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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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Abstract

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Draft for consultation

1 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 <input type="checkbox"/> Lot number: <input type="checkbox"/> Batch number: <input type="checkbox"/> Item number: <input type="checkbox"/> EU control number: <input type="checkbox"/> Custom number: <input type="checkbox"/> Other:	Type of sample <input type="checkbox"/> Article <input type="checkbox"/> Material <input type="checkbox"/> Starting substances <input type="checkbox"/> Product from intermediate stages of manufacturing	Type of business <input type="checkbox"/> Producer <input type="checkbox"/> Manufacture and Converter <input type="checkbox"/> Importer <input type="checkbox"/> Wholesale trade <input checked="" type="checkbox"/> Retailer <input type="checkbox"/> 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: <input type="checkbox"/> Set-off?
Sampling temperature During sampling: °C Surrounding temperature: °C	Transport of sample <input type="checkbox"/> Not cooled <input type="checkbox"/> Cooled at: °C <input type="checkbox"/> Freezed at: °C <input type="checkbox"/> Sealed Sealed sample arrived undamaged: <input type="checkbox"/> Yes <input type="checkbox"/> No Transport of sample via <input type="checkbox"/> Courier <input type="checkbox"/> Inspectors <input type="checkbox"/> Authorized and instructed person:	Spot of Sampling <input type="checkbox"/> Production / -plant: Details: <input type="checkbox"/> Storage <input type="checkbox"/> Sale <input type="checkbox"/> Self-Service <input type="checkbox"/> Cooling unit <input type="checkbox"/> Freezer Possibility of Set-Off <input type="checkbox"/> Yes <input type="checkbox"/> No Details:

Photo taken at the spot of sampling <input type="checkbox"/> Yes, see annex <input type="checkbox"/> No	Reason of sampling <input type="checkbox"/> Routine sample <input type="checkbox"/> Suspicion sample <input type="checkbox"/> Appeal sample <input type="checkbox"/> Import sample <input type="checkbox"/> Import suspicion sample <input type="checkbox"/> Traceability sample <input type="checkbox"/> Sample in the context of Declaration of compliance	Request for Declaration of Compliance <input type="checkbox"/> Yes <input type="checkbox"/> 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

Draft for consultation

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. Criteria 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.

32 **Annex 3 Cleaning procedures for food simulant E**

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

37 Method 1 using acetone (Beldi et al., 2012)

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

47

48 Method 2 using diethylether.

49 The extraction is carried as follows:

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

55

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

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

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

62

63 Annex 4 Background Table 5, 7, 9

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

67 Introduction

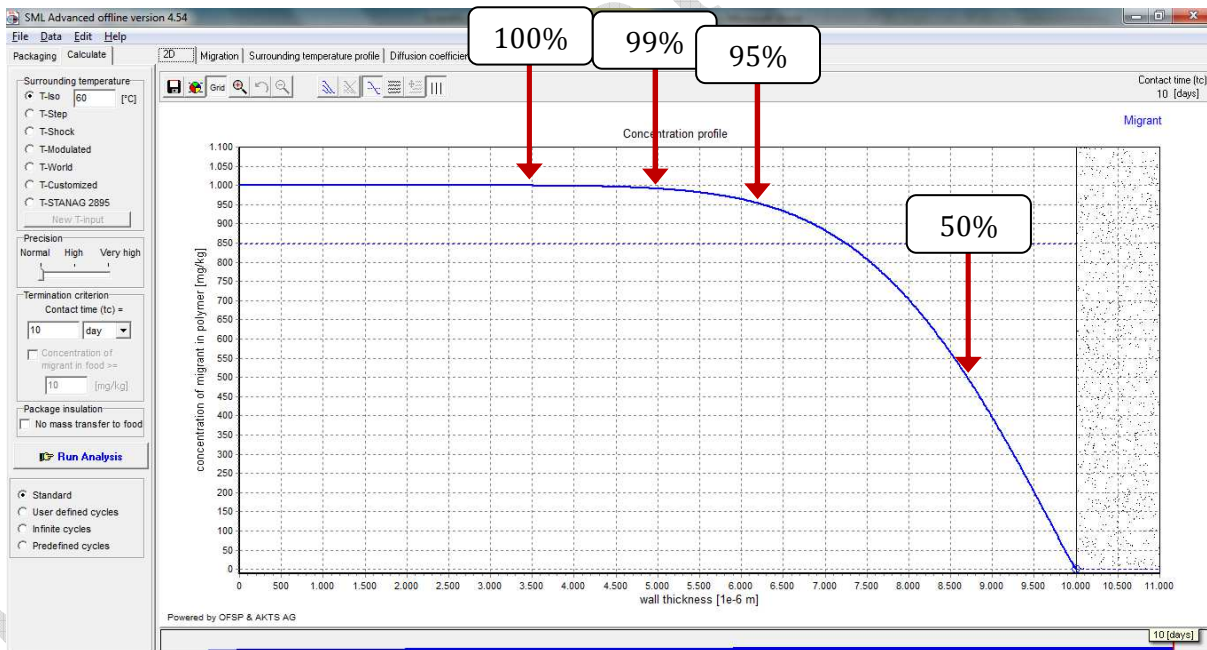
68 The maximum layer thickness (L_{WC} in μm) for worst case migration calculation is 1/2 of
69 the 99% layer thickness.

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

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

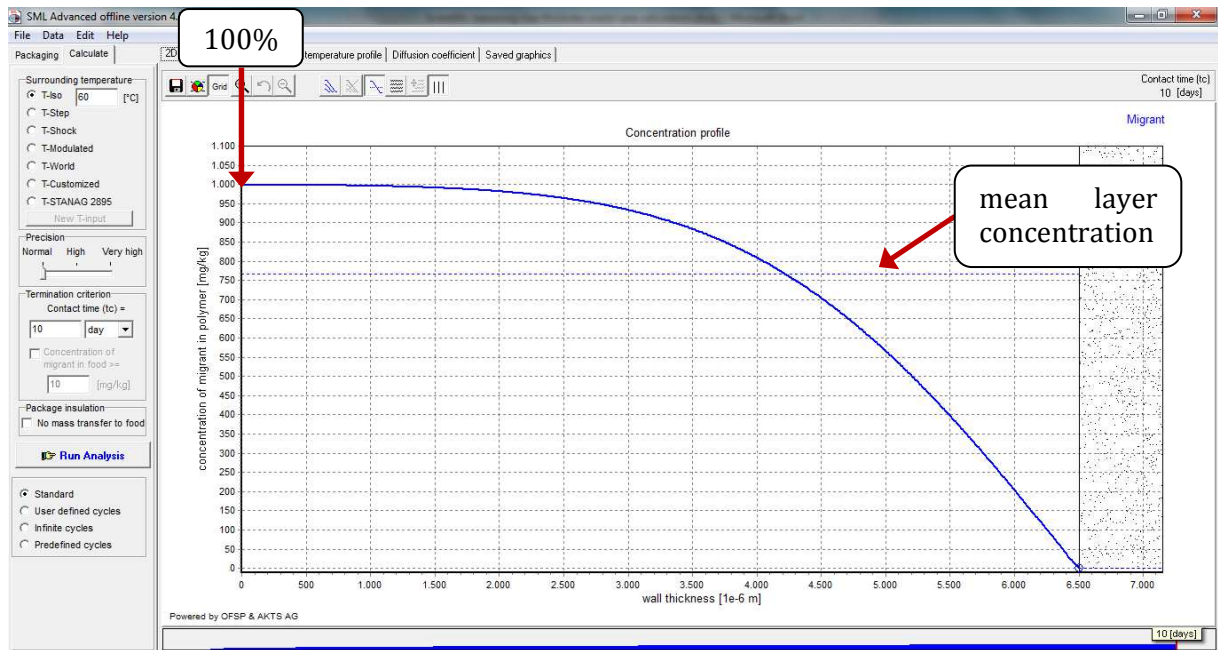
75 To explain the calculation procedure a LDPE monolayer with material thickness 10000 μm (=
76 10 cm) and "upper limit" polymer specific constant $A_p' = 11.5$; $\tau = 0$ was selected. The loss
77 of migrant (specific migration to an infinite contact medium) with molecular mass of 500
78 g/mol (initial concentration 1000 mg/kg plastic) after 10 days at 60°C was calculated. The
79 concentration profile is shown in the figure below.

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



84

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



88

89 For a material with 100% layer thickness (in this example 6500 μm for LDPE) approximate
 90 23.3% = (initial concentration 1000 ppm - mean layer concentration 767 / initial concentration
 91 1000 x 100) of the initial amount of the substance (in this example 500 g/mol molecular mass
 92 of migrant) left the material under the time temperature conditions considered (in this
 93 example 10 days at 60°C).

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

95

96 **LDPE; PP, rubbery**

97 **► molecular mass 100 - 250 g/mol**

98 **10d @ 60°C**

99 => 100% layer thickness = full length

100 no absolute barrier at thicknesses below 10000 μm

101 => 99% layer thickness = full length

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

104 => 2 x 99% layer thickness = none

105 only one side to be considered for calculation of migration if full immersion testing is applied

106

107

108 **10d @ 40°C**

109 => 100% layer thickness = full length

110 no absolute barrier at thicknesses below 10000 μm

111 => 99% layer thickness = full length

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

114 => 2 x 99% layer thickness = none

115 only one side to be considered for calculation of migration if full immersion testing is applied

116

117

118 **10d @ 20°C**

119 => 100% layer thickness = 7000 µm
120 no absolute barrier at thicknesses below 7000 µm

121 => 99% layer thickness = 5000 µm

122 => 1/2 x 99% layer thickness = 2500 µm
123 to be used for worst case calculation of specific migration under assumption of total transfer

124 => 2 x 99% layer thickness = 10000 µm
125 above 10000 µm two sides to be considered for calculation of migration if full immersion
126 testing applied

127

128

129 **2h @ 100°C**

130 => 100% layer thickness = full length
131 no absolute barrier at thicknesses below 10000 µm

132 => 99% layer thickness = full length

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

135 => 2 x 99% layer thickness = none
136 only one side to be considered for calculation of migration if full immersion testing is applied

137

138

139 ► molecular mass 251 - 500 g/mol

140 10d @ 60°C

141 => 100% layer thickness = full length

142 no absolute barrier at thicknesses below 10000 µm

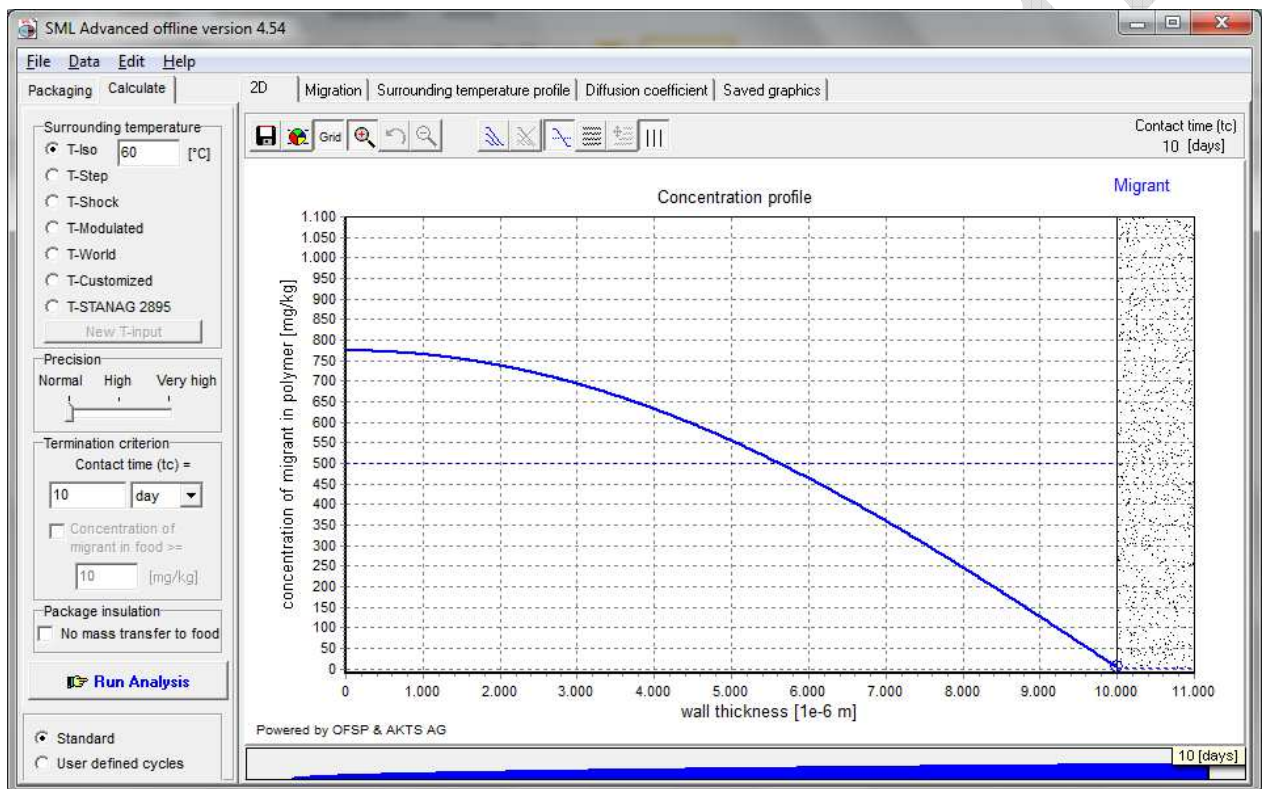
143 => 99% layer thickness = full length

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

146 => 2 x 99% layer thickness = none

147 only one side to be considered for calculation of migration if full immersion testing applied

148



149

150

151

152 **10d @ 40°C**

153 => 100% layer thickness = 8800

154 no absolute barrier at thicknesses below 8800 µm

155 => 99% layer thickness = 6000 µm

156 => 1/2 x 99% layer thickness = 3000 µm

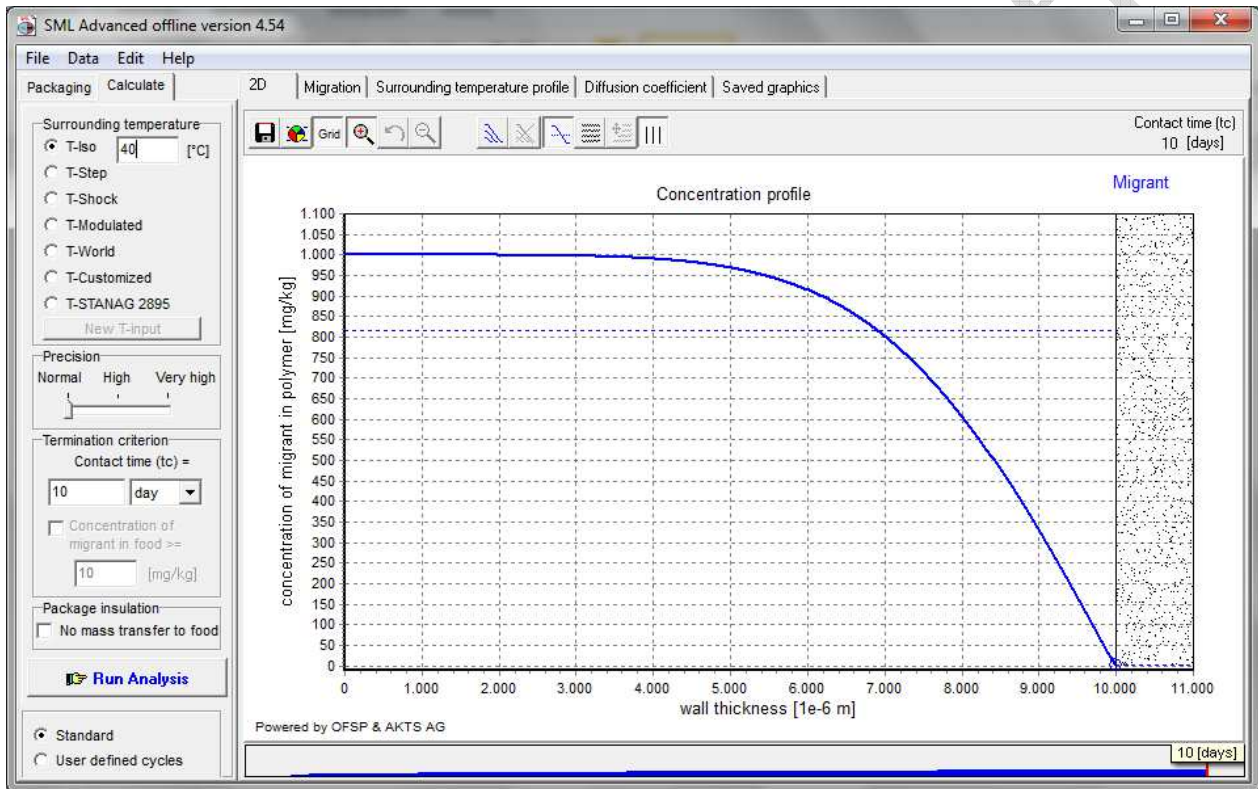
157 to be used for worst case calculation of specific migration under assumption of total transfer

158 => 2 x 99% layer thickness = 12000

159 above 12000 µm two sides to be considered for calculation of migration if full immersion

160 testing applied

161



162

163

164

165 **10d @ 20°C**

166 => 100% layer thickness = 3000 µm

167 no absolute barrier at thicknesses below 3000 µm

168 => 99% layer thickness = 1760 µm

169 => 1/2 x 99% layer thickness = 880 µm

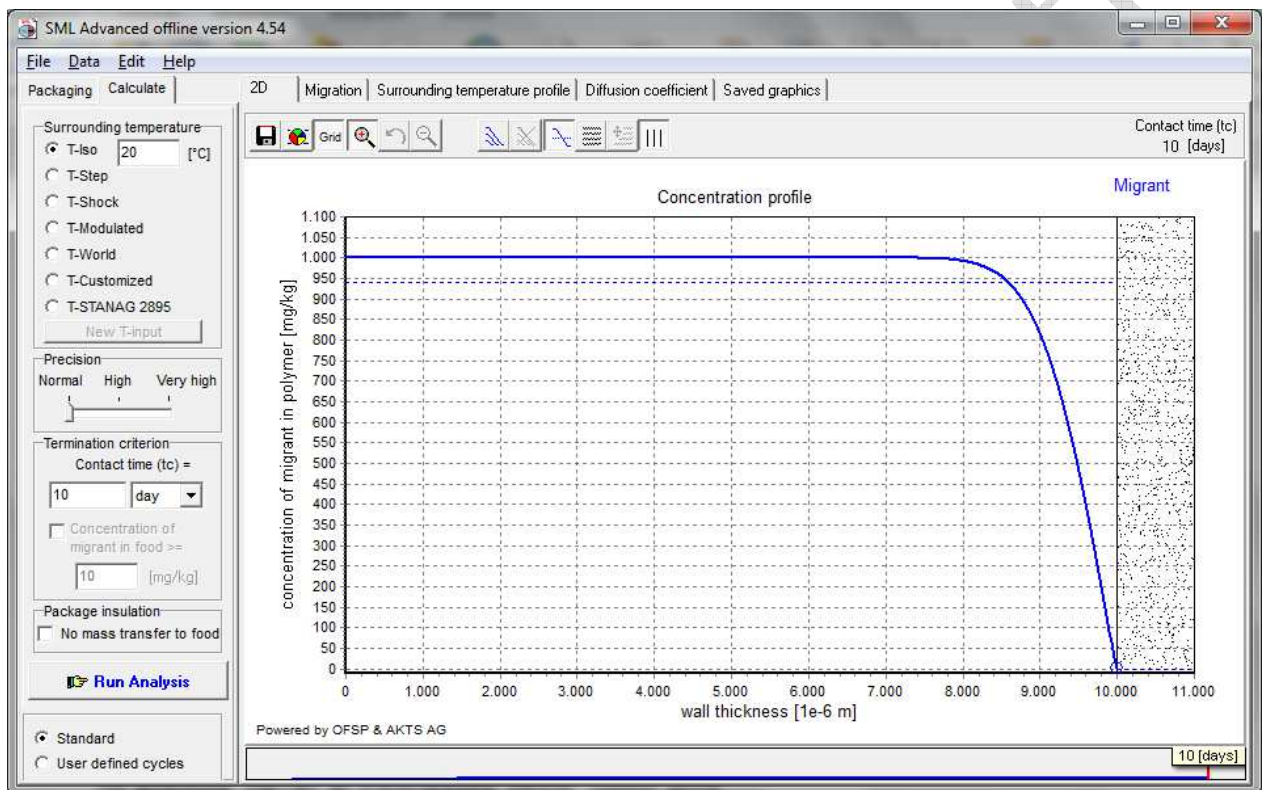
170 to be used for worst case calculation of specific migration under assumption of total transfer

171 => 2 x 99% layer thickness = 3520 µm

172 above 3520 µm two sides to be considered for calculation of migration if full immersion

173 testing applied

174



175

176

177

178 **2h @ 100°C**

179 => 100% layer thickness = 10000

180 no absolute barrier at thicknesses below 10000 µm

181 => 99% layer thickness = 8000

182 => 1/2 x 99% layer thickness = 4000

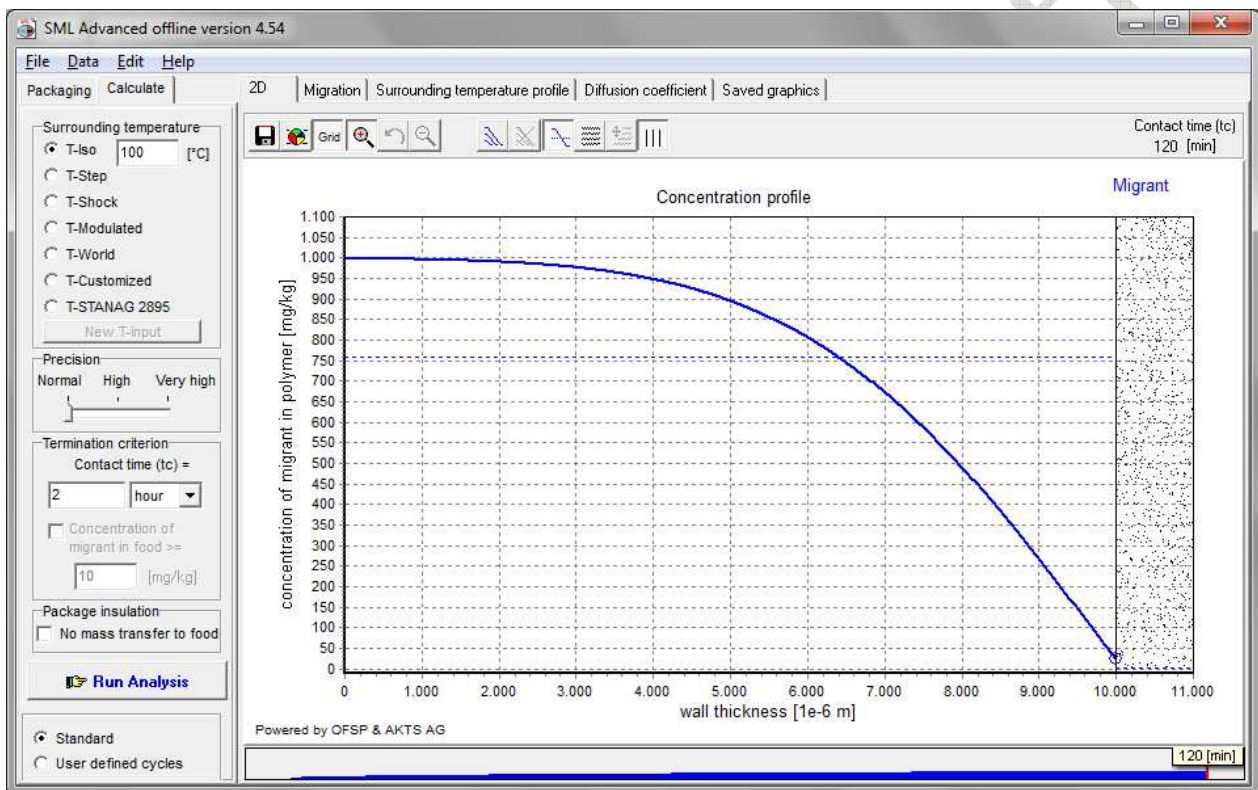
183 to be used for worst case calculation of specific migration under assumption of total transfer

184 => 2 x 99% layer thickness = 16000

185 above 16000 µm two sides to be considered for calculation of migration if full immersion

186 testing applied

187



188

189

190

191 ► molecular mass 501 - 750 g/mol

192 10d @ 60°C

193 => 100% layer thickness = 7000 µm

194 no absolute barrier at thicknesses below 7000 µm

195 => 99% layer thickness = 4800

196 => 1/2 x 99% layer thickness = 2400 µm

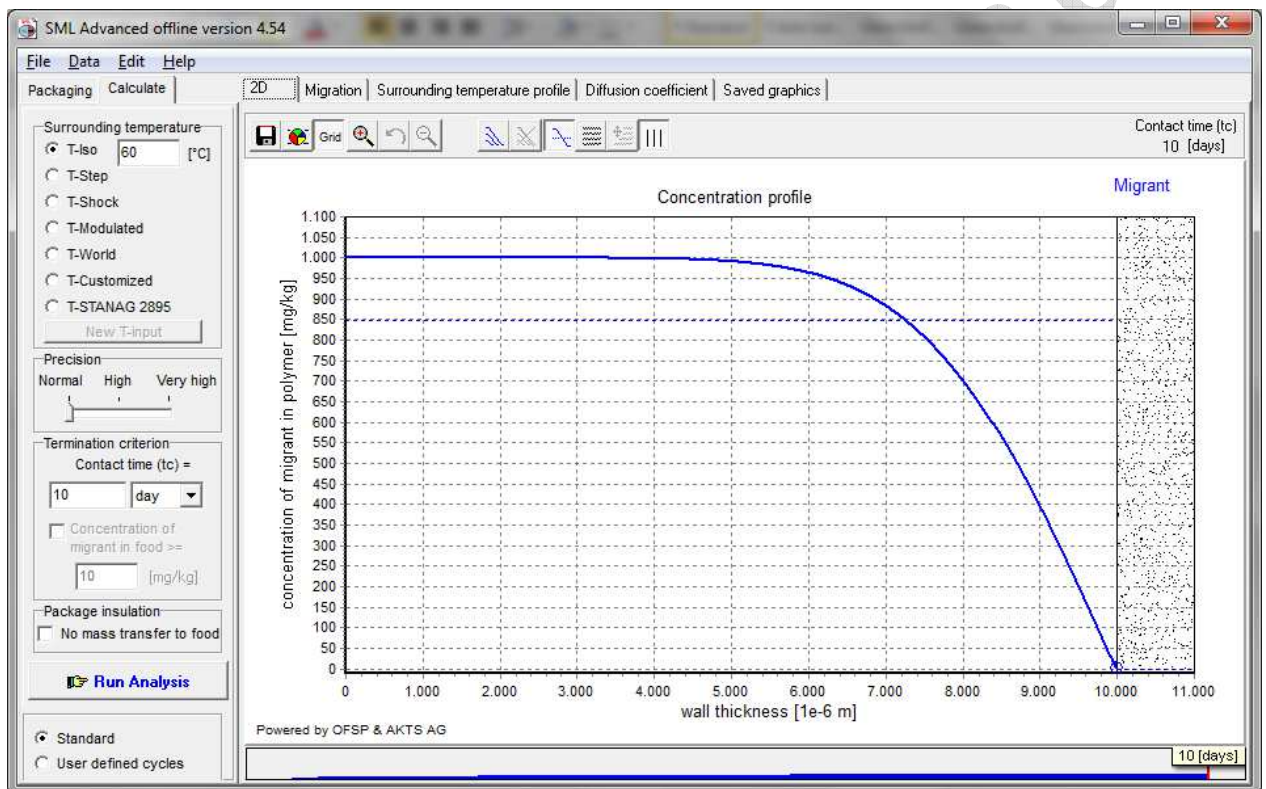
197 to be used for worst case calculation of specific migration under assumption of total transfer

198 => 2 x 99% layer thickness = 9600

199 above 9600 µm two sides to be considered for calculation of migration if full immersion

200 testing applied

201



202

203

204

205 **10d @ 40°C**

206 => 100% layer thickness = 2640

207 no absolute barrier at thicknesses below 2640 µm

208 => 99% layer thickness = 1840 µm

209 => 1/2 x 99% layer thickness = 920 µm

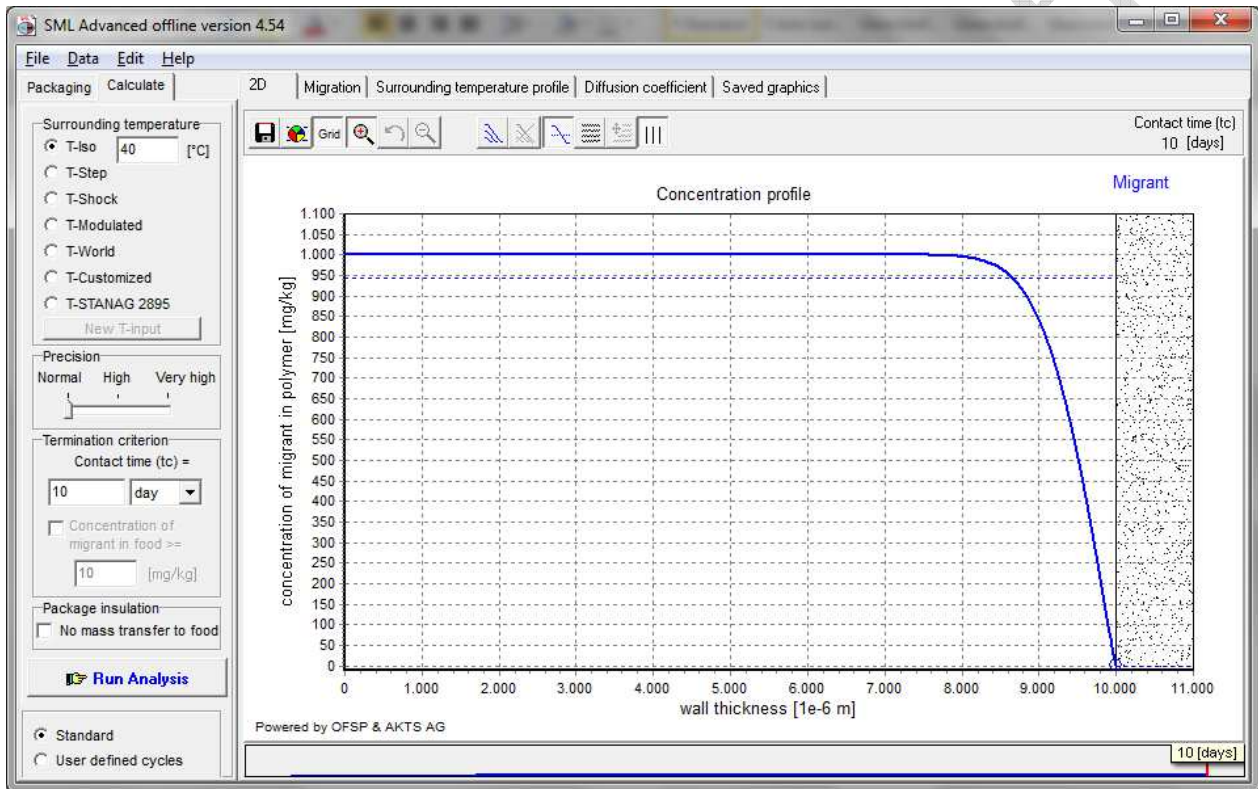
210 to be used for worst case calculation of specific migration under assumption of total transfer

211 => 2 x 99% layer thickness = 3680

212 above 3680 µm two sides to be considered for calculation of migration if full immersion

213 testing applied

214



215

216

217

218 **10d @ 20°C**

219 => 100% layer thickness = 800 µm

220 no absolute barrier at thicknesses below 800 µm

221 => 99% layer thickness = 600 µm

222 => 1/2 x 99% layer thickness = 300 µm

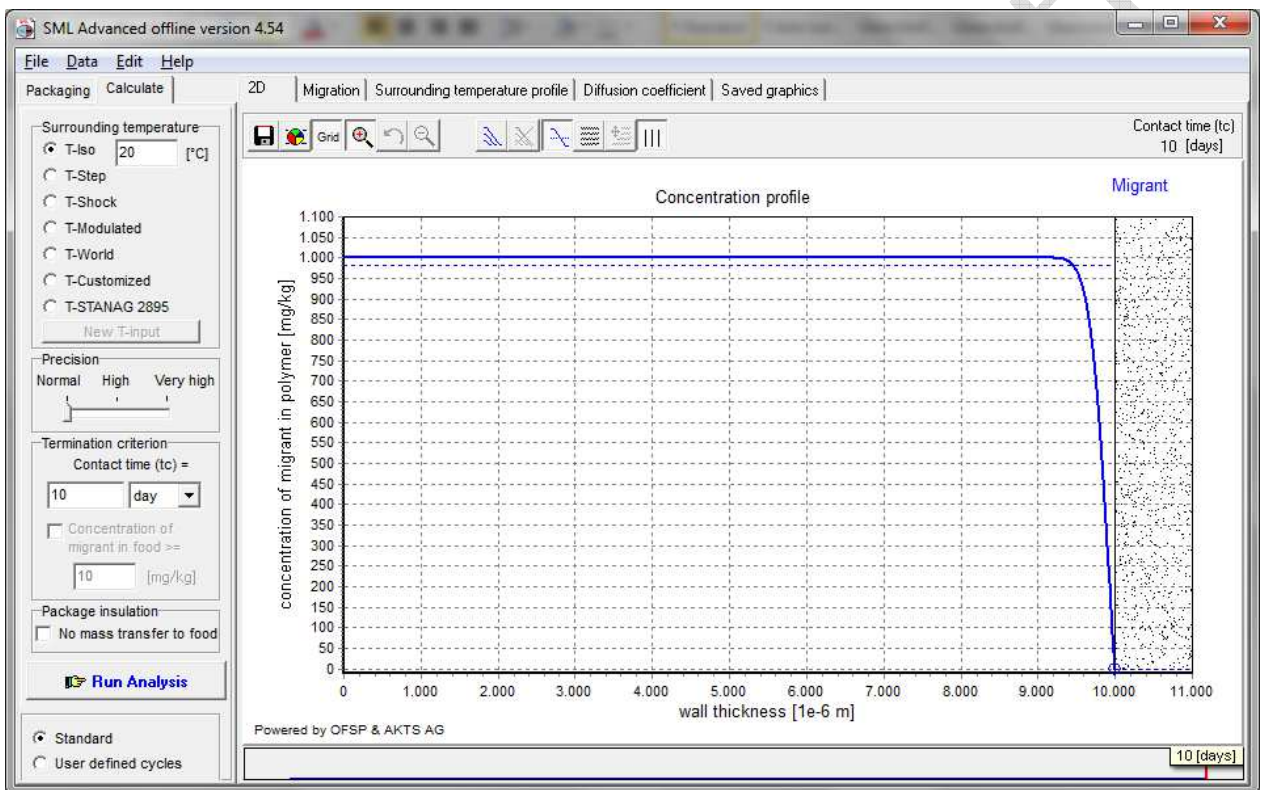
223 to be used for worst case calculation of specific migration under assumption of total transfer

224 => 2 x 99% layer thickness = 1200 µm

225 above 1200 µm two sides to be considered for calculation of migration if full immersion

226 testing applied

227



228

229

230

231

232 **2h @ 100°C**

233 => 100% layer thickness = 3240

234 no absolute barrier at thicknesses below 3240 µm

235 => 99% layer thickness = 2440 µm

236 => 1/2 x 99% layer thickness = 1220 µm

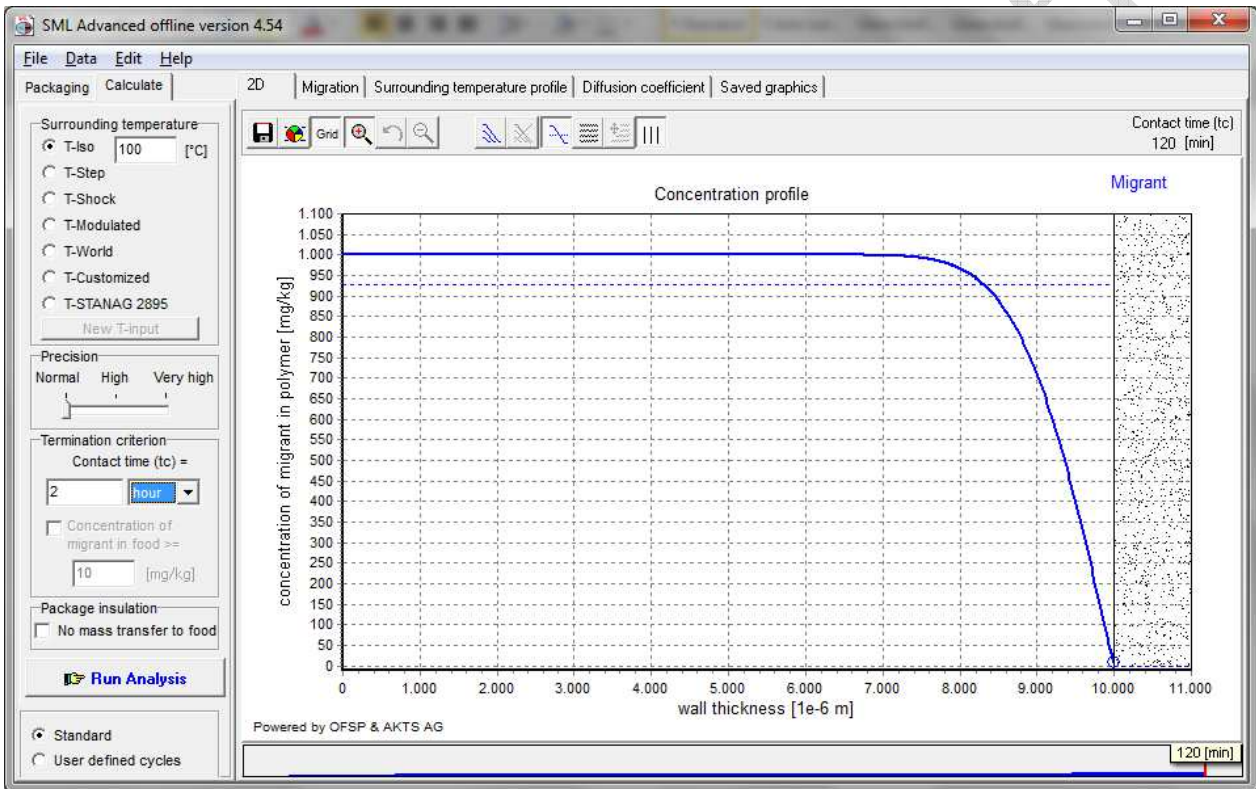
237 to be used for worst case calculation of specific migration under assumption of total transfer

238 => 2 x 99% layer thickness = 4880

239 above 4880 µm two sides to be considered for calculation of migration if full immersion

240 testing applied

241



242

243

244

245

246 ► molecular mass 751 - 1000 g/mol

247 10d @ 60°C

248 => 100% layer thickness = 2600 µm

249 no absolute barrier at thicknesses below 2600 µm

250 => 99% layer thickness = 1920

251 => 1/2 x 99% layer thickness = 960 µm

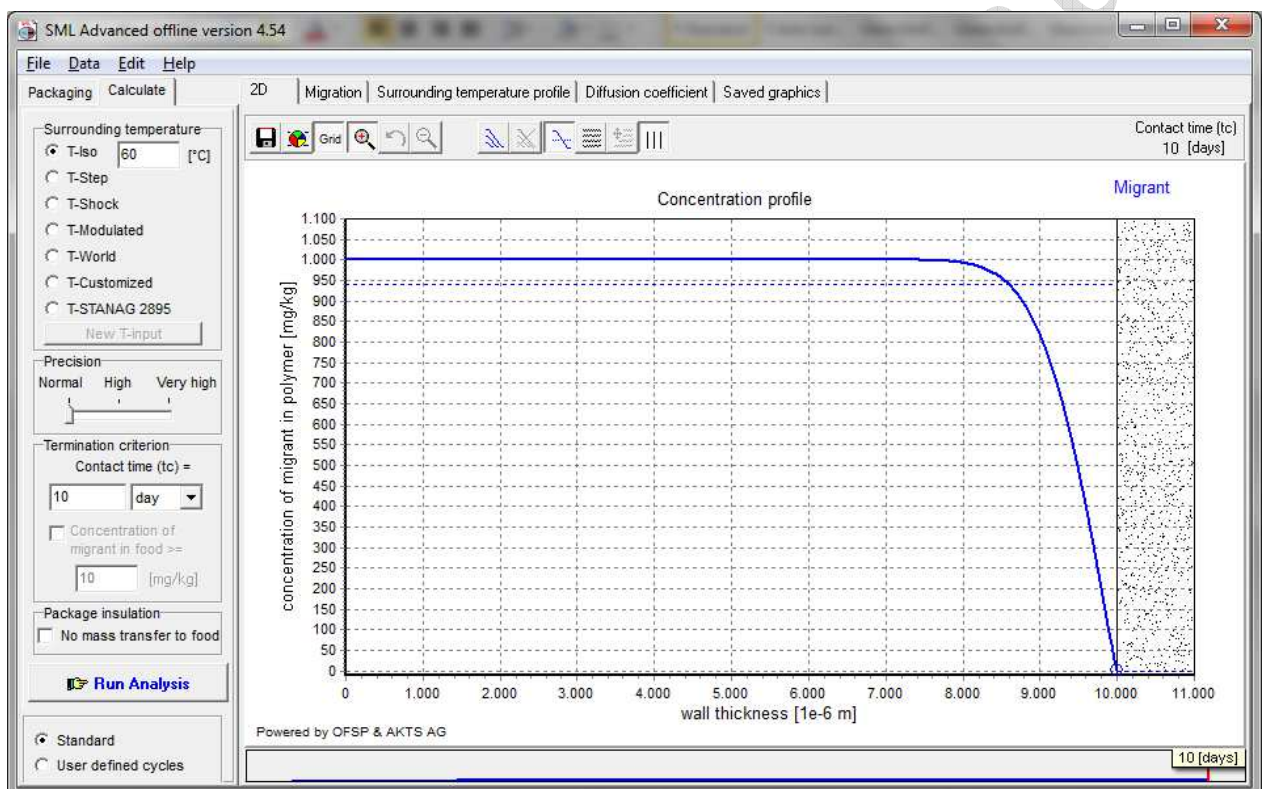
252 to be used for worst case calculation of specific migration under assumption of total transfer

253 => 2 x 99% layer thickness = 2840

254 above 2840 µm two sides to be considered for calculation of migration if full immersion

255 testing applied

256



257

258

259

260

261 **10d @ 40°C**

262 => 100% layer thickness = 1000

263 no absolute barrier at thicknesses below 1000 µm

264 => 99% layer thickness = 720 µm

265 => 1/2 x 99% layer thickness = 360 µm

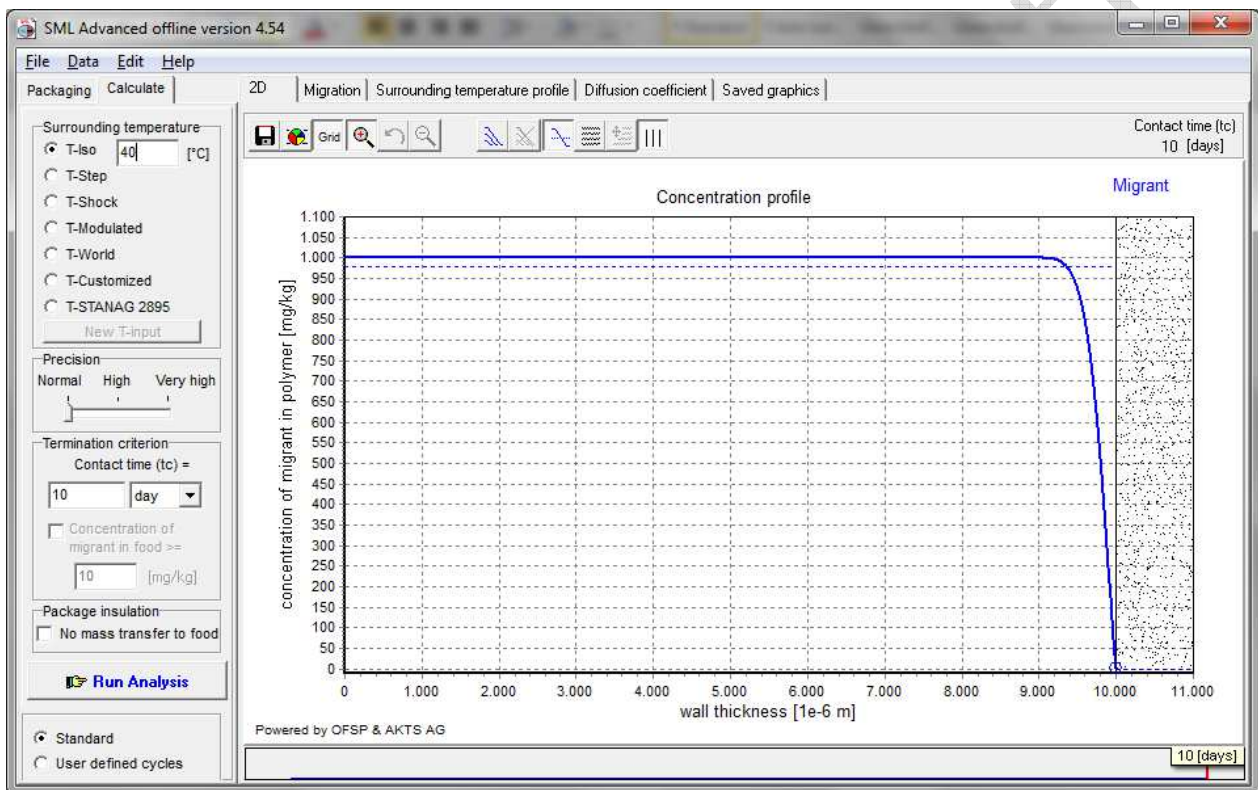
266 to be used for worst case calculation of specific migration under assumption of total transfer

267 => 2 x 99% layer thickness = 1440

268 above 1440 µm two sides to be considered for calculation of migration if full immersion

269 testing applied

270



271

272

273

274

275 **10d @ 20°C**

276 => 100% layer thickness = 340 µm

277 no absolute barrier at thicknesses below 340 µm

278 => 99% layer thickness = 240 µm

279 => 1/2 x 99% layer thickness = 120 µm

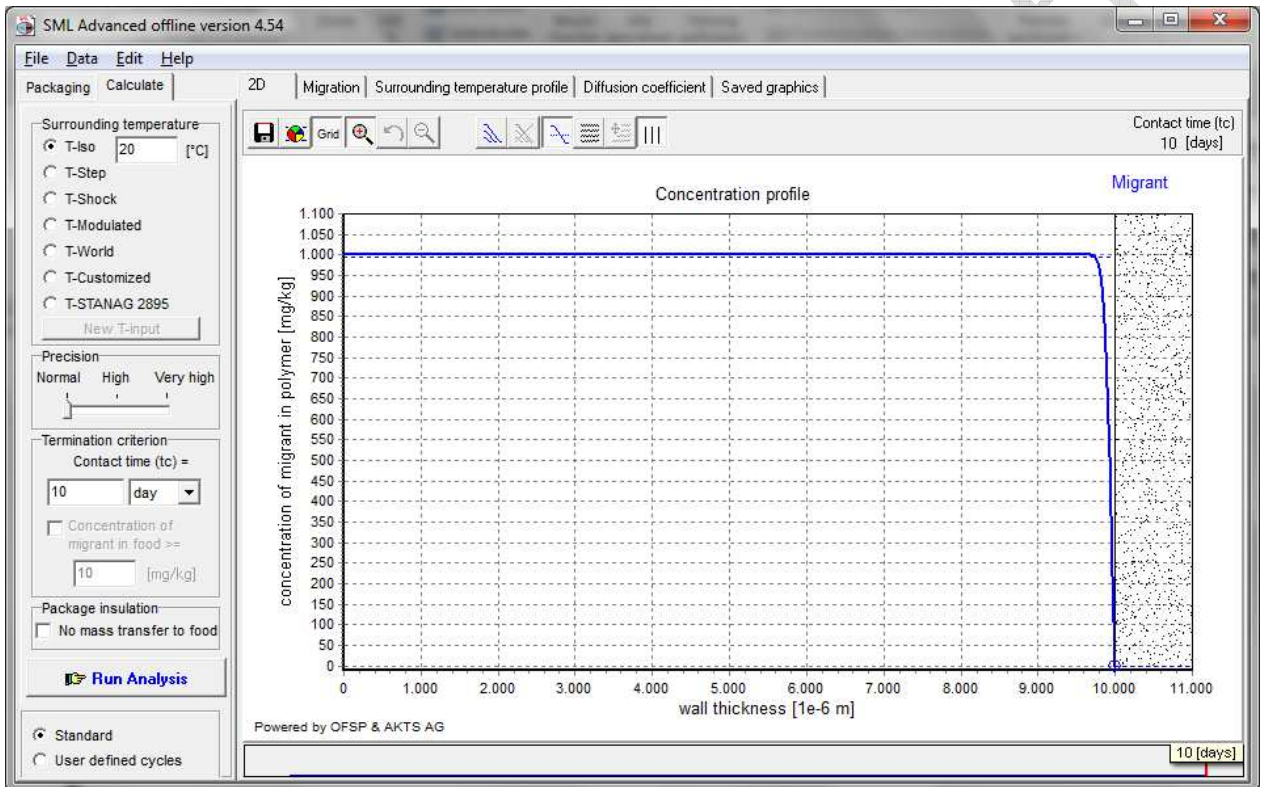
280 to be used for worst case calculation of specific migration under assumption of total transfer

281 => 2 x 99% layer thickness = 480 µm

282 above 480 µm two sides to be considered for calculation of migration if full immersion testing

283 applied

284



285

286

287

288

289

290 **2h @ 100°C**

291 => 100% layer thickness = 1360

292 no absolute barrier at thicknesses below 1360 µm

293 => 99% layer thickness = 960 µm

294 => 1/2 x 99% layer thickness = 480 µm

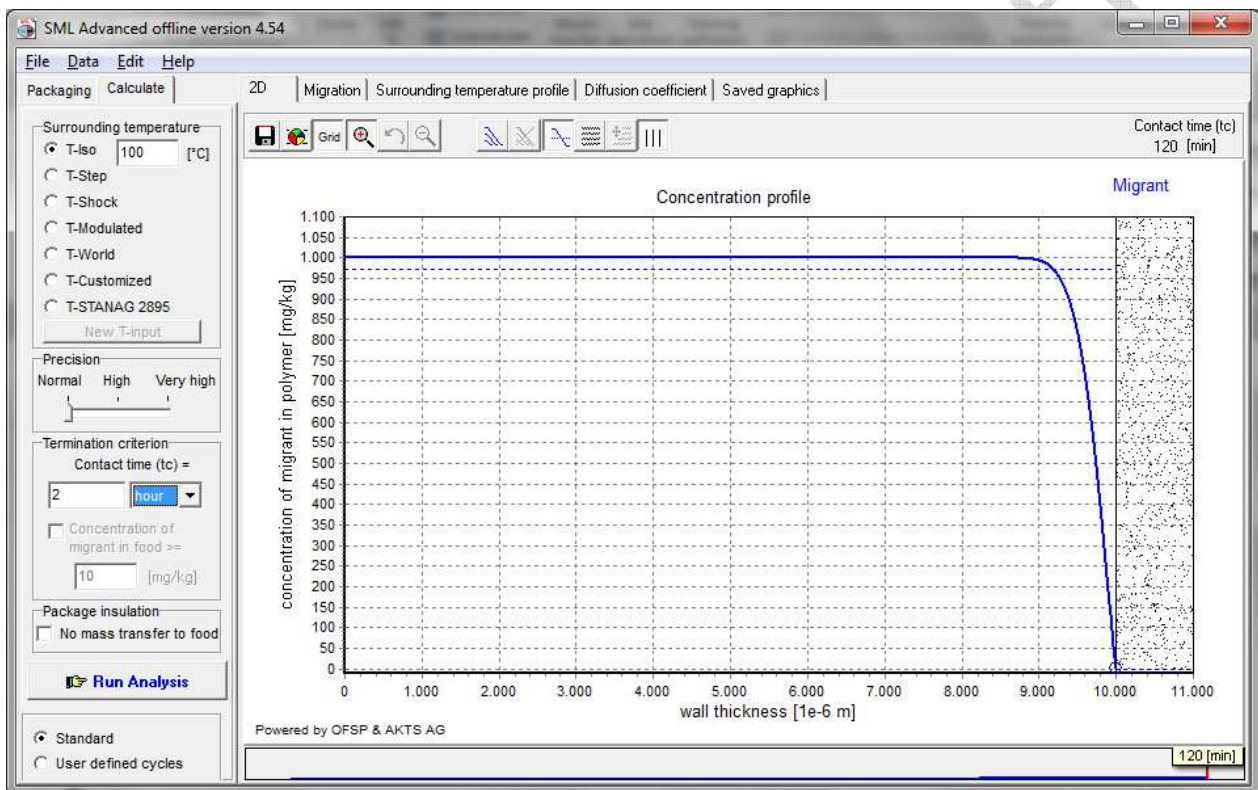
295 to be used for worst case calculation of specific migration under assumption of total transfer

296 => 2 x 99% layer thickness = 1920

297 above 1920 µm two sides to be considered for calculation of migration if full immersion

298 testing applied

299



300

301

302

303

304

305 **HDPE**

306 **► molecular mass 100 - 250 g/mol**

307 **10d @ 60°C**

308 => 100% layer thickness = full length

309 no absolute barrier

310 => 99% layer thickness = full length

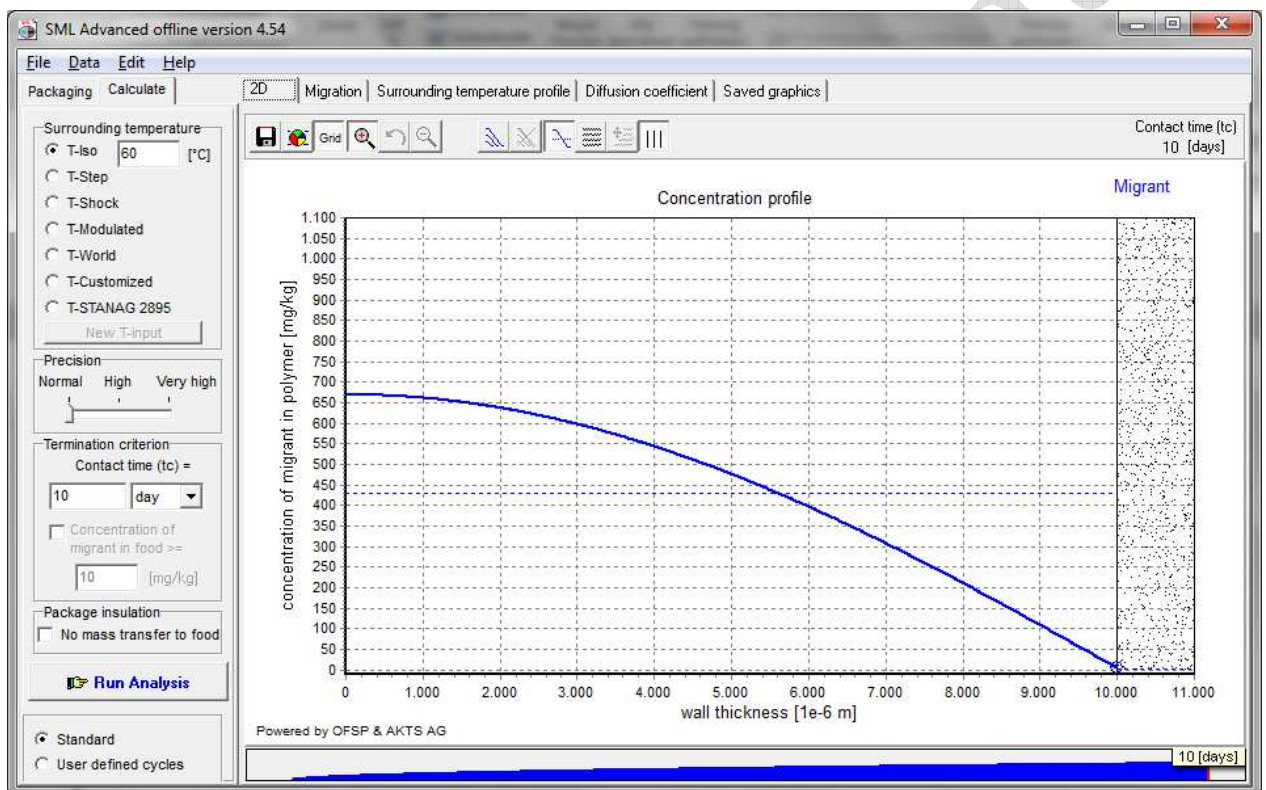
311 => 1/2 x 99% layer thickness = full length

