NRK Masterclass
Deel 1

Praktische implicaties van migratietesten

28/11/2018

Kris Callaert

Projectmanager kunststofverpakkingen en voedselcontact

NRK Verpakkingen

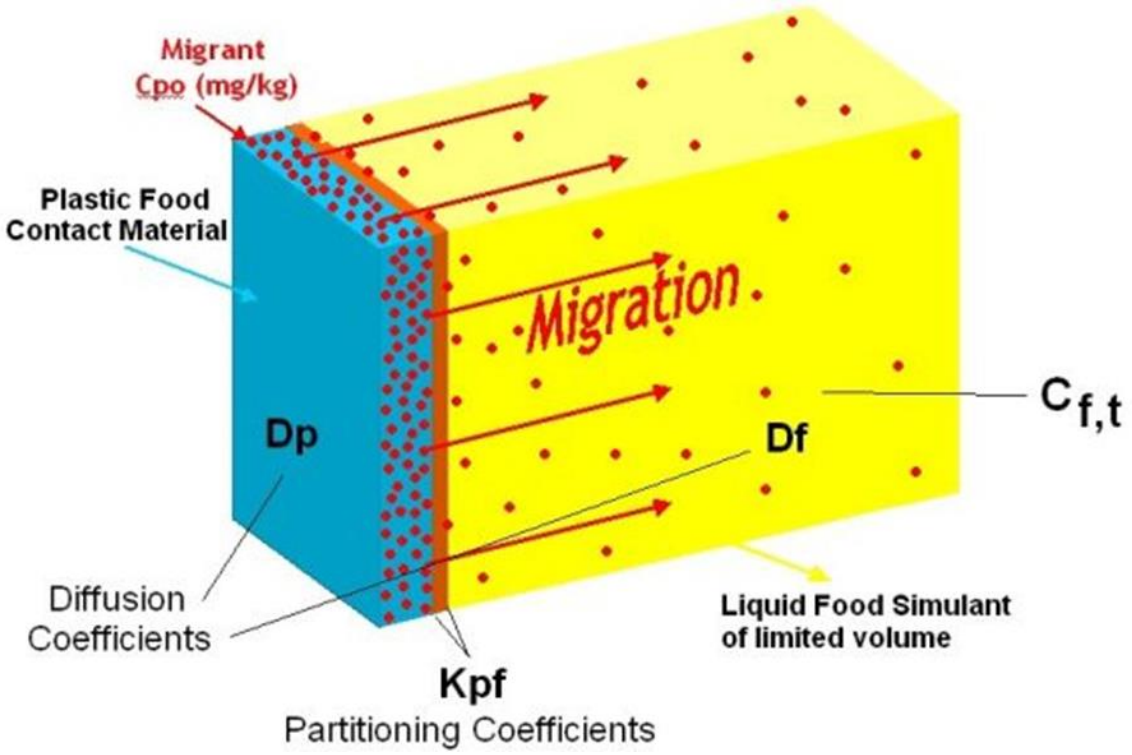
VIAWARE
FOOD CONTACT SAFETY

Praktische implicaties van migratietesten

De praktijk van migratietesten:

- Single use en repeated use
- De klasse van gebruikers
- Screeningtesten versus target testen
- Sample voorbereiding (ageing, set off)
- Onnauwkeurigheid meten, LOD, LOQ
- Het uitdrukken van de migratieresultaten
- Veranderingen in het guidance document
- De analyse voor een NIAS-assessment

Wat is migratie d.m.v. diffusie

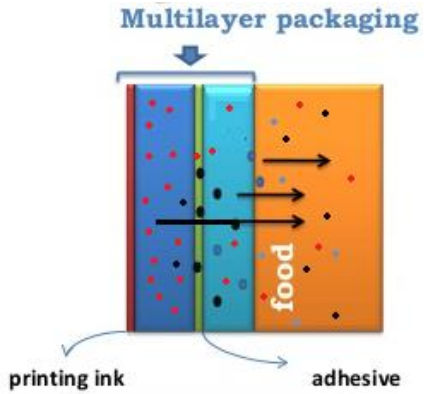


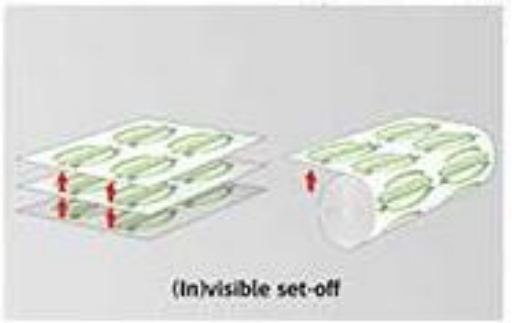
- Food contact material
- Type of food
- Chemical substance
- Initial concentration
- Temperature
- Time
- Surface/volume ratio
- Distribution

Mono layer – multi layer

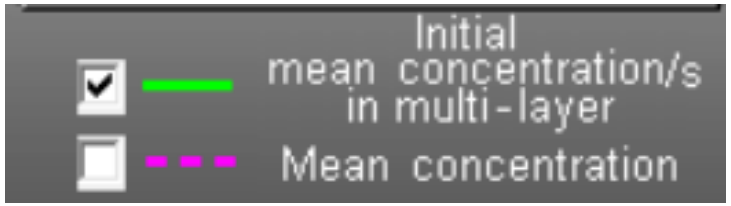
- Mono layer
  - Single side
  - Full immersion

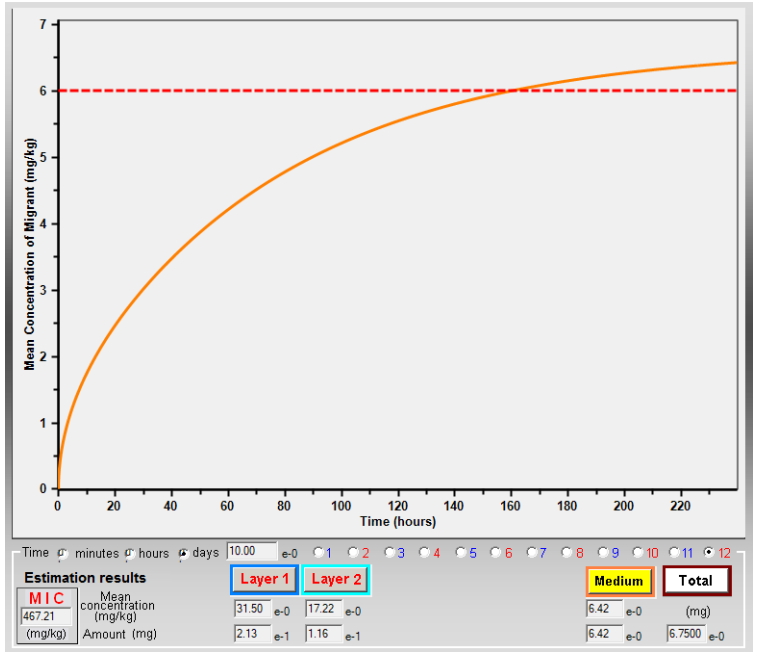
- Multi layer
  - Single side
  - Storage
  - Set-off

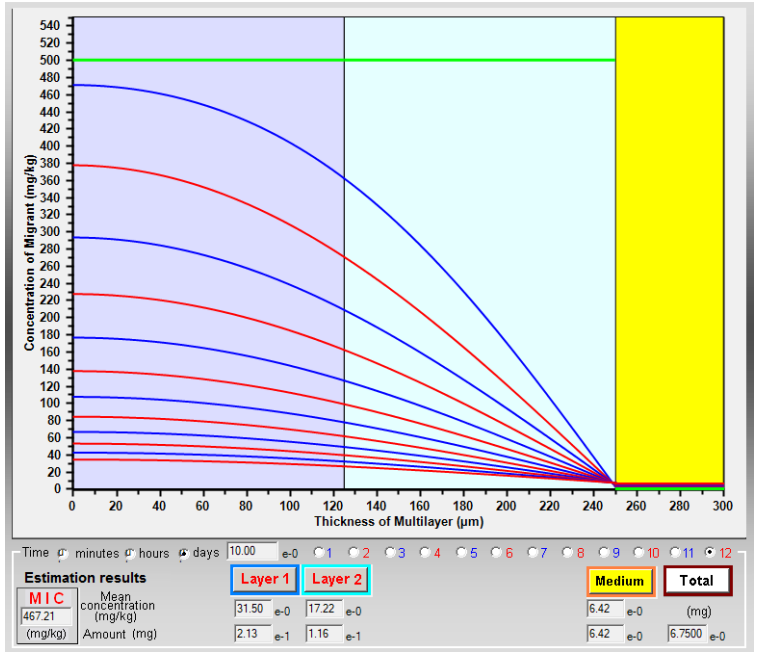


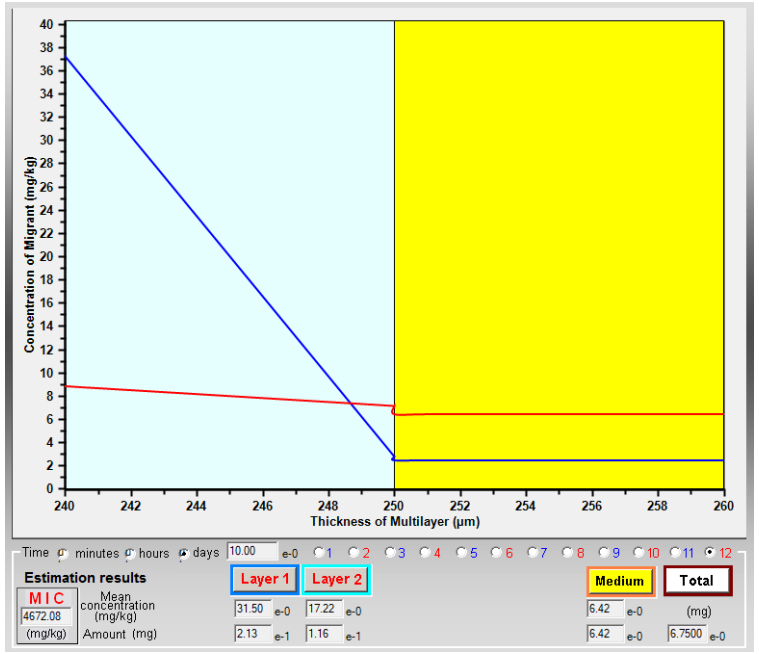


Stof 1: Molecular Mass (MM) = 500 Da Concentratie: 500 ppm (mg/kg FCM) PP homo: 250 micron (density 0,9 g/cm^3)	Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	WCC = 6,75 mg/kg Migratie resultaat: 6,42 mg/kg food

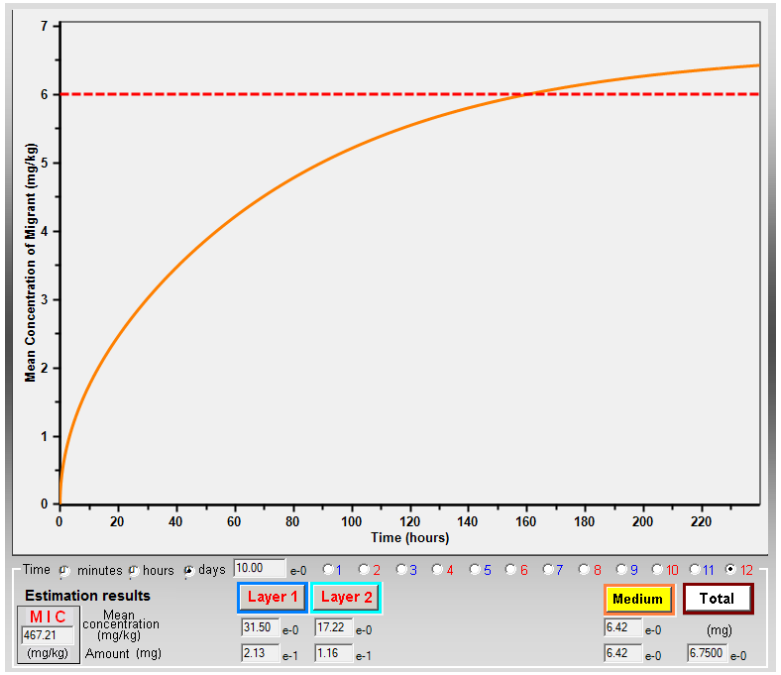


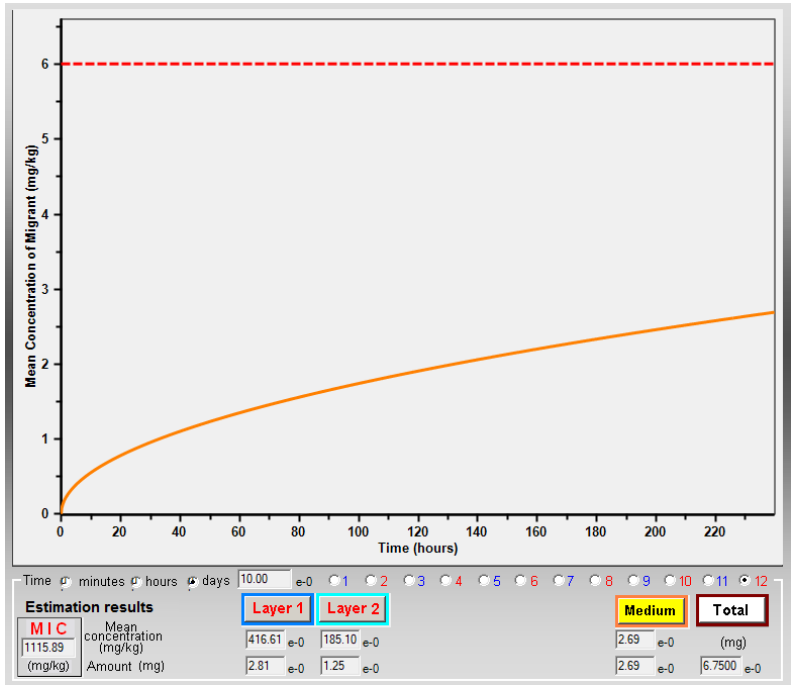


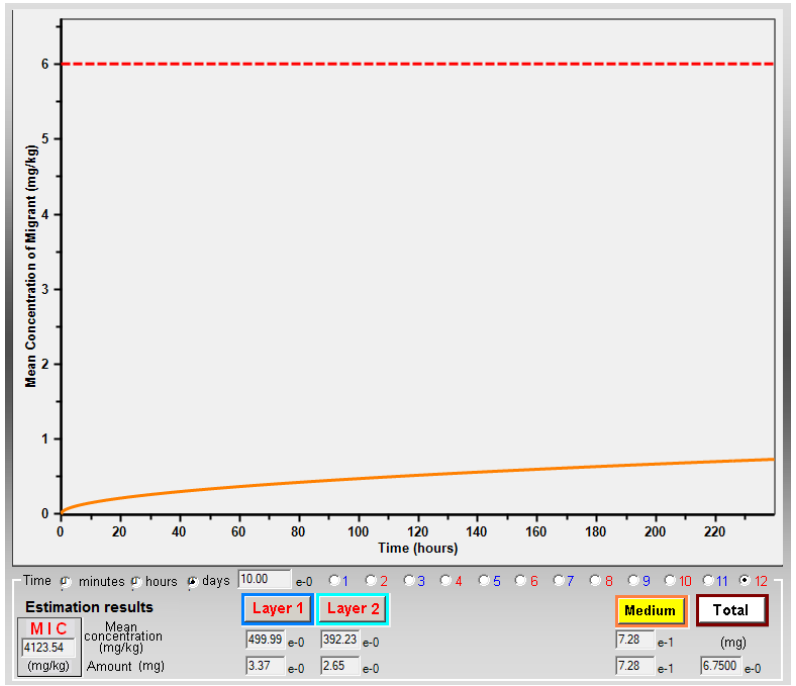




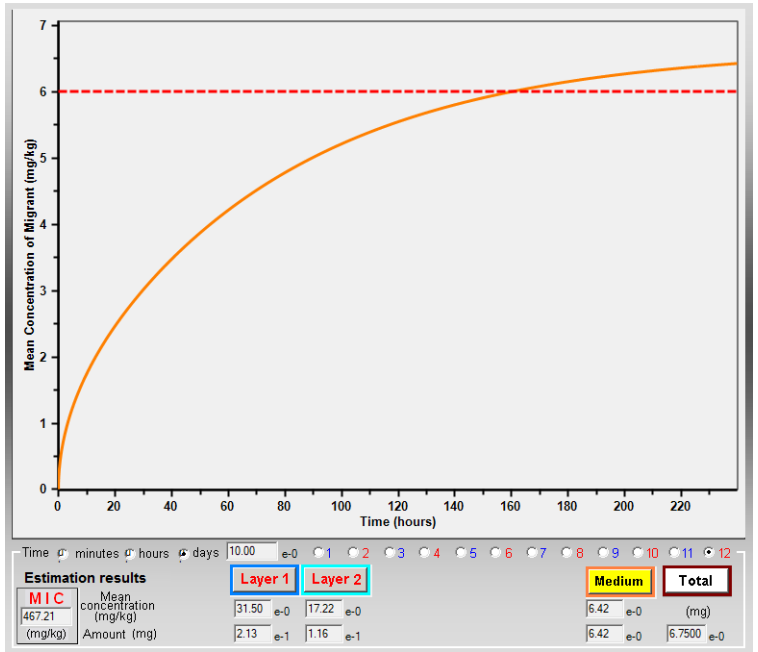
Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) PP homo: 250 micron (density 0,9 g/cm^3) S/V ratio: 6 dm^2/kg food	Condities: - 10d@ 60°C - 10d@ 40°C - 10d@ 20°C	Resultaat: mg/kg food - **6,42** - 2,69 - 0,73

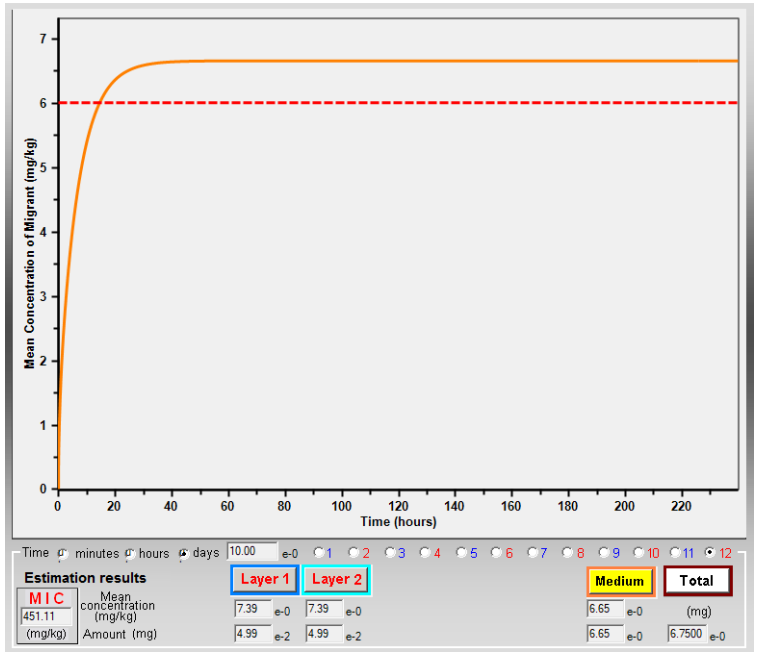


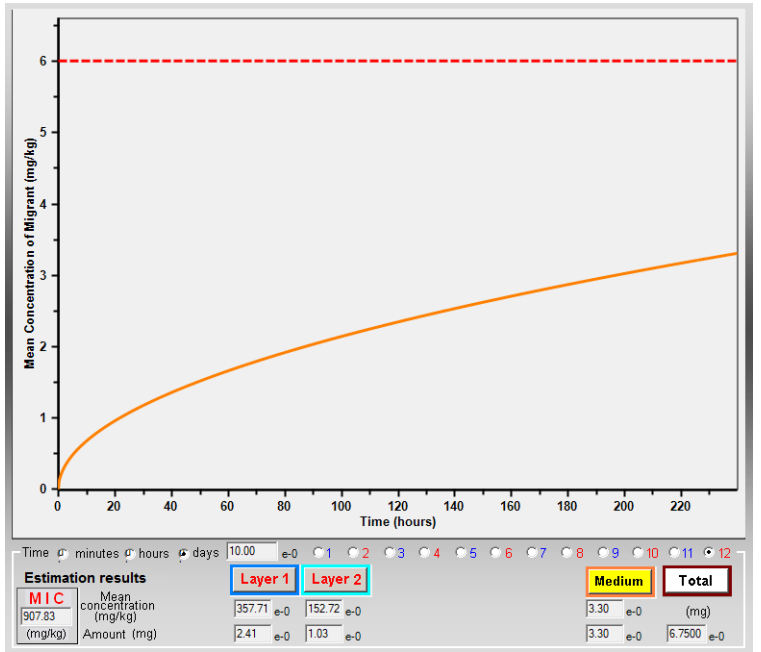




PP homo: 250 micron (density 0,9 g/cm^3) Concentratie: 500 ppm (mg/kg FCM) S/V ratio: 6 dm^2/kg food Condities: 10d@ 60^{o}C	Molecular Mass Stof 1: MM = 500 Da Stof 2: MM = 250 Da Stof 3: MM = 750 Da	Resultaat: mg/kg food - **6,42** - 6,65 - 3,30

