesses below 6 µm

=> 99% layer thickness = 4.4 µm

=> 1/2 x 99% layer thickness = 2.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 8.8 µm
above 8.8 µm two sides to be considered for calculation of migration if full immersion testing applied



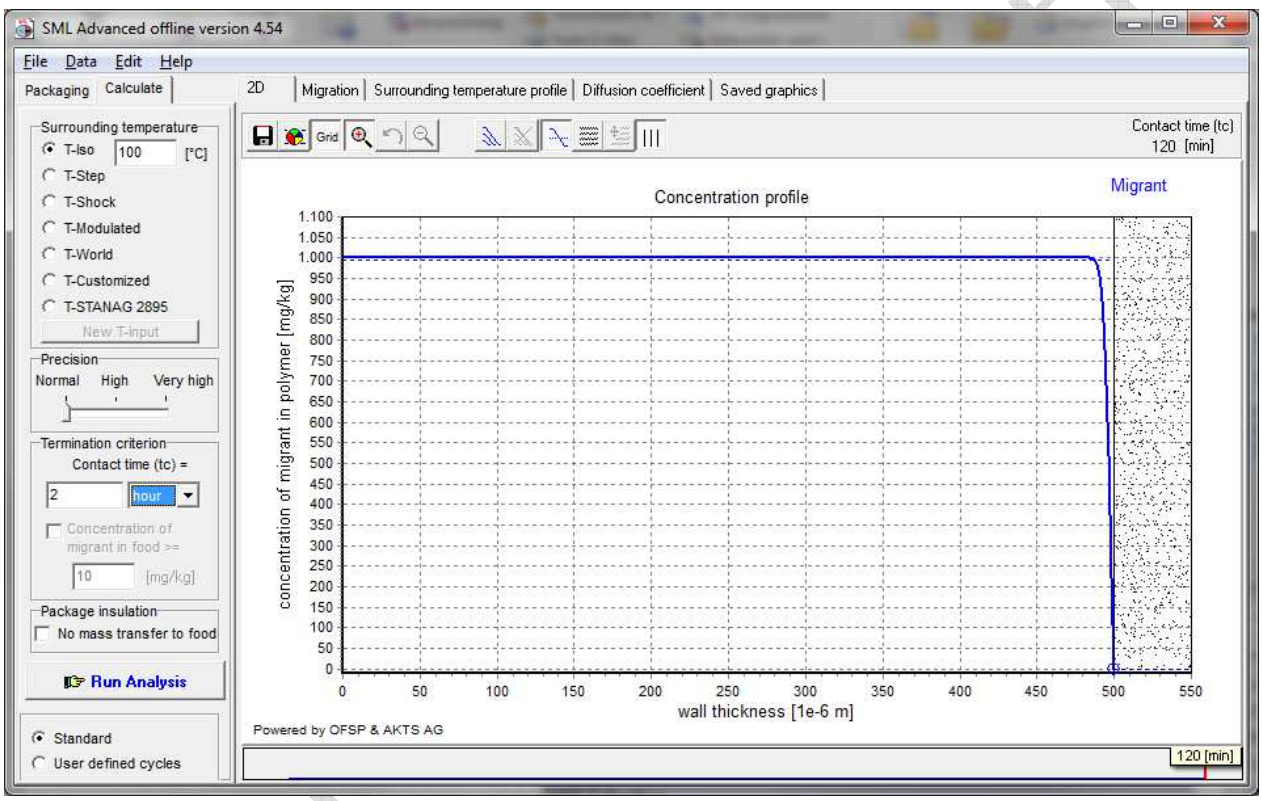
2h @ 100°C

=> 100% layer thickness = 19 µm
no absolute barrier at thicknesses below 19 µm

=> 99% layer thickness = 15 µm

=> 1/2 x 99% layer thickness = 7.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 30 µm
above 30 µm two sides to be considered for calculation of migration if full immersion testing applied



PVC, rigid
(not plasticized; not swollen: e.g. contact with simulant D2, iso-octane)

► molecular mass 100 - 250 g/mol

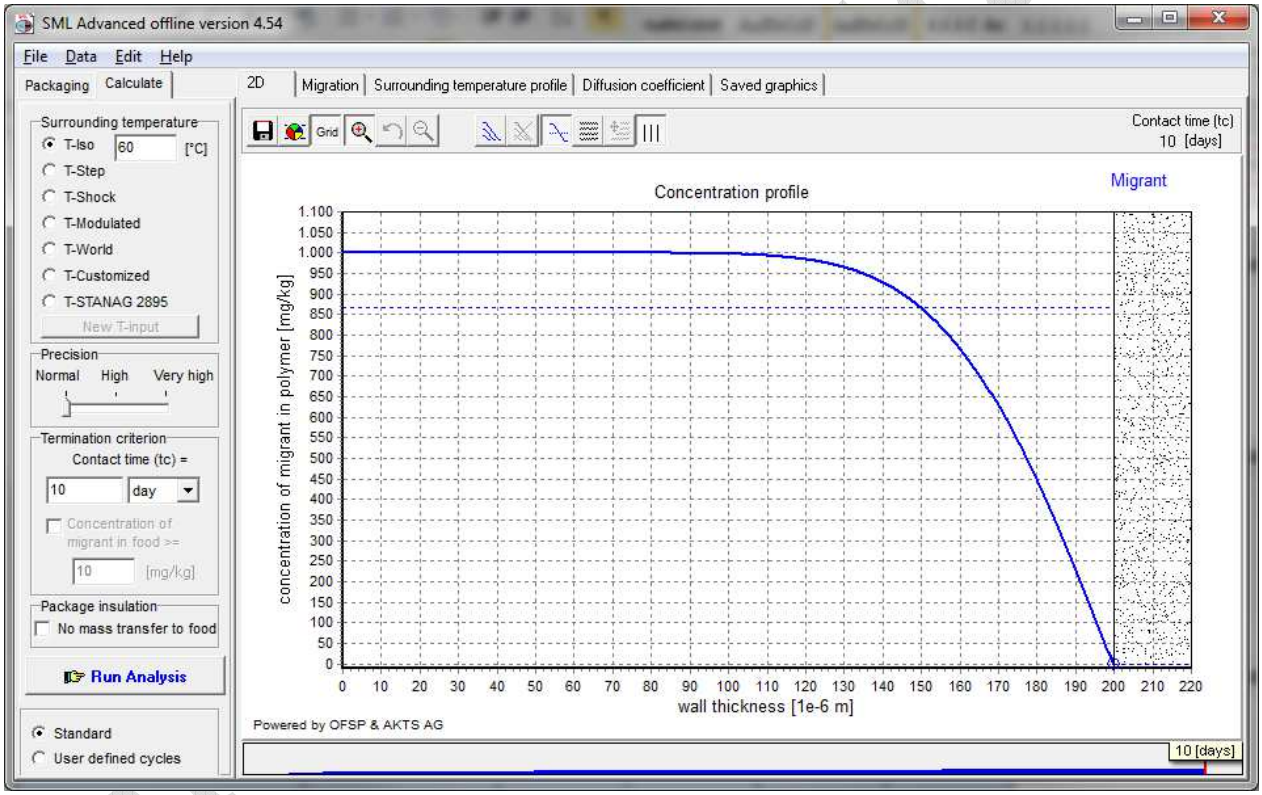
10d @ 60°C

=> 100% layer thickness = 127
no absolute barrier at thicknesses below 127 µm

=> 99% layer thickness = 110 µm

=> 1/2 x 99% layer thickness = 55 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 220 µm
above 220 µm two sides to be considered for calculation of migration if full immersion testing applied



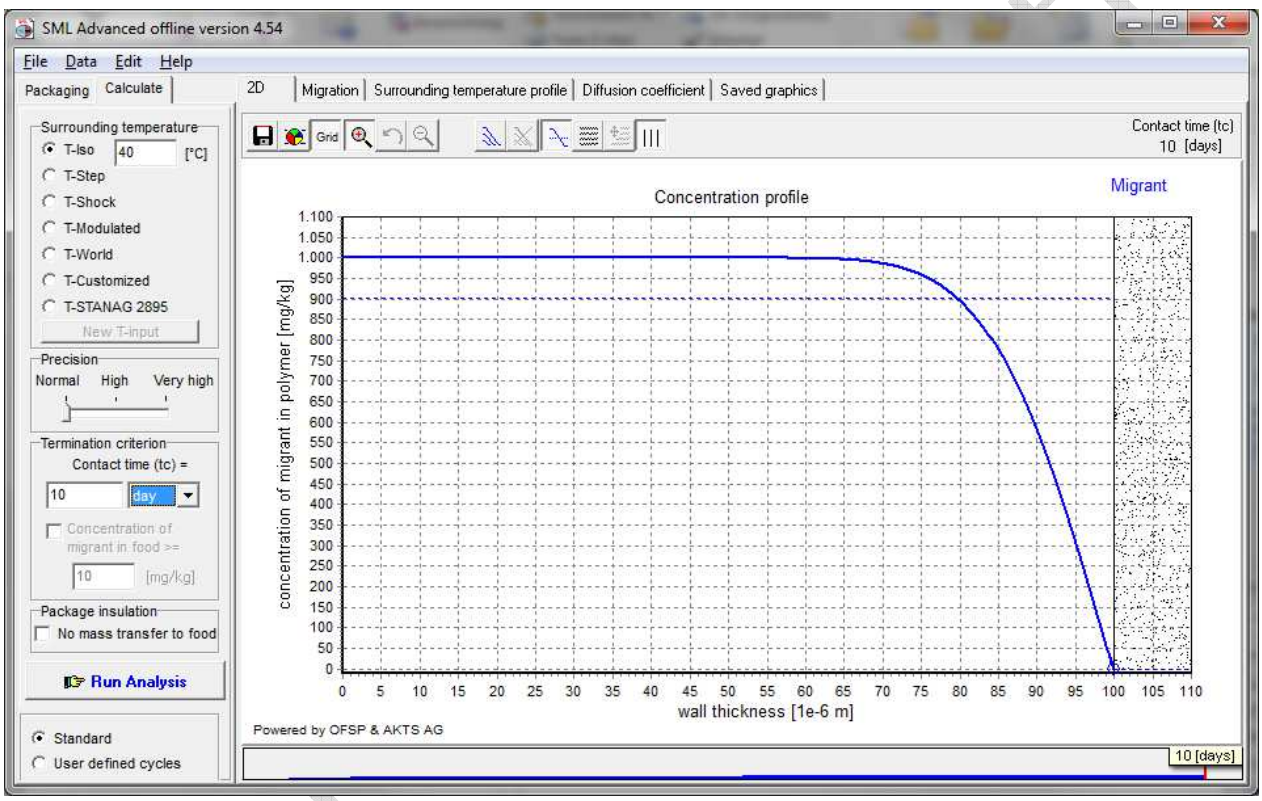
10d @ 40°C

=> 100% layer thickness = 46
no absolute barrier at thicknesses below 46 µm

=> 99% layer thickness = 40 µm

=> 1/2 x 99% layer thickness = 20 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 80
above 80 µm two sides to be considered for calculation of migration if full immersion testing applied



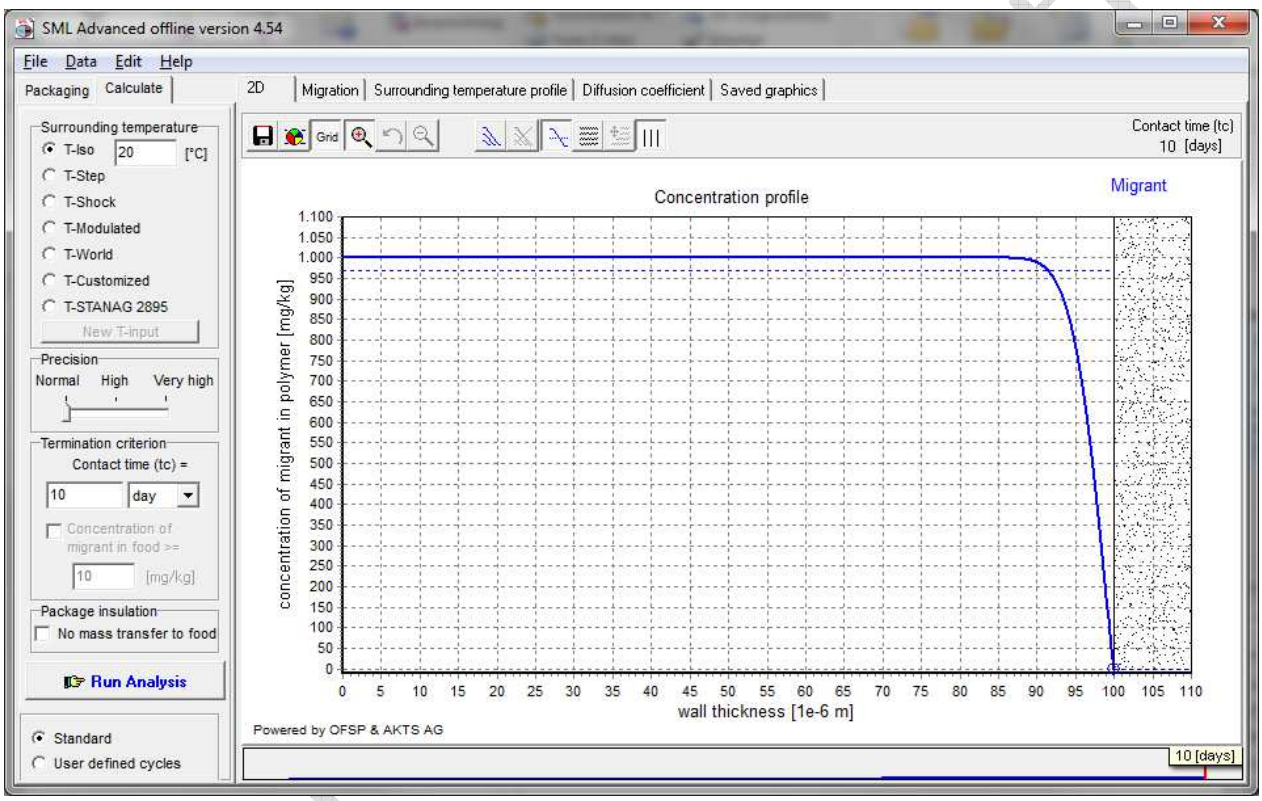
10d @ 20°C

=> 100% layer thickness = 17 µm
no absolute barrier at thicknesses below 17 µm

=> 99% layer thickness = 13 µm

=> 1/2 x 99% layer thickness =7 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 26 µm
above 26 µm two sides to be considered for calculation of migration if full immersion testing applied



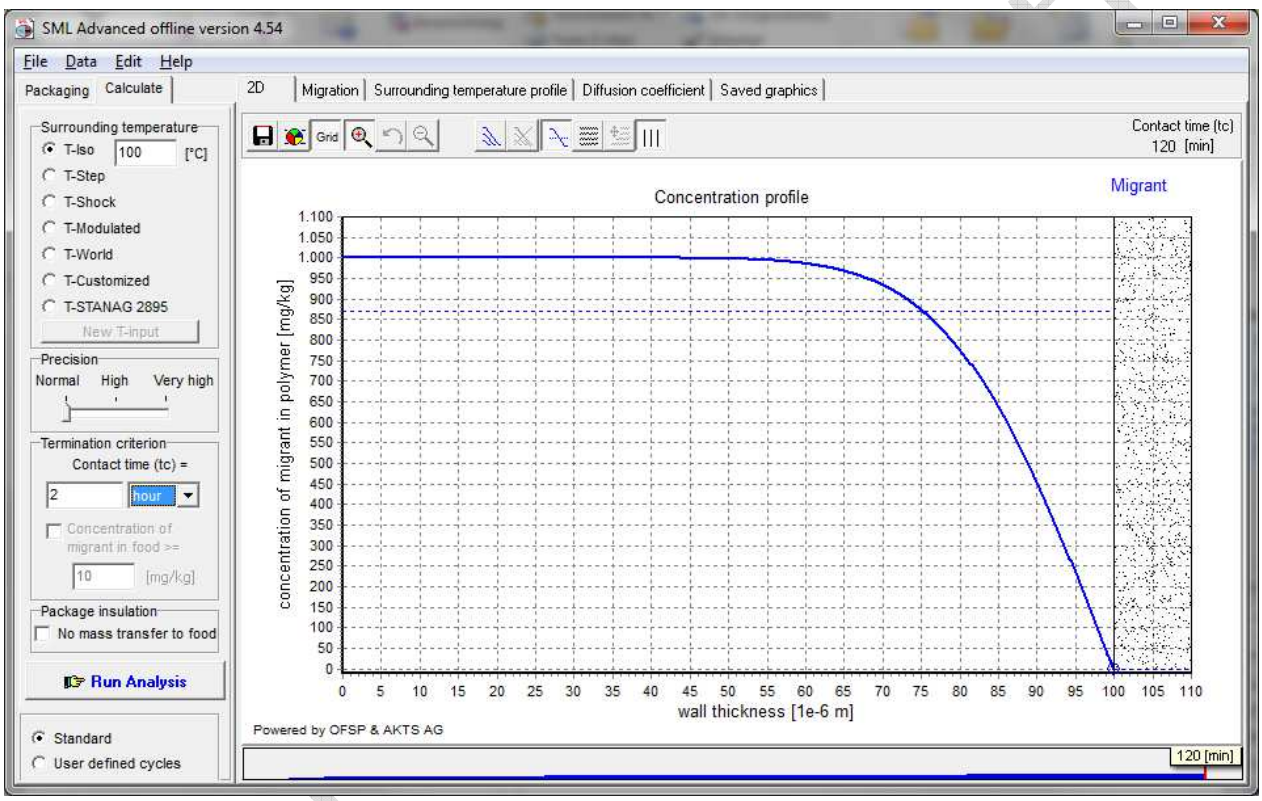
2h @ 100°C

=> 100% layer thickness = 65 µm
no absolute barrier at thicknesses below 65 µm

=> 99% layer thickness = 54 µm

=> 1/2 x 99% layer thickness = 27 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 108 µm
above 108 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 251 - 500 g/mol**

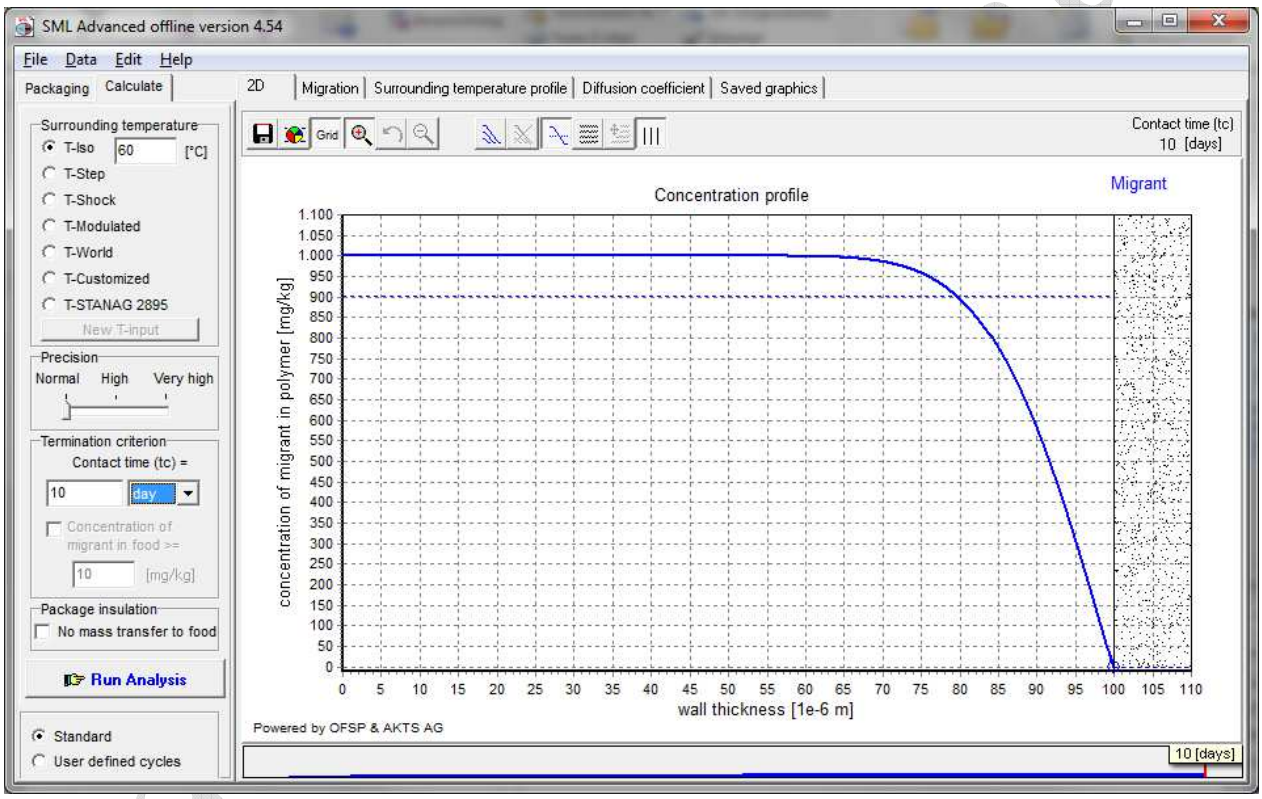
10d @ 60°C

=> 100% layer thickness = 49 µm
no absolute barrier at thicknesses below 49 µm

=> 99% layer thickness = 41 µm

=> 1/2 x 99% layer thickness = 20.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 82 µm
above 82 µm two sides to be considered for calculation of migration if full immersion testing applied



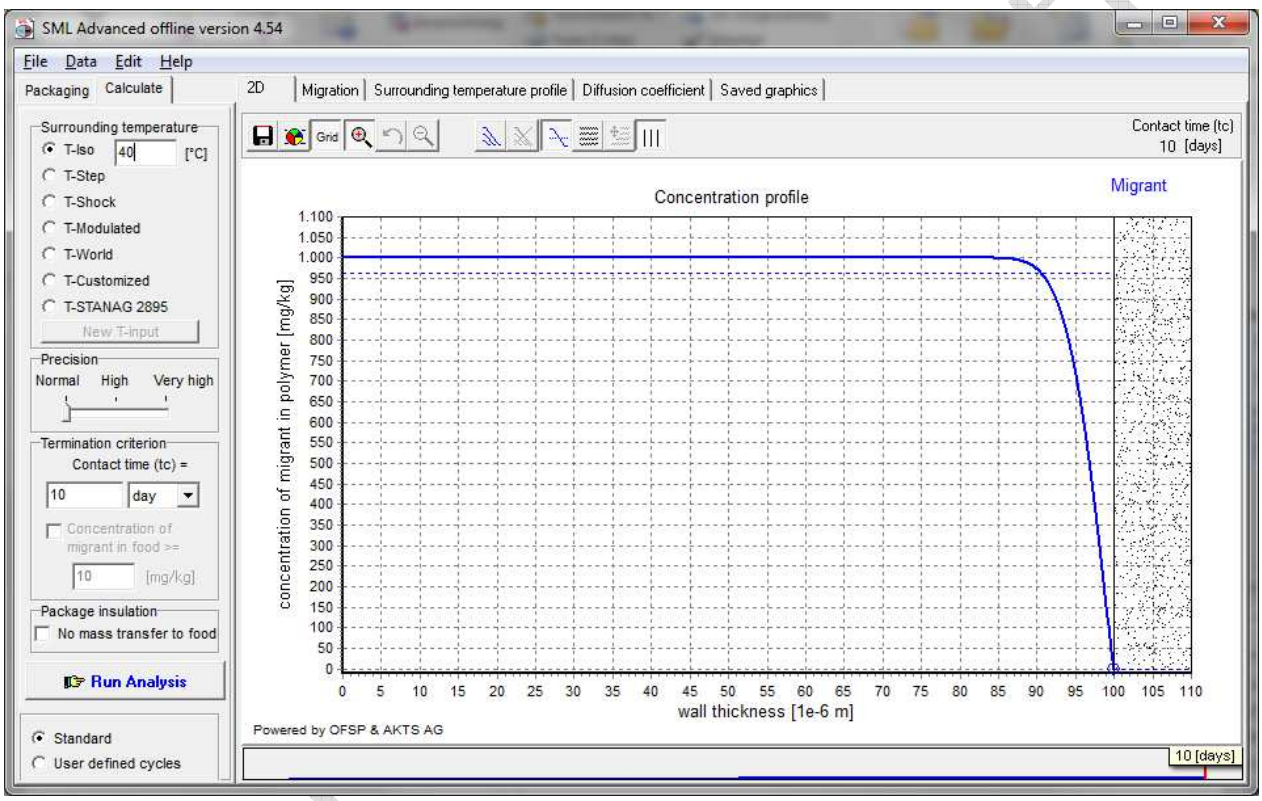
10d @ 40°C

=> 100% layer thickness = 18 µm
no absolute barrier at thicknesses below 18 µm

=> 99% layer thickness = 15 µm

=> 1/2 x 99% layer thickness = 7.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 30 µm
above 30 µm two sides to be considered for calculation of migration if full immersion testing applied



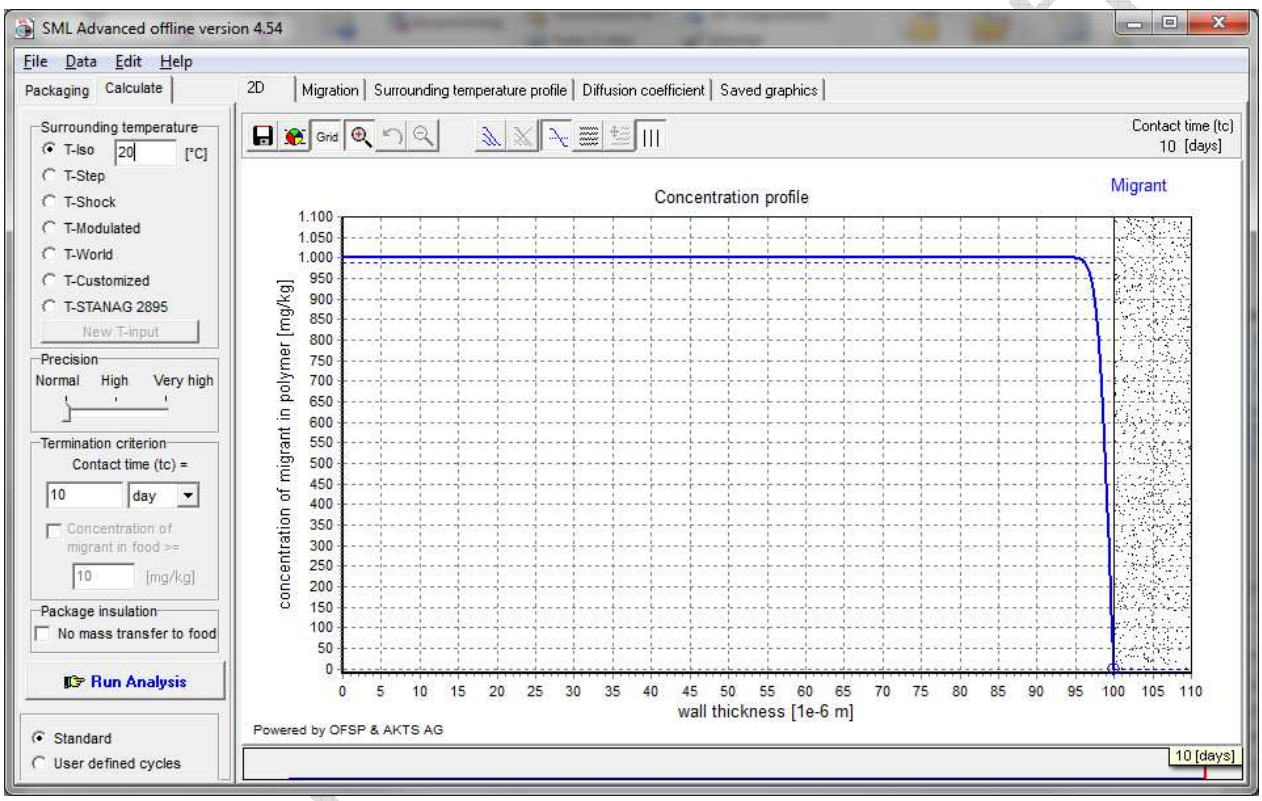
10d @ 20°C

=> 100% layer thickness = 6.2 µm
no absolute barrier at thicknesses below 6.2 µm

=> 99% layer thickness = 5 µm

=> 1/2 x 99% layer thickness = 2.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 10 µm
above 10 µm two sides to be considered for calculation of migration if full immersion testing applied



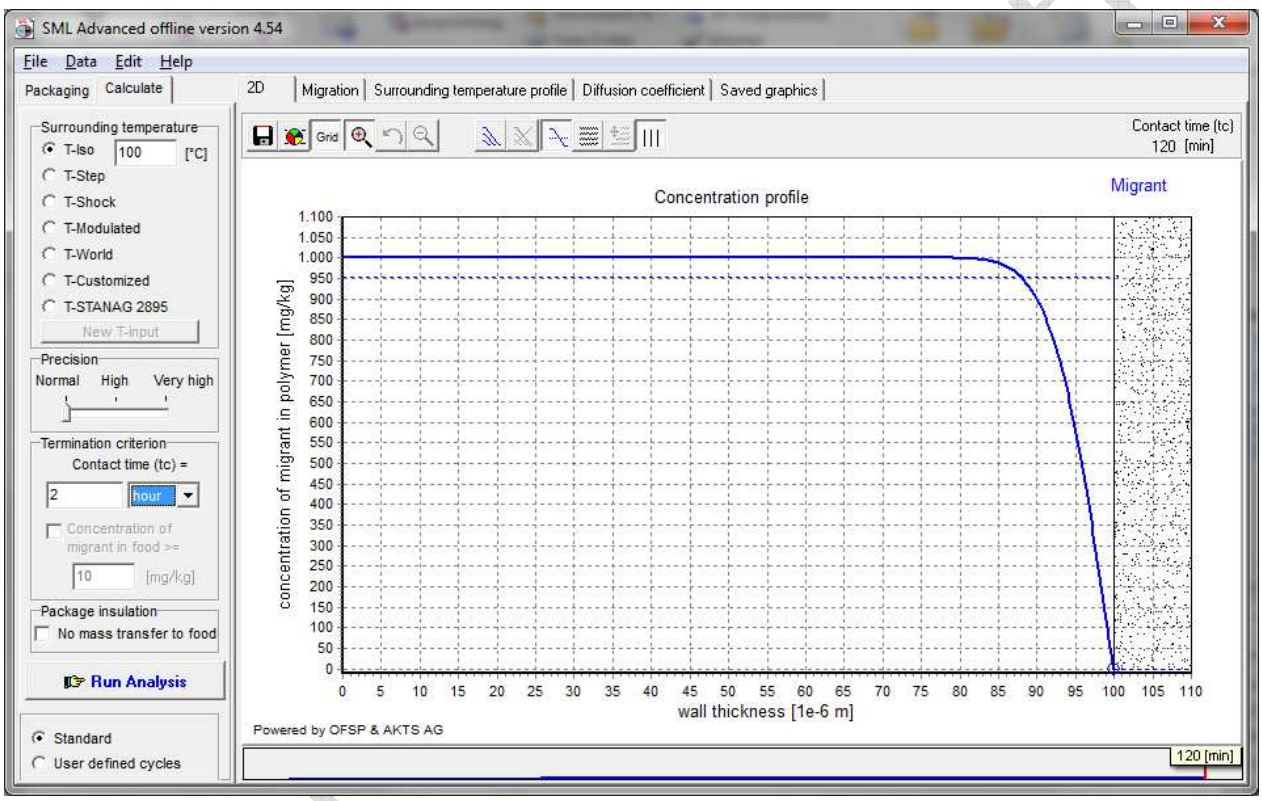
2h @ 100°C

=> 100% layer thickness = 26 µm
no absolute barrier at thicknesses below 26 µm

=> 99% layer thickness = 20 µm

=> 1/2 x 99% layer thickness = 10 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 40 µm
above 40 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 501 - 750 g/mol**

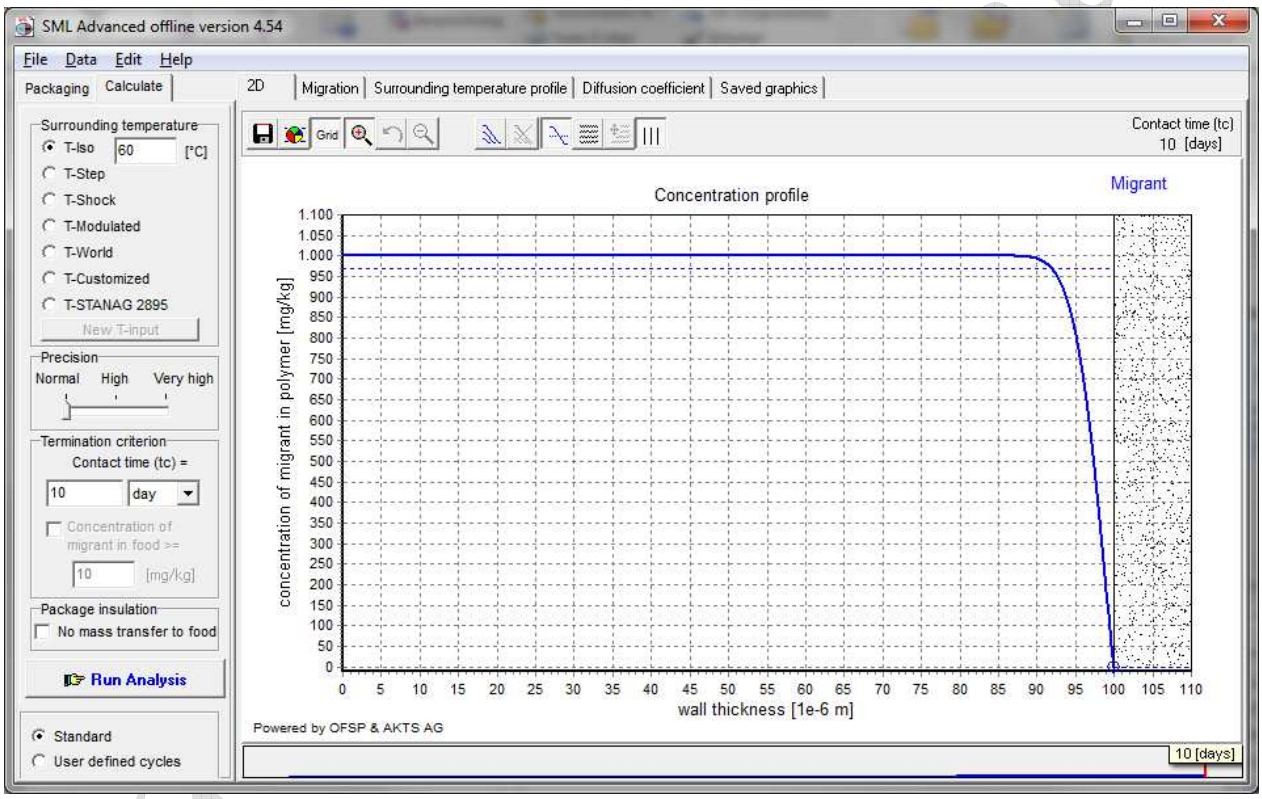
10d @ 60°C

=> 100% layer thickness = 15.2 µm
no absolute barrier at thicknesses below 15.2 µm

=> 99% layer thickness = 12.4 µm

=> 1/2 x 99% layer thickness = 6.2 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 24.8 µm
above 24.8 µm two sides to be considered for calculation of migration if full immersion testing applied