312 to be used for worst case calculation of specific migration under assumption of total transfer

313 => 2 x 99% layer thickness = none

314 one side to be considered for calculation of migration if full immersion testing applied

315



316

317

318

319

320

321 **10d @ 40°C**

322 => 100% layer thickness = 8500

323 no absolute barrier at thicknesses below 8500 µm

324 => 99% layer thickness = 5900 µm

325 => 1/2 x 99% layer thickness = 2950 µm

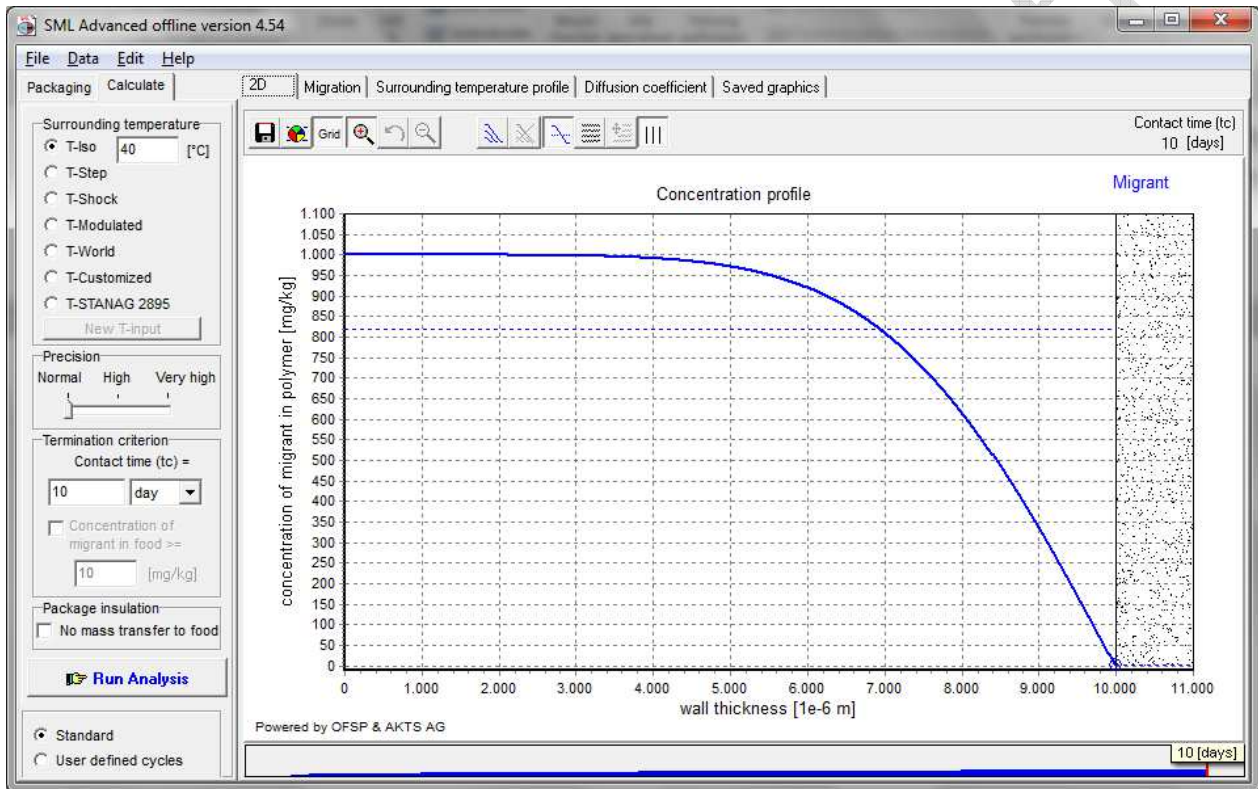
326 to be used for worst case calculation of specific migration under assumption of total transfer

327 => 2 x 99% layer thickness = 11800

328 above 11800 µm two sides to be considered for calculation of migration if full immersion

329 testing applied

330



331

332

333

334

335

336 **10d @ 20°C**

337 => 100% layer thickness = 2280 µm

338 no absolute barrier at thicknesses below 2280 µm

339 => 99% layer thickness = 1600 µm

340 => 1/2 x 99% layer thickness = 800 µm

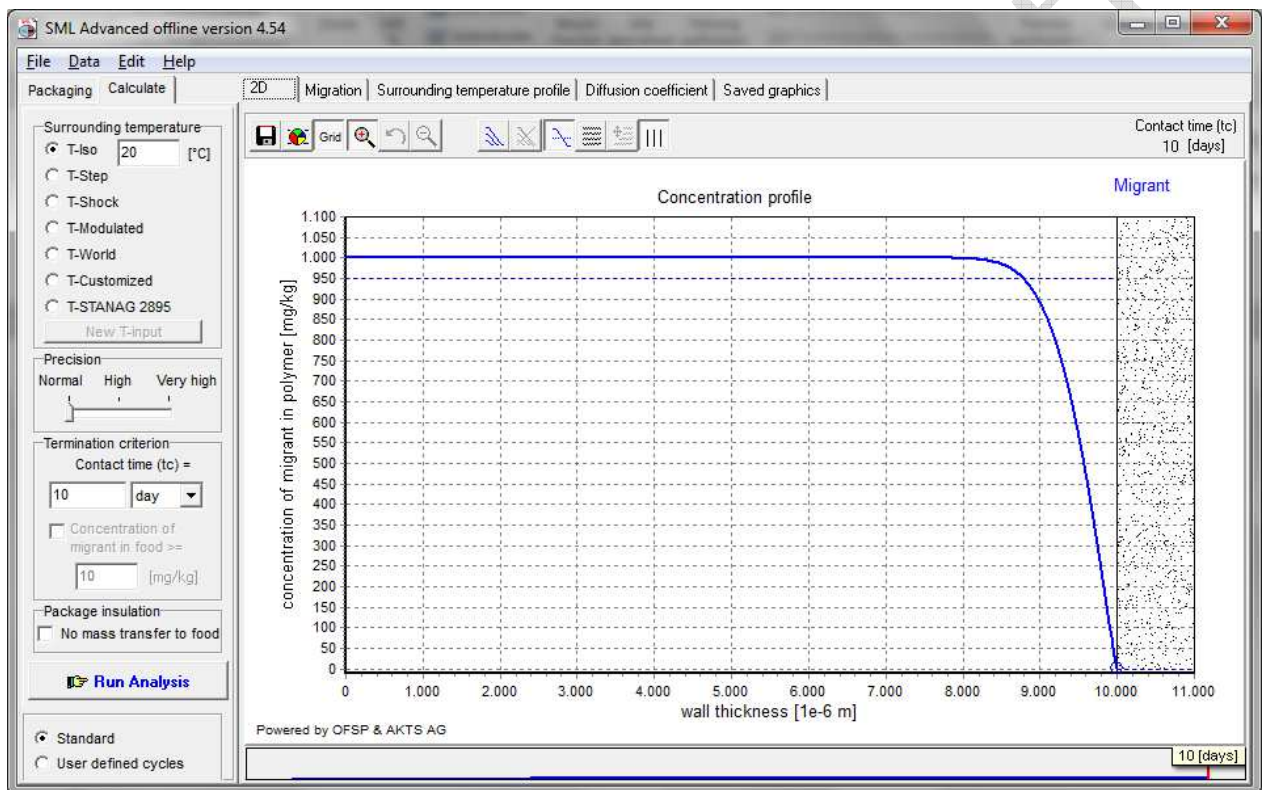
341 to be used for worst case calculation of specific migration under assumption of total transfer

342 => 2 x 99% layer thickness = 3200 µm

343 above 3200 µm two sides to be considered for calculation of migration if full immersion

344 testing applied

345



346

347

348

349

350

351 **2h @ 100°C**

352 => 100% layer thickness = full length

353 no absolute barrier

354 => 99% layer thickness = full length

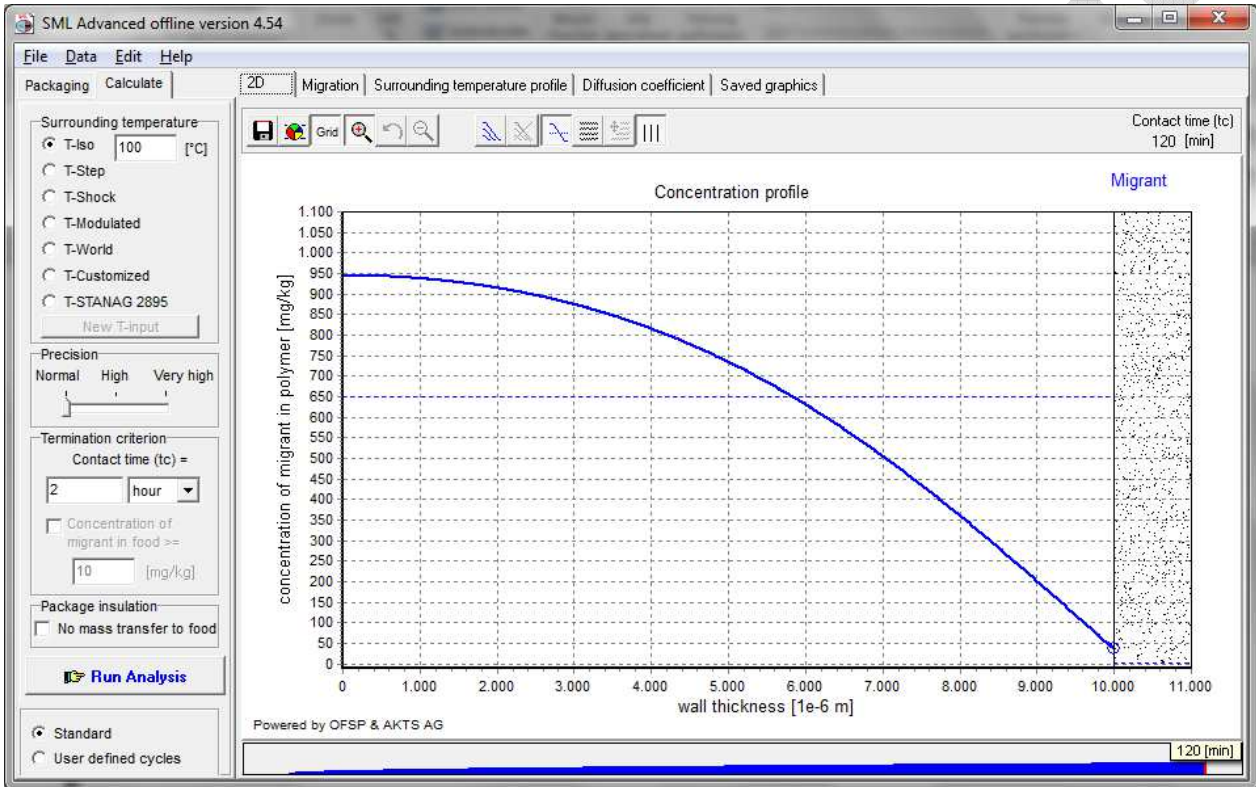
355 => 1/2 x 99% layer thickness = none

356 to be used for worst case calculation of specific migration under assumption of total transfer

357 => 2 x 99% layer thickness = none

358 one side to be considered for calculation of migration if full immersion testing applied

359



360

361

362

363

364

365 ► molecular mass 251 - 500 g/mol

366 10d @ 60°C

367 => 100% layer thickness = 9000 µm

368 no absolute barrier at thicknesses below 9000 µm

369 => 99% layer thickness = 6850 µm

370 => 1/2 x 99% layer thickness = 3425 µm

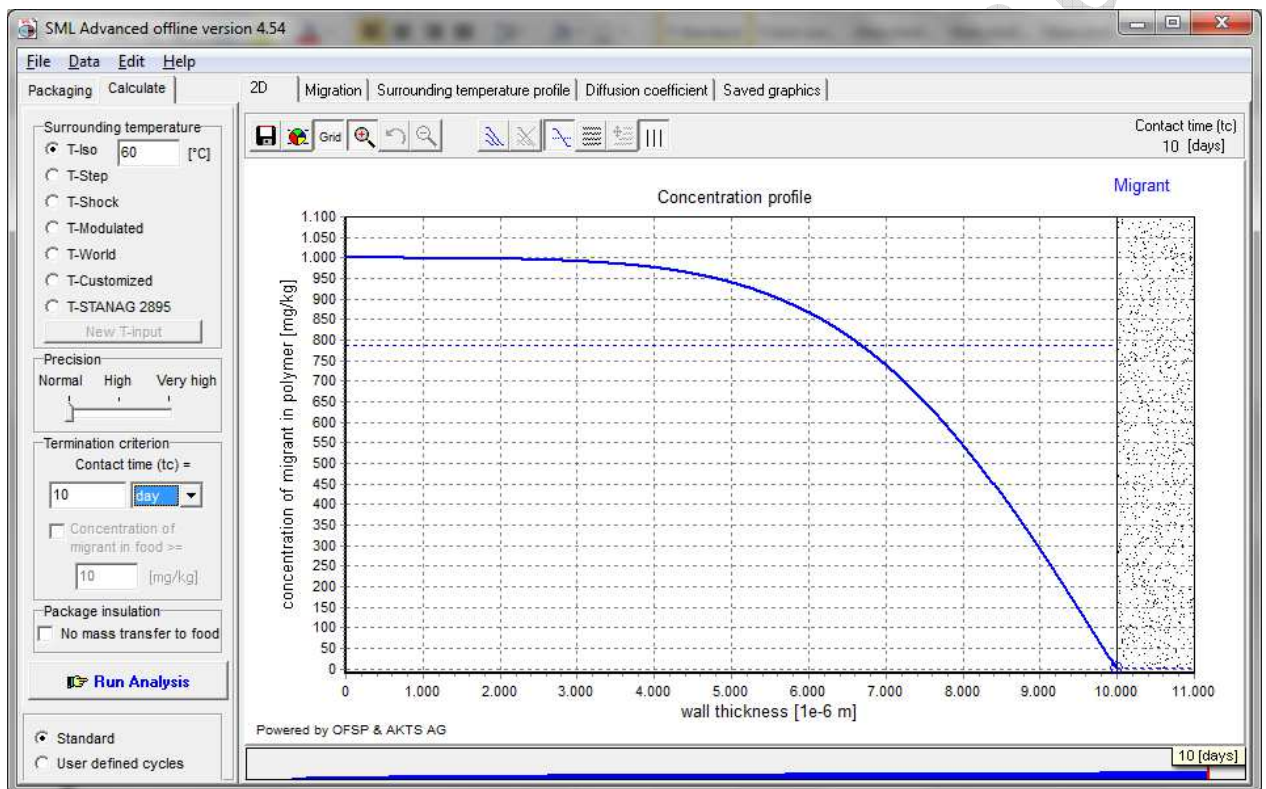
371 to be used for worst case calculation of specific migration under assumption of total transfer

372 => 2 x 99% layer thickness = 13700 µm

373 above 13700 µm two sides to be considered for calculation of migration if full immersion

374 testing applied

375



376

377

378

379

380

381 **10d @ 40°C**

382 => 100% layer thickness = 3000

383 no absolute barrier at thicknesses below 3000 µm

384 => 99% layer thickness = 2200 µm

385 => 1/2 x 99% layer thickness = 1100 µm

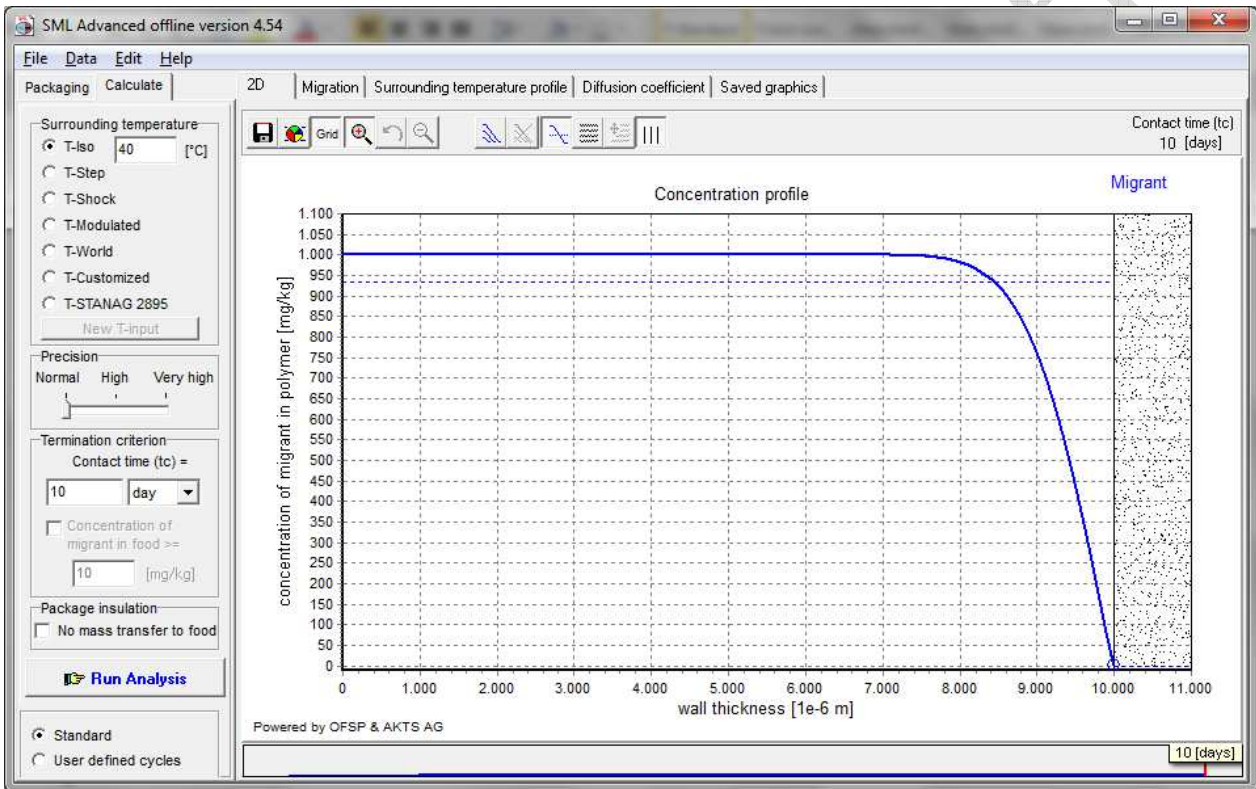
386 to be used for worst case calculation of specific migration under assumption of total transfer

387 => 2 x 99% layer thickness = 4800

388 above 4800 µm two sides to be considered for calculation of migration if full immersion

389 testing applied

390



391

392

393

394

395

396 **10d @ 20°C**

397 => 100% layer thickness = 800 µm

398 no absolute barrier at thicknesses below 800 µm

399 => 99% layer thickness = 600 µm

400 => 1/2 x 99% layer thickness = 300 µm

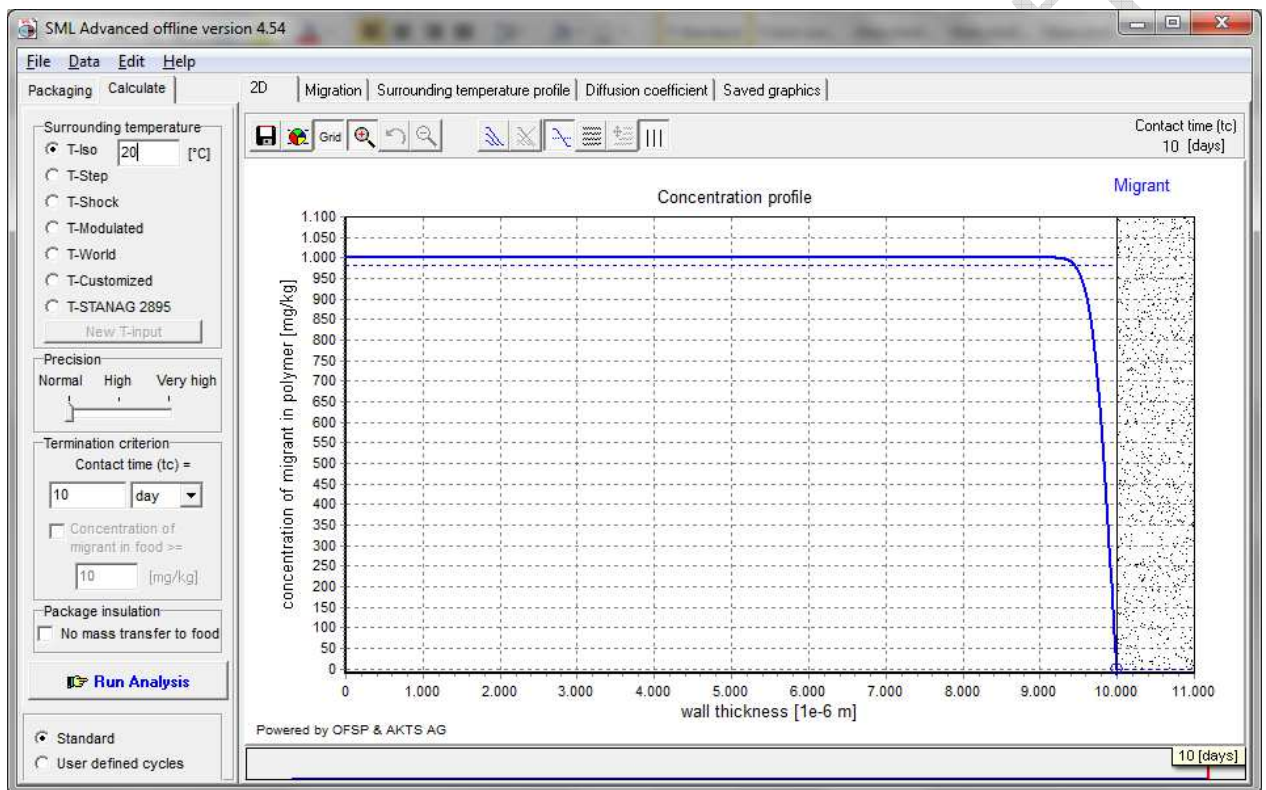
401 to be used for worst case calculation of specific migration under assumption of total transfer

402 => 2 x 99% layer thickness = 1200 µm

403 above 1200 µm two sides to be considered for calculation of migration if full immersion

404 testing applied

405



406

407

408

409

410

411 **2h @ 100°C**

412 => 100% layer thickness = 6400 µm

413 no absolute barrier at thicknesses below 6400 µm

414 => 99% layer thickness = 4300 µm

415 => 1/2 x 99% layer thickness = 2150 µm

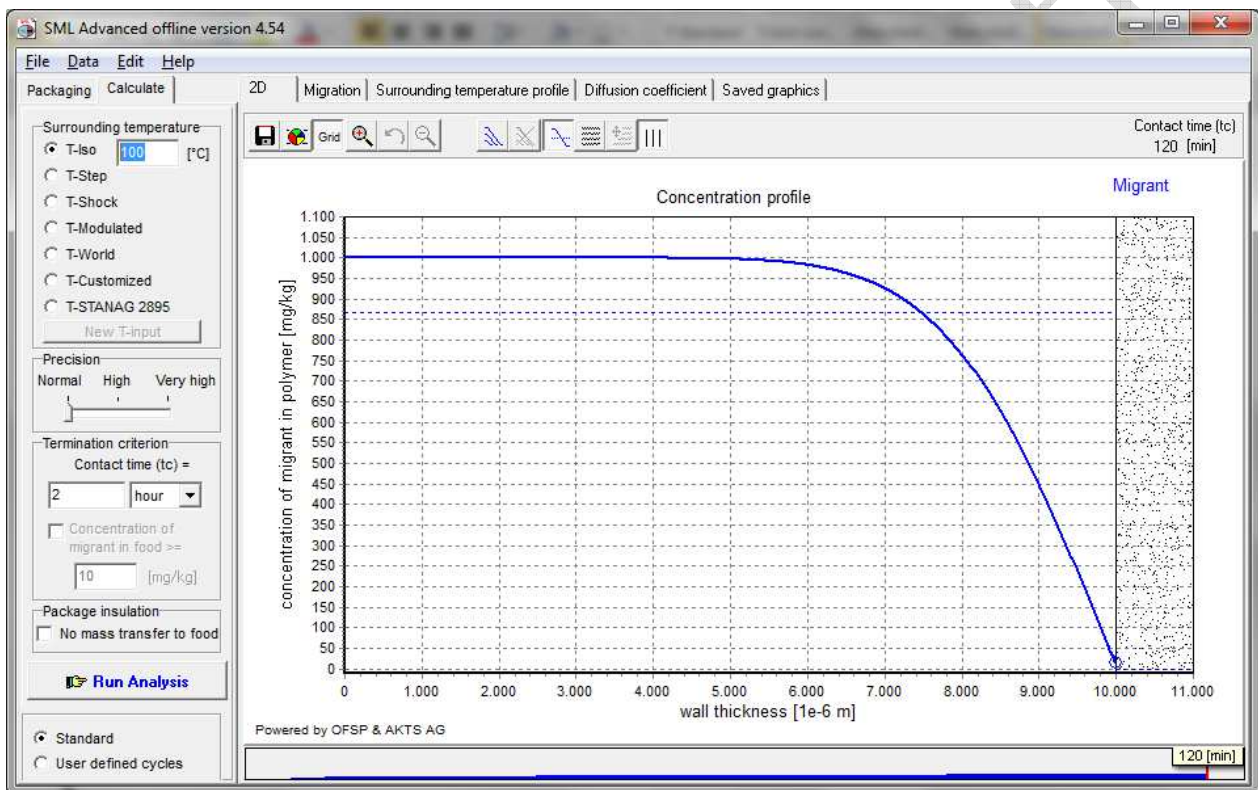
416 to be used for worst case calculation of specific migration under assumption of total transfer

417 => 2 x 99% layer thickness = 8400 µm

418 above 8400 µm two sides to be considered for calculation of migration if full immersion

419 testing applied

420



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426 ► molecular mass 501 - 750 g/mol

427 10d @ 60°C

428 => 100% layer thickness = 3300 µm

429 no absolute barrier at thicknesses below 3300 µm

430 => 99% layer thickness = 2100 µm

431 => 1/2 x 99% layer thickness = 1050 µm

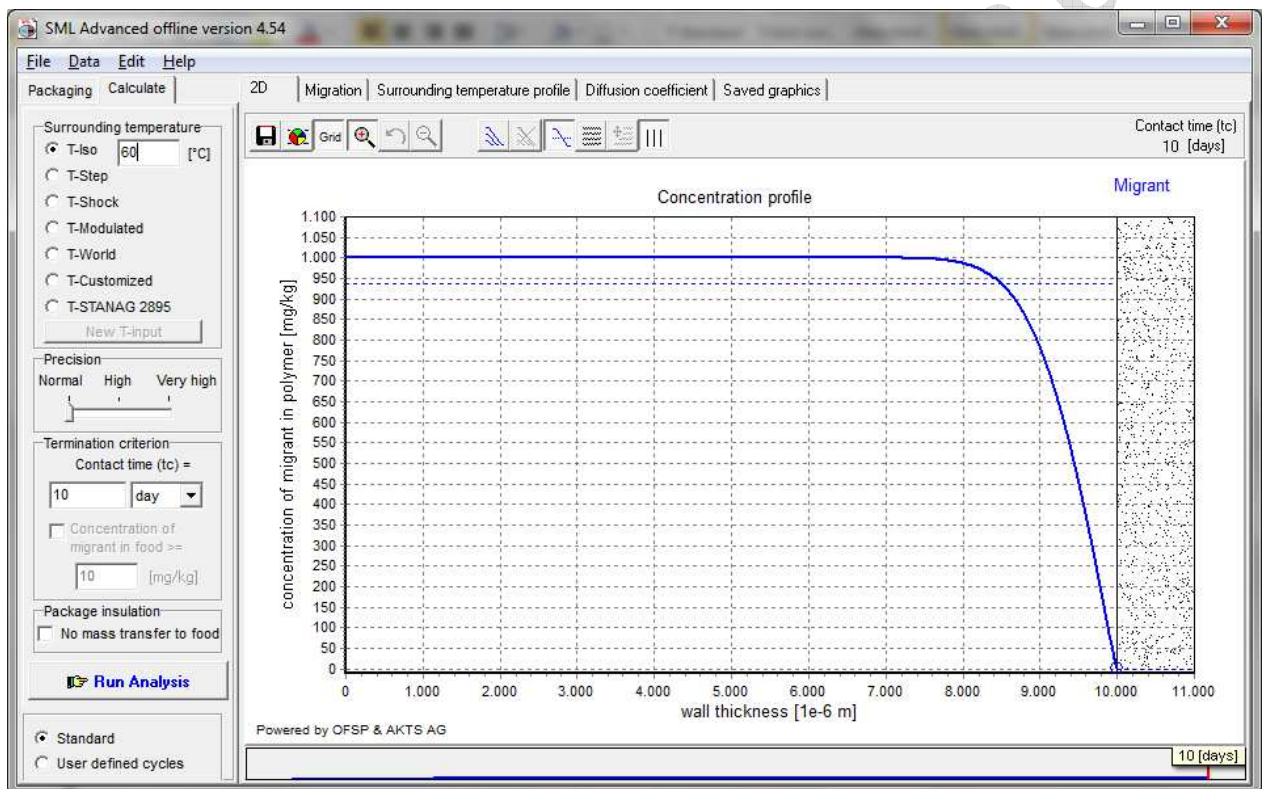
432 to be used for worst case calculation of specific migration under assumption of total transfer

433 => 2 x 99% layer thickness = 4200 µm

434 above 4200 µm two sides to be considered for calculation of migration if full immersion

435 testing applied

436



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442 **10d @ 40°C**

443 => 100% layer thickness = 960

444 no absolute barrier at thicknesses below 960 µm

445 => 99% layer thickness = 660 µm

446 => 1/2 x 99% layer thickness = 330 µm

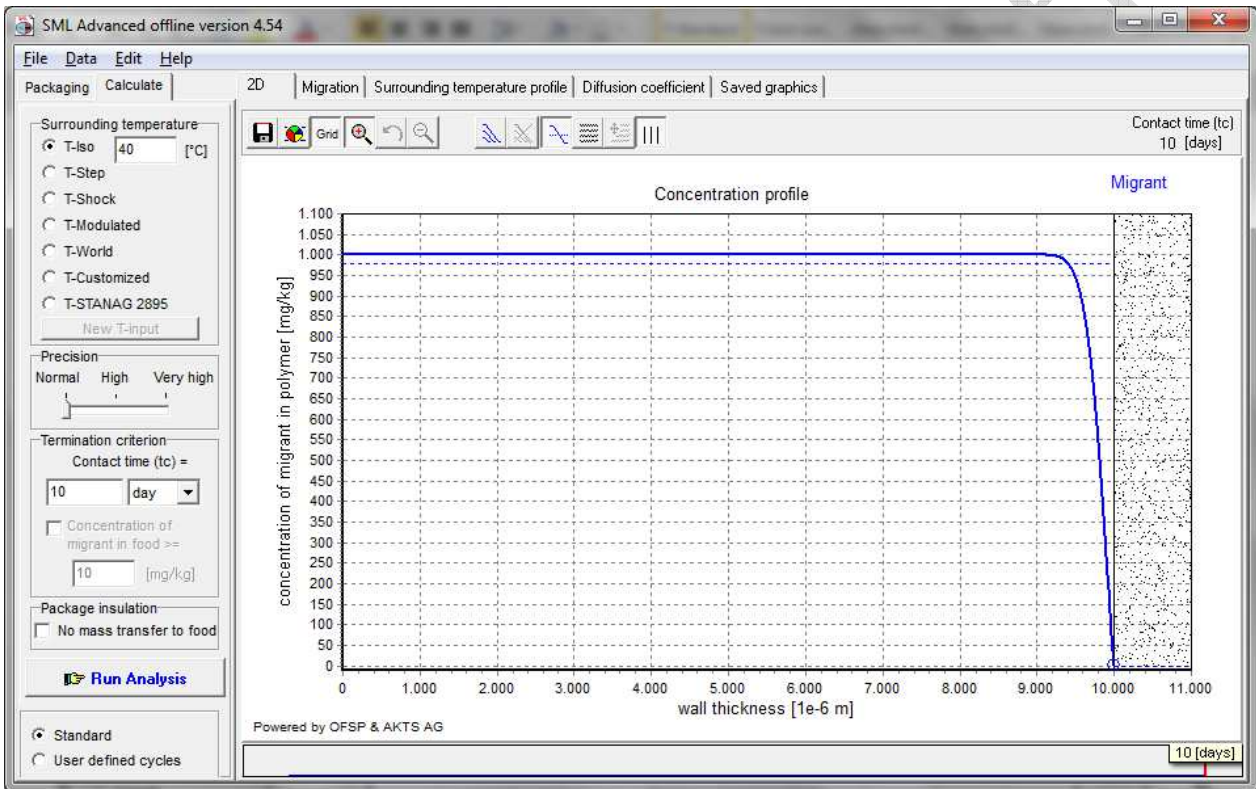
447 to be used for worst case calculation of specific migration under assumption of total transfer

448 => 2 x 99% layer thickness = 1320

449 above 1320 µm two sides to be considered for calculation of migration if full immersion

450 testing applied

451



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457 **10d @ 20°C**

458 => 100% layer thickness = 280 µm

459 no absolute barrier at thicknesses below 280 µm

460 => 99% layer thickness = 200 µm

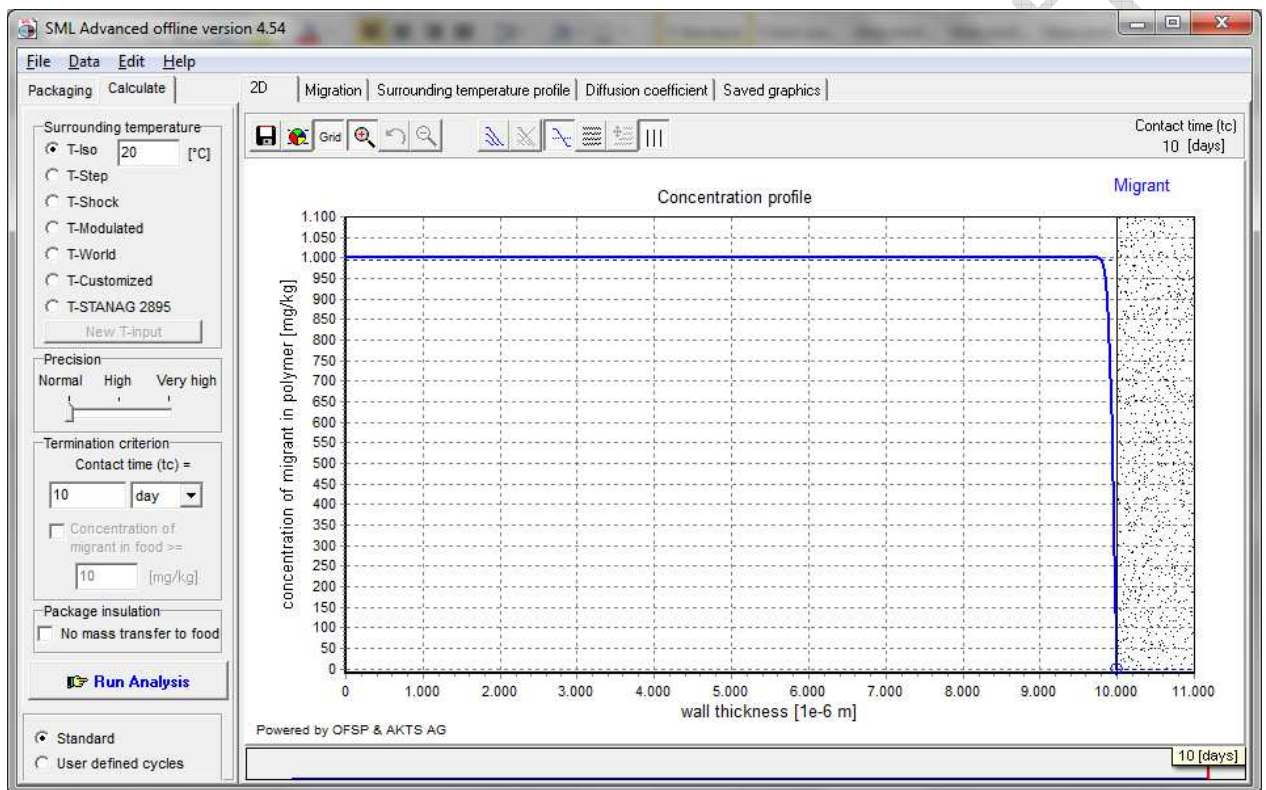
461 => 1/2 x 99% layer thickness = 100 µm

462 to be used for worst case calculation of specific migration under assumption of total transfer

463 => 2 x 99% layer thickness = 400 µm

464 above 400 µm two sides to be considered for calculation of migration if full immersion testing applied

466



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472 **2h @ 100°C**

473 => 100% layer thickness = 1800 µm

474 no absolute barrier at thicknesses below 1800 µm

475 => 99% layer thickness = 1320 µm

476 => 1/2 x 99% layer thickness = 660 µm

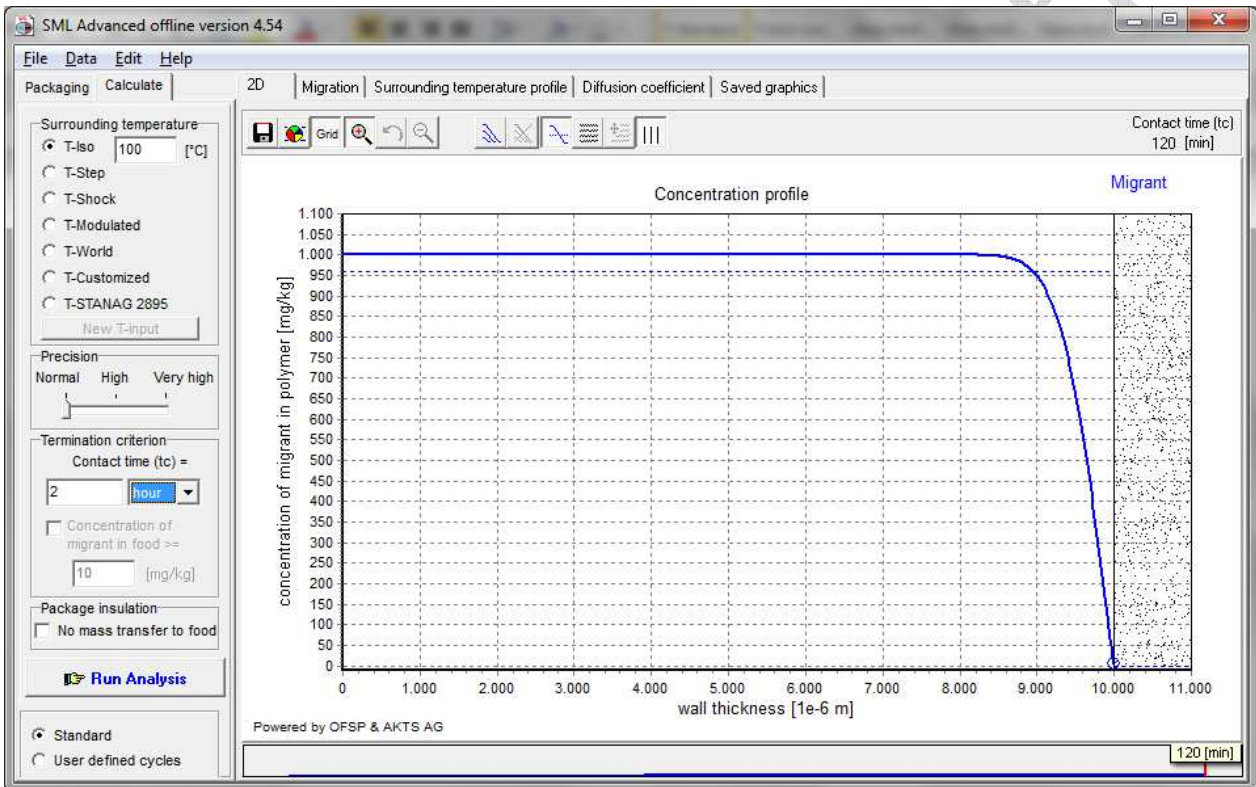
477 to be used for worst case calculation of specific migration under assumption of total transfer

478 => 2 x 99% layer thickness = 2640 µm

479 above 2640 µm two sides to be considered for calculation of migration if full immersion

480 testing applied

481



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487 ► molecular mass 750 - 1000 g/mol

488 10d @ 60°C

489 => 100% layer thickness = 1080 µm

490 no absolute barrier at thicknesses below 1080 µm

491 => 99% layer thickness = 840 µm

492 => 1/2 x 99% layer thickness = 420 µm

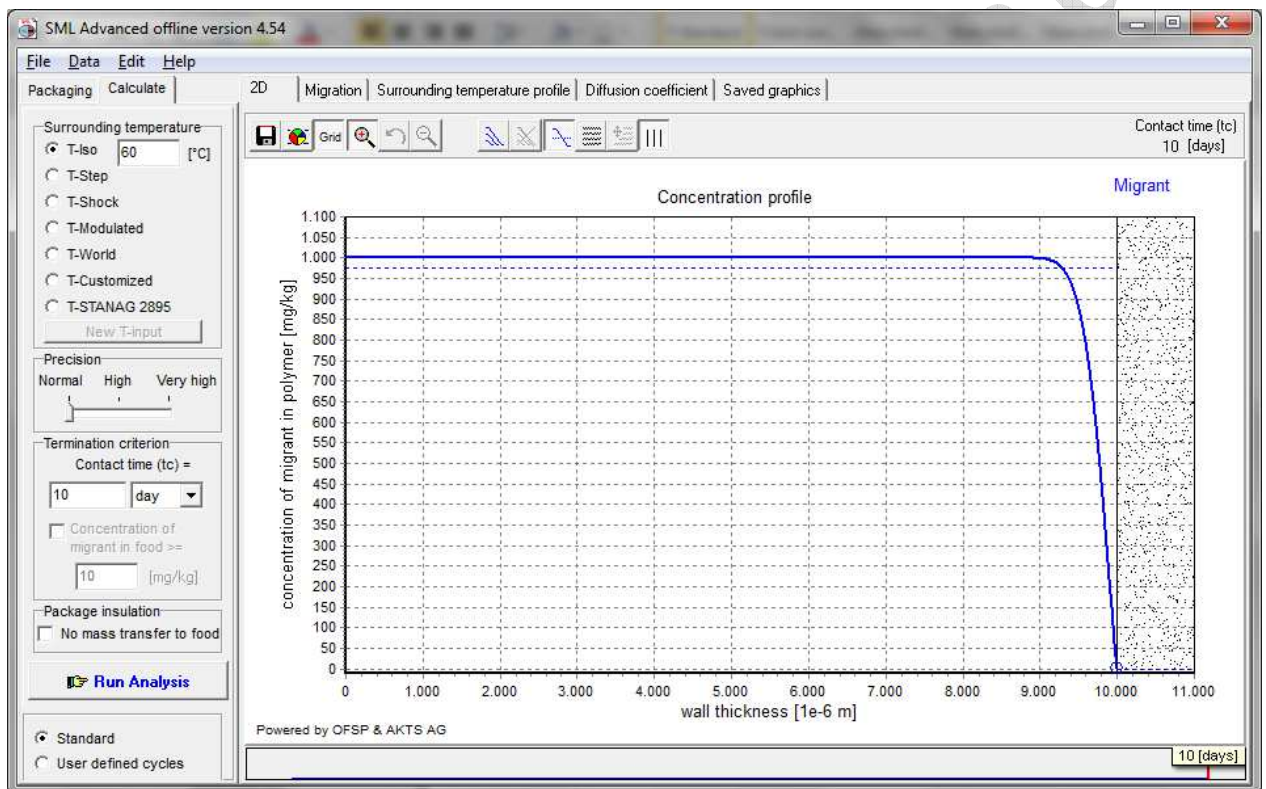
493 to be used for worst case calculation of specific migration under assumption of total transfer

494 => 2 x 99% layer thickness = 1680 µm

495 above 1680 µm two sides to be considered for calculation of migration if full immersion

496 testing applied

497



498

499

500

501

502

503 **10d @ 40°C**

504 => 100% layer thickness = 400

505 no absolute barrier at thicknesses below 400 µm

506 => 99% layer thickness = 270 µm

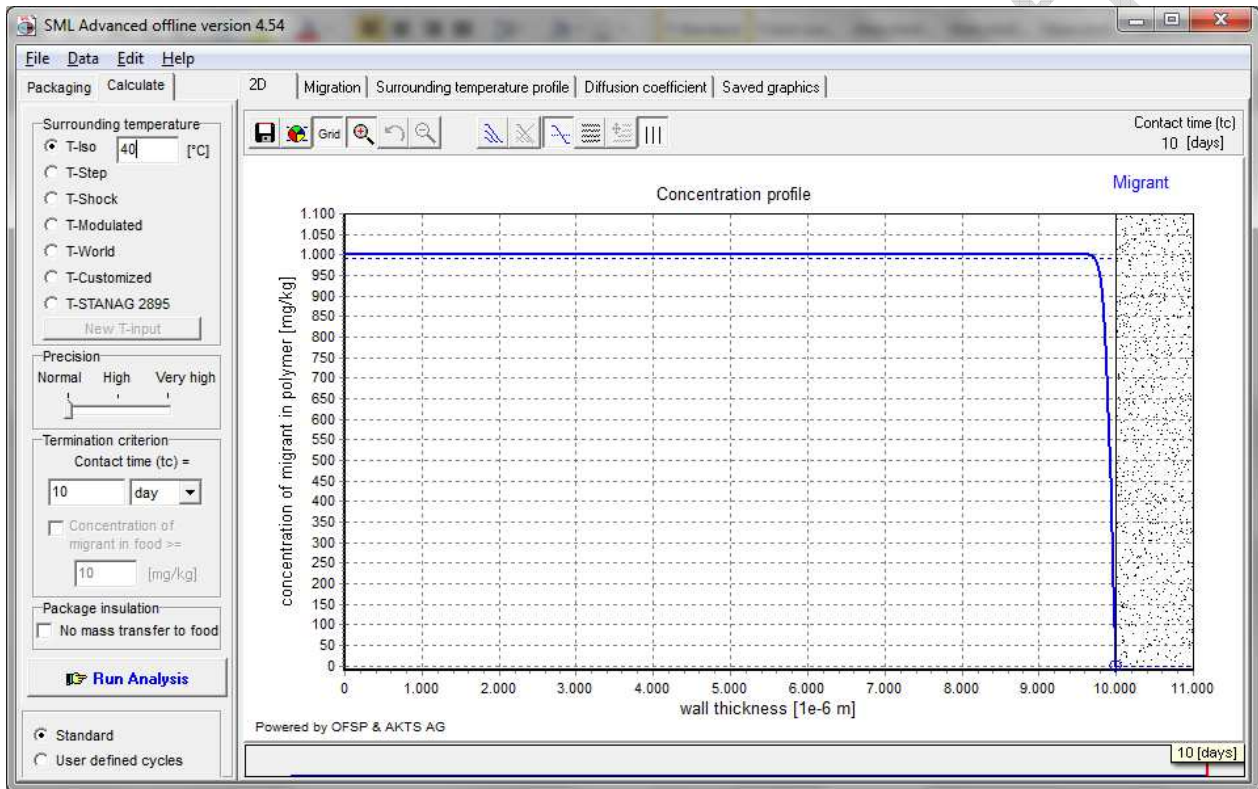
507 => 1/2 x 99% layer thickness = 135 µm

508 to be used for worst case calculation of specific migration under assumption of total transfer

509 => 2 x 99% layer thickness = 540

510 above 540 µm two sides to be considered for calculation of migration if full immersion testing
511 applied

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518 **10d @ 20°C**

519 => 100% layer thickness = 130 µm

520 no absolute barrier at thicknesses below 130 µm

521 => 99% layer thickness = 84 µm

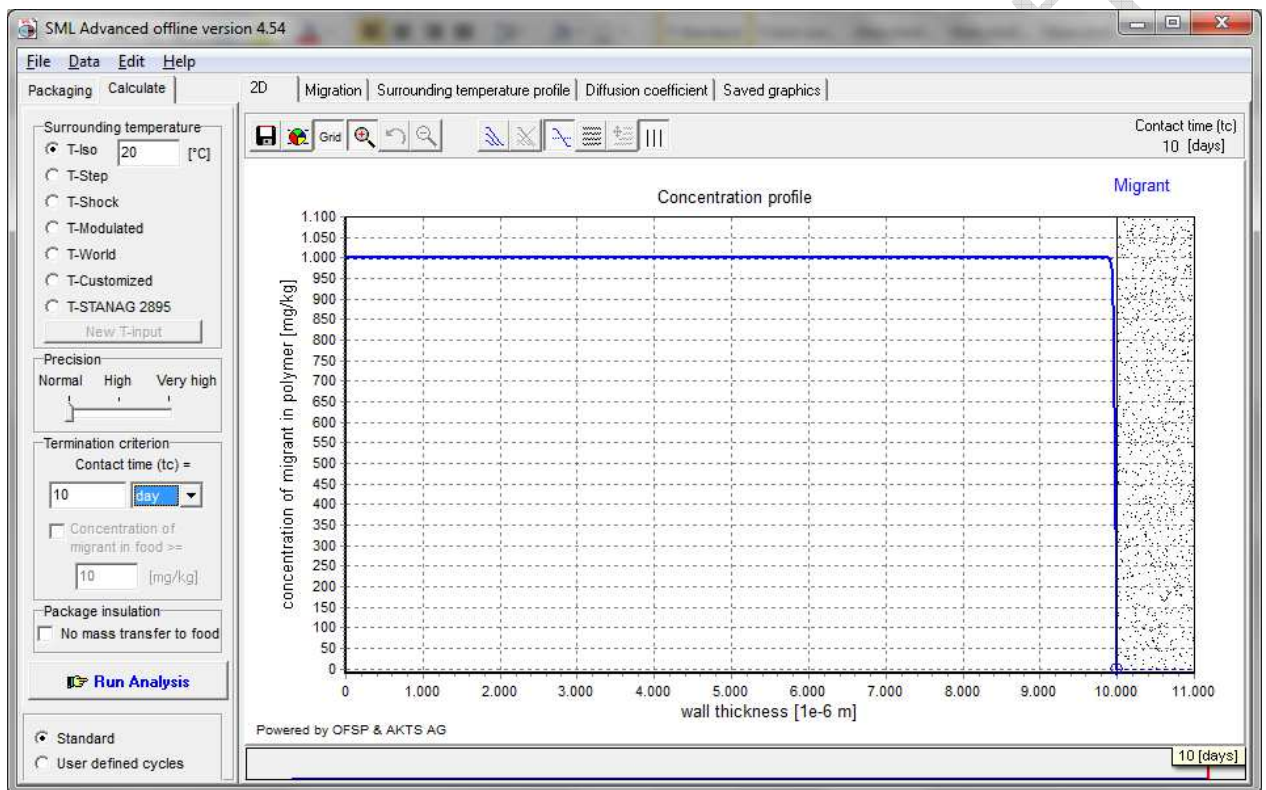
522 => 1/2 x 99% layer thickness = 42 µm

523 to be used for worst case calculation of specific migration under assumption of total transfer

524 => 2 x 99% layer thickness = 168 µm

525 above 168 µm two sides to be considered for calculation of migration if full immersion testing applied

527



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533 **2h @ 100°C**

534 => 100% layer thickness = 700 µm

535 no absolute barrier at thicknesses below 700 µm

536 => 99% layer thickness = 520 µm

537 => 1/2 x 99% layer thickness = 260 µm

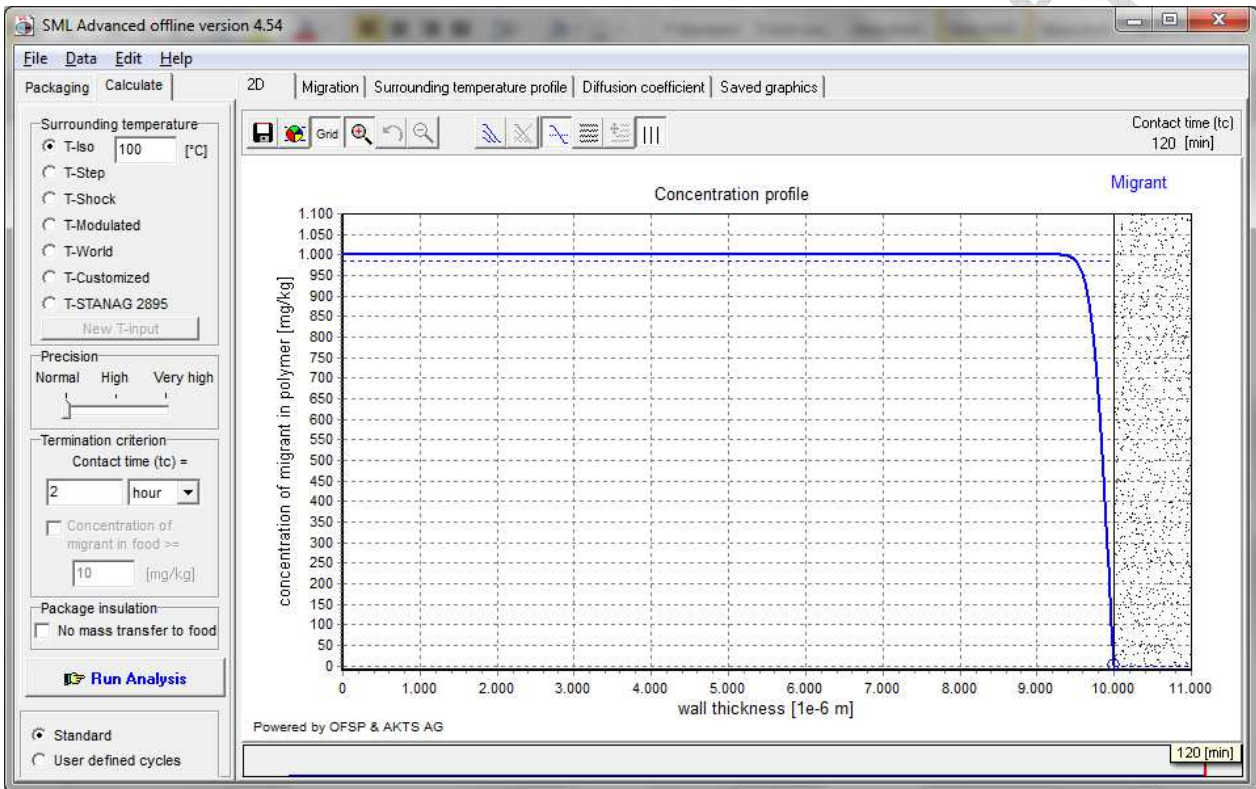
538 to be used for worst case calculation of specific migration under assumption of total transfer

539 => 2 x 99% layer thickness = 1040 µm

540 above 1040 µm two sides to be considered for calculation of migration if full immersion

541 testing applied

542



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548 **PP, homo; PP random**

549 **► molecular mass 100 - 250 g/mol**

550 **10d @ 60°C**

551 => 100% layer thickness = full length

552 no absolute barrier

553 => 99% layer thickness = 10000 µm

554 => 1/2 x 99% layer thickness = 5000 µm

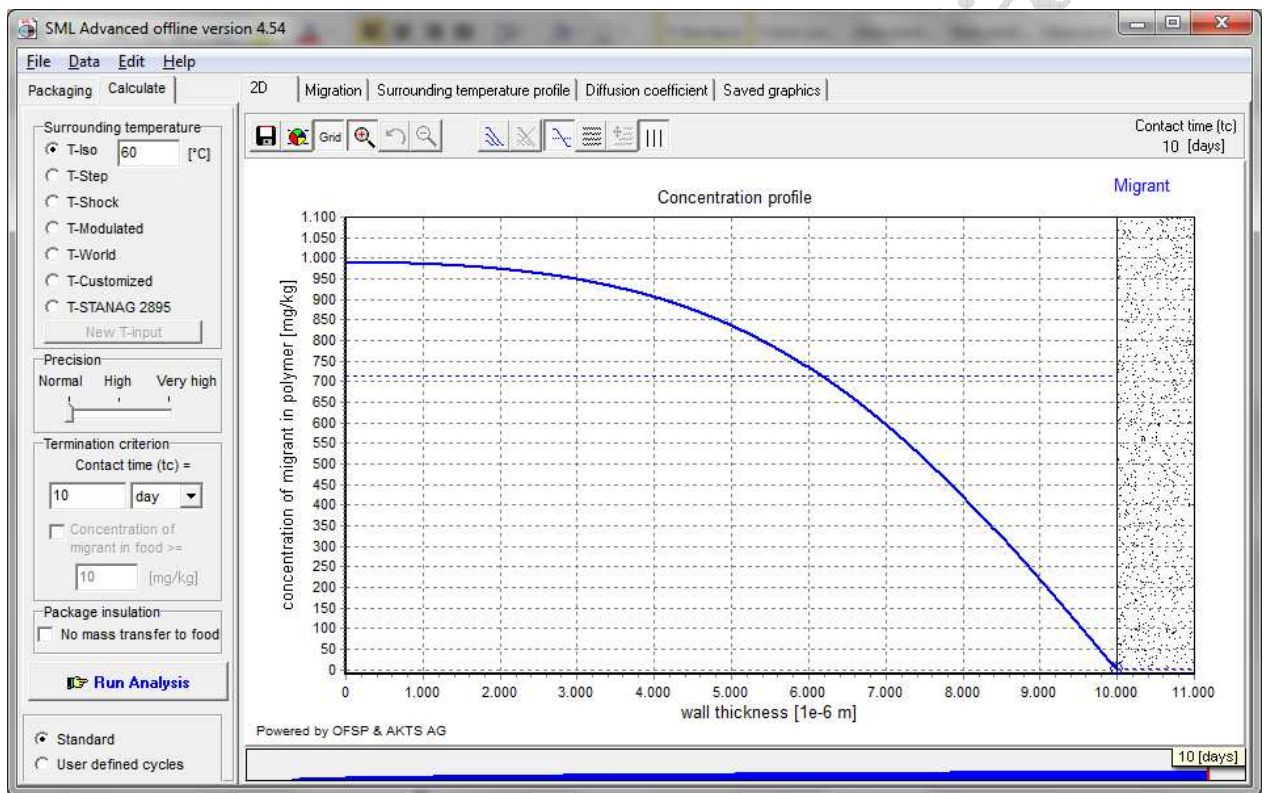
555 to be used for worst case calculation of specific migration under assumption of total transfer