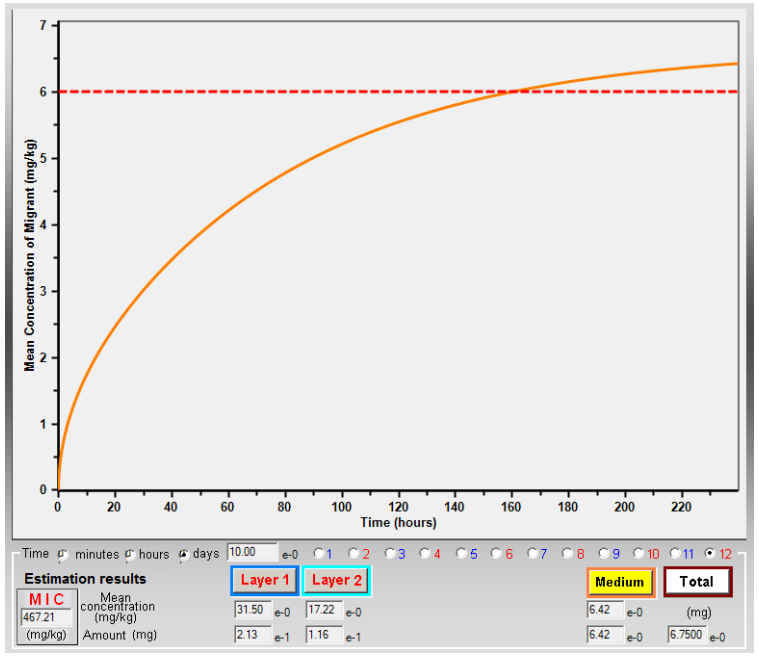


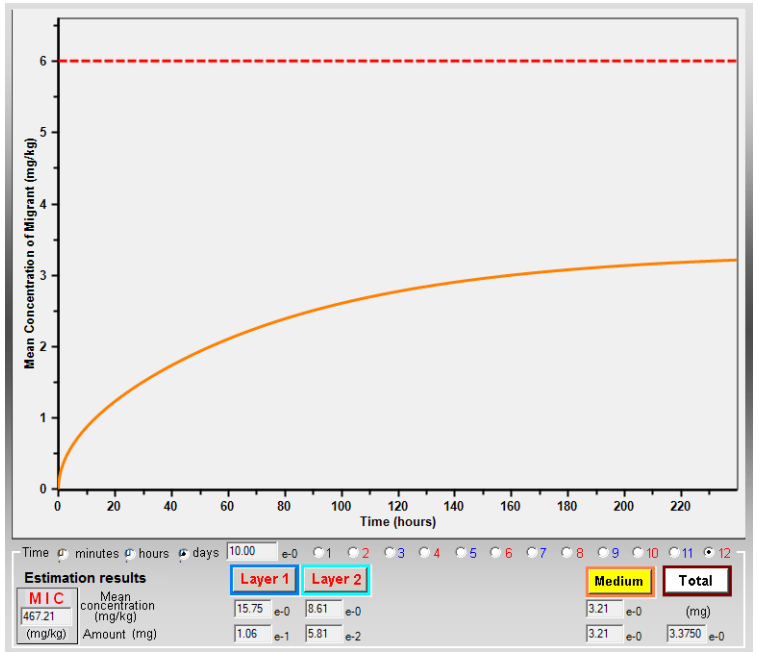


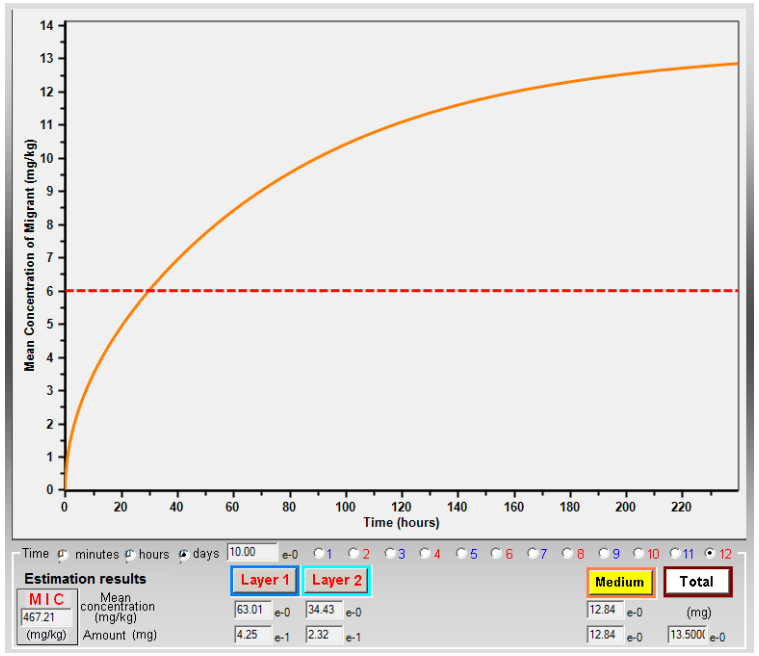


PP homo: 250 micron (density 0,9 g/cm^3) S/V ratio: 6 dm^2/kg food Condities: 10d@ 60^{o}C Stof 1: Molecular Mass = 500 Da	Concentratie (mg/kg FCM): 500 ppm 250 ppm 1000 ppm	Resultaat: mg/kg food - **6,42** - 3,21 - 12,84

	2x t/12	4x t/12	6x t/12	8x t/12	10x t/12	12x t/12
250 ppm	1,73	2,39	2,77	3,00	3,13	3,21
500 ppm	3,47	4,77	5,54	5,99	6,26	6,42
1000 ppm	6,94	9,55	11,08	11,99	12,53	12,84

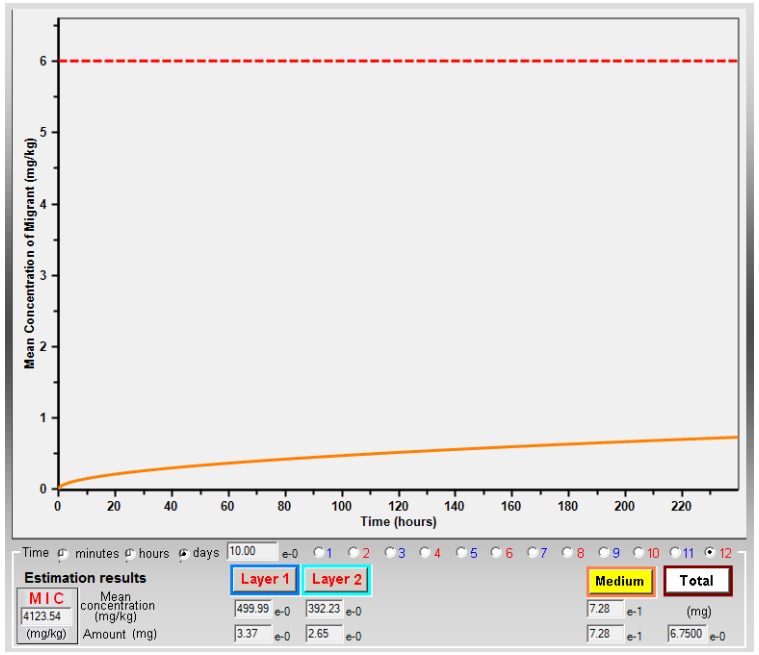


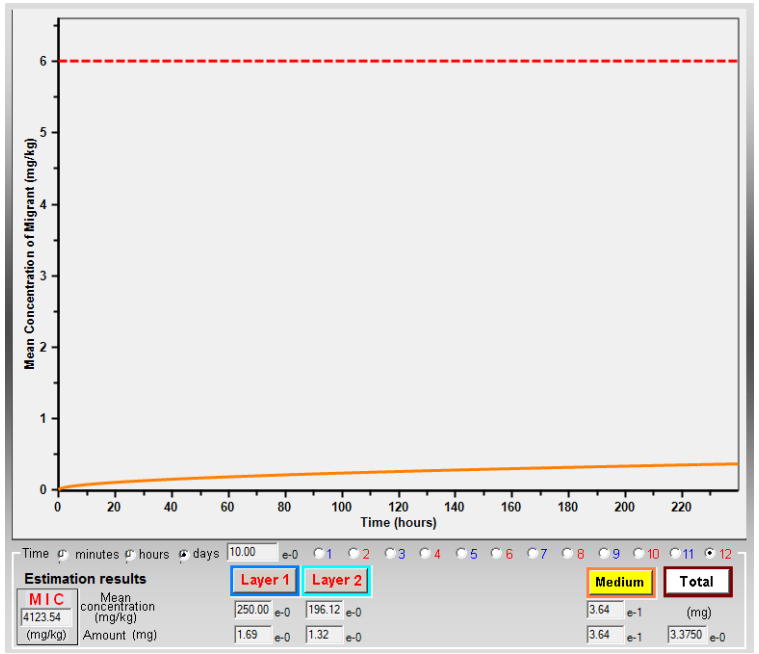


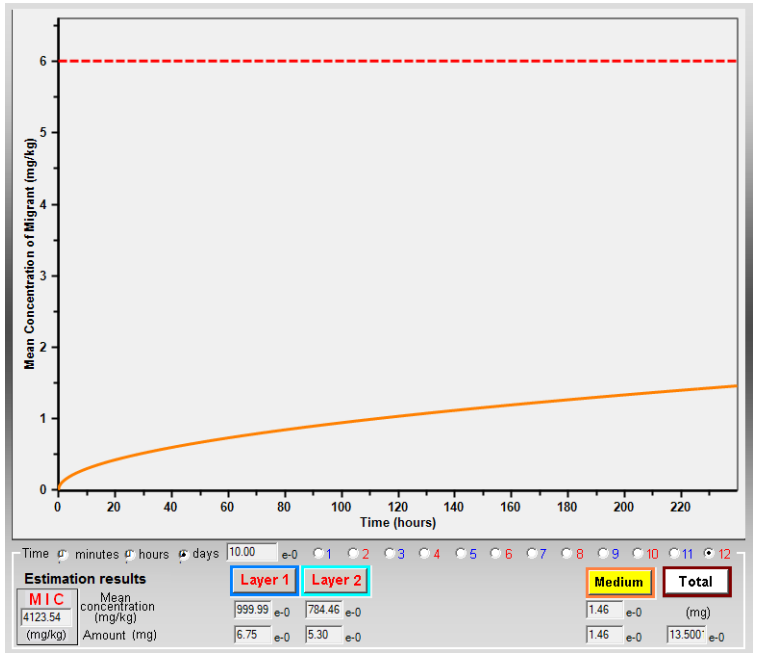


PP homo: 250 micron (density 0,9 g/cm^3) S/V ratio: 6 dm^2/kg food Condities: 10d@ 60°C / 10d@ 20°C Stof 1: Molecular Mass = 500 Da	Concentratie (mg/kg FCM): 500 ppm 250 ppm 1000 ppm	Resultaat: mg/kg food - **6,42**/0,73 - 3,21/0,365 - 12,84/1,46

Lineair verband tussen het migratie resultaat en de concentratie, ongeacht de conditie

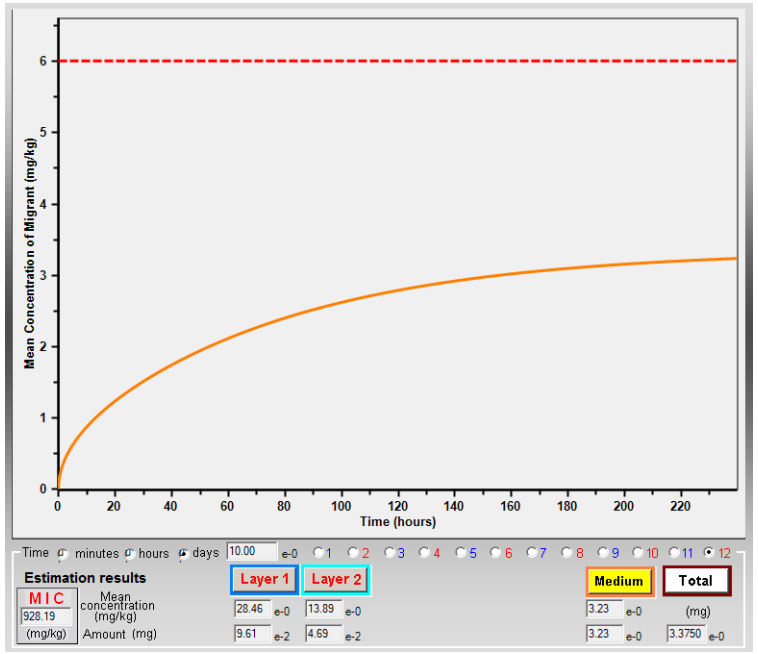


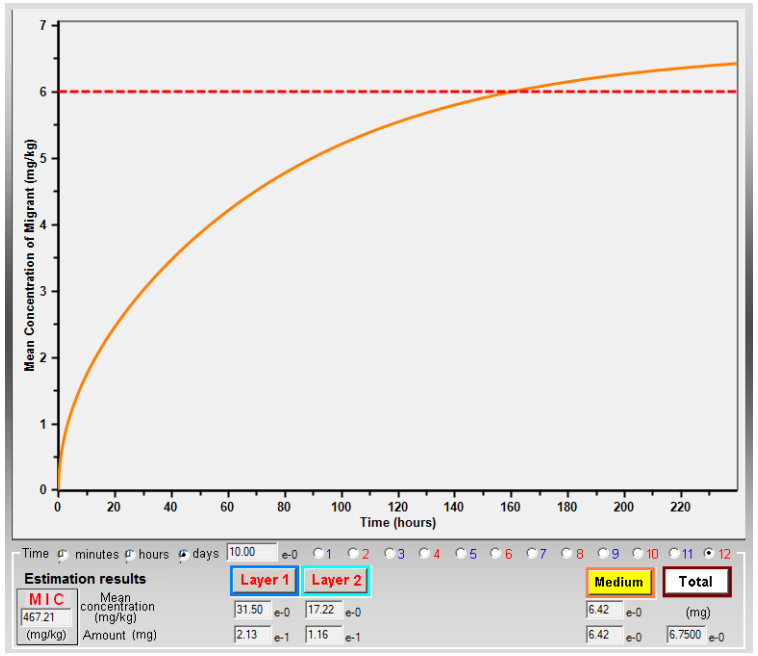


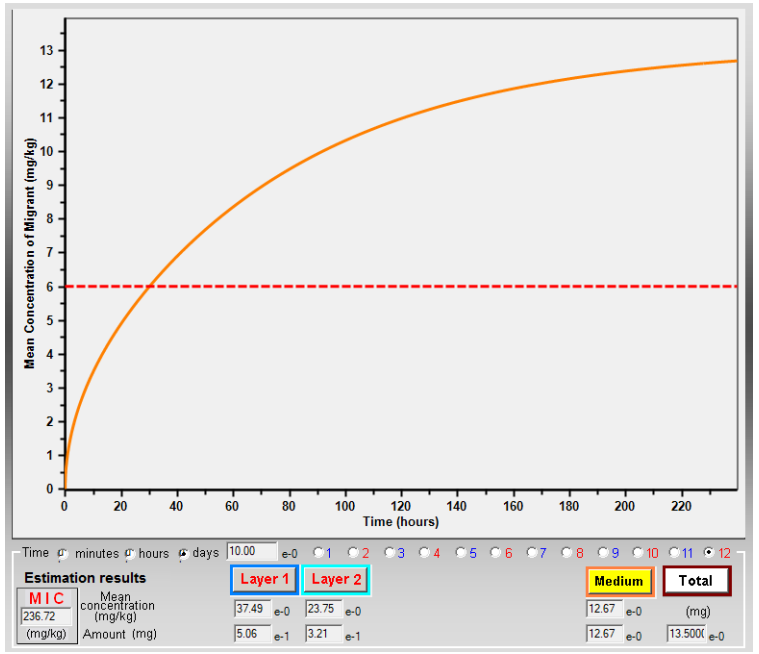


Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) PP homo: 250 micron (density 0,9 g/cm^3) Condities: 10d@ 60^{o}C	S/V ratio: - 3 dm^2/kg food - 6 dm^2/kg food - 12 dm^2/kg food	Resultaat: mg/kg food - 3,23 - **6,42** - 12,67

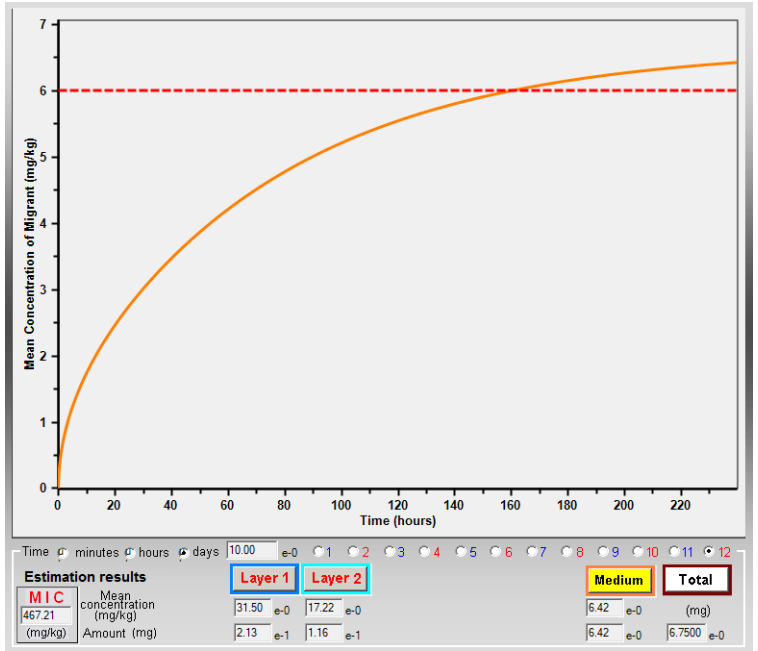
	2x t/12	4x t/12	6x t/12	8x t/12	10x t/12	12x t/12
3 dm^2/kg food	1,74	2,40	2,78	3,01	3,15	3,23
6 dm^2/kg food	3,47	4,77	5,54	5,99	6,26	6,42
12 dm^2/kg food	6,90	9,46	10,97	11,85	12,37	12,67

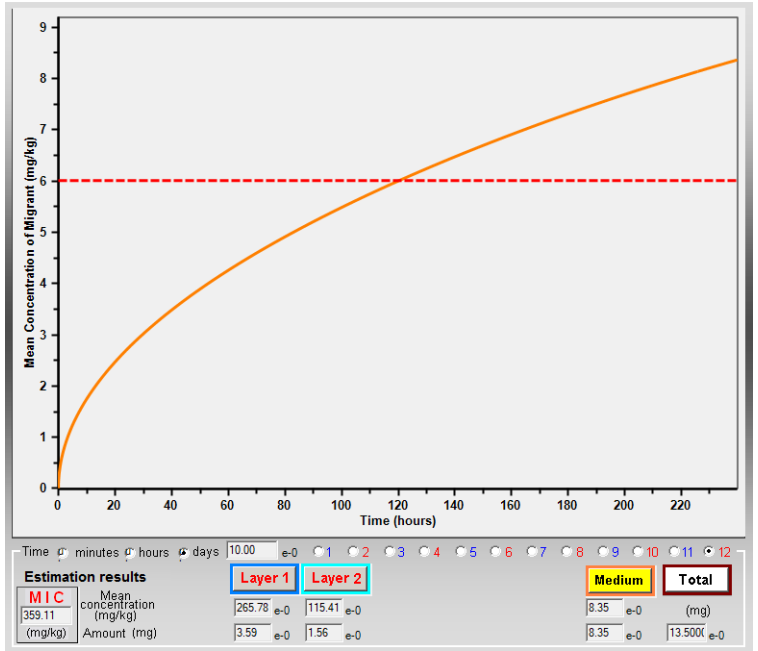


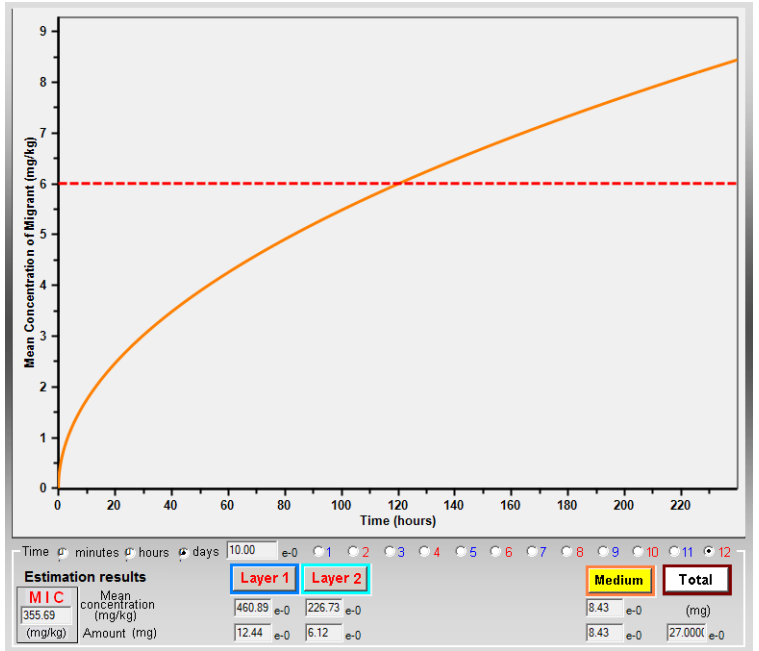




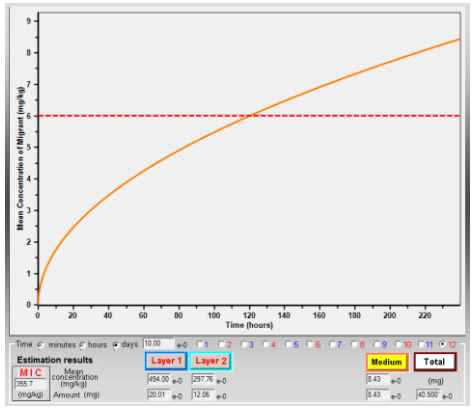
PP homo (density 0,9 g/cm^3) Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	Polymeer dikte - 250 micron - 500 micron - 1000 micron	Resultaat: mg/kg food - **6,42** - 8,35 - 8,43