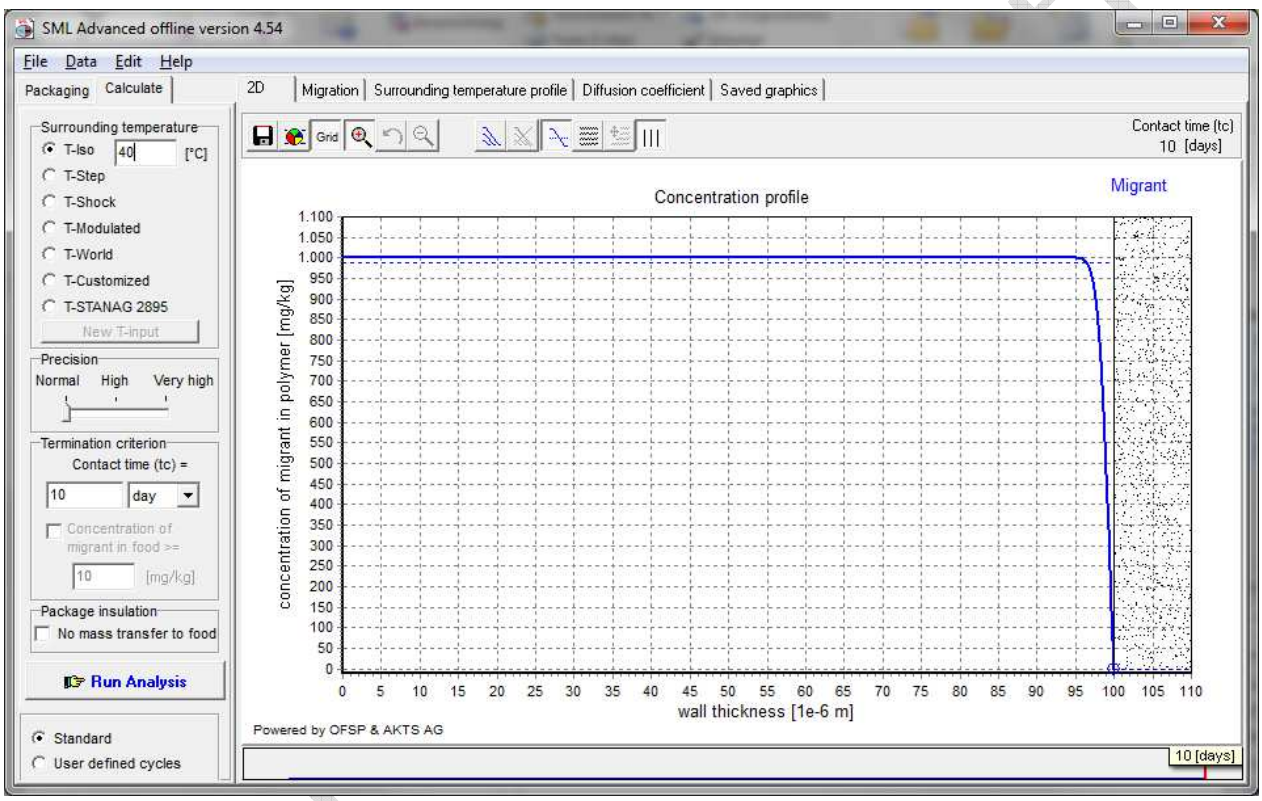
10d @ 40°C

=> 100% layer thickness = 6 µm
no absolute barrier at thicknesses below 6 µm

=> 99% layer thickness = 5 µm

=> 1/2 x 99% layer thickness = 2.5 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 10 µm
above 10 µm two sides to be considered for calculation of migration if full immersion testing applied



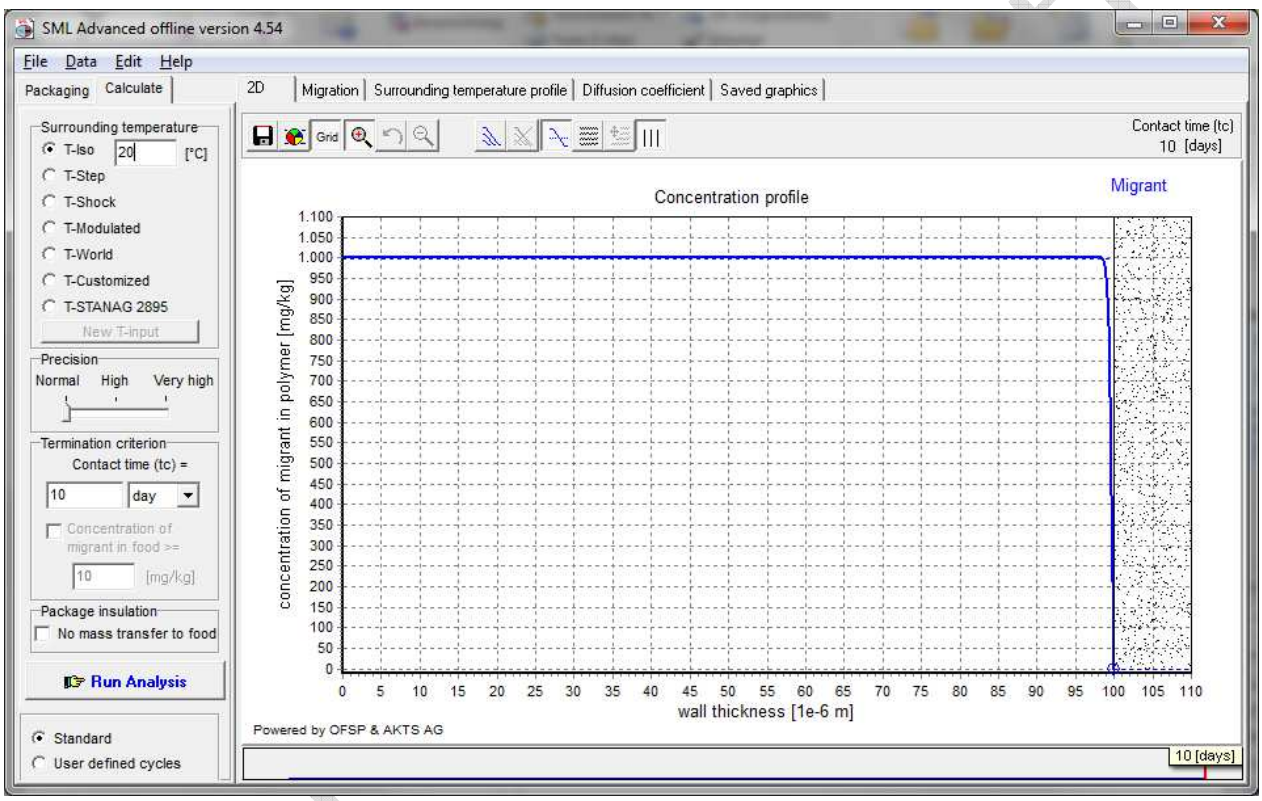
10d @ 20°C

=> 100% layer thickness = 2.2 µm
no absolute barrier at thicknesses below 2.2 µm

=> 99% layer thickness = 1.6 µm

=> 1/2 x 99% layer thickness = 0.8 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 3.2 µm
above 3.2 µm two sides to be considered for calculation of migration if full immersion testing applied



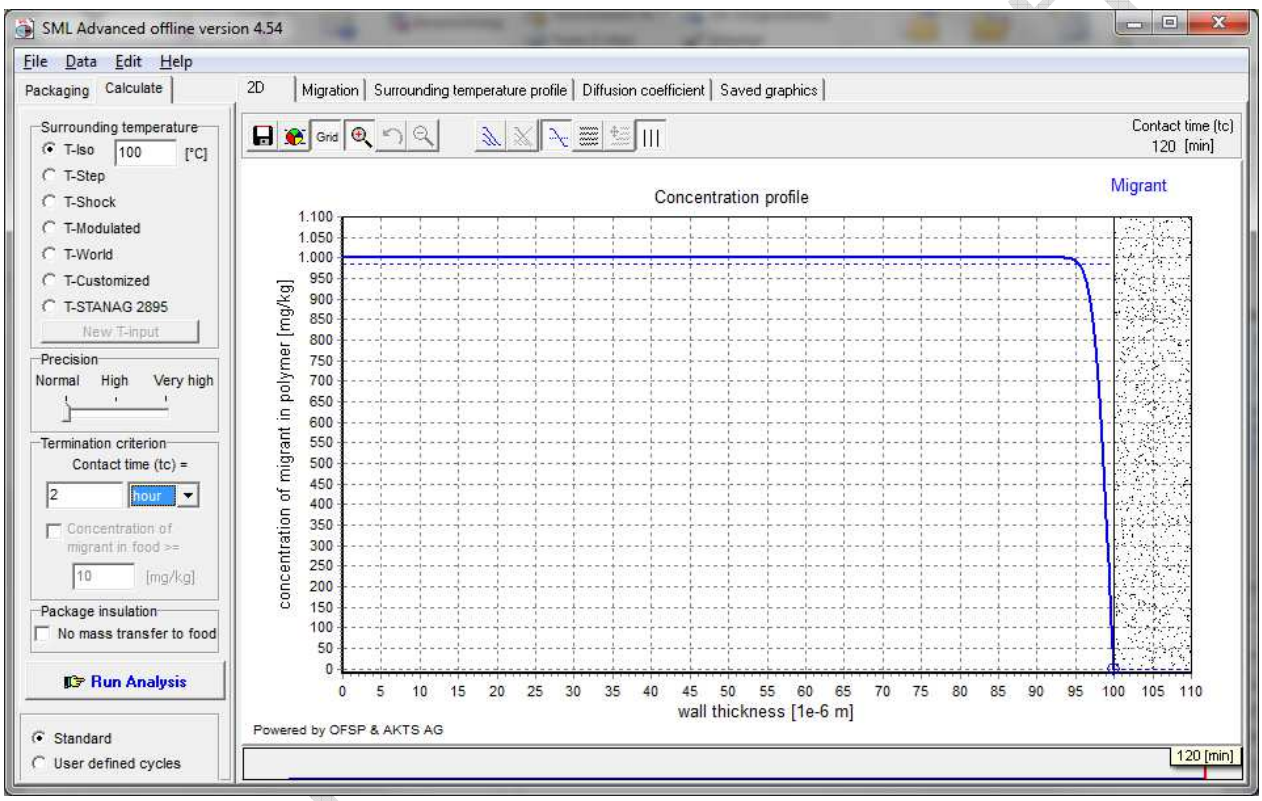
2h @ 100°C

=> 100% layer thickness = 8 µm
no absolute barrier at thicknesses below 8 µm

=> 99% layer thickness = 6.2 µm

=> 1/2 x 99% layer thickness = 3.1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 12.4 µm
above 12.4 µm two sides to be considered for calculation of migration if full immersion testing applied



► **molecular mass 751 - 1000 g/mol**

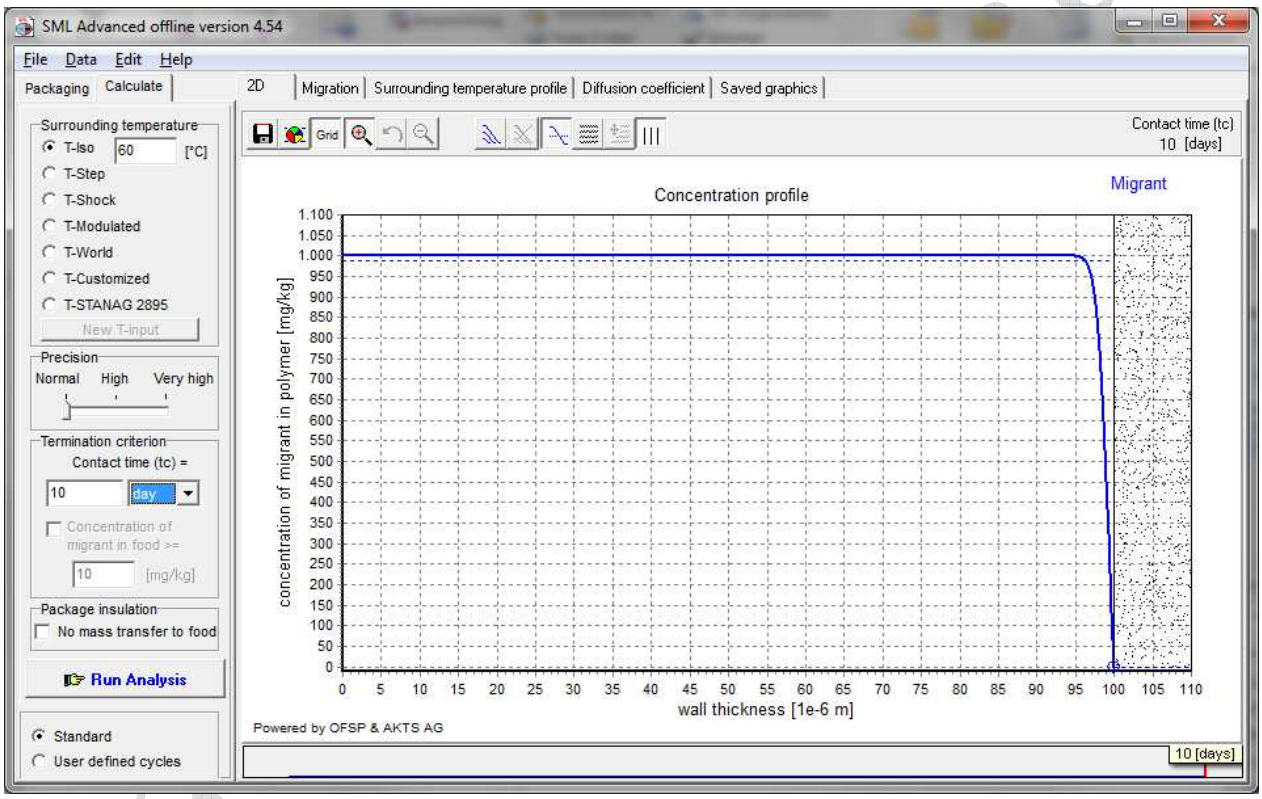
10d @ 60°C

=> 100% layer thickness = 6 µm
no absolute barrier at thicknesses below 6 µm

=> 99% layer thickness = 4.8 µm

=> 1/2 x 99% layer thickness = 2.4 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 9.2 µm
above 9.2 µm two sides to be considered for calculation of migration if full immersion testing applied



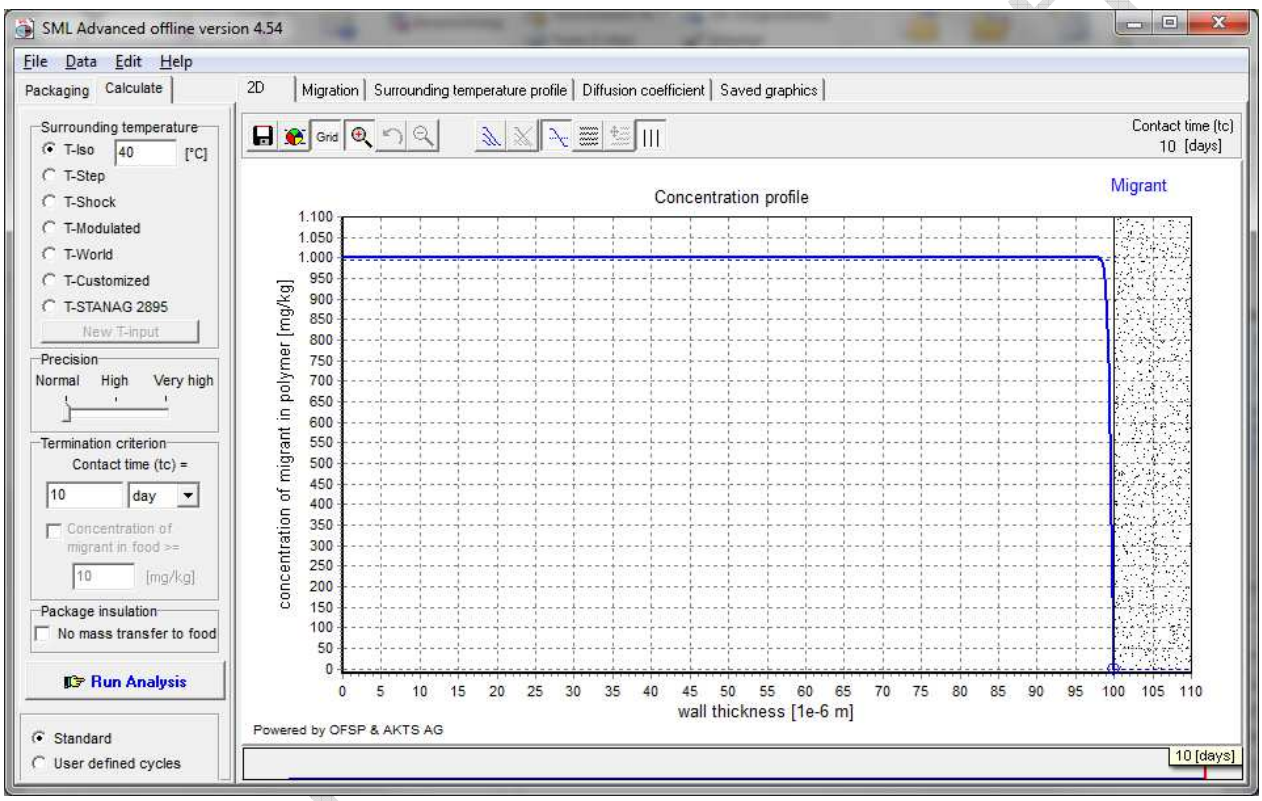
10d @ 40°C

=> 100% layer thickness = 2.8 µm
no absolute barrier at thicknesses below 2.8 µm

=> 99% layer thickness = 2 µm

=> 1/2 x 99% layer thickness = 1 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 4 µm
above 4 µm two sides to be considered for calculation of migration if full immersion testing applied



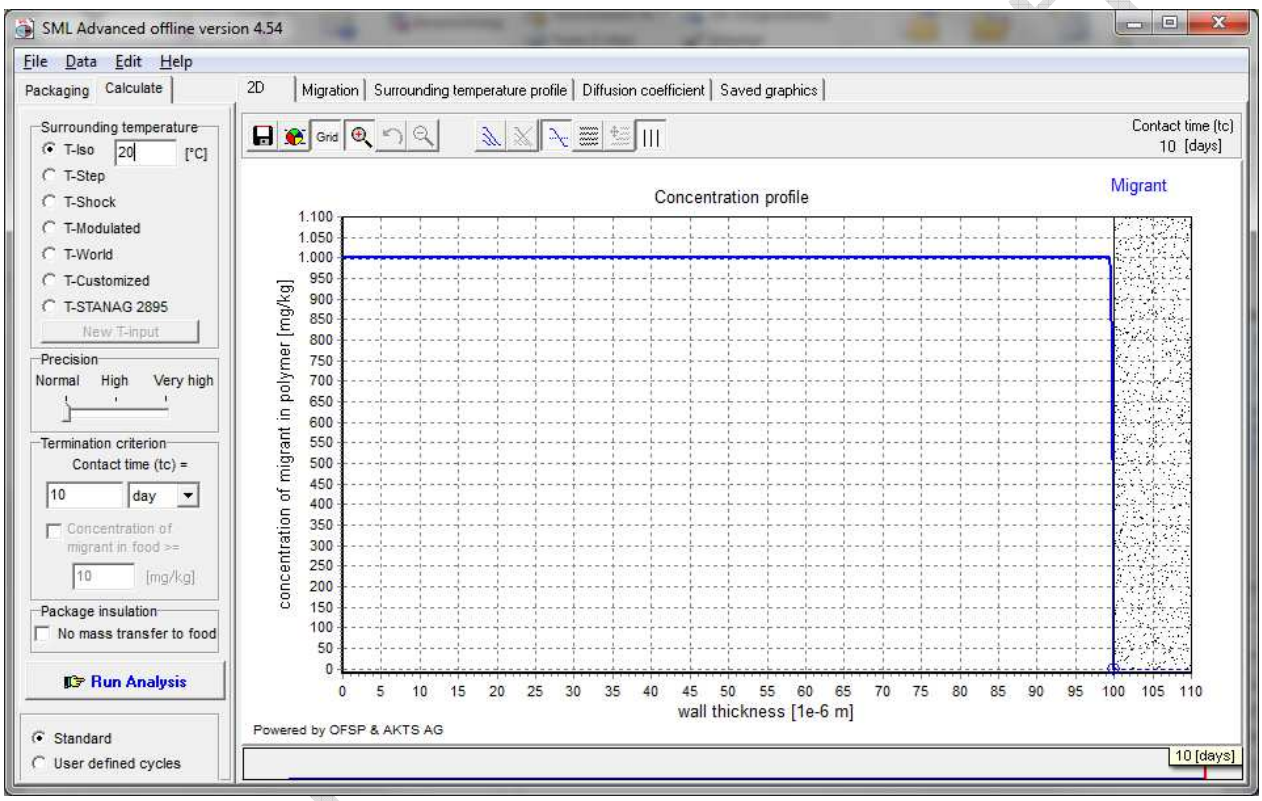
10d @ 20°C

=> 100% layer thickness = 0.8 µm
no absolute barrier at thicknesses below 0.8 µm

=> 99% layer thickness = 0.6 µm

=> 1/2 x 99% layer thickness = 0.3 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 1.2 µm
above 1.2 µm two sides to be considered for calculation of migration if full immersion testing applied



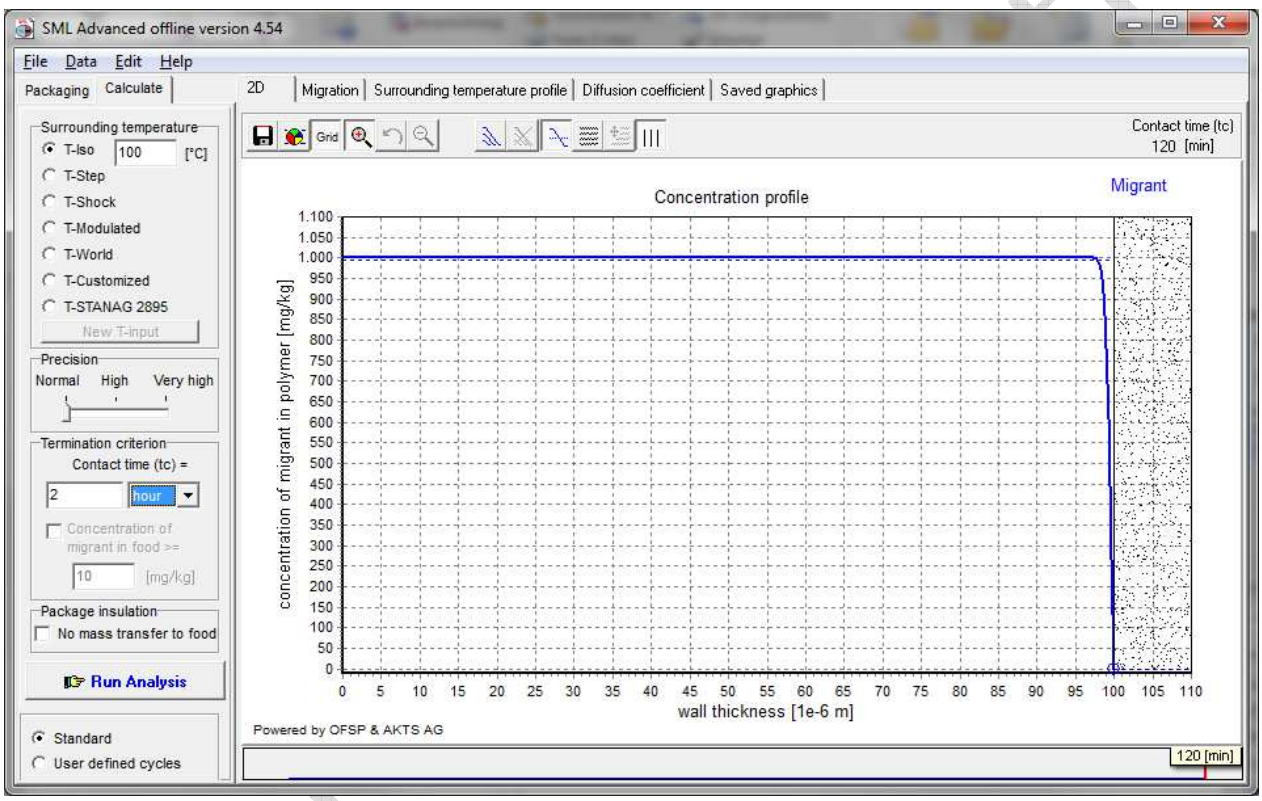
2h @ 100°C

=> 100% layer thickness = 3.3 µm
no absolute barrier at thicknesses below 3.3 µm

=> 99% layer thickness = 2.6 µm

=> 1/2 x 99% layer thickness = 1.3 µm
to be used for worst case calculation of specific migration under assumption of total transfer

=> 2 x 99% layer thickness = 5.2 µm
above 5.2 µm two sides to be considered for calculation of migration if full immersion testing applied



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Annex 5 Section 5.2.1

EXAMPLE 1 – Determination of compliance with an SML using the OM result

SM of 2,4,6-triamino-1,3,5-triazine (CAS No. 108-78-1) into simulant D2

(a) Taking into account the analytical tolerance

Overall migration of a film was determined after 10 days at 40°C in food simulant D2. The average overall migration value was 1 mg/dm^2. The conventional surface-to-volume ratio of 6 dm^2/kg of food leads to an equivalent to 6 mg/kg food simulant. Taking into account the analytical tolerance for the determination of OM into food simulant D2 (18 mg/kg) then the maximum overall migration value (measured value + analytical tolerance) is 24 mg/kg food simulant. The SML for 2,4,6-triamino-1,3,5-triazine given in Regulation (EU) No 10/2011 is 30 mg/kg. Therefore the overall migration value including analytical tolerance is below the SML. Therefore it can be concluded that as long as the migrant is stable in the food simulant during the contact phase and that it is not lost by volatilisation then for all SM conditions equal to or less severe than 10 days at 40°C the overall migration result can be used to confirm that the migration of 2,4,6-triamino-1,3,5-triazine into fatty food is in compliance with the SML.

(b) Taking into account the stability of the analyte

Considering the structure of 2,4,6-triamino-s-triazine it is not expected that it will breakdown during contact but it is expected that it will react with food simulant D2. However the reaction products formed are not volatile and therefore it can be concluded that as long as the migrant and its reaction products is not lost by volatilisation then for all SM conditions equal to or less severe than 10 days at 40°C the overall migration result can be used to confirm that the migration of 2,4,6-triamino-1,3,5-triazine into fatty food is in compliance with the SML.

(c) Taking into account the volatility of the analyte

From the literature the melting point of 345°C is known, i.e. the migrant is non-volatile. Therefore it can be concluded that for all SM conditions equal to or less severe than 10 days at 40°C the overall migration result can be used to confirm that the migration of 2,4,6-triamino-1,3,5-triazine into fatty food is in compliance with the SML.

NOTE: This example is dealing with food simulant D2 however when compliance is determined with food simulant B then the OM may exceed the SML where as the SM of melamine would comply with the SML. It should be noticed that the melamine will react with the acetic acid to form the salt. The molecular mass of melamine is 126 g/mol and the molecular mass of the melamine triacetate is 306 g/mol. So the OM will increase by a factor of (306/126) = 2.4. A SM of 30 mg melamine/kg will result into an OM of 73 mg melamine triacetate /kg. The SM is compliant if the analytical tolerance is included but the OM is non-compliant if the analytical tolerance of 12 mg/kg is included.

EXAMPLE 2 – Determination of compliance with an SML using the OM result

SM of diethylene glycol (CAS No. 111-46-6) into simulant A

(a) Taking into account the analytical tolerance

Overall migration of a film was determined after 10 days at 40°C in food simulant A. The average overall migration value was 1 mg/dm^2. The conventional surface-to-volume ratio of 6 dm^2/kg of food leads to an equivalent to 6 mg/kg food simulant. Taking into account the analytical tolerance for the determination of OM into food simulant A (12 mg/kg) then the maximum overall migration value (measured value + analytical tolerance) is 18 mg/kg food simulant. The SML(T) for diethylene glycol given in Regulation (EU) No 10/2011 is 30 mg/kg. Therefore it can be concluded that as long as the migrant is stable in the food simulant during the contact phase

and that it is not lost by volatilisation then for all SM conditions equal to or less severe than 10 days at 40°C the overall migration result can be used to confirm that the migration of diethylene glycol into food simulant A is in compliance with the SML.

(b) Taking into account the stability of the analyte

Considering the structure of diethylene glycol it is not expected that it will breakdown during the contact time and nor is it expected that it will react with simulant A and therefore it can be concluded that as long as the migrant is not lost by volatilisation then for all SM conditions equal to or less severe than 10 days at 40°C the overall migration result can be used to confirm that the migration of diethylene glycol into simulant A is in compliance with the SML.

(c) Taking into account the volatility of the analyte

From the literature the boiling point of 244°C is known, i.e. the analyte is semi-volatile. It has previously been demonstrated Bradley et al. (2009) that the recovery of diethylene glycol following evaporation of aqueous simulants is 0%. Therefore for this substance the volatility is such that the compliance with the SML cannot be demonstrated by the determination of the OM.

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FCM Subst. No	PM Ref. No	CAS No	Substance name	SML group	SML mg/kg	b.p. [°C] @ 760Torr	Simulant	Recovery [%]	SM through OM possible
14	33801		n-alkyl(C10-C13) benzenesulphonic acid		30	---		No data available	YES (1)
15	34130		alkyl, linear with even number of carbon atoms (C12-C20) dimethylamines		30	---		No data available	YES (1)
69	74400		phosphorous acid, tris(nonyl-and/or dinonylphenyl) ester		30	---		No data available	YES (1)
74	77440		polyethyleneglycol diricinoleate		42	---		No data available	YES (1)
91	92320		tetradecyl-polyethyleneglycol (EO = 3-8) ether of glycolic acid		15	---		No data available	YES (3)
159	74560	0000085-68-7	phthalic acid, benzyl butyl ester	32	30	370		No data available	YES (1)
160	84800	0000087-18-3	salicylic acid, 4-tert-butylphenyl ester		12	368		No data available	YES (4)
177	16955	0000096-49-1	ethylene carbonate		30	248		No data available	YES (1)
207	31920	0000103-23-1	adipic acid, bis(2-ethylhexyl) ester	32	18	390 - 417		No data available	YES (2)
209	17050	0000104-76-7	2-ethyl-1-hexanol		30	185		No data available	YES (1)
217	15565	0000106-46-7	1,4-dichlorobenzene		12	175-180			NO

226	15272	0000107-15-3	ethylenediamine		12	118			NO
230	16150	0000108-01-0	dimethylaminoethanol		18	135			NO
231	10120	0000108-05-4	acetic acid, vinyl ester		12	73			NO
239	19975	0000108-78-1	2,4,6-triamino-1,3,5-triazine		30	Decompos .@ 352	3%Acetic acid	76 +/-5	YES (1) Recovery correction factor 100/71
264	22660	0000111-66-0	1-octene		15	121			NO
284	84880	0000119-36-8	salicylic acid, methyl ester		30	222		No data available	YES (1)
324	83700	0000141-22-0	ricinoleic acid		42	245@10T orr	95%EtOH	94+/-5	YES (1)
353	42480	0000584-09-8	carbonic acid, rubidium salt		12	-----		No data available	YES (4)
383	72160	0000948-65-2	2-phenylindole		15	250@10T orr		No data available	YES (3)
384	40000	0000991-84-4	2,4-bis(octylmercapto) -6-(4-hydroxy-3,5-di-tert -butylanilino)-1,3,5-triazine		30	670.7±65. 0		No data available	YES (1)
532	88640	0008013-07-8	soybean oil, epoxidised		60 resp. 30	-----		No data available	YES (1)
563	78320	0009004-97-1	polyethyleneglycol monoricinoleate		42	----		No data available	YES (1)
633	53200	0023949-66-8	2-ethoxy-2′-ethyloxanilide		30	>400		No data available	YES (1)
635	40720	0025013-16-5	tert-butyl-4-hydroxyanisole		30	268		No data available	YES (1)

658	52000	0027176-87-0	dodecylbenzenesulphonic acid		30	-----		No data available	YES (1)
675	38800	0032687-78-8	N,N′-bis(3-(3,5-di-tert-butyl -4-hydroxyphenyl)propionyl) hydrazide		15	652.6±55.0		No data available	YES (3)
688	92560	0038613-77-3	tetrakis(2,4-di-tert-butyl-phenyl)-4,4′-biphenylylene diphosphonite		18	854.2±65.0		No data available	YES (2)
708	77520	0061791-12-6	polyethyleneglycol ester of castor oil		42	-----		No data available	YES (1)
710	38700	0063397-60-4	bis(2-carbobutoxyethyl)tin-bis (isooctyl mercaptoacetate)		18	-----		No data available	YES (2)
711	42000	0063438-80-2	(2-carbobutoxyethyl)tin-tris (isooctyl mercaptoacetate)		30	-------		No data available	YES (1)
716	60800	0065447-77-0	1-(2-hydroxyethyl)-4-hydroxy -2,2,6,6-tetramethyl piperidine-succinic acid, dimethyl ester, copolymer		30	------		No data available	YES (1)
760	83595	0119345-01-6	reaction product of di-tert-butylphosphonite with biphenyl, obtained by condensation of 2,4-di-tert-butylphenol with Friedel Craft reaction product of phosphorous trichloride and biphenyl		18	------		No data available	YES (2)
798	92200	0006422-86-2	terephthalic acid, bis(2-ethylhexyl)ester	32	60	400		No data available	YES (1)
811	80077	0068441-17-8	polyethylene waxes, oxidised		60	------		No data available	YES (1)

89	89440		stearic acid, esters with ethyleneglycol	2	30	-------		No data available	YES (1)
227	16990	0000107-21-1	ethyleneglycol	2	30	198	H2O 95%EtOH	0 3+/-3	NO
263	13326	0000111-46-6	diethyleneglycol	2	30	248	H2O 95%EtOH	0 6+/-7	NO
248	19540	0000110-16-7	maleic acid	3	30	110-138	H2O	72+/-15	YES (1) Recovery correction. factor 100/60
248	19540	0000110-16-7	maleic acid	3	30	110-138	95%EtOH	41+/-10	NO for ethanolic solutions
234	19960	0000108-31-6	maleic anhydride	3	30	202		No data available	As for maleic acid
212	14200	0000105-60-2	caprolactam	4	15	270	H2O 95%EtOH	0 10+/-3	NO
435	14230	0002123-24-2	caprolactam, sodium salt	4	15	-------			NO
444	61440	0002440-22-4	2-(2′-hydroxy-5′-methylphenyl)benzotriazole	12	30	225@10Torr		No data available	YES (1)
469	60480	0003864-99-1	2-(2′-hydroxy-3,5′-di-tert-butylphenyl)-5-chlorobenzotriazole	12	30	469.2±55.0		No data available	YES (1)
470	60400	0003896-11-5	2-(2′-hydroxy-3′-tert-butyl-5′-methylphenyl)-5-chlorobenzotriazole	12	30	460.4±55.0		No data available	YES (1)
98	17260	0000050-00-0	formaldehyde	15	15	-21			NO

196	18670	0000100-97-0	hexamethylenetetramine	15	15	280	H2O 95%EtOH	0 4+/-4	NO
290	55360	0000121-79-9	gallic acid, propyl ester	20	30	448.6±40.0		No data available	
386	55280	0001034-01-1	gallic acid, octyl ester	20	30	------		No data available	
390	55200	0001166-52-5	gallic acid, dodecyl ester	20	30	-------		No data available	
73	76866		polyesters of 1,2-propanediol and/or 1,3- and/or 1,4-butanediol and/or polypropyleneglycol with adipic acid, which may be end-capped with acetic acid or fatty acids C12-C18 or n-octanol and/or n-decanol	31	30	------		No data available	YES (1)
797	76807	0007328-26-5	polyester of adipic acid with 1,3-butanediol, 1,2-propanediol and 2-ethyl-1-hexanol	31	30	------		No data available	YES (1)
FCM no.'s 8, 72, 73, 138, 140, 157, 159, 207, 242, 283, 532, 670, 728, 729, 775, 783, 797, 798, 810,				32	60	-------	all	No data available	YES (1)

815									

Explanations:

YES (1): all food simulants, if recovery is ok

YES (2): for volatile food simulants, if recovery is ok
borderline for oils as simulant, if recovery is ok

YES (3): for volatile food simulants, if recovery is ok
NOT for oils as food simulant

YES (4): borderline for volatile food simulants, if recovery is ok
NOT for oils as food simulants

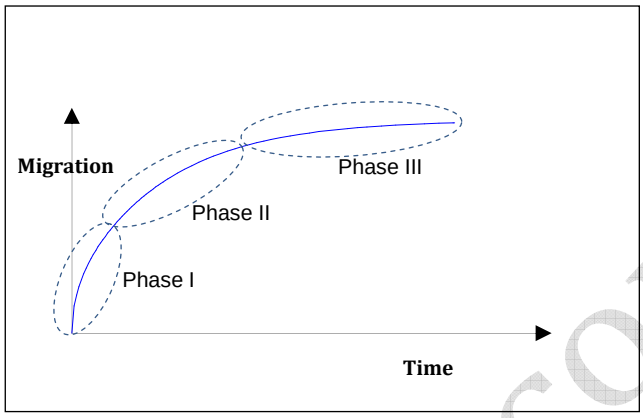
NO: all food simulants, because SML too low or due to poor recovery

Annex 6 Section 5.2.5 background document

Scientific reasoning for use of screening food simulants instead of vegetable oils and related testing conditions.

The following two principal contributions to the migration behaviour in the system food contact material in contact with food simulants will determine the result of a migration test: 1) thermodynamic contribution, i.e. relative solubility of the migrant between plastic and simulant, described by the partition coefficient of the migrant between both matrices 2) kinetic contribution, i.e. diffusion rate of the migrant in and from the plastic into the simulant. Typically a migration curve (understood as migration as a function of time) for polymers in contact with liquids consists of 3 phases: an initial phase (phase I) where the migration is controlled only or largely by the kinetic contribution and final phase (phase III) where the migration it controlled by the thermodynamic contribution. In between of both phase the migration is influenced by both elements (Figure 1).

Figure 1: Typical migration curve showing three basic phases of migration



A specific migration test for a plastic with an screening food simulant will be more severe compared to the conventional migration test if: (a) both of the two contributions are more severe, or (b) one contribution dominates the other, i.e. the more severe contribution compensates the less severe contribution, resulting overall again in a more severe migration test.