556 => 2 x 99% layer thickness = 20000 µm

557 above 20000 µm two sides to be considered for calculation of migration if full immersion

558 testing applied

559



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565 **10d @ 40°C**

566 => 100% layer thickness = 3900

567 no absolute barrier at thicknesses below 3900 µm

568 => 99% layer thickness = 2920 µm

569 => 1/2 x 99% layer thickness = 1460 µm

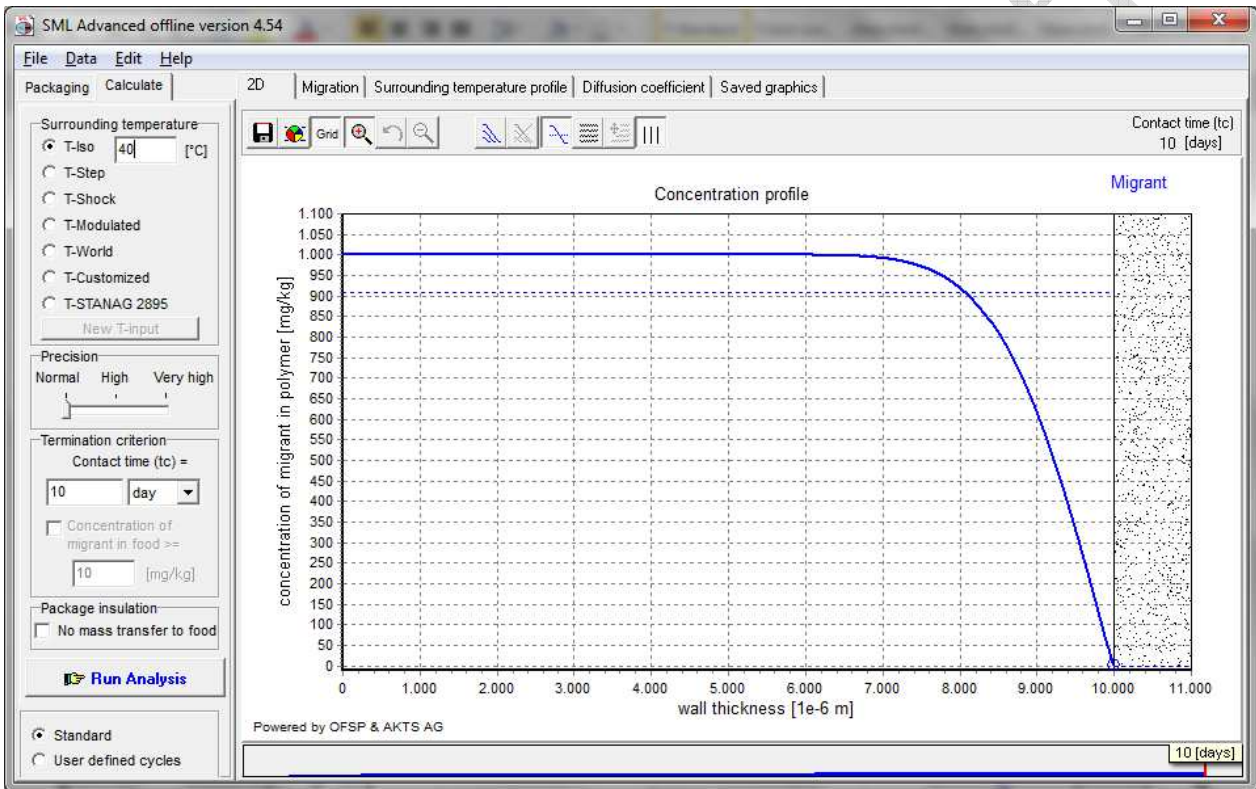
570 to be used for worst case calculation of specific migration under assumption of total transfer

571 => 2 x 99% layer thickness = 5840

572 above 5840 µm two sides to be considered for calculation of migration if full immersion

573 testing applied

574



579

580 **10d @ 20°C**

581 => 100% layer thickness = 1080 µm

582 no absolute barrier at thicknesses below 1080 µm

583 => 99% layer thickness = 800 µm

584 => 1/2 x 99% layer thickness = 400 µm

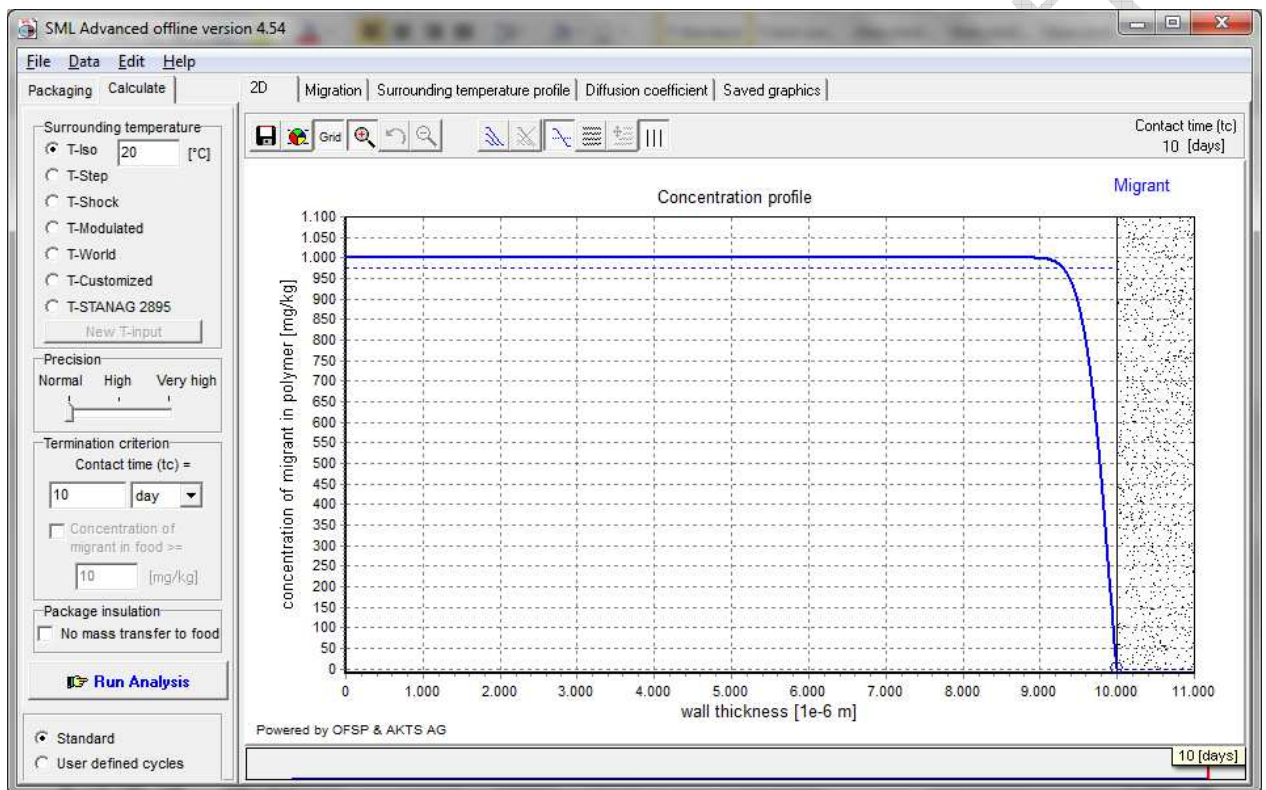
585 to be used for worst case calculation of specific migration under assumption of total transfer

586 => 2 x 99% layer thickness = 1600 µm

587 above 1600 µm two sides to be considered for calculation of migration if full immersion

588 testing applied

589



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595 **2h @ 100°C**

596 => 100% layer thickness = 8000 µm

597 no absolute barrier at thicknesses below 8000 µm

598 => 99% layer thickness = 5850 µm

599 => 1/2 x 99% layer thickness = 2925 µm

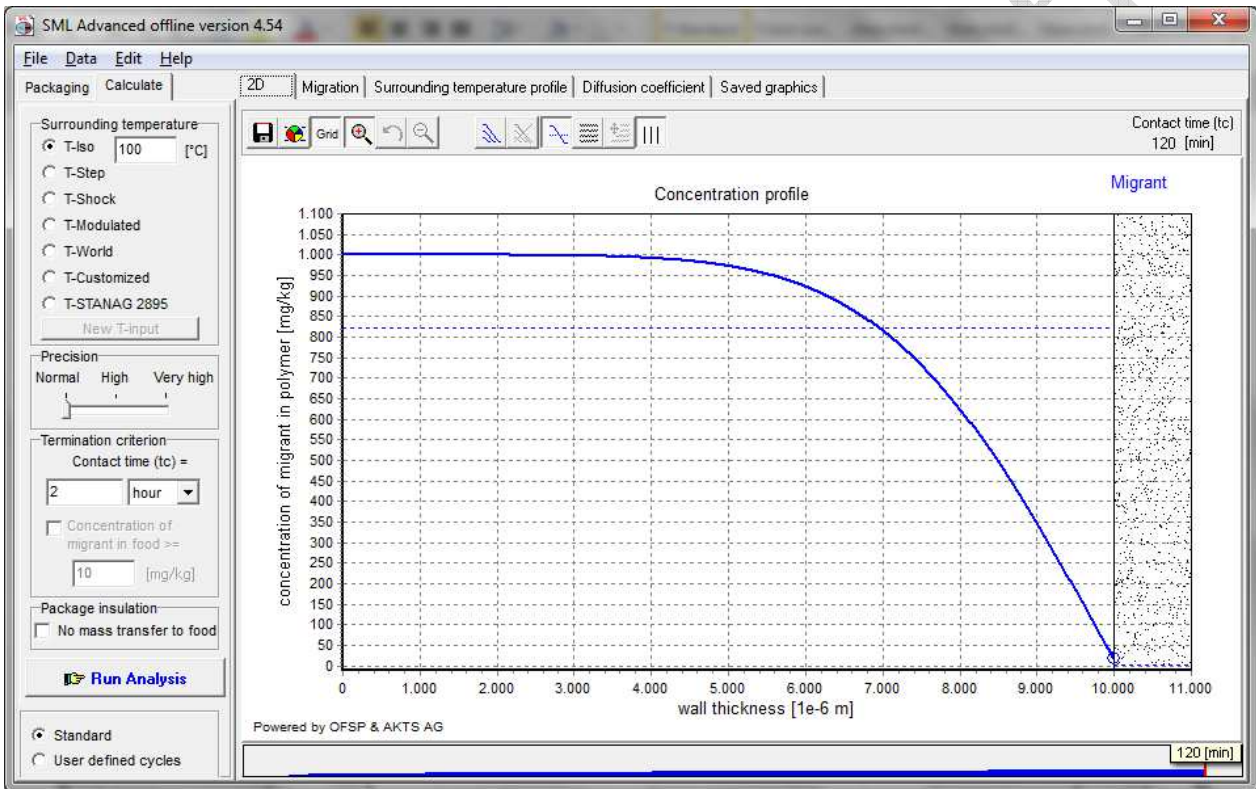
600 to be used for worst case calculation of specific migration under assumption of total transfer

601 => 2 x 99% layer thickness = 11700 µm

602 above 11700 µm two sides to be considered for calculation of migration if full immersion

603 testing applied

604



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610 ► molecular mass 251 - 500 g/mol

611 10d @ 60°C

612 => 100% layer thickness = 4600 µm

613 no absolute barrier at thicknesses below 4600 µm

614 => 99% layer thickness = 3400 µm

615 => 1/2 x 99% layer thickness = 1700 µm

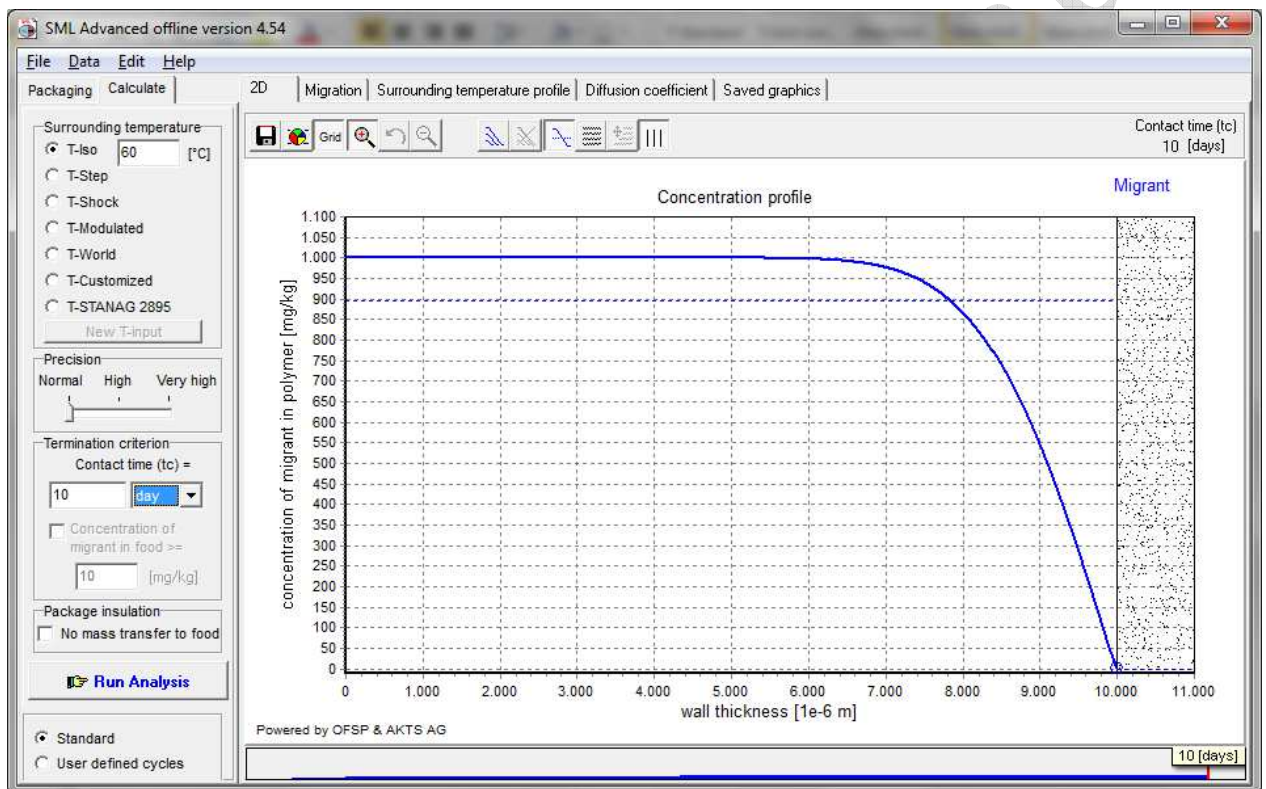
616 to be used for worst case calculation of specific migration under assumption of total transfer

617 => 2 x 99% layer thickness = 6800 µm

618 above 6800 µm two sides to be considered for calculation of migration if full immersion

619 testing applied

620



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626 **10d @ 40°C**

627 => 100% layer thickness = 1480 µm

628 no absolute barrier at thicknesses below 1480 µm

629 => 99% layer thickness = 1100 µm

630 => 1/2 x 99% layer thickness = 550 µm

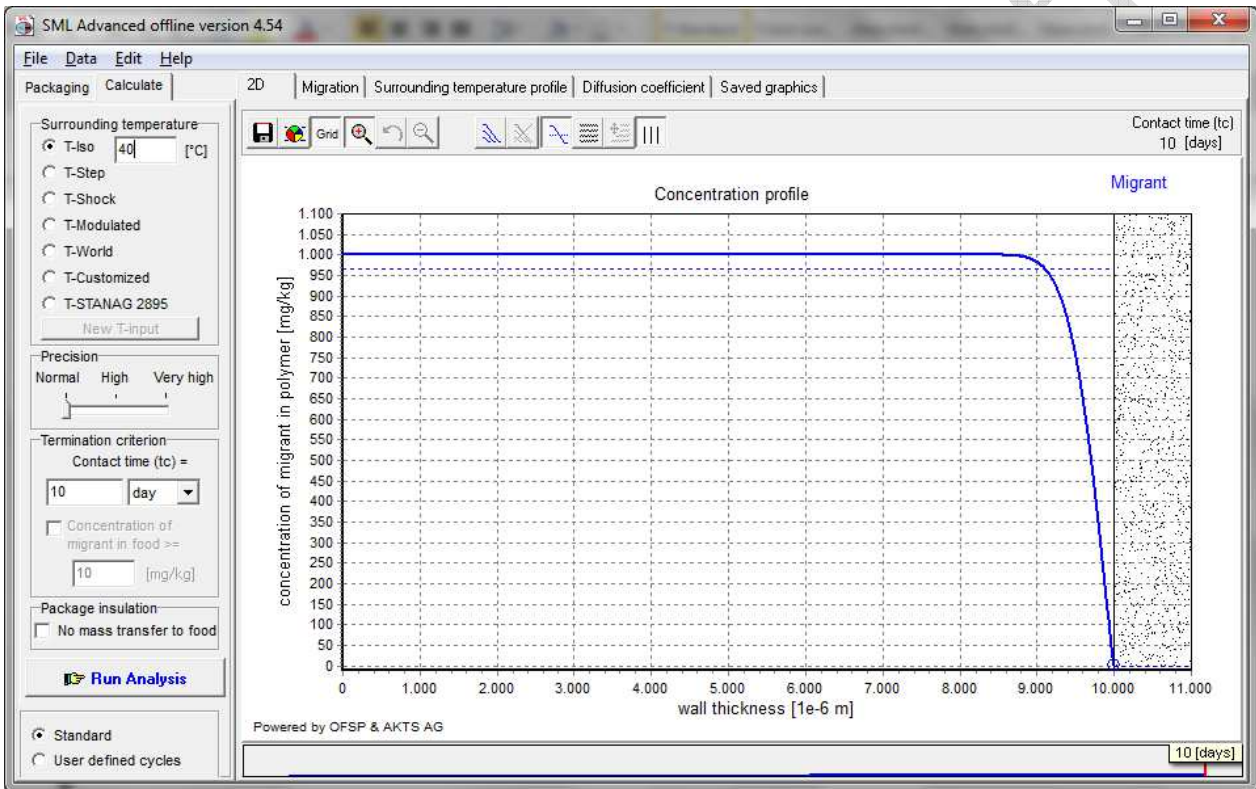
631 to be used for worst case calculation of specific migration under assumption of total transfer

632 => 2 x 99% layer thickness = 2200 µm

633 above 2200 µm two sides to be considered for calculation of migration if full immersion

634 testing applied

635



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641 **10d @ 20°C**

642 => 100% layer thickness = 440 µm

643 no absolute barrier at thicknesses below 440 µm

644 => 99% layer thickness = 310 µm

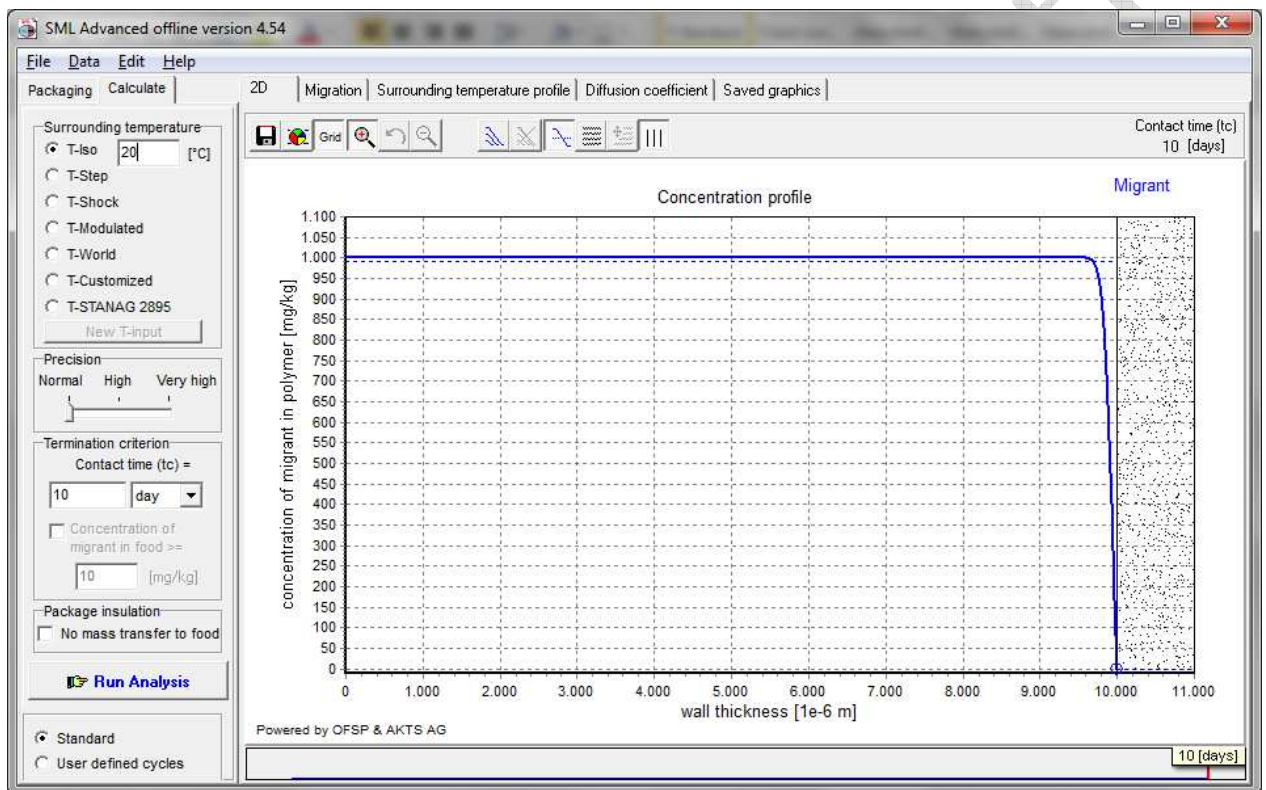
645 => 1/2 x 99% layer thickness = 155 µm

646 to be used for worst case calculation of specific migration under assumption of total transfer

647 => 2 x 99% layer thickness = 620 µm

648 above 620 µm two sides to be considered for calculation of migration if full immersion testing applied

650



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656 **2h @ 100°C**

657 => 100% layer thickness = 3000 µm

658 no absolute barrier at thicknesses below 3000 µm

659 => 99% layer thickness = 2160 µm

660 => 1/2 x 99% layer thickness = 1080 µm

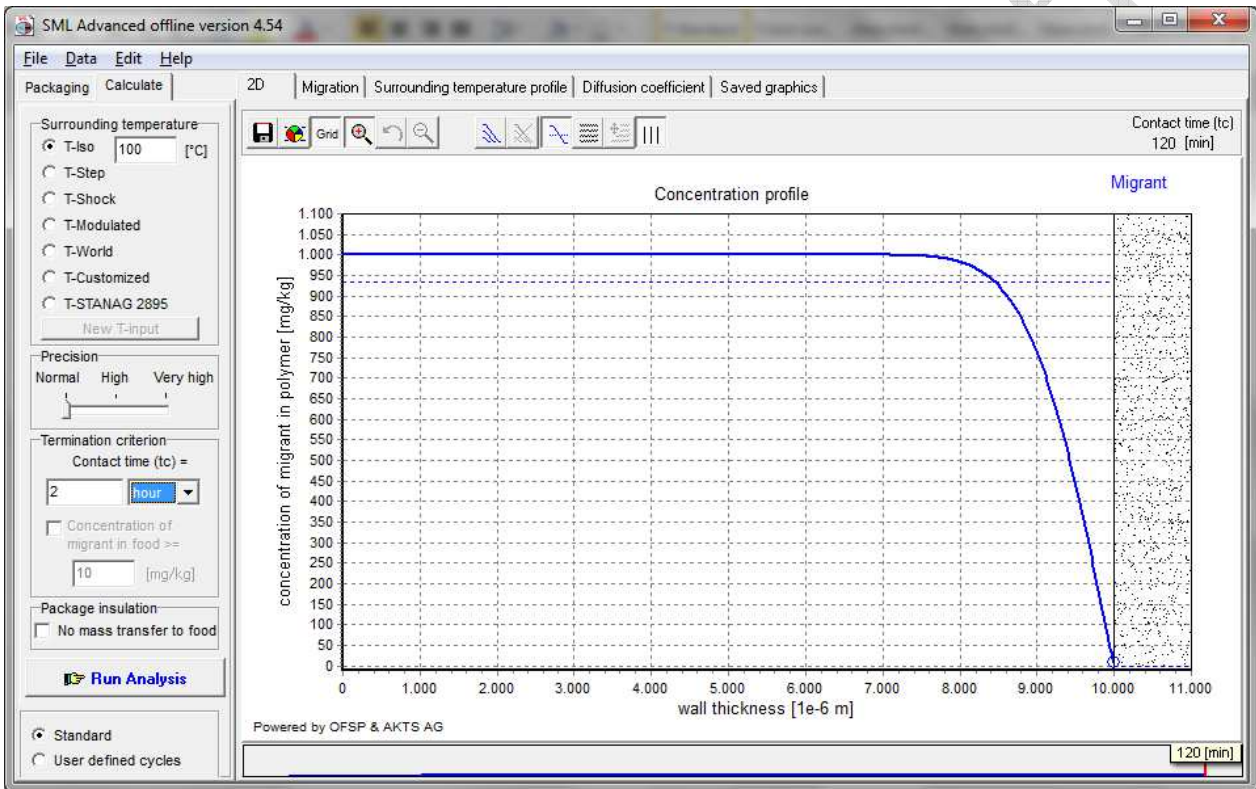
661 to be used for worst case calculation of specific migration under assumption of total transfer

662 => 2 x 99% layer thickness = 4320 µm

663 above 4320 µm two sides to be considered for calculation of migration if full immersion

664 testing applied

665



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671 ► molecular mass 501 - 750 g/mol

672 10d @ 60°C

673 => 100% layer thickness = 1400 µm

674 no absolute barrier at thicknesses below 1400 µm

675 => 99% layer thickness = 1040 µm

676 => 1/2 x 99% layer thickness = 520 µm

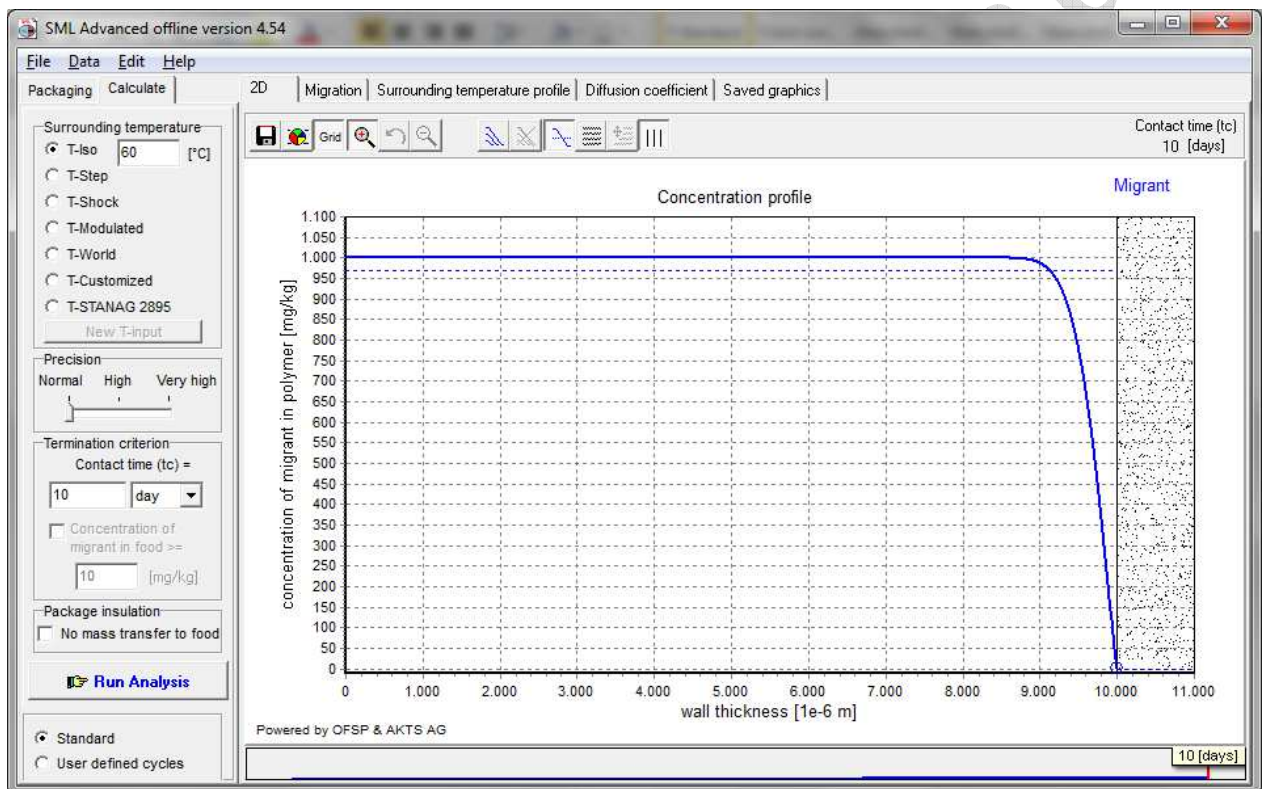
677 to be used for worst case calculation of specific migration under assumption of total transfer

678 => 2 x 99% layer thickness = 2080 µm

679 above 2080 µm two sides to be considered for calculation of migration if full immersion

680 testing applied

681



686

687 **10d @ 40°C**

688 => 100% layer thickness = 500 µm
689 no absolute barrier at thicknesses below 500 µm

690 => 99% layer thickness = 340 µm

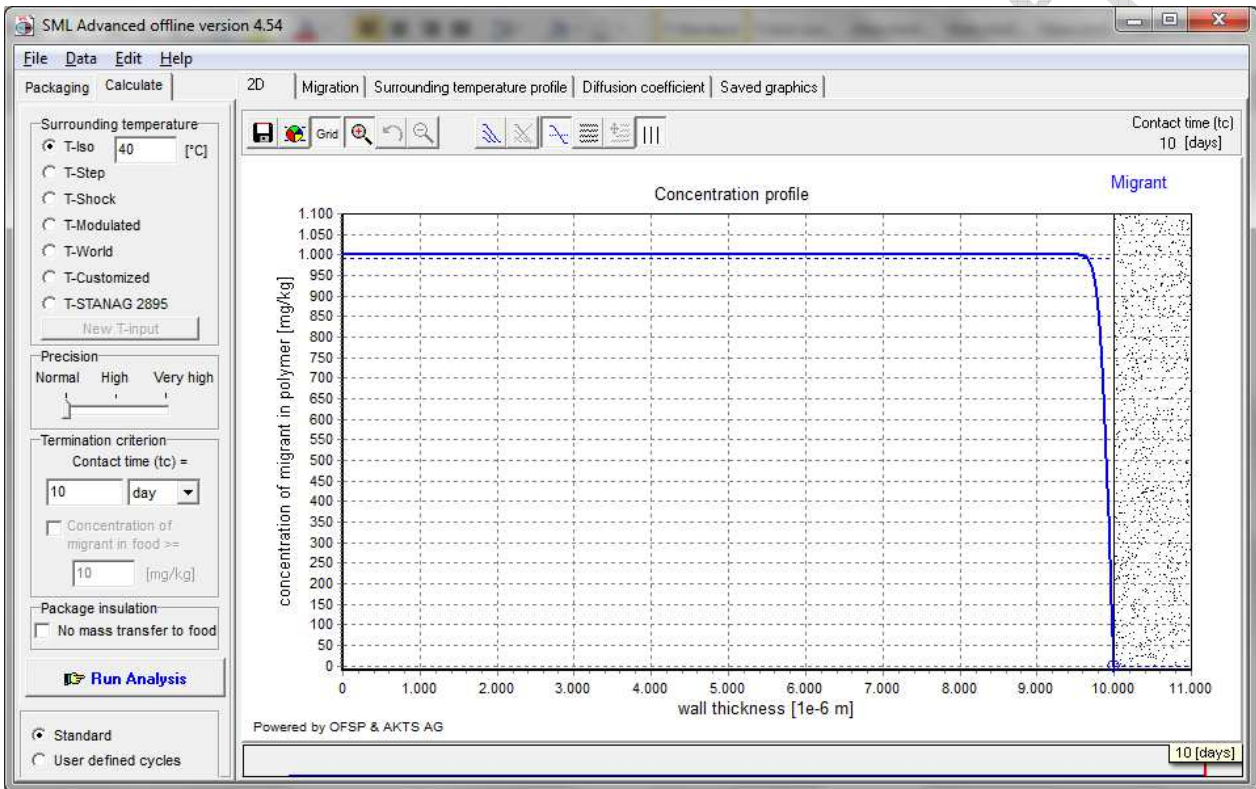
691 => 1/2 x 99% layer thickness = 170 µm

692 to be used for worst case calculation of specific migration under assumption of total transfer

693 => 2 x 99% layer thickness = 680 µm

694 above 680 µm two sides to be considered for calculation of migration if full immersion testing
695 applied

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702 **10d @ 20°C**

703 => 100% layer thickness = 160 µm

704 no absolute barrier at thicknesses below 160 µm

705 => 99% layer thickness = 110 µm

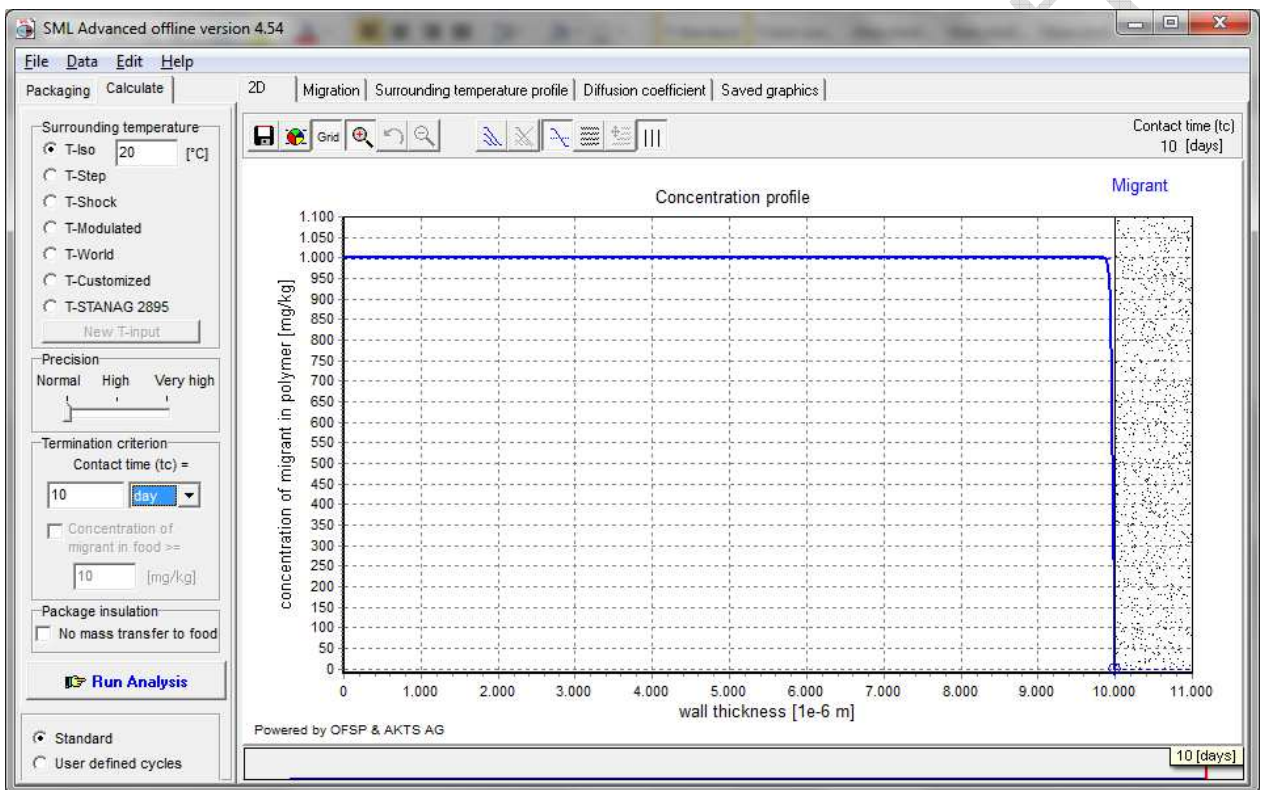
706 => 1/2 x 99% layer thickness = 55 µm

707 to be used for worst case calculation of specific migration under assumption of total transfer

708 => 2 x 99% layer thickness = 220 µm

709 above 220 µm two sides to be considered for calculation of migration if full immersion testing applied

711



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717 **2h @ 100°C**

718 => 100% layer thickness = 900 µm

719 no absolute barrier at thicknesses below 900 µm

720 => 99% layer thickness = 660 µm

721 => 1/2 x 99% layer thickness = 330 µm

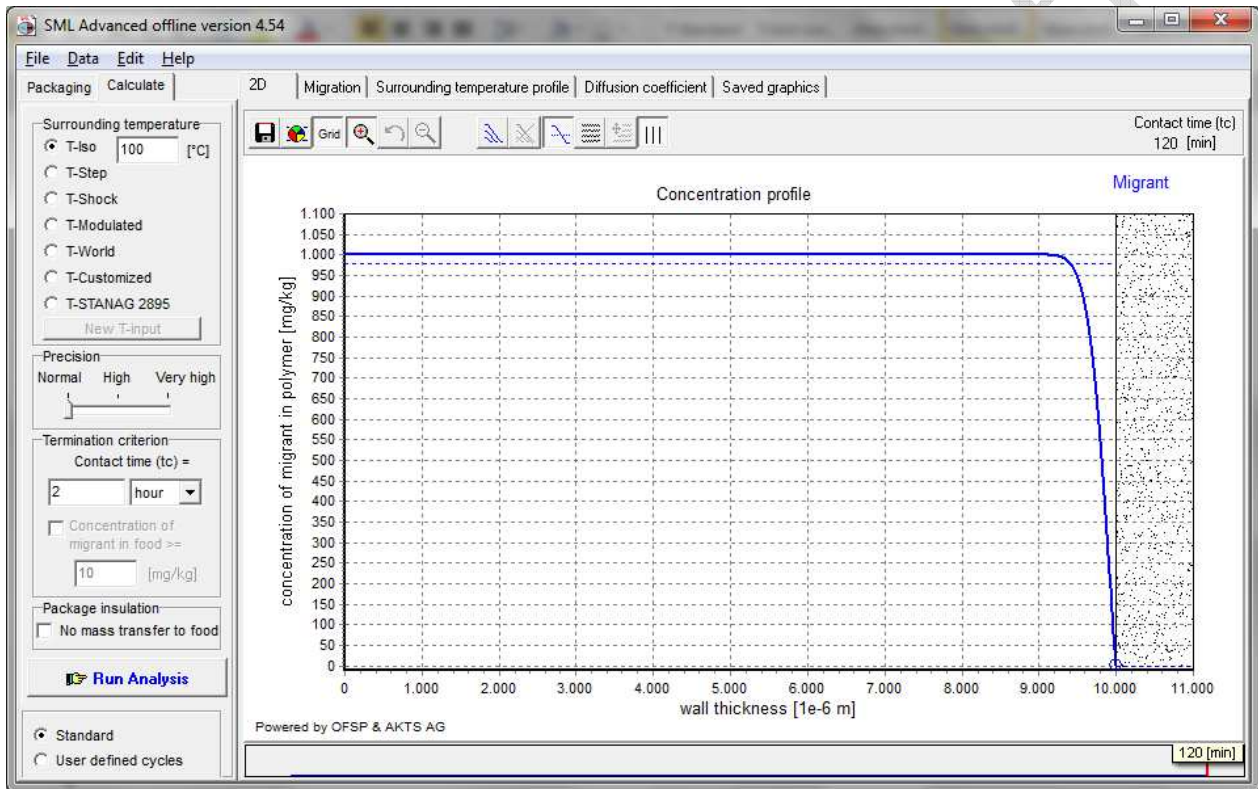
722 to be used for worst case calculation of specific migration under assumption of total transfer

723 => 2 x 99% layer thickness = 1320 µm

724 above 1320 µm two sides to be considered for calculation of migration if full immersion

725 testing applied

726



731

732 ► molecular mass 751 - 1000 g/mol

733 10d @ 60°C

734 => 100% layer thickness = 580 µm

735 no absolute barrier at thicknesses below 580 µm

736 => 99% layer thickness = 420 µm

737 => 1/2 x 99% layer thickness = 210 µm

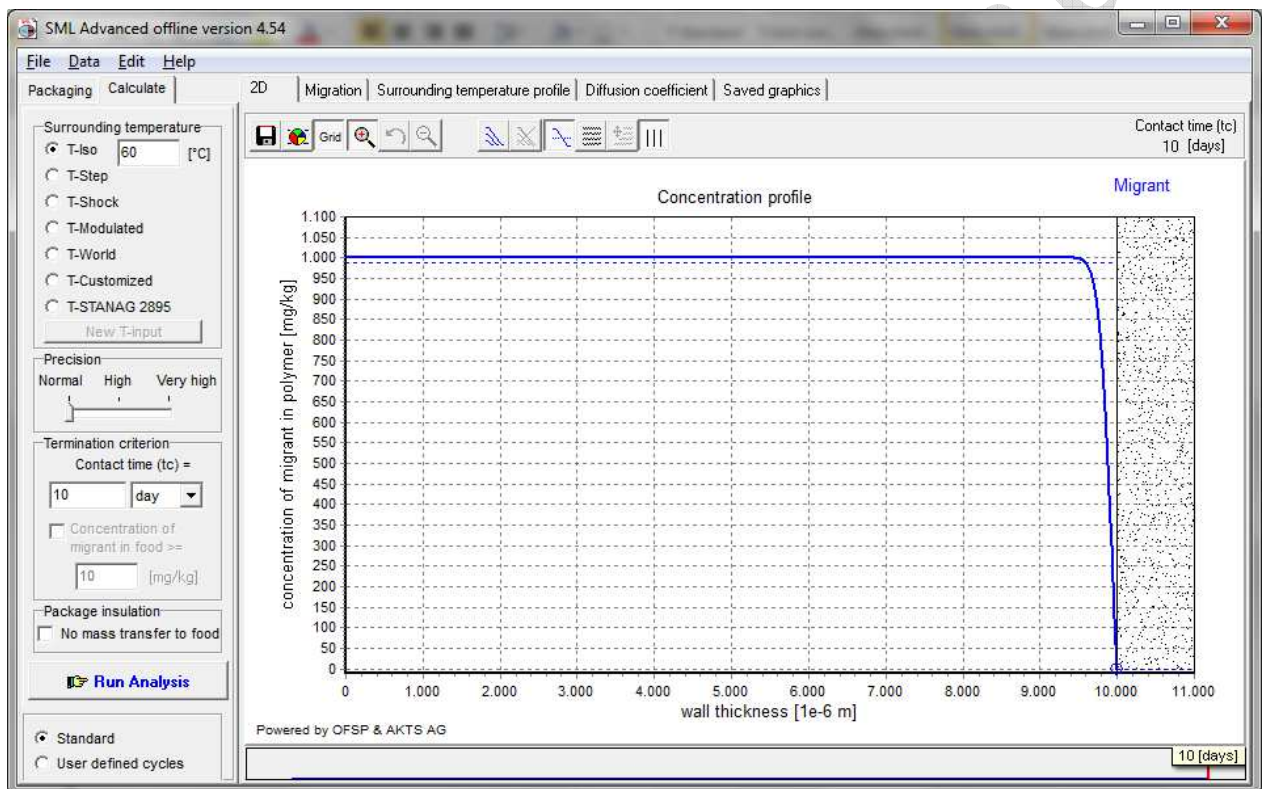
738 to be used for worst case calculation of specific migration under assumption of total transfer

739 => 2 x 99% layer thickness = 840 µm

740 above 840 µm two sides to be considered for calculation of migration if full immersion testing

741 applied

742



743

744

745

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748 **10d @ 40°C**

749 => 100% layer thickness = 220 µm

750 no absolute barrier at thicknesses below 220 µm

751 => 99% layer thickness = 144 µm

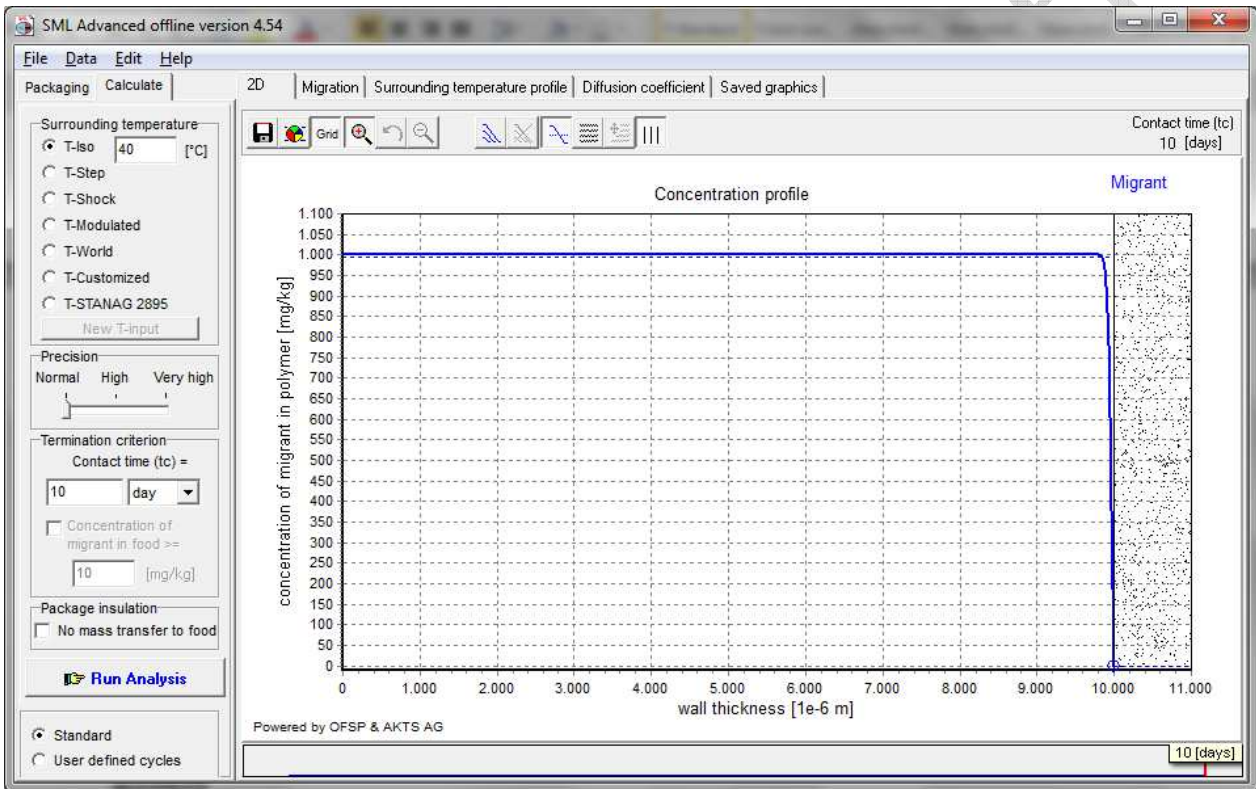
752 => 1/2 x 99% layer thickness = 72 µm

753 to be used for worst case calculation of specific migration under assumption of total transfer

754 => 2 x 99% layer thickness = 288 µm

755 above 288 µm two sides to be considered for calculation of migration if full immersion testing
756 applied

757



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763 **10d @ 20°C**

764 => 100% layer thickness = 70 µm

765 no absolute barrier at thicknesses below 70 µm

766 => 99% layer thickness = 40 µm

767 => 1/2 x 99% layer thickness = 20 µm

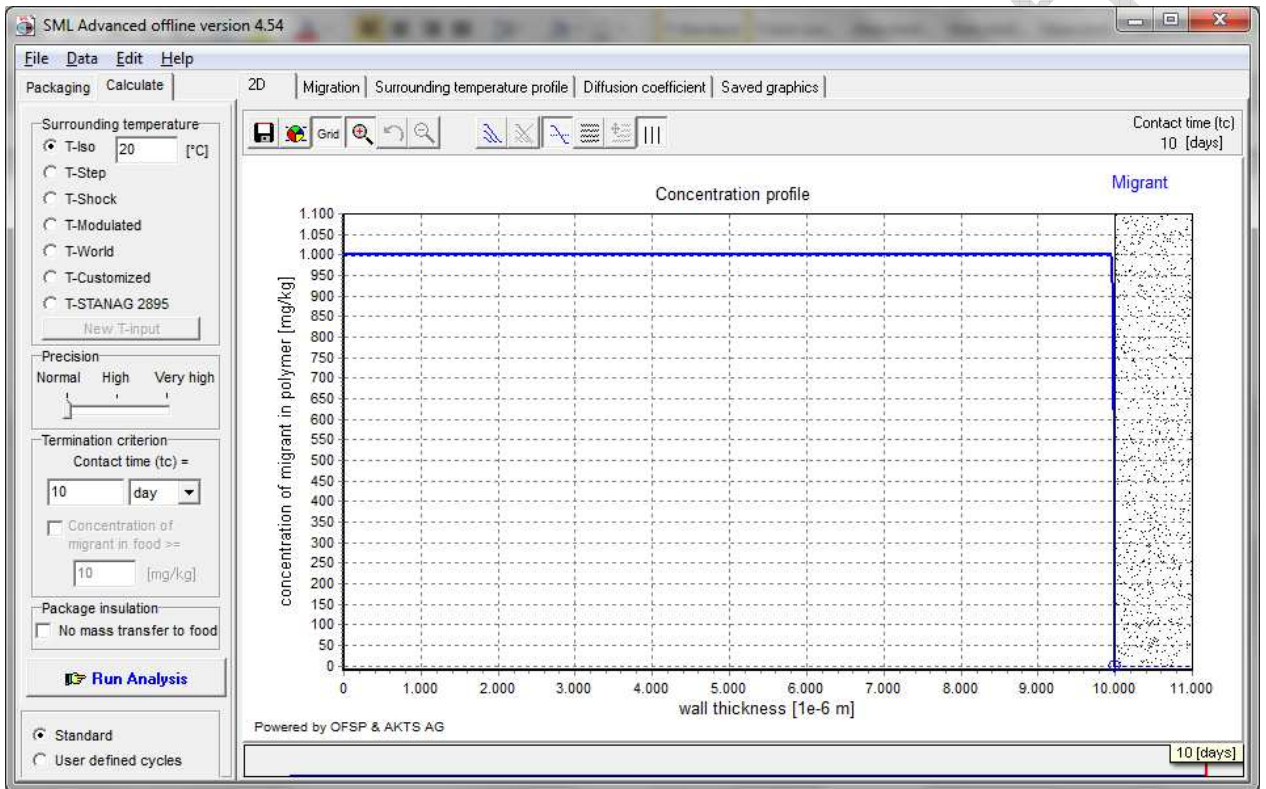
768 to be used for worst case calculation of specific migration under assumption of total transfer

769 => 2 x 99% layer thickness = 80 µm

770 above 80 µm two sides to be considered for calculation of migration if full immersion testing

771 applied

772



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778 **2h @ 100°C**

779 => 100% layer thickness = 380 µm

780 no absolute barrier at thicknesses below 380 µm

781 => 99% layer thickness = 270 µm

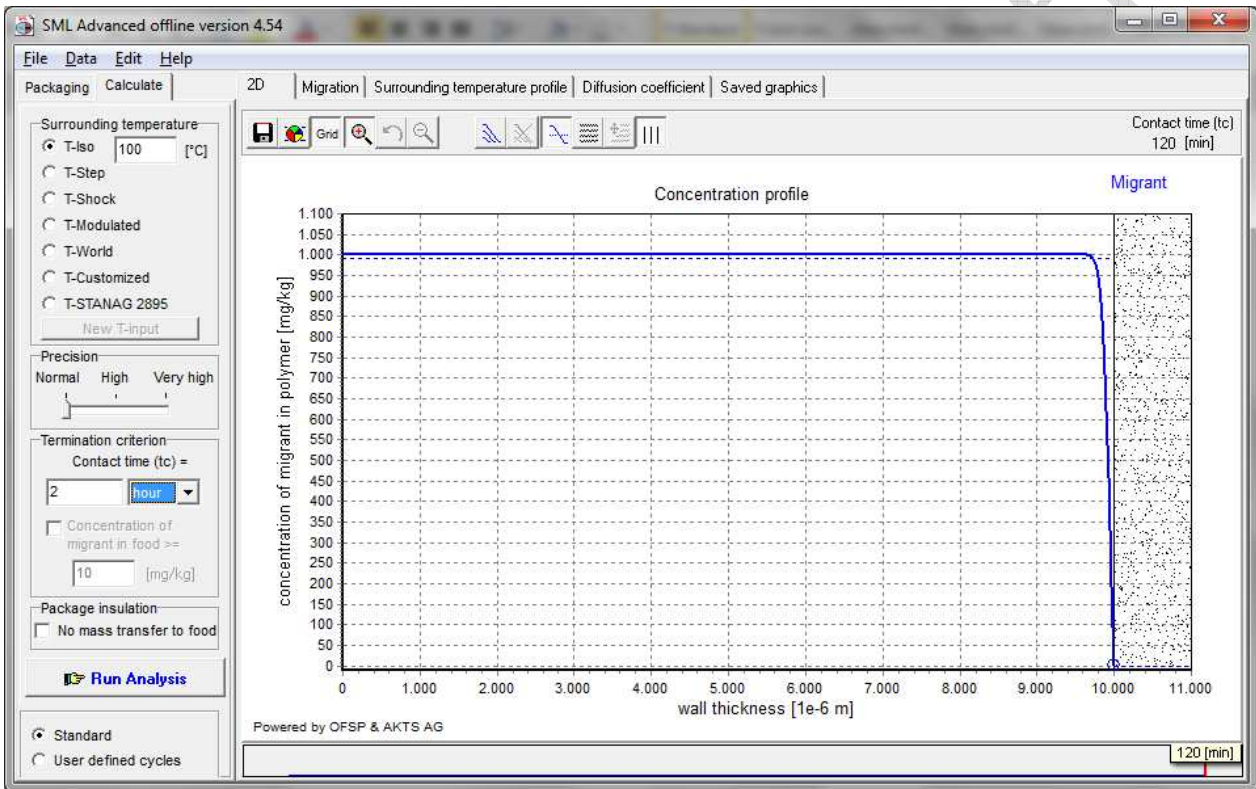
782 => 1/2 x 99% layer thickness = 135 µm

783 to be used for worst case calculation of specific migration under assumption of total transfer

784 => 2 x 99% layer thickness = 540 µm

785 above 540 µm two sides to be considered for calculation of migration if full immersion testing
786 applied