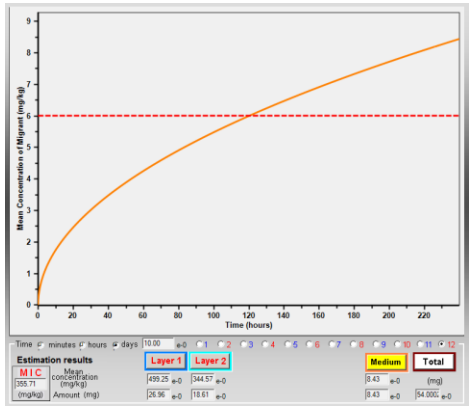


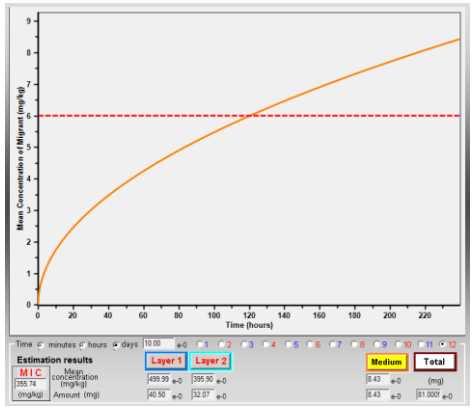


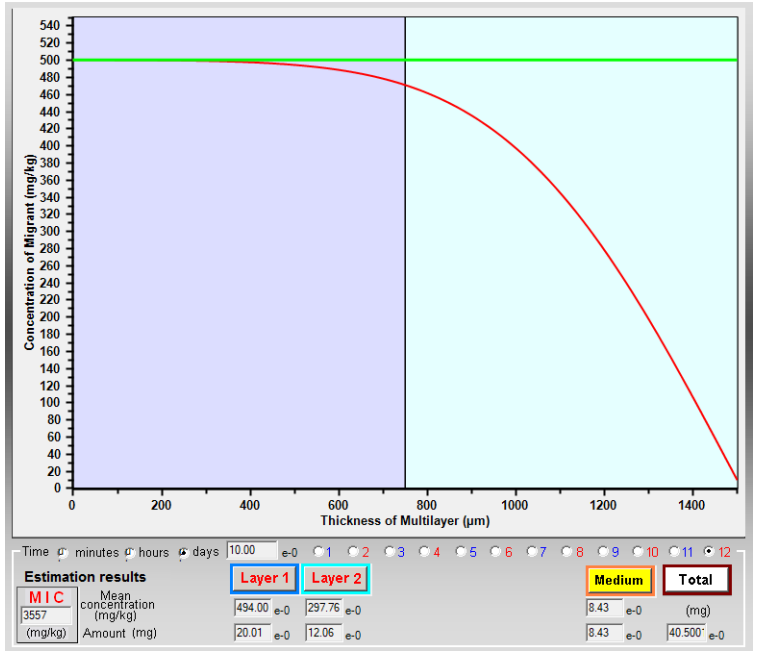


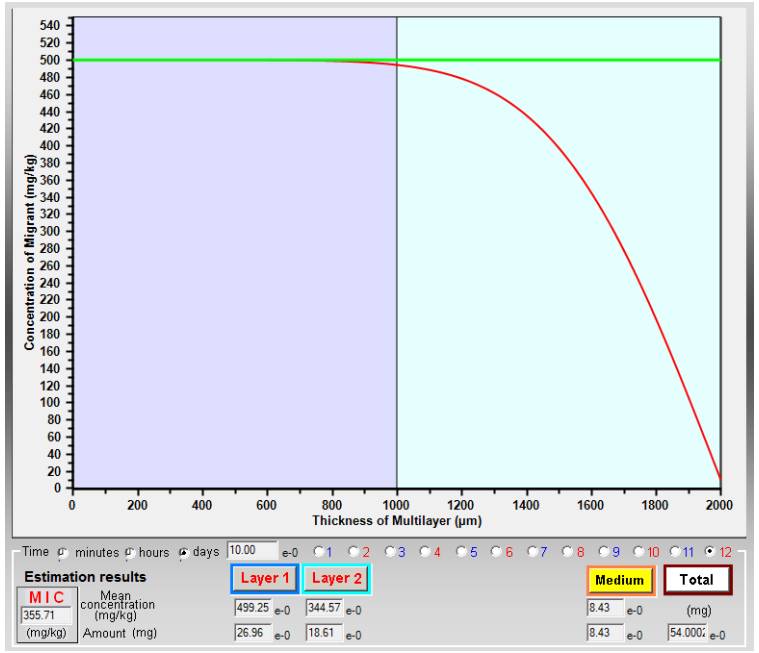
PP homo (density 0,9 g/cm^3) Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	Polymeer dikte - 1500 micron - 2000 micron - 3000 micron	Resultaat: mg/kg food - **8,43 (WCC = 40,5)** - 8,43 (WCC = 54) - 8,43 (WCC = 81)











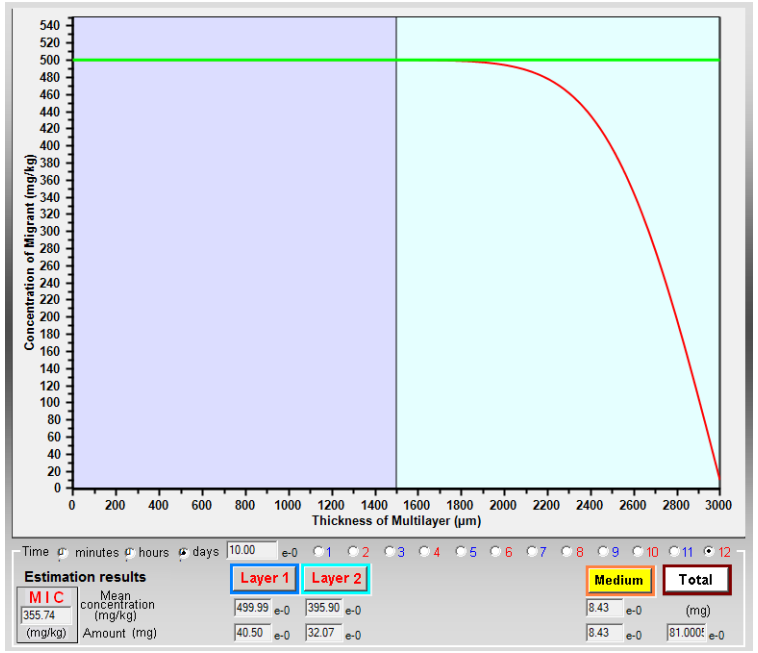
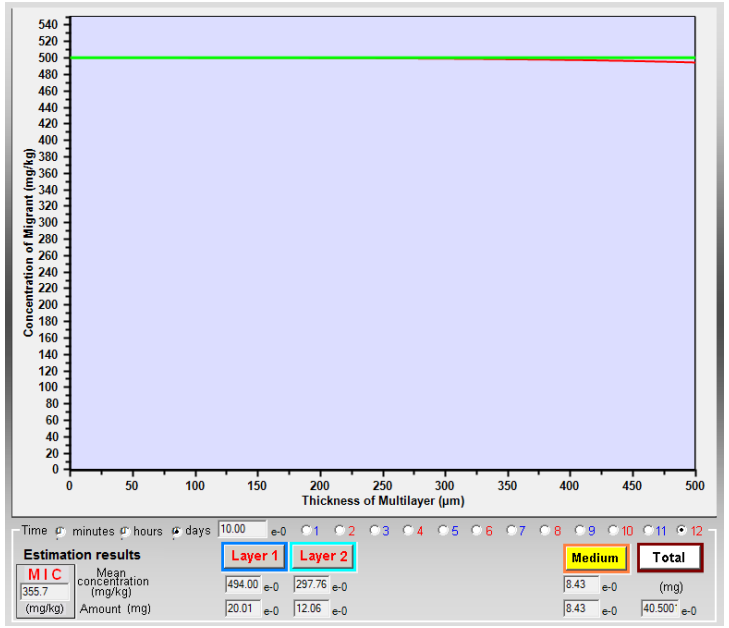
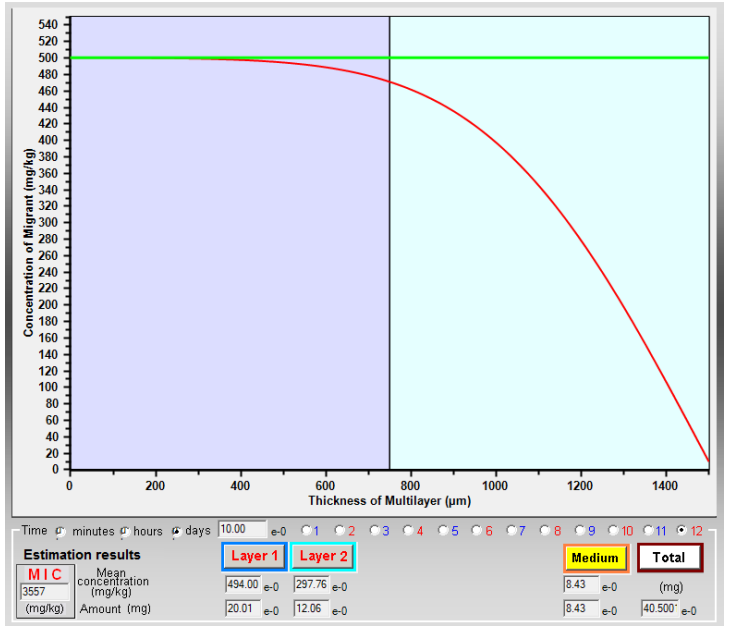


Table 7 Layer thickness L (in µm) for which total mass transfer assumption can be made at different contact conditions for four different molecular mass ranges

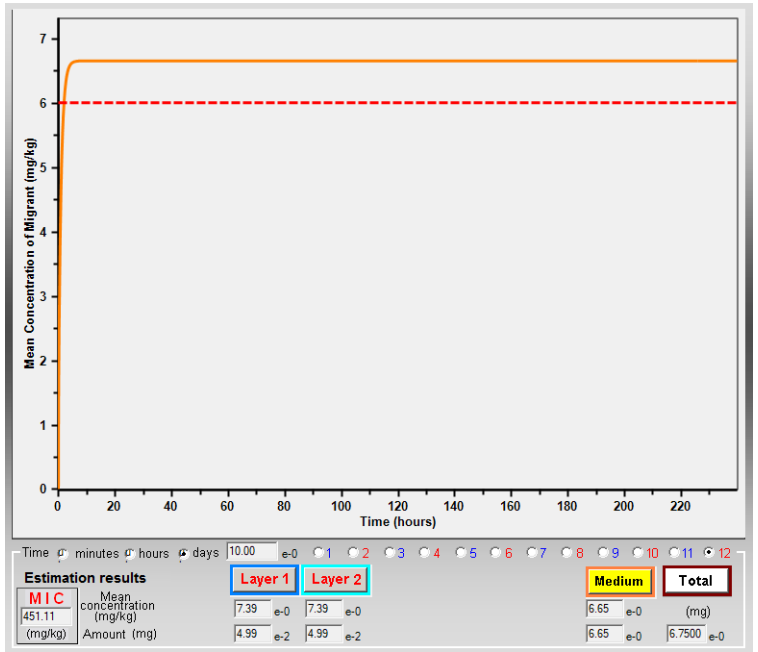
Polymer type	time/Temp	layer thickness L in [µm] for			
molecular mass of migrant (g/mol)		100-250	251-500	501-750	751-1000*
LDPE,	10 days at 60°C	Full L	Full L	2400	960
PP rubbery[8]	10 days at 40°C	Full L	3000	920	360
	10 days at 20°C	2500	880	300	120
	2h at 100°C	Full L	4000	1220	480
HDPE	10 days at 60°C	Full L	3425	1050	420
	10 days at 40°C	2950	1100	330	135
	10 days at 20°C	800	300	100	42
	2h at 100°C	Full L	2150	660	260
PP isotactic/homo PP random	10 days at 60°C	5000	1700	520	210
	10 days at 40°C	1460	550	170	72
	10 days at 20°C	400	155	55	20
	2h at 100°C	2925	1080	330	135
PET, PBT, PEN	10 days at 60°C	40	15	5	2
	10 days at 40°C	13	5	2	1
	10 days at 20°C	4	2	1	0.5
	2h at 100°C	25	10	3	2
PS	10 days at 60°C	55	21	7	3

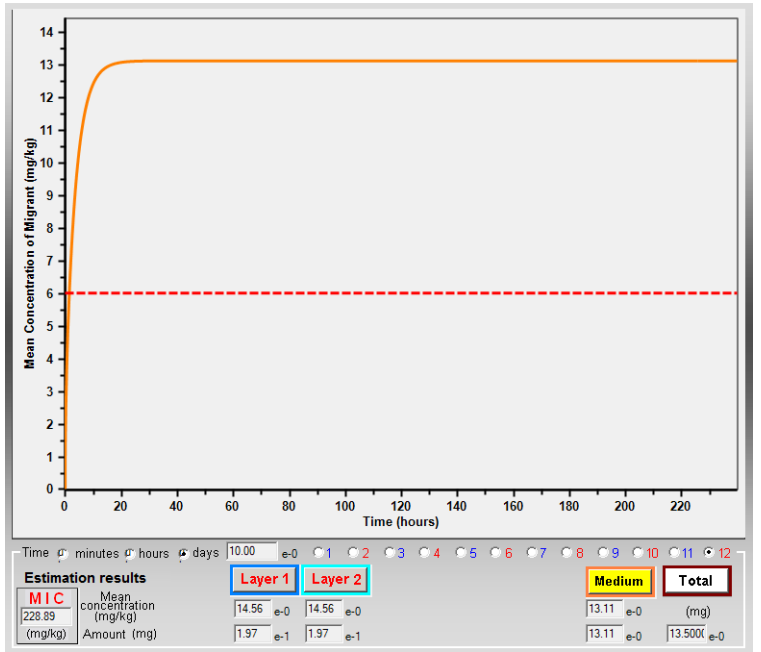


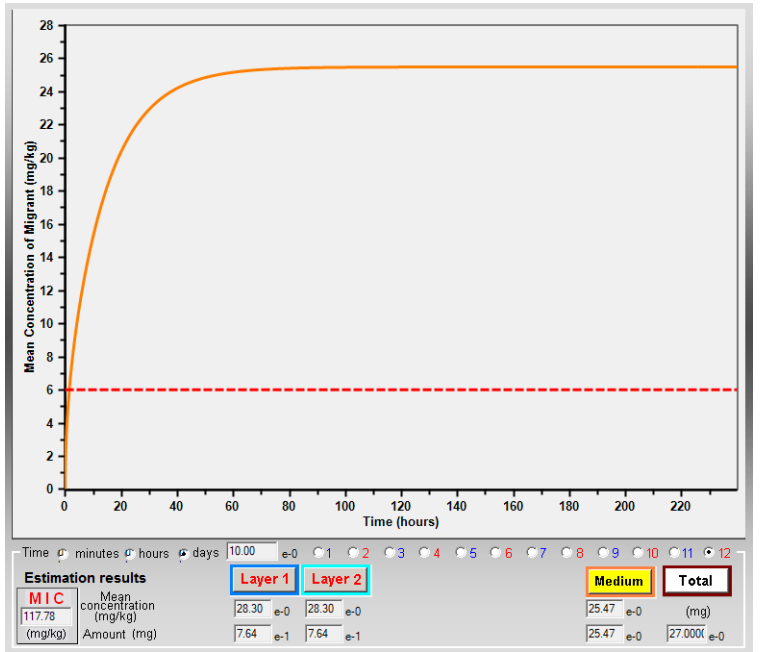


PP homo (density 0,9 g/cm^3) **Stof 1: Molecular Mass = 100 Da** Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	Polymeer dikte - 250 micron - 500 micron - 1000 micron	Resultaat: mg/kg food - **6,65** - 13,11 - 25,47

Full migration or equilibrium.
Migration testing = WCC

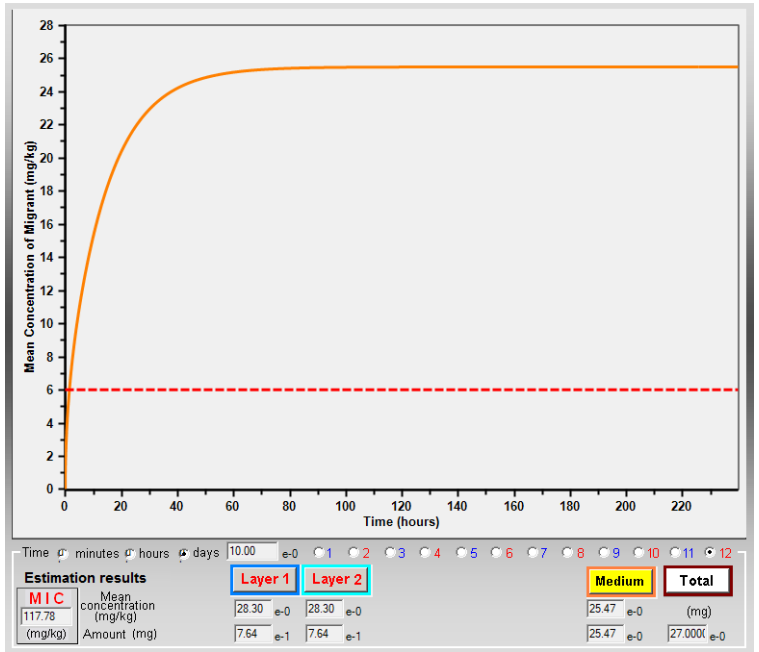


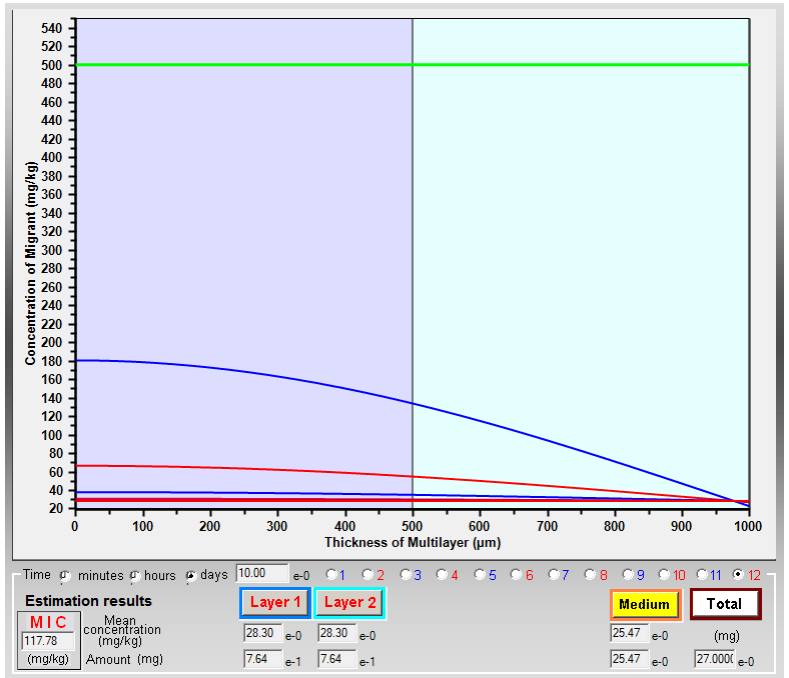


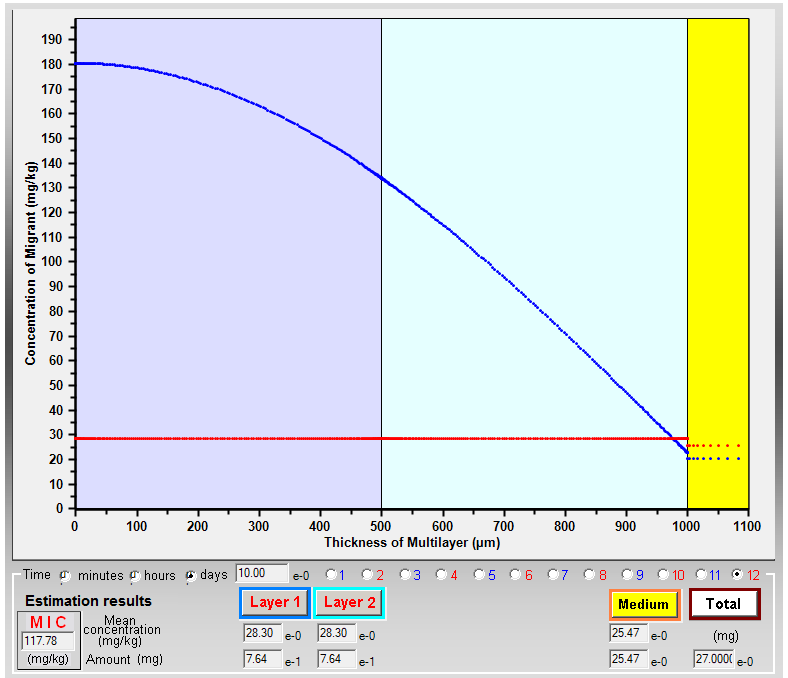


PP homo (density 0,9 g/cm^3) **Stof 1: Molecular Mass = 100 Da** Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	Polymeer dikte - 250 micron - 500 micron - 1000 micron	Resultaat: mg/kg food - **6,65** - 13,11 - 25,47