To prevent testing under to severe conditions resulting in extremely overestimating and totally unrealistic results testing migration with an screening simulant may not employ only an screening food simulant but also appropriately different time/temperature testing conditions.

Deviation from food simulants and conventional time - temperature conditions for migration testing according to PIM is possible based on scientific evidence. However, currently not enough data and knowledge exists to establish a general rule covering all possible migration test cases. Especially, the increase of migration rates due to swelling effects in the polymer strongly depends on the nature of the polymer and the type of simulant and is not predictable based on today's knowledge.

Thermodynamic considerations

Thermodynamic considerations should focus in the first place on the solubility of the migrant in the screening food simulant. The relative solubility of the migrant, understood as the ratio of concentrations of the migrant in the FC polymer and the food simulant at equilibrium (which is the partition coefficient) determines in which amounts the migrant will migrate in phases II and III of the migration process.

From a purely thermodynamic point of view the requirement that the migration tests with the screening food simulants must be more severe compared to the test with vegetable oil implies

that the solubility of the migrants in the screening food simulant is equal to or higher than in vegetable oils. As a consequence, when applying the same conventional time and temperature conditions as applicable for vegetable oil, at least the same or higher migration test result should be obtained.

Solubility of migrants in screening food simulants

There is a well-known chemical rule of the thumb stating "similar dissolves similar". For screening food simulants this means that they need to have a similar polarity as vegetable oils, which ensures that the solubility of the migrants in the screening food simulant is comparable to or even higher than in vegetable oils.

With introduction of the solubility parameter an important scientific approach which makes the quantification of polarities for liquid media like food simulants or typical solvents and polymers possible was published by Hildebrandt in 1950 [1]. The solubility parameter δ is defined as the square root of the cohesive energy density at 20°C. There are three main contributions to the cohesive energy: (1) contribution of dispersion forces, (2) contribution of polar forces and (3) contribution of hydrogen bonding. The solubility parameter for a medium (solvent or polymer) is calculated $\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$. For a mixture of two solvents the solubility parameter of the mixture is calculated based on the volume fraction of the two solvents.

A substance is soluble in a medium (solvent or polymer) if the solubility parameters are equal or close to each other. According to Yalkowsky [2], any two liquids will be completely miscible at room temperature if their solubility parameters do not differ by more of three units.

Regarding interaction between a polymer and the contact medium the extent of swelling of the polymer by the solvent will be more pronounced if their solubility parameters are close to each other. Solubility parameters can be estimated by the group contribution method introduced by van Krevelen [3].

Based on the above considerations an appropriate screening food simulant (FS) may be selected for a given polymer – migrant system.

In this context it is important to note that due to the lower molecular weight of screening food simulants the entropic contribution of the screening food simulants to the solubility is higher compared to vegetable oils. It can therefore be expected that at the same polarity of an screening FS versus vegetable oil, migrants are better soluble in the screening food simulants compared to vegetable oil or, in other word, the solubility of a migrant in an screening FS is sufficiently high even if the polarity of this FS deviates from that of the edible oil. .

Selection of screening food simulants

Based on the above considerations the selection of an screening food simulant can be made. The requirement is that the result of the migration tests with the screening food simulants must be at least as severe as the test with vegetable oil. This implies that the solubility of the migrants in the screening food simulant is at a minimum as high as in vegetable oils. In combination with the use of conventional time and temperature conditions as applicable for vegetable oil, in general a more severe migration test results will be obtained.

General recommendation for selection of screening food simulants

[1] Hildebrand, J. H.; Scott, R. L. The Solubility of Nonelectro-lytes, 3rd ed. Reinhold: New York, 1950.

[2] Yalkowsky, S. H.; unpublished results. Cited from foot note **Error! Bookmark not defined.**.

[3] VAN KREVELEN D. W., 1990: Properties of polymers. Their correlation with chemical structure; their numerical estimation and prediction from additive group contributions. Elsevier, 3rd Edition, Amsterdam pp. 189-225

According to the scientific considerations [4] esters built from C2 to C4 acids with C2 to C4 alcohols and mixtures of these with aliphatic hydrocarbons with C6 to C8 carbon atoms can be recommended as screening food simulants for migration testing (specific and overall), which most likely will satisfy the requirement that the solubility of the migrants in the screening food simulant is as high as in vegetable oils, due to similar polarity of the screening food simulant with vegetable oil.

For the above general recommendation regarding nature of screening food simulants it was considered that the screening food simulants should exhibit the same chemical functionalities, i.e. ester functionality (dipol-dipol interaction) and segments of hydrocarbons (Van der Waals or dispersion interaction) as vegetable oils.

Because vegetable oils are not capable to undergo hydrogen bonding screening food simulants capable to undergo hydrogen bonding like alcohols are taken into consideration as screening food simulants for vegetable oils only for specific cases, i.e. dedicated polymers. This mean that alcohols like for example ethanol are eligible in specific situations, but from generic point of view due to the hydroxyl functionality they show a different solubilizing mechanism compared to fatty acid esters, i.e. vegetable oils. Similar limitation is valid for use of pure hydrocarbons like iso-octane which are eligible in specific situations, but from general point of view due to lag of functional groups they show a different solubilizing mechanism compared to fatty acid esters, i.e. vegetable oils.

In the further considerations distinction between specific migration testing and overall migration testing is required.

Screening food simulants selection for specific migration testing

The recommendation on screening food simulants selection is based on the rule "similar solves similar", i.e. the closer the polarity of the migrant and the simulant is, the better the solubility of the migrant will be in the simulant. As a measure of polarity the octanol to water partition coefficient ($K_{O/W}$) is used because plenty of scientific literature is in place and numerous estimation procedures including software tools exist.

A migration experiment from thermodynamic point of view involves three parties: (A) the simulant, (B) the migrant and (C) the polymer. Each of these exhibits a polarity which can be expressed by an octanol to water partition coefficient. Establishing octanol to water partition coefficient for polymers is unlikely, but based on some conventional assumption an estimate can be made.

Starting point is a specific migration experiment for a migrant from a plastic with vegetable oil as required by legislation. The migrant will exhibit an octanol to water partition coefficient of $K_{O/W}$(mig) and the vegetable oil will exhibit an octanol to water partition coefficient of $K_{O/W}$(oil). Octanol to water partition coefficients for vegetable oils are in the range of 20 to 30 [5], e.g. for Tripalmitoylglycerol $K_{O/W}$(oil) = 21.9 and Tristearoylglycerol $K_{O/W}$(oli) = 25.11.

When substituting the vegetable oil with an screening food simulant this would exhibit an octanol to water partition coefficient of $K_{O/W}$(sim). The following systematic approach is based on the screening food simulants ethanol and iso-octane, because they represent the two extremes from polarity point of view and substantial experience has been gained in the past published in the scientific literature.

$K_{O/W}$(ethanol) ~ 0 (estimated values are scattering around 0 depending on the estimation tool used.

[4] Feigenbaum, A.E., Riquet A.M., Scholler, D., Fatty food Simulants: Solvents to mimic the behaviour of fats in contact with packaging plastics.; ACS Symposium.

[5] Pattone et al; Solubility of fatty acids and other hydrophobic molecules in liquid trioleoylglycerol.; Journal of Lipid Research 1984, (25), p 189-

$K_{O/W}$(iso-octane) = 4.1 (estimated with EPI suite)

Because of the significantly lower molecular weight of screening food simulants compared to vegetable oil the octanol to water partition coefficient $K_{O/W}$(sim) is corrected by the ratio of the molecular weight between vegetable oil and the screening food simulant.

As generic vegetable oil the triglyceride with one stearic acid, one linoleic acid and one palmitic acid was considered showing a molecular weight of 861.44 g/mol and an estimated octanol to water partition coefficient of 22.74 (EPI suite).

Log Kow(version 1.68 estimate): 22.74

SMILES : O=C(CCCCCCC=CCCCCCCCC)OC(COC(=O)CCCCCCCCCCCCCCC)COC(=O)CCCCCCCCCCCCC
CCCC
CHEM :
MOL FOR: C55 H104 O6
MOL WT : 861.44

TYPE	NUM	LOGKOW FRAGMENT DESCRIPTION	COEFF	VALUE
Frag	3	-CH3 [aliphatic carbon]	0.5473	1.6419
Frag	46	-CH2- [aliphatic carbon]	0.4911	22.5906
Frag	1	-CH [aliphatic carbon]	0.3614	0.3614
Frag	2	=CH- or =C< [olefinc carbon]	0.3836	0.7672
Frag	3	-C(=O)O [ester, aliphatic attach]	-0.9505	-2.8515
Const		Equation Constant		0.2290
			Log Kow =	22.7386

Because of the significantly lower molecular weight of screening food simulants compared to vegetable oil the octanol to water partition coefficient $K_{O/W}$(sim) is corrected by the ratio of the molecular weight between vegetable oil and the screening food simulant.

This procedure is reasonable because the most estimation procedures for the octanol to water partition coefficient (e.g. EPI Suite from US EPA) are based on fragment increments. As a consequence bigger molecules with higher molecular weight like vegetable oil exhibit higher octanol to water partition coefficients compared to smaller molecules with lower molecular weight like ethanol or iso-octane, even if they are of similar polarity.

As an example iso-octane with molecular weight of 114 g/mol exhibits an octanol to water partition coefficient of $K_{O/W}$(C8) = 4.1. A saturated hydrocarbon with molecular weight of 254 g/mol exhibits an octanol to water partition coefficient of $K_{O/W}$(C18) = 9.2. A saturated hydrocarbon with molecular weight of 858 g/mol exhibits an octanol to water partition coefficient of $K_{O/W}$(C61) = 30.3.

Correcting the octanol to water partition coefficient for iso-octane by the ratio of the molecular weight between vegetable oil and the screening food simulant means calculating the octanol to water partition coefficient for a saturated hydrocarbon with the same molecular weight as vegetable oil.

When considering ethanol and iso-octane as screening food simulants to vegetable oil and correcting their octanol to water partition coefficients by the molecular weight ratio a polarity scale between 0 and 31 results:

$K_{O/W}^{corr}$(ethanol) = 861.44 / 46 * $K_{O/W}$(ethanol) ~ 0

$K_{O/W}^{corr}$(iso-octane) = 861.44 / 114 * $K_{O/W}$(iso-octane) = 31

The condition for the selection of the screening food simulant is now:

$$ratio^K = [K_{O/W}(sim) - K_{O/W}(mig)] / [K_{O/W}(oli) - K_{O/W}(mig)]$$

$$\mathbf{-1 < ratio^K < 1}$$

If the above $ratio^K$ is above -1 and below 1 the screening food simulant can be considered to be an screening for vegetable oil. If the above $ratio^K$ is between -1.5 and -1 respectively 1 and 1.5 the food simulant may be a reasonable screening for vegetable oil, but a certain risk of underestimation exists.

The condition "-1 < ratio^K < 1" is a strong requirement if migration comes close to equilibrium concentration. For -1.5 < ratio^K < -1 or 1 < ratio^K < 1.5 no underestimation is expected if:

a) the migrating amount of the substance will be significantly lower than its equilibrium concentration, or

b) the migrant is sparingly soluble in vegetable oil and as sparingly soluble in the screening food simulant.

This is the case for example because release of the substance from the polymer under testing conditions is diffusion controlled [6] or simply because the total amount of substance present in the material is significantly lower than the equilibrium concentration.

It should be noted that selection of an screening food simulant were the solubility for the migrant is less than in vegetable oil will have an impact on partitioning of the substance between polymer and simulant, which will lower the equilibrium concentration in the simulant.

The suitability of the screening food simulant ethanol 95% and iso-octane for some typical examples (monomers or additives) is considered below including reference to existing literature data.

1) Laurolactam $K_{O/W}$ = 3.6 (estimated with EPI Suite)
2) Tinuvin 1577 $K_{O/W}$ = 6.2 (estimated with EPI Suite)
2) Erucamide $K_{O/W}$ = 8.4 (estimated with EPI Suite)
3) Irganox 1076 $K_{O/W}$ = 13.4 (estimated with EPI Suite)
4) Irgaphos 168 $K_{O/W}$ = 18.1 (estimated with EPI Suite)

For testing the specific migration of laurolactam ethanol is a suitable screening food simulant for vegetable oil because laurolactam is better soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(laurolactem) is closer to $K_{O/W}$(ethanol) than to $K_{O/W}$(oil).

The corresponding calculation is:

ratio^K = [0 - 3.6] / [22.74 - 3.6] = -0.19

-1 < ratio^K < 1

For testing the specific migration of laurolactam iso-octane may be a suitable screening food simulant for vegetable oil because laurolactam is less soluble in iso-octane than in vegetable oil due to the fact that $K_{O/W}$(laurolactem) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).

The corresponding calculation is:

ratio^K = [31 - 3.6] / [22.74 - 3.6] | = 1.43

< 1 ratio^K < 1.5

For laurolactame a systematic migration study from Polyamide 12 in olive oil compared to is-octane exists which demonstrates that iso-octane is a suitable screening simulant compared to olive oil [7]. In this case the suitability of iso-octane vs. olive oil is based on the fact that laurolactam exhibits limited solubility in both simulants.

For testing the specific migration of Tinuvin 1577 ethanol is a suitable screening food simulant for vegetable oil because Tinuvin 1577 is better soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(Tinuvin 1577) is closer to $K_{O/W}$(ethanol) than to $K_{O/W}$(oil).

The corresponding calculation is:

ratio^K = [0 - 6.2] / [22.74 - 6.2] = -0.37

6 Publication Fraunhofer IVV - use of ethanol 50% as alternative food simulant for vegetable oil

7 N. H. STOFFERS, M. DEKKER, J. P. H. Linssen, A. STOERMER, R. FRANZ; "Alternative fatty food simulants and diffusion kinetics of nylon 12 food packaging"; Food Additives and Contaminants, Vol. 20, No. 10 (October 2003), pp. 949–959

$-1 < ratio^K < 1$

For testing the specific migration of Tinuvin 1577 iso-octane may be a suitable screening food simulant for vegetable oil because Tinuvin 1577 is less soluble in iso-octane than in vegetable oil due to the fact that $K_{O/W}$(Tinuvin 1577) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).

The corresponding calculation is:

$ratio^K = [31 - 6.2] / [22.74 - 6.2] = 1.5$

$1 < ratio^K = 1.5$

For Tinuvin 1577 no comparative systematic migration investigation exists. Instead for Tinuvin 234 (log $K_{O/W}$ = 7.67; calculated with EPI Suite) a systematic migration study from PET into ethanol 95%, iso-octane and Miglyol (a fractionated coconut oil) exists [8].

$ratio^K = [31 - 7.67] / [22.74 - 7.67] = 1.55$; $ratio^K > 1.5$

Migration results obtained with iso-octane lightly overestimate those obtained with Miglyol.

For testing the specific migration of Erucamide ethanol is a suitable screening food simulant because Erucamide is better soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(Erucamide) is closer to $K_{O/W}$(ethanol) than to $K_{O/W}$(oil).

The corresponding calculation is:

$ratio^K = [0 - 8.4] / [22.74 - 8.4] | = -0.59$

$-1 < ratio^K < 1$

For testing the specific migration of Erucamide iso-octane is not expected to be a suitable screening food simulant because Erucamide is less soluble in iso-octane than in vegetable oil due to the fact that $K_{O/W}$(Erucamide) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).

The corresponding calculation is:

$ratio^K = [31 - 8.4] / [22.74 - 8.4] = 1.58$

$ratio^K > 1.5$

For testing the specific migration of Irganox 1076 ethanol may be a suitable screening food simulant for vegetable oil because Irganox 1076 is less soluble in ethanol than in vegetable oil due to the fact that $K_{O/W}$(Irganox 1076) is closer to $K_{O/W}$(oil) than to $K_{O/W}$(ethanol).

The corresponding calculation is:

$ratio^K = [0 - 13.4] / [22.74 - 13.4] | = -1.43$

$-1.5 < ratio^K < -1$

For testing the specific migration of Irganox 1076 iso-octane is not expected to be a suitable screening food simulant for vegetable oil because Irganox 1076 is much less soluble in iso-octane than in vegetable oil due to the fact that $K_{O/W}$(Irganox 1076) is much closer to $K_{O/W}$(oil) than to $K_{O/W}$(iso-octane).

The corresponding calculation is:

$ratio^K = [31 - 13.4] / [22.74 - 13.4] | = 1.88$

$ratio^K > 1.5$

For Irganox 1076 a systematic migration study from LDPE in various foods and olive oil compared to ethanol 95% and iso-octane exists which demonstrates that ethanol 95% is still a suitable screening simulant compared to olive oil [9].

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[8] T. H. Begley, J. E. Biles, C. Cunningham, O. Piringer; " Migration of a UV stabilizer from polyethylene terephthalate (PET) into food simulants"; Food Additives and Contaminants, Vol. 21, No. 10 (October 2004), pp. 1007–1014

The systematic migration study also indicates that iso-octane might be a suitable screening food simulant as well. Due to strong swelling of the LDPE film by iso-octane and sufficient solubility for the amount of Irganox 1076 present in the LDPE film in iso-octane the migration into iso-octane is higher compared to olive oil.

For testing the specific migration of Irgafos 168 ethanol is not expected to be a suitable screening food simulant for vegetable oil because Irgafos 168 is much less soluble in ethanol than in vegetable oil due to the fact that $K_{o/w}$(Irgafos 168) is much closer to $K_{o/w}$(oil) than to $K_{o/w}$(ethanol).

The corresponding calculation is:

$ratio^K = [0 - 18.1] / [22.74 - 18.1] = -3.9$

$ratio^K < -1.5$

This consideration is substantiated by comparative experimental results were migration values obtained for Irgafos 168 with ethanol 95% are significantly lower compared to olive oil [10].

For testing the specific migration of Irgafos 168 iso-octane is not expected to be a suitable screening food simulant for vegetable oil because Irgafos 168 is much less soluble in iso-octane than in vegetable oil due to the fact that $K_{o/w}$(Irgafos 168) is much closer to $K_{o/w}$(oil) than to $K_{o/w}$(iso-octane).

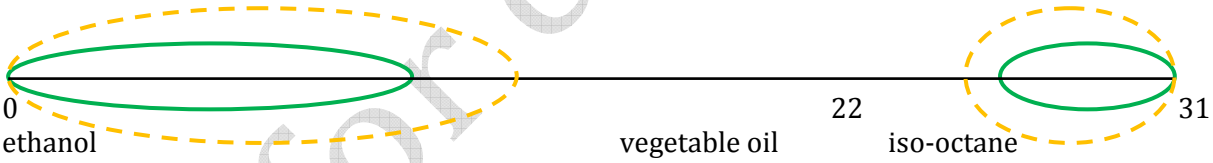
The corresponding calculation is:

$ratio^K = [31 - 18.1] / [22.74 - 18.1] = 2.78$

$ratio^K > 1.5$

However the same considerations made for Irganox 1076 may apply for Irgafos 168 but no published comparative experimental data exist.

Polarity scale based on octanol to water partition coefficients:



In most of the cases specific migration testing can be performed with the screening food simulants ethanol 95% for substances with an octanol to water partition coefficient log $K_{o/w}$ < 14 and with iso-octane for substances with an octanol to water partition coefficient log $K_{o/w}$ > 26. There is a gap for substances exhibiting a polarity similar to vegetable oil which is too far from that of ethanol 95% but not close enough to that of iso-octane. In these cases it is considered that iso-octane is the suitable screening food simulant, provided the residual content of the substance in the material is below 100 mg/dm^2 of plastic material investigated, because in this case solubility of the migrant in iso-octane is expected to be sufficiently high.

Finally solubility of substances in liquid media strongly depends on temperature. Because most of the literature data cited are at or below 60°C it is considered that the recommendations made

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[9] G. Beldì, S. Pastorelli, F. Franchini, C. Simoneau; "Time-and temperature-dependent migration studies of Irganox 1076 from plastics into foods and food simulants."; Food Additives and Contaminants, Vol. 29, No. 5, May 2012, 836–845

[10] N. H. STOFFERS, R. BRANDSCH, E. L. BRADLEY, I. COOPER, M. DEKKER, A. STORMER, R. FRANZ; " Feasibility study for the development of certified reference materials for specific migration testing. Part 2: Estimation of diffusion parameters and comparison of experimental and predicted data"; Food Additives and Contaminants, February 2005; 22(2): 173–184

above are valid up to a maximum test temperature of 70°C. Above 70°C the use of screening food simulants in many cases will induces physical changes of the materials investigated and from laboratory point of view their use above 70°C is dangerous due to their flammability. At higher temperatures (above 100°C) it is considered that food simulant E might be the more appropriate screening food simulant for vegetable oils.

The closer the polarity of the screening food simulant to the polarity of the plastic is, the higher is the risk of interaction between polymer and simulant, i.e. swelling of the polymer by uptake of screening food simulant. Depending on (a) the amount of simulant taken up by the polymer and (b) the extent of the plasticising effect related to the amount of simulant taken up, an increase of the migration rate compared to testing with vegetable oil can be expected.

The swelling effect may open the possibility for deviations from the conventional time and temperature testing conditions for vegetable oil when testing with screening food simulants. This option will be discussed below in the kinetic considerations section in more detail.

Screening food simulants selection for overall migration testing

Under the assumption, that the nature of the migrating substances contributing to the overall migration from the plastic is known, the considerations for screening simulant selection made for specific migration testing are applicable as well.

Regarding screening food simulants for overall migration testing and in the view of the fact that overall migration testing is an inertness test without toxicological relevance it is considered that the testing scheme based on use of ethanol 95% and iso-octane established with Directive 82/711/EEC including amendments is still feasible due to its long term use and existing experience.

The two solvents ethanol 95% and iso-octane span the polarity range of migrants from plastics encountered in practice. Substituting the overall migration test with vegetable oil requires testing with both solvents under consideration of the highest result for compliance evaluation. Regarding ethanol 95% its polarity is much higher compared to vegetable oil, reason for which solubility of non-polar migrants in ethanol 95% is expected to be lover compared to olive oil and as a consequence contribution of non-polar migrants (e.g. polyolefine oligomers or typical antioxidants) to overall migration may be underestimated. Regarding isooctane its polarity is lower compared to olive oil, reason for which solubility of polar migrants in isooctane is expected to be lover compared to vegetable oil and as a consequence contribution of polar migrants (e.g. polyamide oligomers or residual monomers) to overall migration for may be underestimated.

To minimize the risk of underestimation testing with both screening food simulants is requested and the highest migration result needs to be considered for compliance evaluation.

However the use of ethanol 95% and isooctane as screening food simulants was historically associated with time/temperature conditions which deviate from those for vegetable oil. This turned out to be a serious source of underestimation especially for testing temperatures above 40°C.

Possible deviations from the conventional time and temperature testing conditions for vegetable oil when testing with screening food simulants will be discussed below in the kinetic considerations section.

Kinetic considerations (time/temperature conditions)

Food contact materials most likely interact with foods to a given extent if they come into contact. Interaction means that materials may release components to the contact medium (migration) and/or may take up components from the contact medium or part of the contact medium itself. The migration rate of components from the material in most of the cases is diffusion controlled and may depend on the uptake rate and amount related to the contact medium.

In most food contact material applications the extent of polymer swelling by the packaged food is low, but there are also examples were the extent of swelling is very high like swelling of polyamide based sausage casings by water. Increase of migration rates due to swelling strongly depends on the nature of the polymer and the nature of the food or food simulant.

Due to the fact that screening food simulants most likely are volatile media they may interact with the plastic material during the migration test, i.e. take up simulant in the material to a given extent. Depending on polarity match between plastic and contact medium the uptake of simulant in the material is higher due to similar polarity and consequently the migration rates of components out of the material will increase due to swelling, i.e. the plasticising effect on the plastic material caused by the contact medium.

Because screening food simulants are volatile media they may not be used for migration testing at high temperatures. Instead time and temperature conditions for migration testing (both overall and specific) should be selected according to the formula given in Annex V under 2.1.4, i.e. lower testing temperature at longer testing time.

As a consequence one might tend to lower test time and/or test temperature to account for the swelling effect. However from today's knowledge point of view there are two main effects during swelling: (a) the amount of simulant taken up by the material and (b) the extent of the plasticising effect related to the amount of simulant taken up.

At the moment there is limited scientific background available to make a general recommendation regarding selection of time and temperature conditions for migration testing with respect to the possible combinations of plastic materials and screening food simulants.

Interaction of plastics with screening food simulants

Working with screening food simulants instead of vegetable oil most likely involves working with volatile media (solvents). Depending on the polarity of the plastic and the polarity of the solvent interaction between plastic and solvent may occur. The closer the polarity of plastic and solvent is the higher the extent of interaction, i.e. swelling will be. Due to swelling the diffusion controlled release of components from the plastic will increase. This effect is best described for PVC where the polymer specific constant (A_P-value) will change when adding a highly effective plasticiser to rigid PVC. An upper limit A_P-value of -1 is observed for rigid PVC and an upper limit A_P-value of 14.5 is observed for PVC plasticised with 30% Dioctylphthtalate. Do to that diffusion rates of component from plasticised PVC will be more than ten orders of magnitude faster compared to rigid PVC.

During the migration test of a plastic with a solvent under swelling conditions in fact two parallel processes will take place, i.e. the diffusion controlled uptake of the solvent under non-swollen conditions which will induce the diffusion controlled release of the component under swollen conditions. Because solvents are small molecules in most of the cases they will diffuse faster into the plastic than the bigger component out of the plastic. As a consequence overall a diffusion like release of the component from the plastic under swollen conditions will be observed. In terms of polymer specific constants the A_P-value for the non-swollen polymer and the A_P-value for the swollen polymer will differ significantly. The described behavior will be shown for the example polyamide 6 (PA6) when tested with aqueous food simulants, were water, acetic acid 3% and ethanol 10% are swelling the polymer during testing.

- Example: polyamide, PA6

Polyamides are plastic materials widely used for food contact applications. They are very polar compared to other plastics like polyolefines, polystyrene or polyethyleneterephthalate and hence prone to interact with foods. Prone to interaction with foods mean uptake of water from the food which on one hand will change the mechanic properties of the material and on the other hand will change its migration properties. From mechanical point of view water uptake will increase the flexibility of polyamides because water exhibits a plasticising effect.

Because of the complex migration behaviour of the PA6 film additional migration investigations with the aqueous food simulants water, 10% ethanol and 3% acetic acid were performed to establish a systematic view on the migration properties of polyamide in support of migration modelling. The work is focused on Caprolactam migration from a PA6 film but more general conclusions on the migration behaviour of PA6 can be drawn.

>> Water uptake of PA6

Due to its high polarity PA6 will take up significant amounts of water from the environment or contact medium. The water uptake of PA6 depends on the relative humidity of the surrounding air. Typical values are reported in the technical documentation of leading PA6 producers and are in the range given in Figure 1 [11].

(a) (b)

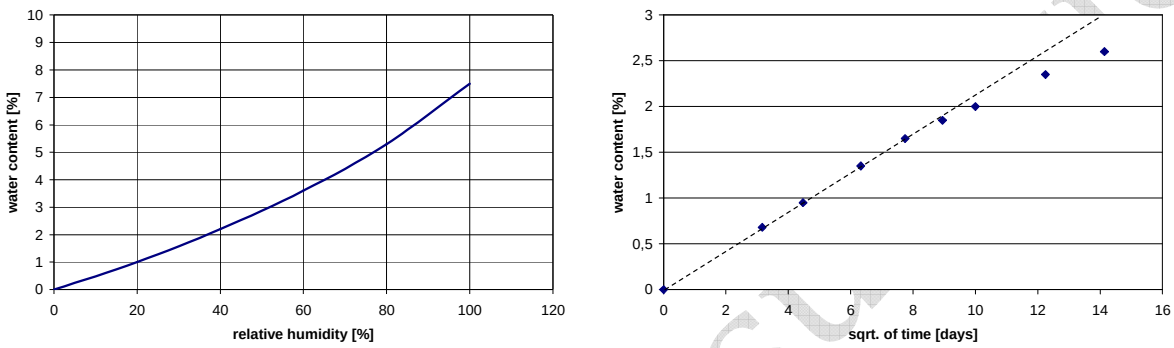


Figure 1 (a) Water content of PA6 at equilibrium in dependence of the relative humidity (b) Water uptake of PA6 (2 mm thickness) with time at 23°C and 50% rel. humidity

The water uptake rate of PA6 is strongly time and temperature dependent and much higher water uptake rates result if water comes in direct contact with PA6 as shown in Figure 2.

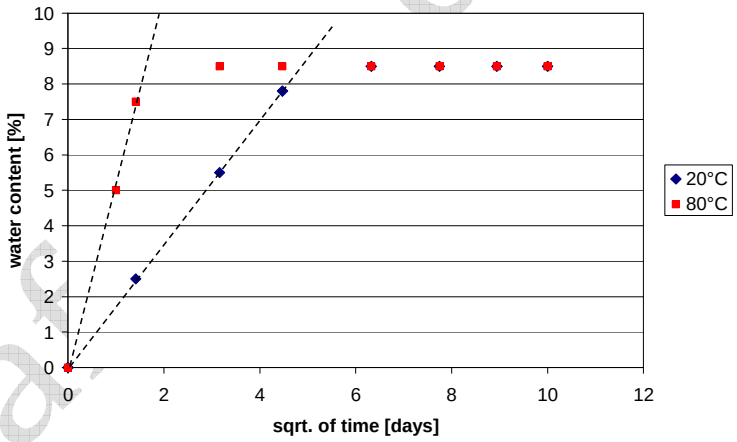


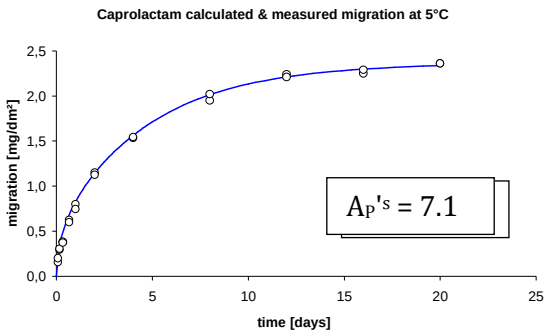
Figure 2 Water uptake of PA6 (2 mm thickness) with time at 20°C and 80°C in direct contact with water

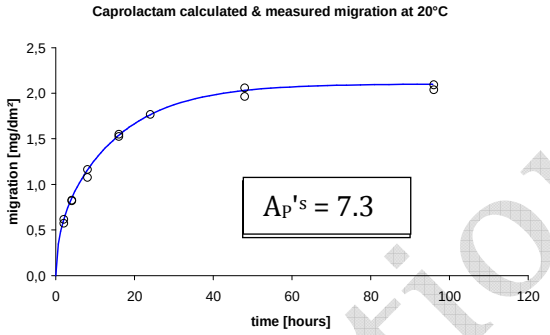
The linear dependence of the water content from the square root of time indicates that the water uptake is diffusion controlled. PA6 will interact with aqueous foods (liquid or semi-solid) similar to pure water when used as direct food contact material.

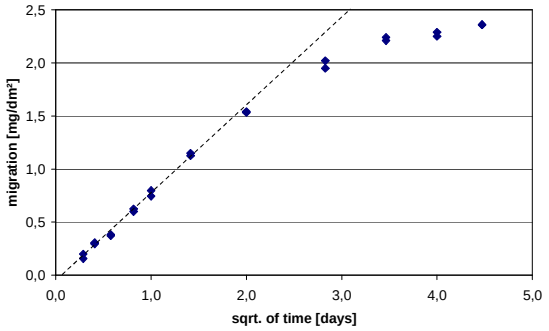
[11] BASF Schrift KTEM 0401 BD Ultramid /Capron www.basf.de/ultramid

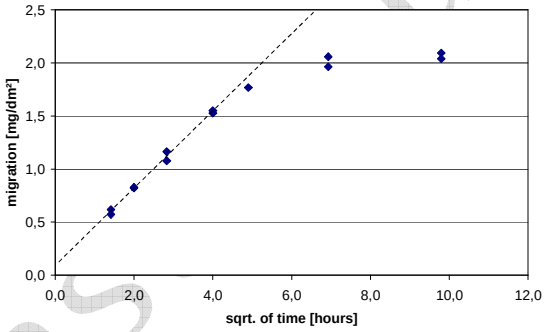
>> Migration kinetics of caprolactam from PA6 film into aqueous food simulants

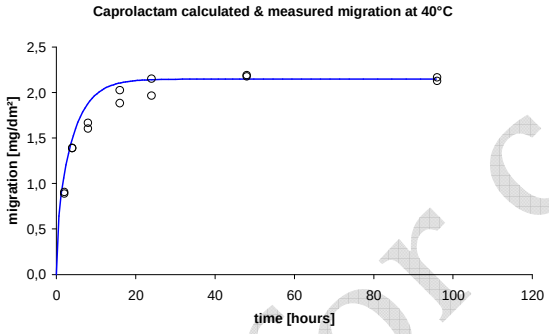
Migration into water at 5°C to 70°C:

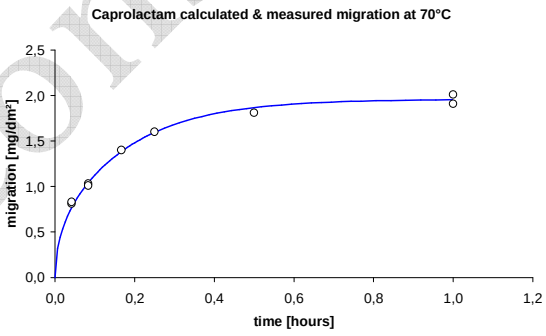


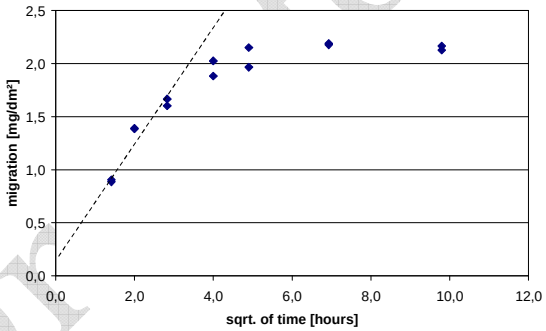


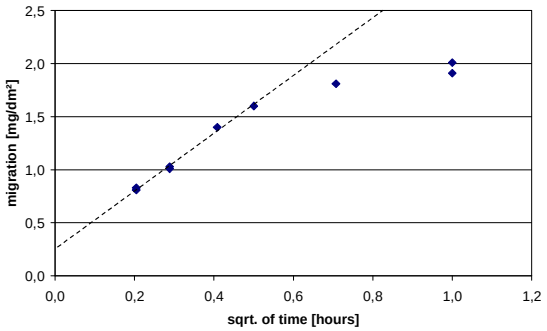






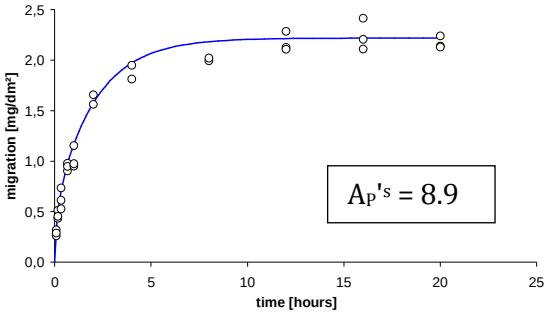


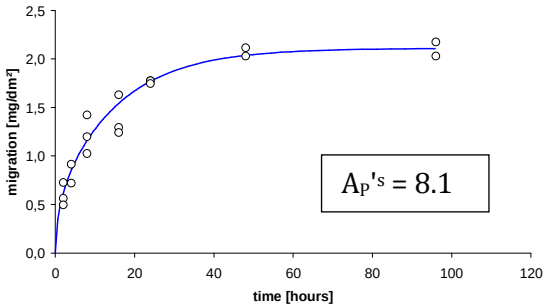




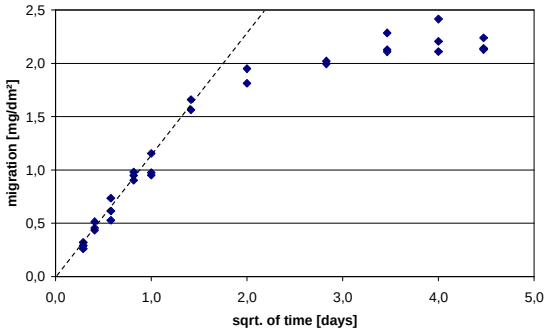
It can be observed that with increasing temperature the linear regression of the migration kinetic when related to the square root of time (up to approximate 60% of the equilibrium value) deviates more and more from the origin which is an indication that the rate of the two processes (a) diffusion controlled sorption of the simulant (manly water) in the non-swollen PA6 and (b) diffusion controlled release of caprolactam from the swollen PA6 are getting closer.