787



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793 **PET, PBT, PEN**

794 **► molecular mass 100 - 250 g/mol**

795 **10d @ 60°C**

796 => 100% layer thickness = 91

797 no absolute barrier at thicknesses below 91 µm

798 => 99% layer thickness = 80 µm

799 => 1/2 x 99% layer thickness = 40 µm

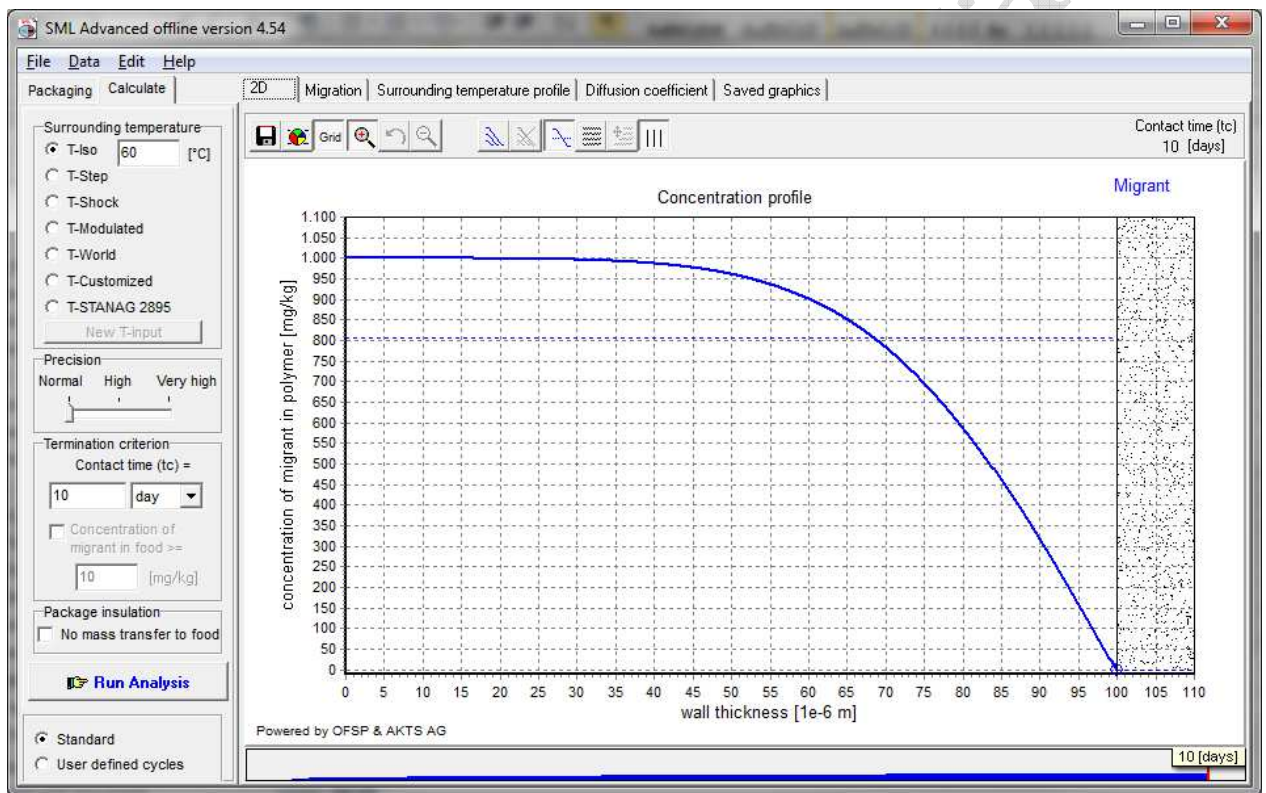
800 to be used for worst case calculation of specific migration under assumption of total transfer

801 => 2 x 99% layer thickness = 160 µm

802 above 160 µm two sides to be considered for calculation of migration if full immersion testing

803 applied

804



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810 **10d @ 40°C**

811 => 100% layer thickness = 31

812 no absolute barrier at thicknesses below 31 µm

813 => 99% layer thickness = 26 µm

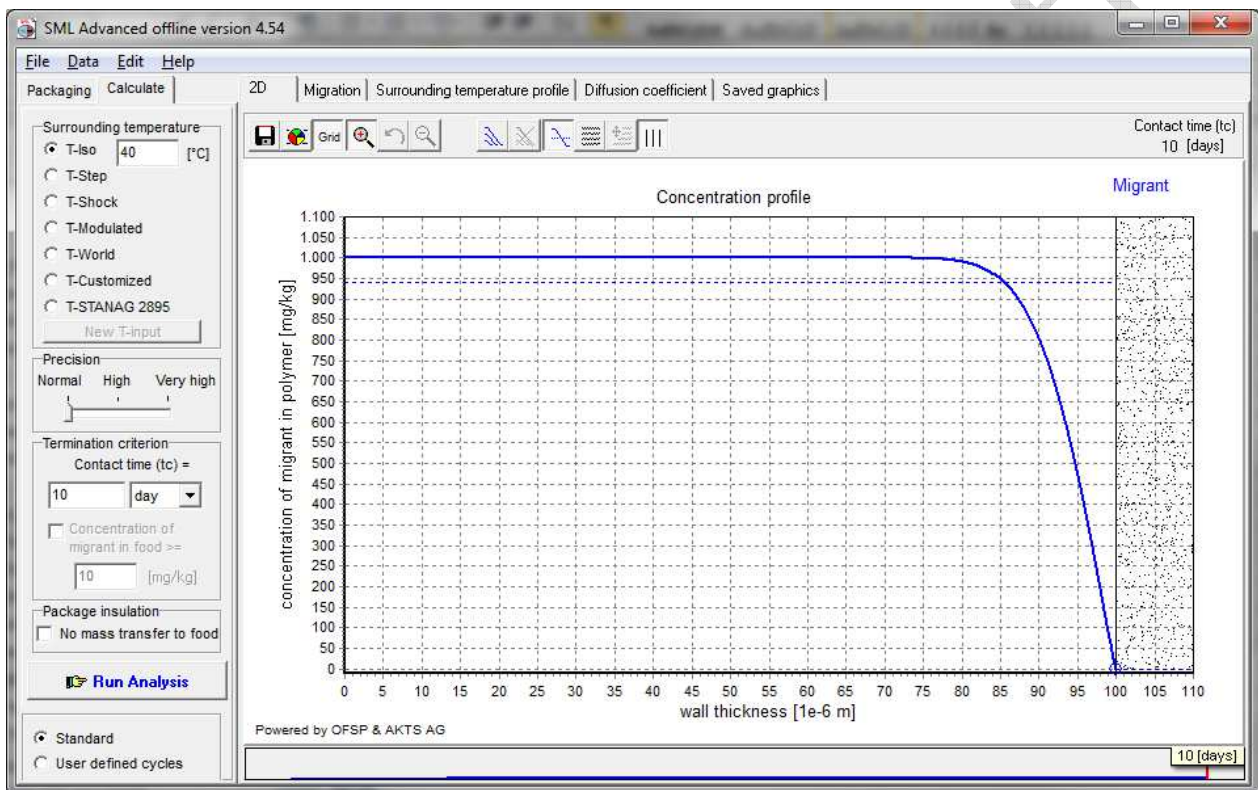
814 => 1/2 x 99% layer thickness = 13 µm

815 to be used for worst case calculation of specific migration under assumption of total transfer

816 => 2 x 99% layer thickness = 52

817 above 52 µm two sides to be considered for calculation of migration if full immersion testing
818 applied

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825 **10d @ 20°C**

826 => 100% layer thickness = 9 µm

827 no absolute barrier at thicknesses below 9 µm

828 => 99% layer thickness = 7 µm

829 => 1/2 x 99% layer thickness = 4 µm

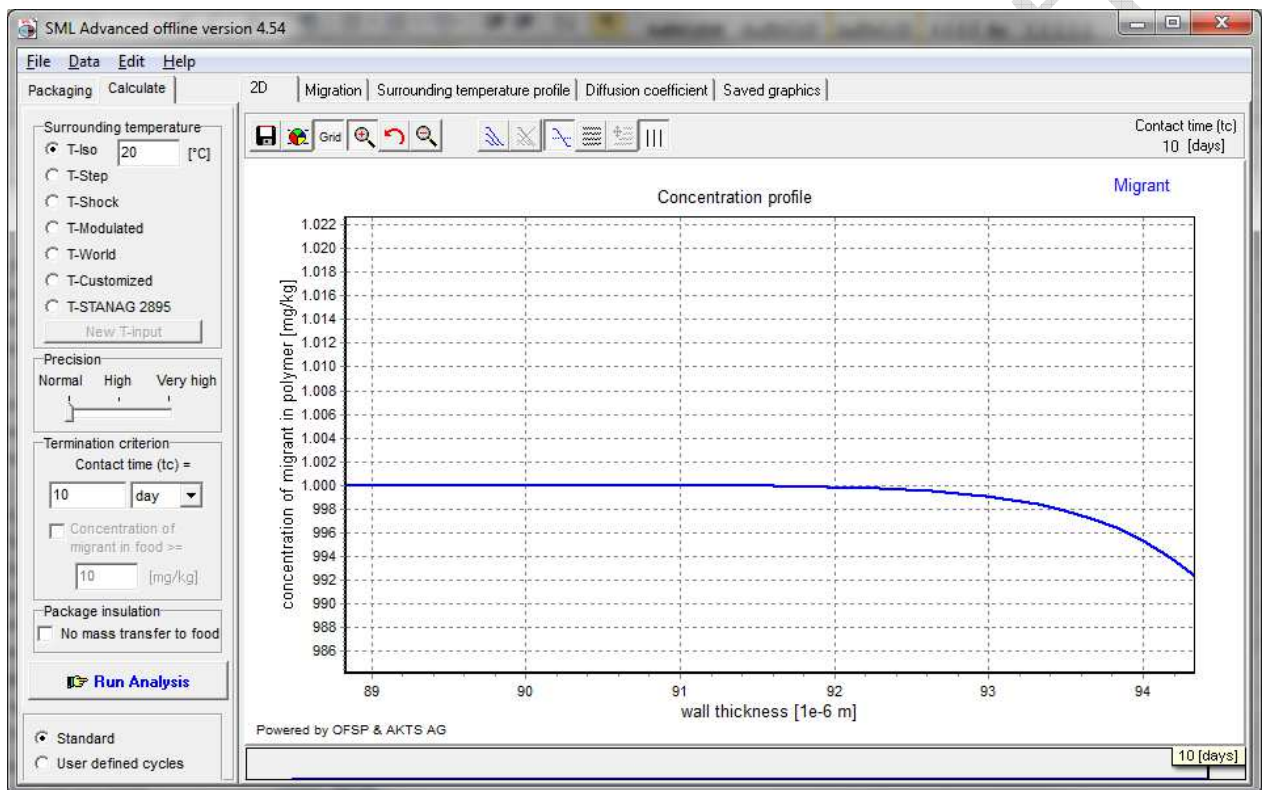
830 to be used for worst case calculation of specific migration under assumption of total transfer

831 => 2 x 99% layer thickness = 14 µm

832 above 14 µm two sides to be considered for calculation of migration if full immersion testing

833 applied

834



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840 **2h @ 100°C**

841 => 100% layer thickness = 61 µm

842 no absolute barrier at thicknesses below 61 µm

843 => 99% layer thickness = 50 µm

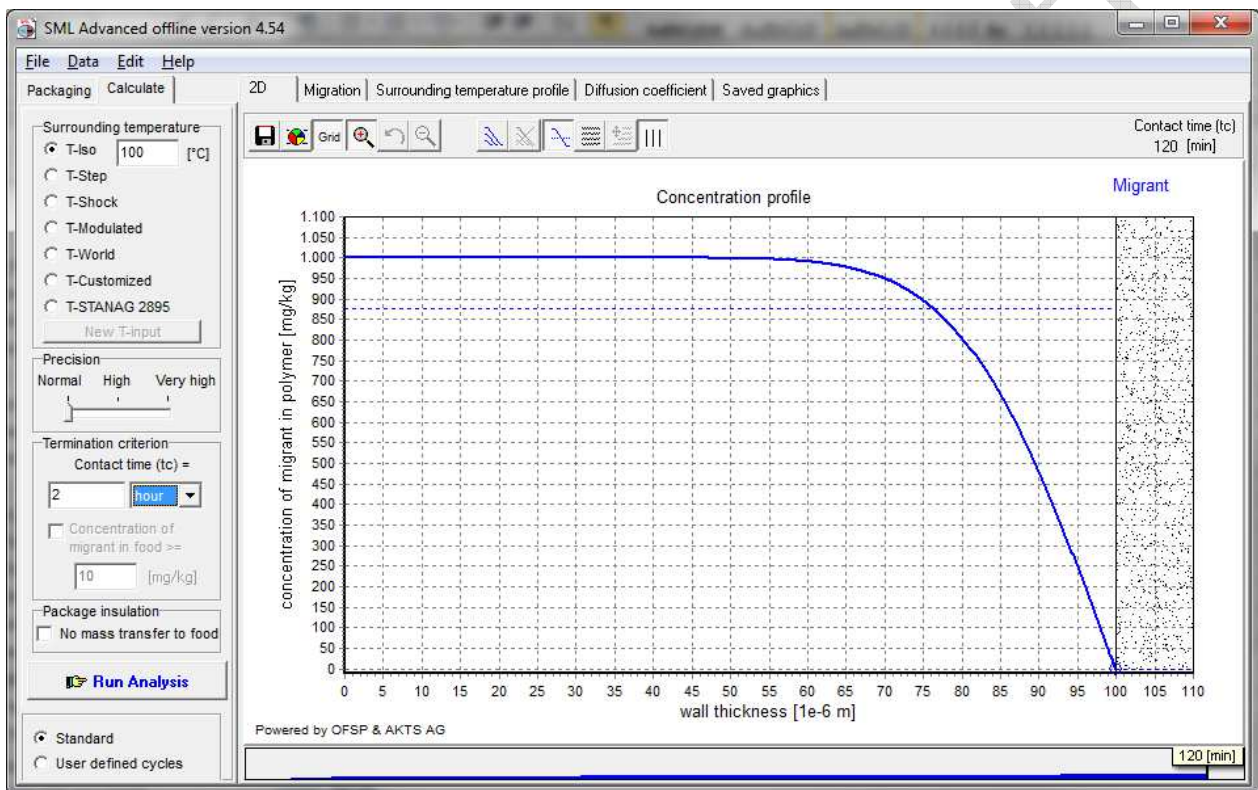
844 => 1/2 x 99% layer thickness = 25 µm

845 to be used for worst case calculation of specific migration under assumption of total transfer

846 => 2 x 99% layer thickness = 100 µm

847 above 100 µm two sides to be considered for calculation of migration if full immersion testing
848 applied

849



850

851

852

853

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855 ► molecular mass 251 - 500 g/mol

856 10d @ 60°C

857 => 100% layer thickness = 35 µm

858 no absolute barrier at thicknesses below 35 µm

859 => 99% layer thickness = 29 µm

860 => 1/2 x 99% layer thickness = 15 µm

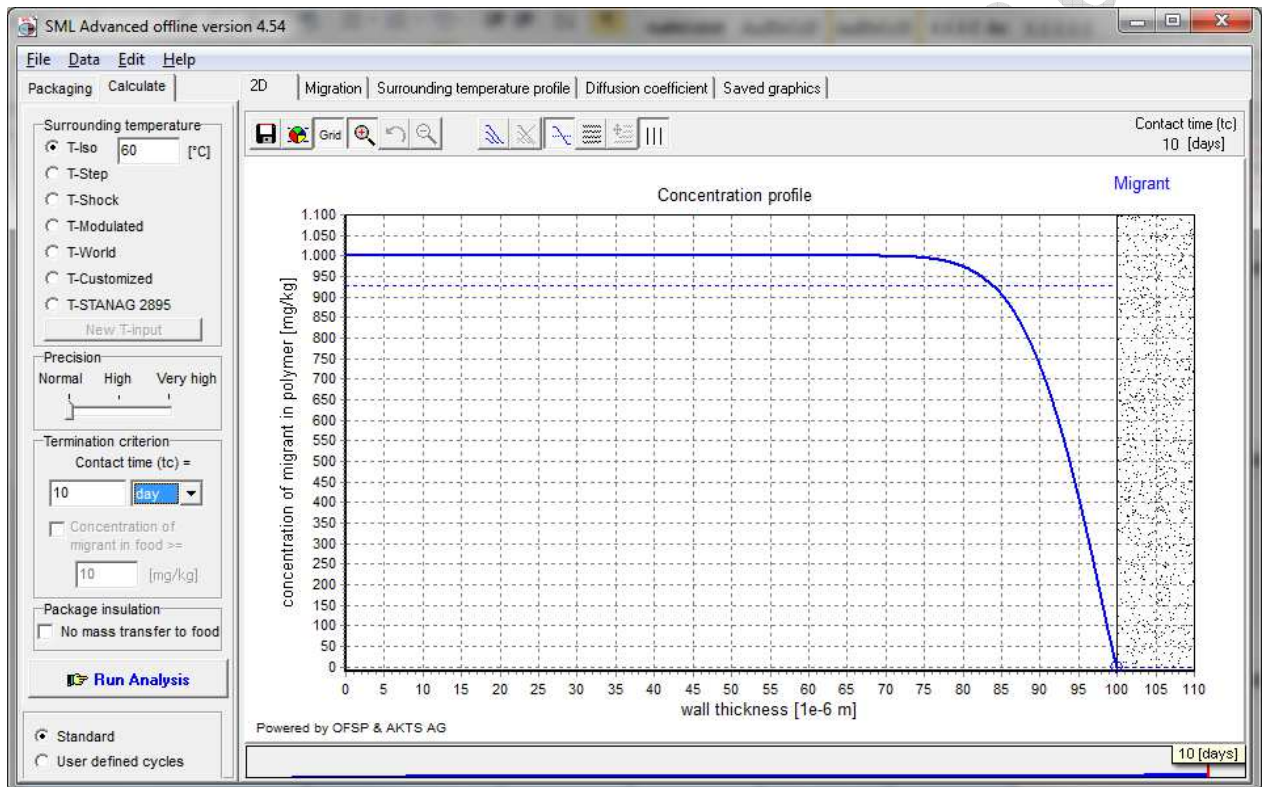
861 to be used for worst case calculation of specific migration under assumption of total transfer

862 => 2 x 99% layer thickness = 58 µm

863 above 58 µm two sides to be considered for calculation of migration if full immersion testing

864 applied

865



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871 **10d @ 40°C**

872 => 100% layer thickness = 14 µm

873 no absolute barrier at thicknesses below 14 µm

874 => 99% layer thickness = 9.4 µm

875 => 1/2 x 99% layer thickness = 5 µm

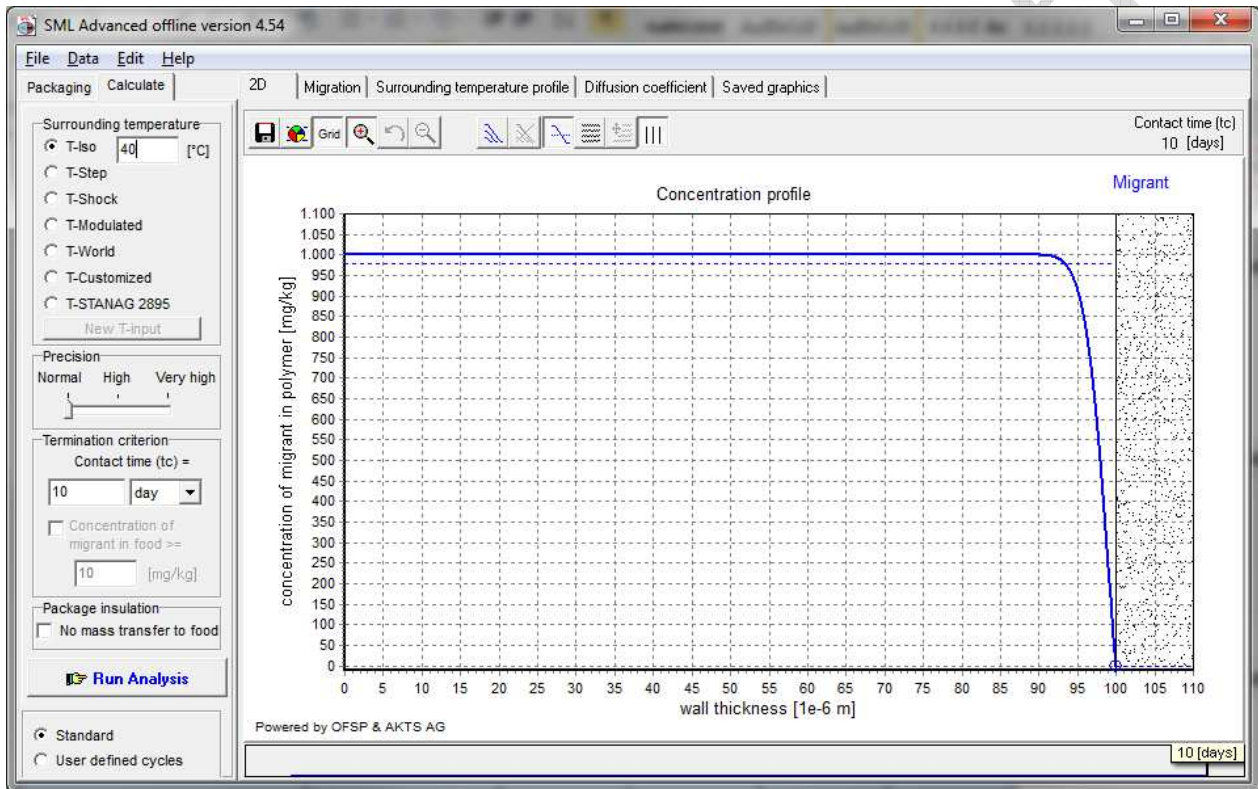
876 to be used for worst case calculation of specific migration under assumption of total transfer

877 => 2 x 99% layer thickness = 19 µm

878 above 19 µm two sides to be considered for calculation of migration if full immersion testing

879 applied

880



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886 **10d @ 20°C**

887 => 100% layer thickness = 4 µm

888 no absolute barrier at thicknesses below 4 µm

889 => 99% layer thickness = 3 µm

890 => 1/2 x 99% layer thickness = 1.5 µm

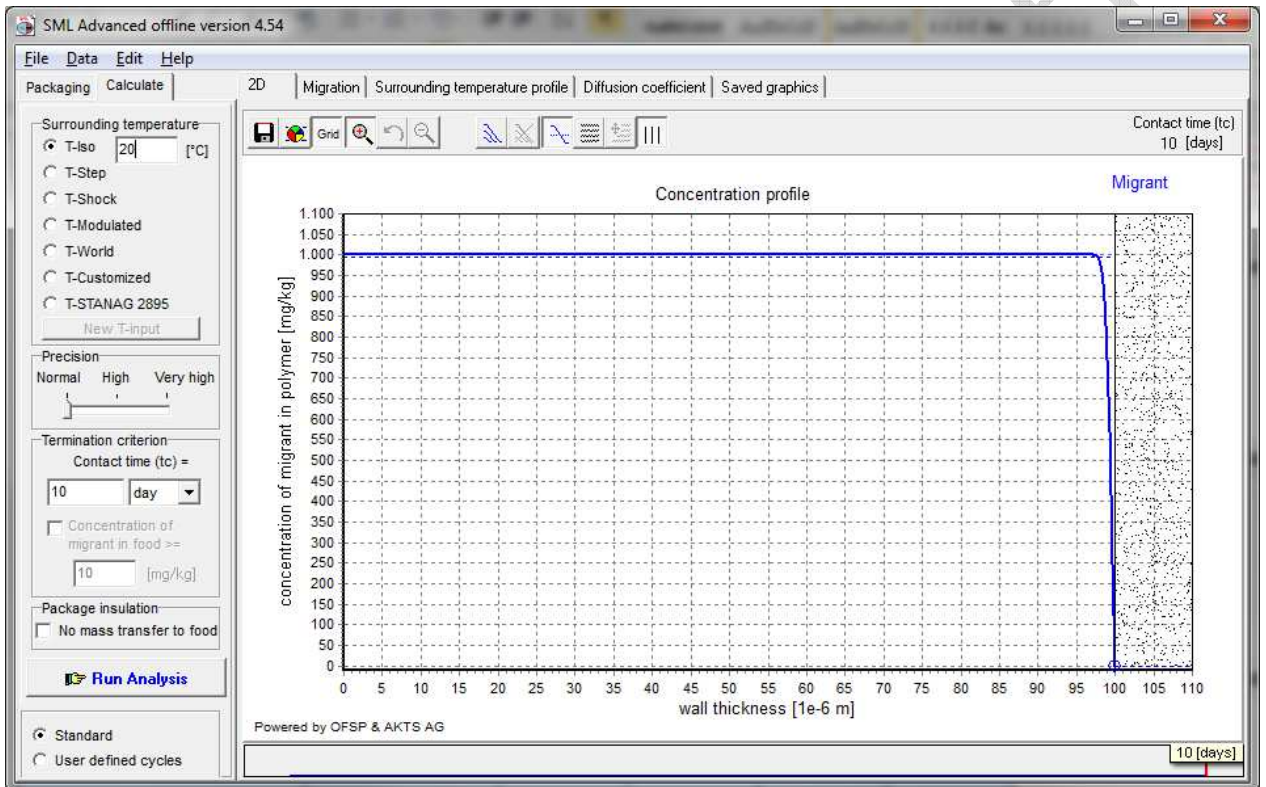
891 to be used for worst case calculation of specific migration under assumption of total transfer

892 => 2 x 99% layer thickness = 6 µm

893 above 6 µm two sides to be considered for calculation of migration if full immersion testing

894 applied

895



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901 **2h @ 100°C**

902 => 100% layer thickness = 23 µm

903 no absolute barrier at thicknesses below 23 µm

904 => 99% layer thickness = 19 µm

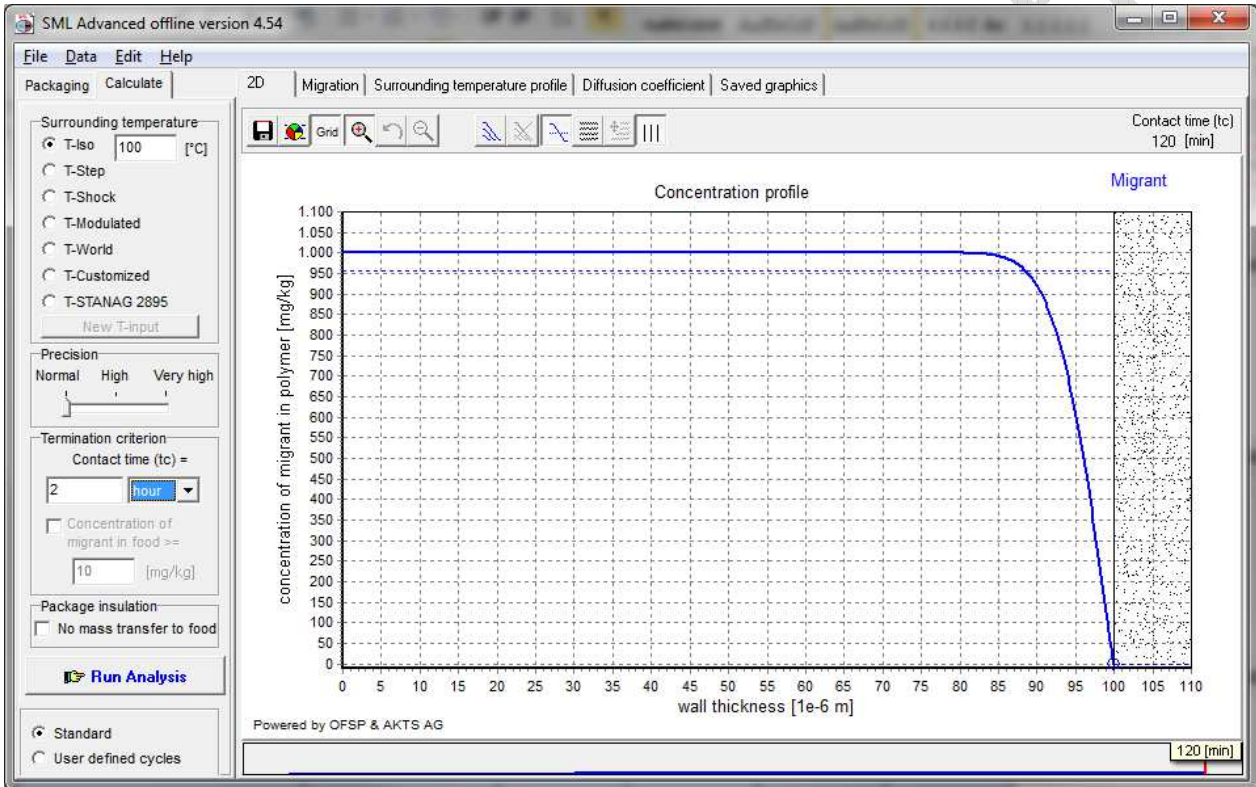
905 => 1/2 x 99% layer thickness = 10 µm

906 to be used for worst case calculation of specific migration under assumption of total transfer

907 => 2 x 99% layer thickness = 38 µm

908 above 38 µm two sides to be considered for calculation of migration if full immersion testing
909 applied

910



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916 ► molecular mass 501 - 750 g/mol

917 10d @ 60°C

918 => 100% layer thickness = 12 µm

919 no absolute barrier at thicknesses below 12 µm

920 => 99% layer thickness = 9 µm

921 => 1/2 x 99% layer thickness = 5 µm

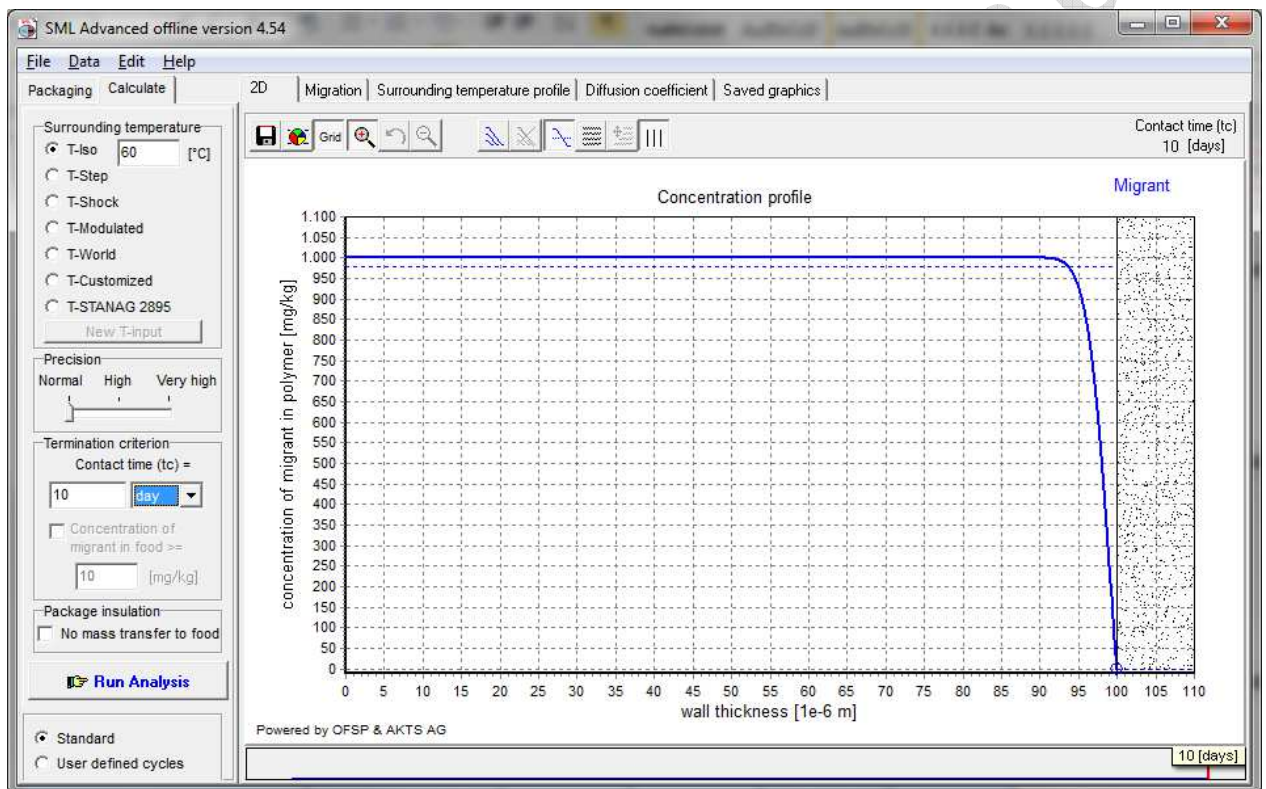
922 to be used for worst case calculation of specific migration under assumption of total transfer

923 => 2 x 99% layer thickness = 18 µm

924 above 18 µm two sides to be considered for calculation of migration if full immersion testing

925 applied

926



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932 **10d @ 40°C**

933 => 100% layer thickness = 4 µm

934 no absolute barrier at thicknesses below 4 µm

935 => 99% layer thickness = 3 µm

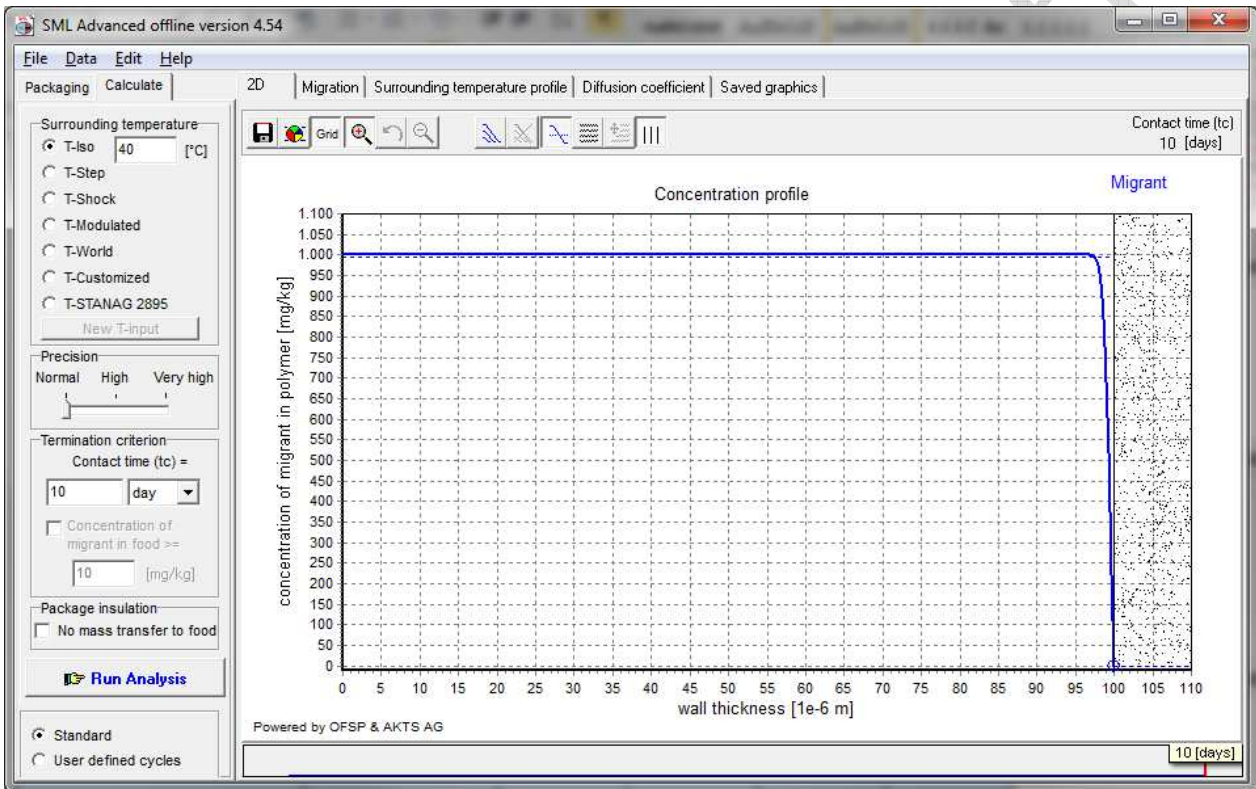
936 => 1/2 x 99% layer thickness = 1.5 µm

937 to be used for worst case calculation of specific migration under assumption of total transfer

938 => 2 x 99% layer thickness = 6 µm

939 above 6 µm two sides to be considered for calculation of migration if full immersion testing
940 applied

941



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947 **10d @ 20°C**

948 => 100% layer thickness = 1.3 µm

949 no absolute barrier at thicknesses below 1.3 µm

950 => 99% layer thickness = 1 µm

951 => 1/2 x 99% layer thickness = 0.5 µm

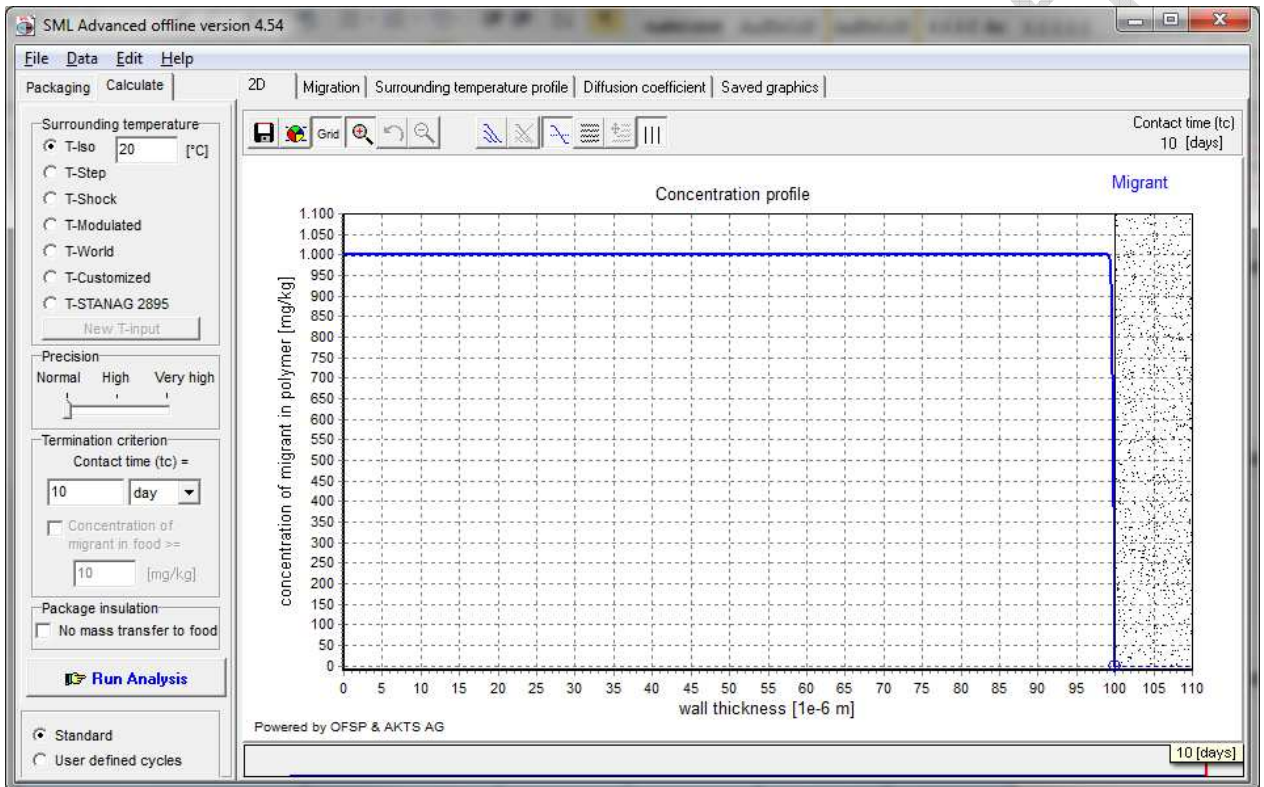
952 to be used for worst case calculation of specific migration under assumption of total transfer

953 => 2 x 99% layer thickness = 2 µm

954 above 2 µm two sides to be considered for calculation of migration if full immersion testing

955 applied

956



957

958

959

960

961

962 **2h @ 100°C**

963 => 100% layer thickness = 7 µm

964 no absolute barrier at thicknesses below 7 µm

965 => 99% layer thickness = 6 µm

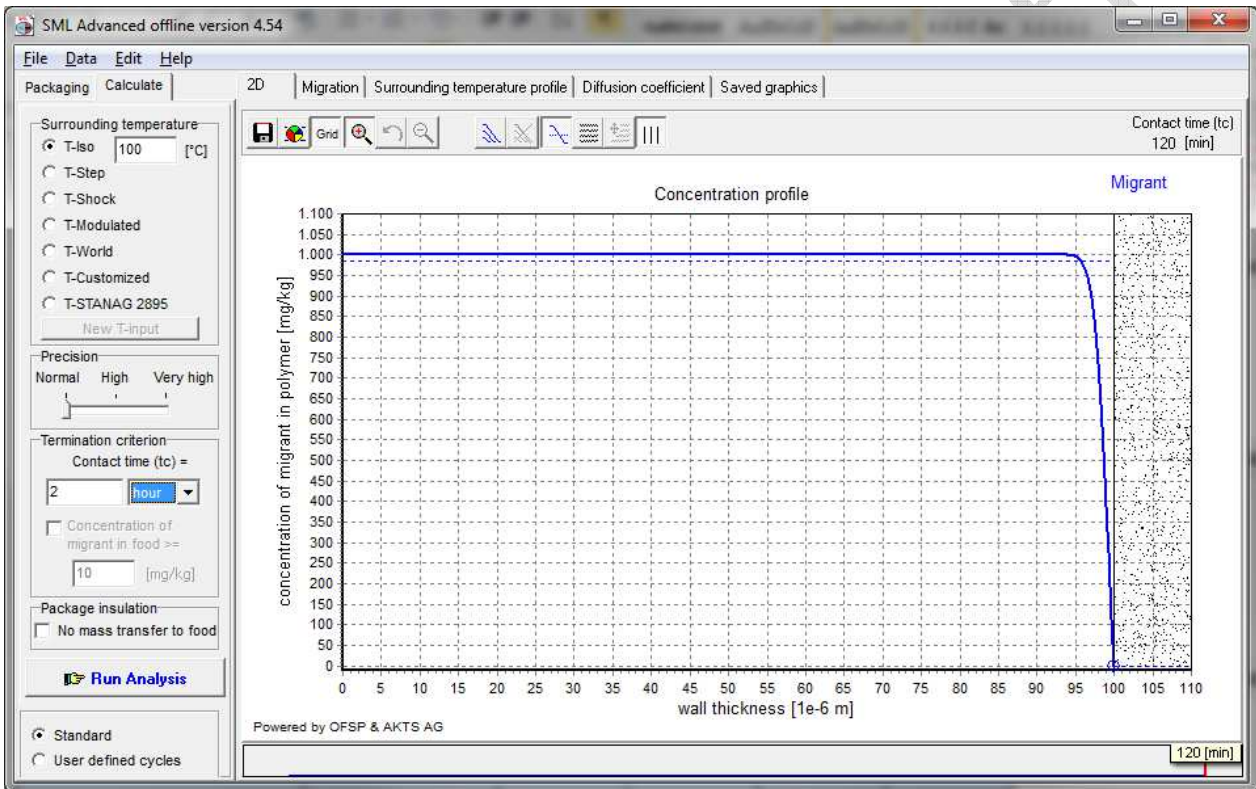
966 => 1/2 x 99% layer thickness = 3 µm

967 to be used for worst case calculation of specific migration under assumption of total transfer

968 => 2 x 99% layer thickness = 12 µm

969 above 12 µm two sides to be considered for calculation of migration if full immersion testing
970 applied

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977 ► molecular mass 751 - 1000 g/mol

978 10d @ 60°C

979 => 100% layer thickness = 4.4 µm

980 no absolute barrier at thicknesses below 4.4 µm

981 => 99% layer thickness = 3.6 µm

982 => 1/2 x 99% layer thickness = 2 µm

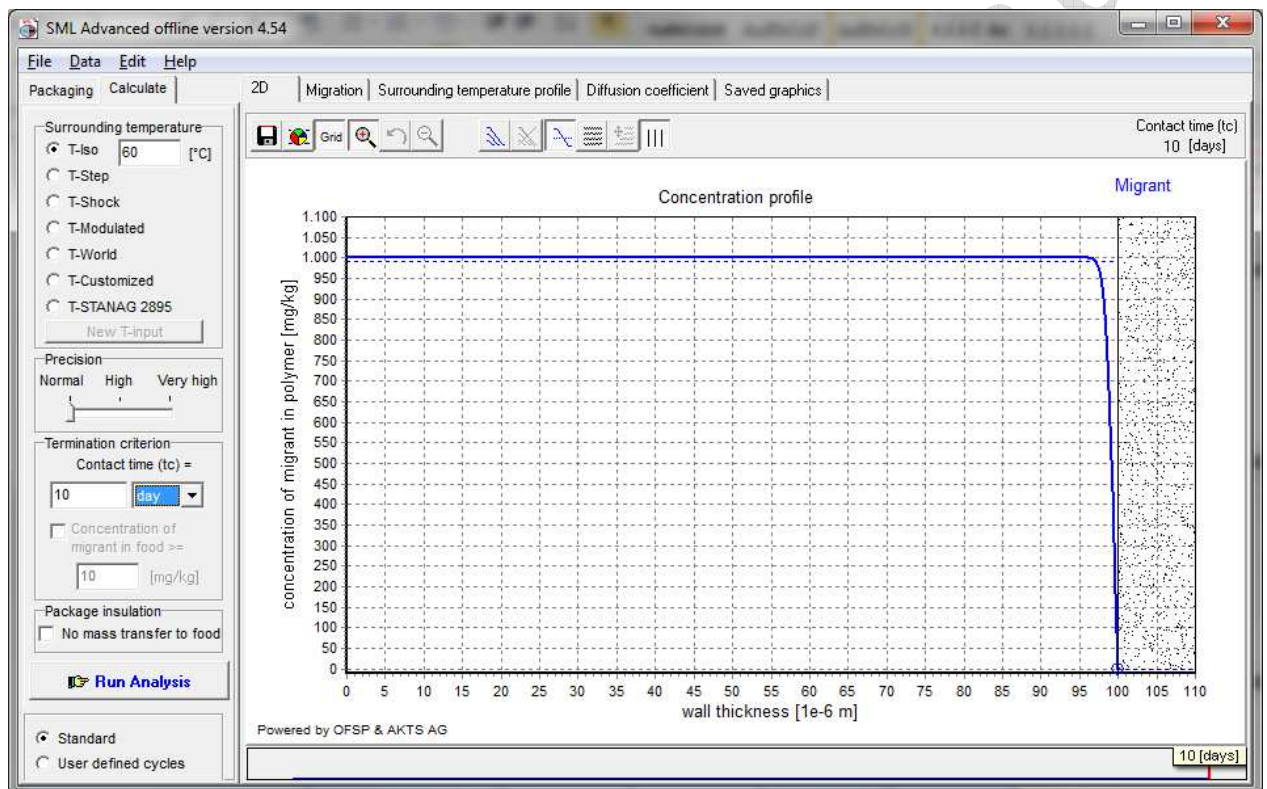
983 to be used for worst case calculation of specific migration under assumption of total transfer

984 => 2 x 99% layer thickness = 7 µm

985 above 7 µm two sides to be considered for calculation of migration if full immersion testing

986 applied

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993 **10d @ 40°C**

994 => 100% layer thickness = 1.7 µm

995 no absolute barrier at thicknesses below 1.7 µm

996 => 99% layer thickness = 1.3 µm

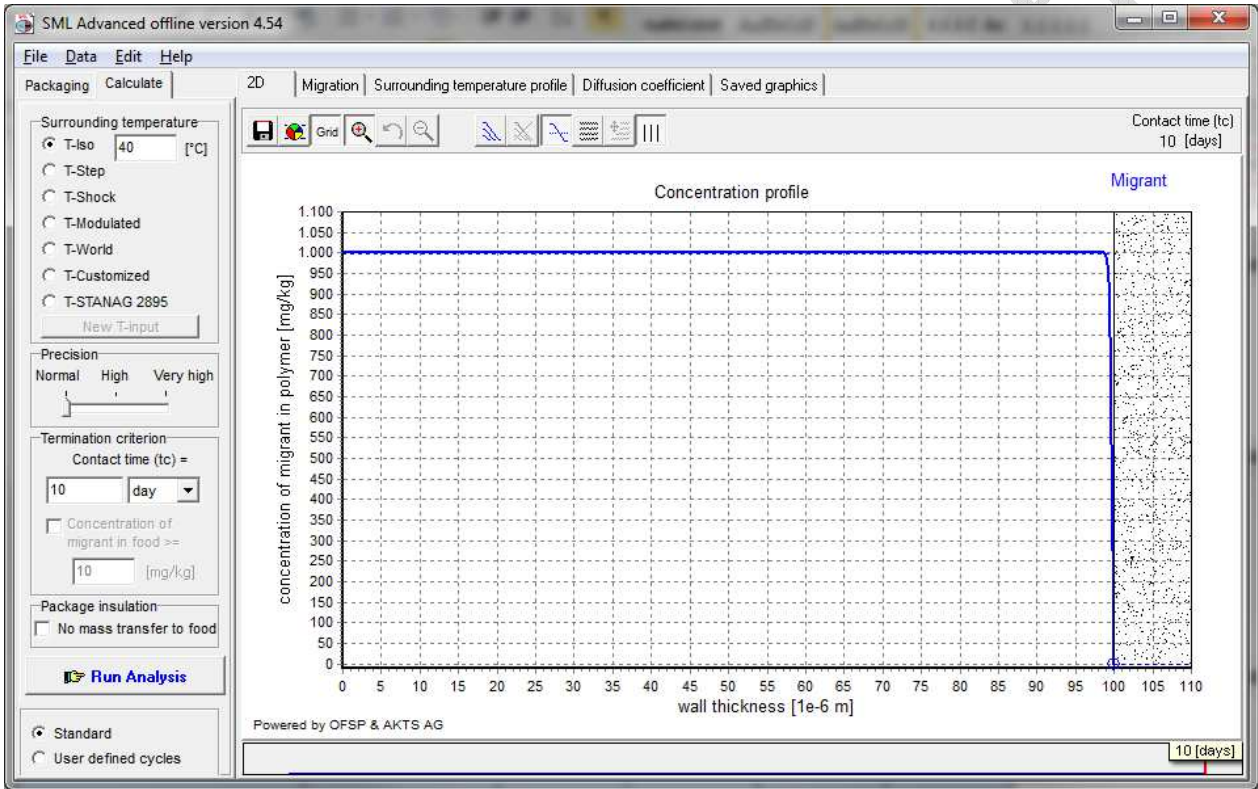
997 => 1/2 x 99% layer thickness = 0.7 µm

998 to be used for worst case calculation of specific migration under assumption of total transfer

999 => 2 x 99% layer thickness = 2.6 µm

1000 above 2.6 µm two sides to be considered for calculation of migration if full immersion testing
1001 applied

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1008 **10d @ 20°C**

1009 => 100% layer thickness = 0.6 µm

1010 no absolute barrier at thicknesses below 0.6 µm

1011 => 99% layer thickness = 0.4 µm

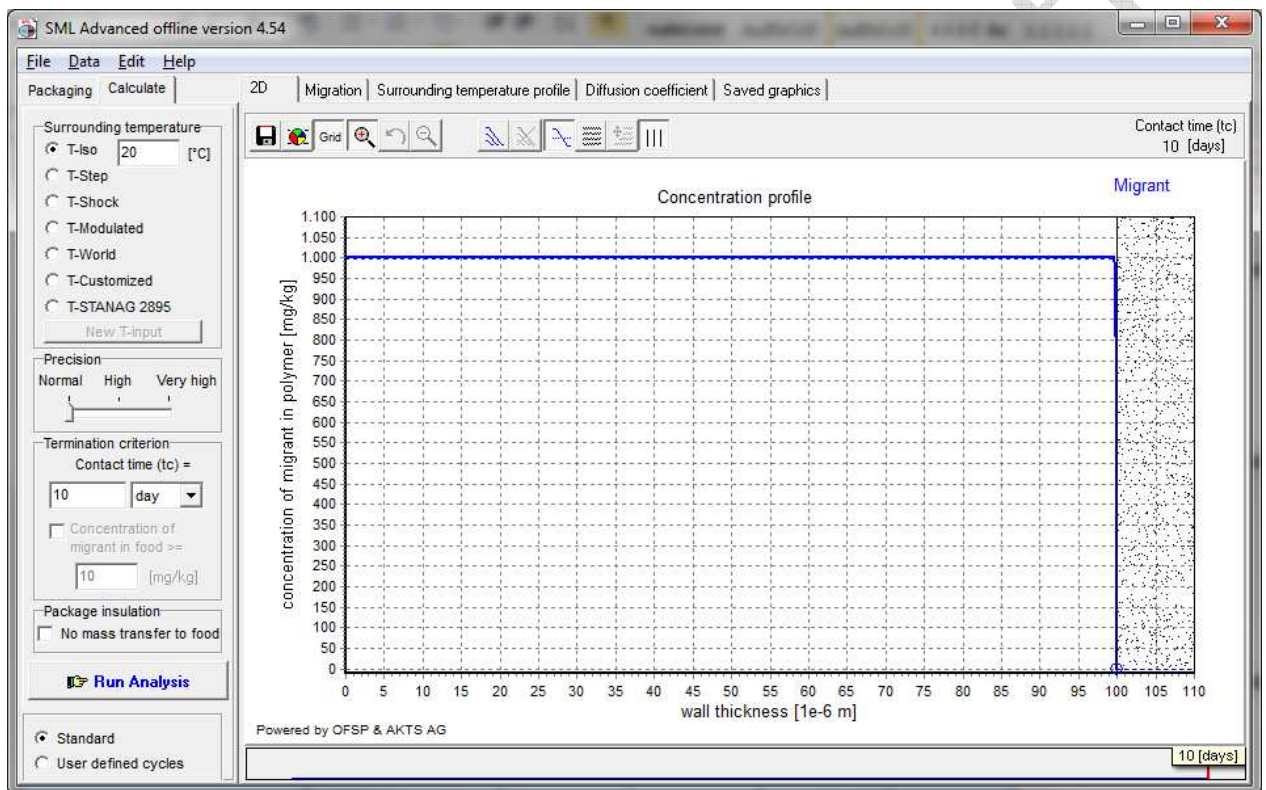
1012 => 1/2 x 99% layer thickness = 0.2 µm

1013 to be used for worst case calculation of specific migration under assumption of total transfer

1014 => 2 x 99% layer thickness = 0.8 µm

1015 above 0.8 µm two sides to be considered for calculation of migration if full immersion testing applied

1017



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1023 **2h @ 100°C**

1024 => 100% layer thickness = 3 µm

1025 no absolute barrier at thicknesses below 3 µm

1026 => 99% layer thickness = 2.4 µm

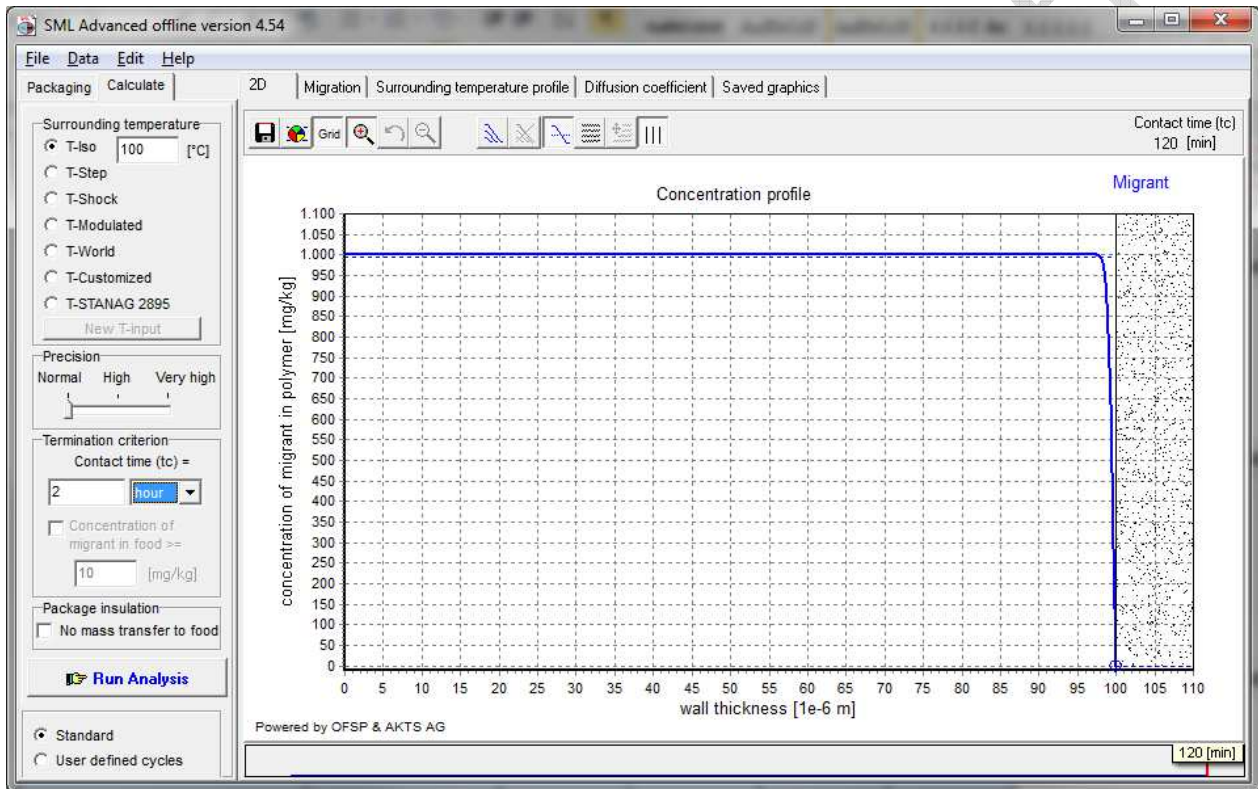
1027 => 1/2 x 99% layer thickness = 1.2 µm