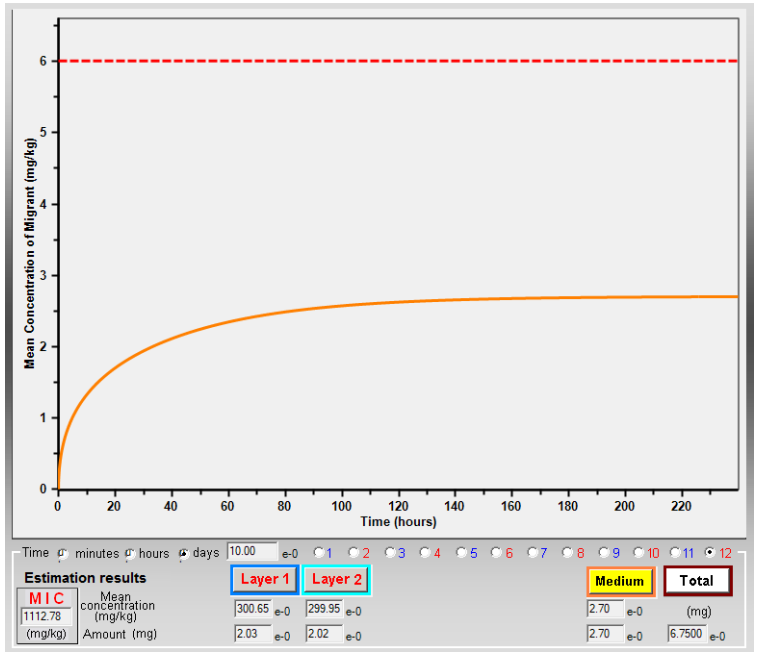
Full migration or equilibrium.
Migration testing = WCC

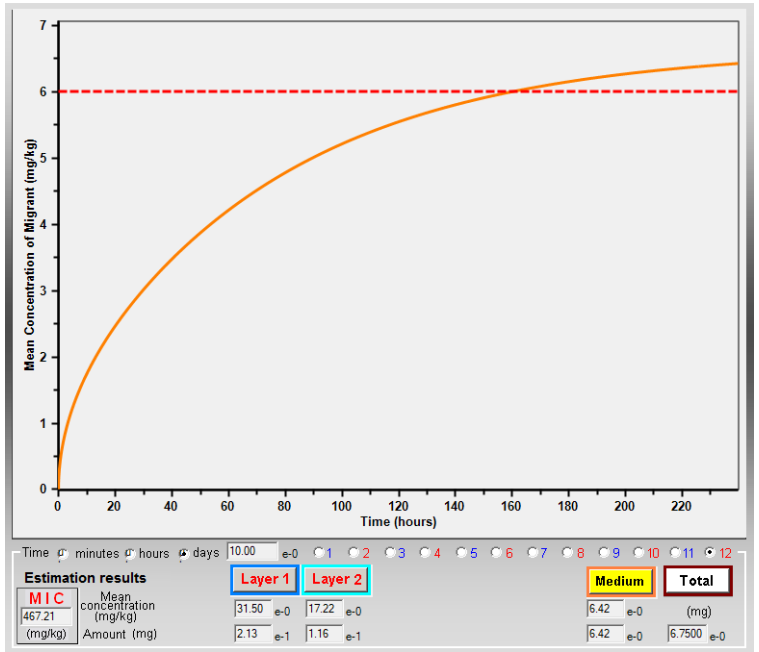


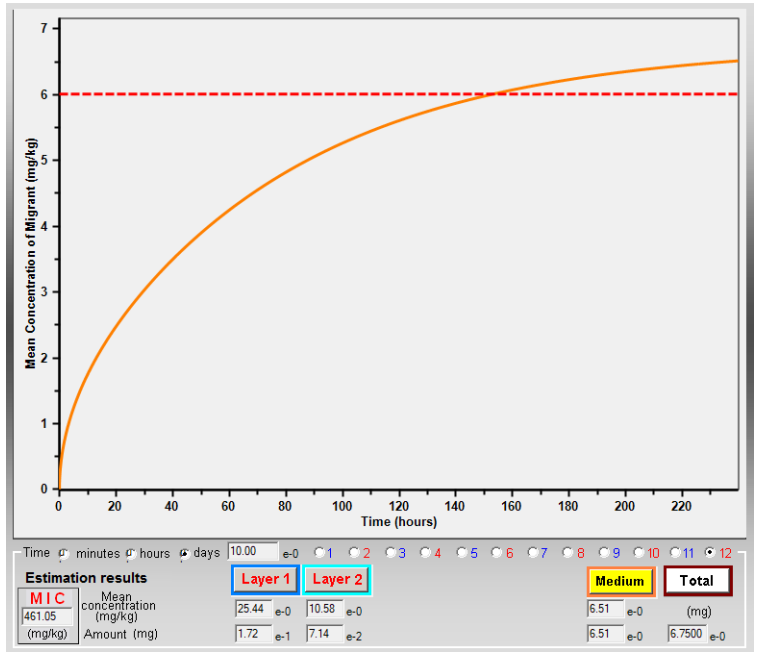




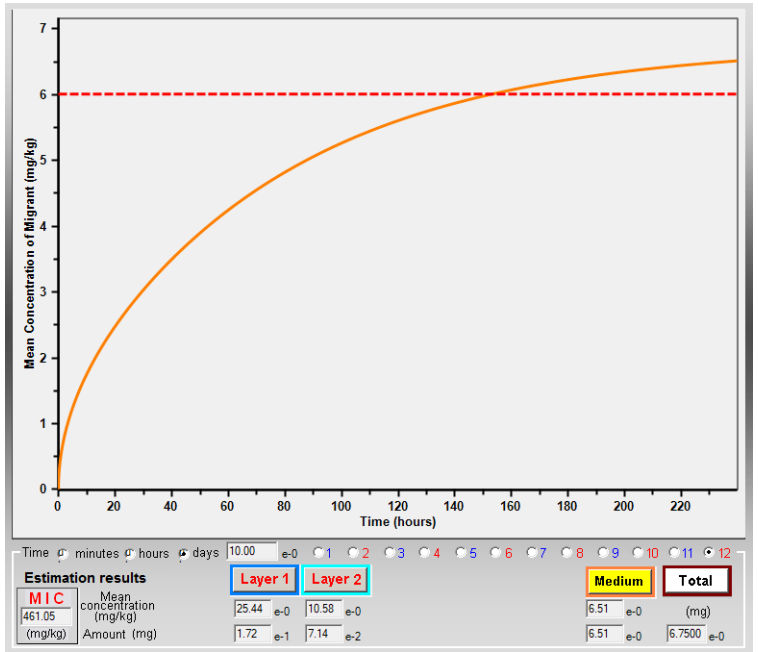
PP homo: 250 micron (density 0,9 g/cm^3)	Polariteit (partitioning factor)	Resultaat: mg/kg food
Concentratie: 500 ppm (mg/kg FCM)	- PP/Food = 100	- 2,70
Condities: 10d@ 60^{o}C	- PP/Food = 1	- **6,42**
S/V ratio: 6 dm^2/kg food	- PP/Food = 0,01	- 6,51

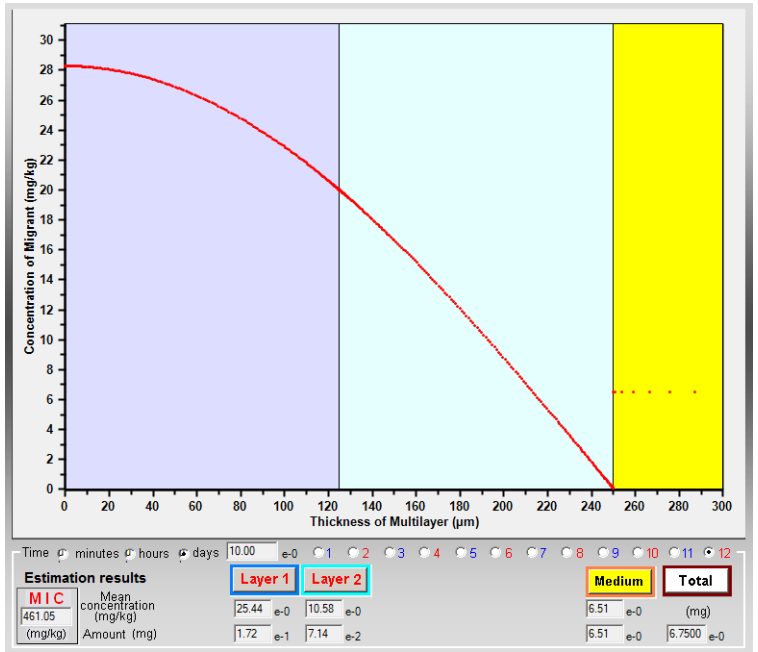


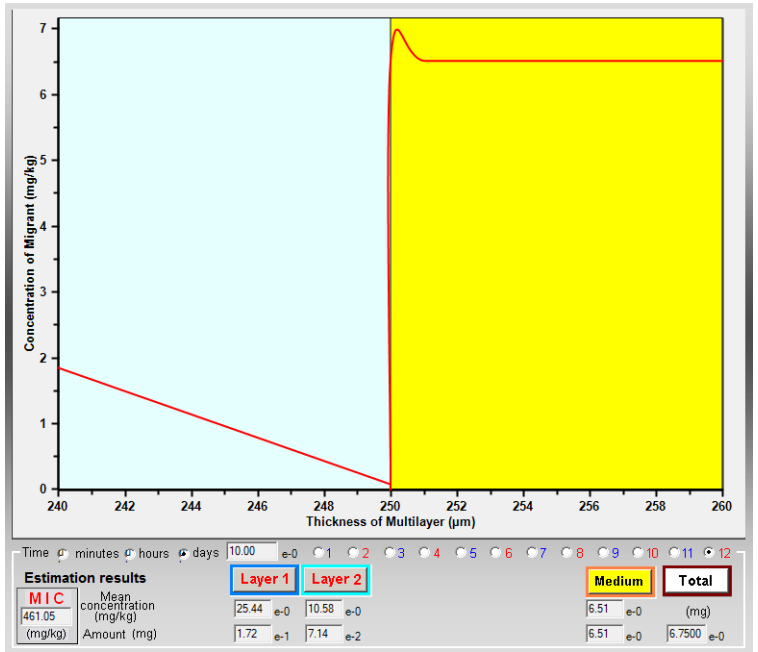




PP homo: 250 micron (density 0,9 g/cm$^{3)}$ Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^{2}/kg food	Polariteit (partitioning factor) - PP/Food = 100 - PP/Food = 1 - PP/Food = 0,01	Resultaat: mg/kg food - 2,70 - **6,42** - 6,51



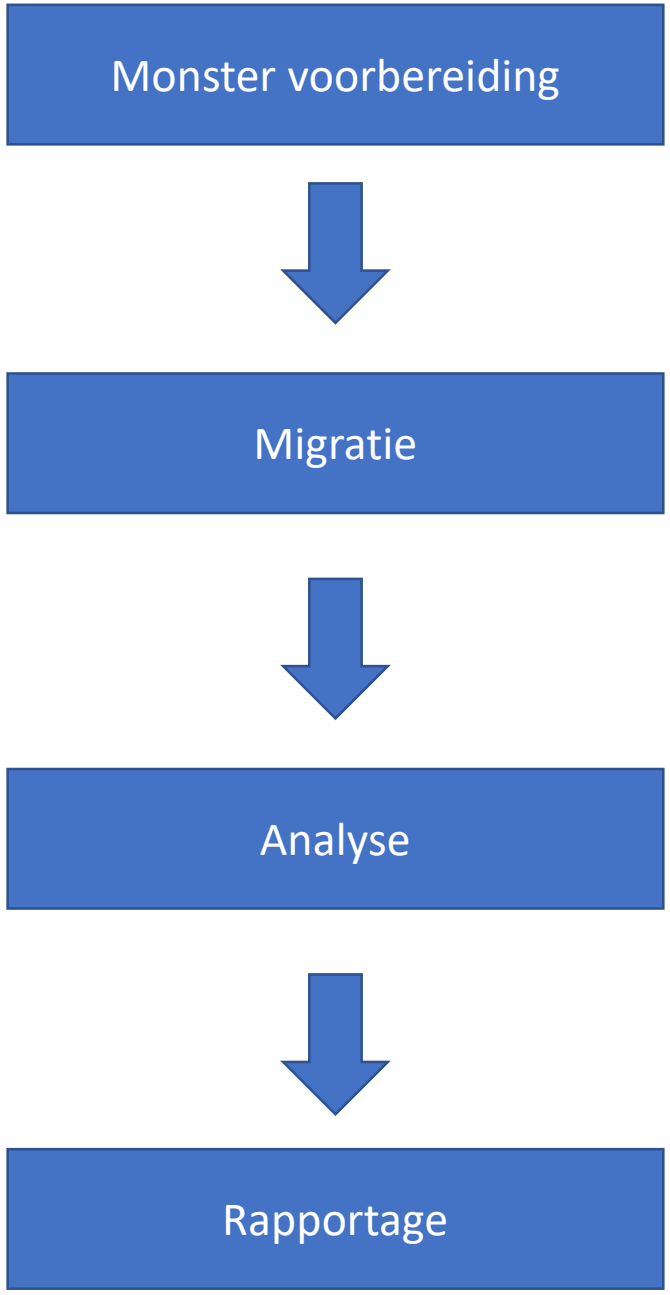




Migratietesten

- Veiligheid
  - Specifieke migratie
  - NIAS screening

- Inertie
  - Totale migratie
  - Color bleeding
  - Organoleptic testing



- Shelf life van de verpakking
- Set-off

Tijd
Temperatuur

- Type test
  - Total immersion
  - Single side
- Test conditions
- Repeated use
- Simulants

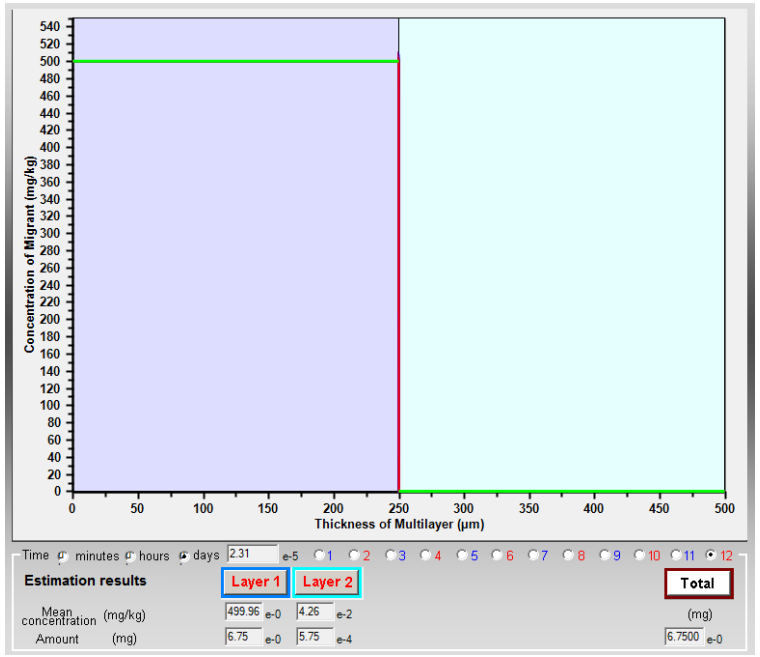
Tijd
Temperatuur
Simulant

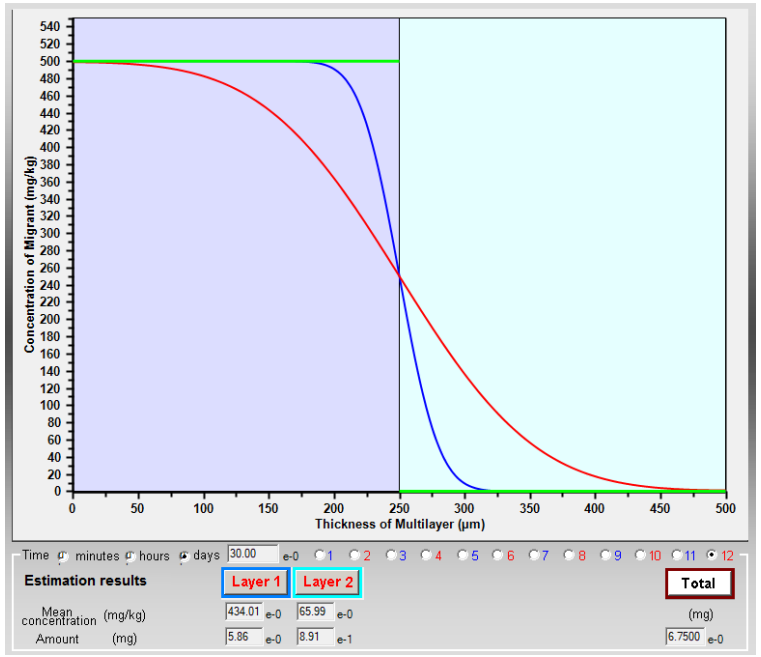
- Specifieke migratie
  - Screening test
  - Target test
- LOD (Limit of detection)
- LOQ (limit of quantification)
- Meetonnauwkeurigheid

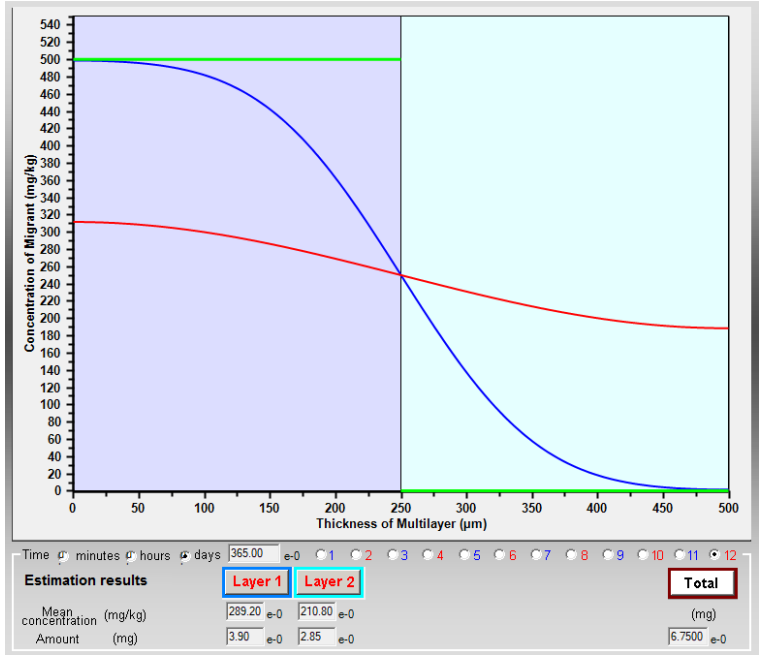
- S/V ratio
- Reductiefactoren
- Baby food