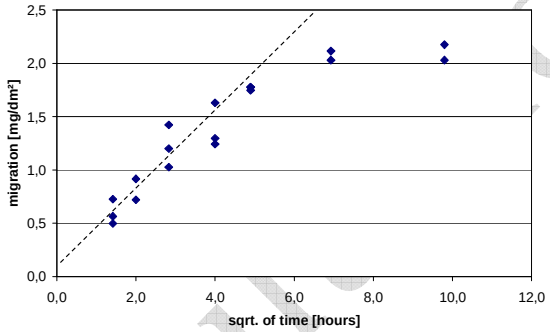
Migration into acetic acid 3% at 5°C and 20°C:





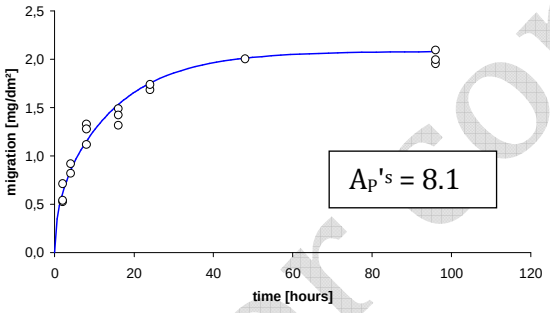
3013



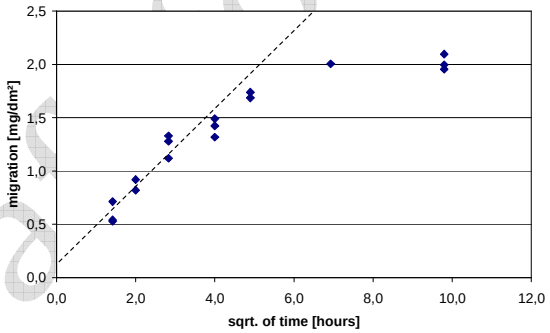


3014
3015

3016 *Migration into ethanol 10% at 20°C:*



3017



3018

3019 The polymer specific constant describing the diffusion properties of PA6 swollen by the aqueous
3020 simulants is in the range of $A_P's$ = 8.9 to 7.1 and decrease slightly with increasing temperature
3021 (from 5°C to 70°C).

3022 In the EU-Project "Certified Reference Materials" [12] the diffusion properties of polyamide 6
3023 (PA6) were investigated. Migration kinetics from the PA6 into iso-octane where performed for
3024 caprolactame. A summary of the results is given in the table below.

12 EU-Project "Certified Reference Materials"

Table 1 Summary of statistical results regarding initial concentration, specific migration, diffusion- and partition coefficients of caprolactam from PA6 into isooctane.

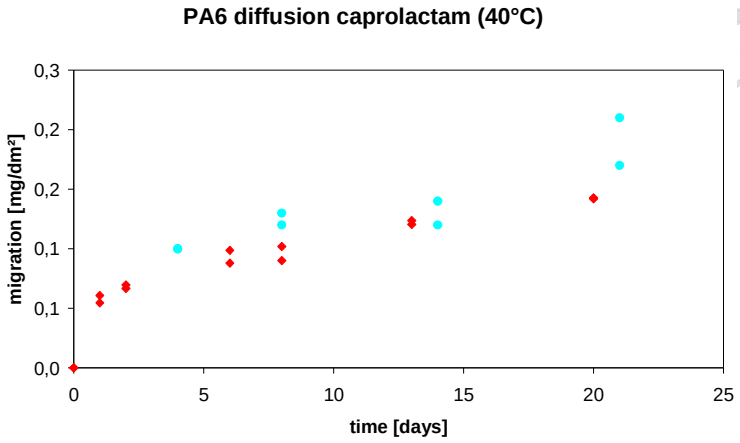
Summary of statistical results caprolactam

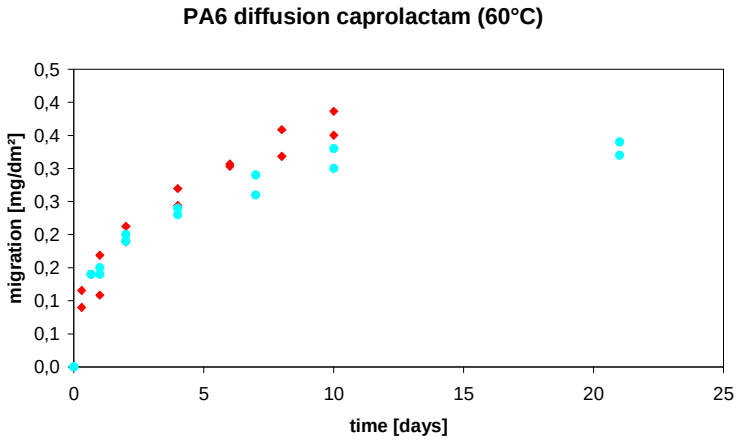
Certified property	$C_{P,0}$	SM	D_P			A_P			K_{PF}		
	mg/kg	mg/dm²	40°C	60°C	80°C	40°C	60°C	80°C	40°C	60°C	80°C
Number of accepted Data sets (Labs)	3(1)	4	3	4	4	3	4	4	3	4	4
Number of Individual Data (analysed samples)	9	15	3	4	4	3	4	4	3	4	4
All Data Sets compatible two by two? (Scheffe's multiple t-test)	yes	no									
Outlying lab means? (Dixon, Nalimov, Grubbs)	no	no									
Outlying variances (Cochran)	yes(2)	no									
Mean of lab means	**2116**	**0.99**	**8E-13**	**4E-12**	**5E-11**	**-1**	**-1**	**-1**	**844**	**325**	**149**
Within labs standard deviation	28	0.09									
Between labs standard deviation	30	0.20									
Minimum value			7E-13	2E-12	1E-11	-0.2	-0.6	0.01	1015	210	62
Maximum value			1E-12	8E-12	8E-11	-1	-2	-2	600	590	400
Lab variances homogeneous? (Bartlett)	o.o.r.	o.o.r.									
Standard deviation of distribution of lab means	24	0.12									
Lab means distribution (Kolmogorov-Smirnov-Lilliefors test) normally distributed?	i.d.	i.d.									
Half-width of 95% confidence interval	60	0.19									

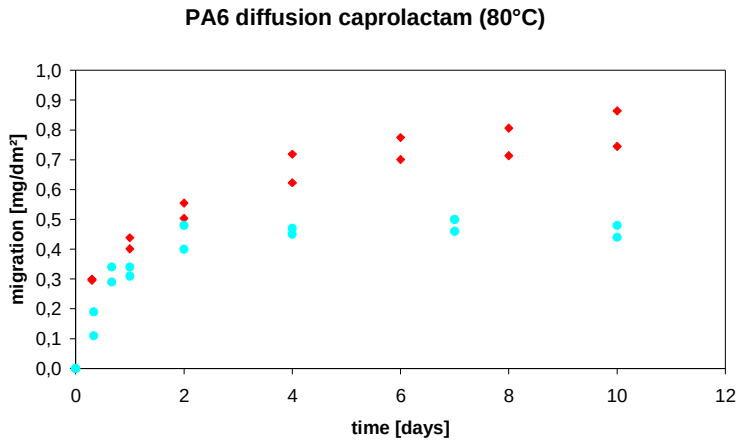
(1) Results of 3 labs were considered after elimination of one outlier.

(2) Cochran test: Lab 0 is outlier at 5% and 1% level. Lab 1 is outlier at 5% and 1% level.

Some representative migration kinetics for caprolactam from PA6 into iso-octane at different temperatures (40°C, 60°C and 80°C) are given in the figures below.







Comparing the A_P'-value at 40°C for non-swollen PA6 and PA6 swollen by water an increase of up to 9 units (from A_P' = -1 to $A_P'^s$ = 8.1) is observed. A similar swelling behavior can be expected for ethanol 95% and ethanol/water mixtures.

- Example Polyethylene terephthalate (PET)

The migration characteristics of the UV stabilizer Tinuvin 234 (2-(2H-benzotriazol-2-yl)-4,6-bis (1-methyl-1-phenylethyl)phenol) into food simulants has been measured from polyethylene terephthalate (PET) using HPLC with UV detection. Ethanol/water, isooctane and a fractionated coconut oil simulant (Miglyol) were used as food simulating solvents. The migration characteristics were measured at temperatures in the range of 40–70C [13].

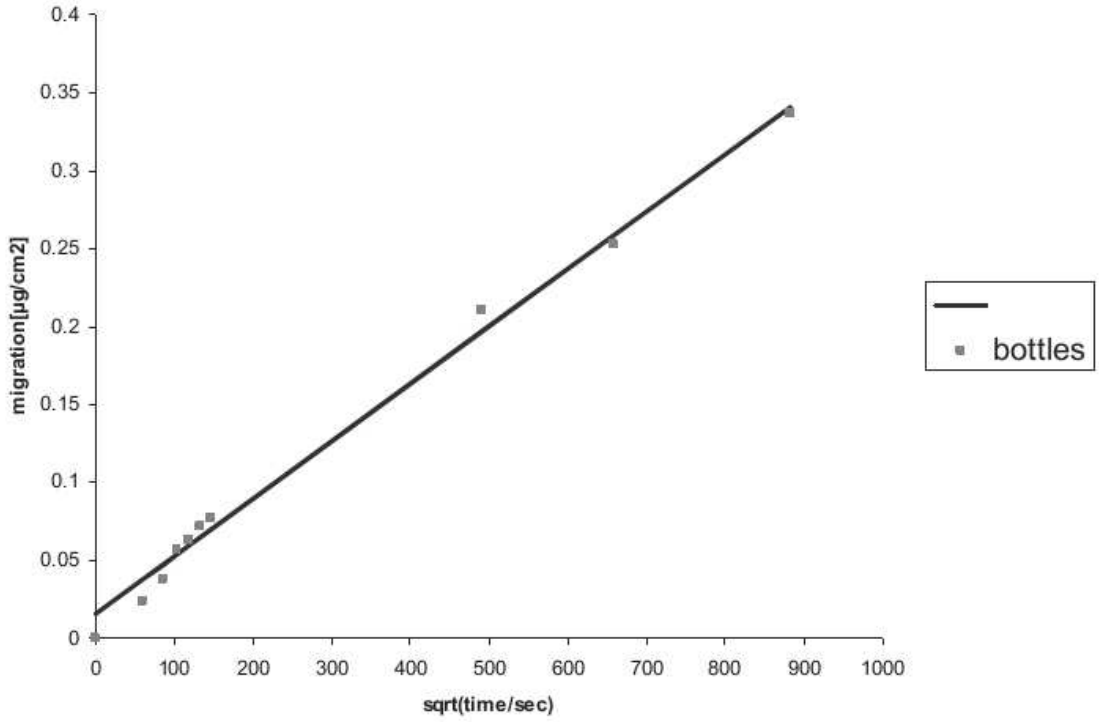


Figure 3. Migration of Tinuvin 234 into 95% ethanol at 60°C from PET bottles.

[13] Begley, T. H. et al.; Migration of UV stabilizer from polyethylene terephthalate (PET) into food simulants.

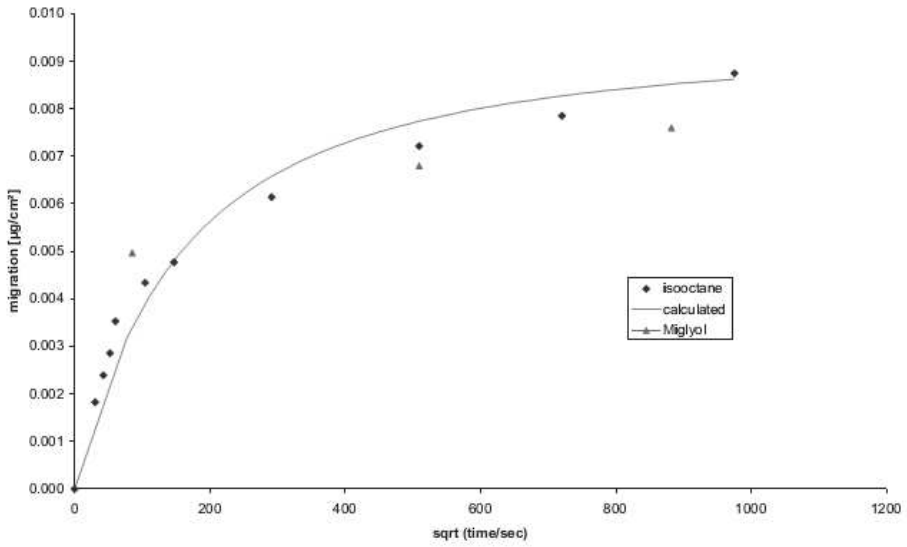


Figure 4. Migration of Tinuvin 234 into isooctane at 60°C from rectangular PET strips. Data represented by triangles (▲) are for migration experiments conducted from rectangular PET strips into Miglyol. The solid line is a calculated migration result according to equation (1).

The authors conclude that: "Using the short-time isooctane migration data, the magnitude of the diffusion of T234 in PET is determined to be ~3 x 10^{-16} cm^2/s. This diffusion value is significantly less than the 1 x 10^{-14} cm^2/s value determined at 60°C with ethanol, indicating that ethanol has an interaction with the PET causing diffusion to be faster. The apparent diffusion of T234 in PET in the presence of 95% ethanol is ~33 times faster than to isooctane."

The diffusion coefficients reported turn into the following polymer specific constants:

Temperature [°C]	**sample type**	**simulant**	**A_P'**	**A_P'**	**tau**
40	PET-bottle	etoh95%		-1.34	1577
50	PET-bottle	etoh95%		-1.14	1577
60	PET-bottle	etoh95%		1.25	1577
60	PET-bottle	iO	-2.26		1577
70	PET-bottle	iO	-2.21		1577
40	PET-bottle	iO	-5.65		1577
40	PET-bottle	etoh95%		-1.18	1577
40	PET-bottle	iO	-6.22		1577
mean			**-4.1**	**-0.6**	**1577**

It can be concluded that swelling of PET by ethanol 95% will increase the polymer specific value for PET by 3.5 units (from A_P' = -4.1 to $A_P's$ = -0.6)

- Example Polyolefines (LDPE, HDPE, PP)

For polyolefines in the swollen state no systematic kinetic data are available. However if for LDPE testing conditions 2 days at 20°C with iso-octane were considered to be equivalent to

testing conditions 10 days at 40°C with olive oil [14] it can be concluded that swelling of LDPE by iso-octane will increase the polymer specific value for LDPE by 5.5 units (from A_P' = 10 to $A_P'^s$ = 15.5). A similar swelling behavior for iso-octane can be expected for HDPE and PP, however the impact of their crystallinity on swelling is not known.

Conclusion

If for a plastic material both (a) the polymer specific constant in the non-swollen (A_P') state assumed to be applicable when in contact with vegetable oil and (b) the polymer specific constant in the swollen state ($A_P'^s$) applicable when in contact with the screening food simulant are available, time and temperature conditions to be used for testing with vegetable oil can be translated into time and temperature conditions to be used for testing with the screening food simulant.

Testing specific migration with screening food simulants

Selection of screening food simulants for specific migration testing should follow the criteria defined for ratio^K described above. As a first recommendation the same time/temperature conditions for specific migration testing with screening food simulants should be used as those to be used with vegetable oil.

If the screening food simulants to be used are ethanol 95% or iso-octane the following time/temperature conditions are considered to be suitable under consideration of polymer swelling:

Table 2 specific migration test conditions for screening food simulants

plastic	vegetable oil	ethanol 95%	iso-octane	Tenax
LDPE, LLDPE PP random PP rubbery	@ > 100°C			same t/T conditions as for vegetable oil
LDPE, LLDPE PP random PP rubbery	10d @ 60°C	10d @ 60°C	2d @ 40°C	
	10d @ 40°C	10d @ 40°C	2d @ 20°C	
	10d @20°C	10d @ 20°C	1d @ 20°	
HDPE	@ > 100°C			same t/T conditions as for vegetable oil
HDPE	10d @ 60°C	10d @ 60°C	1d @ 60°C	
	10d @ 40°C	10d @ 40°C	1d @ 40°C	
	10d @20°C	10d @ 20°C	1d @ 20°	

[14] EU Directive 97/48/EEC

PP isotactic	@ > 100°C			same t/T conditions as for vegetable oil
PP isotactic	10d @ 60°C	10d @ 60°C	1d @ 60°C	
	10d @ 40°C	10d @ 40°C	1d @ 40°C	
	10d @20°C	10d @ 20°C	1d @ 20°	
PET, PBT, PEN	@ > 100°C			same t/T conditions as for vegetable oil
PET, PBT, PEN	10d @ 60°C	1d @ 60°C	10d @ 60°C	
	10d @ 40°C	1d @ 40°C	10d @ 40°C	
	10d @20°C	1d @ 20°C	10d @ 20°	
PS	@ > 100°C			same t/T conditions as for vegetable oil
PS	10d @ 60°C	1d @ 60°C	1d @ 60°C	
	10d @ 40°C	1d @ 40°C	1d @ 40°C	
	10d @20°C	10d @ 20°C	1d @ 20°	
SBS	10d @ 60°C	10d @ 60°C	1d @ 60°C	
	10d @ 40°C	10d @ 40°C	1d @ 40°C	
	10d @20°C	10d @ 20°C	1d @ 20°	
PA 6, PA 6.6	@ > 100°C			same t/T conditions as for vegetable oil
PA 6, PA 6.6	10d @ 60°C	1d @ 60°C	10d @ 60°C	
	10d @ 40°C	1d @ 40°C	10d @ 40°C	
	10d @20°C	1d @ 20°C	10d @ 20°	
PA 12	@ > 100°C			same t/T conditions as for vegetable

				oil
PA 12	10d @ 60°C	1d @ 60°C	10d @ 60°C	
	10d @ 40°C	1d @ 40°C	10d @ 40°C	
	10d @20°C	1d @ 20°C	10d @ 20°	
PVC, rigid	@ > 100°C			same t/T conditions as for vegetable oil
PVC, rigid	10d @ 60°C	1d @ 60°C	10d @ 60°C	
	10d @ 40°C	1d @ 40°C	10d @ 40°C	
	10d @20°C	1d @ 20°C	10d @ 20°	

Time-temperature conditions recommended for screening food simulants deviate from the conventional ones used for vegetable oil due to swelling.

Testing overall migration with screening food simulants

Based on the above conclusion the following time/temperature conditions for overall migration testing with screening food simulants are recommended:

Table 3 overall migration test conditions for screening food simulants

plastic	**vegetable oil**	**ethanol 95%**	**iso-octane**
LDPE, LLDPE PP random PP rubbery	OM2	OM2	2d @ 20°C 1d @ 40°C
	OM1	2d@40°C	1d@20°C
HDPE	OM2	2d@60°C	1d@40°C
	OM1	2d@40°C	1d@20°C
PP isotactic	OM2	2d@60°C	1d@40°C
	OM1	2d@40°C	1d@20°C
PET, PBT, PEN	OM2	1d@40°C 1d @ 50°C	OM2
	OM1	1d@20°C	2d@40°C
PS	OM2	1d@40°C 1d @ 40°C	1d@40°C

	OM1	1d@20°C	1d@20°C
SBS	OM2	2d@60°C	2d@20°C 1d @ 40°C
	OM1	2d@40°C	1d@20°C
PA 6, PA 6.6	OM2	1d@40°C	2d@60°C
	OM1	1d@20°C	2d@40°C
PA 12	OM2	1d@40°C	2d@60°C
	OM1	1d@20°C	2d@40°C
PVC, rigid	OM2	1d@40°C	2d@60°C 1d @ 40°C
	OM1	1d@20°C	2d@40°C

Time / temperature conditions recommended for screening food simulants deviate from the conventional ones used for vegetable oil due to swelling.

Annex 7 Test method for overall migration into vegetable oil

Content

This annex describes the test method for overall migration into vegetable oil. It consists of four main sections:

- Test method for overall migration into vegetable oil in the temperature range of 20-100°C
- Test method for overall migration into vegetable oil in the temperature range of 5-20°C
- Test method for overall migration into vegetable oil in the temperature range of 100-175°C
- Test method for overall migration into vegetable oil in case of incomplete extraction of vegetable oil in the temperature range of 5-175°C

Testing at low temperatures

Testing with oil at 5°C may lead to technical problems if the oil partially solidifies.

Sunflower oil, which is free of components which solidify at the temperature of test (i.e. a "de-waxed" oil), may be used.

However, with olive oil and sunflower oil the test is usually without this problem at 10°C. If the overall migration does not exceed the limit when tested at 10°C this indicates that it would not have exceeded the limit at 5°C.

Testing by total immersion or in a cell or in a pouch is practicable at low temperatures, although if a cell or pouch is used for the vegetable oil where a visual check on solidification is difficult, a de-waxed vegetable oil shall be used.

The method of test for the determination of overall migration at low temperatures in the range of 5-20°C is given in section 7.2.

Testing at high temperature

In practice, severe difficulties have been found in obtaining consistent and comparable results in inter-laboratory trials with the test conditions for simulating contact at temperatures of use in excess of 121°C. The main source of inconsistency appears to be due to variation in the time required to achieve the test contact temperature with olive oil and other fatty food simulants. Various options such as heating of sample tubes in electrically heated cells, etc. are under investigation as possible solutions to the problem. These have been incorporated into the methods described in section 7.3.

7.1 Test method for overall migration into vegetable oil in the temperature range of 20-100°C

7.1.1 Scope

This Annex describes a test method for the determination of the overall migration into vegetable oil from plastics materials and articles. The test method is applicable for temperatures above 20°C and up to, but not including, 100°C. The method describes four different ways to perform the migration, i.e. immersion, filling, pouch forming and filling and cell for one-sided contact.

The immersion method is most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers, from which test pieces of a suitable size can be cut.

The cell method is most suitable for plastics in the form of films and sheets, but is particularly applicable to those materials consisting of more than one layer or surfaces that differ in their migration characteristics and that have to be tested with food simulant in contact with the surface which is intended to come into contact with foodstuffs.

The pouch method is most suitable for plastics in the form of films and sheets, which are sealable by heat or pressure, but it is particularly applicable to those materials consisting of

more than one layer, which has to be tested with the food simulant in contact only with the surface which is intended to come into contact with the foodstuffs.

The filling method is most suitable for plastics in the form of containers and articles that can be filled. Testing samples by this method enables testing of non-homogenous articles provided they are not too large.

NOTE – This test method has been written for use with rectified olive oil. The test method can also be used with appropriate modifications with other vegetable oils as defined in Regulation (EU) No 10/2011. These other vegetable oils will produce different chromatograms for the simulant methyl esters to those of the methyl esters of rectified olive oil. Select suitable chromatogram peaks of the methyl esters of the other vegetable oils for the quantitative determination of the simulant extracted from the test specimens.

The test method described is applicable to most types of plastics, although there are some plastics for which it is known not to be applicable.

7.1.2 **Principe**

The overall migration from a sample of the plastics is determined as the loss in mass per unit of surface area intended to come into contact with foodstuffs.

The selection of the test conditions will be determined by the conditions of use as described in Regulation (EU) No 10/2011.

Test specimens of known mass are immersed in, filled with or put in contact to rectified olive oil at temperatures above 20°C and below 100°C for a relevant contact time during which substances may migrate into the olive oil. Next, the oil is removed. The test specimen, are blotted to remove oil adhering to the surface, and reweighed. Pouches are cut open before blotting.

The test specimens will usually retain absorbed oil that is extracted and determined quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is carried out by hydrolysing the oil with potassium hydroxide followed by methylation with a boron trifluoride/methanol complex. An internal standard, triheptadecanoin, is added prior to the extraction of the absorbed oil from the test specimens. This ensures that any active or extractable component of the plastic that reacts with the extracted oil also reacts with the internal standard. The internal standard is also subjected to hydrolysis and methylation, providing compensation for any inefficiency in the hydrolysis and methylation processes.

Migration into the oil is calculated by subtracting the mass of oil retained by the test specimen from the mass of the test specimen after removal from the oil, then subtracting this mass from the initial mass of the test specimen.

The total loss in mass is expressed in milligrams per square decimetre of surface area of the test specimen.

To allow for inaccuracies which may arise during the procedure and which may be difficult to detect, due for example to contamination or loss of oil during the sample handling stages, four determinations are carried out on the sample allowing for one failing result.

This method includes variations that are applicable to certain plastics.

NOTE – Before starting a migration exercise, the test sample should be examined for the presence of substances interfering in the determination of the amount of extracted oil (see Annex 0). If an unacceptable amount of interference is present then suitability of one of the other vegetable oils should be examined. If an interference is present which would interfere with the triheptadecanain internal standard an alternative internal standard should be used.

7.1.3 **Reagents**

NOTE – All reagents should be of recognized analytical quality, unless otherwise specified.

NOTE – Specifications for solid reagents, used as such in discrete quantities, may not address suitability for use in methods of analysis in this Annex. Solid reagents might not be homogeneous with respect to contaminants not addressed by specifications, therefore it may be necessary to demonstrate that such reagents are suitable for use.

7.1.3.1 Rectified olive oil (see section 4.2.3)

Some substances which may migrate from plastics are capable of interfering with the gas chromatographic method for the determination of olive oil, e.g. glyceryl oleates. When testing articles containing these substances they may be tested with other vegetable oils, such as sunflower oil, corn oil, coconut oil, palm kernel oil.

NOTE: The refining or rectifying process will remove interfering substances and unsaponifiable matter and free fatty acids. In general, oil containing less than 1% of unsaponifiable matter and/or free fatty acids will be suitable for all migration experiments provided the oil is stored in the dark at refrigerated or frozen conditions until it is used.

7.1.3.2 Extraction solvent

For non-polar plastics such as polyethylene and polypropylene:

- Pentane (98 %; boiling point 36°C)

For polar plastics, such as polyamide and polyacetal:

- 95/5 v/v azeotropic mixture of pentane (98 %) and ethanol (99 %).

NOTE 1 – Pentane is a very volatile and highly flammable solvent. Care should therefore be taken when handling this solvent to prevent contact with ignition sources. Ethanol is also a flammable solvent. It is not recommended that extractions with either pentane or pentane/ethanol mixture be left unattended, particularly overnight.

NOTE 2 – Due to the low boiling points of the solvents, cooled condenser water may be required to prevent undue loss of solvent from the condenser.

NOTE 3 – Some solvents can contain non-volatile substances which, after hydrolysis and methylation processes, produce gas chromatography peaks with retention times similar to the retention times oil methyl esters and methyl heptadecanoate from the internal standard. Solvents found to contain such substances should be redistilled before use.

7.1.3.3 Internal standard

Triheptadecanoin (glyceryl trimargarate; CAS No, 2438-40-6) of a quality such that the products from hydrolysis and methylation processes do not contain substances giving detectable gas chromatography peaks with similar retention times to the oil methyl ester peaks. Prepare a solution containing 2.0 mg/ml in cyclohexane.

Other migrating substances may give rise to peaks in the gas chromatogram which interfere with the internal standard peak. Alternative internal standards such as hydrocinnamic acid, ethyl ester or trinonadecanoin may be used in such cases.

7.1.3.4 Potassium hydroxide solution

Prepare a solution of 11.0 g/L in methanol

7.1.3.5 Boron trifluoride/methanol complex

Prepare a solution of approximately 150 g/L BF_3 in methanol.

7.1.3.6 n-Heptane

7.1.3.7 Sodium sulphate

7.1.3.7.1 Sodium sulphate, anhydrous, Na_2SO_4

7.1.3.7.2 Sodium sulphate, saturated solution

7.1.3.8 Diethyl ether

7.1.4 Apparatus

7.1.4.1 Cutting slab

Clean smooth glass, metal or plastics slab of sufficient area to prepare test specimens, 250 mm x 250 mm is suitable.

7.1.4.2 Tweezers, stainless steel, blunt nosed.

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

7.1.4.4 Metal templates

Size 100 ± 0.2 mm x 10± 0.2 mm (square).

7.1.4.5 Rule or template

Size 25 ± 1 mm wide.

7.1.4.6 Rule, graduated in millimetres

with an accuracy of 0.1 mm.

7.1.4.7 Analytical balance

capable of determining a change in mass of 0.1 mg.

7.1.4.8 Test specimen supports

Constructed of stainless steel with cross arms attached by welding or silver soldering. Stainless steel X4 CrNI 18 10 according to EN 10088 or of composition, chromium 17%, nickel 9%, carbon 0.04%, is suitable. Before initial use thoroughly clean the steel supports. The use of a degreasing solvent and then diluted nitric acid has been found to be suitable.

NOTE – The method has been written for the supports shown in Figure 3 that have been found to be suitable for holding thin film and sheet test pieces. However other supports can be used providing they are capable of holding and keeping the test pieces apart and at the same time ensuring complete contact with the food simulant. For rigid samples, supports with a single cross arm can be used.

7.1.4.9 Gauze

Pieces of fine stainless steel gauze, with a mesh size of 1 mm have been found to be suitable, approximately 25 mm x 100 mm for insertion between the test pieces on the supports. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with diluted nitric acid.

7.1.4.10 Conditioning containers

For conditioning test specimens at 50% ± 5% relative humidity and 80% ± 5% relative humidity at 20°C ± 5°C.

NOTE – For 50% relative humidity, 43% w/v sulphuric acid solution in water is suitable and for 80% relative humidity, 27% w/v sulphuric acid solution is suitable. The solutions should be

freshly prepared by adding a weighed amount of acid to a suitable volume of water, cooling to room temperature and making up to the required volume. It is recommended that relative humidity and temperature be maintained during the conditioning period. Therefore the containers should be placed in a thermostatically controlled room or oven, at a temperature of approximately 20°C, the set temperature should not vary by more than ± 1°C.

7.1.4.11 Glass tubes

With ground neck and stoppers, for retaining the oil 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.

Note – the samples are tested at a fixed ratio of surface area of test specimen to food simulant volume. In order to ensure that all parts of the test specimen are in contact with the food simulant, glass tubes of the appropriate diameter are used. Minor adjustments to the level of the simulant in the tubes may be made by adding glass rods or glass beads sufficient to ensure complete immersion of all of the surfaces of the test specimen. Again the dimensions of suitable glass rods and glass beads are specified in the individual methods.

7.1.4.12 Thermostatically controlled oven or incubator

capable of maintaining the set temperature within the tolerances specified in Table 5.

NOTE: An oven with forced air circulation (e.g. GC oven) has been found very suitable and capable to maintain the required temperature very accurate. In addition heat transfer is increased.

7.1.4.13 Filter paper, lint-free

7.1.4.14 Anti-bumping beads

7.1.4.15 Soxhlet type extractors

capable of holding test specimens on the supports, with 250 ml or 500 ml round bottom flasks 10 fit.

NOTE – Alternative extractors capable of satisfactorily extracting absorbed olive oil from the test specimens can be used.

7.1.4.16 Water bath

capable of holding the flasks of soxhlet type extractors (7.1.4.15)

7.1.4.17 Rotary evaporator or distillation apparatus

for evaporation and collection of the extraction solvent.

NOTE Artificially cooled water can be necessary for efficient condensation of a low boiling point solvent.

7.1.4.18 Steam bath or water bath.

7.1.4.19 Flasks

50 ml, long neck with condensers to fit, for methyl ester preparations.

7.1.4.20 Measuring cylinders

complying with the minimum requirements of ISO 4788, 500 ml, 250 ml, 100 ml, 25 ml, and 10 ml. A 10 ml graduated syringe may be used in place of the 10 ml measuring cylinder.

7.1.4.21 Pipettes

complying with the minimum requirements of ISO 648, 5 ml ami 10 ml.

7.1.4.22 Glass beads or rods

Beads: 2-3 mm in diameter; rods: 2-3 mm in diameter and approximately 100 mm long (see note 7.1.4.11).

7.1.4.23 Gas chromatograph

equipped with flame ionisation detector and an appropriate column. When using a polar column, the major peaks of olive oil, such as C16:0, methyl hexadecanoate (methyl palmitate), C16:1, methyl 9-hexadecenoate (methyl palmitoate), C18:0, methyl octadecanoate (methyl stearate), C18:1, methyl 9- octadecenoate (methyl oleate), C18:2, methyl 9,12-octadecadienoate (methyl linoleate) and the internaI standard C17:0, methyl heptadecanoate (methyl margarate) shall demonstrate baseline separation. Optionally, a non-polar column can be used which shall give baseline separation of the methyl esters with 16 and 18 carbon numbers and the internal standard with 17 carbon number.

NOTE The following columns have been found to be suitable:

- Column 1, polar column, WCOT fused silica column, length 50 m, internal diameter 0.25 mm, coated with a 0.21 µm film of cyanopropyl silicone; Column 1 is a column with a polar stationary phase that allows separation of the individual methyl esters of fatty acids according to their carbon number as well as their number of double bonds in the chain, e.g. the methyl esters of stearic acid is separated from the methyl esters of oleic acid and this is separated from the methyl esters of linoleic acid.

- Column 2, non-polar column, 100% dimethyl polysiloxane, length 25 m, internal diameter 0.32 mm, with a 1 µm film thickness; Column 2 is a column with a non-polar coating which allows only separation of the carbon number, e.g. no separation is obtained between the methyl esters of oleic acid and the methyl esters of stearic acid.

Both types of columns have their own specific advantages and disadvantages. A gas chromatogram obtained with column 1 will reveal more information on the distribution of fatty acid in the olive oil extracted from the test specimen than with column 2. To determine the total area of the fatty acids using column 1, the area of at least 5 peaks may be measured and summed. With column 2 only 2 peaks have to be measured. On the other hand the determination will be more sensitive to interferences when using column 2. In the case where interferences occur on one of the minor peaks, when using column 1, it is possible to exclude that peak and to adapt the calibration graph for the excluded peak. It is even possible to measure only the major peak of oleic acid to quantify the total amount of oil, provided the calibration graph is constructed in the same way.

NOTE A polar column is the preferred one

7.1.4.24 Glass tubes

with ground glass necks and stoppers, of a volume of approximately 10 ml, for storing the heptane layer if necessary.

7.1.4.25 Vacuum oven or vacuum desiccator

capable of maintaining a temperature of 60°C ± 2°C. The vacuum oven or vacuum desiccator shall be equipped with or connected to a vacuum pump capable of achieving a vacuum of 1,3 kPa or less. The vacuum pump shall be provided with a time controller to switch on the vacuum pump every hour for 15 min.

NOTE If a vacuum oven is not available, a vacuum desiccator placed in an oven at 60°C can be used.

7.1.4.26 Desiccator

containing self-indicating silica gel or anhydrous calcium chloride,

7.1.4.27 Balance

capable of determining a change of mass of 10 mg.

7.1.4.28 Disposable plastic syringes

with luer fitting. 1 ml or 10 ml size.

7.1.4.29 Wide gauge luer needles

80 mm x 1.2 mm.

7.1.4.30 Cell

Migration cell as shown in Figure 5-Figure **10**

7.1.4.31 Pouch holder

Figure 11 shows a pouch holder that is suitable. It is constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

7.1.4.32 Heat or pressure sealing device

For use in forming pouches

7.1.4.33 Chromatography tank

Or any other airtight container for test sample storage.

7.1.4.34 Glass rods or metal gauze

For use as spacers between test pieces during solvent extraction.

7.1.4.35 Lint-free cloth

7.1.5 Preparation of test specimens

7.1.5.1 General

The sample taken for testing is the final article, in its ready-for-use state. In some cases this may be impracticable and test specimens can be taken from the material, article or, where appropriate, test specimens representative of this material or article can be used.

An example is where an article is filled with food at the time it is formed. In this case the test may be carried out on a test article prepared especially for testing purposes. This article shall be as representative as possible of the article in actual use.

A further example is where the sample to be tested is of inhomogeneous construction and is too large to be tested by filling and no flat surfaces can be cut from the sample for testing in a cell. In this case the test may be carried out on a test article prepared especially for testing purposes. This article shall as representative as possible of the article in actual use.

Where samples are taken at random from a production batch this shall be indicated when reporting the result. The samples shall be representative of normal production material. Similarly if the sample was not a random sample, and it was selected according to some other parameter, e.g. thickness variation, this shall also be reported.

Samples may be inhomogeneous, e.g. varying in crystallinity or in molecular orientation, or of irregular shape or thickness, e.g. sections cut from bottles, trays, work surfaces, cutlery etc., or so small that several samples are required to constitute a test specimen. Replicate samples as similar as possible to each other and proportionally representing the sample article shall be tested and the sampling details shall be included in the final report.

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any

surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. Minimise handling of the samples and, where necessary, wear cotton gloves.

If articles are accompanied with an instruction that they should be cleaned before use then this instruction should be followed before testing. If, however, the instruction prescribes rubbing of the article with e.g. an oil, then this instruction should not be followed as the oil will contribute to the overall migration.

To ensure that test pieces are well separated and that their surfaces are freely exposed to oil during the period of the test, for thin films insert a piece of fine stainless steel gauze (7.1.4.9) between the test pieces or for thick samples not placed on the supports, insert glass rods between the test pieces after immersion in the oil. Where test specimen supports are used, label the supports with a tag bearing the test specimen identification.

7.1.5.1.1 Surface to volume ratio

Where the surface to volume ratio to be used in contact with food is known this is used in the migration testing. An example of this is where a bottle or other container is intended to contain a specified volume of contents even if this does not completely fill the article. In this case the article is tested with the specified volume of simulant.

Where the surface to volume ratio to be used in contact with food is ot known conventional conditions are used, in the following sections.