1028 to be used for worst case calculation of specific migration under assumption of total transfer

1029 => 2 x 99% layer thickness = 5 µm

1030 above 5 µm two sides to be considered for calculation of migration if full immersion testing
1031 applied

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1038 **PS**

1039 **► molecular mass 100 - 250 g/mol**

1040 **10d @ 60°C**

1041 => 100% layer thickness = 127

1042 no absolute barrier at thicknesses below 127 µm

1043 => 99% layer thickness = 110 µm

1044 => 1/2 x 99% layer thickness = 55 µm

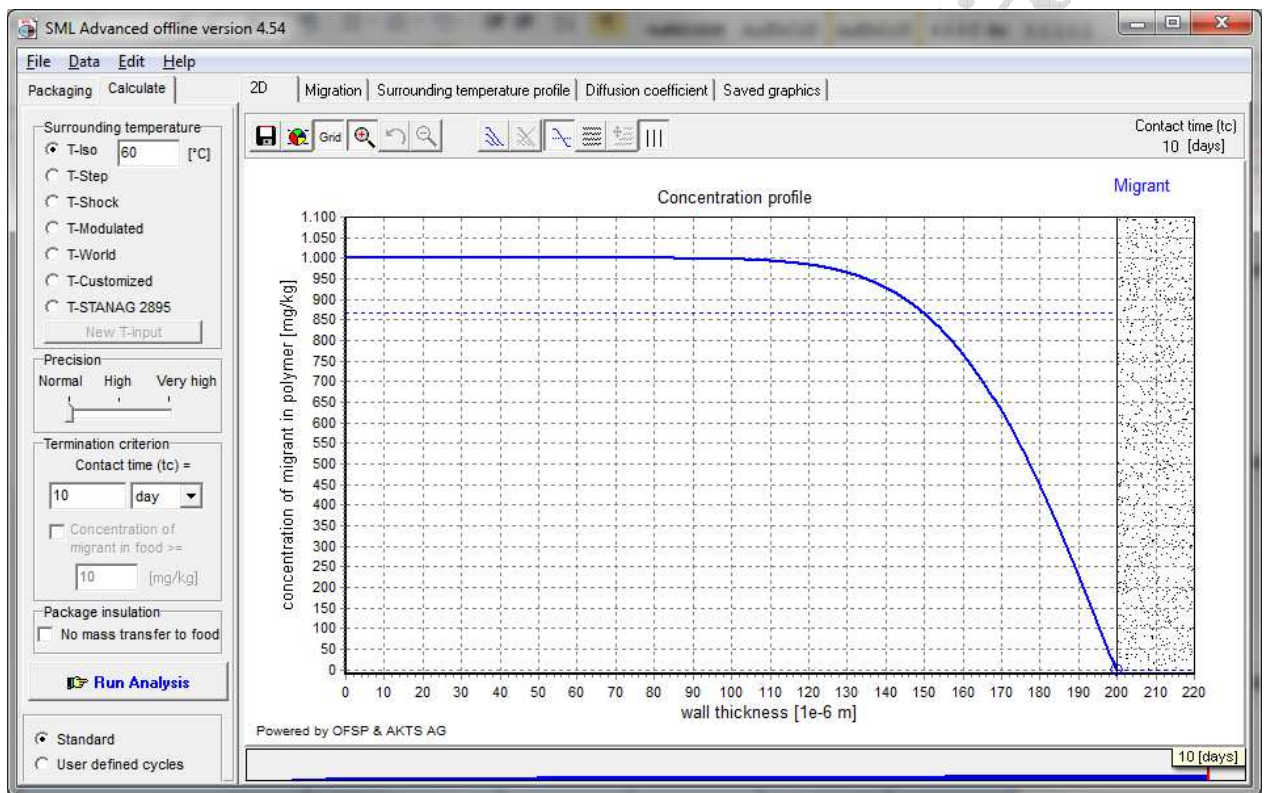
1045 to be used for worst case calculation of specific migration under assumption of total transfer

1046 => 2 x 99% layer thickness = 220 µm

1047 above 220 µm two sides to be considered for calculation of migration if full immersion testing

1048 applied

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1055 **10d @ 40°C**

1056 => 100% layer thickness = 46

1057 no absolute barrier at thicknesses below 46 µm

1058 => 99% layer thickness = 40 µm

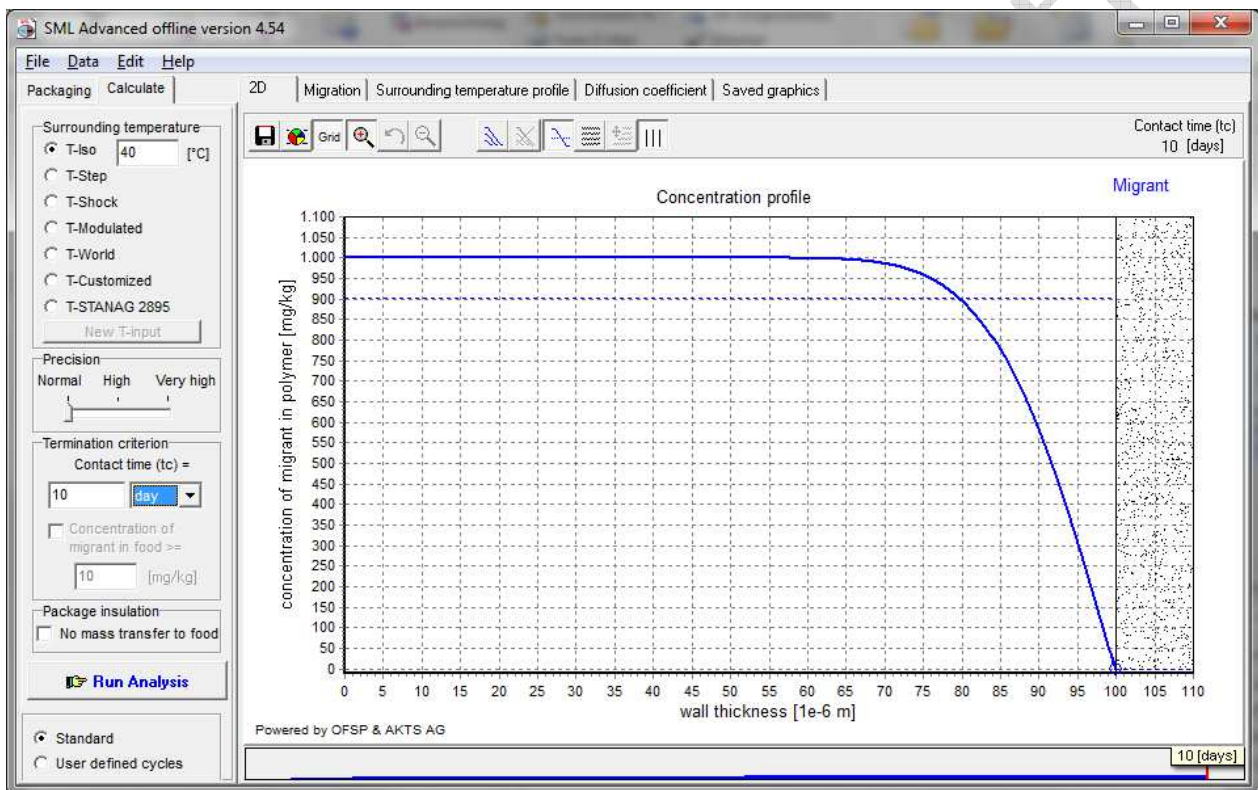
1059 => 1/2 x 99% layer thickness = 20 µm

1060 to be used for worst case calculation of specific migration under assumption of total transfer

1061 => 2 x 99% layer thickness = 80

1062 above 80 µm two sides to be considered for calculation of migration if full immersion testing
1063 applied

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1070 **10d @ 20°C**

1071 => 100% layer thickness = 17 µm

1072 no absolute barrier at thicknesses below 17 µm

1073 => 99% layer thickness = 13 µm

1074 => 1/2 x 99% layer thickness = 7 µm

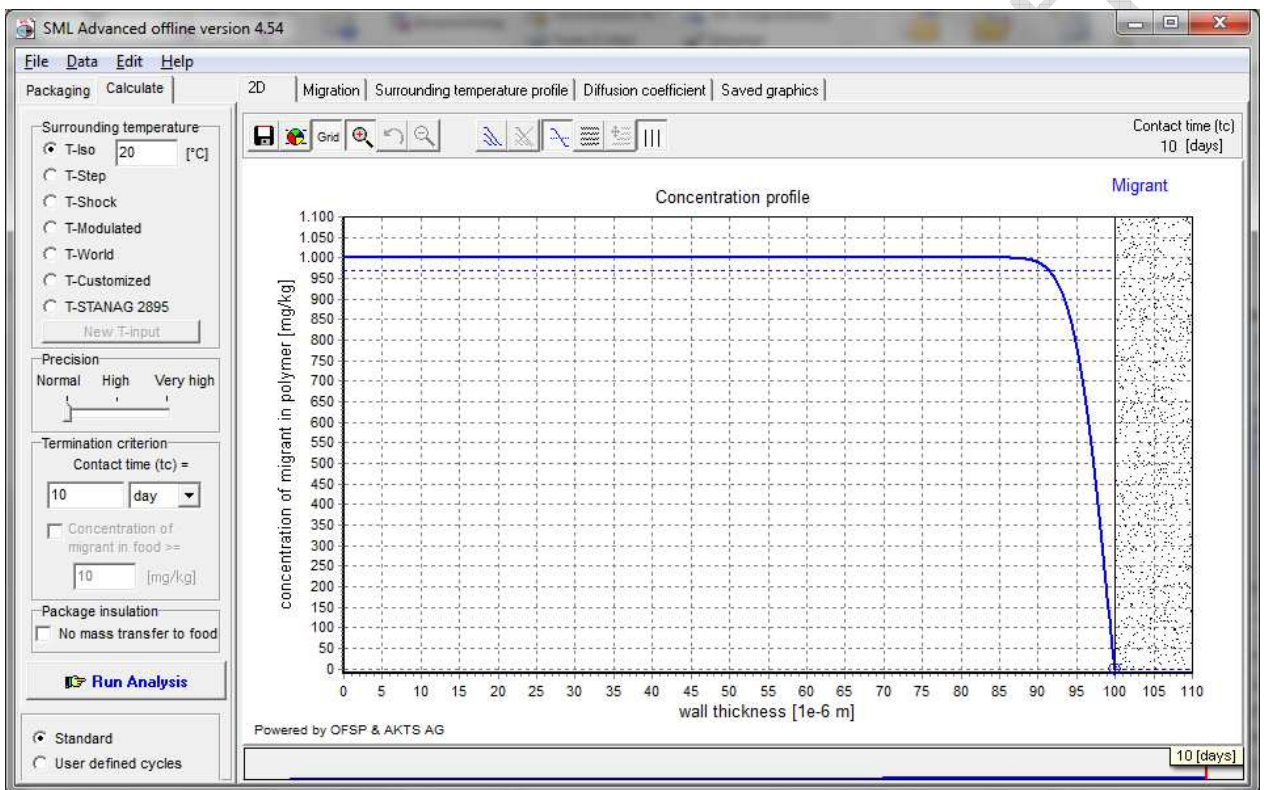
1075 to be used for worst case calculation of specific migration under assumption of total transfer

1076 => 2 x 99% layer thickness = 26 µm

1077 above 26 µm two sides to be considered for calculation of migration if full immersion testing

1078 applied

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1085 **2h @ 100°C**

1086 => 100% layer thickness = 65 µm

1087 no absolute barrier at thicknesses below 65 µm

1088 => 99% layer thickness = 54 µm

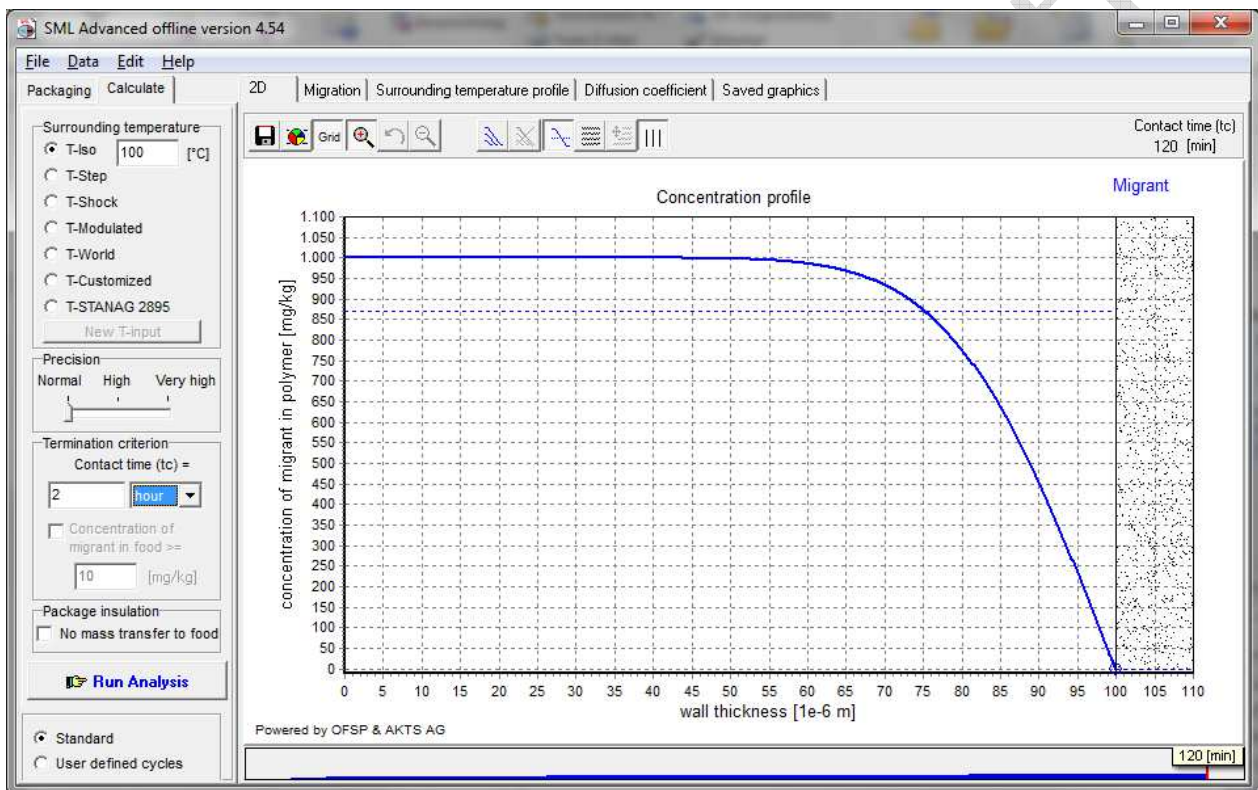
1089 => 1/2 x 99% layer thickness = 27 µm

1090 to be used for worst case calculation of specific migration under assumption of total transfer

1091 => 2 x 99% layer thickness = 108 µm

1092 above 108 µm two sides to be considered for calculation of migration if full immersion testing
1093 applied

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1100 ► molecular mass 251 - 500 g/mol

1101 10d @ 60°C

1102 => 100% layer thickness = 49 µm

1103 no absolute barrier at thicknesses below 49 µm

1104 => 99% layer thickness = 41 µm

1105 => 1/2 x 99% layer thickness = 20.5 µm

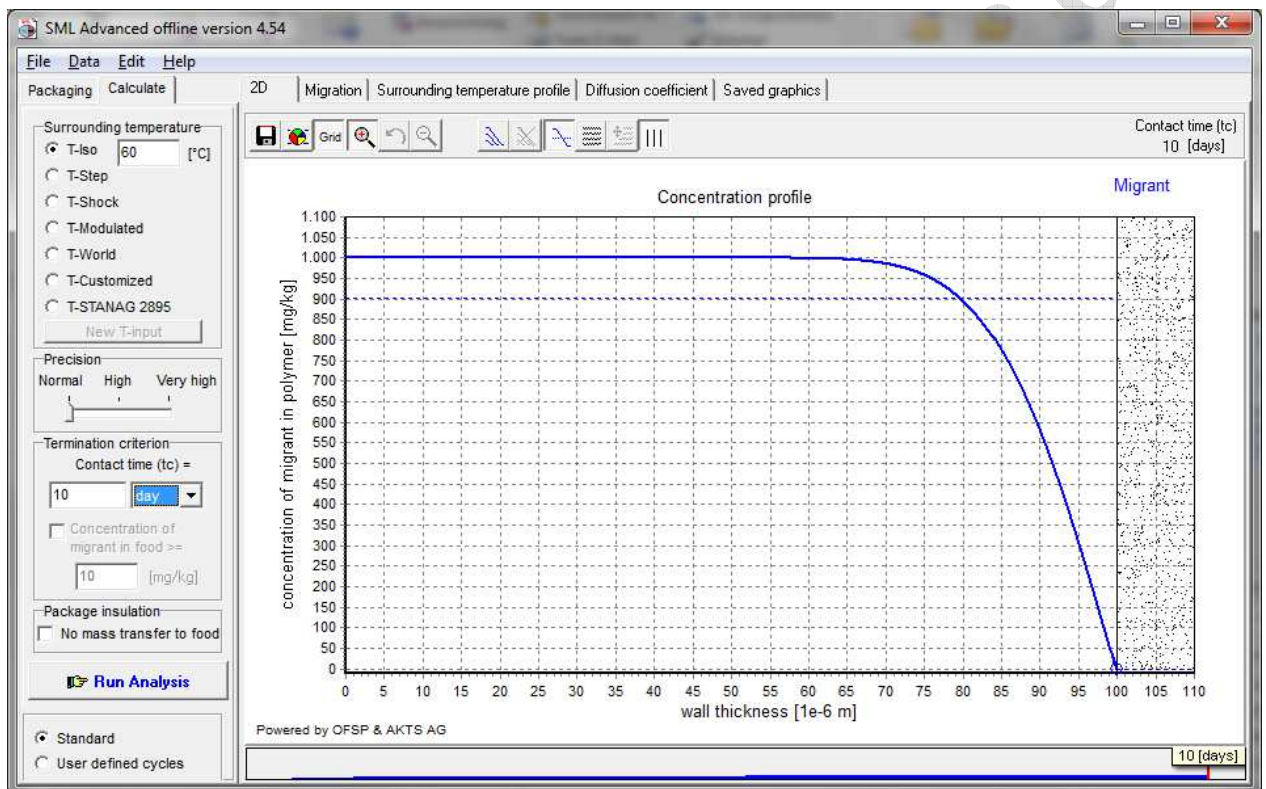
1106 to be used for worst case calculation of specific migration under assumption of total transfer

1107 => 2 x 99% layer thickness = 82 µm

1108 above 82 µm two sides to be considered for calculation of migration if full immersion testing

1109 applied

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1116 **10d @ 40°C**

1117 => 100% layer thickness = 18 µm

1118 no absolute barrier at thicknesses below 18 µm

1119 => 99% layer thickness = 15 µm

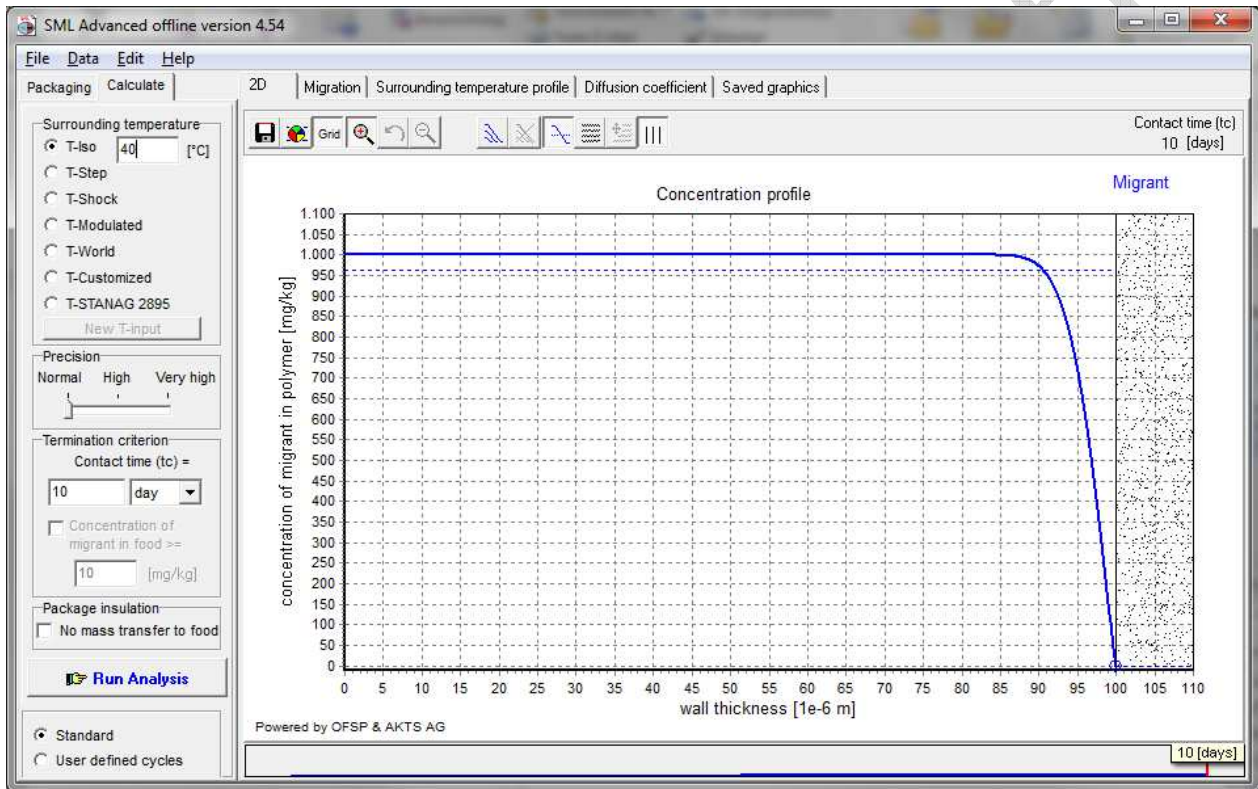
1120 => 1/2 x 99% layer thickness = 7.5 µm

1121 to be used for worst case calculation of specific migration under assumption of total transfer

1122 => 2 x 99% layer thickness = 30 µm

1123 above 30 µm two sides to be considered for calculation of migration if full immersion testing
1124 applied

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1131 **10d @ 20°C**

1132 => 100% layer thickness = 6.2 μm

1133 no absolute barrier at thicknesses below 6.2 μm

1134 => 99% layer thickness = 5 μm

1135 => 1/2 x 99% layer thickness = 2.5 μm

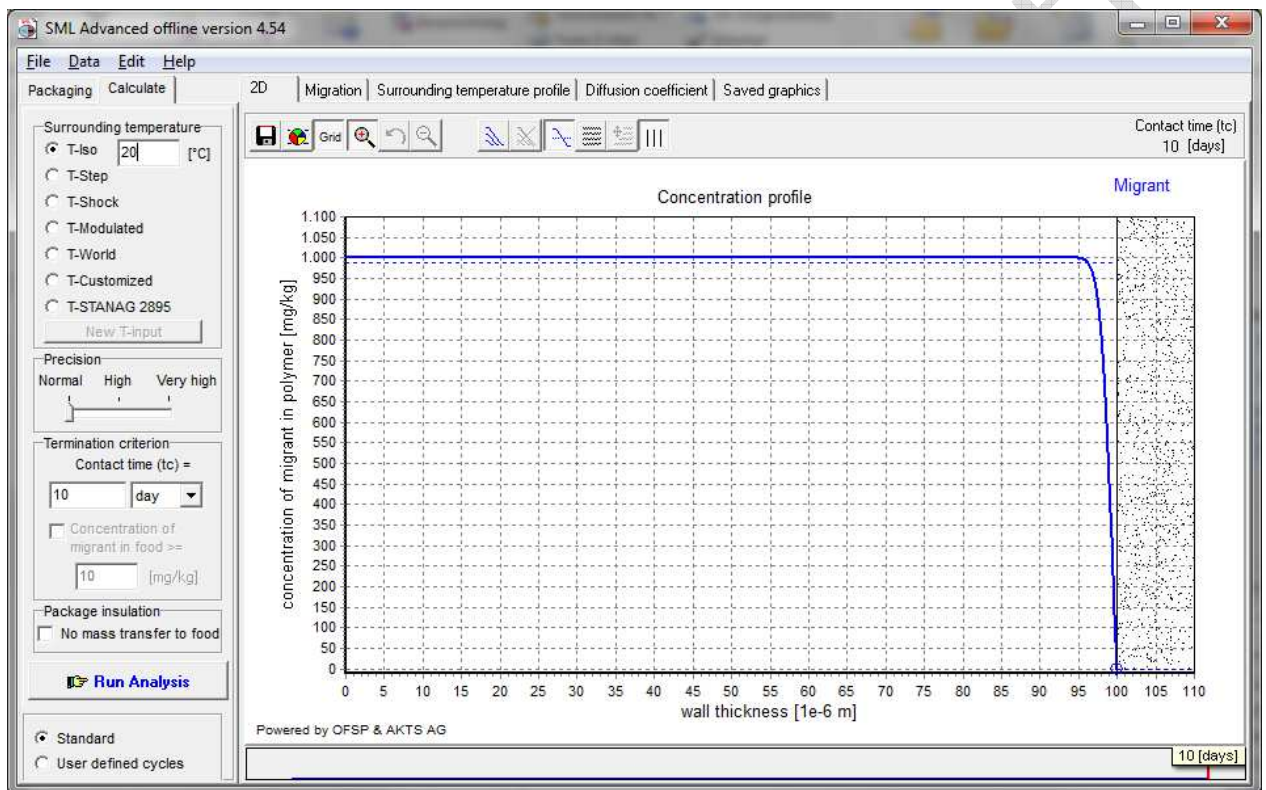
1136 to be used for worst case calculation of specific migration under assumption of total transfer

1137 => 2 x 99% layer thickness = 10 μm

1138 above 10 μm two sides to be considered for calculation of migration if full immersion testing

1139 applied

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1146 **2h @ 100°C**

1147 => 100% layer thickness = 26 µm

1148 no absolute barrier at thicknesses below 26 µm

1149 => 99% layer thickness = 20 µm

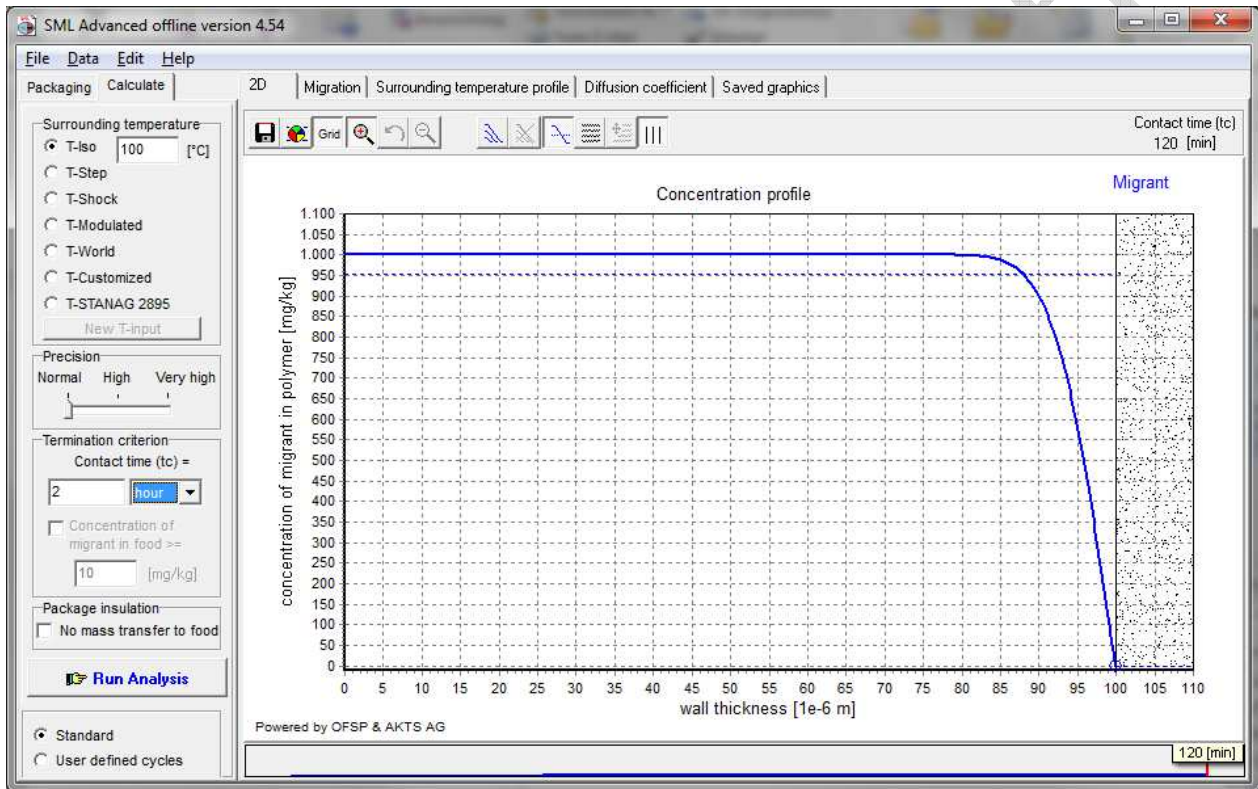
1150 => 1/2 x 99% layer thickness = 10 µm

1151 to be used for worst case calculation of specific migration under assumption of total transfer

1152 => 2 x 99% layer thickness = 40 µm

1153 above 40 µm two sides to be considered for calculation of migration if full immersion testing
1154 applied

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1161 ► molecular mass 501 - 750 g/mol

1162 10d @ 60°C

1163 => 100% layer thickness = 15.2 µm

1164 no absolute barrier at thicknesses below 15.2 µm

1165 => 99% layer thickness = 12.4 µm

1166 => 1/2 x 99% layer thickness = 6.2 µm

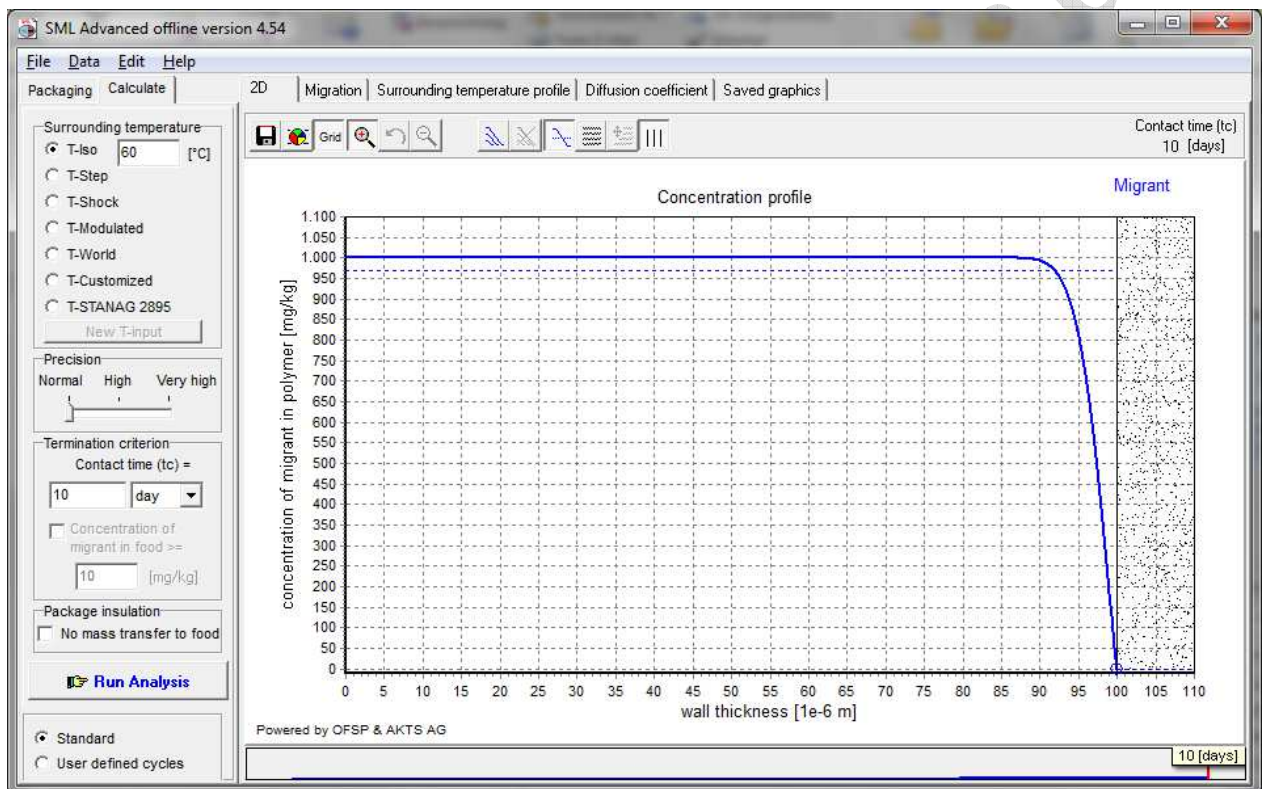
1167 to be used for worst case calculation of specific migration under assumption of total transfer

1168 => 2 x 99% layer thickness = 24.8 µm

1169 above 24.8 µm two sides to be considered for calculation of migration if full immersion testing

1170 applied

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1177 **10d @ 40°C**

1178 => 100% layer thickness = 6 µm

1179 no absolute barrier at thicknesses below 6 µm

1180 => 99% layer thickness = 5 µm

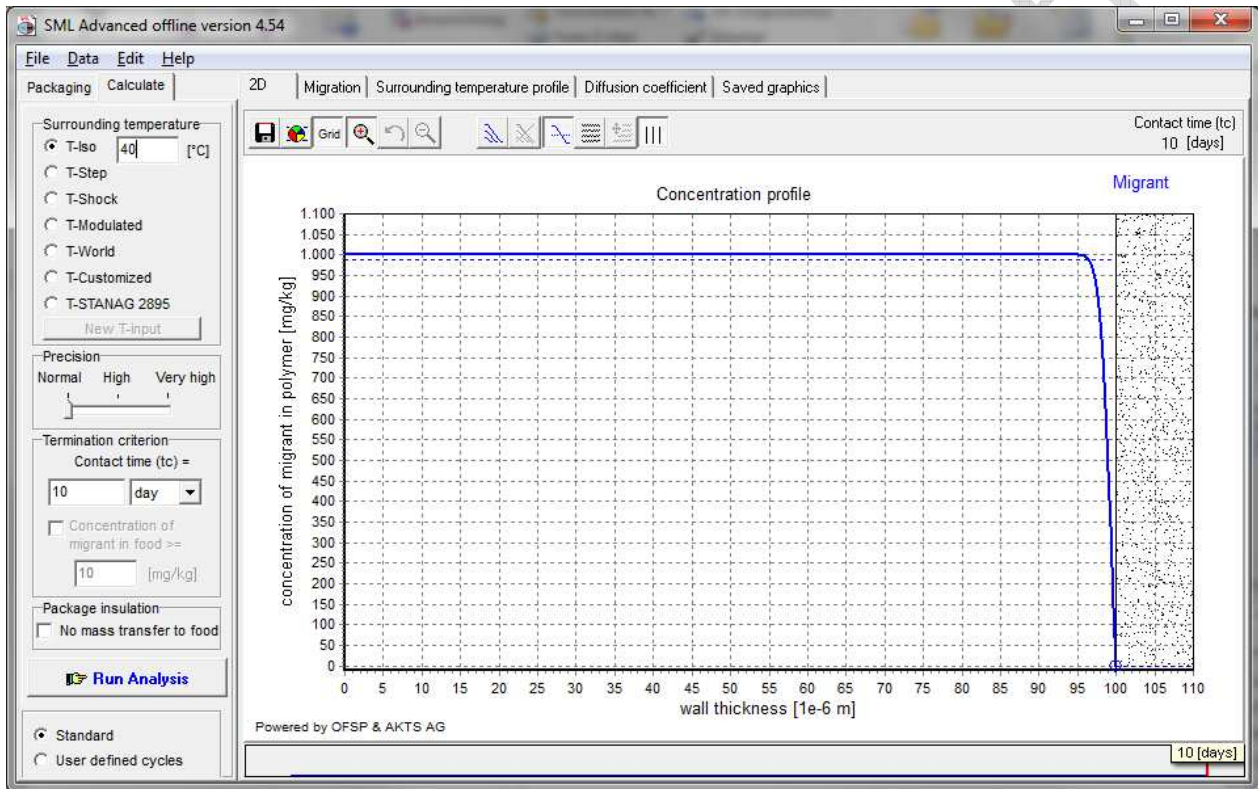
1181 => 1/2 x 99% layer thickness = 2.5 µm

1182 to be used for worst case calculation of specific migration under assumption of total transfer

1183 => 2 x 99% layer thickness = 10 µm

1184 above 10 µm two sides to be considered for calculation of migration if full immersion testing applied

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1192 **10d @ 20°C**

1193 => 100% layer thickness = 2.2 µm

1194 no absolute barrier at thicknesses below 2.2 µm

1195 => 99% layer thickness = 1.6 µm

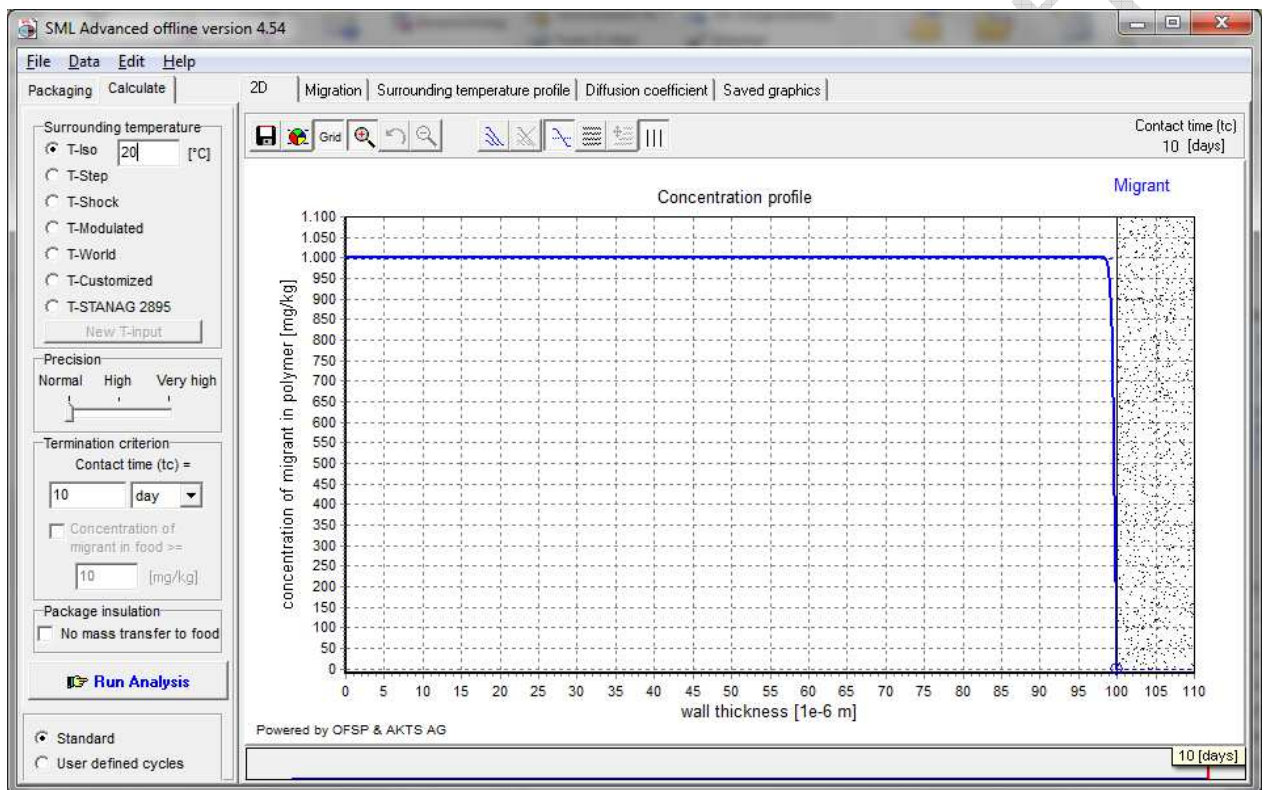
1196 => 1/2 x 99% layer thickness = 0.8 µm

1197 to be used for worst case calculation of specific migration under assumption of total transfer

1198 => 2 x 99% layer thickness = 3.2 µm

1199 above 3.2 µm two sides to be considered for calculation of migration if full immersion testing applied

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1207 **2h @ 100°C**

1208 => 100% layer thickness = 8 µm

1209 no absolute barrier at thicknesses below 8 µm

1210 => 99% layer thickness = 6.2 µm

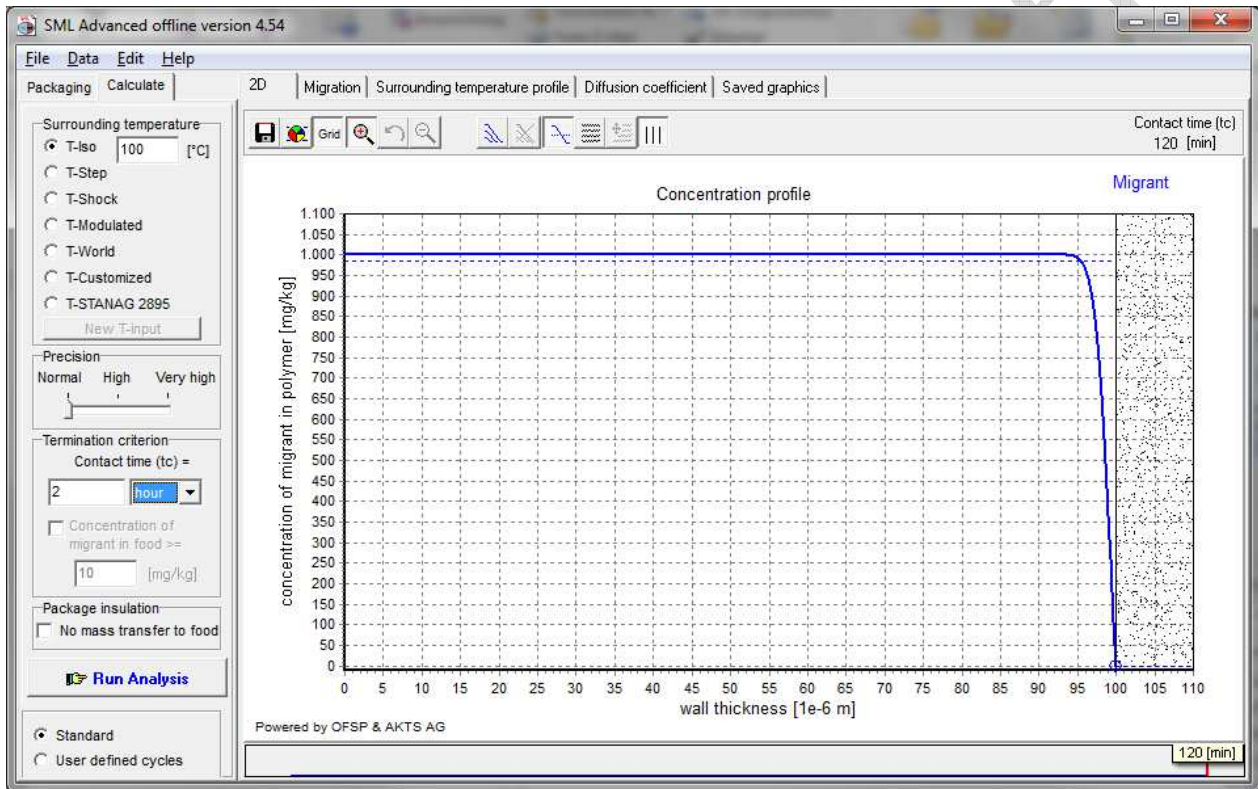
1211 => 1/2 x 99% layer thickness = 3.1 µm

1212 to be used for worst case calculation of specific migration under assumption of total transfer

1213 => 2 x 99% layer thickness = 12.4 µm

1214 above 12.4 µm two sides to be considered for calculation of migration if full immersion testing
1215 applied

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1222 ► molecular mass 751 - 1000 g/mol

1223 10d @ 60°C

1224 => 100% layer thickness = 6 µm

1225 no absolute barrier at thicknesses below 6 µm

1226 => 99% layer thickness = 4.8 µm

1227 => 1/2 x 99% layer thickness = 2.4 µm

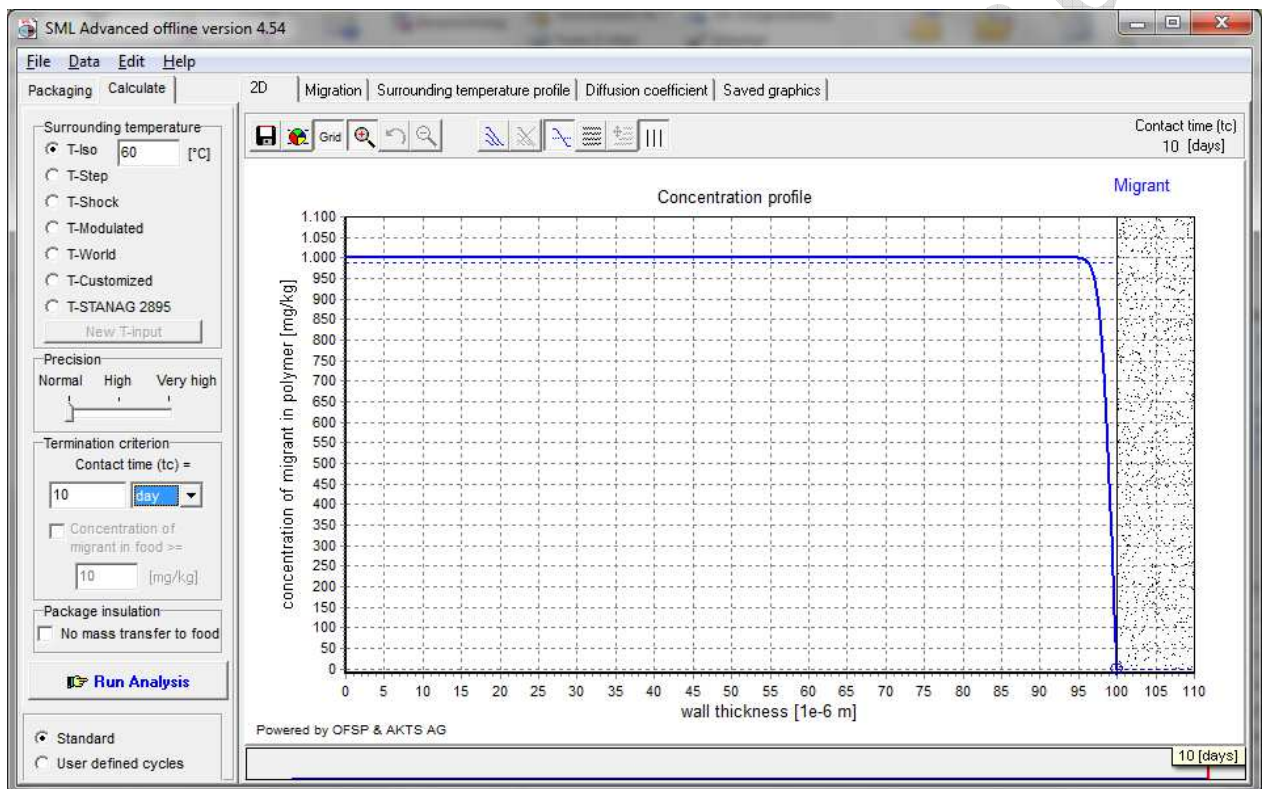
1228 to be used for worst case calculation of specific migration under assumption of total transfer

1229 => 2 x 99% layer thickness = 9.2 µm

1230 above 9.2 µm two sides to be considered for calculation of migration if full immersion testing

1231 applied

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1238 **10d @ 40°C**

1239 => 100% layer thickness = 2.8 µm

1240 no absolute barrier at thicknesses below 2.8 µm

1241 => 99% layer thickness = 2 µm

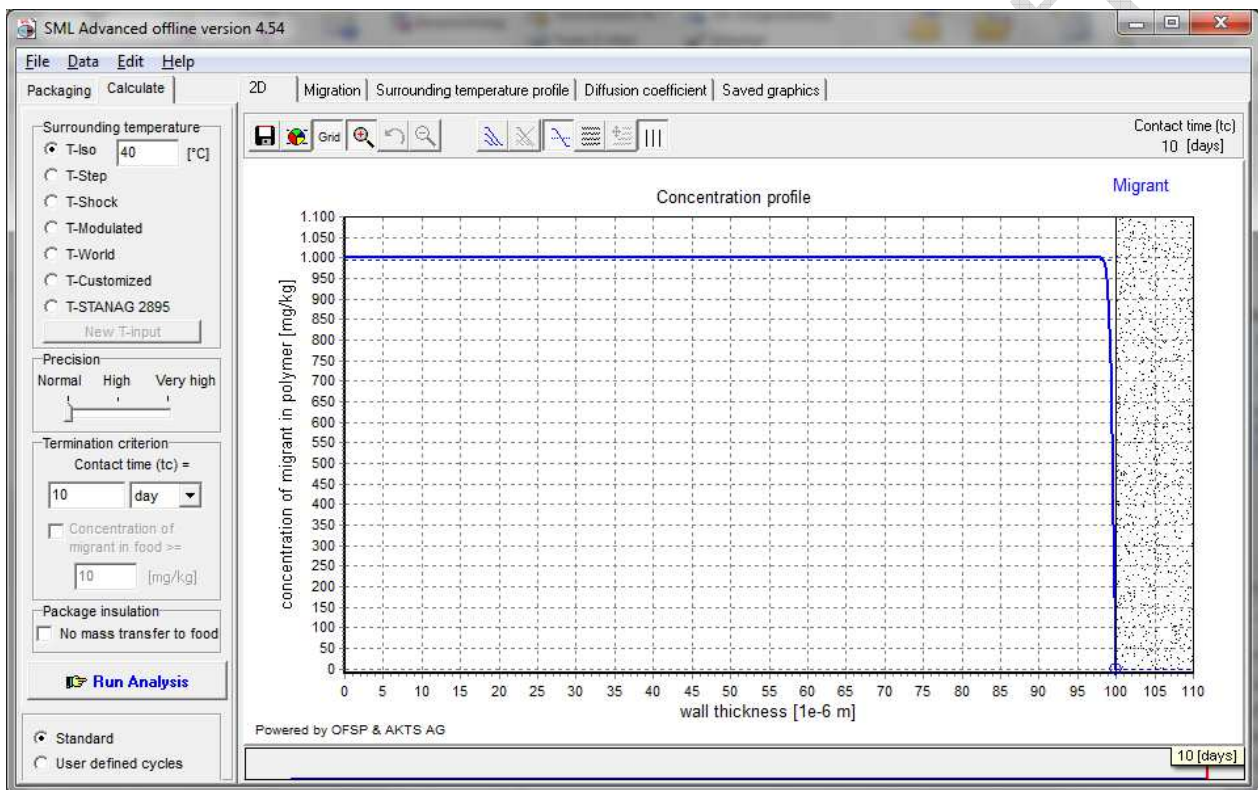
1242 => 1/2 x 99% layer thickness = 1 µm

1243 to be used for worst case calculation of specific migration under assumption of total transfer

1244 => 2 x 99% layer thickness = 4 µm

1245 above 4 µm two sides to be considered for calculation of migration if full immersion testing
1246 applied

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1253 **10d @ 20°C**

1254 => 100% layer thickness = 0.8 µm

1255 no absolute barrier at thicknesses below 0.8 µm

1256 => 99% layer thickness = 0.6 µm

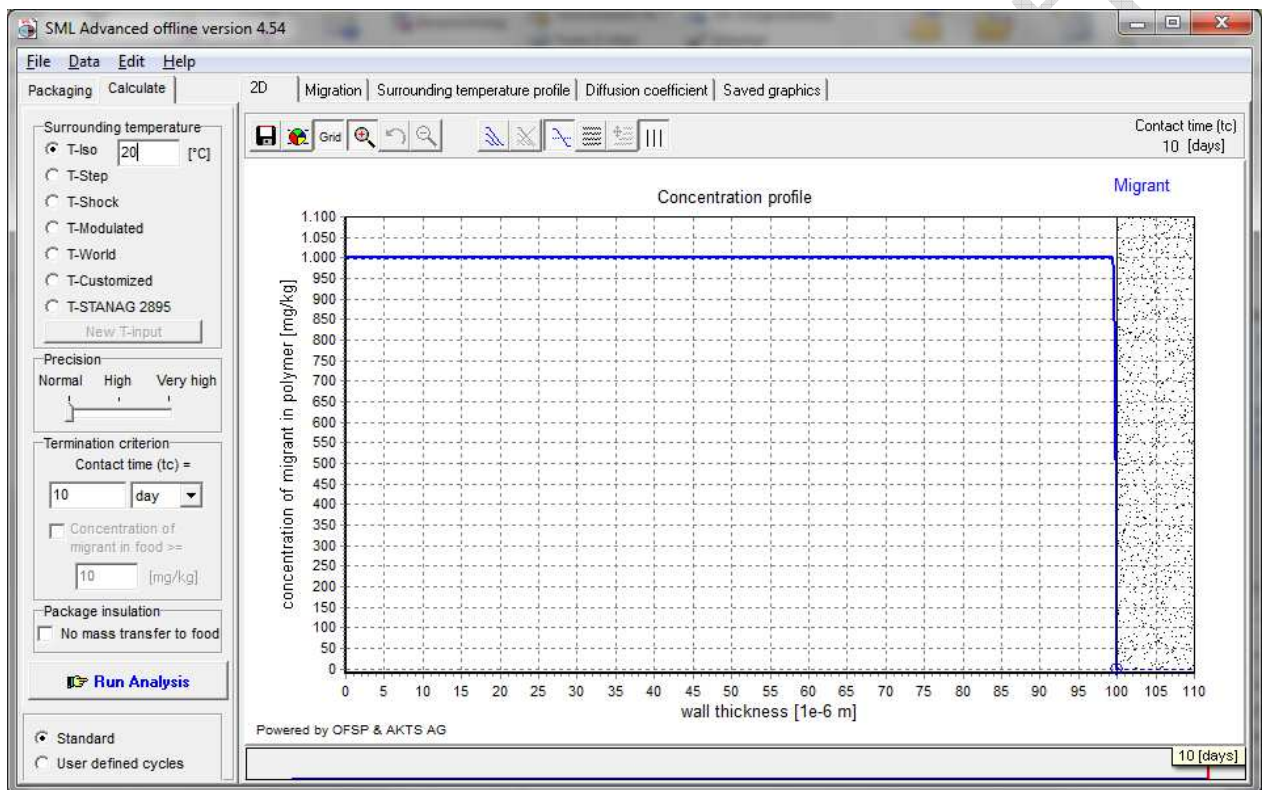
1257 => 1/2 x 99% layer thickness = 0.3 µm

1258 to be used for worst case calculation of specific migration under assumption of total transfer

1259 => 2 x 99% layer thickness = 1.2 µm

1260 above 1.2 µm two sides to be considered for calculation of migration if full immersion testing applied