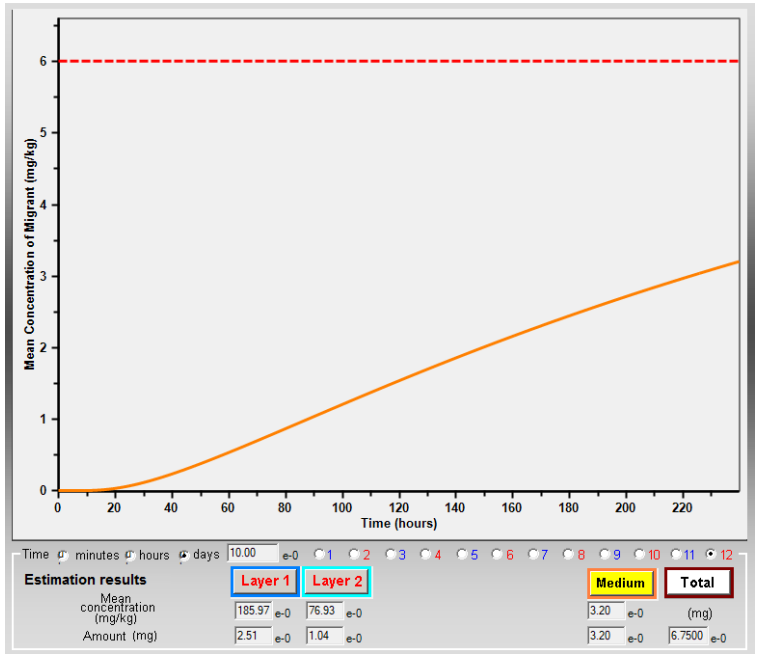
Monster voorbereiding

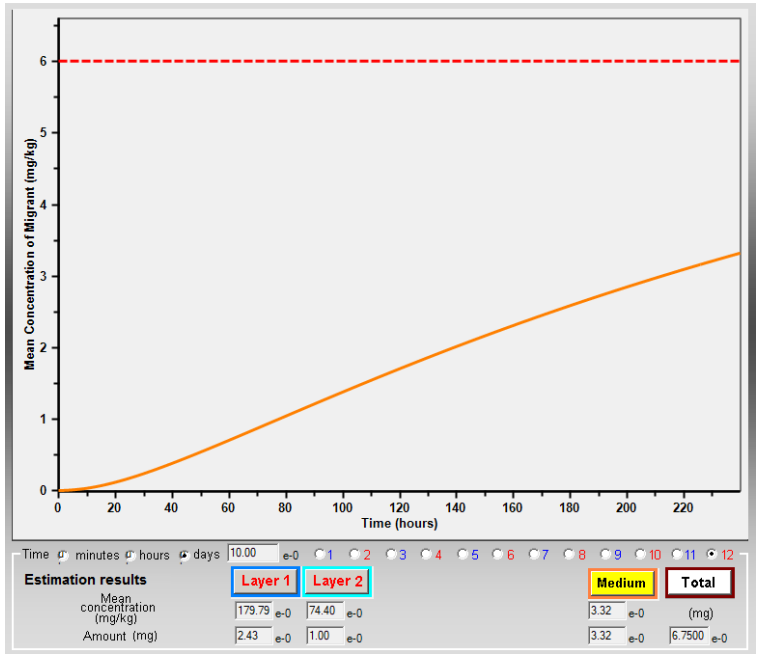
- Shelf life van de verpakking
- Set-off

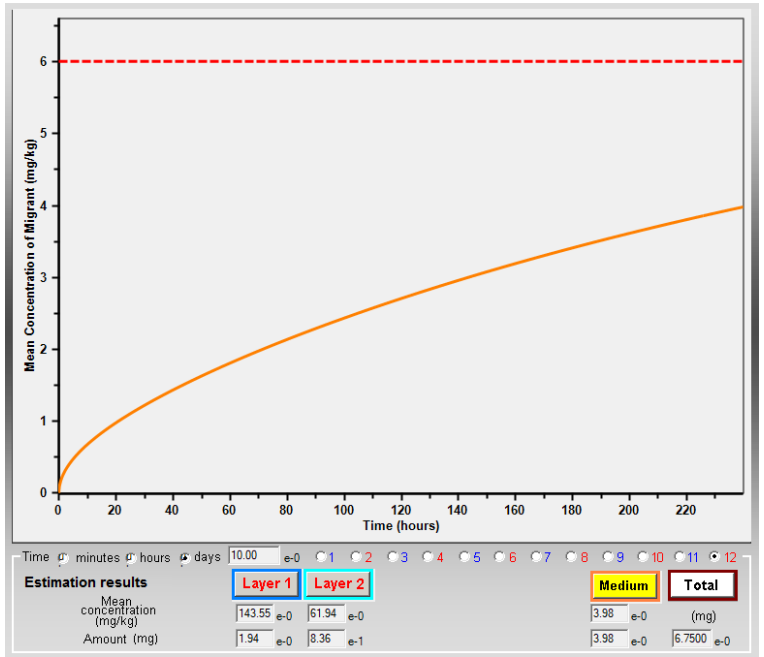

Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 2.31 e-5 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Total
Mean concentration (mg/kg) 499.96 e-0 4.26 e-2
Amount (mg) 6.75 e-0 5.75 e-4
(mg) 6.7500 e-0


Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 30.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Total
Mean concentration (mg/kg) 434.01 e-0 65.99 e-0
Amount (mg) 5.86 e-0 8.91 e-1
(mg) 6.7500 e-0


Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 365.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Total
Mean concentration (mg/kg) 289.20 e-0 210.80 e-0
Amount (mg) 3.90 e-0 2.85 e-0
(mg) 6.7500 e-0


Mean Concentration of Migrant (mg/kg)
Time (hours)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Medium
Total
Mean concentration (mg/kg) 185.97 e-0 76.93 e-0 3.20 e-0
Amount (mg) 2.51 e-0 1.04 e-0 3.20 e-0 6.7500 e-0
(mg)

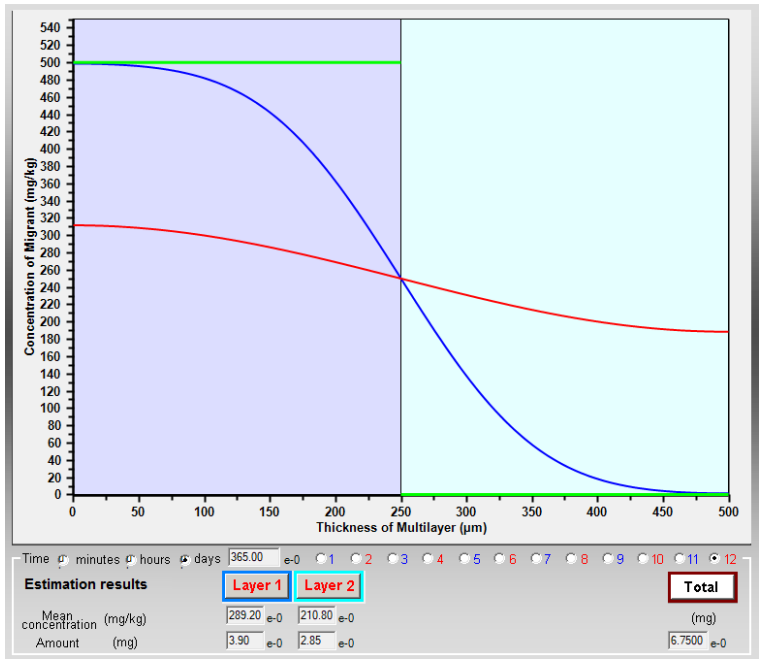

Mean Concentration of Migrant (mg/kg)
Time (hours)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Medium
Total
Mean concentration (mg/kg) 179.79 e-0 74.40 e-0 3.32 e-0
Amount (mg) 2.43 e-0 1.00 e-0 3.32 e-0 6.7500 e-0
(mg)


Mean Concentration of Migrant (mg/kg)
Time (hours)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
Layer 1
Layer 2
Medium
Total
Mean concentration (mg/kg) 143.55 e-0 61.94 e-0 3.98 e-0
Amount (mg) 1.94 e-0 8.36 e-1 3.98 e-0 6.7500 e-0
(mg)

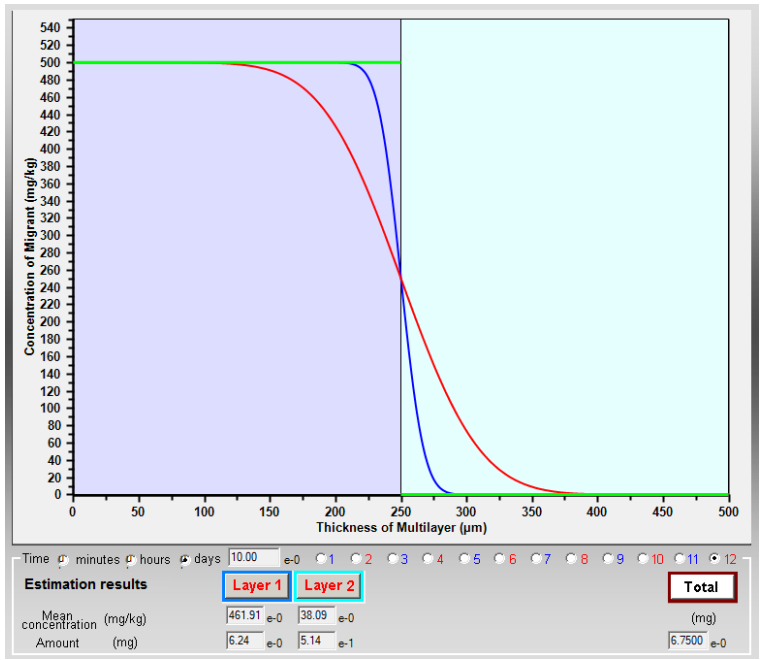
Veroudering door labo

- Versnelde veroudering door labo
  Plaatsen van stalen in oven, voorafgaan aan de test
- Testen op verouderde stalen

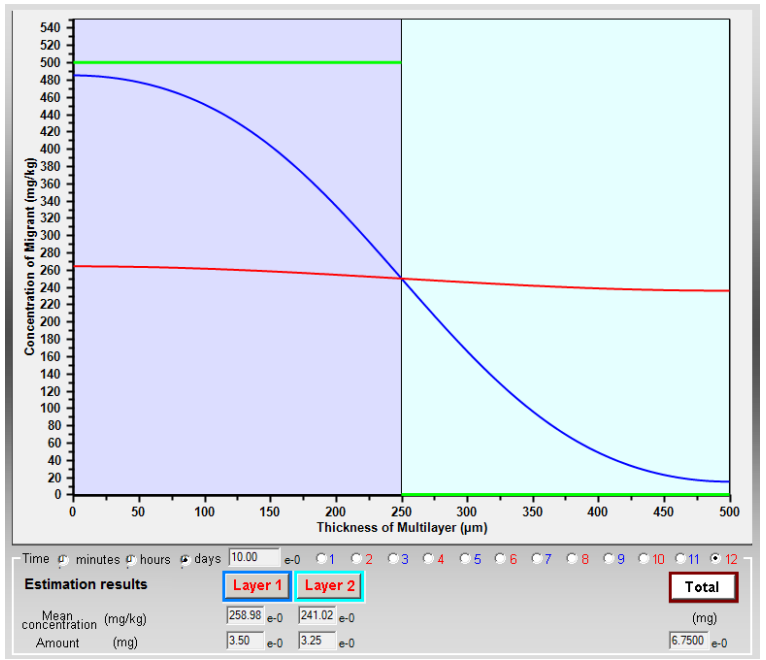
365d @ 25°C



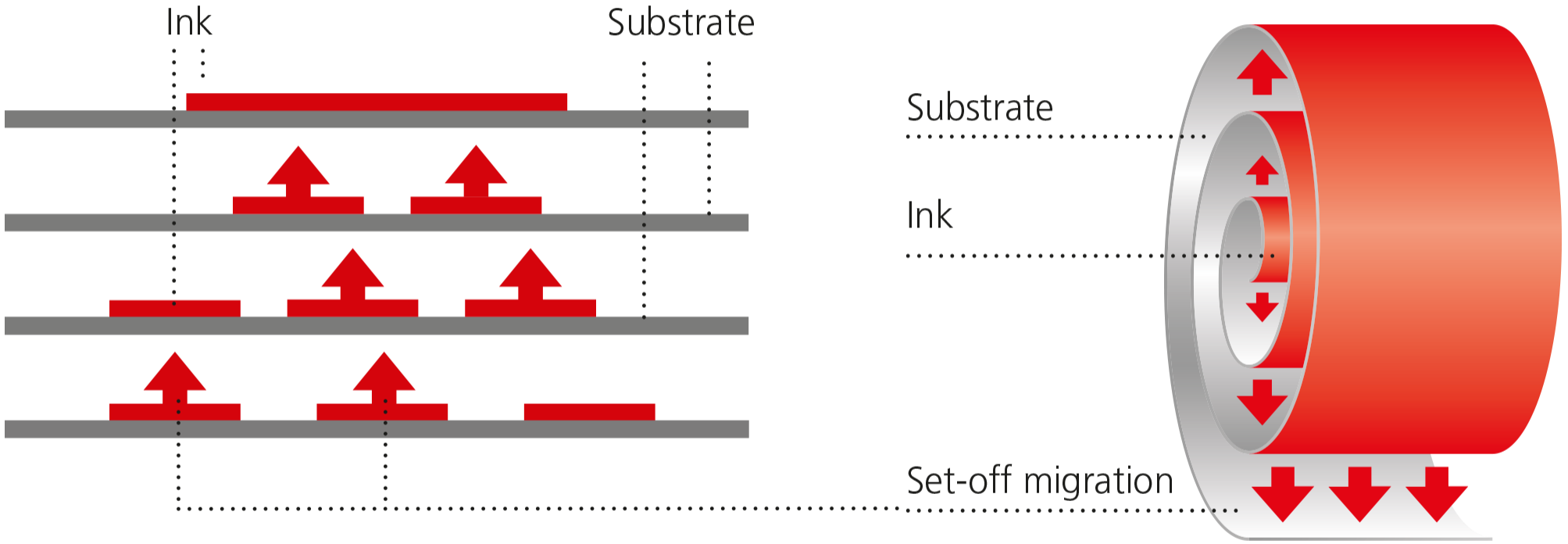
10d@40°C



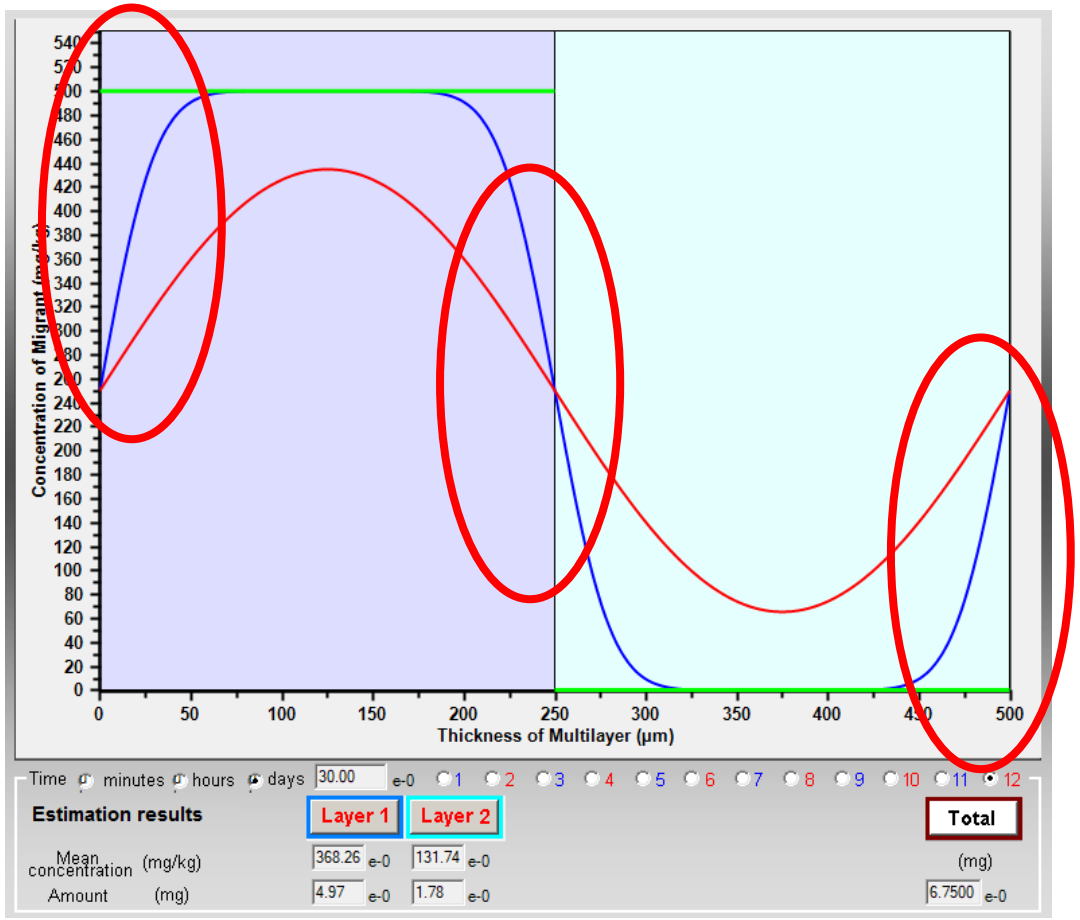
10d@60°C



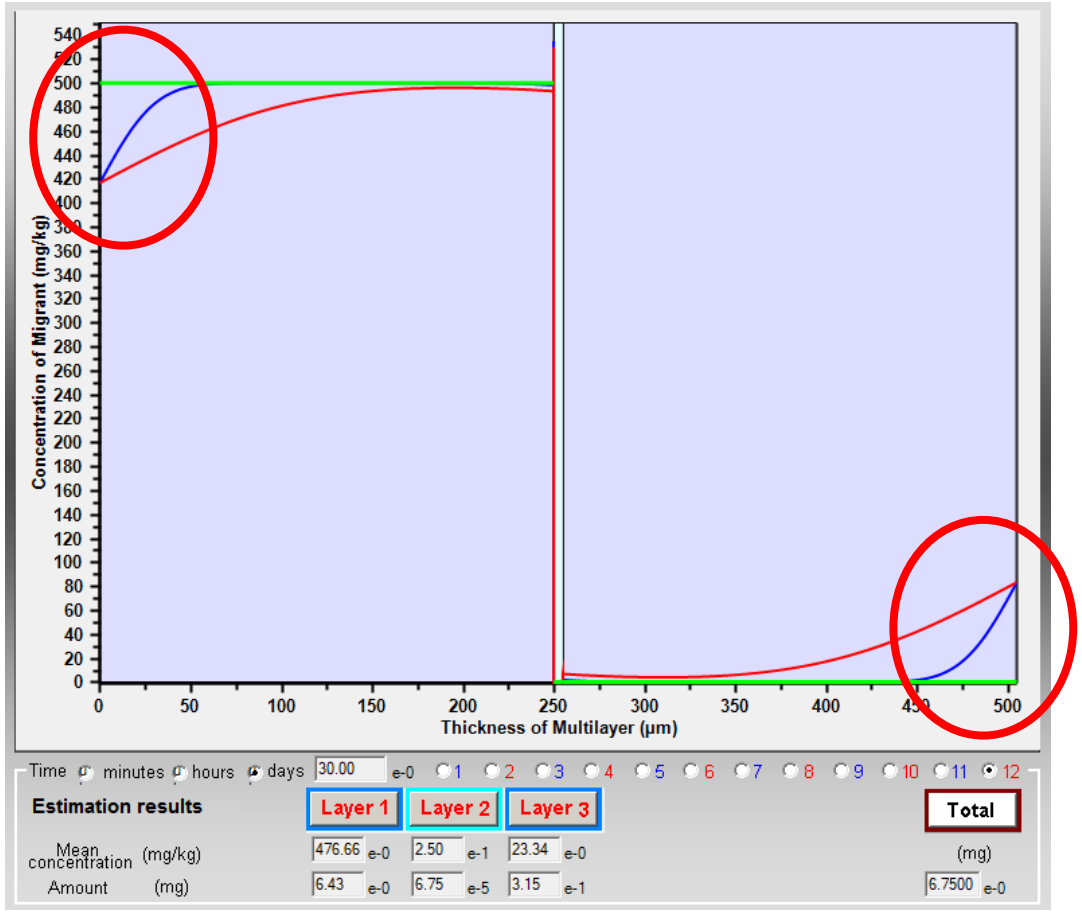
Set-off



Set-off zonder barrière



Set-off met barrière



Barrière materialen

- Polymeren
  - Functionele barrière
- Complexe meerlaagse materialen
  - Absolute barrière
  - Functionele barrière

Polymer	time/Temp	FB layer thickness (µm)			
Molecular mass range of migrant (g/mol)		100-250	251-500	501-750	751-1500
LDPE, PP rubber	10 days at 60°C	no FB	no FB	7000	2600
	10 days at 40°C	no FB	8800	2640	1000
	10 days at 20°C	7000	3000	800	340
	2 hours at 100°C	no FB	10000	3240	1360
HDPE	10 days at 60°C	no FB	9000	3300	1080
	10 days at 40°C	8500	3000	960	400
	10 days at 20°C	2280	800	280	130
	2 hours at 100°C	no FB	6400	1800	700
PP homo/isotactic; random	10 days at 60°C	no FB	4600	1400	580
	10 days at 40°C	3900	1480	500	220
	10 days at 20°C	1080	440	160	70
	2 hours at 100°C	8000	3000	900	380
PET, PBT, PEN	10 days at 60°C	91	35	12	5
	10 days at 40°C	31	14	4	2
	10 days at 20°C	9	4	2	1
	2 hours at 100°C	61	23	7	3
PS	10 days at 60°C	127	49	16	6
	10 days at 40°C	46	18	6	3
	10 days at 20°C	17	7	3	1
	2 hours at 100°C	65	26	8	4

Barrière materialen

- Polymeren
  - Functionele barrière

- Complexe meerlaagse materialen
  - Absolute barrière
  - Functionele barrière

Table 10 **Barrier films which act as a general FB in reducing any migration down to levels below of 10 ppb at test contact conditions of 10 d @ 60°C.**

Film structure	Base polymer	Barrier material
36 µm O-PET corona treated	PET	PET
12 µm PET metallised	PET	metallisation
12 µm PET-SiOx 80 nm [1]	PET	SiOx
12 µm PET-SiOx 50 nm Ormocer-Laquer[1]	PET	SiOx / Ormocer
12 µm PET / SiOx[1]	PET	SiOx
12 µm PET / AlOx / adhesive / 30 µm PP	PP	PET-AlOx
6 µm aluminium[*]		*Aluminium*
6 µm aluminium[*] */ PE*	*PE*	*Aluminium*

[1] It should be noted that this is only the case when no pinholes or other damages are present.