7.1.5.1.1.1 Single surface versus double surface testing by total immersion

For verification of compliance section 4.4.3 of this document needs to be taken into account. Overall migration tests shall be performed in such a way that only those parts of the sample intended to come into contact with foodstuffs in actual use will be in contact with the foodstuff or simulant. In cases where the overall migration limit is exceeded when testing by total immersion, the test shall be repeated using a method applying single sided contact.

However, it is permissible to demonstrate compliance with an overall migration limit by the use of a more severe test (screening). In the total immersion test, both the surface which is intended to come into contact with the foodstuff and the outside surface are in contact with the food simulant. No allowance is made for this in the calculation of migration per unit of surface area. Although the total surface exposed is 2 dm^2, only 1 dm^2, i.e. the food contact surface, is taken into account in the calculation. It is therefore a more severe test than testing in a pouch or in a cell or by filling. However, if it is possible to demonstrate experimentally that the value obtained in a total immersion test is double that obtained in a single surface test, the value obtained in the total immersion test shall be divided by the total surface area exposed.

Test specimens with cut edges tend to give higher results than those without. In use, the plastics material or article would not normally have cut edges in contact with the foodstuff. The process of cutting may have an irreversible effect on the morphology of the edges of the sample. As a result, the obtained overall migration value is not a true reflection of the real migration under actual conditions of use. Therefore the number of cut edges shall be limited, where possible, and in the case that the overall migration limit is exceeded the test shall be repeated using a method applying single sided contact.

If the area of the cut edges of the test specimen exceeds 10% of the measured area of the sample then this area has to be included in the calculation of the surface area used in the calculation of overall migration.

Testing samples with the test specimens prepared by cutting sections from the plastic and totally immersing in the food simulant, is a more severe test.

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.

7.1.5.1.1.2 Single surface testing using a cell

Where single surface testing is the preferred procedure, particularly important for multi-layer articles, this may be carried out in a cell. For samples that may be obtained in flat form, e.g. film or sheet, testing in the cell has the advantage of readily reproducible sample geometry.

The surface to volume ratio in the type A cell as used in this method, is conventionally 2.5 dm^2 of food contact area to 125 ml of food simulant. Inter-laboratory trials carried out by experienced laboratories have shown that consistent overall migration results can be obtained using cell type A. Comparative studies carried out on the performance of cells type A, B, C, D, E and F revealed that these cells gave similar results. Therefore the cells referred to in Figure 5-**Figure 10** are considered equivalent.

7.1.5.1.1.3 Single surface testing by pouch

For flat articles which have sufficient seal strength to form durable pouches, single surface testing in a pouch may be preferred as this does not require specialized apparatus and allows more efficient use of oven space. Inter-laboratory collaborative testing studies using pouches of precisely specified dimensions have shown that variations in pouch geometry (particularly varying areas outside the seals) can lead to significant variability in the final result.

The surface to volume ratio in the pouch is conventionally 2 dm^2 of food contact area to 100 ml of food simulant.

NOTE Generally pouches are filled with preheated simulant. However for test temperatures above 40°C the pouches may be filled with food simulant at ambient temperature and then the test specimens preheated in a microwave oven to reach the test temperature. A procedure that has been found to be suitable is to insert into the simulant of one of the test specimens a fibre optic probe or to check the temperature after heating by a thermometer. The filled pouches are placed in a microwave oven and heated until the simulant has attained the test temperature. The test specimens are removed to a thermostatically controlled oven or incubator that is preheated to the test temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss. The pouches are left for the selected test period.

7.1.5.1.1.4 Single surface testing using a reverse pouch

As an alternative to using a pouch, a reverse pouch may be used. In this case the surface intended to come into contact with the foodstuff is the outer surface and the pouch is exposed to the food simulant by total immersion.

The use of a reverse pouch offers advantages over the pouch. Since pouches are filled with food simulant, the sealed edges have to be capable of bearing the mass of that simulant; if they are not the seals give way and the pouches are prone to leakage. With the reverse pouch the seals do not have to withstand the pressure of the food simulant and consequently are less likely to leak and the sealed area can be reduced. The use of a reverse pouch permits a more accurate determination of the area exposed to food simulant. However, it is possible that food simulant may leak into the reverse pouch thus increasing the area exposed to food simulant. A way of checking if leaks have occurred, is to seal into the reverse pouch a piece of filter paper which is of similar dimensions to the pouch. If the pouch leaks the paper will absorb the food simulant and this will be visible. This method may not be applicable for overall migration into fatty food simulants, as the mass of the inserted paper may change during storage due to loss of water. Any pouch that leaks shall be discarded and the test shall be repeated.

Where the surface to volume ratio to be used in contact with food is not known, the conventional conditions are used, i.e. 2 dm^2 of surface in contact with 100 ml of simulant.

7.1.5.1.1.5 Single surface testing by filling

For articles in container form, e.g. bottles and trays, it is often most convenient to test them by filling with food simulant. For very large containers testing by filling may not be practicable and it may be necessary to fabricate smaller test specimens representing the article to be tested.

7.1.5.1.1.6 Articles intended for repeated use

It is accepted that where a material or article is intended to come into repeated contact with foodstuffs, the migration tests are carried out three times on the same test sample in accordance with the conditions laid down, using a fresh sample of the food simulant on each occasion. However, if there is conclusive proof that the level of migration does not increase in the second and third test and if the migration limit is not exceeded on the first test, no further test is necessary.

7.1.5.1.1.7 Caps, closures and other sealing devices

Caps, sealing gaskets and other sealing devices shall be tested under conditions that, as far as possible, simulate actual conditions of use.

The test is carried out on closures in the state and form in which they are intended to be used.

The simulants are placed in jars, known to give only consistently low migration, and the jars are closed with the test closures. The jars are then inverted and subjected to the test conditions appropriate for the actual conditions of use. The surface to volume ratio used shall be the same as that intended for use.

In many cases lids and closures may be expected to come into contact with foodstuffs and are tested under similar conditions to the rest of the container.

7.1.5.1.1.8 Large containers

Large containers, where filling is not practicable, may be tested by cutting test specimens from them and testing these by total immersion or by the cell method or using an equivalent cell. Alternatively, smaller test samples representing the large container may be fabricated and tested by filling.

7.1.5.1.1.9 Tubing, taps, valves and filters

Articles such as tubing, taps, valves etc. may be in contact with flowing foodstuff, this may be considered to be repeated brief contact for the purposes of migration testing. Such articles may be tested by repeated total immersion or by repeated filling; tubing may be stoppered with an inert stopper.

7.1.5.1.1.10 Fibres and cloths

Polymeric fibres and cloths are used to make such articles as sacks, filters, conveyor belting and bags for the infusion of beverages. In these circumstances it is not practicable to determine the surface area of the individual fibres in contact with the foodstuffs. Where limits of overall migration are expressed in milligrams per square decimetre of surface area the surface area may be taken as the superficial or projected area of the article.

7.1.5.1.1.11 Articles of irregular shape

Many articles that are required to be tested are of irregular shape or dimensions, e.g. thickness. Examples of these are sinks and work surfaces, eating and cooking utensils, shaped bottles and containers. When portions of these samples are taken for test by total immersion or in a cell care has to be exercised to ensure that the test specimens selected are representative of the whole of those parts of the article intended to come into contact with food. Also, care shall be taken to ensure that replicate test specimens are sufficiently dimensionally similar, one to another, to allow valid replication of results.

7.1.5.2 Number of test specimens

Five test specimens are required for samples, in the form of fillable article, thin films, sheets, cut sections from containers or similar articles. Seven test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape.

These test specimens are utilized as follows:

a) four test specimens for the migration test;

b) one test specimen to determine the suitability of oil as the fatty food simulant and triheptadecanoin as the internal standard (see Annex 0);

c) two test specimens for determination of the surface area, in the case of samples of irregular shape (see 7.1.5.5).

If previous testing has established that interference in the gas chromatography procedure is unlikely and section 0 is omitted, one test specimen less will be required.

Testing in triplicate is allowed but in this case if one test result is invalid repeat the entire procedure.

7.1.5.3 Cutting films, sheets and other flat materials or articles

7.1.5.3.1 Immersion method

Lay the sample on the cutting slab (7.1.4.1) and cut test specimens of 1 dm^2 (7.1.5.1.1.1) using the 100 mm x 100 mm template (7.1.4.4). Check, using the rule (7.1.4.6), that the dimensions of the test specimen are within the specified deviation (± 1 mm).

Cut each test specimen into four test pieces 25 mm x 100 mm using the rule (7.1.4.5). 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. Repeat this procedure for all remaining test specimens.

7.1.5.3.2 Cell method

Lay the sample with its non-food contact surface on the cutting slab (7.1.4.1). Take the ring from the cell type A (7.1.4.30) and place it on the food contact surface of the test sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (7.1.4.3)

7.1.5.3.3 Pouch method

Lay the sample with its non-food contact surface on the cutting slab (7.1.4.1) and cut test pieces using the 120 mm x 120 mm template (7.1.4.4). Two test pieces are required for each test specimen.

Place pairs of the test pieces together with the surfaces to be in contact with the olive oil facing. Use the heat or pressure sealer (7.1.4.32) to form pouches with four seals parallel to all four edges, 10 mm from the edge. Measure the distances between the inner edges of the seals to the nearest 1 mm and calculate the total surface area of the test specimen which will be exposed to olive oil, to the nearest 0.01 dm^2. This shall be approximately 2 dm^2. Using the cutting implement, remove excess film from the sealed area (to reduce the area of film not directly exposed to olive oil) whilst leaving enough to withstand the test conditions without leaking.

Measure and record the surface area of the pouch which will be in contact with the simulant and the total external area of the pouch after trimming excess malarial.

Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently large to insert a 100 ml pipette.

7.1.5.4 Cutting containers and other articles

7.1.5.4.1 Immersion method

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm^2. For articles with individual areas less than 1 dm^2, use a number of articles to provide each test specimen.

Measure the dimensions of each test specimen to the nearest 1 mm, using the rule (see 7.1.5.1.1.1).

Calculate the area of each test specimen to the nearest 0.01 dm^2 and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (7.1.4.11). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

NOTE Cutting the test specimens into smaller pieces will increase the area of cut edges, so that the area of cut edges exceeds 10% of the test specimen area (see 7.1.5.1.1.1).

7.1.5.4.2 Filling method

If the article is large, to avoid handling and weighing problems or using excessive amounts of olive oil it may be preferable to cut it so that the surface of the test specimen in contact with the olive oil does not exceed 3 dm^2.

If this is done, take care that olive oil does not come into contact with the cut edges of the test specimen. It is important that the area in contact with the oil is determined as it will be incorporated into the calculation later.

Scratch lightly an identification code on the external surface of each test specimen.

NOTE If only part of a test specimen is tested, this part should be representative of the whole in terms of composition and wall or layer thickness.

7.1.5.5 Cutting articles of irregular shape for immersion method

Select representative portions of the article, or multiples of the article for small articles, to give nine dimensionally similar test specimens each with a known total surface area of at least 1 dm^2. Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0.05 dm^2 using the Schlegel Method, as described in annex B of EN ISO 8442-2:1997, or any other suitable method. Record the surface area of each test specimen.

7.1.6 **Procedure**

The procedure consists of nine sections:

1. Check whether the vegetable oil and the internal standard are suitable for use in the procedure
2. Check the presence of volatile migrants in the test specimen
3. Check the sensitivity of the test specimen for humidity
4. Initial weighing of the test specimen
5. Contact of the test specimen to the vegetable oil for single use
6. Contact of the test specimen to the vegetable oil for repeated use
7. Final weighing of the test specimen
8. Extraction of the absorbed vegetable oil from the test specimen
9. Quantification of the amount of the adsorbed vegetable oil

7.1.6.1 Determination of the suitability of vegetable oil for overall migration testing

This procedure is carried out to verify that rectified olive oil is suitable as the fatty food simulant, and that triheptadecanoin is suitable for use as an internal standard (7.1.3.3) for the gas chromatographic determination of oil as its methyl esters.

7.1.6.1.1 Chromatogram of vegetable oil and internal standard

Weigh 45 mg to 55 mg of rectified olive oil (7.1.3.1) into a 50 ml flask (7.1.4.19) and add 10.0 ml of the cyclohexane solution of triheptadecanoin (7.1.3.3) by pipette (7.1.4.21). Remove the cyclohexane using a rotary evaporator or water bath (7.1.4.17 or 7.1.4.18) and add, by

measuring cylinder or graduated syringe (7.1.4.20), 10 ml ± 0.2 ml of n-heptane (7.1.3.6). Ensure the residue of rectified olive oil is well dispersed by shaking, warming or by ultrasonic treatment.

7.1.6.1.1.1 Methyl ester preparation procedure

Add by measuring cylinder or graduated syringe (7.1.4.20), 10 ml ± 0.2 ml of the potassium hydroxide solution (7.1.3.4) and a few anti-bumping beads (7.1.4.14). Connect a condenser to the flask and boil the mixture under reflux for 10 min ± 0.5 min.

Add through the condenser by measuring cylinder, or graduated syringe (7.1.4.20), 5 ml ± 0.2 ml of the methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2 min ± 0.25 min.

Cool to room temperature and add, by measuring cylinder (7.1.4.20), 15 ml to 20 ml of saturated sodium sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulphate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

If there will be a delay of more than seven days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

NOTE The methyl esters for the subsequent gas chromatographic determination are in the upper, n-heptane, layer.

Inject the methyl ester solution into the gas chromatograph (7.1.4.23).

NOTE A volume of 1-3 µl has been found suitable for the columns described in the note to 7.1.4.23.

Retain the chromatogram for comparison.

7.1.6.1.2 Chromatogram of test specimen extract

Take one of the test specimens, as prepared in section 7.1.5, and place it in a soxhlet type extractor (7.1.4.15). Take a 250 ml or 500 ml flask (7.1.4.15) and add 10 ml of cyclohexane without the internal standard and sufficient extraction solvent (7.1.3.2) to allow cycling of the soxhlet type extractor (approximately 200 ml or 400 ml, according to the size of the flask) with anti-bumping beads (7.1.4.14)) to control boiling. Using either a water bath or steam bath (7.1.4.18) extract for a period of 7 h (0/+1 h), with not less than six extraction cycles per hour, ensuring that the test pieces are totally submerged in the solvent during each soxhlet cycle, and that they remain separated from each other.

Drain all of the solvent from the soxhlet type extractor into the flask, remove the flask from the soxhlet type extractor and evaporate the solvent to a volume of approximately 10 ml using a rotary evaporator or simple distillation apparatus (7.1.4.17). Transfer the solution of the test specimen extract to a separate 50 ml flask (7.1.4.19) and wash the flask with three portions of 5 ml of solvent. Add the washings to the 50 ml flask. Evaporate to dryness using a rotary evaporator or water bath (7.1.4.17 or 7.1.4.18).

Add 10.0 ml ± 0.2 ml of the n-heptane to the 50 ml flask. Ensure that the test specimen extract is well dispersed by shaking, warming or by ultrasonic treatment. Then subject the contents of the flask to the methyl ester preparation procedure, described in 7.1.6.1.1.1 and inject the same volume of the resulting solution into the gas chromatograph (7.1.4.23). Retain the chromatogram.

7.1.6.1.3 Comparison of chromatograms

Compare the chromatogram of the methyl esters produced from the rectified olive oil and internal standard in the procedure in 7.1.6.1.1 with the chromatogram of the preparation from the test specimen extract produced in procedure 7.1.6.1.2. If peaks are present in the chromatogram of the extract with similar retention times to those of the peaks of rectified olive

oil methyl esters, and equate to 2 mg or more of olive oil, then the method is unsuitable for the material under examination. If a polar column has been used and interferences are observed on the peaks of the C18:0 and/or C18:2 peak, but not on other olive oil methyl ester peaks, then olive oil may be considered to be a suitable fat simulant, following method 3 given in 7.1.6.9.2.2.

If a peak is present in the chromatogram of the extract with similar retention time to that of the peak for methyl heptadecanoate, originating from triheptadecanoin, the internal standard, and is more than 1 % of the height or area of that peak, then consider an alternative internal standard.

NOTE A suitable alternative internal standard is trinonadecanoin or hydrocinnamic acid, ethyl ester.

NOTE **Figure 12** and **Figure 13** show typical chromatograms of the methyl esters of olive oil and triheptadecanoin using columns 1 and 2, respectively.

7.1.6.2 Determination of the presence of volatile substances

Determine the need for removing volatile substances of the test specimens by carrying out the procedure described in this section. If prior tests have established that sample conditioning for removing volatile substances is not required then follow Annex 7.1.6.4.1.

Take one test specimen, as prepared in 7.1.5 and determine the mass to the nearest milligram. Place the test specimen in a vacuum oven (7.1.4.25) at 60±5°C. Reduce the pressure in the oven to 1.3 kPa or less. Leave the test specimen in the oven for 60±10 min. Release the pressure and transfer the test specimen from the vacuum oven to a desiccator (7.1.4.26) containing self-indicating silica gel or anhydrous calcium chloride. Determine, after cooling for 60±10 min the mass of the test specimen. Calculate the difference between the mass of the test specimen before and after the one hour vacuum conditioning. Discard the test specimen.

If the difference between the masses of the test specimen is greater than 2 mg/dm^2, then conditioning of the test specimens to be used in the test will be necessary before each weighing operation in the test procedure (7.1.6.4.2). Is the difference between the masses of the test specimen is less than 2 mg/dm^2, then conditioning of the test specimens to be used in the test will not be necessary before each weighing operation in the test procedure.

7.1.6.3 Determination of the moisture sensitivity of the test specimen

This procedure determines whether the conditioning of test specimens with respect to moisture content will be required.

Take one test specimen, as prepared in 7.1.5 and place in a container (7.1.4.10) maintained at 80% relative humidity for 24 ± 4 h. Remove the test specimen and weigh as quickly as possible after its removal from the controlled environment, to minimise loss of moisture and change in mass.

Place the same test specimen in a container (7.1.4.10) maintained at 50% relative humidity for 24 ± 4 h. Remove the test specimen and weigh, taking the same precautions as above.

If the difference between the masses of the test specimen is greater than 2 mg/dm^2, then conditioning of the test specimens will be necessary before each weighing operation in the test procedure. If the difference between the masses of the test specimen is less than 2 mg/dm^2, then conditioning of the test specimens will not be necessary before each weighing operation in the test procedure.

7.1.6.4 Initial weighing of test specimens

Before weighing, discharge any build-up of static electricity with an antistatic gun or other suitable means.

7.1.6.4.1 Determination of initial weight of non-moisture sensitive test specimen in absence of volatiles

If the test in section 7.1.6.2, shows that there is no substantial amount of volatile substances present in the test specimen and if the test in section 7.1.6.3 shows that the test specimen is not moisture sensitive, determine and record the mass of each test specimen (m_a).

7.1.6.4.2 Determination of initial weight of non-moisture sensitive test specimen in presence of volatiles

If the test in section 7.1.6.2 shows that there is a substantial amount of volatile substances present and if the test in section 7.1.6.3 shows that the test specimen is not moisture sensitive, follow the directions in this section.

7.1.6.4.2.1 Vaccuum conditioning of non-moisture sensitive test specimen in presence of volatiles

Weigh the four test specimens (w_a), as prepared in 7.1.5. Then transfer to a vacuum oven at 60±5°C and reduce the pressure to approximately 1.3 kPa using a high vacuum pump. The vacuum pump can be turned off provided the pressure is maintained. Turn on the vacuum pump every hour for a period of 10-15 min to remove moisture from the oven and to refresh the vacuum. Leave the test specimens under this condition in the vacuum oven for a period of 24±2 h. Transfer the test specimens from the vacuum oven to a desiccator containing self-indicating silica gel or anhydrous calcium chloride. Determine, after cooling for 60±10 min, the mass of the test specimen. Repeat the conditioning procedure until the change in mass between two consecutive weighing's is less than 2 mg/dm^2. Record the final mass of each test specimen (w_b).

7.1.6.4.2.2 Reconditioning of the test specimen after vacuum conditioning

Place the test specimens at ambient humidity (not less than 60%) and determine, after cooling for 60±10 min, the mass of the test specimen. Repeat the conditioning procedure until the change in mass between two consecutive weighing's is less than 2 mg/dm^2. Record the final mass of each test specimen (m_a).

NOTE: Reconditioning is only required for test specimen that have lost water and that may have influenced the physical properties of the test specimen. E.g. polyamide samples need reconditioning to regain their initial properties. If it is known that the loss of mass is due to volatile substances, other than water, then reconditioning is not needed.

NOTE The difference of the mass before and after the reconditioning, w_b and m_a, respectively, indicates the amount of volatiles, including water, that is present in the test specimen.

Usually 70% of the mass lost will be regained. However if significantly less mass is regained then this is an indication for the loss of organic volatiles.

7.1.6.4.3 Determination of initial weight of moisture sensitive test specimen in absence of volatiles

If the test in section 7.1.6.2 shows that there is no substantial amount of volatile substances present and if the test in section 7.1.6.3 shows that the test specimen is moisture sensitive, follow the directions in this section.

Place the test specimen in the container maintained at 50% relative humidity, weigh at intervals of about 24 h, until the change in mass between consecutive weighings of each test specimen is less than 2 mg/dm^2 and record the final mass of each test specimen (m_a).

NOTE: This determination is also applied in case the sample cannot be conditioned using the vacuum method in section 7.1.6.4.2.1 due to decomposition or irreversible changes of the test specimen. However volatiles may then be included.

NOTE: This determination may be used in the screening procedure in which the migration of volatiles is included to demonstrate compliance with generic SML's of volatile organic substances.

7.1.6.4.4 Determination of initial weight of moisture sensitive test specimen in presence of volatiles

If the test in section 7.1.6.2 shows that there is a substantial amount of volatile substances present and if the test in section 7.1.6.3 shows that the test specimen is moisture sensitive, follow the directions in section 7.1.6.4.2.1 followed by section 7.1.6.4.3.

7.1.6.5 Contact with food simulant for single use

7.1.6.5.1 Immersion method

Take six of the glass tubes (7.1.4.11), mark them for identification purposes. Measure 100 ml ± 5 ml of olive oil (7.1.3.1) into each tube by measuring cylinder and stopper the tube.

Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Place the six tubes in the thermostatically controlled oven or incubator (7.1.4.12) set at the test contact temperature. Leave until the olive oil has attained the test contact temperature, using the thermometer or thermocouple to monitor the temperature. Take all tubes from the oven and place into five of the tubes containing olive oil, weighed test specimens prepared as in section 7.1.5 and conditioned if necessary as in section 7.1.6.4. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Ensure that the test specimens are totally immersed in olive oil; if they are not, add then either glass beads or glass rods (7.1.4.22) preheated at the test contact temperature to raise the level of the olive oil until total immersion is achieved.

NOTE 2 The olive oil in the sixth tube is used as a reference standard in constructing the calibration graph (see 7.1.6.9.2.2).

NOTE – To ensure that test pieces are well separated and that their surfaces are freely exposed to oil during the period of the test, insert a piece of fine stainless steel gauze (10.2.4.9) between the test pieces for thin films or insert glass rods between the test pieces after immersion in the oil for thick samples not placed on the supports,. Where test specimen supports are used, label the supports with a tag bearing the test specimen identification.

NOTE the tube with the thermometer or thermocouple is only used for monitoring the temperature

Replace all six tubes in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the olive oil (see NOTE 5) in the sixth tube and leave the tubes for the selected test period, taking into account the tolerances specified in Table 4, after the olive oil in the sixth tube has reached a temperature within the tolerance specified in Table 5.

NOTE 4 Table 4 and Table 5 include tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this standard.

NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

NOTE 5a – in some cases it is difficult to keep the test contact temperature as soon as the test specimen is immersed in the food simulant and put in the oven at the test contact temperature, taking into account the tolerances specified in Table 4. It is very important that all materials except the test specimen used in the test are at the test contact temperature. An extra measure can be to put a water bath with circulation in the oven in which the tubes are put. If the test contact temperature cannot be maintained it is allowed to ensure that the test contact temperature is correct by setting the oven temperature higher.

Take the tubes from the oven or incubator and immediately remove the test specimens from the tubes. For those specimens that have been in olive oil, allow the oil to drain. Remove any

adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the pressing procedure until the filter paper shows no spots of olive oil. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of oil by washing with the extraction solvent and replace the test pieces on them.

7.1.6.5.2 Cell method

Take five type A cells (7.1.4.30), mark them for identification purposes. Place in the thermostatically controlled oven or incubator (7.1.4.12), which is set at the test contact temperature and leave until the test contact temperature has been attained.

Take six glass tubes (7.1.4.11), measure 125±5 ml of olive oil (7.1.3.1) into each tube by measuring cylinder and stopper the tubes.

Alternatively mark the tubes for a volume of 125 ml and fill with olive oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Place the six tubes in the thermostatically controlled oven or incubator (7.1.4.12) set at the test temperature. Leave until the olive oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

NOTE the tube with the thermometer or thermocouple is only used for monitoring the temperature. This tube is also used for filling the cell used for temperature monitoring.

Remove the cells from the thermostatically controlled oven or incubator, dismantle the cells and place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Remove all tubes from the thermostatically controlled oven or incubator or refrigerator and transfer the olive oil from each tube to each of the cells through the filler hole. Remove the thermometer or thermocouple from the tube and insert, if applicable see NOTE 5, in one of the cells and replace the filler plugs.

NOTE 2 The olive oil in the sixth tube is used as a reference standard in constructing the calibration graph (see 7.1.6.9.2.2).

Replace the five cells and the remaining tube in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the cells and olive oil. Observe the temperature of the thermostatically controlled oven or incubator or the olive oil (see NOTE 5) in the one of the cells and leave the cells and tubes for the selected test period, taking into account the tolerances specified in Table 4, after the olive oil in the cell has reached a temperature within the tolerance specified in Table 5.

NOTE 3 The above procedure is typically for cell A (7.1.4.30). The procedure may deviate if another type of cell is used. In all cases attention shall be given to reaching the intended test contact temperature of the oil in the cell to establish the start of the migration period.

NOTE 4 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this method.

NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the air-bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the cells and tube from the oven or incubator and immediately remove the test specimens from the cells and allow the oil to drain. Remove any adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the pressing procedure until the filter paper shows no spots of olive oil.

7.1.6.5.3 Pouch method

Take six of the glass tubes (7.1.4.11), measure 100 ± 5 ml of olive oil (7.1.3.1) into each tube by measuring cylinder (7.1.4.20) and stopper the tube.

NOTE 1 The pouch holder (7.1.4.31) should be cleaned before use, if necessary, using solvents, such as acetone and or detergents. For olive oil that is difficult to remove, use proprietary solvent mixtures.

WARNING Proprietary solvent mixtures usually contain caustic substances and also volatile solvents. Handle with care, using protective gloves and eye protection, in a fume cupboard. Information regarding sources of the proprietary mixtures specified in this test method is available from National Standards Bodies.

Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place the six tubes and the pouch holder, in the thermostatically controlled oven or incubator (7.1.4.12) set at the test temperature.

NOTE 3 Leakage can occur from the pouches and it is advisable to have a drip tray in the oven.

NOTE the tube with the thermometer or thermocouple is only used for monitoring the temperature. This tube is also used for filling the pouch used for temperature monitoring.

Leave until the olive oil has attained the test temperature, using the thermometer or thermocouple to monitor the temperature.

Remove the pouch holder from the thermostatically controlled oven or incubator and place the test specimens between the spacers.

Remove all tubes containing olive oil from the oven and pipette sufficient olive oil into four pouches. This shall be approximately 100 ml, but for thick/semi-rigid materials the quantity will be less. Place a thermocouple in one pouch and close the open corners with a clip.

NOTE 4 The olive oil in the sixth tube is used as a reference standard for constructing the calibration graph (see 7.1.6.9.2.2).

Replace the pouch holder, containing the four pouches, and the tube in the thermostatically controlled oven or incubator set at the test temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or incubator or the olive oil (see NOTE 7) in the pouch and leave the pouches and tubes for the selected test period, taking into account the tolerances specified in Table 4, after the olive oil in the pouch has reached a temperature within the tolerance specified in Table 5.

NOTE 6 Table 4 and Table 5 include tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this part of the test method.

NOTE 7 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Take the pouch holder and the tubes containing olive oil form the thermostatically controlled oven or incubator.

If an evident leak has occurred with more than one pouch the test is invalid and shall be repealed.

If no evident leaks have occurred in at least three pouches, then remove the pouches from the holder.

Pour the olive oil from each pouch and wipe any excess from the outside with filter paper (7.1.4.13). Take each of the four pouches in turn, lay them on the cutting slab (7.1.4.1) and open

them carefully by cutting through one layer along the inner edges of the seals using the cutting implement (10.2.4.3).

Take the two portions of each pouch and remove adhering olive oil by gently pressing between filter papers. Repeat the pressing procedure until the filter paper shows no spots of olive oil.

7.1.6.5.4 Filling method

Place a sufficient volume of olive oil in a beaker in the thermostatically controlled oven or incubator (7.1.4.12) which is set at the test temperature and leave until the test temperature has been attained.

Place a sufficient volume of olive oil in five beakers, to fill each of five test specimens to the nominal volume or to 5 mm from the top if the nominal volume is not known. Insert a thermometer or thermocouple in one of the beakers containing the olive oil for a test specimen.

NOTE the beaker with the thermometer or thermocouple is only used for monitoring the temperature. This beaker is also used for filling the test specimen used for temperature monitoring.

Take one glass tube (10.1.4.11), measure 100 ± 5 ml of olive oil (10.1.3.1) into it by measuring cylinder (10.1.4.20) and stopper the tube. This tube is used as reference standard in constructing the calibration graph (see 7.1.6.9.2.2).

Place the beakers and the tube in the thermostatically controlled oven or incubator or refrigerator set at the test temperature and leave until the olive oil has attained the test temperature.

Place each test specimen on a clean, oil free surface and fill five test specimens with olive oil to within 0.5 cm of the top. If the container has a specified nominal volume of contents, see section 7.1.5.1.1. Place into one of the filled test specimens a thermometer or thermocouple.

NOTE 2 Care should be taken not to spill any oil on the external surfaces.

Place the five filled test specimens and the tube in the thermostatically controlled oven or incubator set at the test contact temperature. This part of the operation should be carried out in the minimum time possible to prevent undue heat loss.

Observe the temperature of the thermostatically controlled oven or incubator or the olive oil (see NOTE 5) in the filled test specimen and leave the test specimens for the selected test period, taking into account the tolerances specified in Table 4, after the olive oil in the test specimen has reached a temperature within the tolerance specified in Table 5.

NOTE 4 Table 4 and Table 5 include tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this part of the test method.

NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

Remove the test specimens and the tube from the thermostatically controlled oven or incubator and immediately empty the test specimens that contained olive oil and allow the oil to drain. Remove any adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the pressing procedure until the filter paper shows no spots of olive oil.

7.1.6.6 Contact with food simulant for repeated use

With vegetable oil, the repeated contact of the same test specimen to fresh portions of food simulant is not a feasible procedure, since the procedure requires solvent extraction to remove the oil. Therefore, the test is carried out on three sets of five test specimens from the same sample of the material or article. One set is subjected to the test appropriate for articles intended for single use by the standard procedure and the mean result calculated (M1) (see section 7.1.6.5). The other two sets for the second and third migration are exposed in a manner identical

in every respect to those of the first migration except for the period of contact. The test specimens of the second migration (M2) are exposed for a period of twice that of sample one and the test specimens of the third migration (M3) are exposed for a period three times that of those of the first migration.

NOTE: the sets dedicated for the second and third migration may have four test specimens if the migration of all test specimens is carried out in one oven. Then the fifth test specimen of the first migration containing the thermocouple serves for all sets.

7.1.6.7 Final weighing of test specimens

7.1.6.7.1 Determination of final weight of non-moisture sensitive test specimen

Weigh all four test specimens and record their mass (m_b).

7.1.6.7.2 Determination of final weight of moisture sensitive test specimen

Place the four test specimen in the container maintained at 50% relative humidity, weigh at intervals of about 24 h, until the change in mass between consecutive weighings of each test specimen is less than 2 mg/dm^2 and record the final mass of each test specimen (m_b).

7.1.6.8 Extraction of absorbed vegetable oil

Take four flasks, 250 ml or 500 ml as appropriate to the size of the soxhlet type extractor (7.1.4.15) to be used for the extraction, and place in each flask 10.0 ml of the internal standard cyclohexane solution of triheptadecanoin (7.1.3.3), using a pipette (7.1.4.21), or an alternative higher quantity if more than 100 mg of olive oil is present.

NOTE 1 if the test specimens have retained more than 100 mg of olive oil, 10.0 ml of the internal standard solution will be insufficient for optimum precision in the gas chromatography determination after extraction. Before commencing the operations in this section an estimation of the quantity of olive oil retained in the test specimens should be obtained by comparing the final masses of the test specimens with their initial masses. If considered necessary the quantity of internal standard solution can be increased from 10 ml although it is essential that the same quantity is used for each test specimen, and that this quantity is also used with the olive oil standards for the calibration graph (see 7.1.6.9.2.2). As a guide, approximately 0.5 mg of the internal standard is required for every mg of extracted olive oil.

Add sufficient extraction solvent (7.1.3.2) to allow cycling of the soxhlet type extractor (approximately 200 ml or 400 ml, according to the size of the flask) with anti-bumping beads (7.1.4.14) to control boiling.

Cut the test specimens into suitable sized strips, not wider than 30 mm and of correct length such that the strips shall be totally immersed during the soxhlet cycle.

NOTE 1a Care should be taken when carrying out the cutting operations to ensure that slivers are not produced and lost.

Place the four test specimens that have been in contact with olive oil into four soxhlet type extractors. Couple each soxhlet to a flask containing the internal standard prepared as above. Using either a water bath or steam bath (7.1.4.16), extract for a period of 7 h (0/+1h), with a minimum of six cycles per hour, ensuring that the test pieces are totally submerged in the solvent during each soxhlet cycle, and that they remain separated from each other.

Drain all of the solvent from the soxhlet type extractors, remove the flasks from the soxhlet type extractors and evaporate the solvent to approximately 10 ml using a rotary evaporator, or simple distillation apparatus (7.1.4.17). Transfer the solutions containing the extracted olive oil and internal standard to separate 50 ml flasks (7.1.4.19), and wash each flask with three portions of 5 ml of solvent. Add the three washings to the respective individual 50 ml flasks. Evaporate to dryness using a rotary evaporator or a water bath.

NOTE 2 Oxidation of the olive oil should be avoided where possible. Therefore evaporation of the solvent to dryness should be carried out under mild conditions of temperature. In addition contact of the olive oil to oxygen should be limited.

NOTE 3 Some types of plastic are known to retain some of the absorbed olive oil despite prolonged soxhlet extraction with pentane. In these cases extraction of the olive oil is incomplete. This is known to give falsely low results in the test procedure. This difficulty may be overcome by subjecting the test specimens to a second extraction, this time with diethyl ether, or to the dissolution/precipitation method set out in section 7.4. The amount of oil obtained in the diethyl ether extract or in the solution after precipitation of the polymer is added to the amount of oil obtained in the pentane extract. To obtain reliable results the migration test shall be repeated using the dissolution/precipitation method.

Repeat the extraction of the test specimens for an additional 7 h (0/+1h), with diethyl ether (7.1.3.8), adding a further quantity of the internal standard solution.

NOTE 4 The same quantity of internal standard solution is used as for the first 7 h extraction. This quantity may not be the optimum if the quantity of olive oil in the first 7 h extraction is high. Good precision is not required for the second 7 h determinations since they are intended primarily as a check on the efficiency of the first 7 h extraction and using the same quantity of internal standard enables one calibration graph to be used.

If previous testing has established that all of the olive oil will be extracted from the test specimens during the first 7 h extraction then the second 7 h extraction may be omitted.

Isolate the residues in 50 ml flasks, using the procedure described above.

Determine the extracted olive oil in both the first 7 h and the second 7 h extraction by the procedure described in 7.1.6.8, but retain the test specimens in the soxhlet type extractors until the extracted olive oil has been determined for the second extraction.

7.1.6.9 Determination of extracted olive oil

7.1.6.9.1 Preparation of fatty acid methyl esters

Add 10 ± 0.2 ml of n-heptane to each of the 50 ml flasks containing die first 7 h extraction residue, by measuring cylinder (7.1.4.20), ensuring that the residues of olive oil and plastics extractables dissolve or are well dispersed by shaking, warming or by ultrasonic treatment.

NOTE 1 Unless die residues in the flasks are dissolved or well dispersed in the n-heptane, quantitative hydrolysis or methylation of the olive oil and of the internal standard might not be obtained under the conditions described particularly when these residues contain extractables from plastics in excess of 50 mg. The internal standard might not react with the plastics extractables to the same degree as does the olive oil and correct results for olive oil might not be obtained.

Add by measuring cylinder or graduated syringe (7.1.4.20), 10 ± 0.2 ml of the potassium hydroxide solution (7.1.3.4) and a few anti-bumping beads (7.1.4.14). Connect a condenser to the flask and boil the mixture under reflux for 10 ± 1.0 min.

Add through the condenser by measuring cylinder, or graduated syringe, 5.0 ± 0.2 ml of the methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2 ± 0.25 min.