1262



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1268 **2h @ 100°C**

1269 => 100% layer thickness = 3.3 µm

1270 no absolute barrier at thicknesses below 3.3 µm

1271 => 99% layer thickness = 2.6 µm

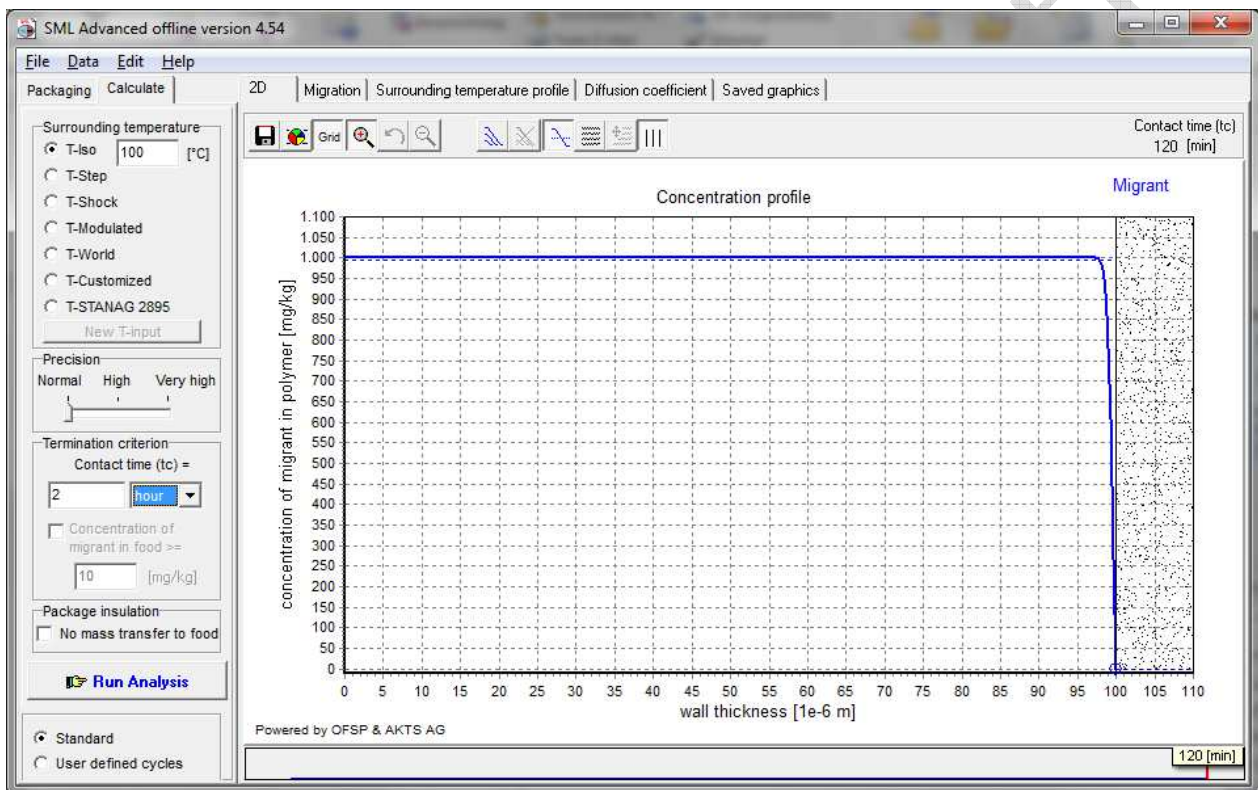
1272 => 1/2 x 99% layer thickness = 1.3 µm

1273 to be used for worst case calculation of specific migration under assumption of total transfer

1274 => 2 x 99% layer thickness = 5.2 µm

1275 above 5.2 µm two sides to be considered for calculation of migration if full immersion testing
1276 applied

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1283 **SBS**

1284 **► molecular mass 100 - 250 g/mol**

1285 **10d @ 60°C**

1286 => 100% layer thickness = full length

1287 no absolute barrier at thicknesses below 10000 µm

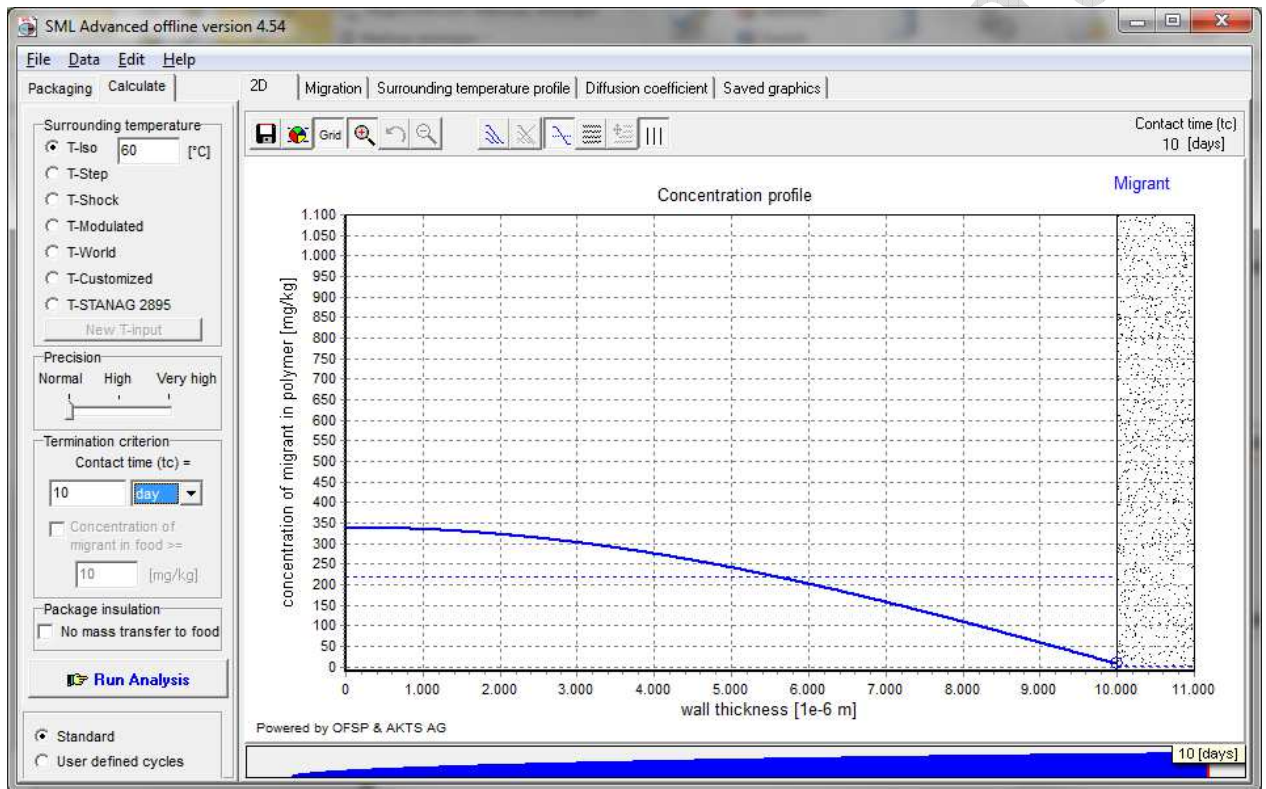
1288 => 99% layer thickness = full length

1289 => full length to be used for worst case calculation of specific migration under
1290 assumption of total transfer

1291 => 2 x 99% layer thickness = none

1292 only one side to be considered for calculation of migration if full immersion testing is applied

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1299 **10d @ 40°C**

1300 => 100% layer thickness = full length

1301 no absolute barrier at thicknesses below 10000 µm

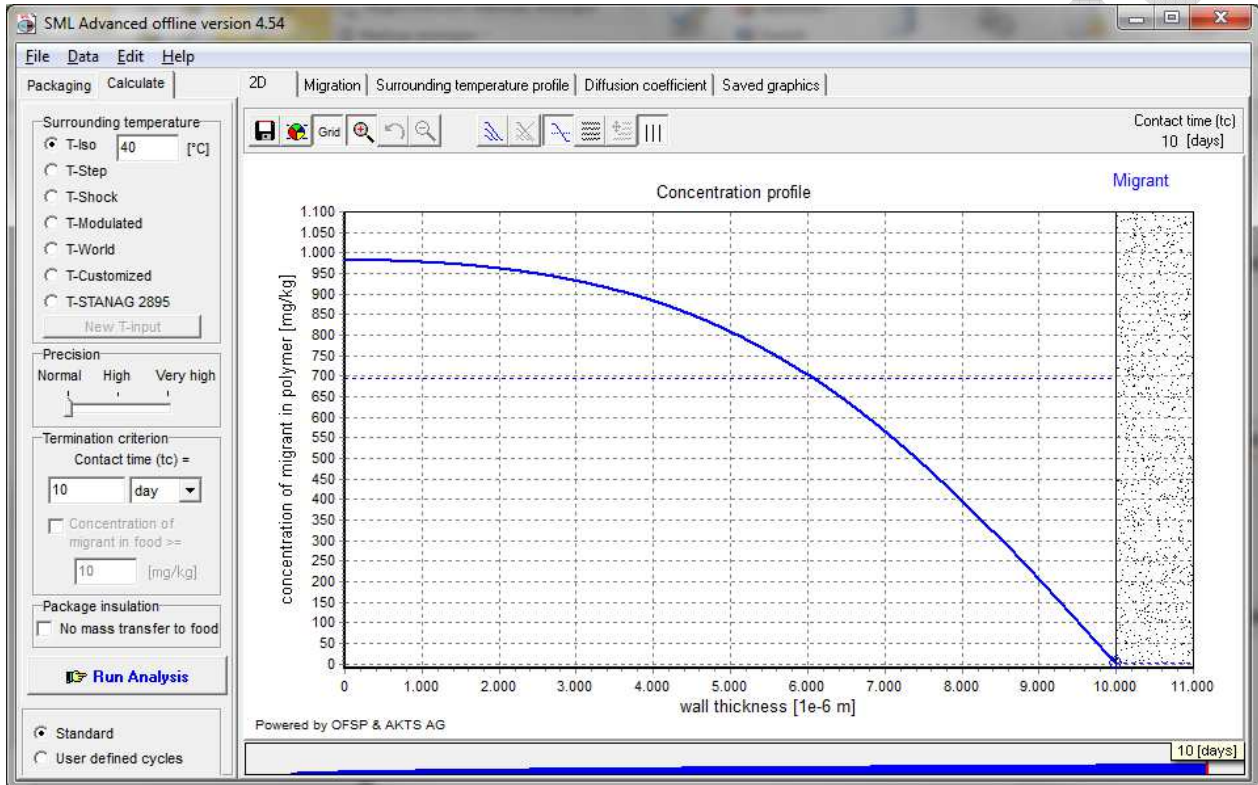
1302 => 99% layer thickness = full length

1303 => full length to be used for worst case calculation of specific migration under
1304 assumption of total transfer

1305 => 2 x 99% layer thickness = none

1306 only one side to be considered for calculation of migration if full immersion testing is applied

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1313 **10d @ 20°C**

1314 => 100% layer thickness = 5000 µm

1315 no absolute barrier at thicknesses below 5000 µm

1316 => 99% layer thickness = 4200 µm

1317 => 1/2 x 99% layer thickness = 2100 µm

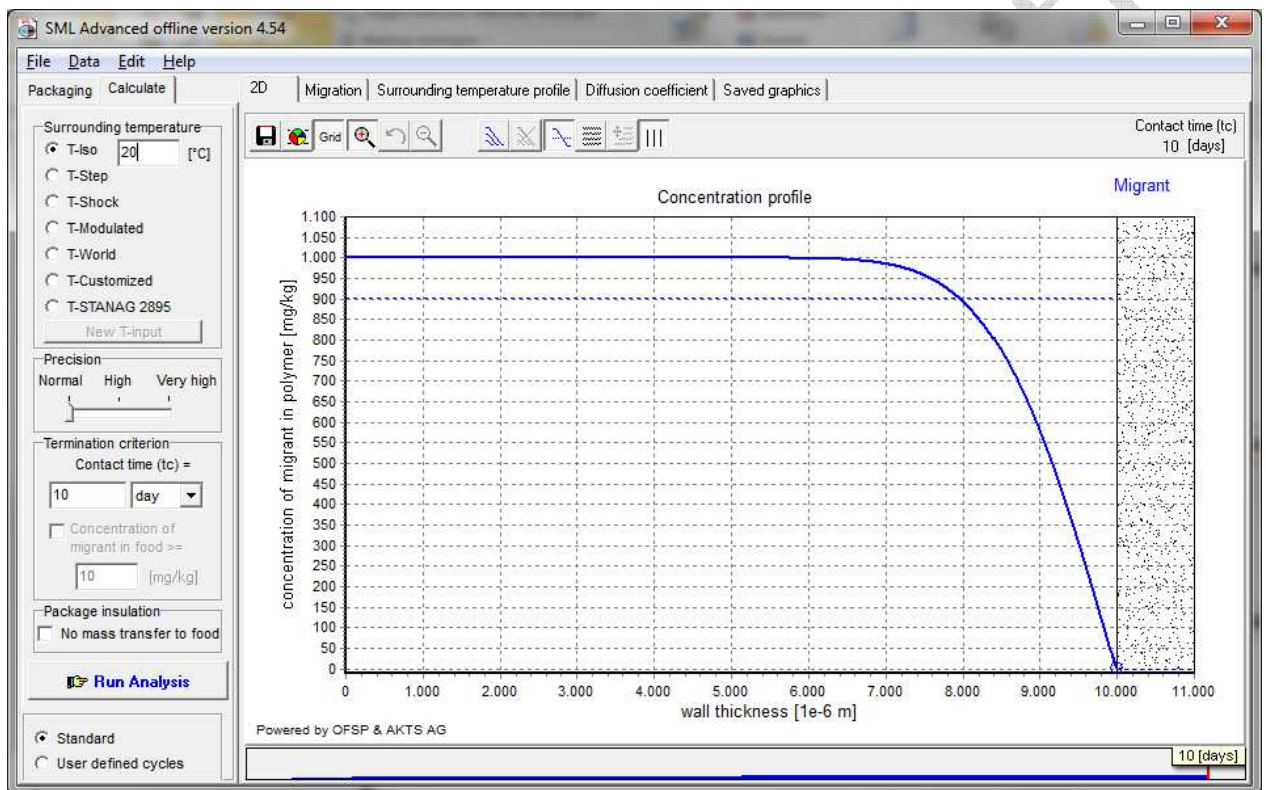
1318 to be used for worst case calculation of specific migration under assumption of total transfer

1319 => 2 x 99% layer thickness = 8400 µm

1320 above 8400 µm two sides to be considered for calculation of migration if full immersion

1321 testing applied

1322



1323

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1328 **2h @ 100°C**

1329 => 100% layer thickness = full length

1330 no absolute barrier at thicknesses below 10000 µm

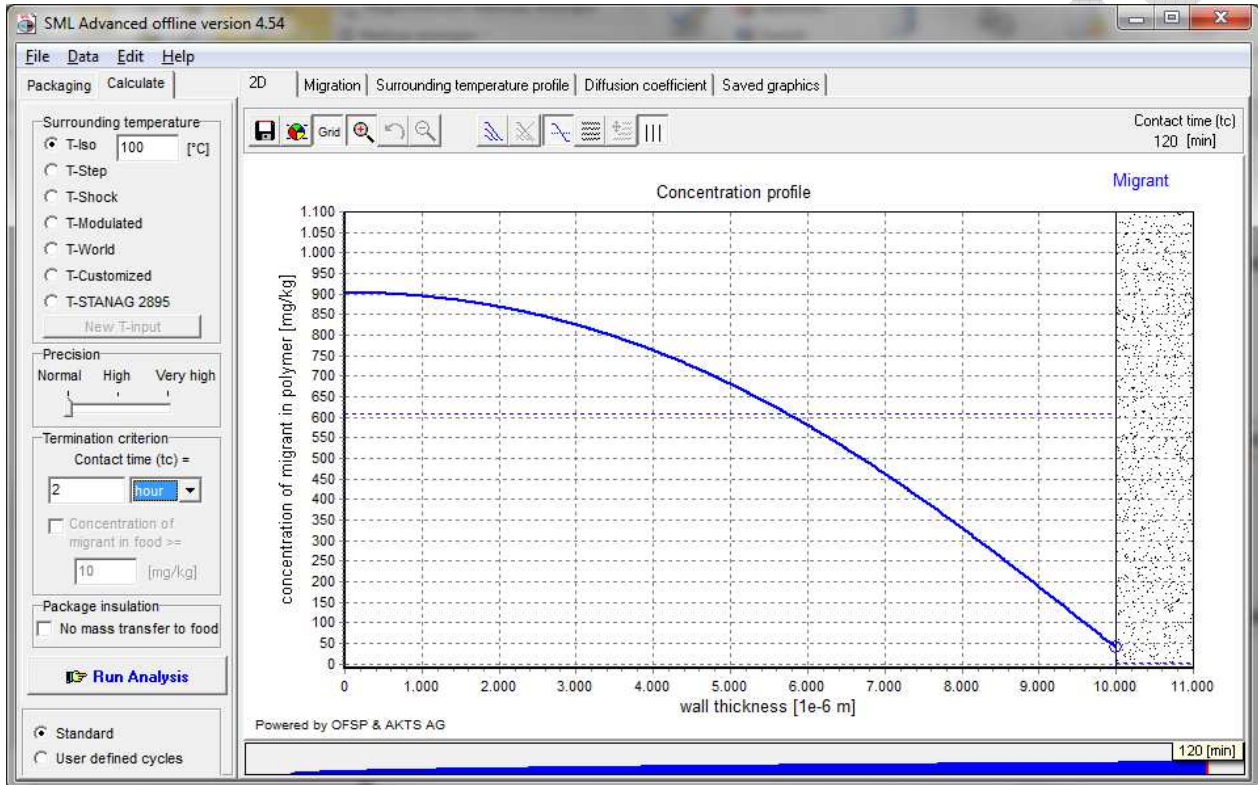
1331 => 99% layer thickness = full length

1332 => full length to be used for worst case calculation of specific migration under
1333 assumption of total transfer

1334 => 2 x 99% layer thickness = none

1335 only one side to be considered for calculation of migration if full immersion testing is applied

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1342 ► molecular mass 251 - 500 g/mol

1343 10d @ 60°C

1344 => 100% layer thickness = full length

1345 no absolute barrier at thicknesses below 10000 µm

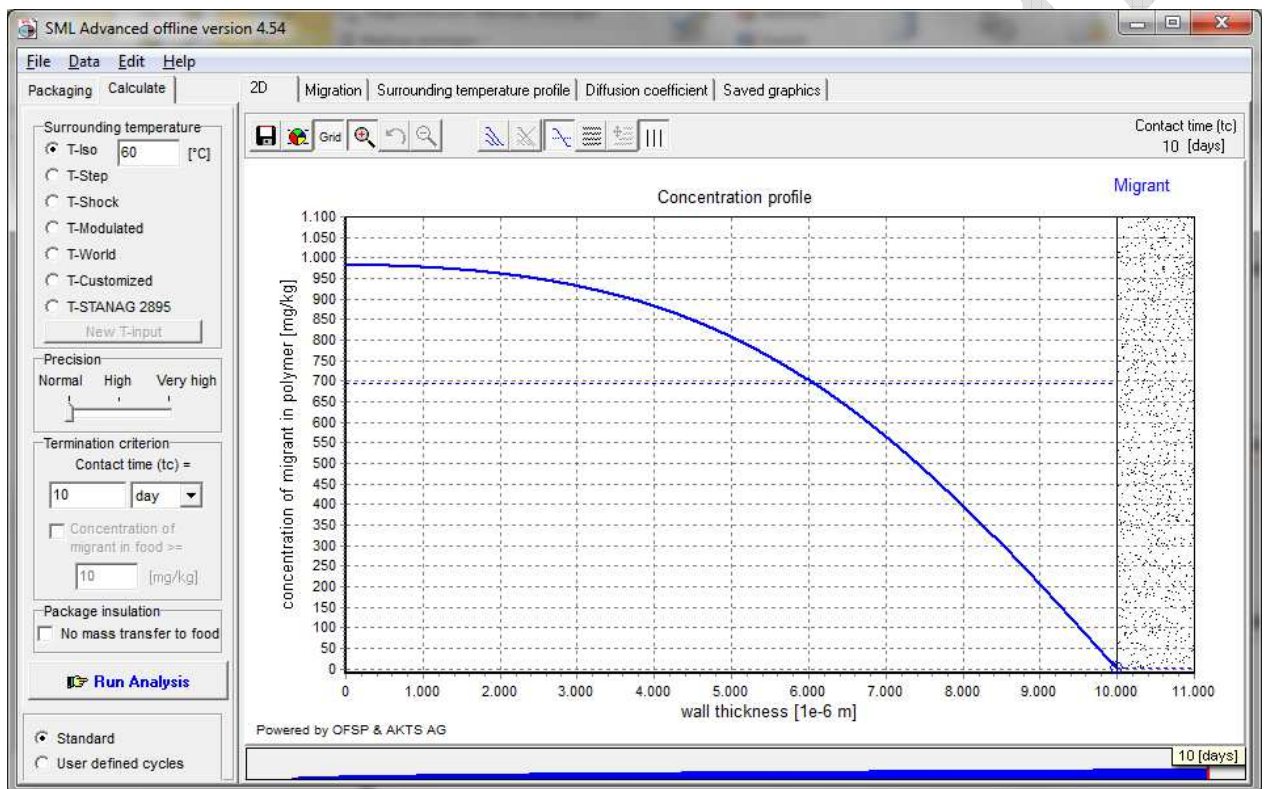
1346 => 99% layer thickness = full length

1347 => full length to be used for worst case calculation of specific migration under
1348 assumption of total transfer

1349 => 2 x 99% layer thickness = none

1350 only one side to be considered for calculation of migration if full immersion testing is applied

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1357 **10d @ 40°C**

1358 => 100% layer thickness = 5800 µm

1359 no absolute barrier at thicknesses below 5800 µm

1360 => 99% layer thickness = 4600 µm

1361 => 1/2 x 99% layer thickness = 2300 µm

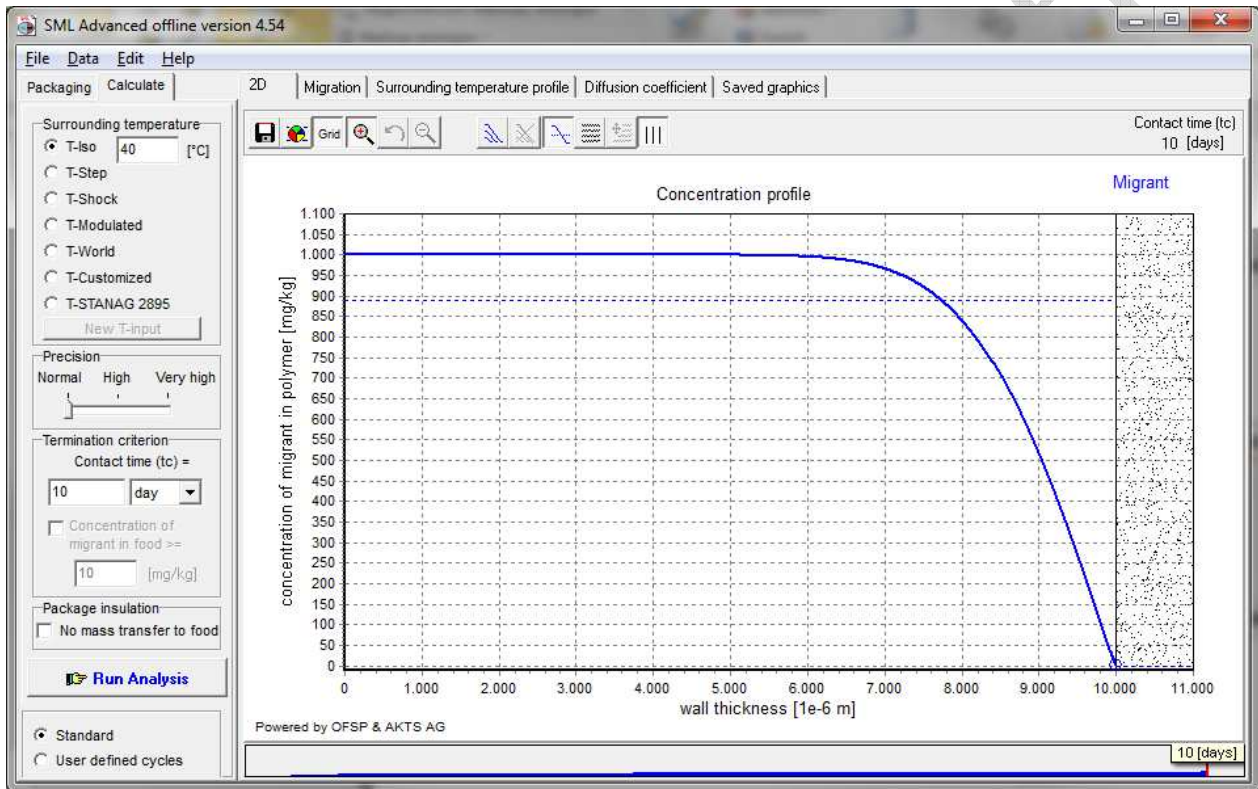
1362 to be used for worst case calculation of specific migration under assumption of total transfer

1363 => 2 x 99% layer thickness = 9200 µm

1364 above 9200 µm two sides to be considered for calculation of migration if full immersion

1365 testing applied

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1372 **10d @ 20°C**

1373 => 100% layer thickness = 1900 µm

1374 no absolute barrier at thicknesses below 1900 µm

1375 => 99% layer thickness = 1500 µm

1376 => 1/2 x 99% layer thickness = 750 µm

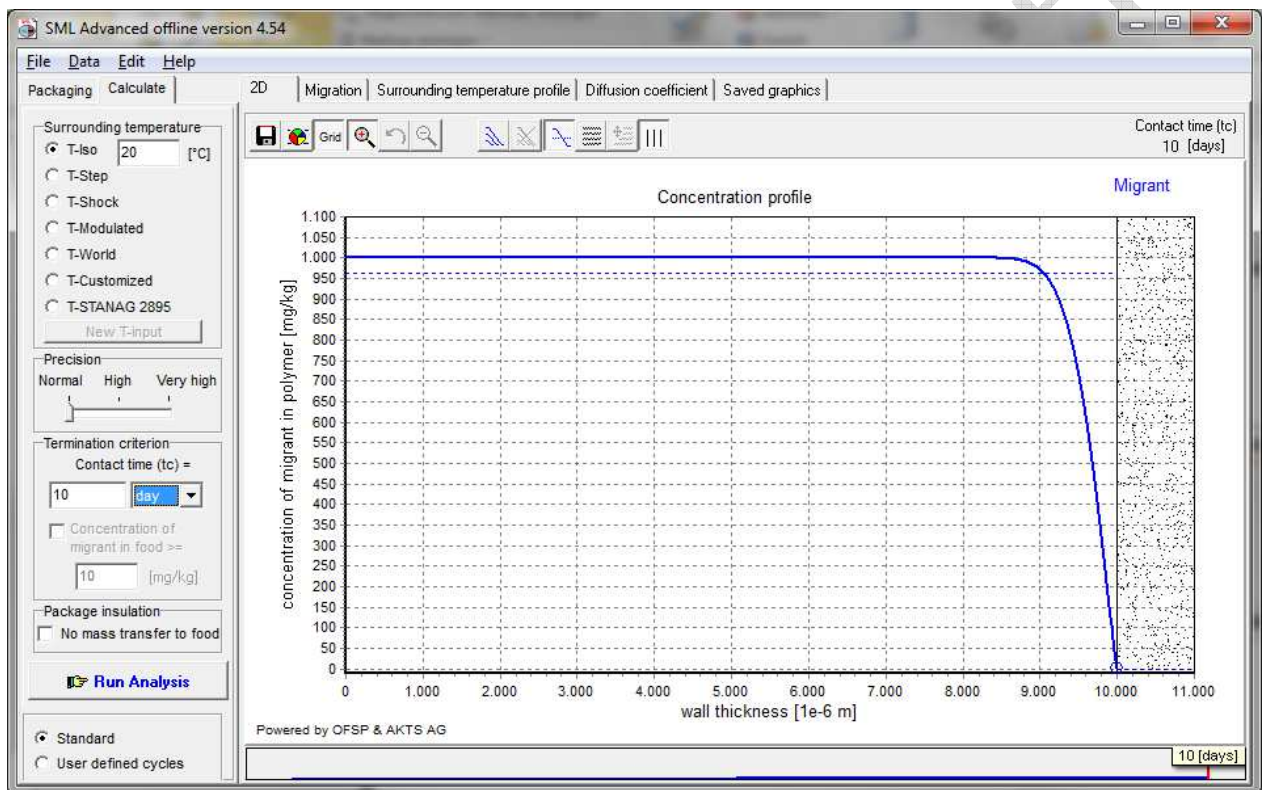
1377 to be used for worst case calculation of specific migration under assumption of total transfer

1378 => 2 x 99% layer thickness = 3000 µm

1379 above 3000 µm two sides to be considered for calculation of migration if full immersion

1380 testing applied

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1387 **2h @ 100°C**

1388 => 100% layer thickness = 7600 μm

1389 no absolute barrier at thicknesses below 7600 μm

1390 => 99% layer thickness = 6200 μm

1391 => 1/2 x 99% layer thickness = 3100 μm

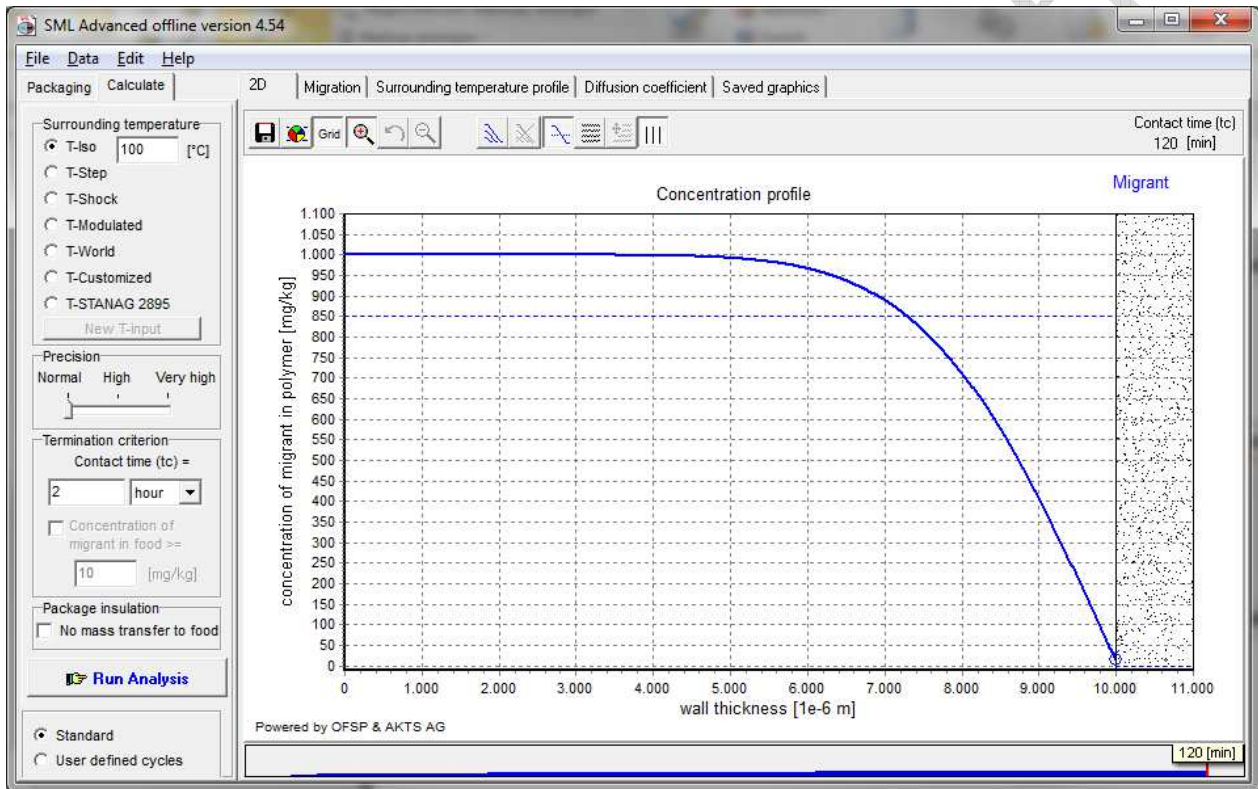
1392 to be used for worst case calculation of specific migration under assumption of total transfer

1393 => 2 x 99% layer thickness = 12400 μm

1394 above 12400 μm two sides to be considered for calculation of migration if full immersion

1395 testing applied

1396



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1402 ► molecular mass 501 - 750 g/mol

1403 10d @ 60°C

1404 => 100% layer thickness = 4600 µm

1405 no absolute barrier at thicknesses below 4600 µm

1406 => 99% layer thickness = 3800 µm

1407 => 1/2 x 99% layer thickness = 1900 µm

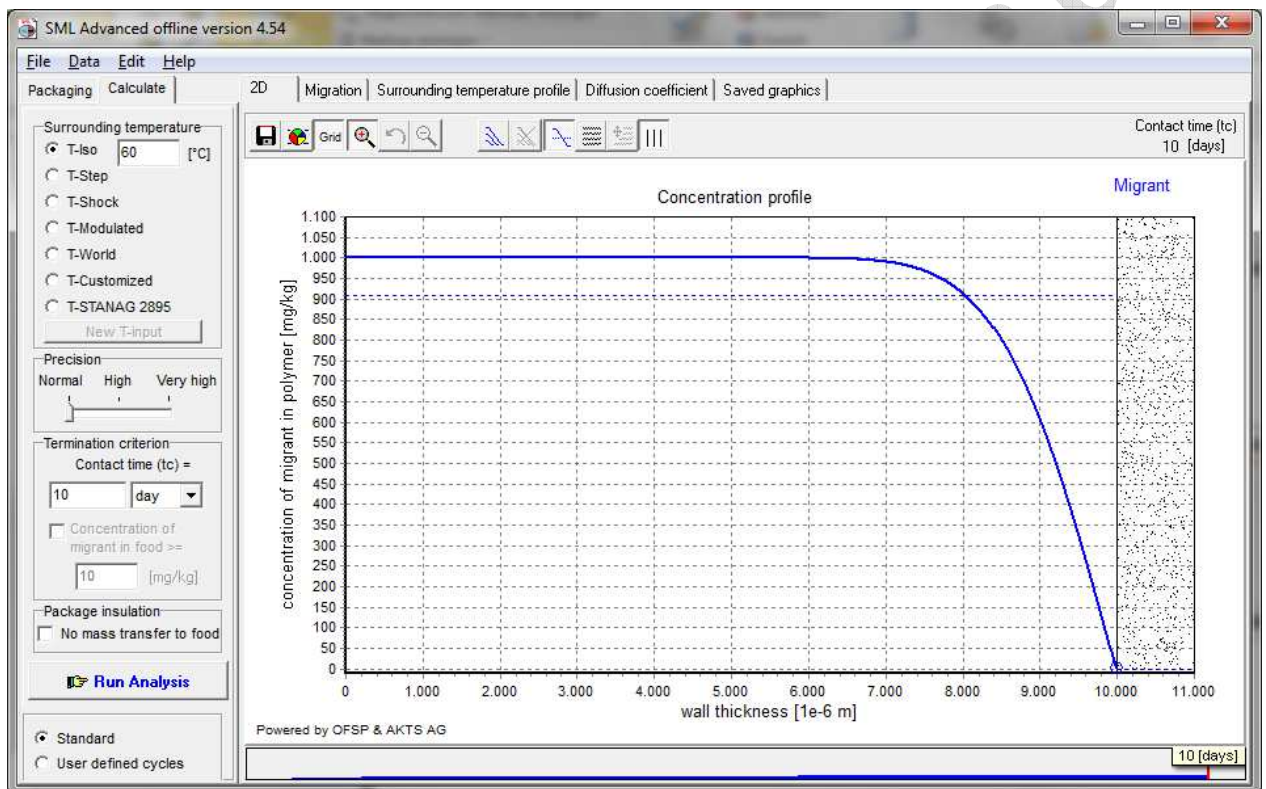
1408 to be used for worst case calculation of specific migration under assumption of total transfer

1409 => 2 x 99% layer thickness = 7200 µm

1410 above 7200 µm two sides to be considered for calculation of migration if full immersion

1411 testing applied

1412



1417

1418 **10d @ 40°C**

1419 => 100% layer thickness = 1750 µm

1420 no absolute barrier at thicknesses below 1750 µm

1421 => 99% layer thickness = 1400 µm

1422 => 1/2 x 99% layer thickness = 700 µm

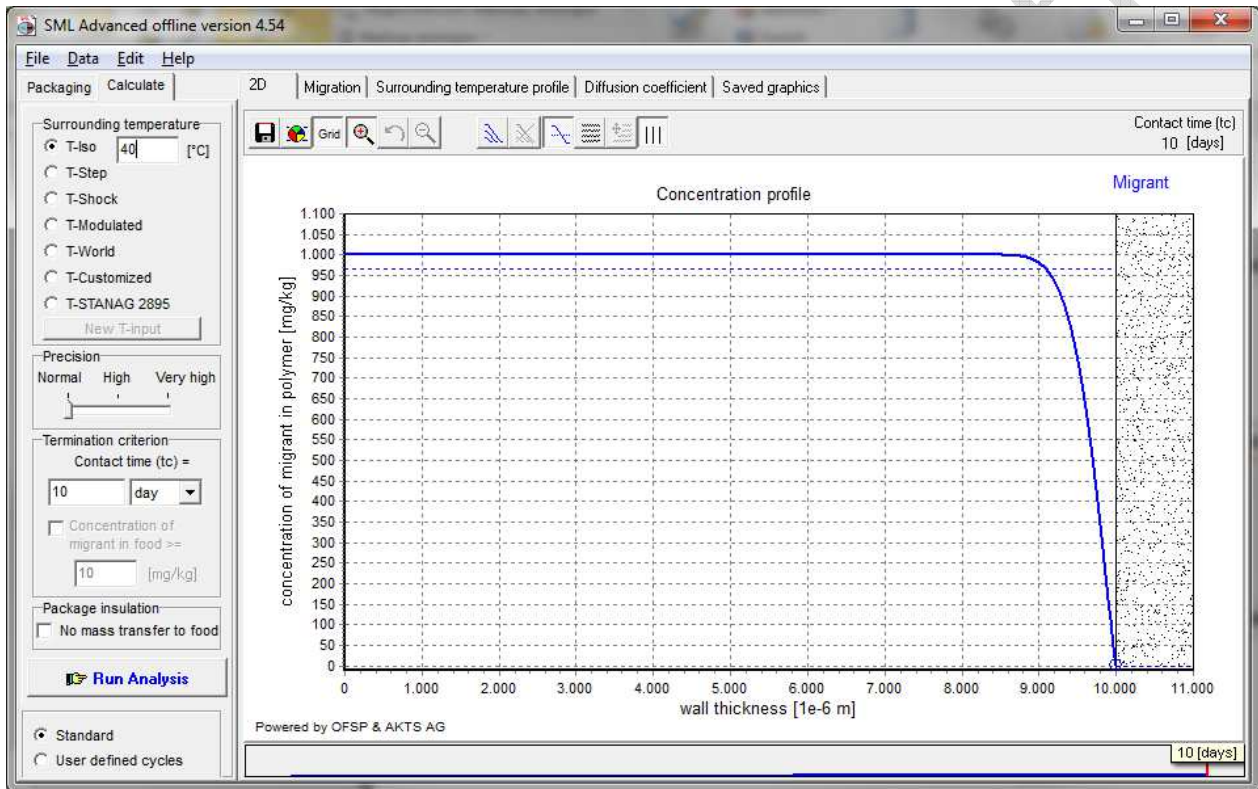
1423 to be used for worst case calculation of specific migration under assumption of total transfer

1424 => 2 x 99% layer thickness = 2800 µm

1425 above 2800 µm two sides to be considered for calculation of migration if full immersion

1426 testing applied

1427



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1433 **10d @ 20°C**

1434 => 100% layer thickness = 600 µm

1435 no absolute barrier at thicknesses below 600 µm

1436 => 99% layer thickness = 470 µm

1437 => 1/2 x 99% layer thickness = 235 µm

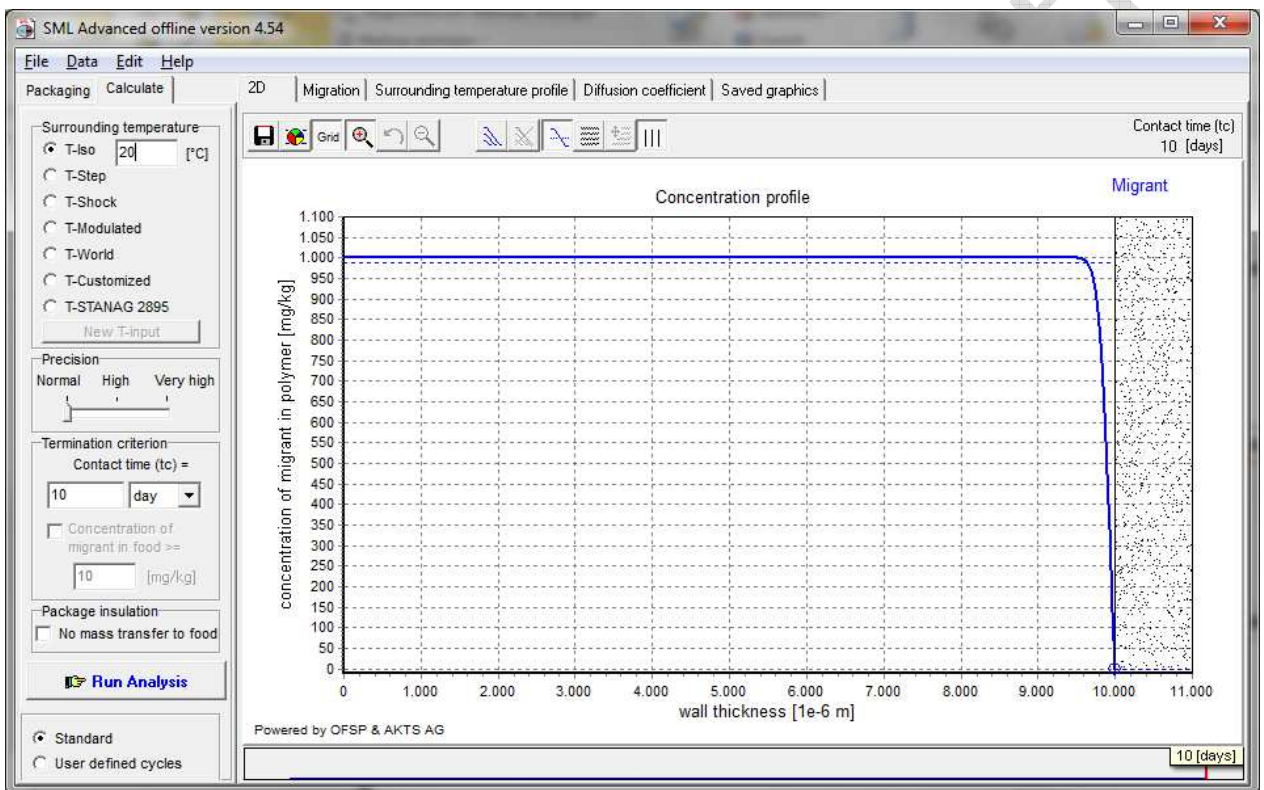
1438 to be used for worst case calculation of specific migration under assumption of total transfer

1439 => 2 x 99% layer thickness = 940 µm

1440 above 940 µm two sides to be considered for calculation of migration if full immersion testing

1441 applied

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1448 **2h @ 100°C**

1449 => 100% layer thickness = 3300 µm

1450 no absolute barrier at thicknesses below 3300 µm

1451 => 99% layer thickness = 1900 µm

1452 => 1/2 x 99% layer thickness = 950 µm

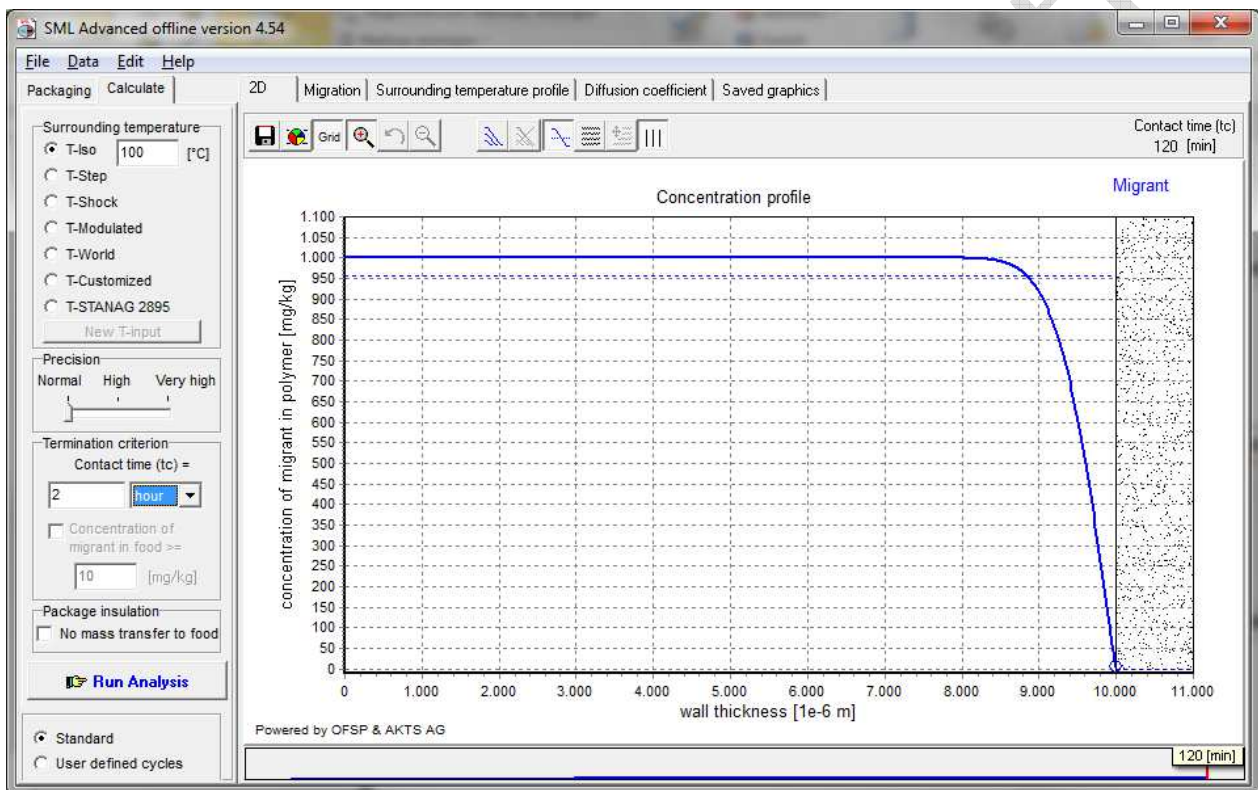
1453 to be used for worst case calculation of specific migration under assumption of total transfer

1454 => 2 x 99% layer thickness = 3800 µm

1455 above 3800 µm two sides to be considered for calculation of migration if full immersion

1456 testing applied

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1463 ► molecular mass 751 - 1000 g/mol

1464 10d @ 60°C

1465 => 100% layer thickness = 1900 µm

1466 no absolute barrier at thicknesses below 1900 µm

1467 => 99% layer thickness = 1500 µm

1468 => 1/2 x 99% layer thickness = 750 µm

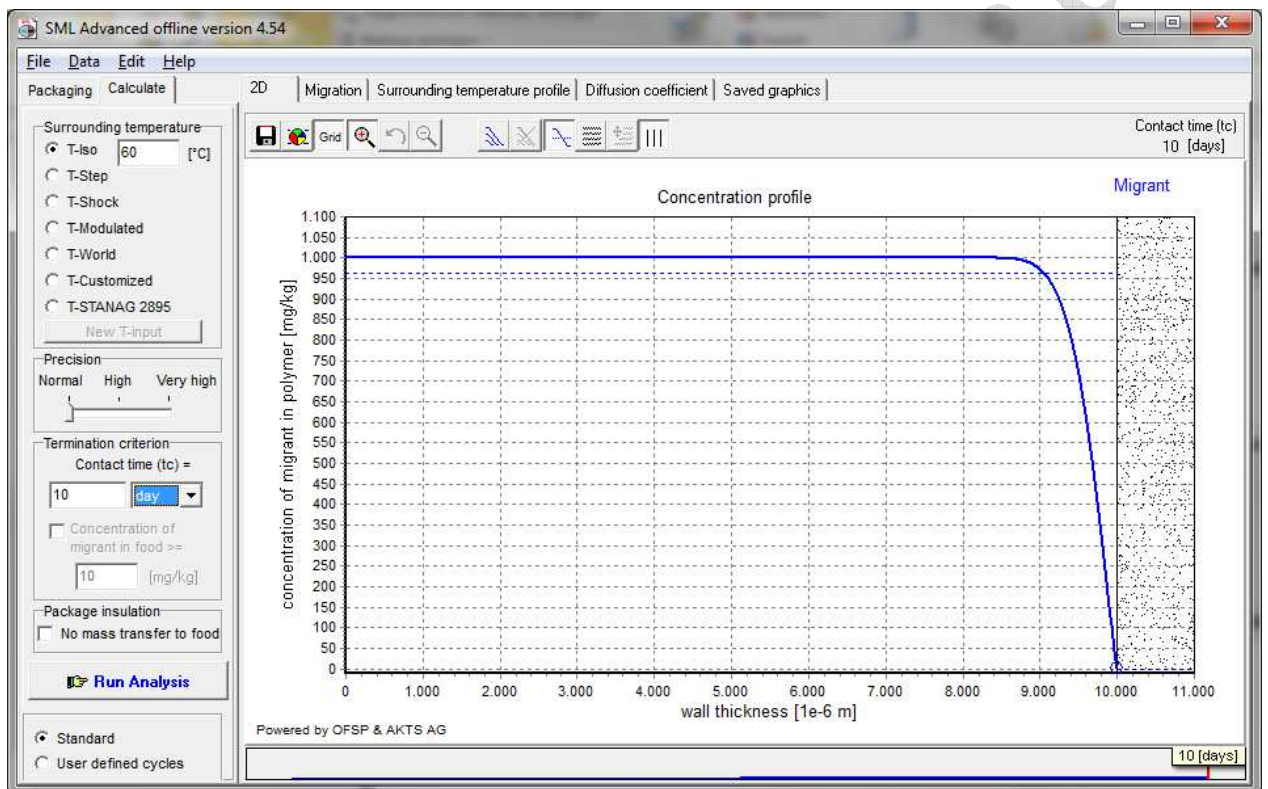
1469 to be used for worst case calculation of specific migration under assumption of total transfer

1470 => 2 x 99% layer thickness = 3000 µm

1471 above 3000 µm two sides to be considered for calculation of migration if full immersion

1472 testing applied

1473



1478

1479 **10d @ 40°C**

1480 => 100% layer thickness = 700 µm

1481 no absolute barrier at thicknesses below 700 µm

1482 => 99% layer thickness = 570 µm

1483 => 1/2 x 99% layer thickness = 285 µm

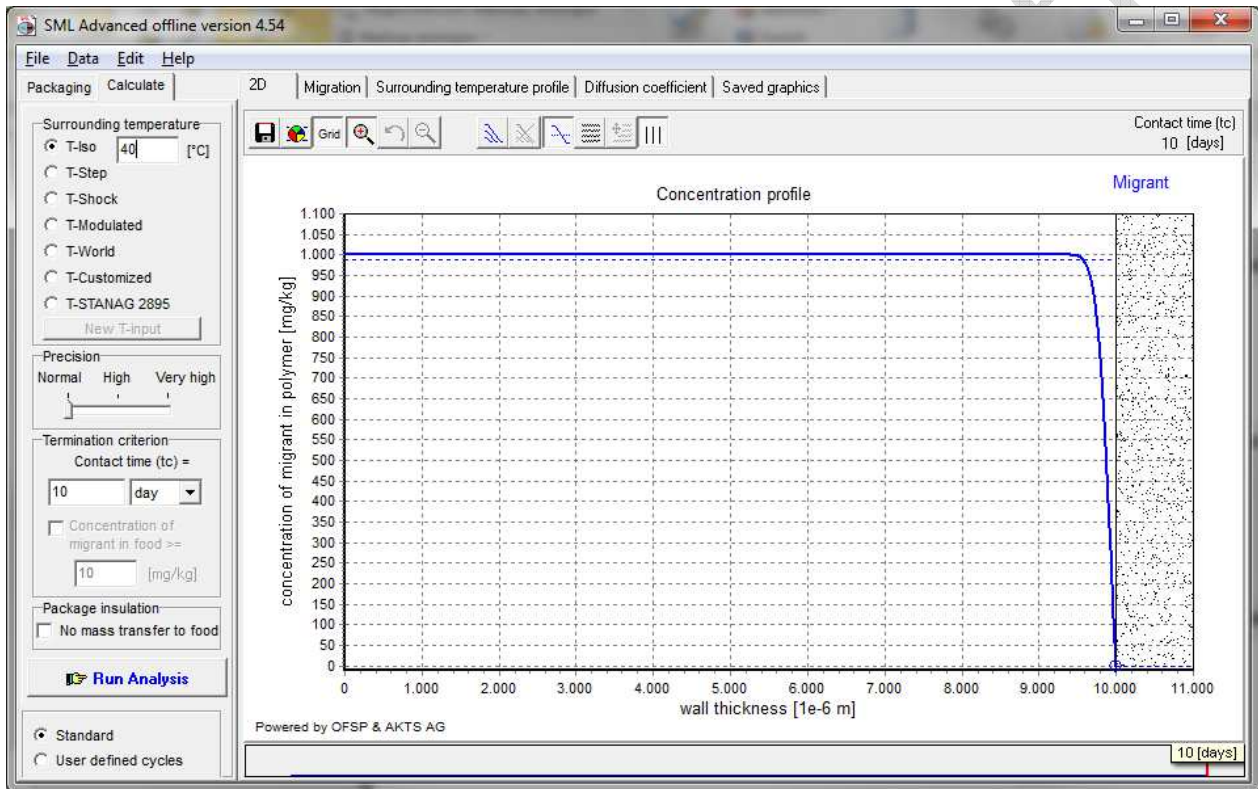
1484 to be used for worst case calculation of specific migration under assumption of total transfer

1485 => 2 x 99% layer thickness = 1140 µm

1486 above 1140 µm two sides to be considered for calculation of migration if full immersion

1487 testing applied

1488



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1494 **10d @ 20°C**

1495 => 100% layer thickness = 280 µm

1496 no absolute barrier at thicknesses below 280 µm

1497 => 99% layer thickness = 200 µm

1498 => 1/2 x 99% layer thickness = 100 µm

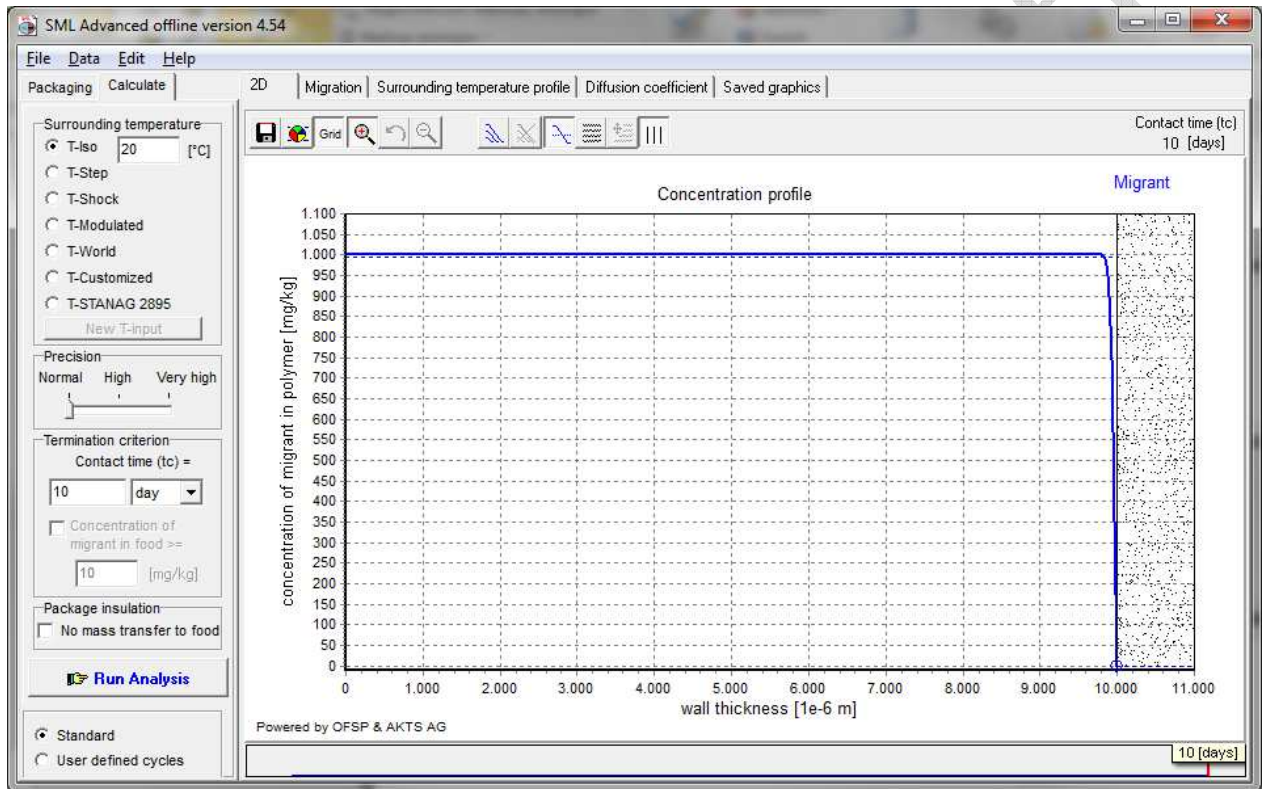
1499 to be used for worst case calculation of specific migration under assumption of total transfer

1500 => 2 x 99% layer thickness = 400 µm

1501 above 400 µm two sides to be considered for calculation of migration if full immersion testing