Table 11 **Barrier films which act as a FB in reducing any migration down to levels below of 10 ppb when used for long term storage at room temperature.**

Film structure	Base polymer	Barrier material
15 µm OPA[*]	PA	PA
12 µm PET	PET	PET
12 µm PVDC coated transparent Polyester film	PET	PVDC
PE / EVOH 3 µm / PE total 30 µm	PE	EVOH

[*] This efficiency is only ensured when no swelling occurs

Migratie

- Test condities (Annex V)
  - OM
    - 7 standard condities + 2 aanvullende condities voor lang contact
    - Testen voor screening
  - SM
    - Kort contact, tot maximaal 30 dagen
    - Langdurige opslag, langer dan 30 dagen
    - Combinaties van contact condities
- Simulanten (Annex III)
  - Welk type voedsel?
  - Simulant A, B, D2 → all types of food
  - Vervangede simulanten D2
- Type test
  - Enkelzijdig
  - Onderdompeling (mono layers)
  - Testen voor herhaald gebruik

Vervangende simulanten
Simulant keuze

- EU 10/2011
  - Waarom?
    - Indien technisch niet mogelijk om te testen met D2
      - Zwelling
      - Porositeit
    - Screening
  - Vervangende simulanten
    - 95% ethanol
    - Iso-octaan
    - Tenax
  - Totale migratie
    - Alle simulanten
    - Tenax enkel bij testen vanaf 100°C

    → **Slechtste resultaat is representatief voor D2**
  - Specifieke migratie
    - Worst case op basis van polariteit
- Draft guidance
  - Simulant keuze op basis van "**octanol/water partitioning coefficient**"

What about ethanol 95% and iso-octane ?

Polarity scale based on octanol to water partition coefficients:

Based on the above considerations it can be considered that in most of the cases specific migration testing can be performed with the alternative 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 iso-octane.

European Commission

Vervangende simulanten
Test condities

- 10/2011
  Geen
- 82/711
- Draft guidance
  - Enforcement
  - Screening

Test condition with simulant D	Test conditions with isooctane	Test conditions with ethanol 95 %	Test conditions with MPPO (1)
10 d at 5 °C	0,5 d at 5 °C	10 d at 5 °C	—
10 d at 20 °C	1 d at 20 °C	10 d at 20 °C	—
10 d at 40 °C	2 d at 20 °C	10 d at 40 °C	—
2 h at 70 °C	0,5 h at 40 °C	2,0 h at 60 °C	—
0,5 h at 100 °C	0,5 h at 60 °C (2)	2,5 h at 60 °C	0,5 h at 100 °C
1 h at 100 °C	1,0 h at 60 °C (2)	3,0 h at 60 °C (2)	1 h at 100 °C
2 h at 100 °C	1,5 h at 60 °C (2)	3,5 h at 60 °C (2)	2 h at 100 °C
0,5 h at 121 °C	1,5 h at 60 °C (2)	3,5 h at 60 °C (2)	0,5 h at 121 °C
1 h at 121 °C	2,0 h at 60 °C (2)	4,0 h at 60 °C (2)	1 h at 121 °C
2 h at 121 °C	2,5 h at 60 °C (2)	4,5 h at 60 °C (2)	2 h at 121 °C
0,5 h at 130 °C	2,0 h at 60 °C (2)	4,0 h at 60 °C (2)	0,5 h at 130 °C
1 h at 130 °C	2,5 h at 60 °C (2)	4,5 h at 60 °C (2)	1 h at 130 °C
2 h at 150 °C	3,0 h at 60 °C (2)	5,0 h at 60 °C (2)	2 h at 150 °C
2 h at 175 °C	4,0 h at 60 °C (2)	6,0 h at 60 °C (2)	2 h at 175 °C

Table 1 Polymers that only contain carbon and hydrogen (LDPE, LLDPE, HDPE, PP (homo, random, rubbery), PS, SBS): conventional test conditions and food simulants that has all to be performed when testing in food simulant D2 is technically not feasible

food simulant D2	ethanol 95%	iso-octane	food simulant E
10 d at 5°C	same t/T conditions as for food simulant D2	0.5 d at 5°C	no
1 d at 20°C	same t/T conditions as for food simulant D2	2.5 h at 20°	no
3 d at 20°C	same t/T conditions as for food simulant D2	8 h at 20°C	no
10 d at 20°C	same t/T conditions as for food simulant D2	1 d at 20°	no
1 d at 40°C	same t/T conditions as for food simulant D2	5 h at 20°C	no
3 d at 40°C	same t/T conditions as for food simulant D2	16 h at 20°C	no
10 d at 40°C	same t/T conditions as for food simulant D2	2 d at 20°C	no
[illegible]	same t/T conditions as for food simulant D2	[illegible]	no
10 d at 60°C	same t/T conditions as for food simulant D2	10 d at 20°C	no
2 h at 70°C	4 h at 60°C	0.5 h at 40°C	no
0.5 h at 100°C	12 h at 60°C	0.5 h at 60°C	same t/T conditions as for food simulant D2
1 h at 100°C	1 d at 60°C	1 h at 60°C	same t/T conditions as for food simulant D2
2 h at 100°C	2 d at 60°C	1.5 h at 60°C	same t/T conditions as for food simulant D2

Table 2 Polymers that contain also other atoms than carbon and hydrogen (PET, PBT, PEN, PA6, PA66, PA12, PVC (rigid), PC, PMMA): conventional test conditions and food simulants that has all to be performed when testing in food simulant D2 is technically not feasible

food simulant D2	ethanol 95%	iso-octane	food simulant E
10 d at 5°C	0.5 d at 5°C	same t/T conditions as for food simulant D2	no
1 d at 20°C	2.5 h at 20°	same t/T conditions as for food simulant D2	no
3 d at 20°C	8 h at 20°C	same t/T conditions as for food simulant D2	no
10 d at 20°C	1 d at 20°	same t/T conditions as for food simulant D2	no
1 d at 40°C	5 h at 20°C	same t/T conditions as for food simulant D2	no
3 d at 40°C	16 h at 20°C	same t/T conditions as for food simulant D2	no
10 d at 40°C	2 d at 20°C	same t/T conditions as for food simulant D2	no
[illegible]	[illegible]	same t/T conditions as for food simulant D2	no
10 d at 60°C	10 d at 20°C	same t/T conditions as for food simulant D2	no
2 h at 70°C	0.5 h at 40°C	4 h at 60°C	no
0.5 h at 100°C	0.5 h at 60°C	12 h at 60°C	same t/T conditions as for food simulant D2
1 h at 100°C	1 h at 60°C	1 d at 60°C	same t/T conditions as for food simulant D2
2 h at 100°C	1.5 h at 60°C	2 d at 60°C	same t/T conditions as for food simulant D2

Specifieke migratie

Table 8 test conditions for screening food simulants compared to vegetable oil for specific migration

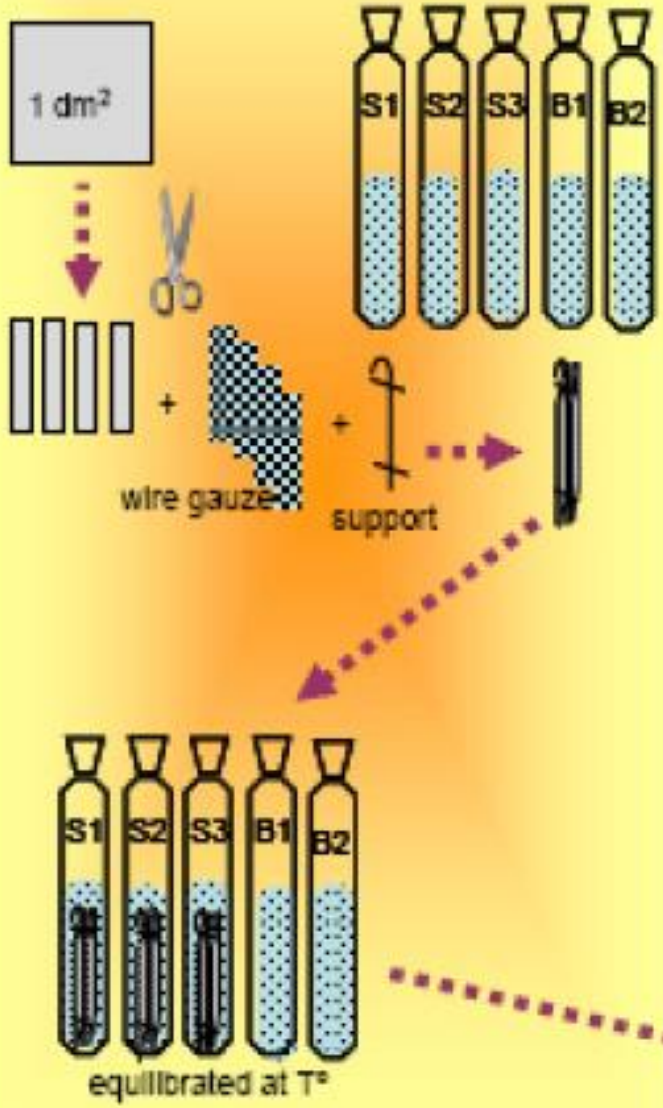
plastic	vegetable oil	ethanol 95%	iso-octane	food simulant E
LDPE, LLDPE PP random PP rubbery	@ ≥ 100°C			same t/T conditions as for vegetable oil
	10d @ 60°C	10d @ 60°C	2d @ 40°C	
	10d @ 40°C	10d @ 40°C	2d @ 20°C	
	10d @20°C	10d @ 20°C	1d @ 20°	
	–	–		
HDPE	@ ≥ 100°C			same t/T conditions as for vegetable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

Totale migratie

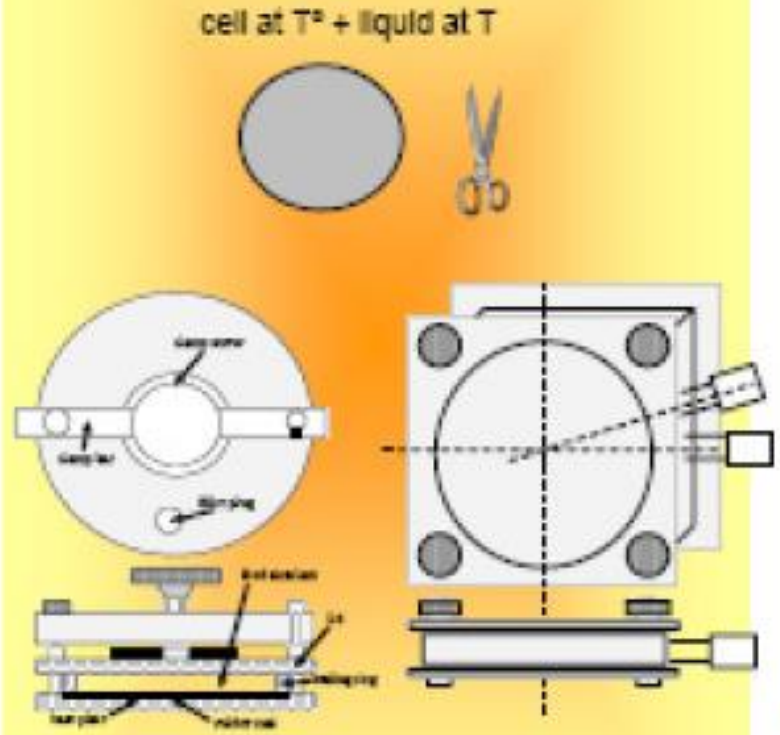
Table 12 Test conditions for screening food simulants compared to vegetable oil for overall migration

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
	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

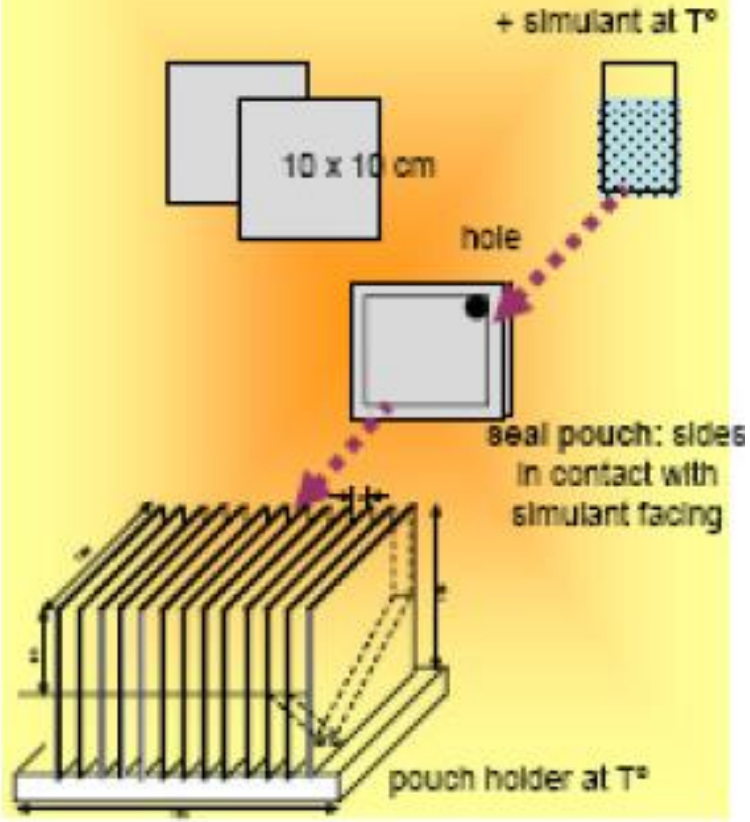
Double face testing by immersion



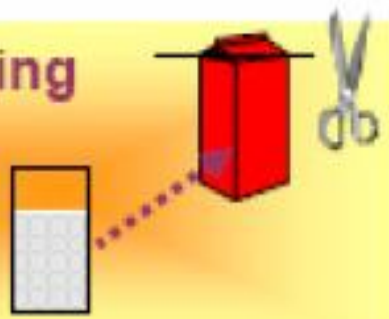
Single surface testing using cells



Single surface testing using pouches



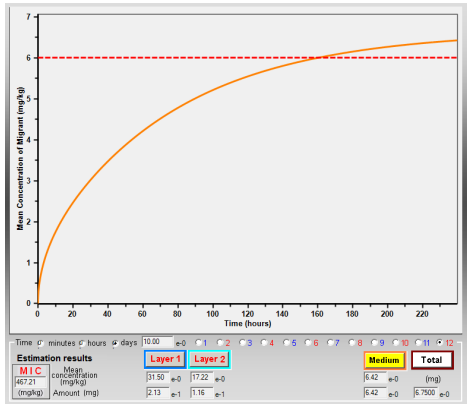
article filling

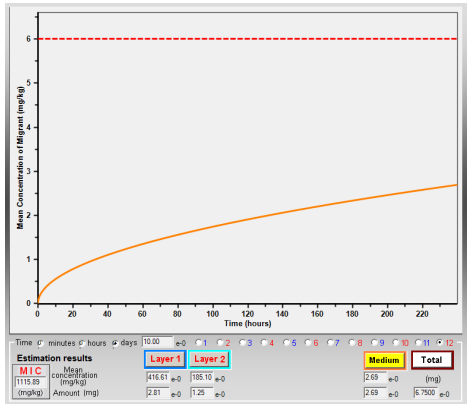


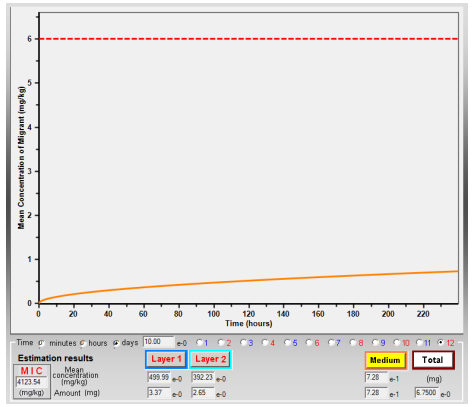
EXPOSURE (t, T)

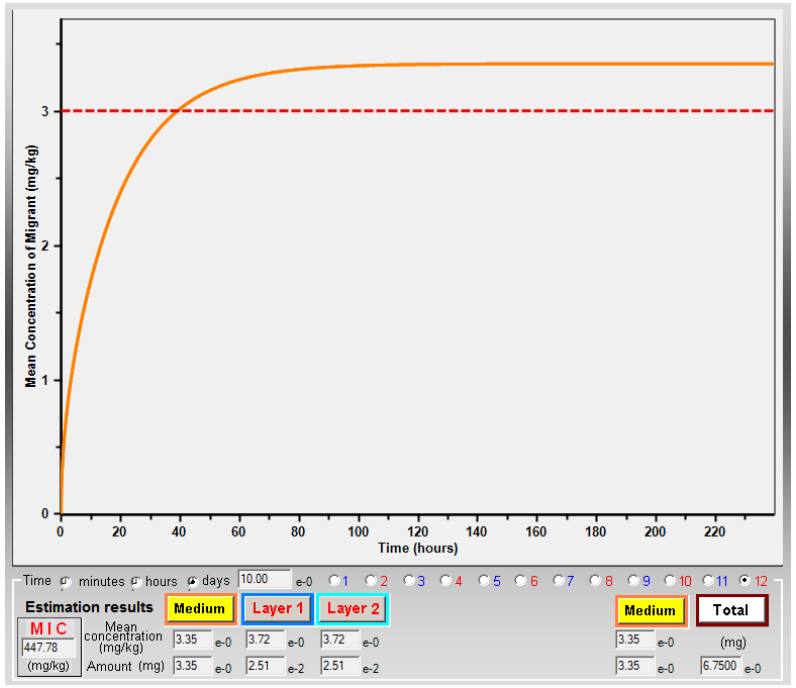
Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) PP homo: 250 micron (density 0,9 g/cm^3) S/V ratio: 6 dm^2/kg food	Condities: - 10d@ 60°C - 10d@ 40°C - 10d@ 20°C	Resultaat: mg/kg food - **6,42 / 6,70** - 2,69 / 5,14 - 0,73 / 1,46