Cool to room ternperature and add, by measuring cylinder. 15-20 ml of saturated sodium sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulfate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the upper layer of n-heptane.

Treat the residues from the second 7 h extraction as described above.

If there will be a delay of more than 7 days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

7.1.6.9.2 Determination of fatty acid methyl esters

7.1.6.9.2.1 Instrument

Determine the methyl esters of the olive oil fatty acids using a gas chromatograph (7.1.4.23).

NOTE 1 For column 1 described in the note to 7.1.4.23 the following operating conditions have been found to be suitable:

carrier gas	helium at 2 ml/min
injector	spit (ratio 40:1)
detector	flame ionisation
temperature programme	initially 1 min at 140°C then ramped at 5°C/min to 190°C and maintained at 190°C for 8 min.
injector temperature	220°C
detector temperature	240°C

For column 2 described in the note to 7.1.4.23 the following operating conditions have been found to be suitable:

carrier gas	helium
oven temperature	250°C isothermal
injector temperature	320 °C
detector temperature	320 °c

Use an integrator to measure the area of each of the olive oil peaks and the internal standard.

NOTE 2 The use of an integrator and measurement of the peak area is the preferred method.

7.1.6.9.2.2 Calibration graph

Weigh a range of quantities of the blank reference olive oil which has been subjected to the same test conditions as the test specimens into 50 ml flasks (7.1.4.19). Weigh a range of olive oil quantities spanning the quantities of olive oil in the first 7 h extractions, taking no fewer than four standards.

Add 10.0 ml of the internal standard cyclohexane solution of triheptadecanoin (7.1.3.3) to each flask using a pipette (7.1.4.21), or the alternative quantity which has been added to the extraction flasks in section 7.1.6.8. Remove the cyclohexane using a rotary evaporator or water bath (7.1.4.18 or 7.1.4.17). Subject the olive oil quantities, with the added internal standard, to the methyl ester preparation procedure described in section 7.1.6.9.1.

Inject each of the n-heptane methyl ester solutions in duplicate, as a minimum, into the gas chromatographic column.

NOTE 1 Typical chromatograms generated using columns 1 and 2 are shown respectively in Figure 12 and Figure 13,

Construct a calibration graph, plotting the ratios of olive oil methyl esters to the internal standard peak on the y-axis and against the weighed quantities of olive oil on the x-axis.

Various methods for the construction of a calibration graph are suitable and the choice of method depends on the equipment and chromatographic column used. The following methods are acceptable:

Method 1 Peak height method

Measure the peak height of the internal standard peak and of the methyl oleate (C18:1) peak, when a polar column has been employed. In the case where a non-polar column has been used for the separation of the methyl ester, then measure the internal standard peak and the C18 peak of the olive oil. Calculate the ratio of the measured C18 peaks to the internal standard peak and plot the ratios versus the weighed quantities of olive oil.

Method 2 Peak area method

Measure the peak area of the internal standard peak and of each of the methyl esters originating from the olive oil. Add together the peak areas of the C16 and C18 peaks if a non-polar column was employed. If a polar column was used, sum the areas of all the peaks (C16:0, C16:1, C18:0, C18:1 and C18:2) originating from the olive oil. Calculate the ratio of the combined areas of the measured peaks to the area of the internal standard peak and plot the ratio versus the weighed quantities of olive oil.

Method 3 Peak area method in the case of interference from the test sample.

In the event that the analysis of a blank test sample, see Annex 0, has revealed an interference with one or more of the olive oil methyl esters, but not all of the peaks, then this peak or peaks shall be excluded from the calculation of the total area of the olive oil methyl esters. Calculate the ratio of the total area of the methyl esters originating from olive oil and which are free from interference and the area of the internal standard and plot the ratios versus the weighed quantities of oil.

NOTE 2 A typical calibration graph is shown in Figure 14.

Calculate from each calibration standard chromatogram the C18:1/C16:0 ratio if a polar column was used or C18/C16 ratio in the case of a non-polar column. Determine the mean ratio value from the duplicate or multiple injections for comparison with the same ratio obtained from the test specimen extracts, see 7.1.6.9.2.3.

NOTE 3 If another vegetable oil than olive oil is used then the ratio of the main peaks in the chromatogram shall be calculated

7.1.6.9.2.3 Determination of olive oil absorbed by test specimens

Inject into the gas chromatograph (7.1.4.23) a suitable quantity from each of the n-heptane methyl ester solutions prepared from the residues containing the extracted olive oil (see 7.1.6.9.1). Inject in duplicate, as a minimum.

For each chromatogram, measure the height or area of the olive oil methyl ester peak or peaks and the internal standard peak using the same peaks and method as used in the construction of the calibration graph, see 7.1.6.9.2. Calculate the ratio of the relevant peaks to the internal standard peak for each chromatogram and for each solution determine the mean ratio value from the duplicate or multiple injections.

Calculate the amount of olive oil extracted from the test specimen from the regression parameters

If the regression line equation is

$$y = ax = b \quad (1)$$

then:

$$m_{\infty} = \frac{(y-b)}{a} \quad (2)$$

where

m_{∞} is the mass of olive oil extracted from the sample, in milligrams:

a is the slope of the calibration graph;

b is the intercept of the calibration graph;

x is the mass of olive oil in the standard, in milligrams;

y is the ratio of olive oil methyl esters to internal standard.

The procedure yields directly the amount of olive oil extracted from the test specimen, in milligrams.

NOTE 1 The method applying calculation from the regression parameters is the preferred method.

If olive oil is found in the second extract from more than one of the test specimens and the amount is less than 10 mg, but measurable, add this to the amount determined from the first 7 h extraction and record the total mass of extracted olive oil for each test specimen in grams.

If more than 10 mg of olive oil is found in the second extract, see NOTE 3 of Annex 7.1.6.8

If the ratio C18 to the C16 peaks has changed, read NOTE 1a

NOTE 1a A difference in C18:1/C16:0 ratio (using column 1) between the olive oil extracted from the test specimen and the olive oil applied as the fatty food simulant in the migration test indicates that the composition of the extracted oil for some reason is different from the composition of the oil that has not been in contact with a test specimen. Possible causes for the changes of the composition are:

- reaction of olive oil constituents with plastics constituents;
- oxidation of unsaturated constituents of the olive oil. This has been observed to occur when rather long periods for conditioning the test specimen after contact with the oil are necessary;
- incomplete methylation of fatty acids in the trans-esterification procedure, such difficulties arise with some types of high impact polystyrene (HIPS) and acrylonitrile-butadiene-styrene (ABS);
- selective absorption of oil constituents by test specimens. Polyolefins for example do absorb selectively mono- and diglycerides of saturated free fatty acids in some cases, whereas HIPS, ABS and nitrilebutadiene rubber (NBR) often selectively absorb diglycerides, and to a lesser extent also monoglycerides of unsaturated fatty acids;
- interference by plastics constituents having the same retention time as C16:0 or C18:1 methyl ester or forming those esters in the trans-esterification stage.

Whether a change in the C18:1/C16:O ratio acts upon the final result of the overall migration determination to an extent which is not acceptable depends mainly on the magnitude of the change and on the amount of oil recovered from the test specimen, e.g. a 25 % change in the C18:1/C16:O ratio may result in a 25 % lower result in the amount of fat extracted, which would mean 2.5 mg when only 10 mg fat is absorbed by the test specimen but 25 mg when 100 mg of fat is absorbed. So a proportional change in C18:1/C16:0 ratio will result in an absolute difference in the amount of fat calculated, and consequently in an absolute difference in the overall migration values. Whilst an absolute difference of 2.5 mg is acceptable, because it is within the accepted analytical tolerance, one of 25 mg is not.

Whether there might be a possibility of obtaining false results because of a change in the C18:1/C16:0 ratio, can easily be established by measuring the amount of oil extracted from the test specimen using two different calibration graphs. In one graph the ratio C16:0/C17:0 is plotted versus the amount of olive oil and in the other one the ratio C18:1/C17:0. The amount of oil calculated using the C16:0/C17:0 graph shall differ from the amount calculated using the C18:1/C17:0 graph by no more than 2 mg/dm^2. In case a larger difference is observed the cause of it has to be identified and an appropriate action be taken. Remedies for problems could be:

- if reaction of oil constituents with plastics constituents is suspected a less reactive oil, e.g coconut oil or palmkernel oil, can be used;
- if oxidation of unsaturated fatty acids is suspected a less vulnerable fatty food simulant, e.g coconut oil or palmkernel oil, can be used;

- if incomplete methylation of fatty acids during trans-esterification is suspected the heptane layer obtained in the normal trans-esterification procedure is subjected to an additional trans-esterification treatment;
- if selective absorption of fatty simulant constituents by the test specimen is suspected, which can be ascertained by thin layer chromatography comparison of the composition of extracted and olive oil a fatty food simulant low in free fatty acids and mono- and diglycerides can be used;
- if interference of oleic acid (C18:1) or heptadecanoic acid (C17:0) peak area measurement by plastic constituents is suspected which can be ascertained by running a blank experiment with a sample of the final article in question, the palmitic acid (C16:0) peak area of olive oil can be used as a reference. It is preferable however to use, if possible, sunflower, coconut or palmkernel oil as the food simulant instead.

For each chromatogram from the first 7 h extractions, calculate the ratio of the height or area of the C18 peak to the height or area of the C16 peak. Determine the mean value of these ratios and compare to the similar ratio determined in 7.1.6.9.2.2 from the olive oil calibration chromatograms. Establish whether the difference between the two ratios values is acceptable.

NOTE 2 A change in the C18/C16 ratio for extracted olive oil samples compared with the same ratio for olive oil used for the calibration graph indicates that some reaction or fractionation of the olive oil has occurred, either during the test period or during extraction of the test specimens. Such changes will have an adverse effect on the overall migration result.

7.1.7 Expression of results

7.1.7.1 Method of calculation for single use

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

$$M = \frac{(m_a - (m_b - m_c)) \cdot 1000}{S} \quad (3)$$

where

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

m_a is the initial mass of the test specimen, before contact with the olive oil, in grams (see section 7.1.6.4);

m_b is the mass of the test specimen after contact with olive oil, in grams (see section 7.1.6.5.2);

m_c is the mass of olive oil absorbed by test specimen, in grams (see section 7.1.6.9.2.3);

S is the surface area of the test specimen in contact with food simulant in square decimetres (see 7.1.5.1.1). Table 4 in section 4.4.3 gives guidelines for selection of the surface area in case of the immersion test.

Calculate the result for each test specimen to the nearest 0.1 mg/dm^2.

7.1.7.2 Method of calculation for repeated use

The mean result for the test specimens of the second migration is calculated (M2) as is that for the test specimens of the third migration (M3).

The migration as a result of the second or third period is calculated as follows:

- migration caused by first period = M1

- migration caused by the second period = M2 - M1

- migration caused by the third period = M3 - M2.

No increase in migration into fatty food simulant is deemed to have occurred if the results (M3 - M2) and (M2 - M1) do not exceed M1 by more than the analytical tolerance.

The true values for M1, M2 or M3 are subject to uncertainty owing to the lack of precision inherent in the method. Systematic errors in the determination of the overall migration are likely to occur equally to the determination of M1, M2 or M3 and therefore need not be allowed for. Random errors do need to be recognized and allowed for.

When repeated testing is used to determine the overall migration into a vegetable oil the individual results for each set of the determinations (M1, M2 or M3) shall be deemed valid if at least three results are obtained in each set which do not differ from the mean for that set by more than 30% for results above 10 mg/dm2 or by more than 3 mg/dm2 for results below 10 mg/dm2. Results which exceed this tolerance shall be discarded according to the procedure given in 11.1.7.2.

7.1.7.3 Validity of individual results

The following analytical tolerances are allowed:

20 mg/kg or 3 mg/dm^2 for all vegetable oils.

If a reduction factor does not apply, results above 10 mg/dm^2 shall not differ by more than 30% from the mean of the set of results.

The determination of overall migration into the fatty food simulant is normally carried out in quadruplicate to allow three valid results to be obtained even if one determination is discarded.

Where four results have been obtained from four determinations, i.e. no single determination has been rejected because of an obvious manipulative error, all four results are valid when each individual result differs from the mean of the four results by not more than the analytical tolerance. However:

- If one of the four results is greater or less than the mean by an amount more than the tolerance, then this result can be rejected and the mean recalculated on the remaining three results.
- If two results are greater or less than the mean by amounts more than the tolerance, the result with the largest difference from the mean can be rejected and a new mean calculated from the remaining three results. The remaining three test results are valid if they are within the analytical tolerance.

If a minimum of three results do not meet the above criteria of being within the analytical tolerance, then the test shall be repeated using fresh test specimens from the sample.

7.1.7.4 Precision

Evaluation of the results of a collaborative trial with a plastic film having a mean overall migration of 6.6 mg/dm^2, determined by the total immersion method, has given the following values for repeatability (r) and reproducibility (R):

r = 2.0 mg/dm^2

R = 2.9 mg/dm^2

The precision data were determined from an experiment conducted in 1996 involving 11 laboratories and six replicates.

Evaluation of the results of a further collaborative trial with a plastic film having a mean overall migration of 8.3 mg/dm^2 and determined by the total immersion method, has given the following values for repeatability and reproducibility:

r = 1.8 mgldm^2

R = 3.7 mg/dm^2

The precision data were determined from an experiment conducted in 1997 involving eight laboratories and six replicates.

7.1.8 Test report

The test report shall include the following:

- reference to this annex of the guidance document;
- all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number(s), thickness;
- conditions of contact time and temperature to food simulants;
- Details of the options in this test method used for the determination
- deviations from the specified procedure and reasons for these;
- individual test results including measurement uncertainty;
- relevant comments on the test results;

Table 4 tolerances related to the test contact time

Contact times	Tolerance min/max
30 min	0/+1 min
60 min	0/+1 min
90 min	0/+3 min
120 min	0/+5 min
150 min	0/+5 min
180 min	0/+7 min
210 min	0/+8 min
240 min	0/+9 min
270 min	0/+10 min
300 min	0/+12 min
360 min	0/+15 min
24 h	0/+0.5 h
48 h	0/+0.5 h
240 h	0/+5 h

Table 5 tolerances related to test contact temperature

Temperature	Tolerance
°C	°C
5	±1
20	±1
30	±1

40	±1
50	±2
60	±2
70	±2
80	±3
90	±3
100	±3
121	±3
130	±5
140	±5
150	±5
160	±5
170	±5
175	±5

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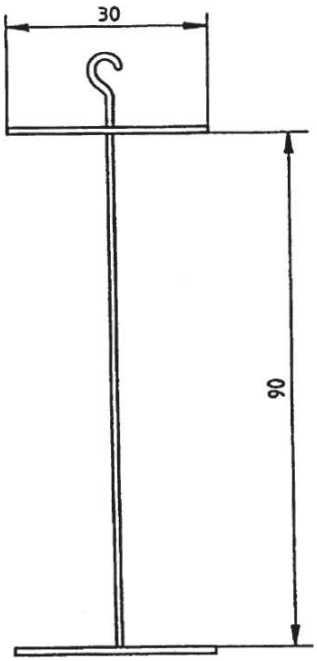
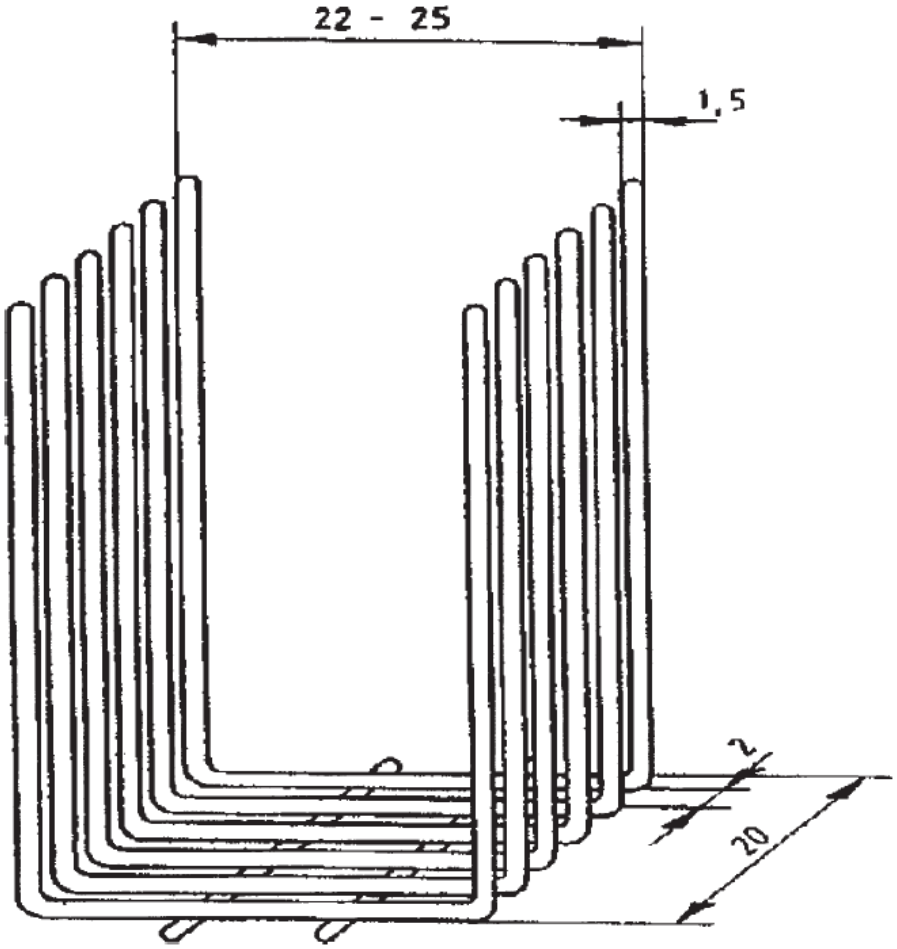


4338

4339 **Figure 3 support; dimensions in mm**



4340

4341 **Figure 4 support; dimensions in mm**

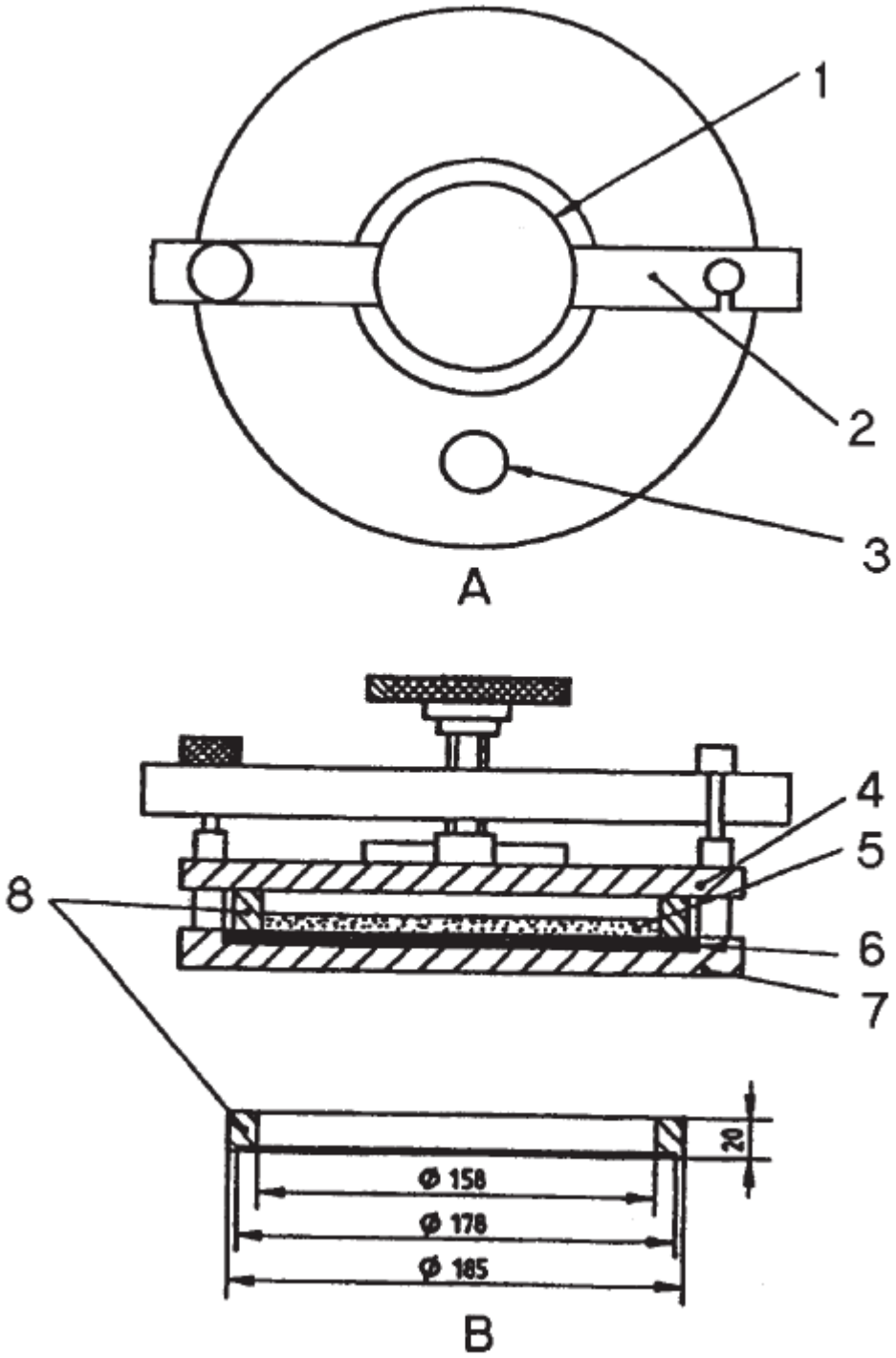


Figure 5 **Cell type A. Dimensions in mm. A, plan elevation; B, side elevation; 1, clamp screw; 2, clamp bar; 3, filler plug; 4, lid; 5, food simulant; 6, rubber mat; 7, base plate; 8, sealing ring**

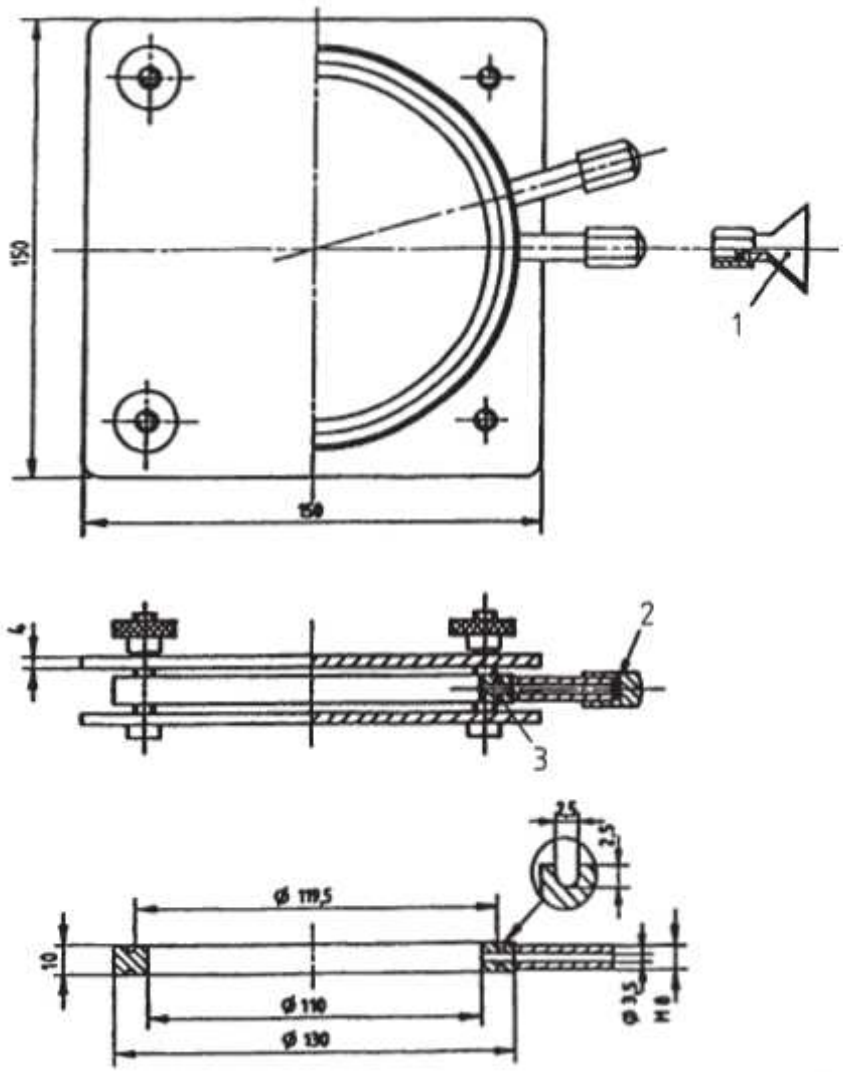
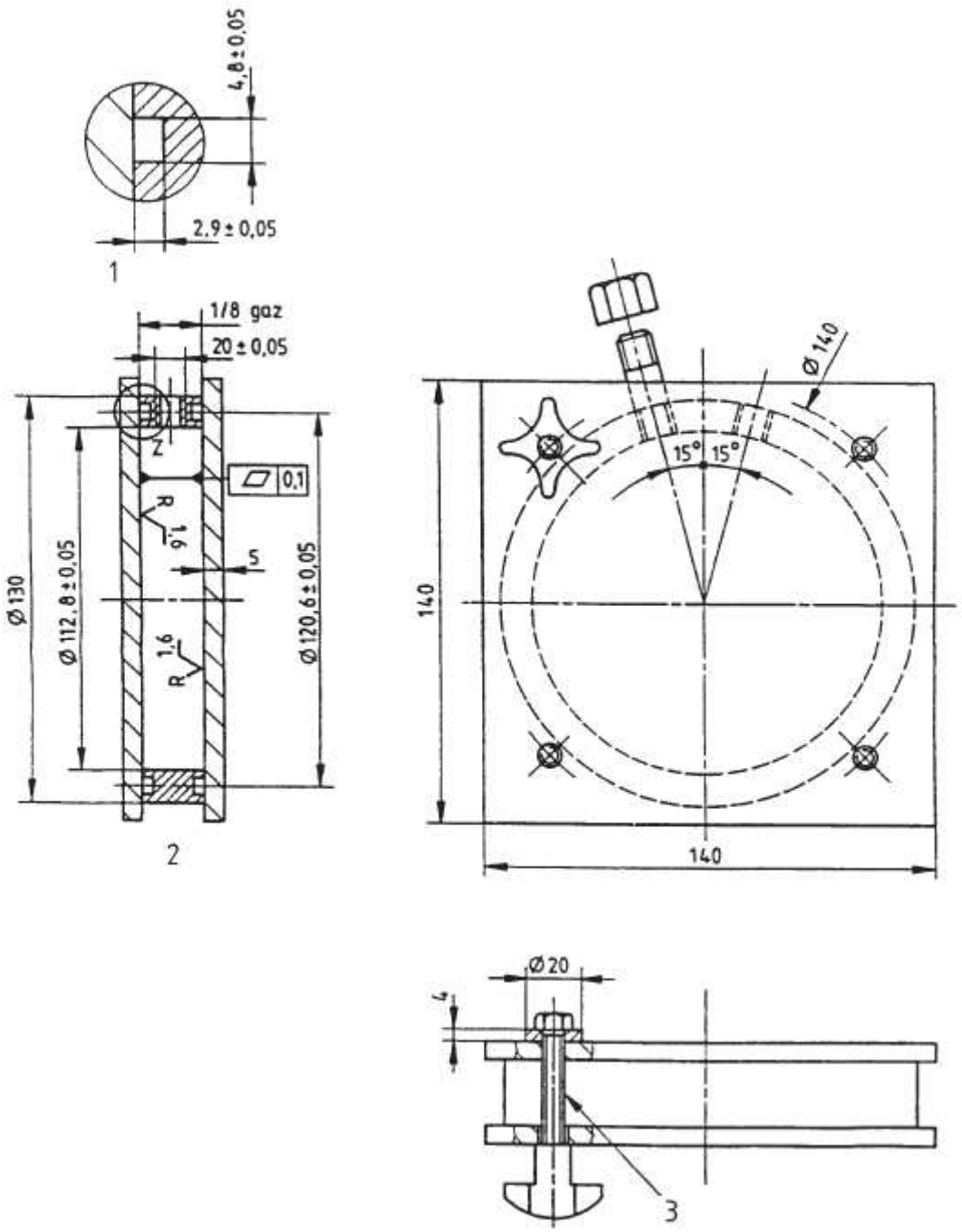


Figure 6 Cell type B. Dimensions in mm. 1, funnel for filling; 2, PTFE disk; 3, PTFE O-ring (119.5 x ∅ 3)



4349

4350 **Figure 7 Cell type C. Dimensions in mm. 1, detail of Z; 2, O-ring (∅ 117.07 x 124.13 x 3.53); 3,**
4351 **screw HM8-50**

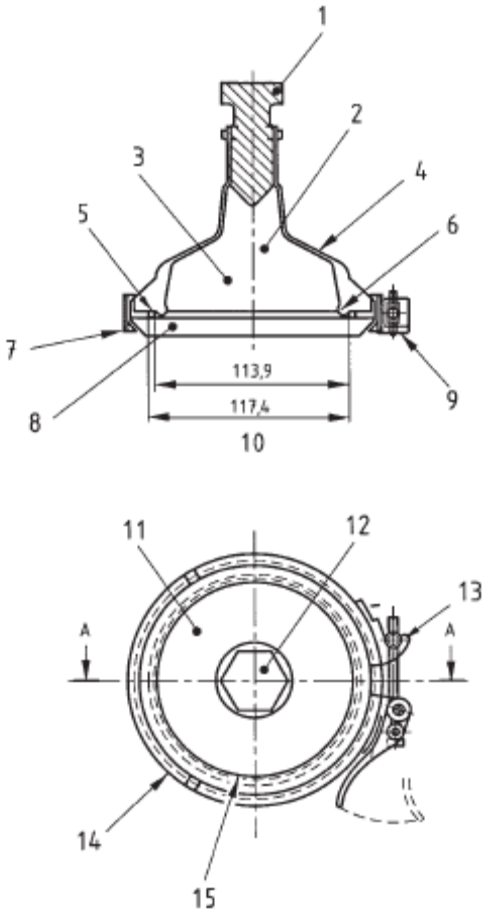
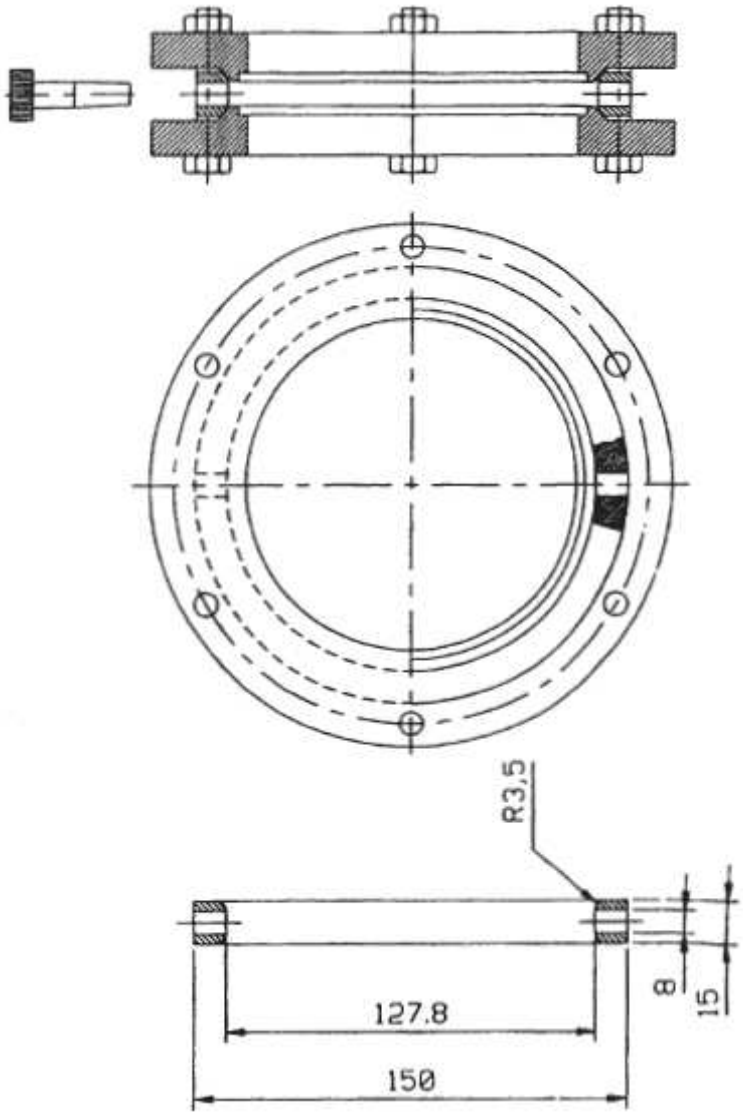


Figure 8 **Cell type D. Dimensions in mm. 1, glass stopper, 2, total inner volume of 296 ml (maximum volume of simulant: 250 ml); 3, exposed surface area of circular test specimens of 1,019 dm^2; 4, glass bell; 5, sealing O-ring (silicon rubber sheathed in PTFE); 6, raised edge to fix the O-ring in place; 7, tension ring (stainless steel); 8, PTFE plate; 9, tensioning seal (stainless steel); 10, sectional view A-A; 11, glass bell; 12, glass stopper; 13, tensioning seal (stainless steel); 14, tension ring (stainless steel); 15, sealing ring**



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4361 **Figure 9 Cell type E. Dimensions in mm.**

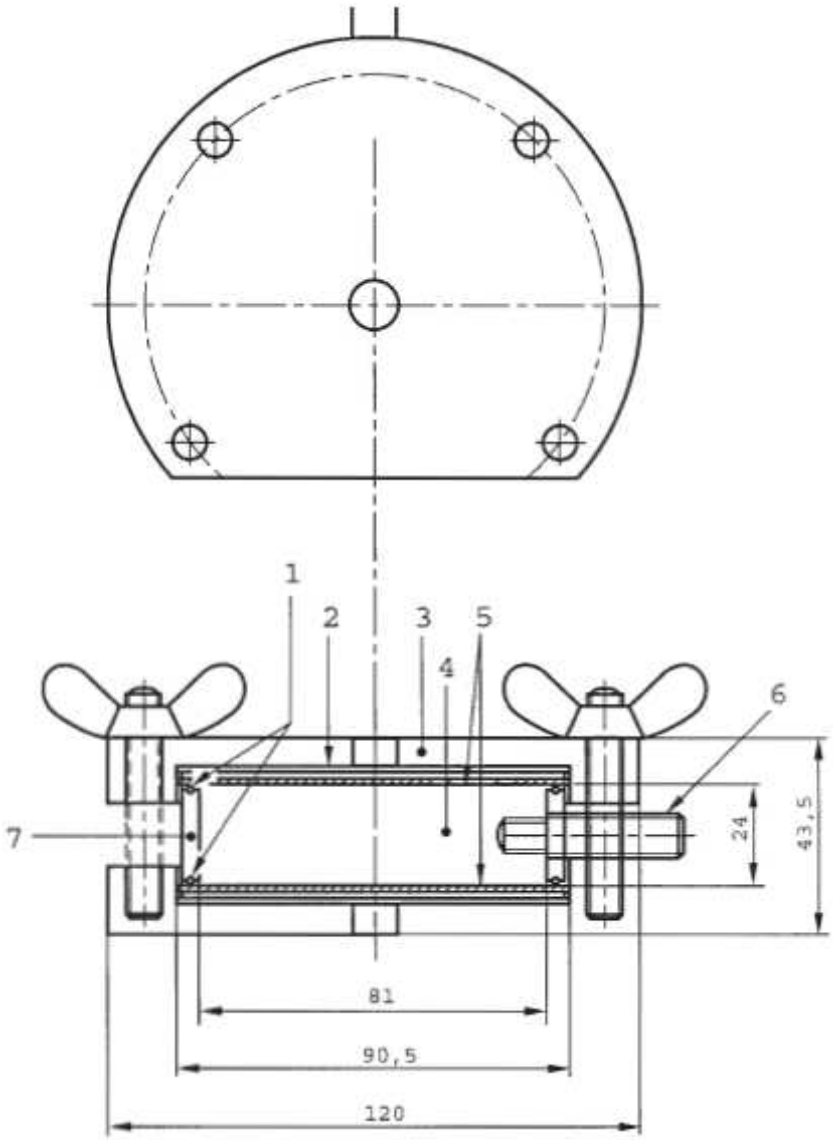


Figure 10 Cell type F. Dimensions in mm. 1, sealing ring; 2, lid (stainless steel); 3, body (aluminium); 4, food simulant; 5, test sample; 6, stopper (PTFE); 7, ring (stainless steel)