1502 applied

1503



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1509 **2h @ 100°C**

1510 => 100% layer thickness = 1000 µm

1511 no absolute barrier at thicknesses below 1000 µm

1512 => 99% layer thickness = 750 µm

1513 => 1/2 x 99% layer thickness = 375 µm

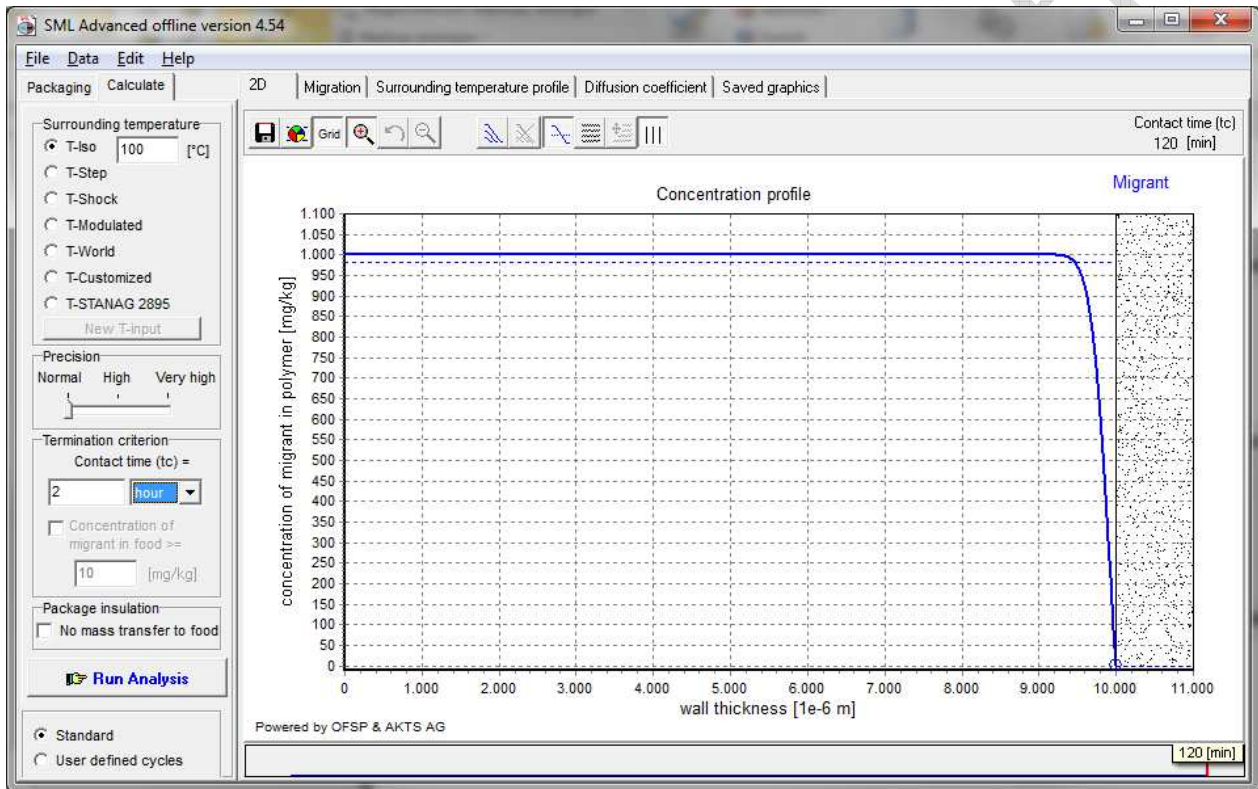
1514 to be used for worst case calculation of specific migration under assumption of total transfer

1515 => 2 x 99% layer thickness = 1500 µm

1516 above 1500 µm two sides to be considered for calculation of migration if full immersion

1517 testing applied

1518



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1524 **PA6**

1525 *(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct*
1526 *contact, e.g. plastic multilayer)*

1527 ► **molecular mass 100 - 250 g/mol**

1528 **10d @ 60°C**

1529 => 100% layer thickness = 210 µm

1530 no absolute barrier at thicknesses below 210 µm

1531 => 99% layer thickness = 182 µm

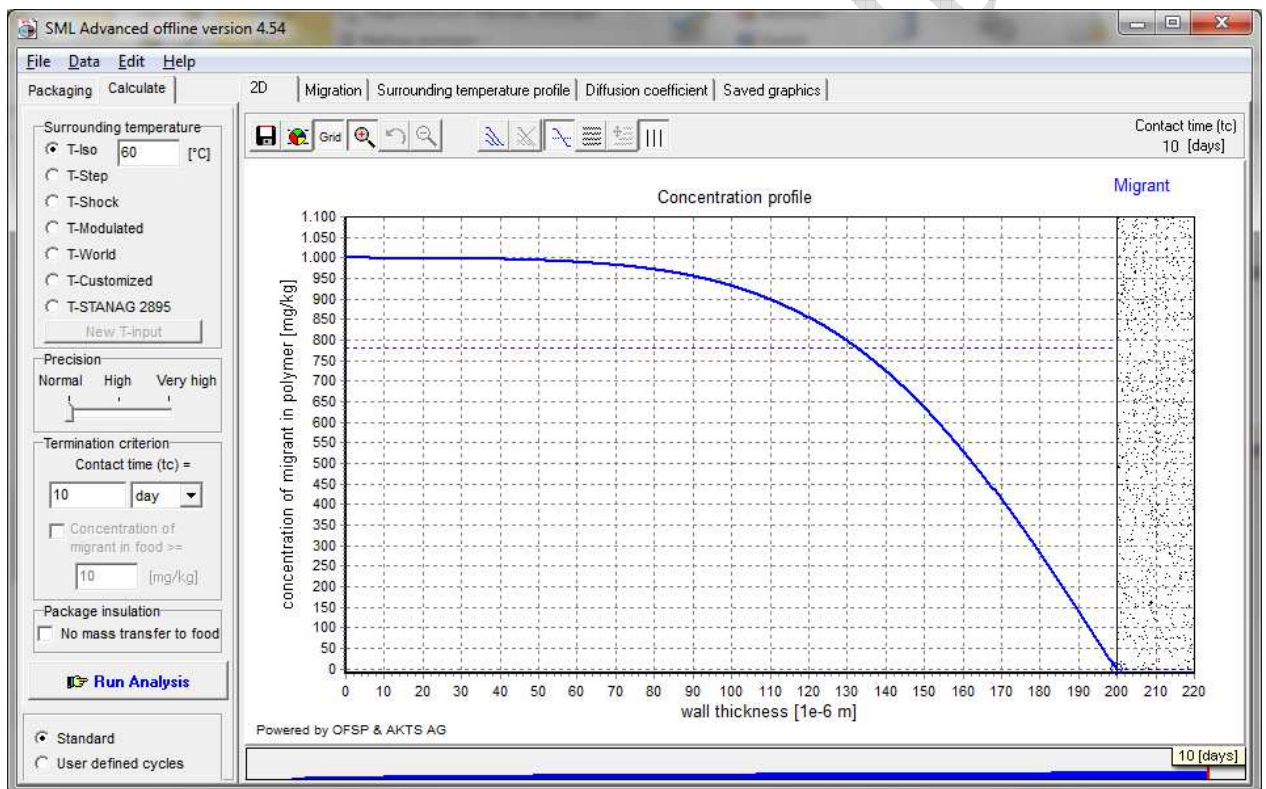
1532 => 1/2 x 99% layer thickness = 91 µm

1533 to be used for worst case calculation of specific migration under assumption of total transfer

1534 => 2 x 99% layer thickness = 364 µm

1535 above 364 µm two sides to be considered for calculation of migration if full immersion testing
1536 applied

1537



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1543 **10d @ 40°C**

1544 => 100% layer thickness = 80 µm

1545 no absolute barrier at thicknesses below 80 µm

1546 => 99% layer thickness = 68 µm

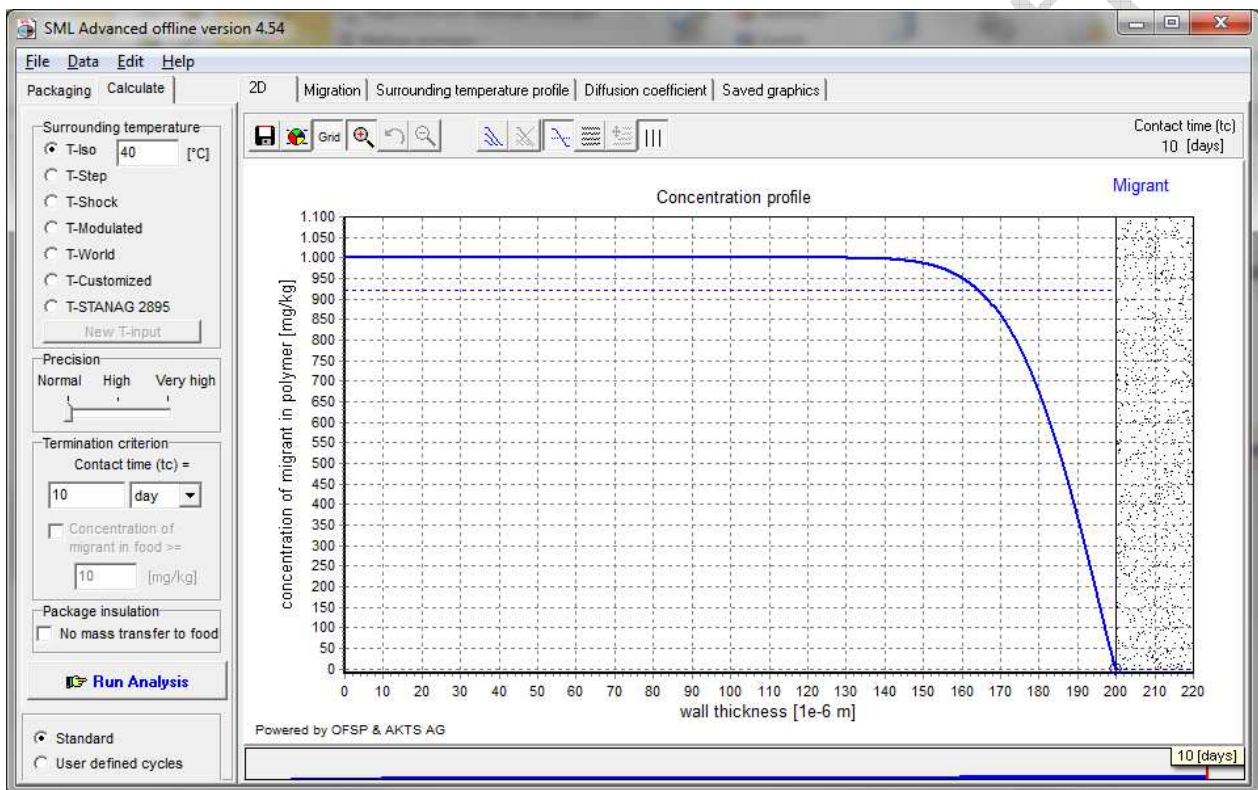
1547 => 1/2 x 99% layer thickness = 34 µm

1548 to be used for worst case calculation of specific migration under assumption of total transfer

1549 => 2 x 99% layer thickness = 136 µm

1550 above 136 µm two sides to be considered for calculation of migration if full immersion testing
1551 applied

1552



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1558 **10d @ 20°C**

1559 => 100% layer thickness = 26 µm

1560 no absolute barrier at thicknesses below 26 µm

1561 => 99% layer thickness = 22 µm

1562 => 1/2 x 99% layer thickness = 11 µm

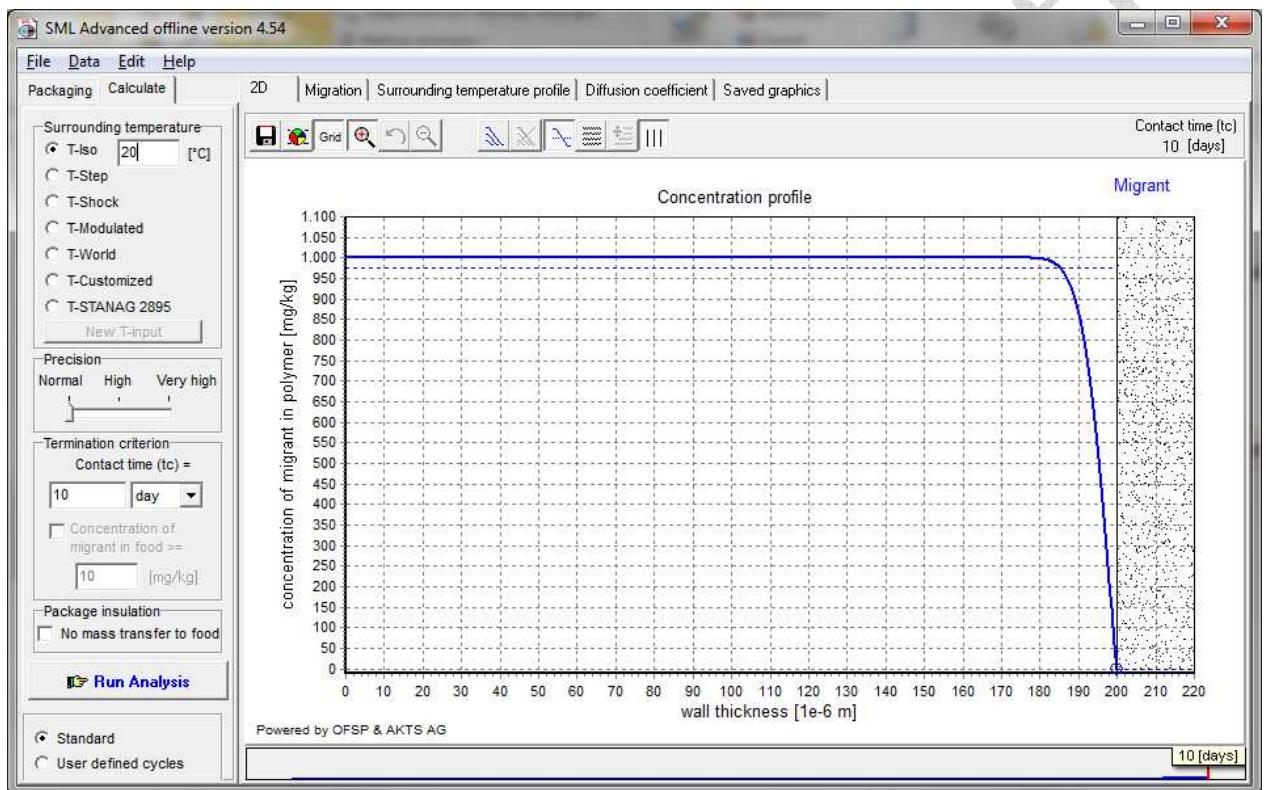
1563 to be used for worst case calculation of specific migration under assumption of total transfer

1564 => 2 x 99% layer thickness = 44 µm

1565 above 44 µm two sides to be considered for calculation of migration if full immersion testing

1566 applied

1567



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1573 **2h @ 100°C**

1574 => 100% layer thickness = 105 µm

1575 no absolute barrier at thicknesses below 105 µm

1576 => 99% layer thickness = 88 µm

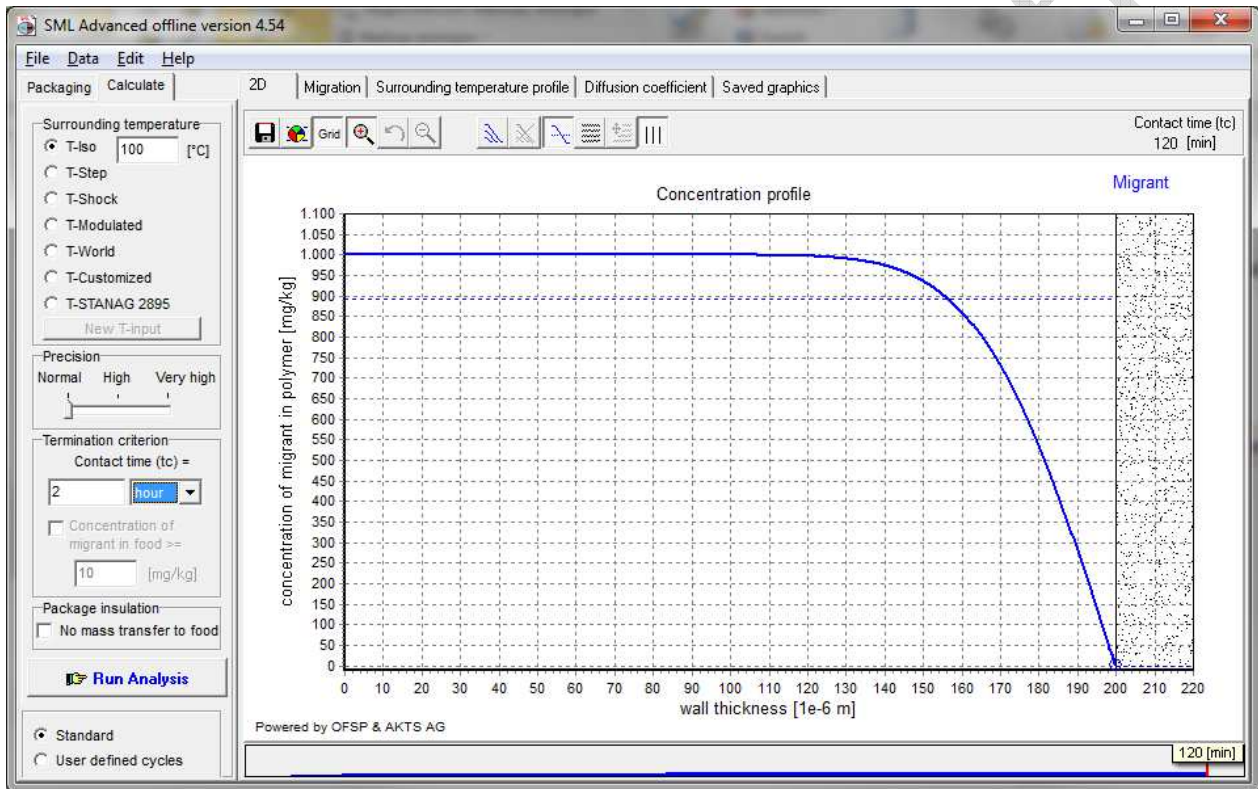
1577 => 1/2 x 99% layer thickness = 44 µm

1578 to be used for worst case calculation of specific migration under assumption of total transfer

1579 => 2 x 99% layer thickness = 176 µm

1580 above 176 µm two sides to be considered for calculation of migration if full immersion testing applied

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1588 ► molecular mass 251 - 500 g/mol

1589 10d @ 60°C

1590 => 100% layer thickness = 82 µm

1591 no absolute barrier at thicknesses below 82 µm

1592 => 99% layer thickness = 66 µm

1593 => 1/2 x 99% layer thickness = 33 µm

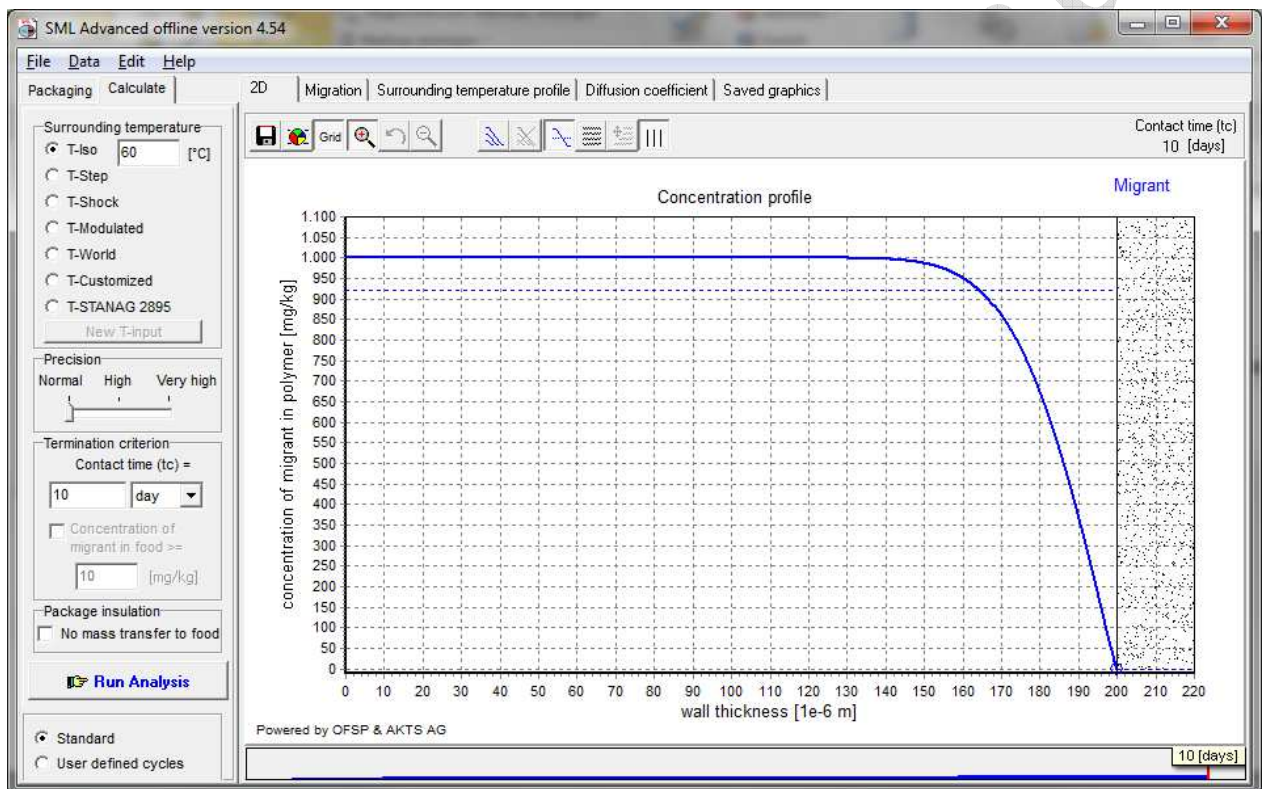
1594 to be used for worst case calculation of specific migration under assumption of total transfer

1595 => 2 x 99% layer thickness = 132 µm

1596 above 132 µm two sides to be considered for calculation of migration if full immersion testing

1597 applied

1598



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1604 **10d @ 40°C**

1605 => 100% layer thickness = 32 µm

1606 no absolute barrier at thicknesses below 32 µm

1607 => 99% layer thickness = 25 µm

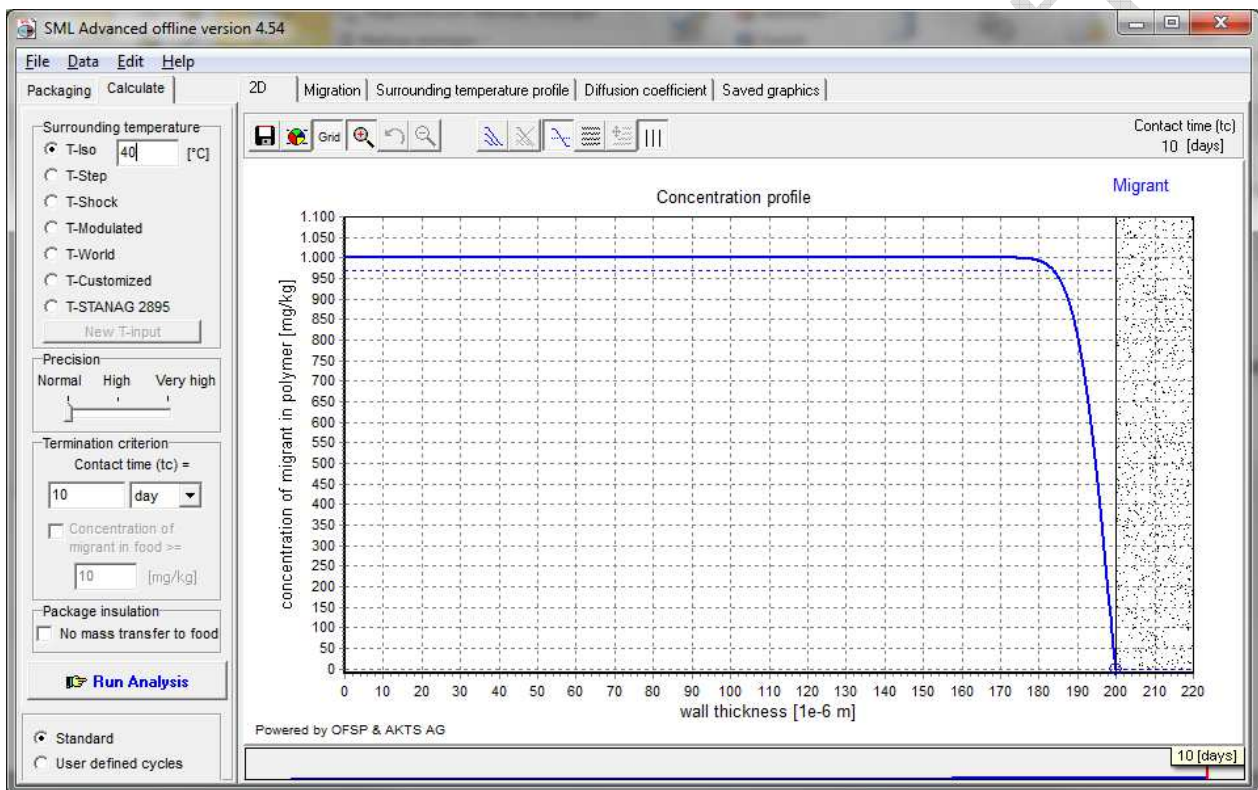
1608 => 1/2 x 99% layer thickness = 12.5 µm

1609 to be used for worst case calculation of specific migration under assumption of total transfer

1610 => 2 x 99% layer thickness = 50 µm

1611 above 50 µm two sides to be considered for calculation of migration if full immersion testing
1612 applied

1613



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1619 **10d @ 20°C**

1620 => 100% layer thickness = 11 µm

1621 no absolute barrier at thicknesses below 11 µm

1622 => 99% layer thickness = 8.2 µm

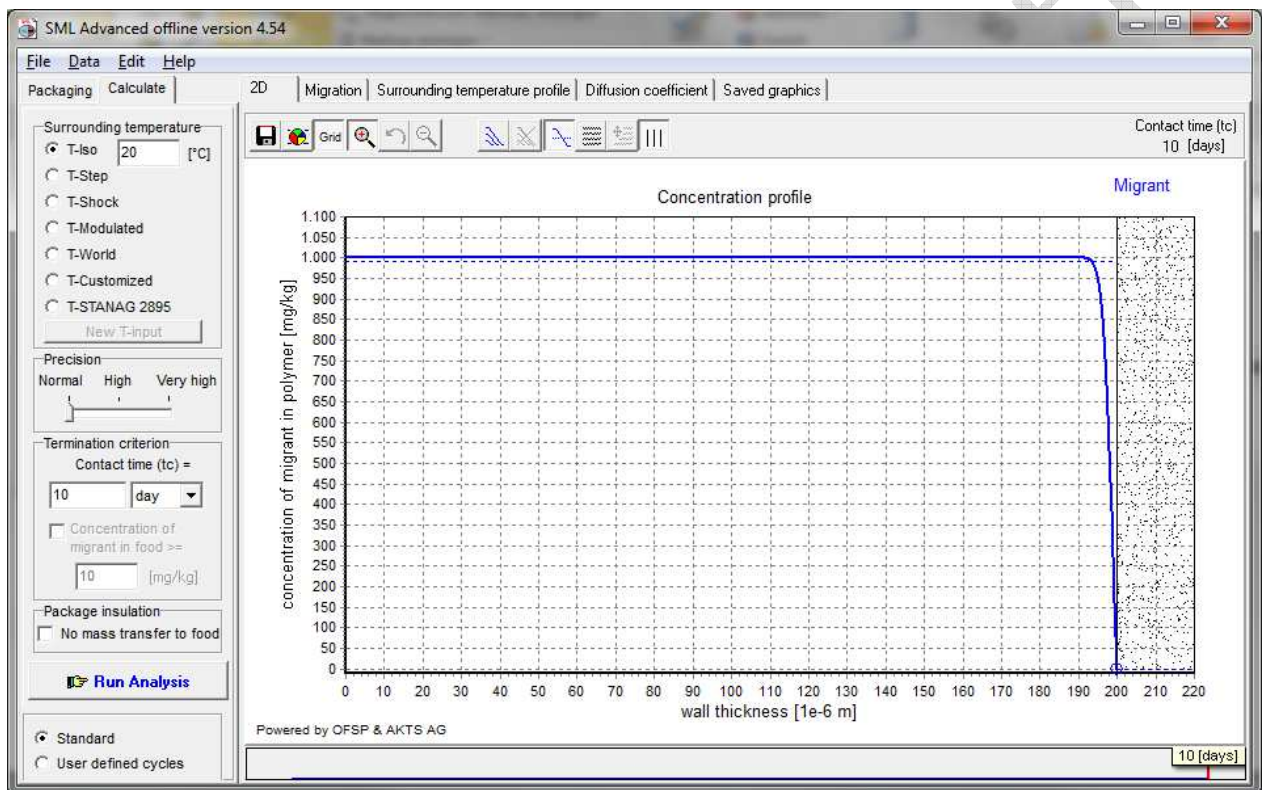
1623 => 1/2 x 99% layer thickness = 4.1 µm

1624 to be used for worst case calculation of specific migration under assumption of total transfer

1625 => 2 x 99% layer thickness = 16.4 µm

1626 above 16.4 µm two sides to be considered for calculation of migration if full immersion testing applied

1628



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1634 **2h @ 100°C**

1635 => 100% layer thickness = 40 µm

1636 no absolute barrier at thicknesses below 40 µm

1637 => 99% layer thickness = 33 µm

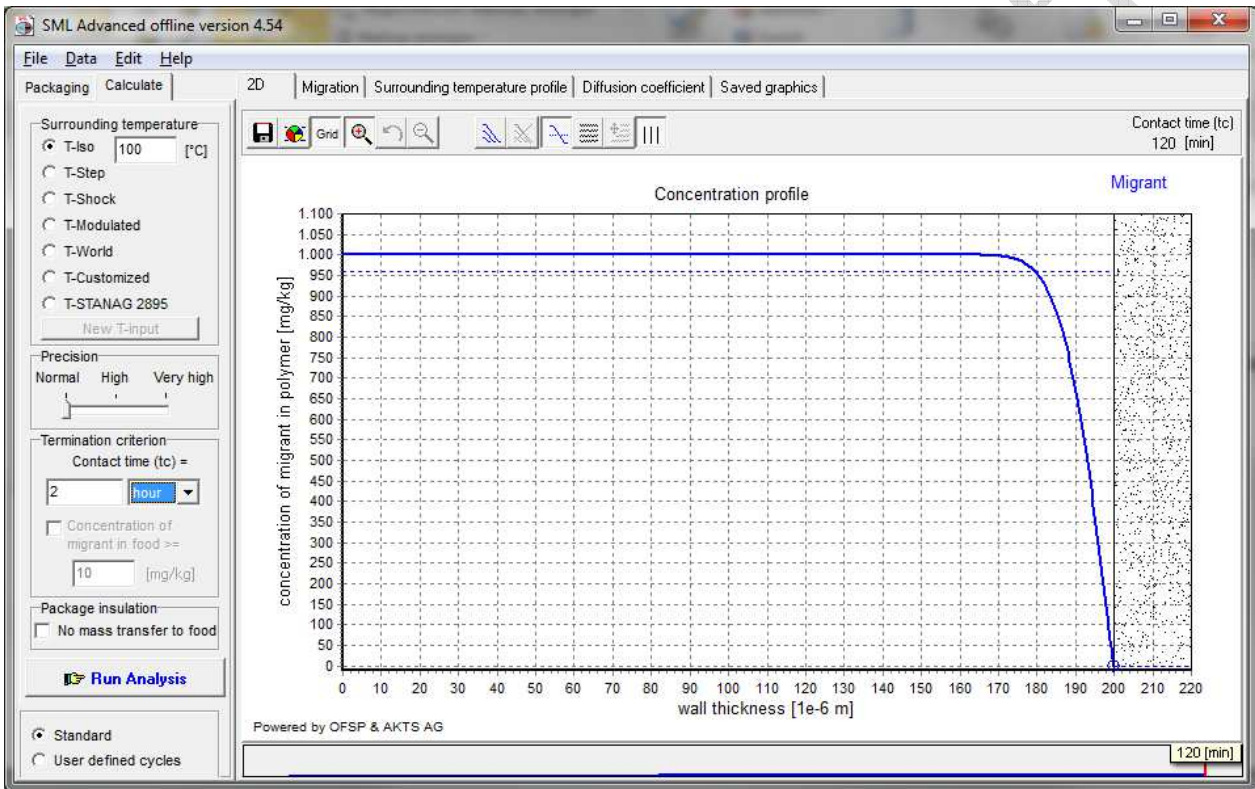
1638 => 1/2 x 99% layer thickness = 16.5 µm

1639 to be used for worst case calculation of specific migration under assumption of total transfer

1640 => 2 x 99% layer thickness = 66 µm

1641 above 66 µm two sides to be considered for calculation of migration if full immersion testing
1642 applied

1643



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1649 ► molecular mass 501 - 750 g/mol

1650 10d @ 60°C

1651 => 100% layer thickness = 25 µm

1652 no absolute barrier at thicknesses below 25 µm

1653 => 99% layer thickness = 20 µm

1654 => 1/2 x 99% layer thickness = 10 µm

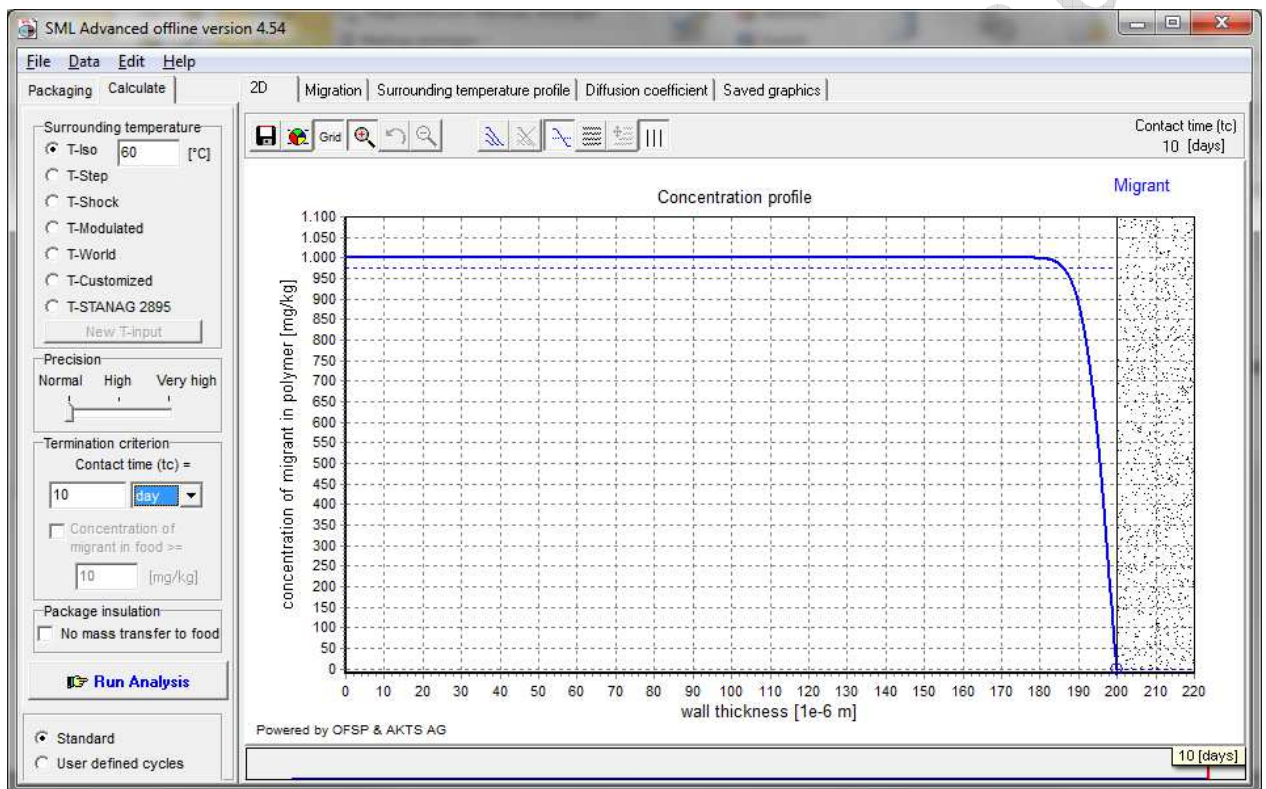
1655 to be used for worst case calculation of specific migration under assumption of total transfer

1656 => 2 x 99% layer thickness = 40 µm

1657 above 40 µm two sides to be considered for calculation of migration if full immersion testing

1658 applied

1659



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1665 **10d @ 40°C**

1666 => 100% layer thickness = 10.3 µm

1667 no absolute barrier at thicknesses below 10.3 µm

1668 => 99% layer thickness = 7.6 µm

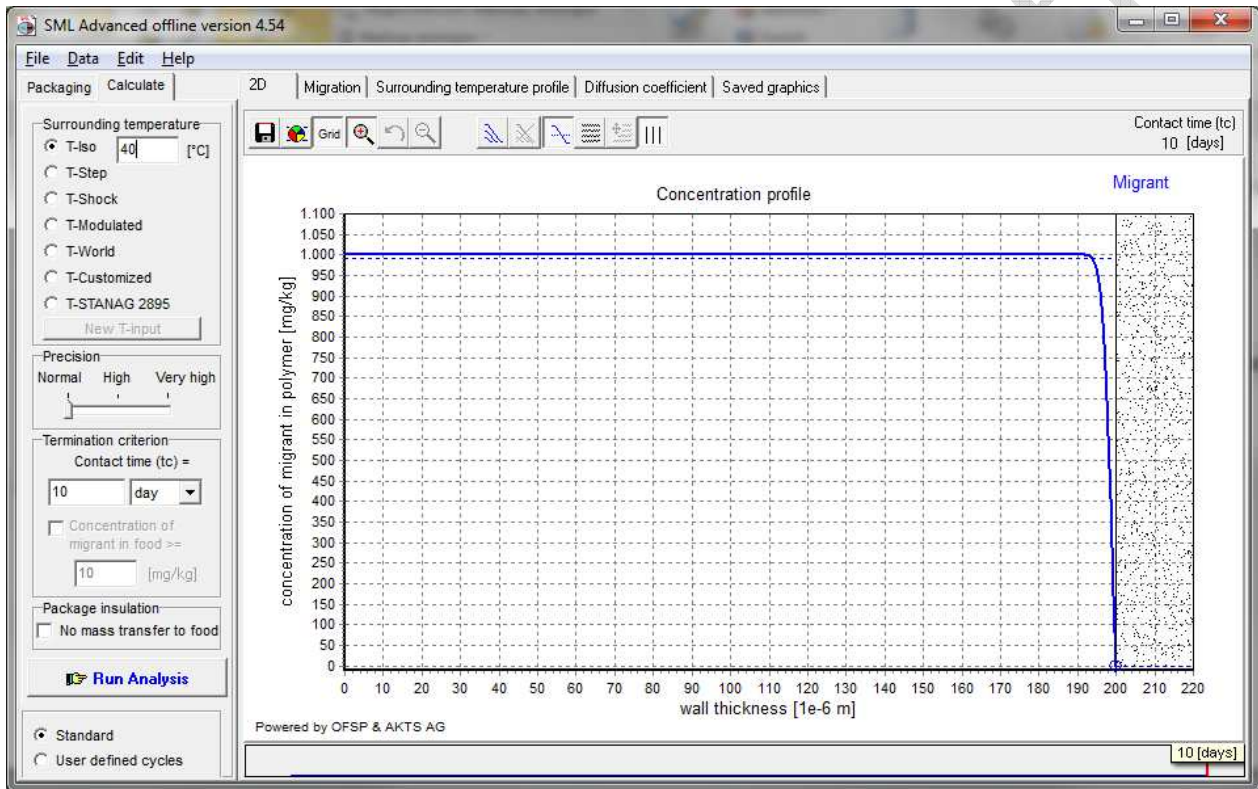
1669 => 1/2 x 99% layer thickness = 3.8 µm

1670 to be used for worst case calculation of specific migration under assumption of total transfer

1671 => 2 x 99% layer thickness = 15.2 µm

1672 above 15.2 µm two sides to be considered for calculation of migration if full immersion testing
1673 applied

1674



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1680 **10d @ 20°C**

1681 => 100% layer thickness = 4 µm

1682 no absolute barrier at thicknesses below 4 µm

1683 => 99% layer thickness = 2.8 µm

1684 => 1/2 x 99% layer thickness = 1.4 µm

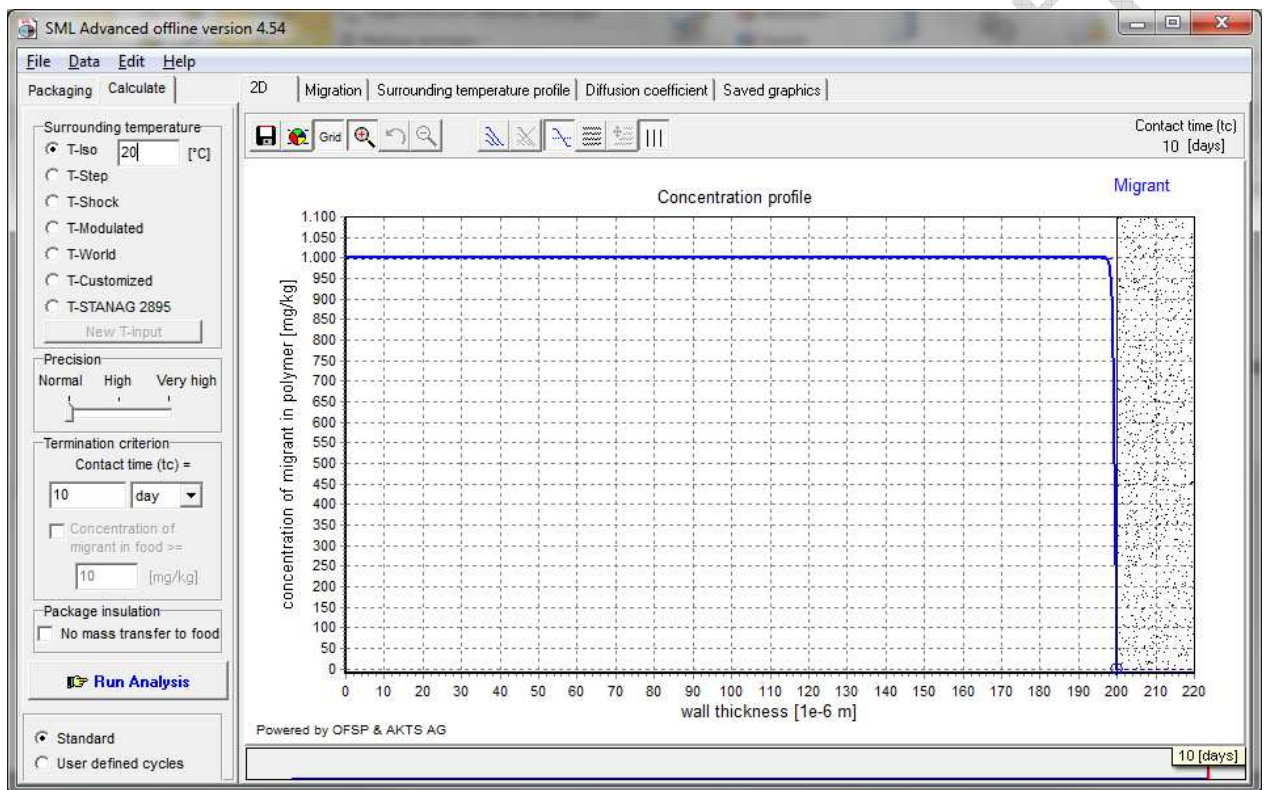
1685 to be used for worst case calculation of specific migration under assumption of total transfer

1686 => 2 x 99% layer thickness = 5.6 µm

1687 above 5.6 µm two sides to be considered for calculation of migration if full immersion testing

1688 applied

1689



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1695 **2h @ 100°C**

1696 => 100% layer thickness = 14 µm

1697 no absolute barrier at thicknesses below 14 µm

1698 => 99% layer thickness = 10.2 µm

1699 => 1/2 x 99% layer thickness = 5.1 µm

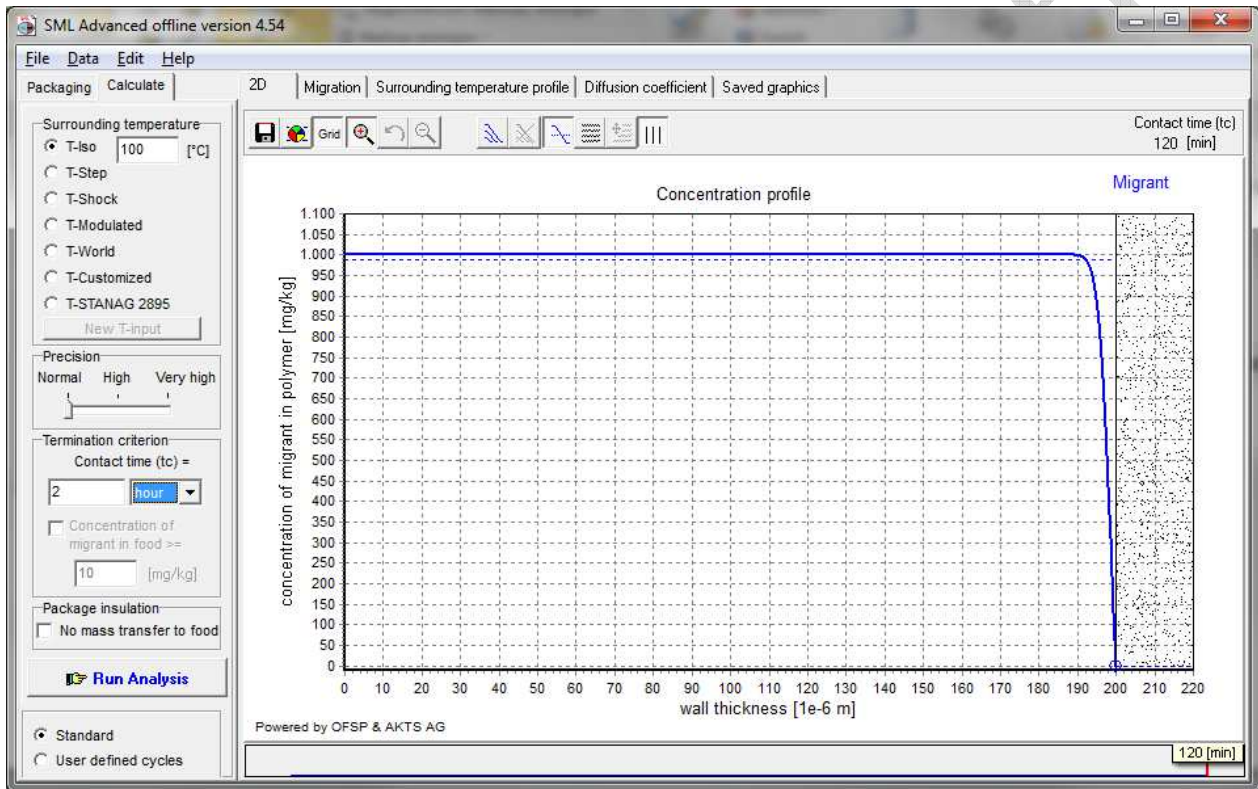
1700 to be used for worst case calculation of specific migration under assumption of total transfer

1701 => 2 x 99% layer thickness = 20.4 µm

1702 above 20.4 µm two sides to be considered for calculation of migration if full immersion testing

1703 applied

1704



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1710 ► molecular mass 751 - 1000 g/mol

1711 10d @ 60°C

1712 => 100% layer thickness = 10 µm

1713 no absolute barrier at thicknesses below 10 µm

1714 => 99% layer thickness = 8 µm

1715 => 1/2 x 99% layer thickness = 4 µm

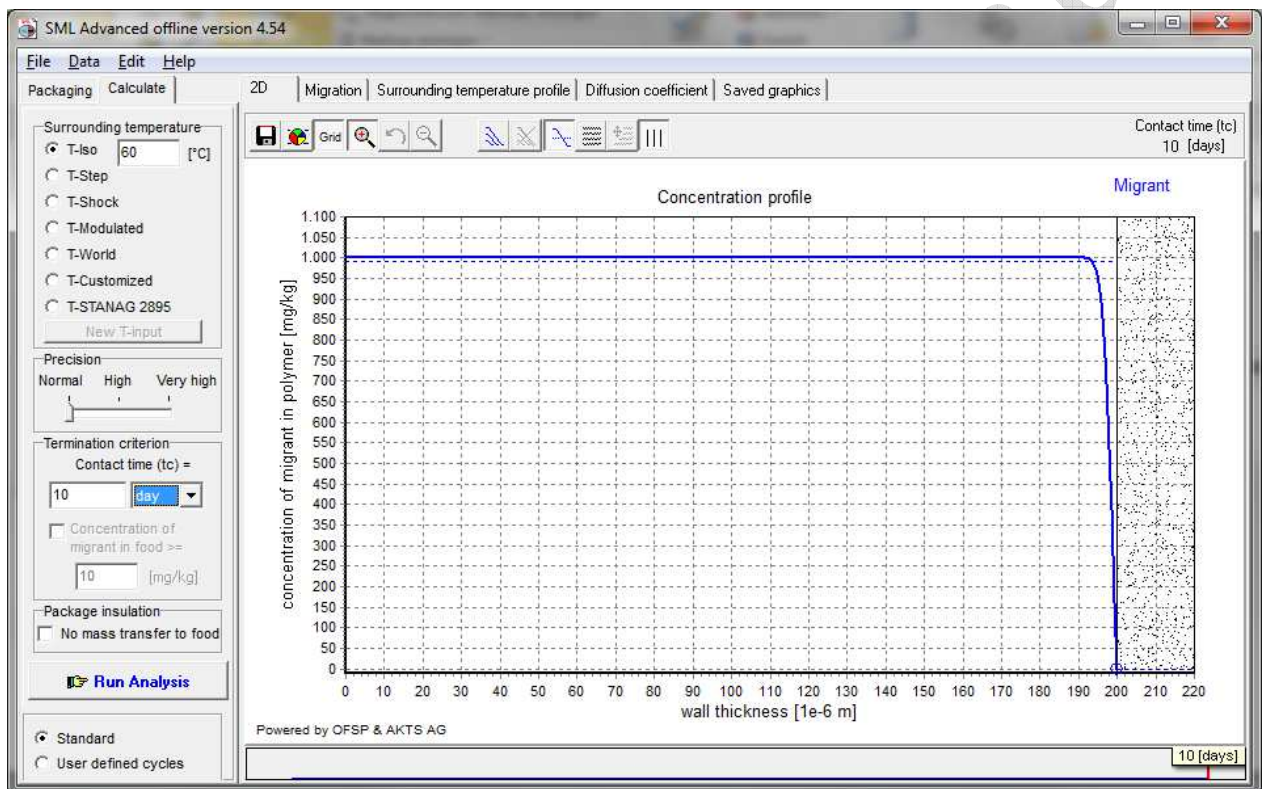
1716 to be used for worst case calculation of specific migration under assumption of total transfer

1717 => 2 x 99% layer thickness = 16 µm

1718 above 16 µm two sides to be considered for calculation of migration if full immersion testing

1719 applied

1720



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1726 **10d @ 40°C**

1727 => 100% layer thickness = 4.2 µm

1728 no absolute barrier at thicknesses below 4.2 µm

1729 => 99% layer thickness = 3.4 µm

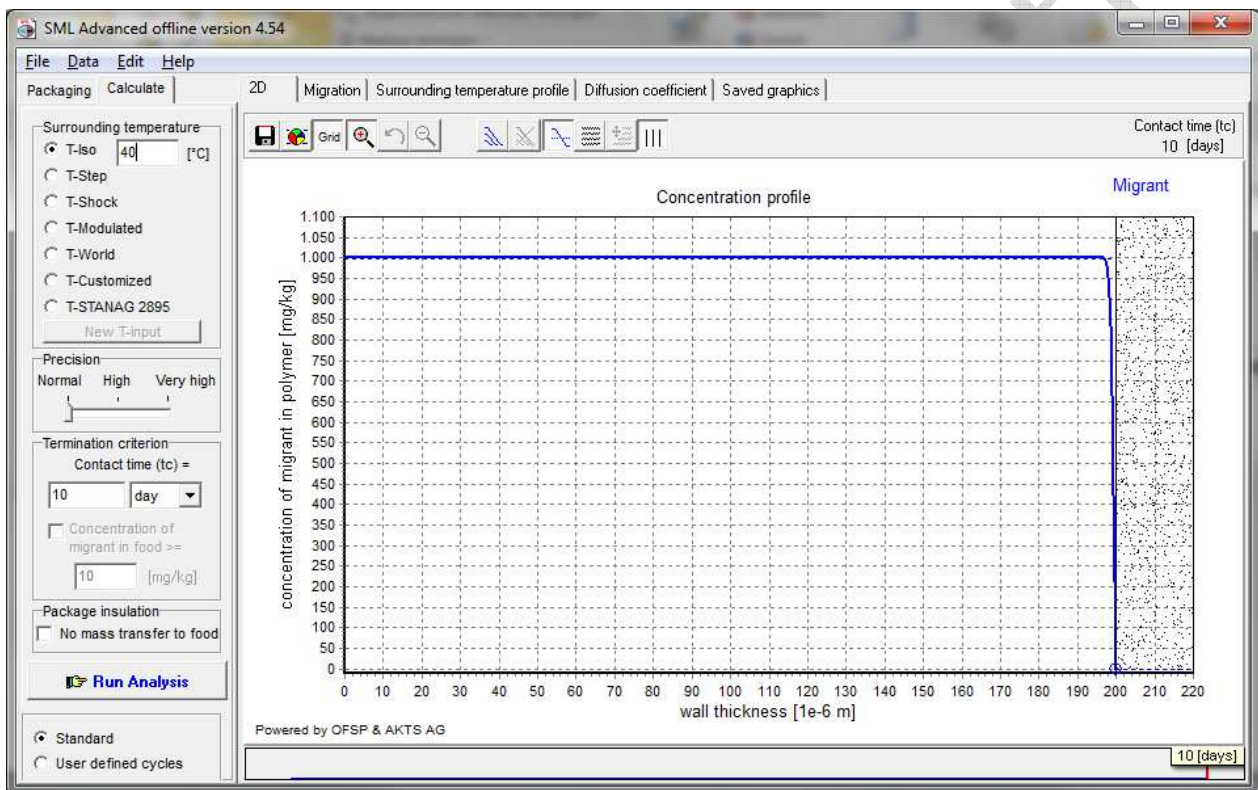
1730 => 1/2 x 99% layer thickness = 1.7 µm

1731 to be used for worst case calculation of specific migration under assumption of total transfer

1732 => 2 x 99% layer thickness = 6.8 µm

1733 above 6.8 µm two sides to be considered for calculation of migration if full immersion testing
1734 applied

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1741 **10d @ 20°C**

1742 => 100% layer thickness = 1.8 µm

1743 no absolute barrier at thicknesses below 1.8 µm

1744 => 99% layer thickness = 1.2 µm

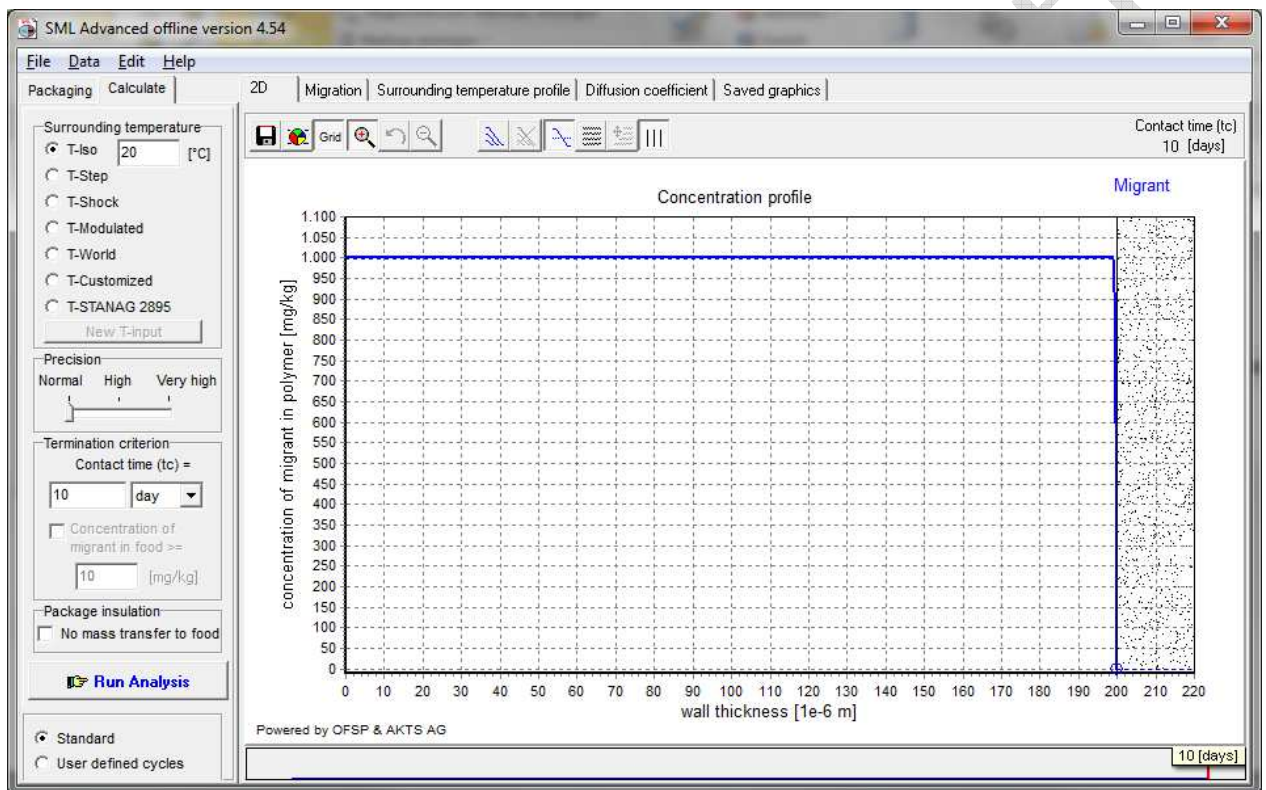
1745 => 1/2 x 99% layer thickness = 0.6 µm