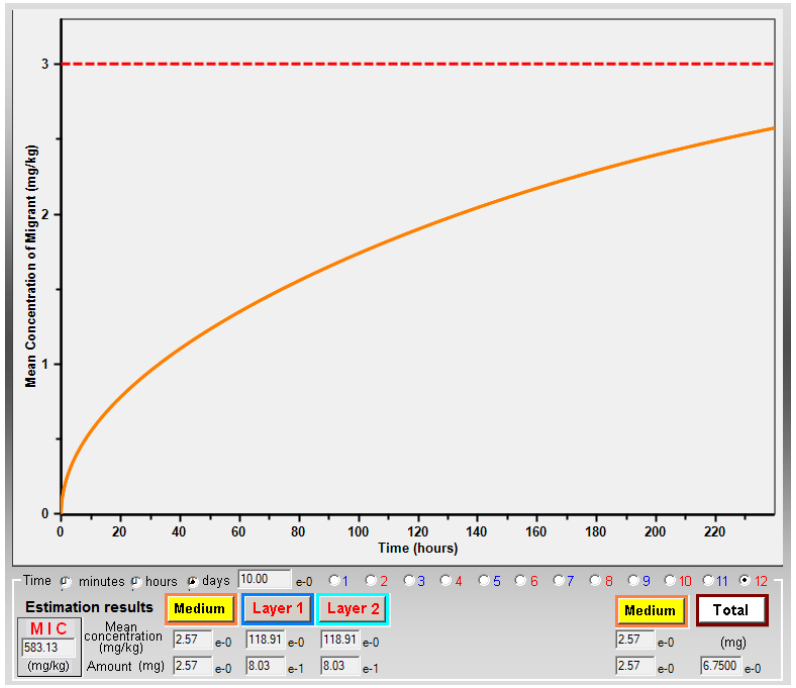
Enkelzijdig

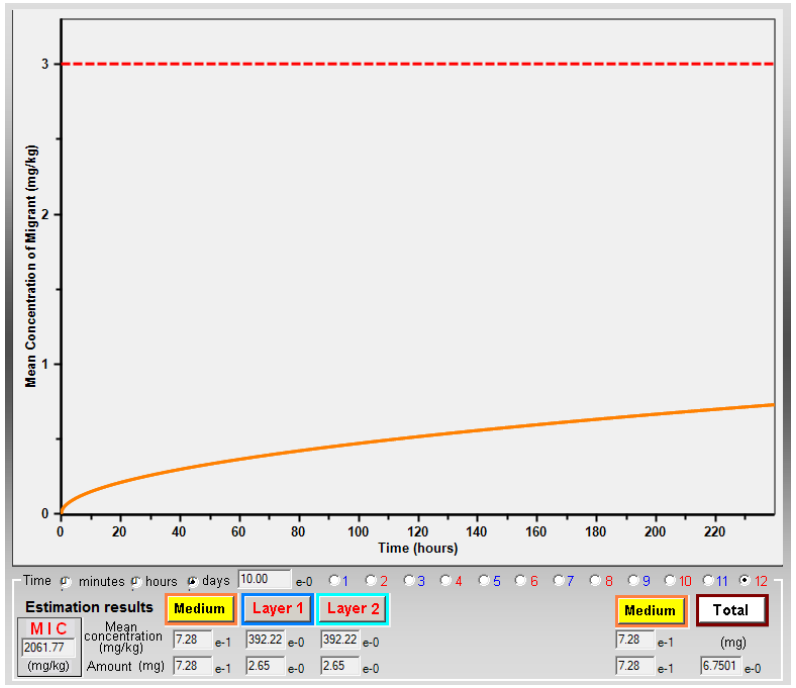


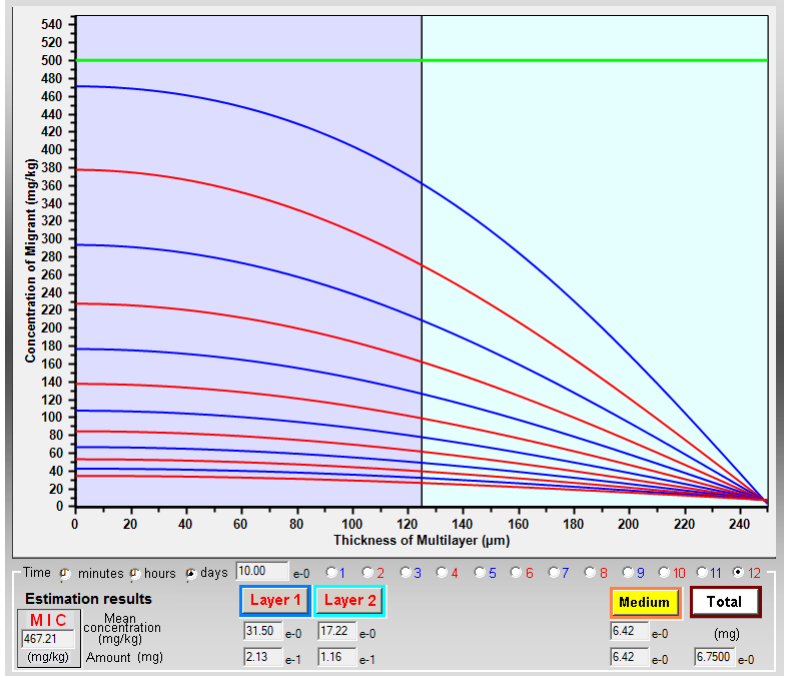


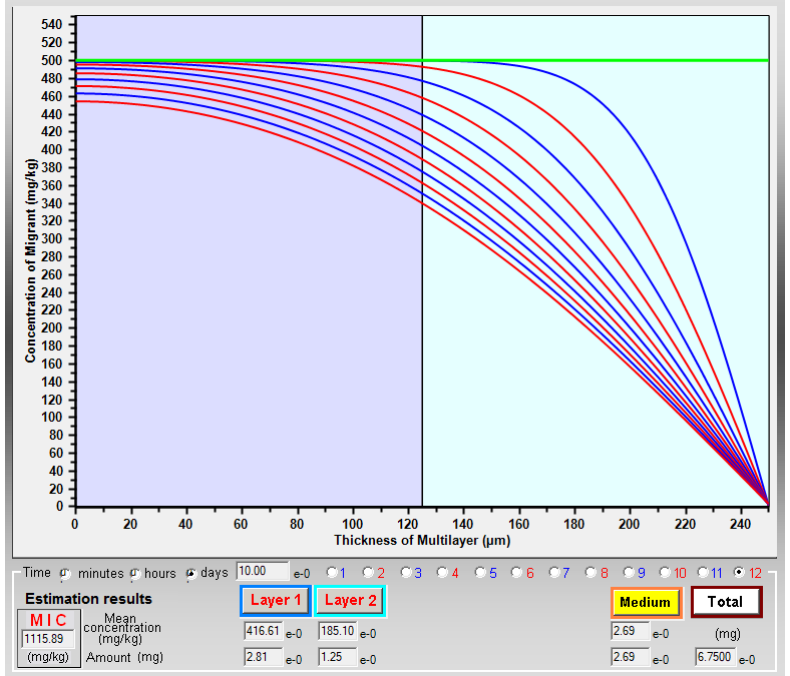


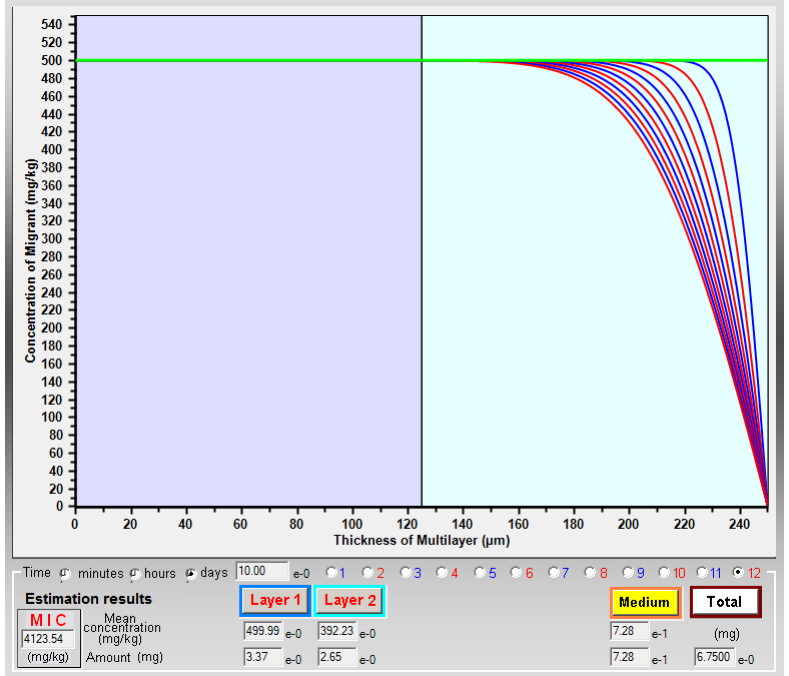


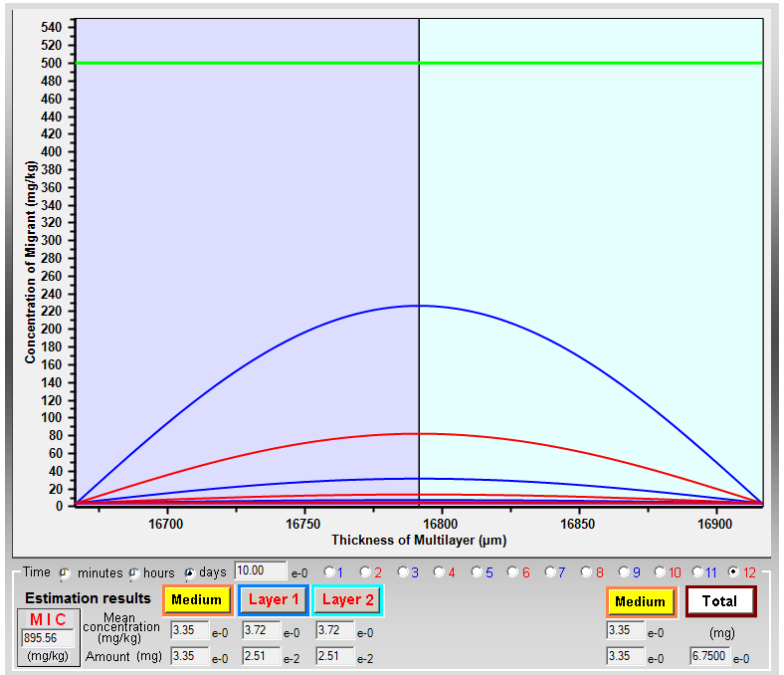


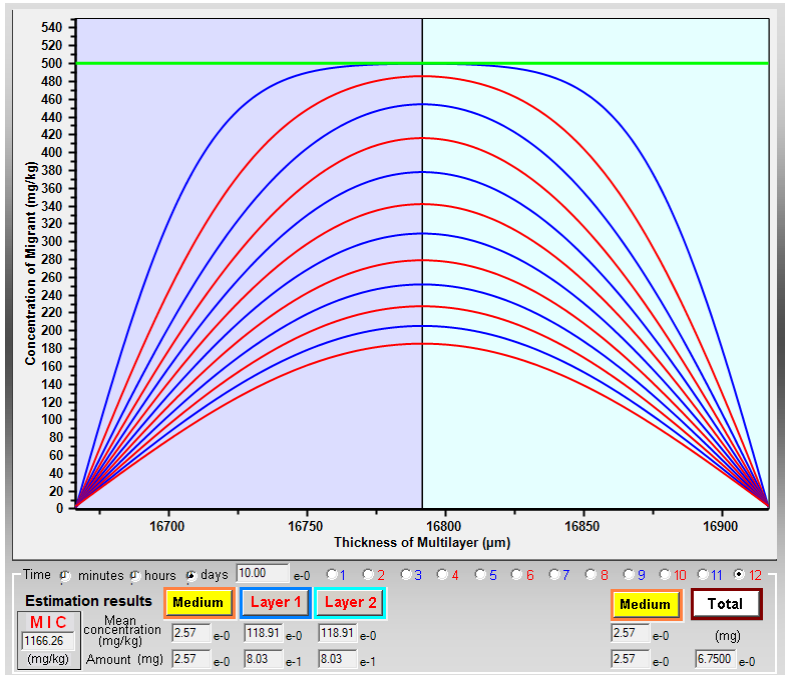


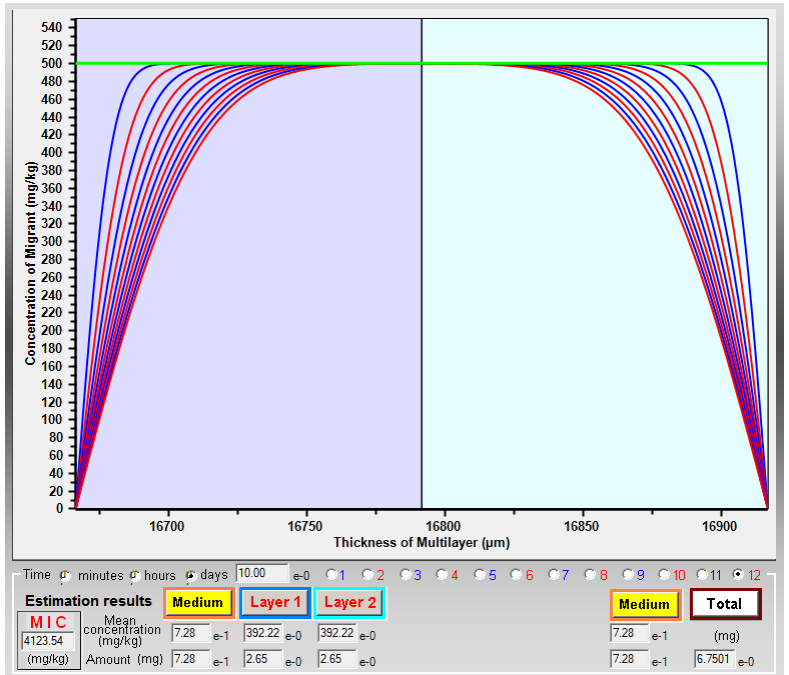

Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 467.21 (mg/kg)
Mean concentration (mg/kg) 31.50 e-0 17.22 e-0 6.42 e-0 (mg)
Amount (mg) 2.13 e-1 1.16 e-1 6.42 e-0 6.7500 e-0
Layer 1 Layer 2 Medium Total


Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 1115.89 (mg/kg)
Mean concentration (mg/kg) 416.61 e-0 185.10 e-0 2.69 e-0 (mg)
Amount (mg) 2.81 e-0 1.25 e-0 2.69 e-0 6.7500 e-0
Layer 1 Layer 2 Medium Total

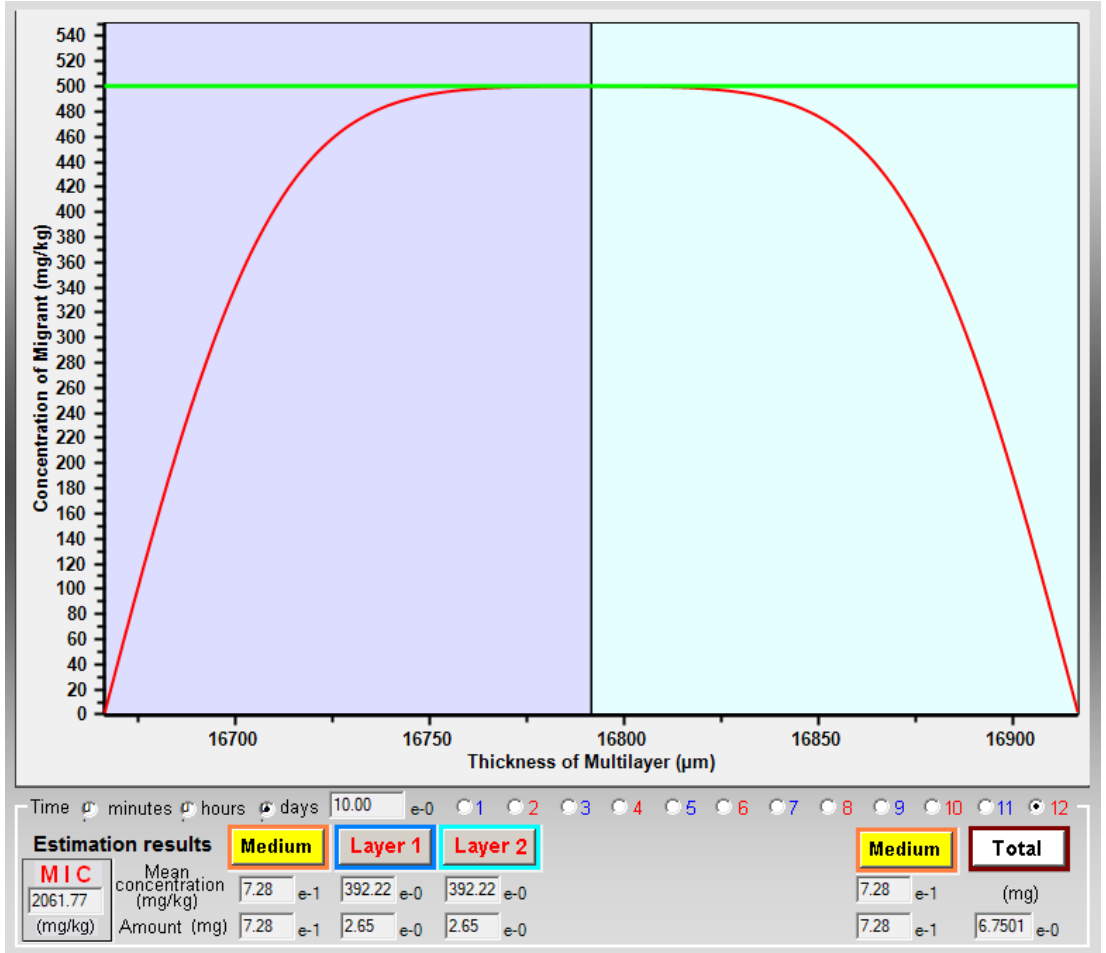

Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 4123.54 (mg/kg)
Mean concentration (mg/kg) 499.99 e-0 392.23 e-0 7.28 e-1 (mg)
Amount (mg) 3.37 e-0 2.65 e-0 7.28 e-1 6.7500 e-0
Layer 1 Layer 2 Medium Total

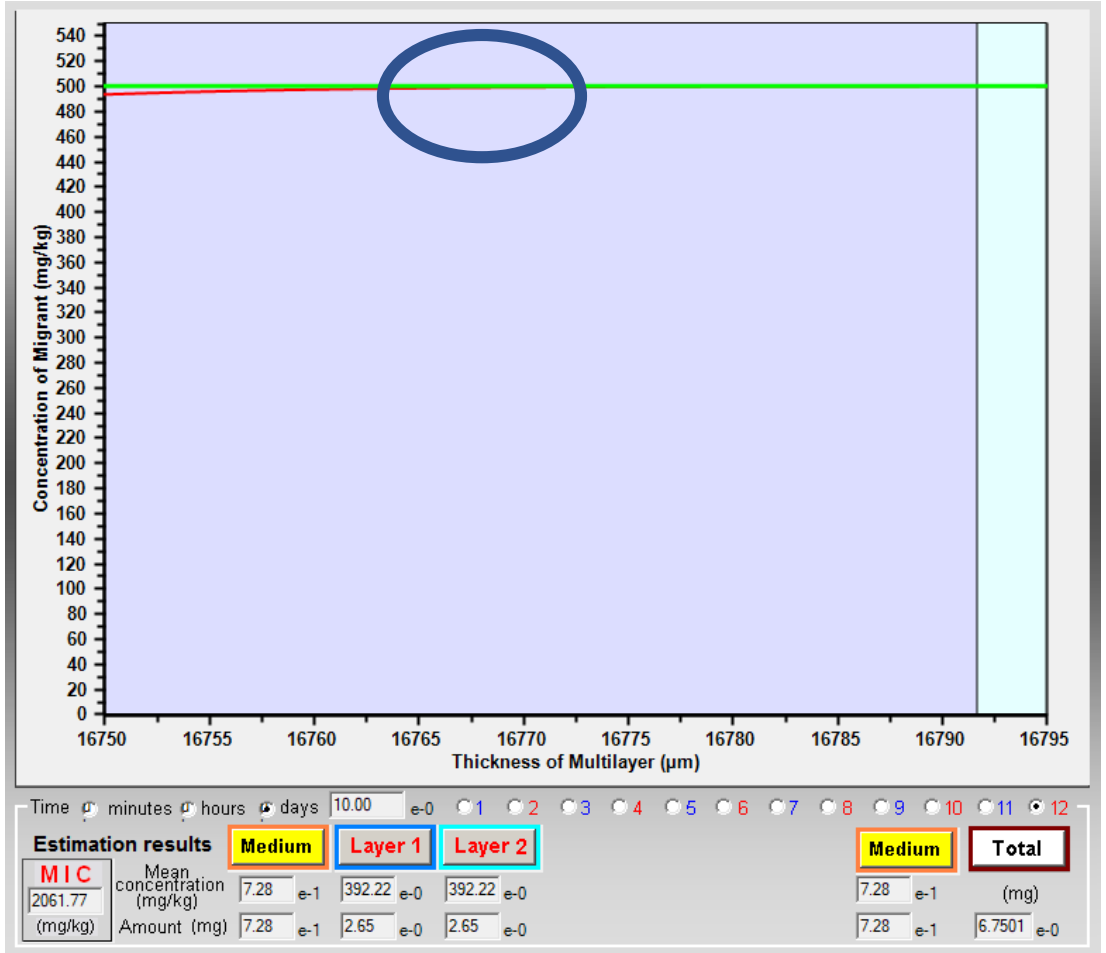

Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 895.56 (mg/kg)
Mean concentration (mg/kg) 3.35 e-0 3.72 e-0 3.72 e-0 (mg)
Amount (mg) 3.35 e-0 2.51 e-2 2.51 e-2 3.35 e-0 6.7500 e-0
Medium Layer 1 Layer 2 Medium Total


Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 1166.26 (mg/kg)
Mean concentration (mg/kg) 2.57 e-0 118.91 e-0 118.91 e-0 2.57 e-0 (mg)
Amount (mg) 2.57 e-0 8.03 e-1 8.03 e-1 2.57 e-0 6.7500 e-0
Medium Layer 1 Layer 2 Medium Total

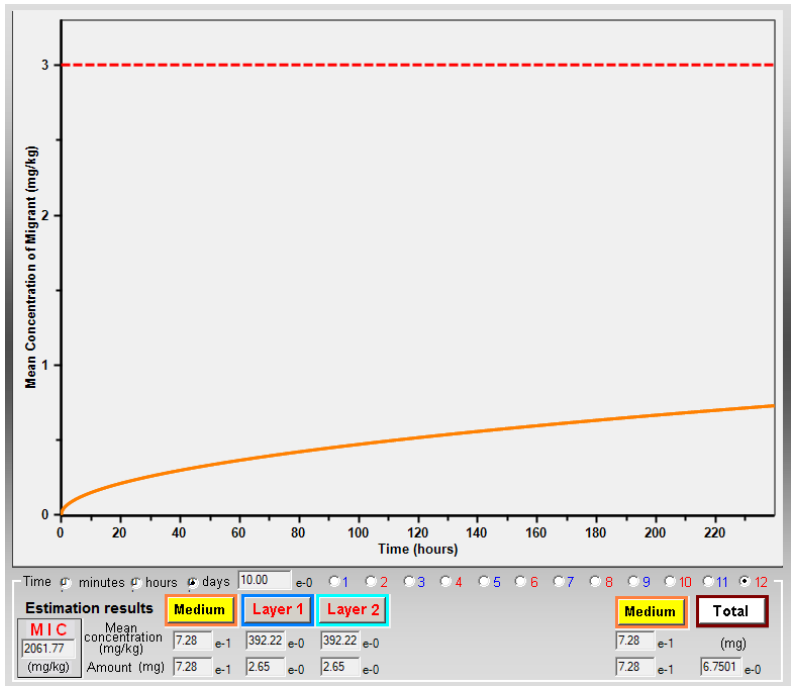

Concentration of Migrant (mg/kg)
Thickness of Multilayer (μm)
Time minutes hours days 10.00 e-0 1 2 3 4 5 6 7 8 9 10 11 12
Estimation results
MIC 4123.54 (mg/kg)
Mean concentration (mg/kg) 7.28 e-1 392.22 e-0 392.22 e-0 7.28 e-1 (mg)
Amount (mg) 7.28 e-1 2.65 e-0 2.65 e-0 7.28 e-1 6.7501 e-0
Medium Layer 1 Layer 2 Medium Total

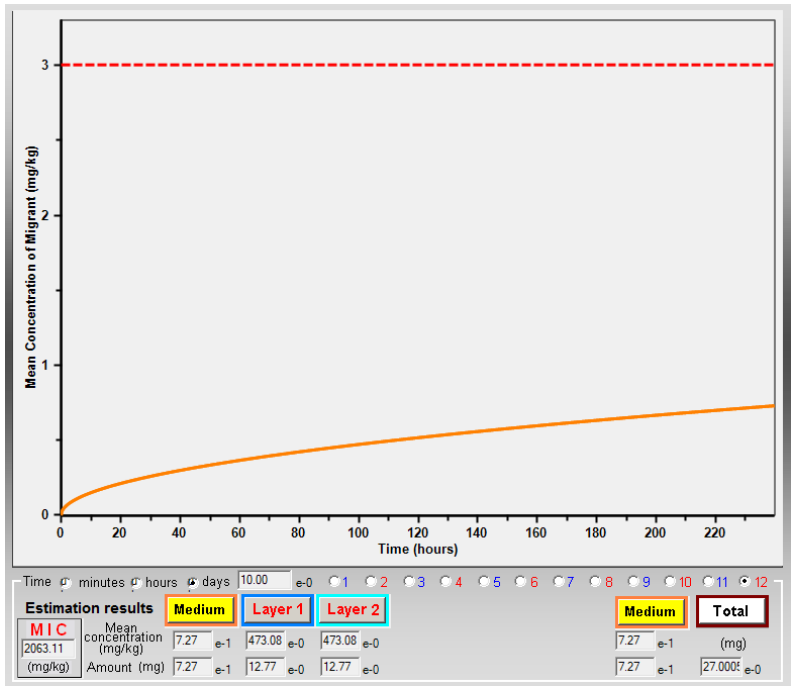
Kritische dikte waarbij migratie van beide zijden mag verondersteld worden.
Migratie = migratie resultaat / 2
Grotere dikte zal het resultaat NIET meer veranderen





PP homo (density 0,9 g/cm^3) Stof 1: Molecular Mass = 500 Da Concentratie: 500 ppm (mg/kg FCM) Condities: 10d@ 60^{o}C S/V ratio: 6 dm^2/kg food	Polymeer dikte - 250 micron - 500 micron - 1000 micron	Resultaat: mg/kg food - **1,46** - 1,46 - 1,46





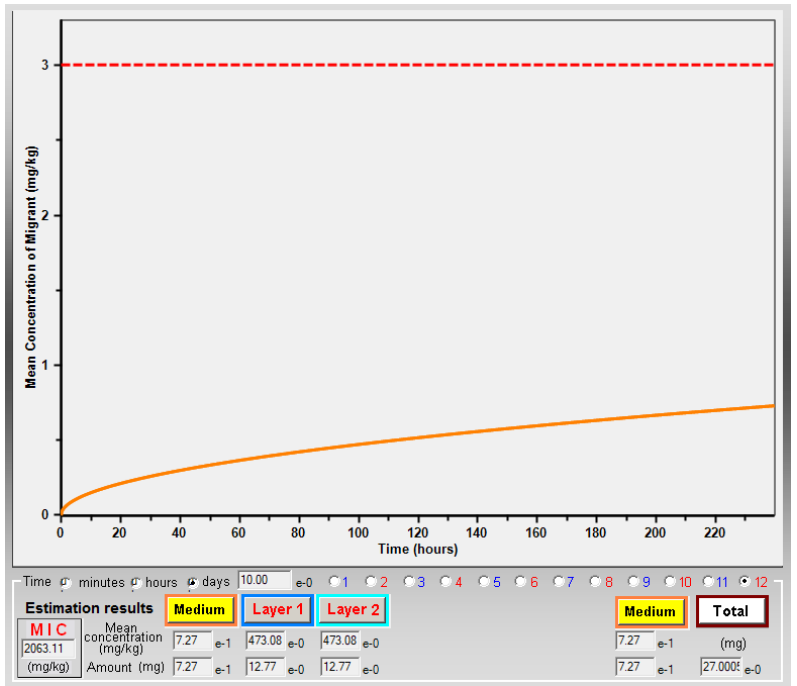
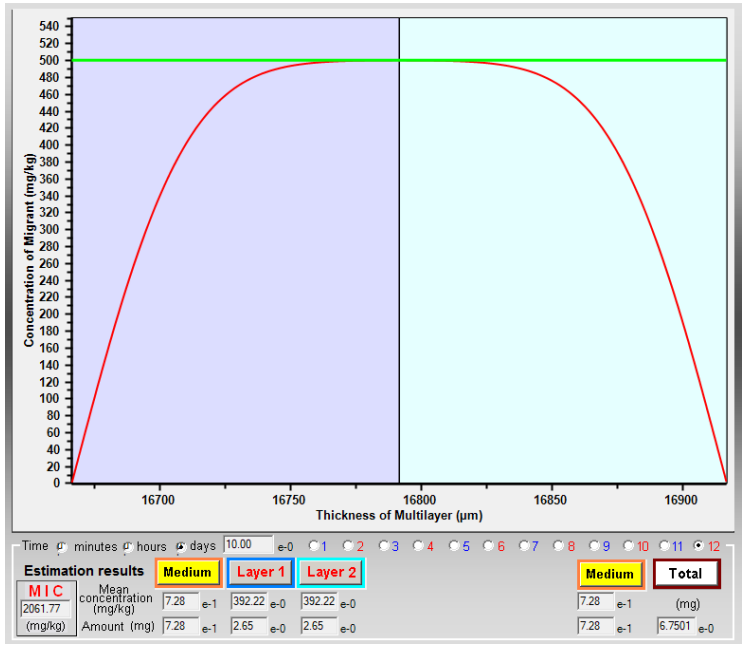
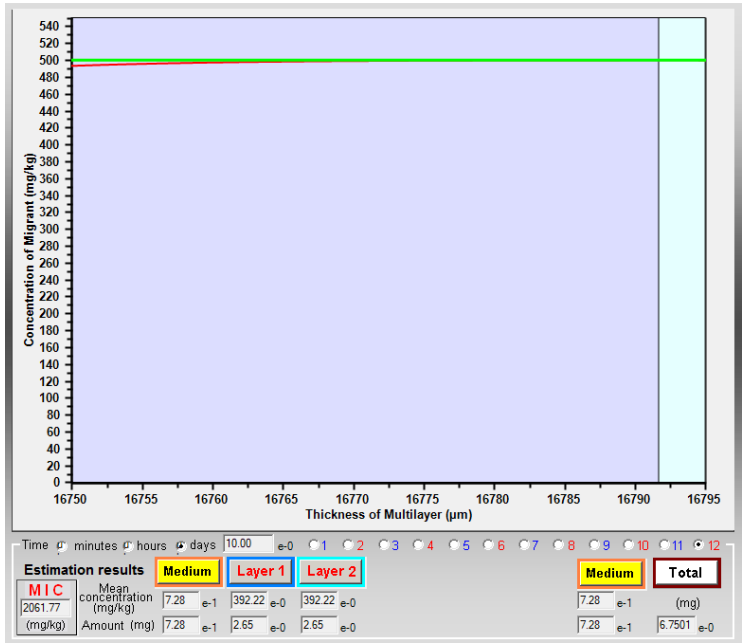


Table 5 Layer thickness L (in µm) and above which both side of the sample can be considered for calculation of migration if tested by full immersion at different contact conditions for four different molecular mass ranges

Polymer type	time/Temp	layer thickness L in [µm] for			
molecular mass of migrant (g/mol)		100-250	251-500	501-750	751-1000*
LDPE, PP blockcopolymer [5]	10 d at 60°C	none	none	9600	3840
	10 d at 40°C	none	12000	3680	1440
	10 d at 20°C	10000	3520	1200	480
	2h at 100°C	none	16000	4880	1920
HDPE	10 d at 60°C	none	13700	4200	1680
	10 d at 40°C	11800	4800	1320	540
	10 d at 20°C	3200	1200	400	168
	2h at 100°C	none	8600	2640	1040
PP isotactic/homo PP random	10 d at 60°C	20000	6800	2080	840
	10 d at 40°C	5840	2200	680	288
	10 d at 20°C	1600	620	220	80
	2h at 100°C	11700	4320	1320	540
PET, PBT, PEN	10 d at 60°C	160	60	20	8
	10 d at 40°C	52	20	8	4
	10 d at 20°C	12	8	4	2
	2h at 100°C	100	40	12	6





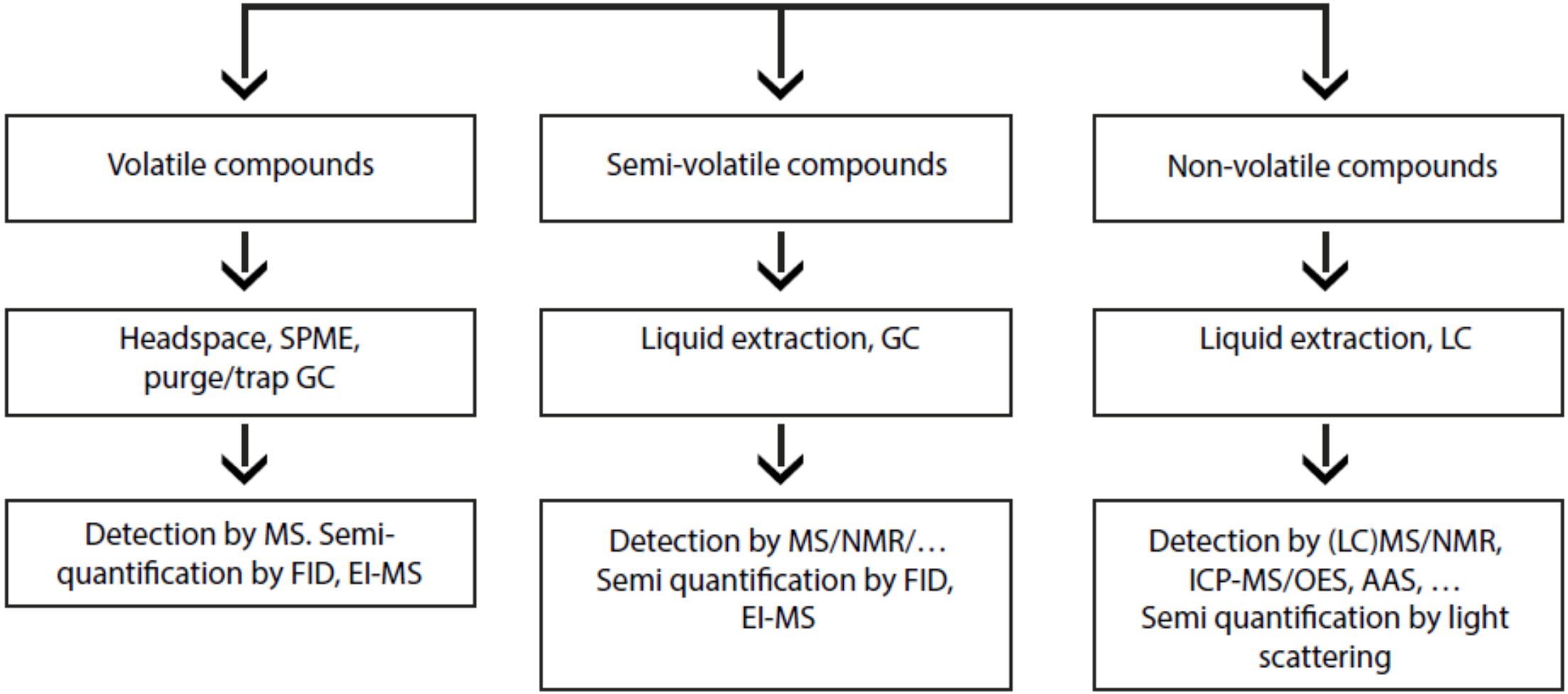
Old EFSA document: Migration from food only takes place the first 250 micron.

Labo's delen resultaten bij dikte > 500 micron!

Analyse

- Specifieke migratie
  - Screening test
    - Niet- stof specifieke test met lagere nauwkeurigheid
    - Semi-kwantitatief via interne standaarden
  - Target test
    - Stof specifiek met grote nauwkeurigheid
    - Kwantitatieve bepaling op basis van geijkte methode
- LOD (detectielimiet)
- LOQ (kwantificatielimiet)
- Meetonnauwkeurigheid
  - Totale migratie
    - Waterige simulanten: 20% (2 mg/dm^2)
    - Plantaardige olie: 30% (3 mg/dm^2)
  - Specifieke migratie
    - Test specifiek

Chemische analyse technieken



Meetonnauwkeurigheid

- Guidance on migration testing
  Vanuit een handhavingsstandpunt: het individuele testresultaat minus de analytische onzekerheid (uitgebreide meetonzekerheid) moet boven de wettelijke limiet liggen om een steekproef te beschouwen als niet-compliant.

- Non-compliance in voedsel gaat boven compliance op food simulanten

Rapportage

Minimum information in the test report

- Identification of the sample (i.e. consignment number, batch number, sample number)
- All information necessary for complete description of the sample, e.g. chemical type, trade mark, grade, dimensions, shape etc – pictures should be included together with a ruler.
- Date and method of sampling
- Name of laboratory; Name of person responsible for analysis; Date of report;
- Analyte(s); A reference to the method(s) used.
- **The type of the migration test (i.e. immersion or article fill, number of contacts)**;
- The duration and temperature;
- **The surface area exposed and volume of food simulant used**;
- **The individual test results, expressed in the correct units. Expanded measurement uncertainty should be reported (used S/V ratio for reporting)**
- Any relevant comments on the test results;
- Details of any confirmation procedure(s), if any.
- Any deviations from the standards

S/V ratio

Artikel 17

Uitdrukking van de resultaten van migratietesten

1. Om de overeenstemming na te gaan worden de specifieke migratiewaarden uitgedrukt in mg/kg onder gebruikmaking van de werkelijke oppervlakte-volumeverhouding bij feitelijk of te verwachten gebruik.

2. In afwijking van lid 1 wordt voor:

a) recipiënten en andere voorwerpen die minder dan 500 ml of gram, dan wel meer dan 10 l bevatten of voor dergelijke hoeveelheden bestemd zijn,

b) materialen en voorwerpen waarvoor vanwege hun vorm een schatting van de relatie tussen de oppervlakte van dergelijke materialen of voorwerpen en de hoeveelheid levensmiddelen die hiermee in contact komt, onuitvoerbaar is,

c) folie en film die nog niet met levensmiddelen in contact komen,

d) folie en film die minder dan 500 ml of gram, dan wel meer dan 10 l bevatten,

de migratie uitgedrukt in mg/kg onder gebruikmaking van een oppervlakte-volumeverhouding van 6 dm^2 per kg levensmiddel.

De eerste alinea is niet van toepassing op materialen en voorwerpen van kunststof die bestemd zijn om in contact te komen of al in contact komen met levensmiddelen voor zuigelingen en peuters zoals omschreven in de Richtlijnen 2006/141/EG en 2006/125/EG.

Omrekenen van het migratie resultaat wanneer de S/V ratio van de test niet gelijk is aan de werkelijke (worst case) S/V ratio,

$$M_{S/V} = M_{test} \times \frac{S/V_{(actual)}}{S/V_{(test)}}$$

Vetreductiecoëfficiënten

- D2 reductie factor (DRF)
  Standaard EU consumptie van 100% vet = 0,2 kg/person/day
  - Consumptie factor afhankelijk van de hoeveelheid vet in het voedsel
    FRF = (g vet in levensmiddel/kg levensmiddel)/200 = (% vet × 5)/100
    Bv mayonaise met 73% vet: DFR = 73*5/100 = 3,65%
  - Resultaat van migratietest in D2, mag gedeeld worden door consumptie factor
  - Niet van toepassingen indien SML=ND en stoffen achter FB

A. patates frites, oliebollen en dergelijke	X				X/5
B. van dierlijke oorsprong	X				X/4

- Stof specifieke vet reductie factor (FRF)
  - Enkel voor SM van lipofiele stoffen
  - Niet van toepassing bij
    - “Baby food”
    - Producten waarvan S/V ratio niet kan bepaald worden (Art. 17.2(b))
    - Migratie voor toepassen van FRF<OM

- Totale reductie factor
  - TRF = DRF x FRF ≤ 5
  - $M_{TRF} = M_{S/V} / TRF$

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
FCM-stofnr.	Ref.-nr.	CAS-nr.	Naam van de stof	Gebruik als additief of polymerisatiehulpmiddel (ja/neen)	Gebruik als monomeer of andere uitgangsstof of door microbiële fermentatie verkregen macromolecuul (ja/neen)	FRF van toepassing (ja/neen)	SML [mg/kg]	SML(T) [mg/kg] (groepsbeperking nr.)	Beperkingen en specificaties	Noten betreffende de controle op de naleving
1	12310	0266309-43-7	albumine	neen	ja	neen				
2	12340	—	albumine, gecoaguleerd door formaldehyd	neen	ja	neen				

Baby food

- Overall migratie
  - Uitgedrukt in mg/kg food
  - Werkelijke S/V ratio
  - Limiet = 60 mg/kg
- Specifieke migratie
  - Werkelijke S/V ratio (artikel 17.2 is niet van toepassing)
  - Geen FRF van toepassing

NRK Verpakkingen

Kris Callaert
NRK Projectmanager kunststofverpakkingen en voedselcontact

T: 0032 475 70 32 36
E: callaert@nrk.nl
Bereikbaar via Viaware: 0031 (0)74 76 00 420

VIAWARE
FOOD CONTACT SAFETY