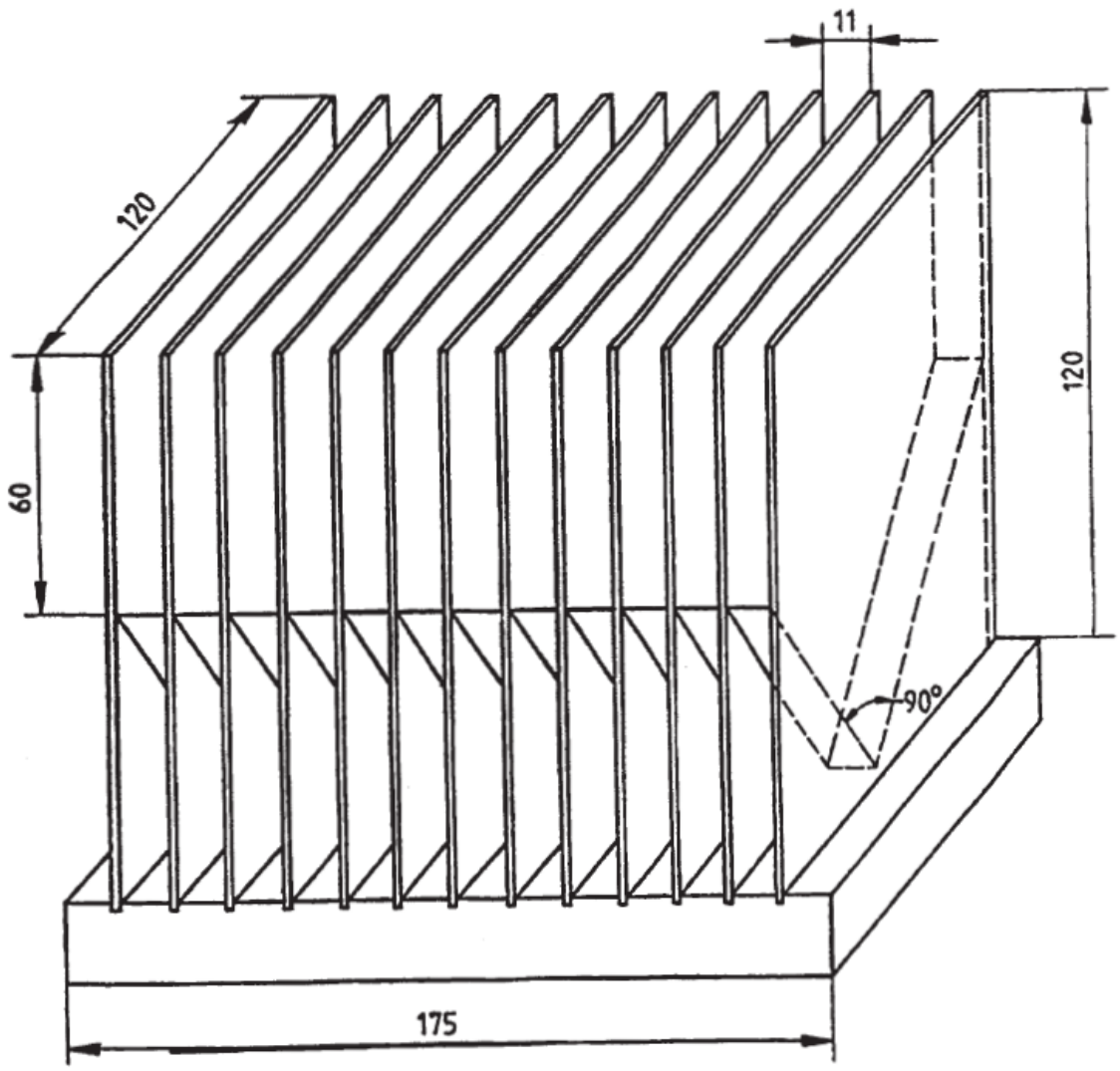
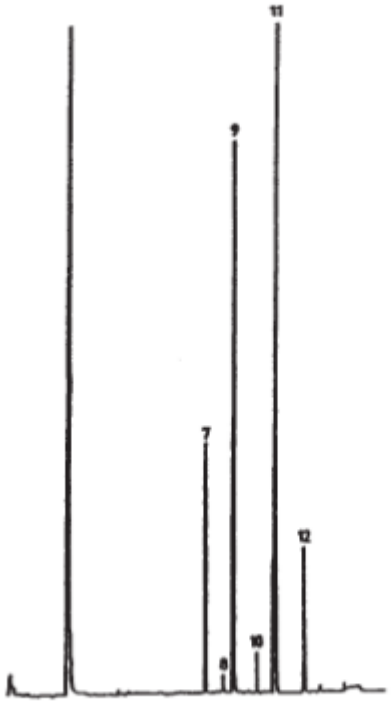
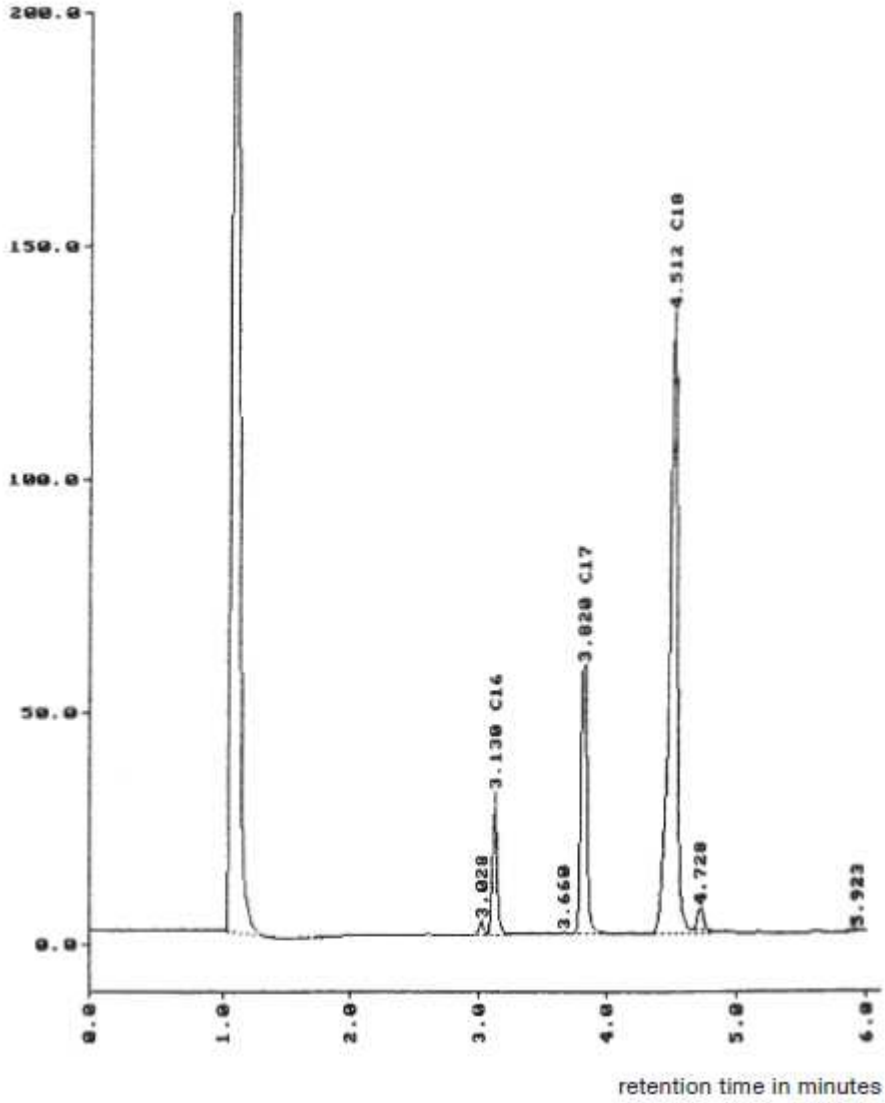


Figure 11 Pouch holder. Dimensions in mm



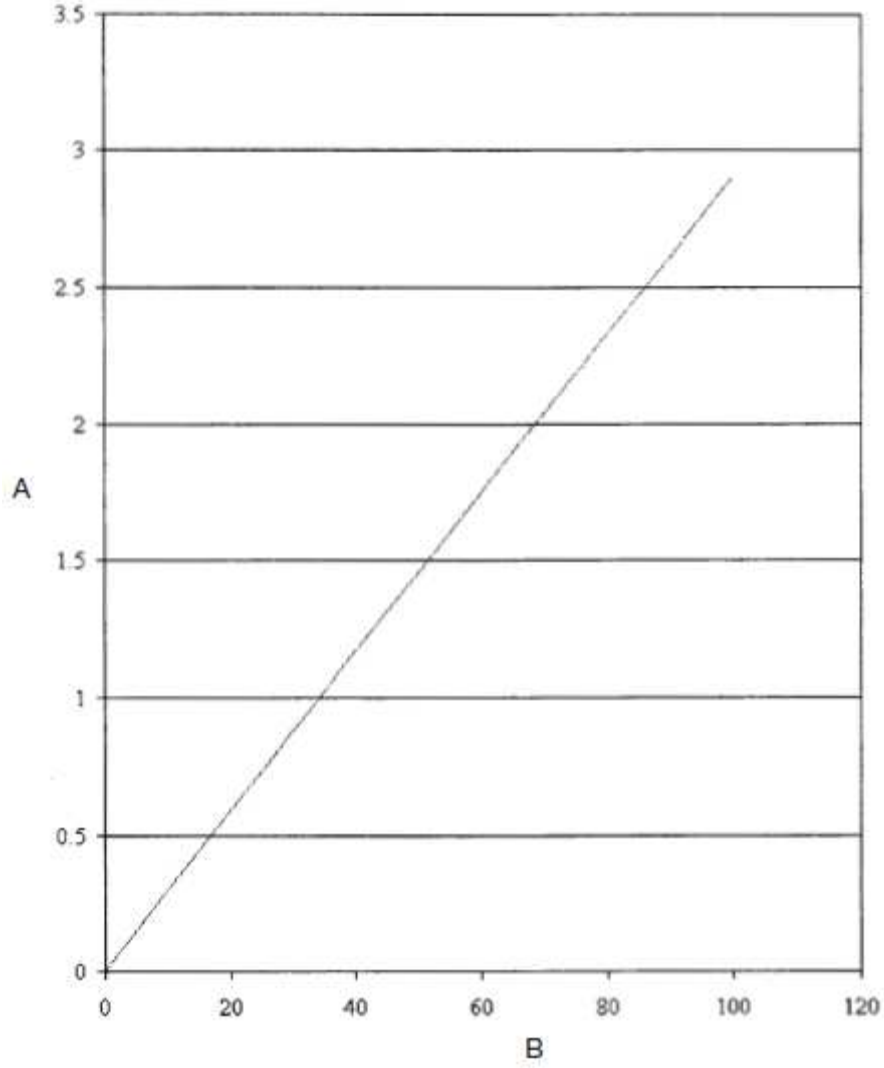
7 = C16:0 8 = C16:1 9 = C17:0 10 = C18:0 11 = C18:1 12 = C18:2

Figure 12 Typical chromatogram of column 1 (7.1.4.23)



4370

4371 **Figure 13 Typical chromatogram of column 2 (7.1.4.23)**



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4373 **Figure 14 Typical calibration graph. A, peak area ratio (C18 + C18)/C17; B, mg of olive oil**

4374

7.2 Test method for overall migration into vegetable oil in the temperature range of 5-20°C

7.2.1 Scope

The scope of this method is described in Annex 7.1.1. Only the deviations are mentioned here.

This method specifies test methods for the determination of the overall migration into vegetable oil from plastics materials and articles, at contact temperatures from 5°C up to and including 20°C.

The oil used in this test method is de-waxed sunflower oil since, unlike olive oil, this remains liquid at the lower test temperature.

7.2.2 Principle

The principle of this method is described in Annex 7.1.2. Only the deviations are mentioned here.

Test specimens of known mass are immersed in, filled with or put into contact to de-waxed sunflower oil for the contact time, at contact temperatures in the range of 5-20°C.

7.2.3 Reagents

The reagents shall be as described in Annex 7.1.3, except that olive oil is replaced by de-waxed sunflower oil.

7.2.4 Apparatus

The apparatus shall be as described in Annex 7.1.4.

7.2.5 Preparation of test specimens

Test specimens shall be prepared as described in Annex 7.1.5.

7.2.6 Procedure

Perform sections 7.1.6.1 to 7.1.6.4

Perform the contact with the de-waxed sunflower oil as described in section 7.1.6.5 or 7.1.6.6 followed by sections 7.1.6.7 and 7.1.6.8.

Determine the extracted de-waxed sunflower oil in accordance with Annex 7.1.6.9.

NOTE: The fatty acid pattern of de-waxed sunflower oil will show C18:2 as the major peak. In addition C18:1 will be present in significant amounts. C16:0 and C18:0 may be present in minor amounts. For quantification of the extracted amount of oil the peaks obtained for C18:1 and C18:2 may be used.

7.2.7 Expression of results

Follow section 7.1.7

7.2.8 Test report

Prepare the test report in accordance with Annex 7.1.8.

7.3 Test method for overall migration into vegetable oil in the temperature range of 100-175°C

7.3.1 Introduction

Migration testing with olive oil at high temperatures introduces a number of analytical difficulties. Experience has shown that it is difficult to achieve reproducible results owing to different laboratories having different equipment which give rise to variations in the time taken to reach the contact temperature. A method is described for determining overall migration by total immersion using an aluminium block with a consistent thermal capacity. Other analytical difficulties with olive oil include possible oxidation of oil at elevated temperatures and the hazard to personnel working with hot oil.

7.3.2 Scope

This method specifies a test method for the determination of the overall migration into fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at temperatures from 100°C up to and including, 175 °C for selected contact times. Another procedure, to be applied at temperatures >175°C, uses food simulant E, i.e. poly(2,6-diphenyl-p-phenylene oxide). In this procedure the mass of substances adsorbed on food simulant E is taken as a measure for the assessment of the overall migration into olive oil.

NOTE 1 The total immersion test method has been written for use with olive oil. The test method can also be used with appropriate modifications with other vegetable oils. These other vegetable oils produce different chromatograms for the simulant methyl esters to those of the methyl esters of olive oil. Suitable chromatogram peaks of the methyl esters of the other vegetable oils should be selected for the quantitative determination of the oil extracted from the test specimens.

NOTE 2: A comparative migration test carried out with polypropylene and polyethylene terephthalate high temperature application containers as test samples at conditions of 2 h at 100 °C and 2 h at 175 °C, respectively, in contact with ^{14}C-labelled synthetic triglyceride and food simulant E provided test results comparable within the analytical tolerance of the methods.

NOTE 3: To obtain reproducible and repeatable results using the food simulant E method, it may be necessary to measure the temperature of the test specimen before starting the migration period. An appropriate method for measuring the temperature of the test specimen needs to be established.

NOTE 4: The described method is written to determine the overall migration by total immersion using an aluminium block for consistent contact temperature and preheating of the simulant. However, when using an oven with forced air circulation (e.g. GC oven), samples may be tested by filling (article, pouch) or in a cell with oil at room temperature, or slightly preheated oil (e.g. 70°C). After bringing the test specimens in contact with the oil, they are placed in the oven and the temperature of the oil of one of the test specimen is recorded. As soon as the oil reaches the intended temperature then the contact time is started. At the end of the contact time the oil is removed immediately or allowed to cool down to a more safe temperature to handle the oil.

The described methods are most suitable for food contact articles in the form of sheets and films, but can also be applied to a wide range of articles and containers.

7.3.3 Method of total immersion in olive oil

7.3.3.1 Principle

The scope of this method is described in Annex 7.1.1. Only the deviations are mentioned here.

Test specimens of known mass are immersed in olive oil for the contact time, at contact temperatures from 100°C up to 175 °C.

7.3.3.2 Reagents

The reagents shall be as described in Annex 7.1.3

7.3.3.3 Apparatus

The apparatus shall be as described in Annex 7.1.4, with the addition of:

Aluminium block or blocks with wells for holding up to ten glass tubes (Annex 7.1.4.11) during the contact time period in the oven or incubator.

NOTE A diagram of a suitable block is shown in Figure 15. The wells in the block should hold the tubes so that there is close contact between the tubes and the block. The block should be of sufficient depth that when the test specimen is placed in the oil in the tubes, the level of the oil is lower than, or equal to the height of the block.

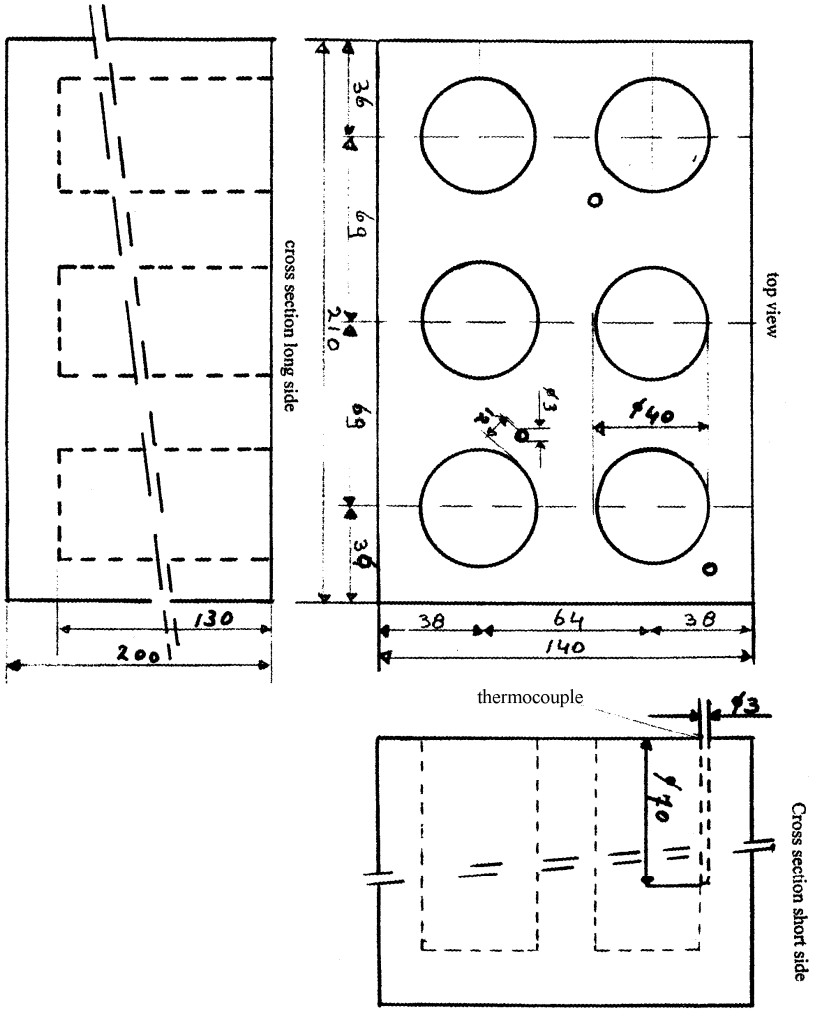

Figure 15 Aluminium block (dimensions in mm). 1) cross section long side, 2) top view, 3) thermocouple and 4) cross section short side.

7.3.3.4 Preparation of test specimens

Test specimens shall be prepared as described in Annex 7.1.5, except that an additional test specimen is required. This test specimen shall be placed in the tube in which the temperature is monitored.

7.3.3.5 Procedure

Perform sections 7.1.6.1 to 7.1.6.4

7.3.3.5.1 Contact with food simulant for single use

Insert a thermocouple in the metal block (Annex 7.3.3.3). Place the metal block and thermocouple in the thermostatically controlled oven or incubator, set at the test temperature and leave for 24 h.

Observe the temperature of the metal block. Confirm that the temperature of the metal block reaches the contact temperature, taking into account the tolerances specified in Table 5.

NOTE 1 If the oven temperature is at the contact temperature, but the temperature of the metal block is not, taking into account the tolerances specified in Table 5, the accuracy of the thermocouple and the temperature control of the oven should be checked. The oven temperature should be adjusted, if necessary, either by increasing or decreasing the oven set temperature, until the temperature of the metal block reaches the contact temperature.

Take six of the glass tubes (7.1.4.11), mark them for identification purposes. Measure 100 ml ± 5 ml of olive oil (7.1.3.1) into each tube by measuring cylinder and stopper the tube.

Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Place the tubes in the thermostatically controlled oven or incubator (7.1.4.12) set at the test contact temperature. Leave until the olive oil has attained the test contact temperature, using the thermometer or thermocouple to monitor the temperature.

NOTE 3 More rapid temperature equilibration of the oil can be established by lifting the tubes from the block periodically and rotating gently before replacing rapidly or by stirring the oil in the tubes with a metal or glass rod, without removing from the block.

Place into five of the tubes containing olive oil, weighed test specimens prepared as in 7.3.3.4 and condition if necessary. Place into one of the tubes a thermometer or thermocouple and stopper the tubes. Ensure that the test specimens are totally immersed in olive oil; if they are not, then add either glass beads or glass rods (Annex 7.1.4.22) to raise the level of the olive oil until total immersion is achieved.

WARNING 1 Take care when handling the hot metal block or blocks during removal from and replacement into the oven or incubator, to prevent skin burns. Take particular care when placing the test specimens in the hot olive oil to prevent splashing or spillage of the olive oil on to the skin. It is recommended that heat protective gloves and a face shield are worn.

NOTE 4 The olive oil in the sixth tube is used as a reference standard in constructing the calibration graph.

Place one test specimen in the sixth tube where the temperature of the oil us being monitored.

Replace the block or blocks containing all tubes in the thermostatically controlled oven or incubator set at the contact temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss.

NOTE 5 Where the contact time during the migration test is in the range of 0,5-4 h, it is particularly important that the time period between placing the test specimens in the olive oil and the olive oil reaching the contact temperature, be kept as short as possible. This time period can be minimized by placing the test specimens in the tubes without removing the tubes from the block and without removing the block from the oven or incubator. It can be necessary to raise the tubes from the block to check that the oil is above the level of the sample and that the test pieces remain separated. The tubes should be raised the minimum amount necessary to carry out these checks and then replaced as quickly as possible. All tubes should be treated in a similar manner.

Using a higher food simulant temperature is a risk for compliance testing but it can be applied for high test contact temperatures of ≥175°C, because the temperature drops very quickly below the 175°C after contact with the test specimen. Be aware that in such cases the temperature profile should be measured and the results should be added to your raw data.

Observe the temperature of the thermostatically controlled oven or incubator or the olive oil in the tube and leave the tubes for the selected test period, taking into account the tolerances specified in Table 4, after the olive oil in the tube has reached a temperature within the tolerance specified in Table 5. For test temperatures in the range of 100-150°C, the time between the immersion of the test specimens and the test temperature being regained shall be 10 min or less. For test temperatures in the range of 151-175 °C the time shall be 15 min or less.

NOTE 7 It has been found that these times can be achieved by using silicone oil in the wells to increase thermal conductivity between the block and the tube.

NOTE 8 Table 4 and Table 5 include tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this standard.

Take the metal block or blocks containing the tubes from the oven or incubator and immediately remove the test specimens from the tubes. Discard the test specimen that was in the tube in which the temperature had been monitored. For the remaining test specimens, which have been in olive oil, allow the oil to drain.

WARNING 2 Take care when handling the metal block or blocks and removing the test specimens from the olive oil at the end of the contact period, to prevent skin bums. Use the protective wear recommended in WARNING 1.

Remove any adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the pressing procedure until the filter paper shows no spots of olive oil. For test specimens on supports, remove the individual test pieces from the supports to carry out this operation. Clean the supports of oil by washing with the extraction solvent and replace the test pieces on them.

7.3.3.5.2 Contact with food simulant for repeated use

Perform the procedure as described in section 7.1.6.6 using section 7.3.3.5.1 in stead of section 7.1.6.5.

7.3.3.5.3

Perform sections 7.1.6.7. to 7.1.6.9.

7.3.3.6 Expression of results

7.3.3.6.1 Method of calculation

Perform sections 7.1.7.1 to 7.1.7.4.

7.3.3.7 Test report

Prepare the test report in accordance with Annex 7.1.8.

7.3.4 Test method of adsorption by poly(2,6-diphenyl-p-phenylene oxide)

7.3.4.1 Principle

The surface of the article to be tested is covered with poly(2,6-diphenyl-p-phenylene oxide) and is held at the selected contact time-temperature test conditions where the maximum temperature applicable is 225°C. If the material is used in a microwave oven the material can be tested in a conventional oven using EN 14233 (see footnote 1 on page 24 of main text). The migration is followed by extraction of the adsorbent using diethyl ether. Finally, the extract is evaporated to dryness using a nitrogen stream and the residue remaining is determined gravimetrically. poly(2,6-diphenyl-p-phenylene oxide) is a porous polymer with a high molecular mass in the range of 500,000-1,000,000 amu, a very high temperature stability (T_m = 350°C), a high specific surface area of 35 m^2/gram and a low specific mass (0.25 g/cm^3). Its pore volume is 2.4 cm^3/g and the average pore size is 200 nm.

7.3.4.2 Reagents

All reagents shall be of recognized analytical quality, unless otherwise specified.

7.3.4.2.1 Diethylether

99.8% purity and stabilized with 1.5-2,5% of ethanol.

7.3.4.2.2 Poly(2,6-diphenyl-p-phenylene oxide)

60-80 mesh; New poly(2,6-diphenyl-p-phenylene oxide) shall be cleaned following one of the procedures in Annex 3.

7.3.4.2.3 Nitrogen

purity 99.999%.

7.3.4.3 Apparatus

7.3.4.3.1 Cutting slab (Annex 7.1.4.1)

7.3.4.3.2 Cutting implement, scalpel, scissors, sharp knife or other suitable device (Annex 7.1.4.3), .

7.3.4.3.3 Rule (Annex 7.1.4.6)

7.3.4.3.4 Analytical balance (Annex 7.1.4.7)

7.3.4.3.5 Oven or incubator (Annex 7.1.4.12)

NOTE In case of an oven with a ventilating system the ventilation rate should be switched to low.

7.3.4.3.6 Petri dishes

Made of glass with an internal diameter of 140 mm; heat resistant.

7.3.4.3.7 Rings

Made of glass with an internal diameter of 125 mm and an external diameter of approximately 130 mm.

7.3.4.3.8 Glass-stoppered Erlenmeyer flasks

capacity of 300 ml.

NOTE The size of the flask that is used to wash poly(2,6-diphenyl-p-phenylene oxide) depends on the mass that is used (see Table 6).

7.3.4.3.9 Glass filter funnels,.

7.3.4.3.10 Folded filter

with a diameter of 125 mm.

7.3.4.3.11 Glass vials

with capacities of 10 ml and 100 ml.

7.3.4.3.12 Dropping pipettes with dropper teats.

7.3.4.3.13 Lint-free cloth (Annex 7.1.4.35)

7.3.4.3.14 Glass plates

to cover the dishes or trays.

7.3.4.3.15 Apparatus to blow off solvent with nitrogen

7.3.4.4 Preparation of test specimens

7.3.4.4.1 General

It is essential that test specimens are clean and free from surface contamination (many plastics can readily attract dust due to static charges). Before preparing test specimens, remove any

surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use (7.1.5.1). Minimize handling of the samples and where necessary, wear cotton gloves.

7.3.4.4.2 Number of test specimens

Three test specimens are required for the test.

7.3.4.4.3 Films and sheets

Lay the sample on a cutting slab (Annex 7.1.4.1). Take the glass ring (Annex 7.3.4.3.7) and place on the surface of the sample. Cut out the test specimen by cutting round the outside of the glass ring, using the cutting implement (Annex 7.1.4.3).

NOTE Taking the inner diameter of the glass ring into account the effective contact area obtained in this way is 1.22 dm^2.

7.3.4.4.4 Containers and other articles

Articles do not have to be cut if it is possible to cover these samples with pieces of glass. In this case, determine the flat bottom area of the article to find the required mass of adsorbent (see Annex 7.3.4.4.5).

7.3.4.4.5 Preparation of poly(2,6-diphenyl-p-phenylene oxide)

To cover the food contact surface sufficiently, 4 g of poly(2,6-diphenyl-p-phenylene oxide) per square decimetre of surface area of the test specimen is required.

7.3.4.5 Procedure

7.3.4.5.1 Contact with poly(2,6-diphenyl-p-phenylene oxide)

For flexible thin film and sheet materials (Annex 7.3.4.4.3), take four Petri dishes (Annex 7.3.4.3.6) and place a prepared test specimen into each dish. Stabilize the test specimen with a glass ring (Annex 7.3.4.3.7) and place 4.8 g poly(2,6-diphenyl-p-phenylene oxide) evenly on the surface of each test specimen, inside the glass ring. Close the Petri dishes.

For rigid containers and other articles (Annex7.3.4.4.4) which are to be tested as a whole, cover the flat bottom of the article with the required amount of poly(2,6-diphenyl-p-phenylene oxide). Calculate the required amount of poly(2,6-diphenyl-p-phenylene oxide) according to the flat bottom food contact surface area which can be covered (Annex 7.3.4.4.5). Weigh the appropriate mass of poly(2,6-diphenyl-p-phenylene oxide) with an accuracy of ± 0.1 g and place it on the flat bottom area of the test specimen. Cover each of the three articles with a glass plate (Annex 7.3.4.3.14).

NOTE 1 When the test sample, prepared as described, is placed in the oven the time required to reach the intended contact temperature can be significant when compared to the intended contact time and the allowed tolerances on contact time and contact temperature. Therefore it can be necessary to include a procedure for the control of the time and temperature of the contact of the test specimens with poly(2,6-diphenyl-p-phenylene oxide), in order to achieve reproducible and repeatable results.

NOTE 2 In the case of articles of irregular geometry and with no flat areas, a corresponding way needs to be found to expose the food contact surface to poly(2,6-diphenyl-p-phenylene oxide). Possible solutions are to cut appropriate parts from the article and cover or mix them with poly(2,6-diphenyl-p-phenylene oxide), using the conventional mass of poly(2,6-diphenyl-p-phenylene oxide) to food contact area ratio (Annex 7.3.4.4.5). If necessary, higher amounts of poly(2,6-diphenyl-p-phenylene oxide) should be used to ensure complete contact between the test specimen and poly(2,6-diphenyl-p-phenylene oxide).

For the blank determination, take an empty Petri dish and put in the same mass of poly(2,6-diphenyl-p-phenylene oxide) as was put on each test specimen, and cover the dish, Set the oven

(section 7.3.4.3.5) at the required test temperature and observe the temperature. When the oven has reached the test temperature place the blank and the four Petri dishes prepared with test specimens in the oven.

Observe the temperature and leave the test specimens for the selected period of contact time after the temperature of the oven has reached a temperature within the permitted tolerance for the test temperature, see Table 4 and Table 5 for tolerances on time and temperature.

Remove the test specimens from the oven and allow them to cool to room temperature without removing the glass covers.

NOTE 3 Cooling to room temperature takes approximately half an hour.

7.3.4.5.2 Determination of the migrating substances

Transfer the poly(2,6-diphenyl-p-phenylene oxide) into the Erlenmeyer flask (Annex 7.3.4.3.8) with the aid of a funnel (7.3.4.3.9). If necessary use a brush for complete transfer of the poly(2,6-diphenyl-p-phenylene oxide).

Calculate, by reference to Table 6, the volume of diethylether needed for extraction of the poly(2,6-diphenyl-p-phenylene oxide) and add this volume to the poly(2,6-diphenyl-p-phenylene oxide) in the Erlenmeyer flask.

Table 6 Volumes of diethylether needed for the extraction of poly(2,6-diphenyl-p-phenylene oxide) (PPPO)

Mass of PPPO	Extraction volume of diethylether		
	1st	2nd	3rd
g	ml	ml	ml
1.0	20	30	30
2.0	30	30	30
3.0	35	30	30
4.0	45	30	30
5.0	50	30	30
6.0	55	30	30
7.0	60	30	30
8.0	70	30	30
9.0	80	40	40
10.0	90	40	40
15.0	120	50	50
20.0	160	60	60

Pour the diethylether through the funnel (Annex 7.3.4.3.9) into the Erlenmeyer flask and shake it manually for 1 min. Allow the Erlenmeyer flask and its contents to stand for 1 min, without shaking.

Place a folded filter (Annex 7.3.4.3.10) into the funnel and insert the funnel into the 100 ml vial (Annex 7.3.4.3.11). Decant the diethylether from the Erlenmeyer flask through the filter into the vial.

Repeat this extraction procedure twice, using the diethylether volumes given in Table 1.

NOTE During the extraction of poly(2,6-diphenyl-p-phenylene oxide) the solvent is decanted from the extract at each washing step. Considerable amounts of solvent remain on the

poly(2,6-diphenyl-p-phenylene oxide) after each washing step which allows the use of a 100 ml volume capacity vial. If necessary, a second 100 ml vial should be used.

Rinse the filter with 10 ml diethylether and concentrate the combined diethylether solutions to approximately 5 ml, first by using a rotary evaporator and finally with the aid of a gentle nitrogen flow (Annex 7.3.4.3.15).

Weigh, with an accuracy of ±0.1 mg, a 10 ml vial (Annex 7.3.4.3.11) for each test specimen and for the blank determination.

Transfer each of the concentrated extracts, quantitatively, into the prepared 10 ml vials using a dropping pipette (Annex 7.3.4.3.12) and include a rinsing step using 5 ml of diethylether.

Evaporate the concentrates to dryness, using a stream of nitrogen, until constant mass has been achieved in the following way; evaporate to dryness (which takes approximately 30 min.) and remove the condensed water formed at the outside of the glass vial. Under a stream of nitrogen monitor the mass of the residue remaining in the glass vial every 5 minutes. Constant mass has been achieved when, after the second weighing, the mass difference is equal to or smaller than 0.5 mg.

Determine the mass of residue by subtracting the original mass of the vial from the stable mass of the vial and residue.

7.3.4.6 Expression of results

7.3.4.6.1 Method of calculation

Express the amount of material adsorbed onto poly(2,6-diphenyl-p-phenylene oxide) as milligrams lost per square decimetre of the test specimen taking only that area into account which was covered by the poly(2,6-diphenyl-p-phenylene oxide). Calculate the adsorbed amount, M, for each specimen according to the following formula:

$$M = \frac{m_a - m_b}{S}$$

where

M is the mass of migrated substances adsorbed onto poly(2,6-diphenyl-p-phenylene oxide) from the test specimen, in milligrams per square decimetre;

m_a is the mass of residue from the poly(2,6-diphenyl-p-phenylene oxide) that had been in contact with the test specimen, in milligrams;

m_b is the mass of residue from the poly(2,6-diphenyl-p-phenylene oxide) that had not been in contact with the test specimen, in milligrams;

S is the surface area of the test specimen that was in contact with the poly(2,6-diphenyl-p-phenylene oxide), in square decimetres.

Calculate the result for each test specimen to the nearest 0.1 mg/dm^2..

7.3.4.6.2 Validity of results

See section 7.1.7.3.

7.3.4.6.3 Precision

No data available.

From a large number of tests, the described procedure provided a maximum repeatability value r = 1 mg/dm^2.

7.3.4.7 Test report

Prepare the test report in accordance with Annex 7.1.8.

7.4 Test method for overall migration into vegetable oil in case of incomplete extraction of vegetable oil in the temperature range of 5-175°C

7.4.1 Scope

This method specifies tests for the determination of the overall migration into fatty food simulants from plastics materials and articles, by total immersion of test specimens in a fatty food simulant at any temperatures above 5°C up to and including 175°C for selected contact times.

When some plastics are tested by the methods in Annex 1, the soxhlet extraction process does not achieve complete recovery of the absorbed olive oil from the test specimens. In this method, the olive oil is released from the plastic test specimens by dissolving them in chloroform, toluene, xylene or tetrahydrofuran.

This method is suitable for plastics that come in contact with olive oil by total immersion as described in Annex 1, 7.2 and 7.3.

This is provided the plastics are soluble in chloroform, toluene, xylene or tetrahydrofuran and insoluble in methanol.

The method can also be suitable for plastics which are only partially soluble in chloroform, toluene, xylene or tetrahydrofuran and insoluble in methanol.

NOTE 1 This test method has been written for use with olive oil. The test method can also be used with appropriate modifications with other vegetable oils, i.e. sunflower oil and corn oil. These other vegetable oils will produce different chromatograms for the methyl esters of the relevant oil compared to those of the methyl esters of olive oil. Select suitable chromatogram peaks of the methyl esters of the other vegetable oils for the quantitative determination of the oil extracted from the test specimens.

NOTE 2 If it has been established that the overall migration into olive oil from the plastics cannot be determined by use of either this method or the methods described in Annex 1 then the use of tests in section 4.2.5 should be considered.

7.4.2 Principle

The principle is described in Annex 7.1.2. Only the deviations are mentioned here.

Test specimens of known mass are exposed to olive oil for the contact time, at contact temperatures varying from 5°C to 175°C, and then taken from the olive oil, blotted to remove oil adhering to the surface, and reweighed.

The absorbed olive oil is extracted by a dissolution and precipitation procedure.

In case the plastic does not dissolve completely, swelling of the plastic in the solvent should be such that the olive oil absorbed can be released from the plastic.

Depending on the type of plastic an appropriate organic solvent is selected in order to dissolve or swell the plastic.

For chloroform soluble plastics like polystyrene and polycarbonate, chloroform is used to release the olive oil absorbed.

For polyolefins, toluene and xylene are used. Low density polyethylene shows good solubility in toluene and high density polyethylene and polypropylene dissolve or swell sufficiently in xylene.

For polyvinylchloride or polyvinylidene chloride, tetrahydrofuran can be applied.

7.4.3 Reagents

The reagents shall be as described in Annex 7.1.3, except that the extraction solvent (Annex 7.1.3.2) is not required and the following reagents are added to the list:

a) chloroform

b) methanol

c) tetrahydrofuran

d) toluene

e) xylene

7.4.4 Apparatus

The apparatus shall be as described in Annex 7.1.4, with the exception of the soxhlet extractors (Annex 7.1.4.15) which are not required and the addition of the following:

a) centrifuge

b) centrifuge tubes 150 ml

c) conical tunnels, 100 mm diameter

d) filter papers, 185 mm diameter

7.4.5 Preparation of test specimens

Prepare the test specimens in accordance with Annex 7.1.5.

7.4.6 Procedure

7.4.6.1 Determination of the suitability of vegetable oil for overall migration

Follow section 7.1.6.1.1

7.4.6.1.1 Chromatogram of test specimen extract

Take one of the test specimens, prepared in Annex 7.4.5, and place it in a 250 ml round bottom flask and add 10 ml of cyclohexane without the internal standard. Add to the flask by measuring cylinder 50-60 ml of chloroform (7.4.3.a), tetrahydrofuran (7.4.3.c), toluene (7.4.3.d) or xylene (7.4.3.e) and a few anti-bumping beads to control boiling.

Couple the flask to a condenser. Place on either a water bath or steam bath and reflux for 30 min (0/+5 min). Slowly add by measuring cylinder or syringe, 10±0.2 ml of the potassium hydroxide down the condenser and continue refluxing for 15-20 min. Add by measuring cylinder at least 50±2 ml of methanol and continue refluxing for 5-6 min.

Remove the flask from the water bath and allow cooling. Transfer the solution from the flask to a 150 ml centrifuge tube (7.4.4.b), washing out the flask with 5-10 ml of methanol into the tube. Centrifuge the solution for 20 min (0/+5 min), at 2000-2500 rpm. Filter the supernatant solution through a filter paper into a 250 ml flask.

Evaporate the solution to 15-20 ml, either using a rotary evaporator or simple distillation apparatus. Transfer the solution to a 50 ml round bottom flask, washing out with 5-7 ml of methanol, and add a few anti-bumping beads. Evaporate the solution to dryness on a water bath.

Subject the extracted material to the methyl ester preparation procedure, described in Annex 7.1.6.1.1.1, but substituting the 10±0.2 ml of the potassium hydroxide solution with 10±0.2 ml of methanol (4.b).

Inject the same volume of resulting solution as used in section 7.1.6.1.1 into the gas chromatograph. Retain the chromatogram,

7.4.6.1.2 Comparison of chromatograms

Draw the conclusions in accordance with section 7.1.6.1.3.

7.4.6.2

Follow sections 7.1.6.2 to 7.1.6.7

7.4.6.3 Extraction of absorbed olive oil

Take four flasks, 250 ml, to be used for the extraction, and place in each flask 10.0 ml of the internal standard cyclohexane solution of triheptadecanoin (7.1.3.3), using a pipette (7.1.4.21), or an alternative higher quantity if more than 100 mg of olive oil is present.

NOTE 1 If the test specimens have retained more than 100 mg of olive oil, 10.0 ml of the internal standard solution is not sufficient for optimum precision in the gas chromatography determination after extraction. Before commencing the operations in this clause an estimation of the quantity of olive oil retained in the test specimens should be obtained by comparing the final masses of the test specimens with their initial masses. If considered necessary the quantity of internal standard solution can be increased from 10 ml although it is essential that the same quantity is used for each test specimen and that this quantity is also used with the olive oil standards for the calibration graph. As a guide, approximately 0.5 mg of the internal standard is required for every milligram of extracted olive oil.

Place the four test specimens in the flasks and add to each flask, by measuring cylinder, 50-60 ml of chloroform (7.4.3.a), tetrahydrofuran (7.4.3.c), toluene (7.4.3.d) or xytene (7.4.3e) and a few anti-bump beads to control boiling. Carefully remove the test specimens from the supports using tweezers. If necessary, carefully cut the test specimens in pieces of approximately 2*2 cm. Wash the tweezers and supports with 50-60 ml of the relevant solvent and transfer the washings to the flask. Couple each flask to a condenser, place on either a water bath or a steam bath and reflux for 30 min. Add slowly down the condenser, by measuring cylinder or syringe, 10±0.2 ml of the potassium hydroxide solution (7.1.3.4) and continue refluxing for 15-20 min. Add by measuring cylinder at least 50±2 ml of methanol slowly down the condenser and continue refluxing for 5-6 min.

NOTE 2 In such cases that the mass of the test sample is relatively high then the amount of the dissolving and precipitating solvent might need to be adapted to the mass of the test sample. In all cases a solution with reasonable viscosity should be obtained.

Remove the flasks from the water bath and allow to cool. Transfer the solution from each flask to individual 150 ml centrifuge tubes (7.4.4.b), washing out each flask with 5-10 ml of methanol into the tube. Centrifuge each solution until a clear supernatant liquid is obtained. Filter the supernatant solutions through a filter paper into 250 ml round bottom flasks. Evaporate the solutions to 15-20 ml, either using a rotary evaporator or a simple distillation apparatus. Transfer the solutions to individual 50 ml round bottom flasks, washing out with 5-7 ml of methanol, add a few anti-bumping beads. Evaporate each solution to dryness on a water bath.

NOTE 3 Oxidation of the olive oil should be avoided where possible. Therefore evaporation of the solvent to dryness should be carried out under mild conditions of temperature. In addition contact of the olive oil with oxygen should be limited.

7.4.6.4 Determination of extracted olive oil

7.4.6.4.1 Preparation of fatty acid methyl esters

Add 10±0.2 ml of n-heptane to each of the 50 ml flasks containing the dry residue as obtained in the previous section 7.4.6.3 by measuring cylinder (7.1.4.20), ensuring that the residues of olive oil and plastic extractables dissolve or are well dispersed by shaking, warming or by ultrasonic treatment.

NOTE 1 Unless the residues in the flasks are dissolved or well dispersed in the n-heptane, quantitative hydrolysis or methylation of the olive oil and of the internal standard might not be obtained under the conditions described particularly where these residues contain extractables from plastics in excess of 50 mg. The internal standard might not react with the plastic extractables to the same degree as does the olive oil and correct results for olive oil might not be obtained.

Add by measuring cylinder or graduated syringe, 10±0.2 ml of methanol (7.4.3.b) and a few anti-bumping beads (7.1.4.14). Connect a condenser to the flask and boil the mixture under reflux for 10±1.0 min.

Add through the condenser by measuring cylinder, or graduated syringe, 5.0±0.2 ml of the methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2±0.25 min.

Cool to room temperature and add, by measuring cylinder, 15-20 ml of saturated sodium sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulphate solution until the liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the upper, n-heptane, layer.

If there is a delay of more than 7 days in using a methyl ester solution for the gas chromatographic determinations, transfer the n-heptane layer to a small stoppered tube (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

7.4.6.4.2 Determination of fatty acid methyl esters

Determine the fatty acid methyl esters in accordance with Annex 7.1.6.9.2.

7.4.7 **Expression of results**

Perform section 7.1.7.1 to 7.1.7.3.

7.4.7.1 Precision

No data

7.4.8 **Test report**

Prepare the test report in accordance to Annex 7.1.8.

Annex 8 Test method for overall migration into water, aqueous food simulants, isooctane and ethanol 95%

Content

8.1 Scope

This section describes test methods for the determination of the overall migration into water, aqueous based food simulants, i.e. A, B, C and D1, and the organic solvents isooctane and ethanol 95% from plastics which are intended to come into contact with foodstuffs. The method describes four different ways to perform the migration, i.e. immersion, filling, pouch forming and filling and cell for one-sided contact.

The immersion method is most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers from which test pieces of suitable size can be cut. The test contact temperature can go up to reflux conditions for water and aqueous food simulants.

The cell method is most suitable for plastics in the form of films and sheets, but is particularly applicable to those materials consisting of more than one layer or for surfaces that differ in their migration characteristics, which should be tested with the food simulant in contact only with the surface which is intended to come into contact with foodstuffs. The test contact temperature can go up to and including 60°C for organic solvents and 70°C for water and aqueous food simulants.

The pouch method is most suitable for plastics in the form of films and sheets which are sealable by heat or pressure. The test is particularly applicable to those materials consisting of more than one layer, which are tested with the food simulant in contact only with the surface which is intended to be in contact with the foodstuffs. The test contact temperature can go up to and including 60°C for organic solvents and 70°C for water and aqueous food simulants.

This filling method is most suitable for plastics in the form of containers and articles that can be filled. The test contact temperature can go up to and including 60°C for organic solvents and 70°C for water and aqueous food simulants.

8.2 Principle

The overall migration of non-volatile substances from a sample of the plastics is determined as the mass of non-volatile residue after evaporation of the solvent, i.e. aqueous food stimulant or organic solvent, following immersion.

The selection of the test conditions will be determined by the worst foreseeable conditions of use as described in Regulation (EU) No 10/2011.

Test specimens are exposed to the food simulant for the contact time at the test contact temperature. At the end of the test period, each test specimen is removed from the food simulant. The food simulant from each test specimen is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area of test specimen.

8.3 Reagents

Each of the food simulants below shall give a non-volatile residue of less than 5 mg/l, when evaporated to dryness and dried to constant mass at 105-110°C.

8.3.1 **Ethanol 10% (v/v) in aqueous solution (food simulant A)**

A solution is prepared by diluting 100 ml of acetic acid with distilled water to a volume of 1 l.

8.3.2 **Acetic acid 3% (w/v) in aqueous solution (food simulant B)**

A solution is prepared by diluting 30 g of acetic acid with distilled water to a volume of 1 l.

8.3.3 **Ethanol 20% (v/v) in aqueous solution (food simulant C)**

A solution is prepared by diluting 200 ml of acetic acid with distilled water to a volume of 1 l.

8.3.4 **Ethanol 50% (v/v) in aqueous solution (food simulant D1)**

A solution is prepared by diluting 500 ml of acetic acid with distilled water to a volume of 1 l.

8.3.5 **Ethanol**

purity 96 % (v/v) or greater. A 95 % (v/v) aqueous solution is prepared.

8.3.6 **Iso-octane, (2,2,4-trimethyl pentane)**

purity 98.5 % (v/v) or greater, CAS No. 540-84-1.

WARNING — isooctane and ethanol are flammable. Take care at all times when handling these organic solvents to prevent contact with sources of ignition.

8.4 Apparatus

8.4.1 **Cutting slab**

Clean smooth glass, metal or plastics slab with a suitable area to prepare test specimens, 250 mm x 250 mm is suitable.

8.4.2 **Tweezers**

Stainless steel, blunt nosed.

8.4.3 **Cutting implement**

Scalpel, scissors or sharp knife or other suitable device

8.4.4 Metal template

(100 ± 0.2 mm) x (100 ± 0.2 mm) (square) or (120 ± 1 mm) x (120 ± 1 mm) (square)

8.4.5 Rule or template

25 ± 1 mm wide

8.4.6 Rule

Graduated in mm, and with an accuracy of 0.1 mm.

8.4.7 Analytical balance

Capable determining a change in mass of 0.1 mg.

8.4.8 Specimen supports

Constructed of stainless steel with cross arms attached by welding or silver soldering, or of glass. Stainless steel X4 CrNi 18 10 according to EN 10088-1:2014 or stailess steel having a composition of 17% chromium, 9% nickel and 0.04% carbon is suitable. Before initial use thoroughly clean the stainless steel supports. The use of a degreasing solvent and then dilute nitric acid has been found to be suitable. For the aqueous acetic acid food simulant, use supports constructed out of glass, as there is a tendency for the acetic acid to corrode stainless steel supports, particularly if the joints are silver soldered.

NOTE 1 However stainless steel supports can be used for acetic acid if it can be demonstrated that when immersed on their own in simulant, for the test period, at the test temperature, the residue after evaporating the simulant to dryness and drying in an oven or incubator or refrigerator to constant mass at 105-110°C is less than 5 mg/l.

NOTE 2 The method has been written for the supports shown in Figure 3 on page 223 which have been found to be suitable for holding thin film and sheet test pieces. However other supports can be used provided that they are capable of holding and keeping the test pieces apart and ensuring complete contact with the food simulant at the same time. For rigid samples, supports with a single cross arm may be used.

8.4.9 Gauze

Pieces of fine stainless steel gauze with a mesh size of 1 mm have been found to be suitable, approximately 25 mm x 100 mm or, glass rods, 2-3 mm in diameter and approximately 100 mm long to be used with the acetic acid food simulant, for insertion between the test pieces. Before initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric acid.

8.4.10 Glass tubes

Ground neck, for retaining the food simulant and test specimens. Tubes with an internal diameter of approximately 35 mm and length of between 100 mm and 200 mm, excluding the ground neck have been found to be satisfactory.

8.4.11 Glass beads

2-3 mm diameter or glass rods of 2-3 mm in diameter and approximately 100 mm long.

8.4.12 Thermostatically controlled oven or incubator or refrigerator

Capable of maintaining the set temperature within the tolerances specified in Table 5 on page 222.

WARNING — The interior/sample space of the oven, incubator or refrigerator should not have any exposed heating elements, to minimise safety hazards arising from any loss of flammable test media from the tubes during the test period.

8.4.13 Dishes

made of stainless steel, nickel, platinum, platinum alloy or gold, 50-90 mm diameter and a maximum mass of 100 g, for evaporation of food simulants and weighing of residues. Glass, glass ceramic or ceramic dishes may be used provided that the surface characteristics are such that the masses of the dishes after evaporation of any specified food simulants followed by conditioning in the desiccator used achieves a constancy of ± 0.5 mg. Stainless steel and nickel dishes are suitable only for aqueous ethanol solutions. Glass, glass ceramic, glazed ceramic, platinum or, platinum alloy or gold dishes are suitable for all food simulants.

8.4.14 Steam bath, hot plate, distillation apparatus or rotary evaporator

For evaporation of food simulant at the end of test period.

8.4.15 Desiccator

Filled with anhydrous calcium chloride or self indicating silica gel.

8.4.16 Measuring cylinder

100 ml, complying with the minimum requirements of ISO 4788:2005.

8.4.17 flasks, 250 ml

Suitable for attaching to reflux condensers

8.4.18 condensers to fit the flasks

8.4.19 heating mantle

for maintaining the food simulants at reflux temperature during contact

8.4.20 glass filter of porosity G1

8.4.21 Cells

type A as shown in Figure 5 on page 225, either the all aluminium (anodized) cells or the cells with the stainless steel (316 grade) lids and rings, are suitable for water and aqueous ethanol food simulants. For the aqueous acetic acid food simulant the cells with the stainless steel lids and rings are used. The internal diameter of the rim of the sealing ring shall be 178.4 ± 0.1 mm, to give an area of the test specimen exposed to the food simulant of 2.5 dm2.

NOTE The cells, type A, are constructed with a rubber mat in the base plate. When using the cells with either the water or aqueous ethanol food simulants it is advised that a disc of aluminium foil is placed on the mat before inserting the test specimen. For the aqueous acetic acid food simulant a disc of polytetrafluoroethylene or other suitable material which is inert to acetic acid can be used. The use of these discs will prevent any substances from the mat influencing the migration result.

For details of equivalent cells see Figure 6 to Figure **10**.

8.4.22 Pipettes

Having volumes of 50, 100 and 200 ml, complying with the minimum requirements of ISO 648:2008.

8.4.23 Measuring cylinders

250 ml, complying with the minimum requirements of ISO 4788:2005.

8.4.24 Metal template

(120 ± 1 mm) x (120 ± 1 mm) (square).

8.4.25 Pouch holder

the example shown in Figure 11 has been shown to be suitable, constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

8.4.26 Heat or pressure sealing device

for use in forming pouches.

8.4.27 Lint-free cloth or soft brush.

8.4.28 Beaker

250 ml and 2 l.

8.5 Preparation of test specimens

8.5.1 General

It is essential that test specimens are clean and free from surface contamination, because many plastics can readily attract dust due to static charges. Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush (8.4.27). Under no circumstances wash the sample with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use see (7.1.5.1). Minimise handling of the samples and where necessary, wear cotton gloves.

To ensure that test pieces are well separated and that the surfaces are freely exposed to the food simulant during the period of the test, for thin films, insert a piece of fine stainless steel gauze, or glass rods with the acetic acid simulant, between the test pieces or for thick samples not placed on the supports, insert glass rods between the test pieces after immersion in the food simulant. Where specimen supports are used, label the supports with a tag bearing the test specimen identification.

When preparing test specimens measure the surface area according to 7.1.5.1.1.

8.5.2 Number of test specimens

Three test specimens are required for samples, in the form of thin films, sheets, cut sections from containers or similar articles. Five test specimens, similar dimensionally one to another, are required for fillable samples or samples of articles of irregular shape. These test specimens are utilized as follows:

a) three test specimens for the migration test;

b) two test specimens for determination of the surface area, in the case of samples of irregular shape (8.5.5).

For articles with a nominal volume of more than 200 ml five articles are required to provide five test specimens. These test specimens are utilized as follows:

a) three test specimens for the migration test;

b) two test specimens for the determination of surface area.

For articles with a nominal volume of less than 200 ml one test specimen shall be made up of sufficient articles to contain a minimum of 200 ml of the food simulant. Record the number of articles used to provide one test specimen.

Five test specimens are required. These test specimens are utilized as follows:

a) three test specimens for the migration test;

b) two test specimens for the determination of surface area.

8.5.3 **Films and sheets**

8.5.3.1 Immersion

Lay the sample on the cutting slab (8.4.1) and cut the test specimens of 1 dm^2 (), using the 100 mm x 100 mm template (8.4.4). Check, using the rule (8.4.6), that the dimensions of the test specimen are within the specified tolerance (± 1 mm).

8.5.3.1.1 Immersion method using oven

Cut each test specimen into four test pieces 25 mm x 100 mm using the rule (8.4.5). Assemble one test specimen onto the support (8.4.8) by piercing suitable holes in the test pieces and placing two test pieces on each side of the cross arms of the support. Repeat this procedure for all remaining test specimens.

8.5.3.1.2 Immersion method using reflux

Cut each test specimen into sixteen test pieces 25 mm x 25 mm using the rule. Repeat this procedure for all remaining test specimens.

8.5.3.2 Cell

Lay the sample on the cutting slab (8.4.1) with the surface to be in contact with the food simulant uppermost. Take the ring from the standard cell (8.4.21) and place on the surface of the sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (8.4.3).

8.5.3.3 Pouch

Lay the sample on the cutting slab (8.4.1) with the surface to be in contact with the food simulant uppermost and cut the test specimens using the 120 mm x 120 mm template (8.4.24).

Place pairs of the test pieces together with the surfaces to be in contact with the food simulant uppermost. Using the heat or pressure sealer (8.4.26), join all four edges 10 mm from the edge to form pouches with four seals. Measure the distances between the inner edges of the seals to the nearest 1 mm and calculate the total surface area of the test specimen which will be exposed to the food simulant, to the nearest 0.01 dm^2. This shall be approximately 2 dm^2. Using the cutting implement (8.4.3), remove excess film from the sealed area (to reduce area of film not directly exposed to food simulant whilst leaving enough to withstand the test conditions without leaking.

Measure and record the surface area of the pouch which will be in contact with the food simulant and the total external area of the pouch after trimming excess material.

Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently large to insert a 100 ml pipette.

NOTE Pouches of dimensions other than 100 mm x 100 mm can be used for testing. These pouches should be prepared where possible so that the total surface area exposed is not less than 1 dm^2.

8.5.4 Containers and other articles

8.5.4.1 Immersion

Cut sections from the walls of the container or article to give test specimens each of area approximately 1 dm^2. For articles with individual areas less than 1 dm^2, use a number of articles to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1 mm, using the rule. Calculate only the surface area of the sample which is intended to come into contact with foodstuffs. Calculate the area of each test specimen to the nearest 0.01 dm^2 and record.

8.5.4.1.1 Immersion method using oven

If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (8.4.10). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

8.5.4.1.2 Immersion method using reflux

Cut each test specimen into test pieces 25 mm x 25 mm.

8.5.4.2 Filling

Determine and record the volume of food simulant required to fill an article to its nominal foodstuff volume. If the nominal volume of foodstuff to fill the article is not known, determine the surface area that will be in contact with the food simulant when filled to 5 mm from the top of the test specimen.

Next determine and record the surface area of the test specimen which is intended to come into contact with its nominal volume of foodstuff.

NOTE 2 In the case of articles with a volume of less than 200 ml this will be the surface area of one article multiplied by the number of articles used to provide one test specimen.

8.5.5 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give five dimensionally similar test specimens each with a known total surface area of at least 1 dm^2. Measure only the surface area intended to come into contact with foodstuffs of two of these test specimens to the nearest 0.05 dm^2 using e.g. the Schlegel Method, as described in Annex B of EN ISO 8442-2:1997, the methods described in Mieth and Hoekstra (2014) or any other suitable method. Record the surface area of each test specimen.

8.6 Procedure

8.6.1 Contact with food simulant

NOTE When testing some samples by single surface testing the small amounts of food simulant may permeate through the sample. For example, there may be a small loss of

ethanol when testing with high strength ethanol/water food simulants by filling. In this case, as the loss in ethanol from the food simulant may be expected to reflect what would happen under actual conditions of use of the alcoholic beverage, this loss may be disregarded. However, if permeation occurs when testing in a cell, care has to be taken to ensure contamination does not arise from contact of the food simulant with components of the cell.

8.6.1.1 Immersion method using oven

Take three glass tubes (8.4.10) for the test specimens and a further two to provide blanks, measure by measuring cylinder 100±2 ml of the food simulant into each tube and stopper the tube. Insert a thermometer or thermocouple, if applicable see NOTE 2, in one of the test specimen tubes, and stopper all five tubes. Place the five tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the food simulant has attained the test temperature.

NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so, then use a separate tube for measuring the temperature and an additional test specimen

Place a test specimen into three tubes, re-insert the thermometer or thermocouple, and stopper the tubes. Mark the tubes for identification. Ensure that the test specimens are totally immersed in the food simulant; if they are not then add either glass beads or rods to raise the level of the food 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 food simulant.

Mark the liquid level on the outside of each tube with a suitable marker.

Replace all of the tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature.

Observe the temperature and leave the tubes for the selected period of contact time after the temperature of the food simulant has reached a temperature within the permitted tolerance for the test temperature, see Table 4 and Table 5 for permitted tolerances on test times and temperature.

NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant.

WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take care to ensure that the tubes are well stoppered to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.

WARNING 2 — If possible place the tubes in a drip container capable of holding the total volume of organic solvent in case of accident.

WARNING 3 — To minimise hazards arising due to the volatile and flammable nature of the organic solvents the maximum test temperature is 60°C. Do not conduct the tests at temperatures above 60°C.

Take the tubes from the oven or incubator or refrigerator and check the level of food simulant If this level has fallen by more than 10 mm below the mark, or has exposed any part of the test pieces, repeat the test using fresh test specimens.

If the level of the food 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 repeat the test.

NOTE 2 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the food simulant.

NOTE 3 For short test contact times (≤6 h) the use of hot stirring plate heating controlled by a thermocouple may be a good alternative for an oven.

8.6.1.2 Immersion method using reflux

Take three flasks (8.4.17) for the test specimens and a further two to provide blanks, measure by measuring cylinder 100 ± 2 ml of food simulant into each flask.

Place the flasks in the heating mantle, connect the condensers.

Turn on the water supply to the condensers.

Switch on the heating mantle and heat the food simulant in each of the flasks to boiling. Turn off the heating, allow the flasks to cool for 2-3 min, remove the condensers from three flasks containing 100 ml of simulant and place a test specimen in each flask. Ensure that the test specimens are totally immersed in the food simulant.

WARNING: Hot fumes can emit from the flasks when the lids are removed.

Replace the condensers and switch on the heating mantles, and heat so that reflux is achieved within 5 min.

Observe the food simulant in the flask, following the onset of reflux, leave for the test time, taking into account the tolerances in Table 4. Turn off the heating mantle, turn off the water to the condenser and remove the flask from the mantle.

NOTE Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant.

To separate the food simulant from the test specimens, pour off the hot food simulant through a glass filter (8.4.20), collecting the filtrate in a clean container. Rinse each of the flasks and the test specimens in the flasks with two portions of 10 ± 1 ml of unused simulant and pour these washings through the filter.

WARNING: Danger hot flasks and contents.

8.6.1.3 Cell

Take three cells (8.4.21), mark these for identification purposes. Place in the thermostatically controlled oven or incubator or refrigerator (8.4.12), which is set at the selected test temperature and leave until the test temperature has been attained.

Take three glass tubes (8.4.10), for the food simulant for contact with the test specimens and a further two to provide blanks, measure by measuring cylinder 125 ± 2 ml of the food simulant into each tube. Insert a thermometer or thermocouple, if applicable, see NOTE 2, in one of the tubes and stopper the tubes. Mark the liquid level on the outside of each tube with a suitable marker.

NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so, then use a separate tube for measuring the temperature and use an additional test specimen and cell.

Place the tubes in the thermostatically controlled oven or incubator or refrigerator, set at the test temperature and leave until the food simulant has attained the test temperature.

Remove the cells from the thermostatically controlled oven or incubator or refrigerator, dismantle and place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the clamping screw wheel is well tightened down.

Remove three tubes containing 125 ml of the food simulant from the thermostatically controlled oven or incubator or refrigerator and transfer the food simulant from each tube to the cells through the filler hole. Remove the thermometer or thermocouple from the tube and, insert, if

applicable see NOTE 2, in one of the cells and replace the filler plugs. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the cells and food simulants. If any leakage is observed reject that cell from further tests.

WARNING 1 — Never place a leaking cell in the oven.

Replace the test cells in the thermostatically controlled oven or incubator or refrigerator, set at the test temperature.

Observe the temperature, leave the cells and the blank tubes for the selected period of time after the temperature of the food simulant in the cell has reached a temperature within the permitted tolerance for the test temperature, see Table 4 and Table 5 for permitted tolerances on test times and temperature.

NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant.

WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take care to ensure that the tubes are well stoppered and pouches are carefully closed at the corner to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.

WARNING 2 — For safety reasons do not load an oven with more than the test specimens of one test sample.

WARNING 3 — Place the pouches and tubes in a drip container capable of holding the total volume of organic solvent in case of an accident.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the organic solvents, the maximum test temperature is 60°C. Do not conduct the tests at temperatures above 60°C.

Take the cells and the two tubes containing the blank food simulant from the thermostatically controlled oven or incubator or refrigerator.

Transfer the solvent from each of the cells into the tubes, check the level of solvent in each, if this has fallen by more than 10 mm below the mark, repeat the test with fresh test pieces. Recover at least 90% of the original volume of food simulant or repeat the test.

Rinse each cell twice with 20 ± 2 ml of unused food simulant, add these rinses to the respective tubes. Add twice 20 ± 2 ml of food simulant to each of the two tubes containing blank simulant.

NOTE 2 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant.

8.6.1.4 Pouch

Take three glass tubes (8.4.10) for the food simulant for filling the pouches and a further two to provide the blanks, measure by measuring cylinder 100 ± 2 ml of the food simulant into each tube. Insert a thermometer or thermocouple, if applicable, see NOTE 3, in one of the tubes and stopper the tubes.

NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so, then use a separate tube for measuring the temperature and use an additional pouch.

Place the tubes and the pouch holder in the thermostatically controlled oven or incubator or refrigerator, set at the test temperature and leave until the test temperature has been attained.

Remove the pouch holder from the thermostatically controlled oven or incubator or refrigerator and place between the spacers the test specimen pouches.

Remove the tubes containing the 100 ml of food simulant from the thermostatically controlled oven or incubator or refrigerator. Pipette sufficient food simulant into three test specimen to just fill the pouch. This shall be about 100 ml, but for thick/semi-rigid materials the quantity will be less. Remove the thermometer or thermocouple from the tube and insert, if applicable, see NOTE 3, in one of the filled pouches. Secure the open corner of each pouch with a clip.

NOTE After filling, the corner of the pouch can be closed by heat sealing for aqueous food simulants.

If not all food simulant is used to fill the pouch, retain the tube and residual contents. Measure and record the volume of the residual food simulant. This part of the operation should be carried out in the minimum time to prevent undue heat loss. If any leakage is observed reject that pouch from further tests.

WARNING 1 — Never place a leaking pouch in the oven.

Replace the pouch holder, containing the test specimen pouches, in the thermostatically controlled oven or incubator or refrigerator, set at the test temperature. Observe the temperature and leave the pouches and blank tubes for the selected contact time after the temperature of the simulant has reached a temperature within the permitted tolerance for the test temperature, see Table 4 and Table 5 for permitted tolerances on test times and temperature.

NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant.

Take the pouch holder and the tubes containing the blank food simulant from the thermostatically controlled oven or incubator or refrigerator.

Examine the pouches for leaks, if at least 90% of original volume of food simulant is not recovered from each pouch, the test is invalid and shall be repeated using fresh pouches.

NOTE 2 For plastics that loose food simulant during the test period due to permeation through the plastic see .

NOTE 3 For contact times 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator or refrigerator, instead of the temperature of the simulant.

WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take care to ensure that the tubes are well stoppered and pouches are carefully closed at the corner to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator and generating an explosive mixture.

WARNING 2 — For safety reasons do not load an oven with more than the test specimens of one test sample.

WARNING 3 — Place the pouches and tubes in a drip container capable of holding the total volume of organic solvent in case of an accident.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the two solvents, the maximum test temperature is 60°C. Do not conduct the tests at temperatures above 60°C.

8.6.1.5 Filling

Mark three test specimen for identification, and where more than one article has been used for a test specimen, also mark individually.

Place, in a beaker, a sufficient volume of the food simulant to fill the three test specimens to the nominal volume or to 5 mm from the top if the nominal volume is not known. and to provide

two 200 ml blanks. Insert a thermometer or thermocouple, if applicable see NOTE 3, in the food simulant. Place the beaker in the thermostatically controlled oven or incubator or refrigerator set at the test temperature and leave until the food simulant has attained the test temperature.

NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so, then use separate beakers filled with food stimulant for each test specimen and one additional for measuring the temperature. Use an additional test specimen for measuring the temperature during the contact time.

Remove the beaker containing the food simulant from the thermostatically controlled oven or incubator or refrigerator. Fill the three test specimens with food simulant to the nominal volume of the article or to 0.5 cm from the top. Insert the thermometer or thermocouple in one of the test specimens containing food simulant, if applicable see NOTE 3. Cover the test specimens and the remaining food simulant with an inert material to prevent evaporation. This part of the operation should be carried out in the minimum time to prevent undue heat loss from the simulant.

WARNING 1 — Covering of articles filled with organic solvents is very important in respect to safety. Due to the variety of fillable articles it is impossible to prescribe one method for covering the filling opening. In general aluminium foil has appeared to be suitable in many cases. Also, a combination of glass plates with aluminium foil can be useful. Containers, like bottles and cups, are easily closed by carefully wrapping aluminium foil over the filling opening. Articles with large open areas, such as trays or dishes, should be covered with a glass plate of an appropriate size. The article and cover should then be placed on a sheet of aluminium foil, which is then folded around and over the article and glass plate. In this way a pouch is made that prevents evaporation of the organic solvent or reduces it to an acceptable level. To insert a thermocouple the glass plate should be provided with a hole that fits the thermocouple or that is large enough to accept a polytetrafluoroethylene stopper with the thermocouple.

WARNING 2— For safety reasons do not load an oven with more than the test specimens of one test sample. In case were the capacity of the article is large, then the test specimens should be placed in the oven one at a time.

WARNING 3 — If possible place the article in a drip container capable of holding the total volume of organic solvent in case of an accident.

WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the organic solvents, the maximum test temperature is 60°C. Do not conduct the tests at temperatures above 60°C.

Place the test specimens in the thermostatically controlled oven or incubator or refrigerator set at the test temperature. Observe the temperature and leave the test specimens and food simulant for the selected contact time after the temperature of the simulant has reached a temperature within the permitted tolerance for temperature, see Table 4 and Table 5 for permitted tolerances on test times and temperature.

NOTE 1 Where the surface of simulant is large, a check should be made to ensure that excessive loss of simulant by evaporation does not occur.

NOTE 2 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant.

Take the marked test specimens and beaker with blank food simulant from the thermostatically controlled oven or incubator or refrigerator.

NOTE 3 For contact times of 24 h or more it is acceptable to monitor the temperature of the air bath of the thermostatically controlled oven or incubator or refrigerator or refrigerator, instead of the temperature of the simulant.

8.6.2 Determination of migrating substances

WARNING — Both iso-octane and ethanol are volatile and flammable organic solvents. Take care when evaporating them to prevent vapours contacting sources of ignition, particularly when using a hot plate to carry out the evaporation. The evaporation should be carried out in a fume cupboard.

8.6.2.1 Preparation of dishes

Take five dishes (8.4.13), marked for identification, place the dishes in an oven maintained at 105-110 °C, for a period of 30 ± 5 min, to dry.

NOTE three dishes for the exposed food simulant and 2 dishes for the blank

Remove the dishes from the oven, place in a desiccator (8.4.15) 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, record their final masses.

8.6.2.2 Evaporation method

Take the tubes, pouches or filled test specimens containing the food simulant and pour 40-50 ml from each into separate dishes. By means of a steam bath, hot plate or other form of heating (8.4.14) evaporate to a low volume, taking care to avoid loss, in particular, by sputtering or overheating of the residues.

NOTE 1 The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

When most of the food simulant has evaporated, pour the remaining food simulant from each of the tubes into the respective dishes and continue the evaporation. Wash out each of the tubes, including the blank tubes with two lots of 10 ± 1 ml of unused simulant and pour these washings into the respective dishes. Continue the evaporation.

NOTE 2 A stream of nitrogen can be used to facilitate evaporation.

When the simulant has almost completely evaporated, place the dish in an oven maintained at 105-110 °C, for a period of 30 ± 5 mm, to complete the evaporation and dry the residue.

Remove the dishes from the oven, place in a desiccator (8.4.15) and allow 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 stable mass of the dish (8.6.2.1) from the stable mass of the dish and residue.

8.6.2.3 Distillation method

Transfer the food simulants to individual round bottom flasks (250 ml are suitable). Wash out each of the tubes, including the blank tubes, with two lots of 10 ± 1 ml of unused food 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 rotary evaporator. Distil off the food simulant until approximately 30-50 ml remains in each flask. Transfer the remaining food simulant from each flask to individual evaporating dishes (8.4.13). Rinse each flask with two lots of 10 ± 1 ml of fresh simulant and add the rinses to the appropriate dishes. Continue the evaporation of the food simulant by means of a steam bath, hot plate or other form of heating, proceeding as in 8.6.2.2.

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

8.6.3 Expression of results

8.6.3.1 Method of calculation

Express the overall migration as milligrams of residue per square 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:

$$M = \frac{(m_a - m_b) \cdot 1000}{S}$$

where

M is the overall migration into the simulant, in milligrams per square decimetre of 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 food simulant in which it had been immersed, in grams;

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

S is the surface area of the test specimen intended to come into contact with foodstuff, in square decimetres, see 7.1.5.1.1. In the case of articles with a nominal volume less than 200 ml, S is the surface area of one article multiplied by the amount of articles that constitutes the test specimen.

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.

8.6.3.2 Validity of results

The following analytical tolerances are allowed:

12 mg/kg or 2 mg/dm^2 for all aqueous food simulants.

The test result for each individual test specimen is valid if it differs from the mean of the triplicate test results by not more than the permitted analytical tolerance. If a minimum of three resuits is not within the analytical tolerance, then the test is repeated using fresh test specimens from the sample.

8.6.3.3 Precision

The precision data were determined for a polyamide sample under the test conditions of 24 h at 40 °C with water, 3% acetic acid and .

The precision data for water were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

The precision data for 3% acetic acid were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 10 laboratories and one sample.

The precision data for ethanol 10% were determined from the BSI/DTI trial conducted in 1991 (Pira Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

Table 7 Precision data (mg/dm^2)

	Level	Repeatability (r)	Reproducibility (R)
Water	6.9	1.3	2.6
3% acetic acid	10.7	1.1	2.3
Ethanol 10%	11.9	1.1	2.9

The difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval can exceed the repeatability value r on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on identical test material reported by two laboratories can differ by more than the reproducibility value R on average not more than once in 20 cases in the normal and correct operation of the method.

8.6.4 **Test report**

The test report shall include the following:

- reference to this method and to the part used for the test procedure;
- all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number, thicknesses;
- conditions of time and temperature of contact with food simulants;
- departures from the specified procedure, and reasons for these:
- individual test results, expressed as milligrams of residue per square decimetre of sample
- f) relevant comments on the test results such as test contact area and contact volume.

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