1746 to be used for worst case calculation of specific migration under assumption of total transfer

1747 => 2 x 99% layer thickness = 2.4 µm

1748 above 2.4 µm two sides to be considered for calculation of migration if full immersion testing applied

1750



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1756 **2h @ 100°C**

1757 => 100% layer thickness = 5.5 µm

1758 no absolute barrier at thicknesses below 5.5 µm

1759 => 99% layer thickness = 4.4 µm

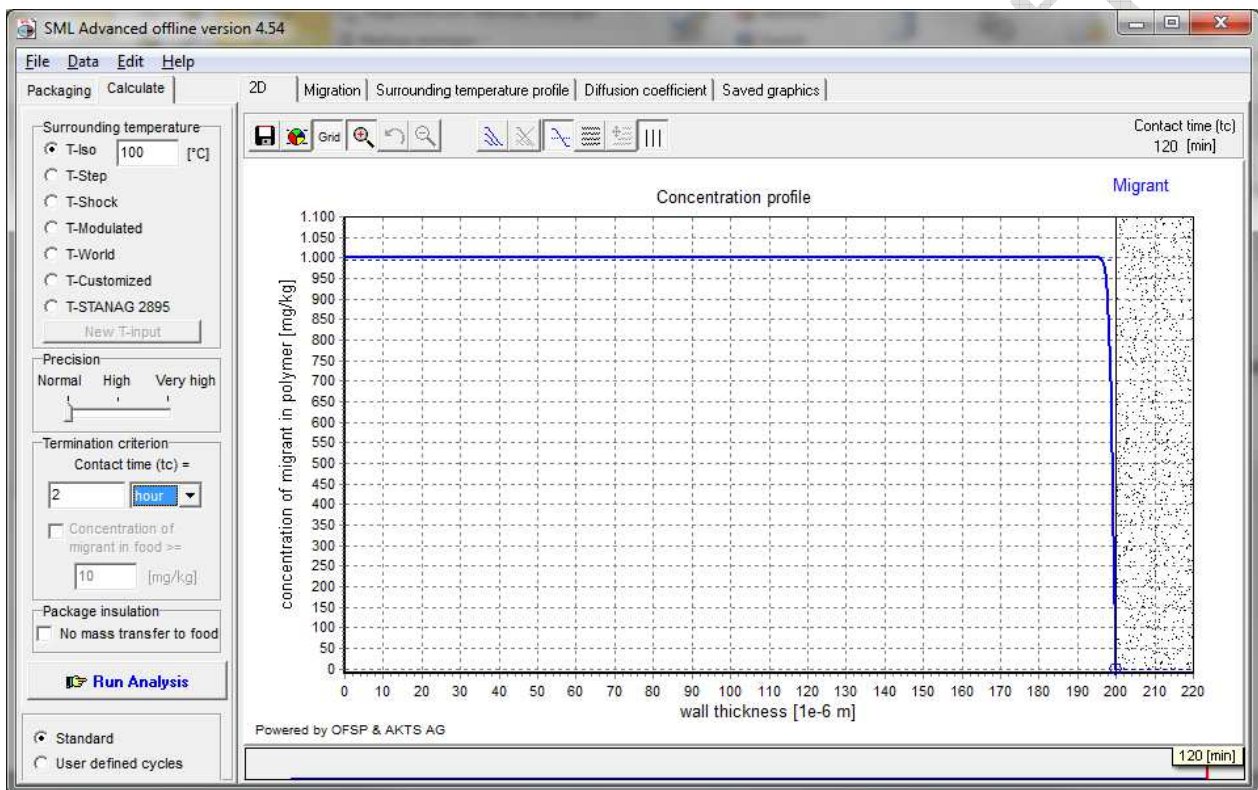
1760 => 1/2 x 99% layer thickness = 2.2 µm

1761 to be used for worst case calculation of specific migration under assumption of total transfer

1762 => 2 x 99% layer thickness = 8.8 µm

1763 above 8.8 µm two sides to be considered for calculation of migration if full immersion testing
1764 applied

1765



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1771 **PA6,6**

1772 *(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct*
1773 *contact, e.g. plastic multilayer)*

1774 ► **molecular mass 100 - 250 g/mol**

1775 **10d @ 60°C**

1776 => 100% layer thickness = 565 µm

1777 no absolute barrier at thicknesses below 565 µm

1778 => 99% layer thickness = 490 µm

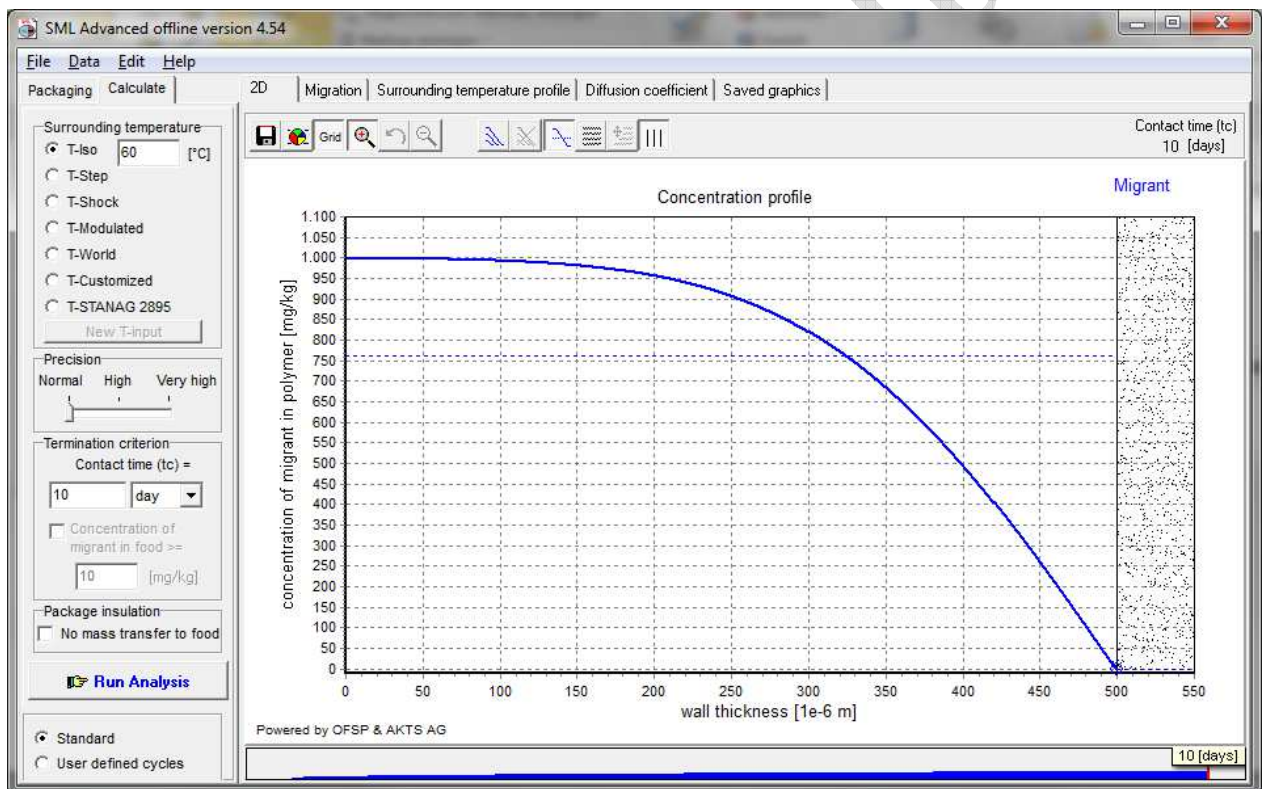
1779 => 1/2 x 99% layer thickness = 245 µm

1780 to be used for worst case calculation of specific migration under assumption of total transfer

1781 => 2 x 99% layer thickness = 980 µm

1782 above 980 µm two sides to be considered for calculation of migration if full immersion testing
1783 applied

1784



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1790 **10d @ 40°C**

1791 => 100% layer thickness = 220 µm

1792 no absolute barrier at thicknesses below 220 µm

1793 => 99% layer thickness = 180 µm

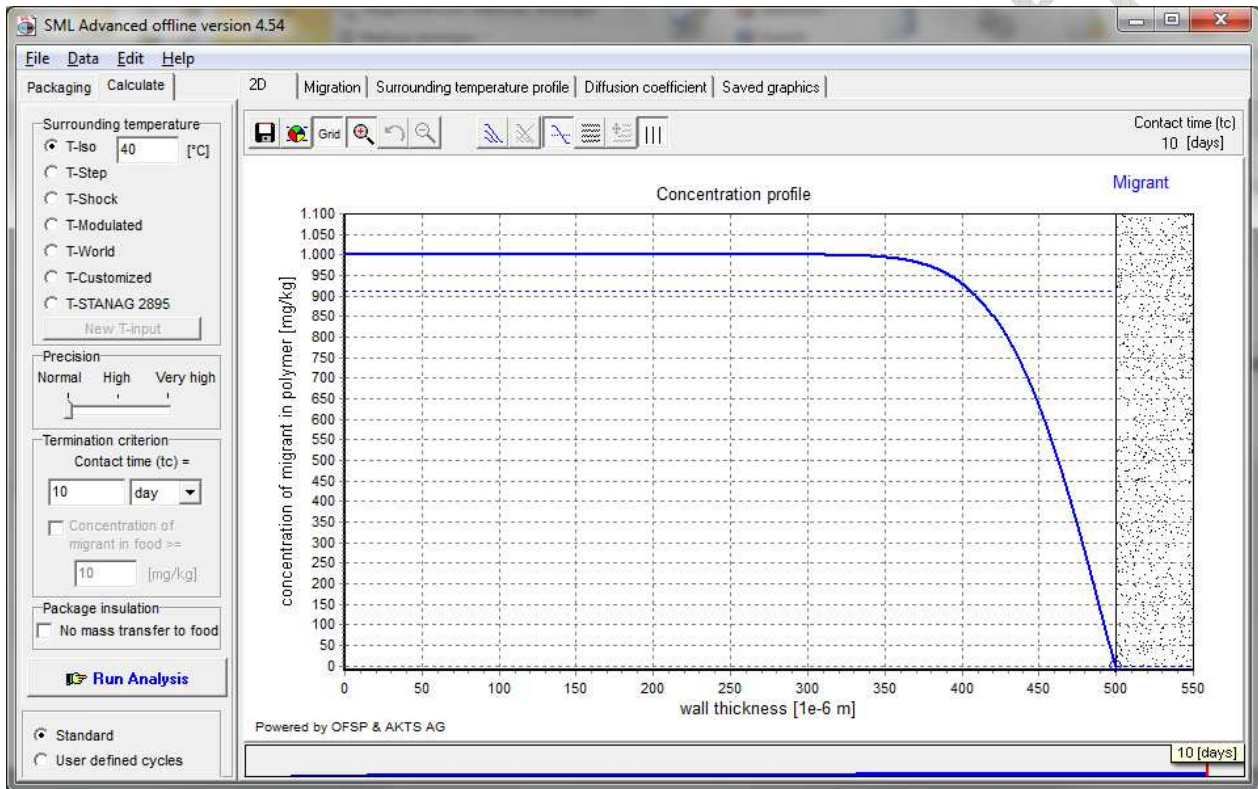
1794 => 1/2 x 99% layer thickness = 90 µm

1795 to be used for worst case calculation of specific migration under assumption of total transfer

1796 => 2 x 99% layer thickness = 360 µm

1797 above 360 µm two sides to be considered for calculation of migration if full immersion testing
1798 applied

1799



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1805 **10d @ 20°C**

1806 => 100% layer thickness = 76 µm

1807 no absolute barrier at thicknesses below 76 µm

1808 => 99% layer thickness = 58 µm

1809 => 1/2 x 99% layer thickness = 29 µm

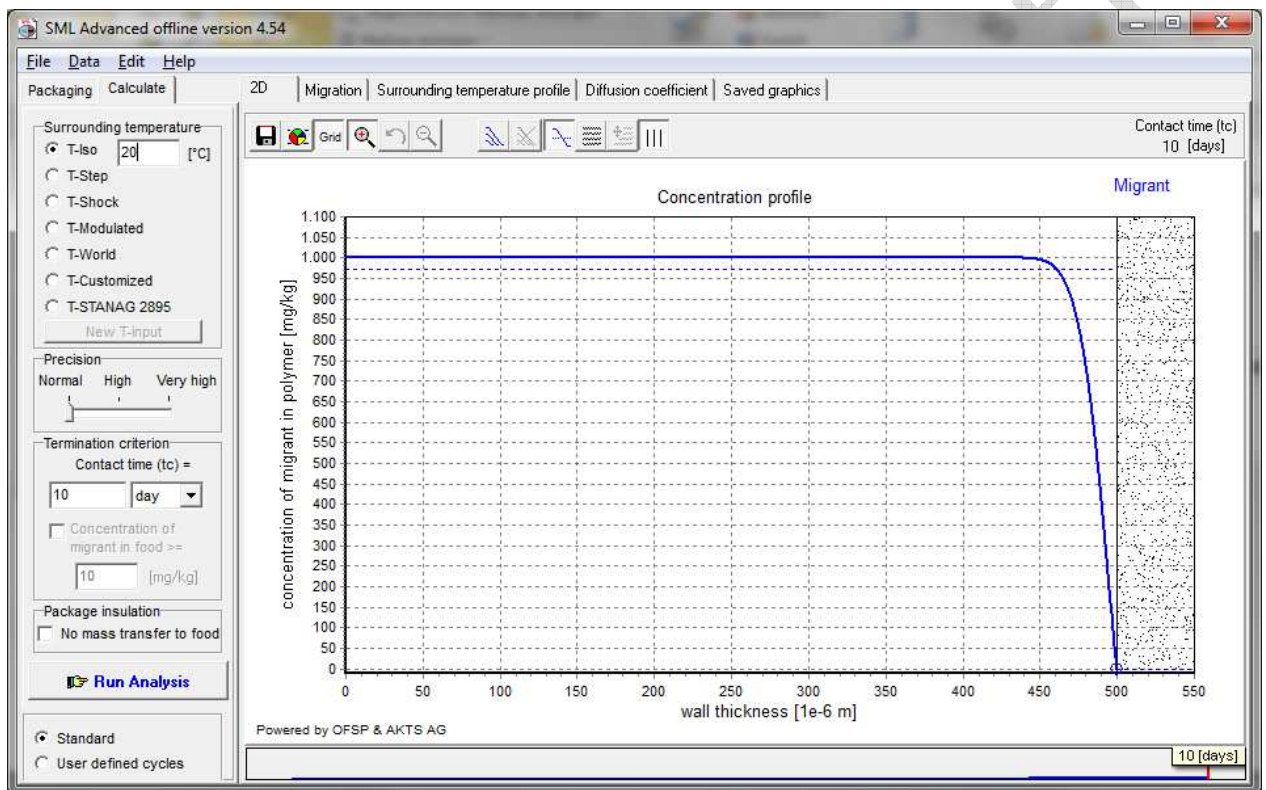
1810 to be used for worst case calculation of specific migration under assumption of total transfer

1811 => 2 x 99% layer thickness = 116 µm

1812 above 116 µm two sides to be considered for calculation of migration if full immersion testing

1813 applied

1814



1815

1816

1817

1818

1819

1820 **2h @ 100°C**

1821 => 100% layer thickness = 300 µm

1822 no absolute barrier at thicknesses below 300 µm

1823 => 99% layer thickness = 240 µm

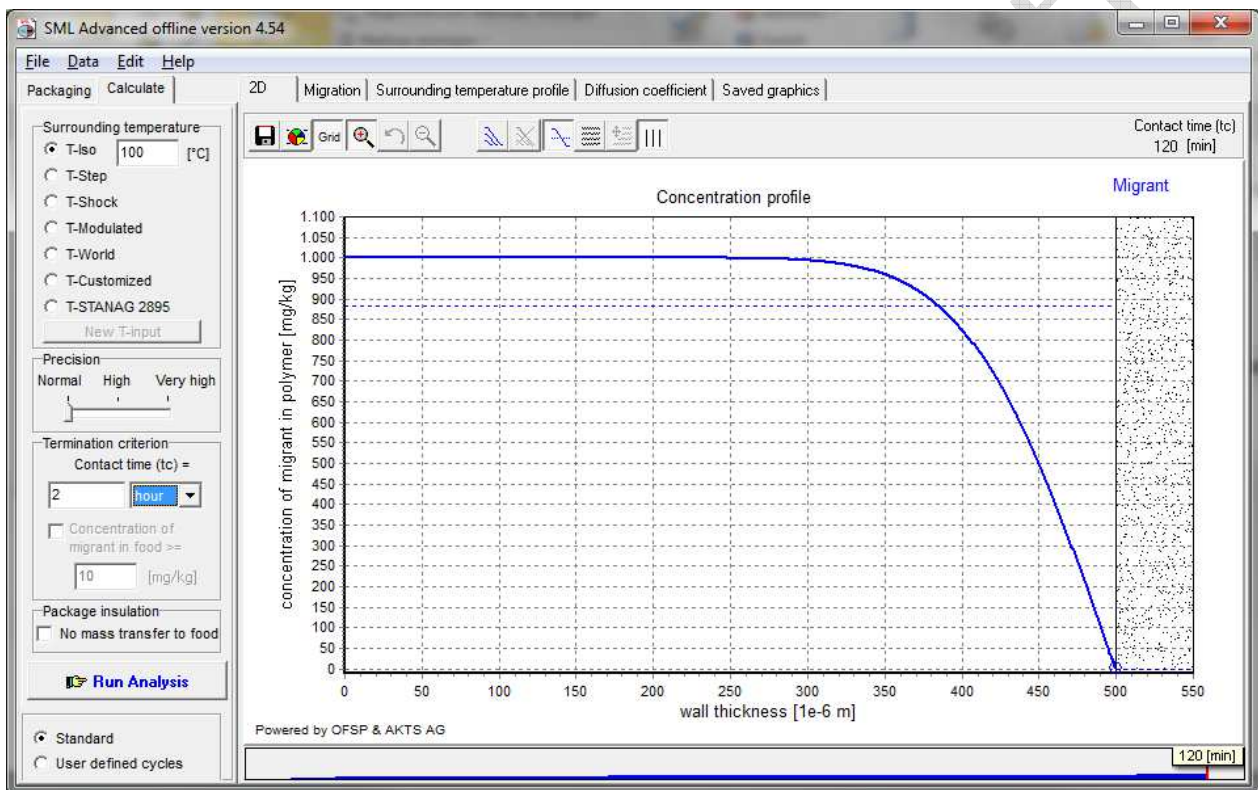
1824 => 1/2 x 99% layer thickness = 120 µm

1825 to be used for worst case calculation of specific migration under assumption of total transfer

1826 => 2 x 99% layer thickness = 480 µm

1827 above 480 µm two sides to be considered for calculation of migration if full immersion testing
1828 applied

1829



1830

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1834

1835 ► molecular mass 251 - 500 g/mol

1836 10d @ 60°C

1837 => 100% layer thickness = 225 µm

1838 no absolute barrier at thicknesses below 225 µm

1839 => 99% layer thickness = 180 µm

1840 => 1/2 x 99% layer thickness = 90 µm

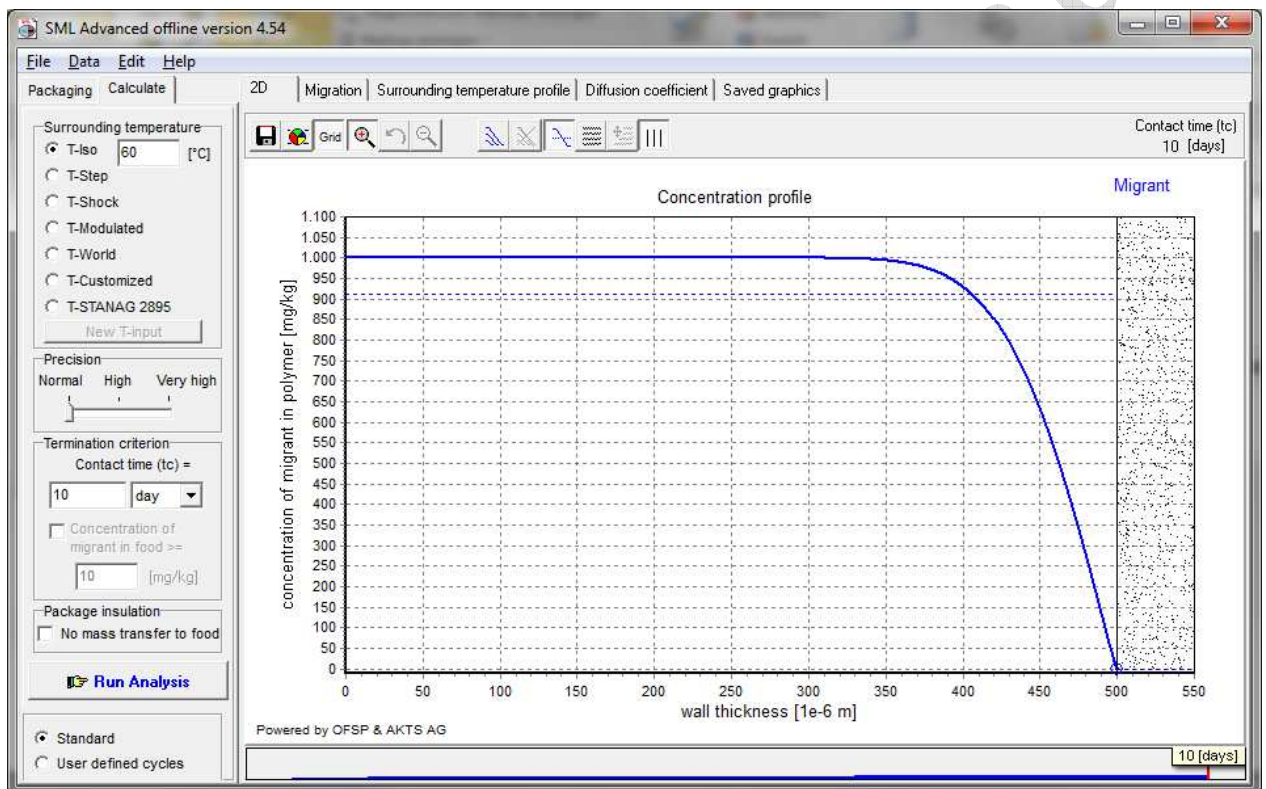
1841 to be used for worst case calculation of specific migration under assumption of total transfer

1842 => 2 x 99% layer thickness = 360 µm

1843 above 360 µm two sides to be considered for calculation of migration if full immersion testing

1844 applied

1845



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1851 **10d @ 40°C**

1852 => 100% layer thickness = 85 µm

1853 no absolute barrier at thicknesses below 85 µm

1854 => 99% layer thickness = 68 µm

1855 => 1/2 x 99% layer thickness = 34 µm

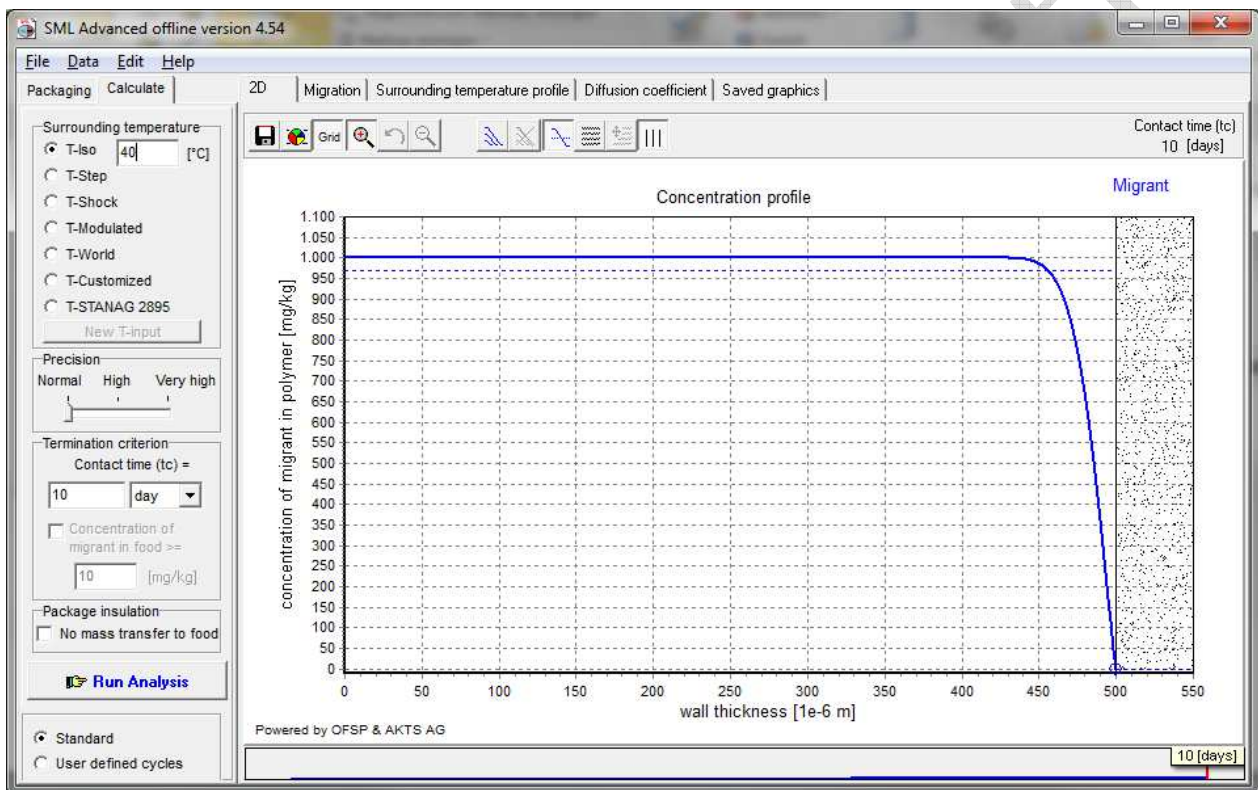
1856 to be used for worst case calculation of specific migration under assumption of total transfer

1857 => 2 x 99% layer thickness = 136 µm

1858 above 136 µm two sides to be considered for calculation of migration if full immersion testing

1859 applied

1860



1861

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1865

1866 **10d @ 20°C**

1867 => 100% layer thickness = 28 µm

1868 no absolute barrier at thicknesses below 28 µm

1869 => 99% layer thickness = 22 µm

1870 => 1/2 x 99% layer thickness = 11 µm

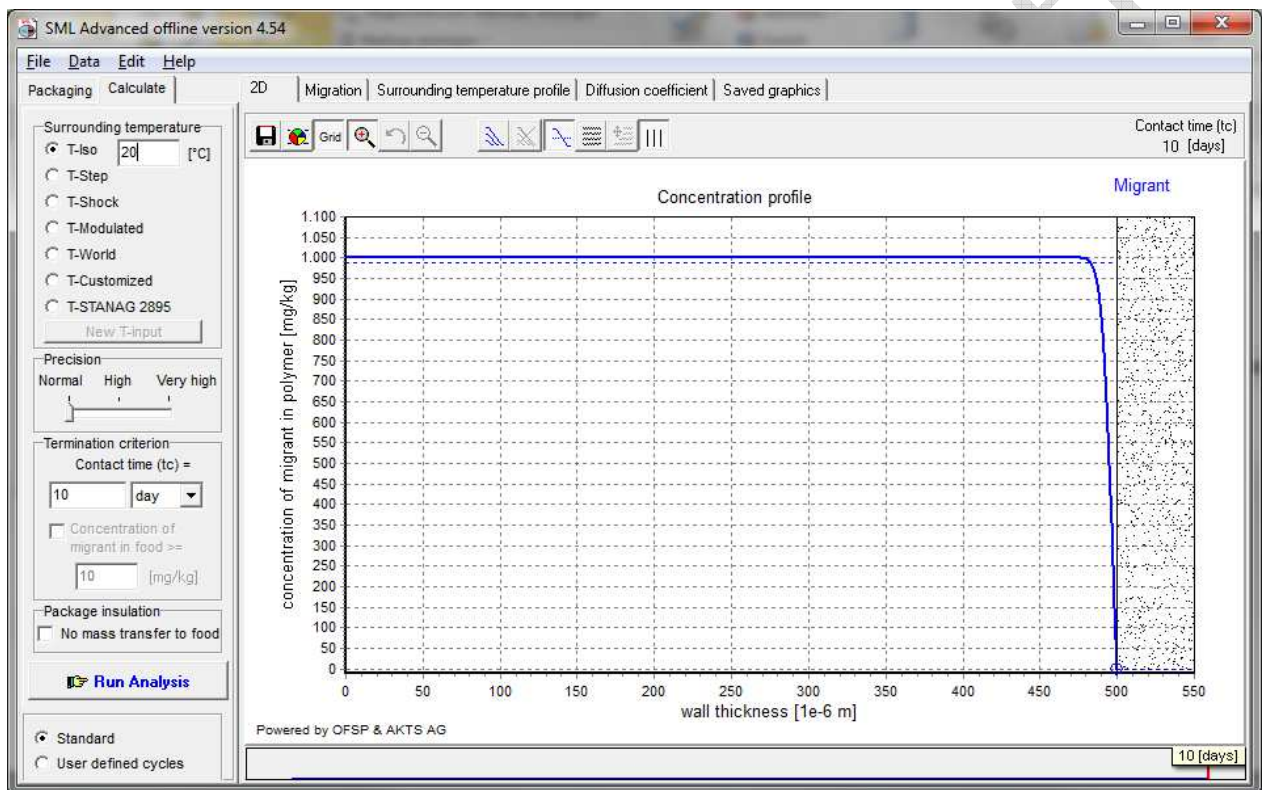
1871 to be used for worst case calculation of specific migration under assumption of total transfer

1872 => 2 x 99% layer thickness = 44 µm

1873 above 44 µm two sides to be considered for calculation of migration if full immersion testing

1874 applied

1875



1876

1877

1878

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1880

1881 **2h @ 100°C**

1882 => 100% layer thickness = 120 µm

1883 no absolute barrier at thicknesses below 120 µm

1884 => 99% layer thickness = 90 µm

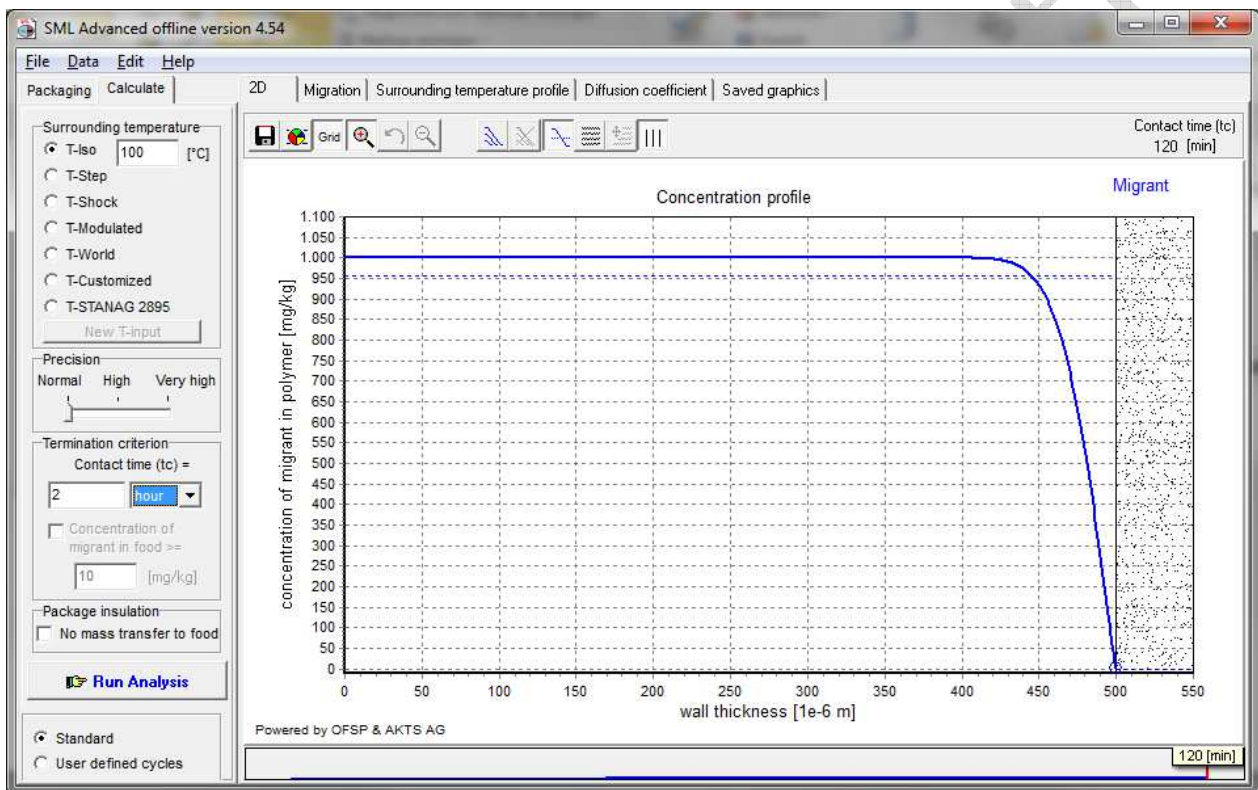
1885 => 1/2 x 99% layer thickness = 45 µm

1886 to be used for worst case calculation of specific migration under assumption of total transfer

1887 => 2 x 99% layer thickness = 180 µm

1888 above 180 µm two sides to be considered for calculation of migration if full immersion testing applied

1890



1891

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1896 ► molecular mass 501 - 750 g/mol

1897 10d @ 60°C

1898 => 100% layer thickness = 70 µm

1899 no absolute barrier at thicknesses below 70 µm

1900 => 99% layer thickness = 54 µm

1901 => 1/2 x 99% layer thickness = 27 µm

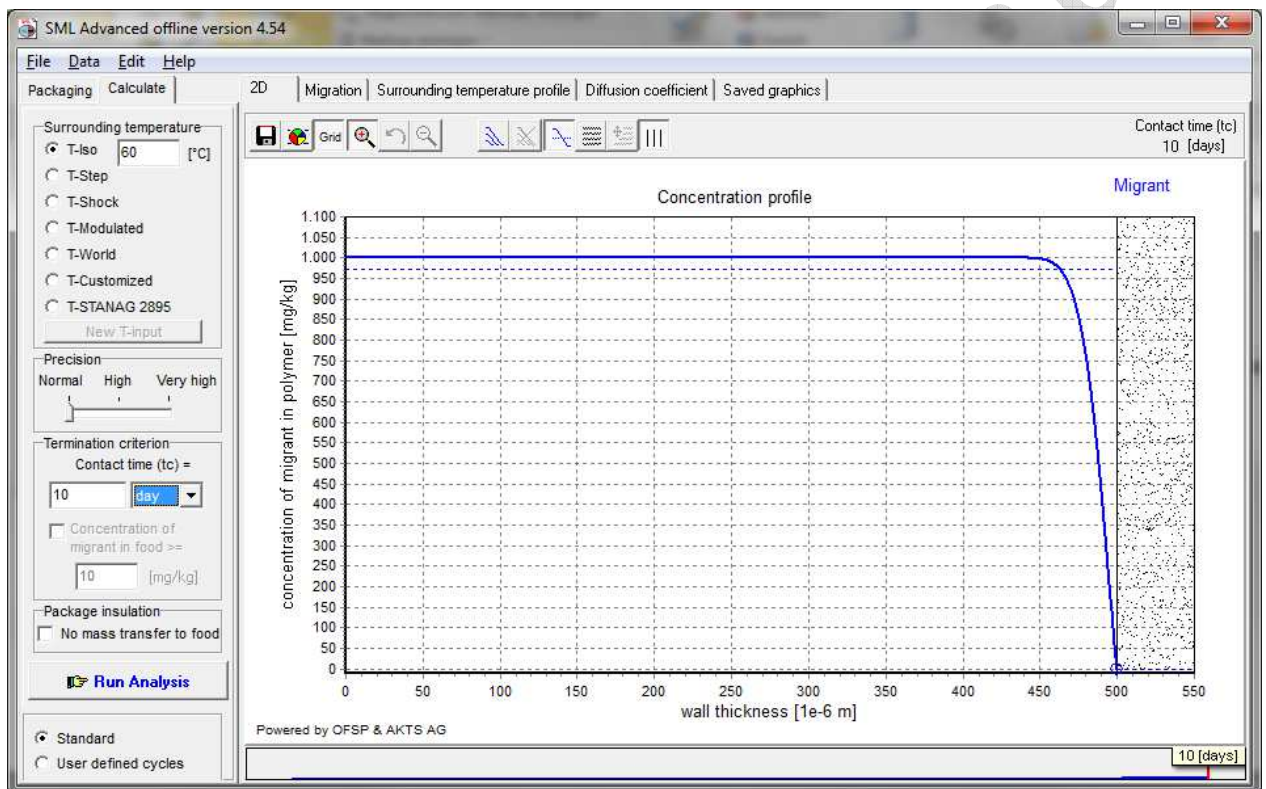
1902 to be used for worst case calculation of specific migration under assumption of total transfer

1903 => 2 x 99% layer thickness = 108 µm

1904 above 108 µm two sides to be considered for calculation of migration if full immersion testing

1905 applied

1906



1907

1908

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1911

1912 **10d @ 40°C**

1913 => 100% layer thickness = 26 µm

1914 no absolute barrier at thicknesses below 26 µm

1915 => 99% layer thickness = 21 µm

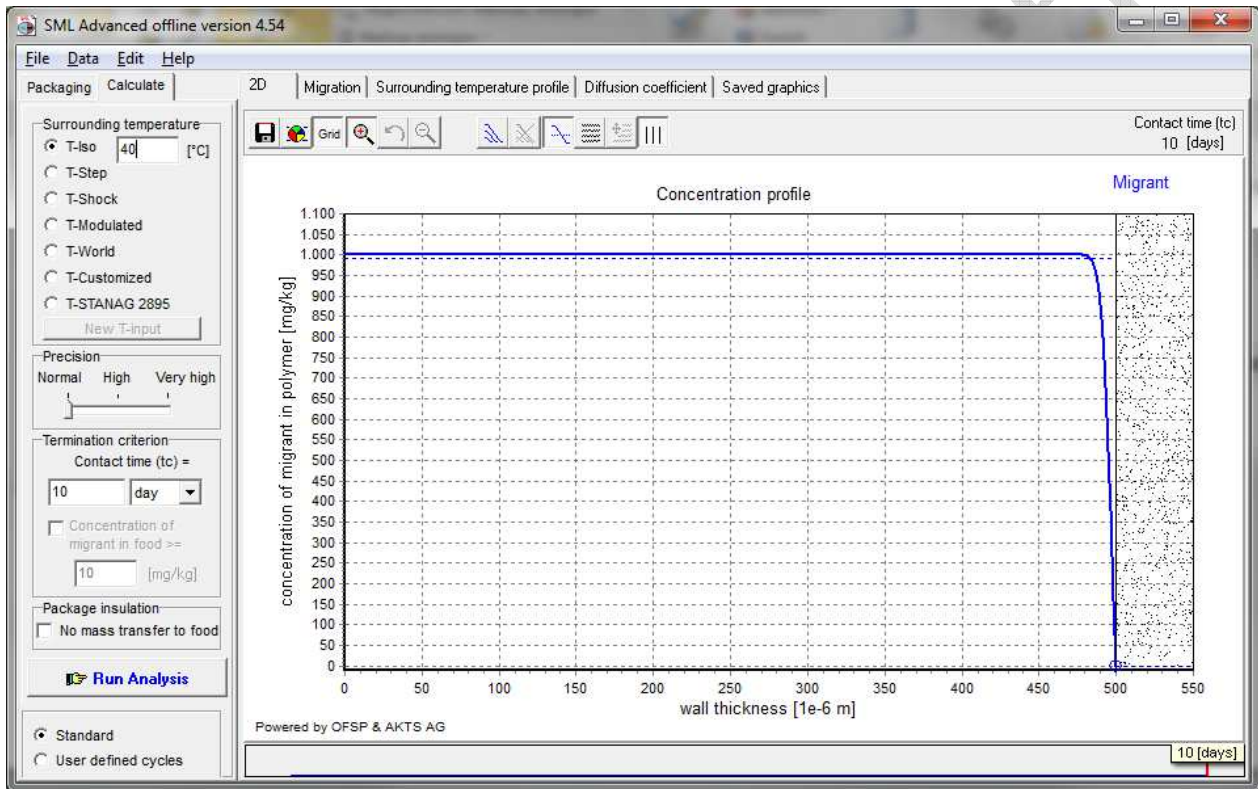
1916 => 1/2 x 99% layer thickness = 10.5 µm

1917 to be used for worst case calculation of specific migration under assumption of total transfer

1918 => 2 x 99% layer thickness = 42 µm

1919 above 42 µm two sides to be considered for calculation of migration if full immersion testing applied

1921



1922

1923

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1927 **10d @ 20°C**

1928 => 100% layer thickness = 10 µm

1929 no absolute barrier at thicknesses below 10 µm

1930 => 99% layer thickness = 7.8 µm

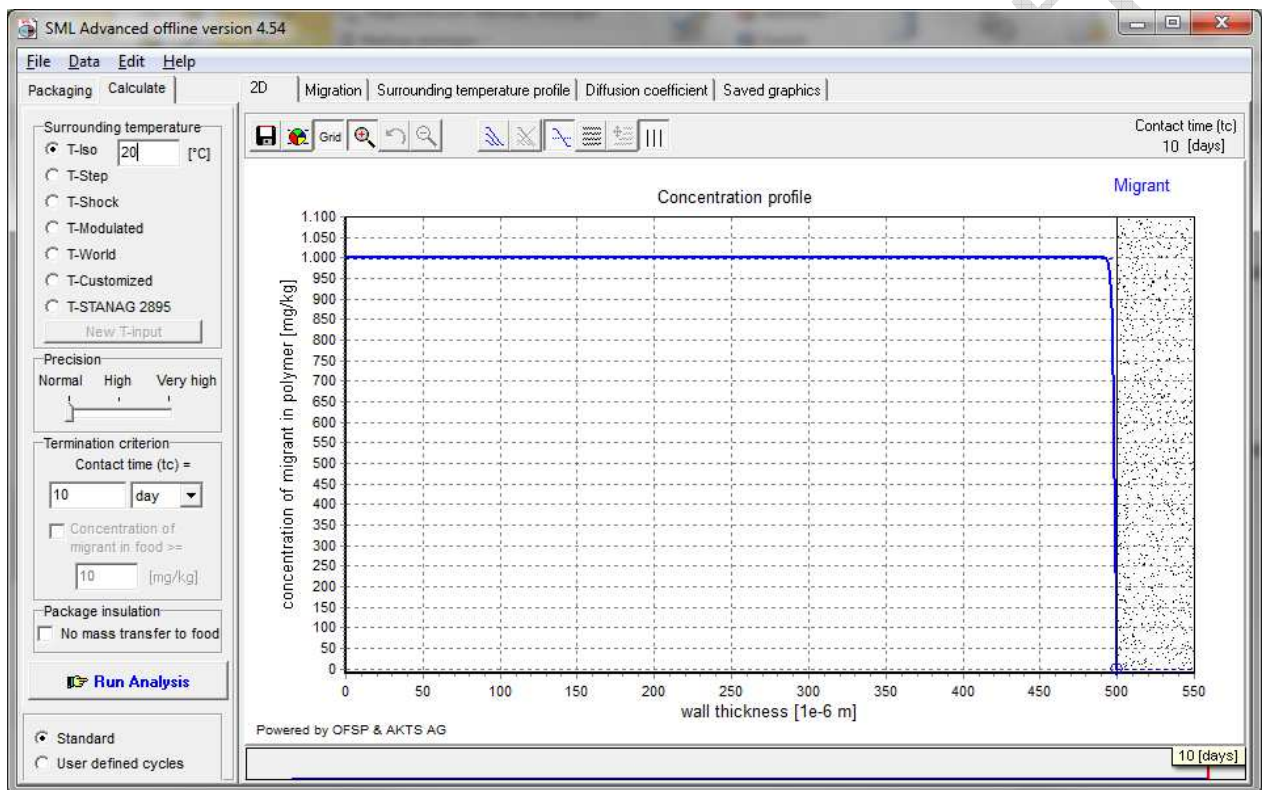
1931 => 1/2 x 99% layer thickness = 3.9 µm

1932 to be used for worst case calculation of specific migration under assumption of total transfer

1933 => 2 x 99% layer thickness = 15.6 µm

1934 above 15.6 µm two sides to be considered for calculation of migration if full immersion testing applied

1936



1937

1938

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1941

1942 **2h @ 100°C**

1943 => 100% layer thickness = 36 µm

1944 no absolute barrier at thicknesses below 36 µm

1945 => 99% layer thickness = 28 µm

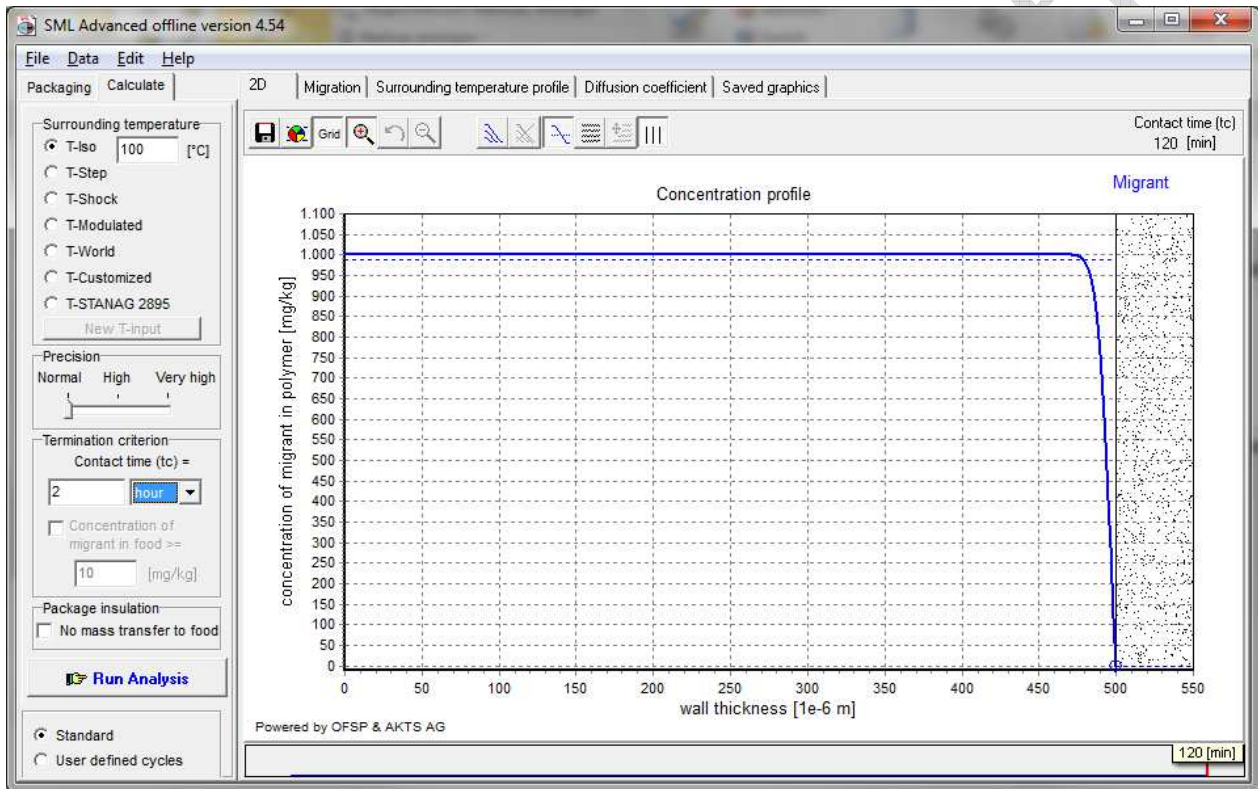
1946 => 1/2 x 99% layer thickness = 14 µm

1947 to be used for worst case calculation of specific migration under assumption of total transfer

1948 => 2 x 99% layer thickness = 56 µm

1949 above 56 µm two sides to be considered for calculation of migration if full immersion testing applied

1951



1952

1953

1954

1955

1956

1957 ► molecular mass 751 - 1000 g/mol

1958 10d @ 60°C

1959 => 100% layer thickness = 28 µm

1960 no absolute barrier at thicknesses below 28 µm

1961 => 99% layer thickness = 22 µm

1962 => 1/2 x 99% layer thickness = 11 µm

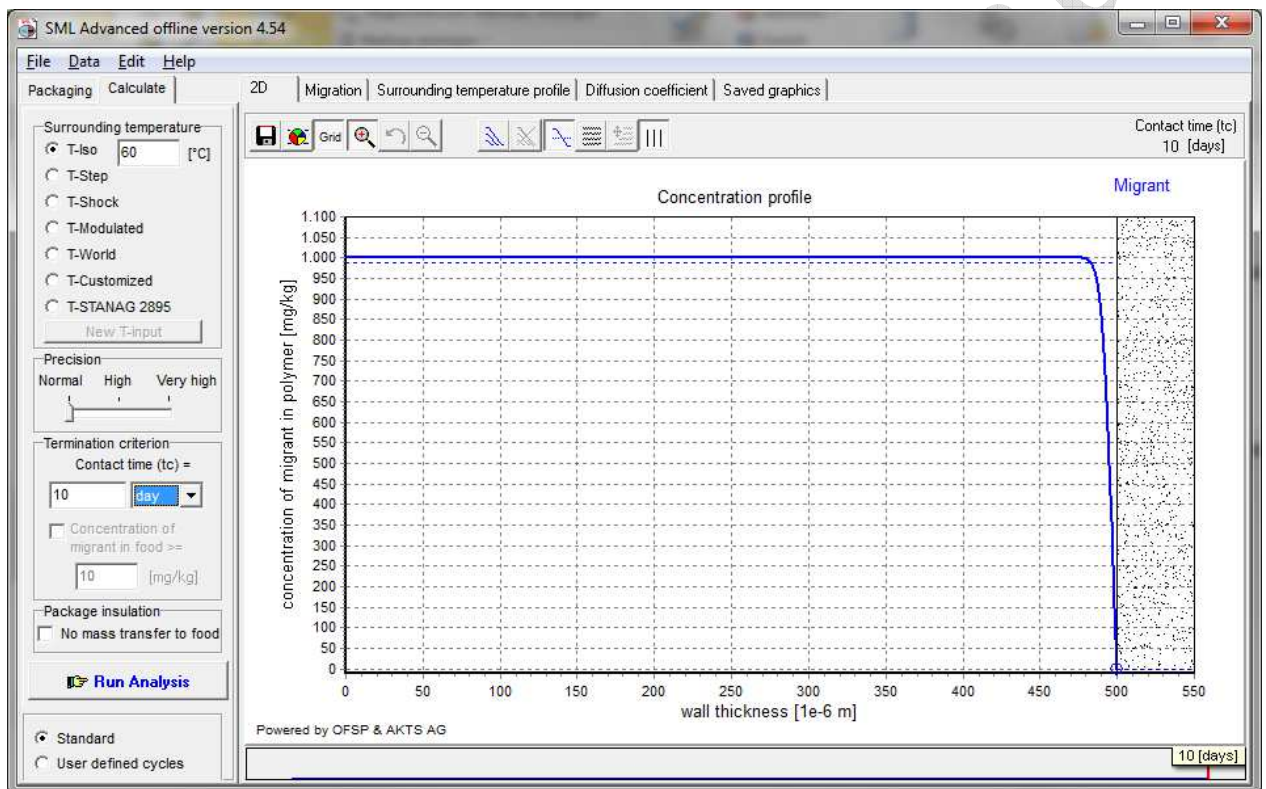
1963 to be used for worst case calculation of specific migration under assumption of total transfer

1964 => 2 x 99% layer thickness = 44 µm

1965 above 44 µm two sides to be considered for calculation of migration if full immersion testing

1966 applied

1967



1968

1969

1970

1971

1972

1973 **10d @ 40°C**

1974 => 100% layer thickness = 13 µm

1975 no absolute barrier at thicknesses below 13 µm

1976 => 99% layer thickness = 9.2 µm

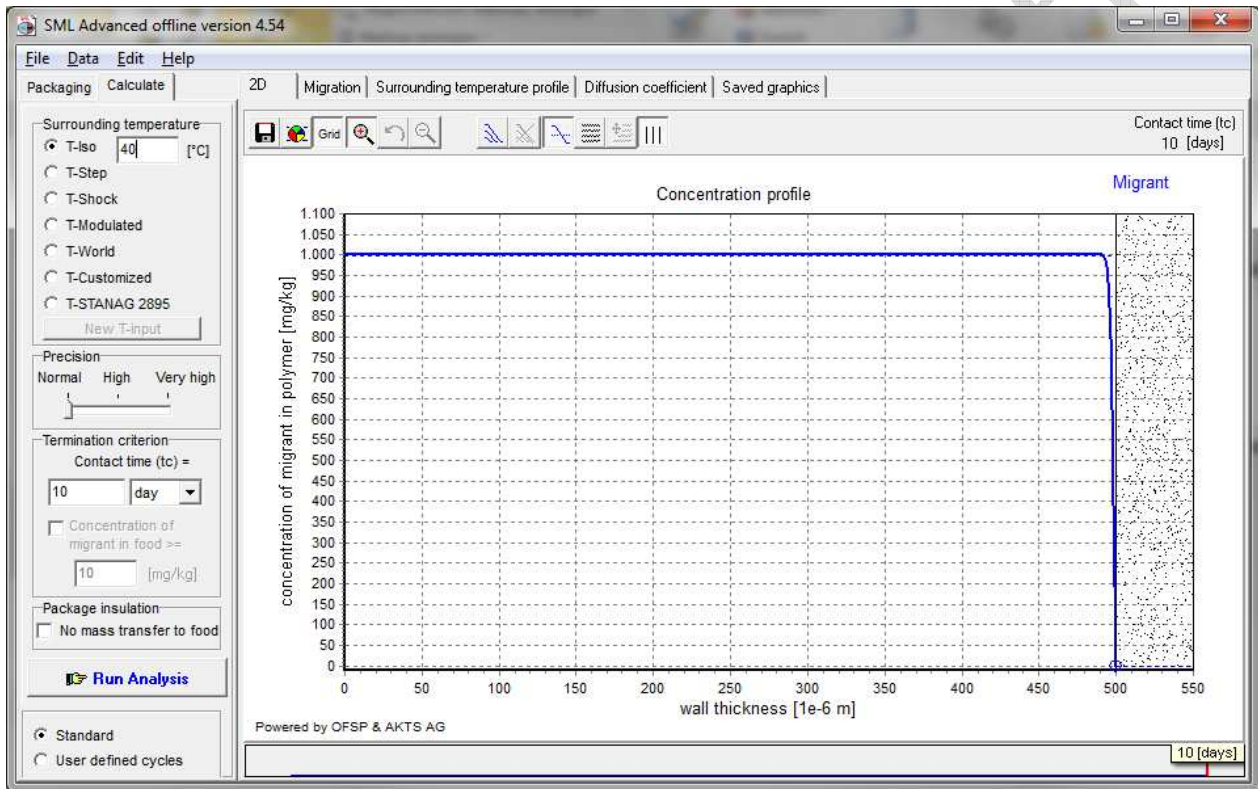
1977 => 1/2 x 99% layer thickness = 4.6 µm

1978 to be used for worst case calculation of specific migration under assumption of total transfer

1979 => 2 x 99% layer thickness = 18.4 µm

1980 above 18.4 µm two sides to be considered for calculation of migration if full immersion testing applied

1982



1983

1984

1985

1986

1987

1988 **10d @ 20°C**

1989 => 100% layer thickness = 4.4 μm

1990 no absolute barrier at thicknesses below 4.4 μm

1991 => 99% layer thickness = 3.2 μm

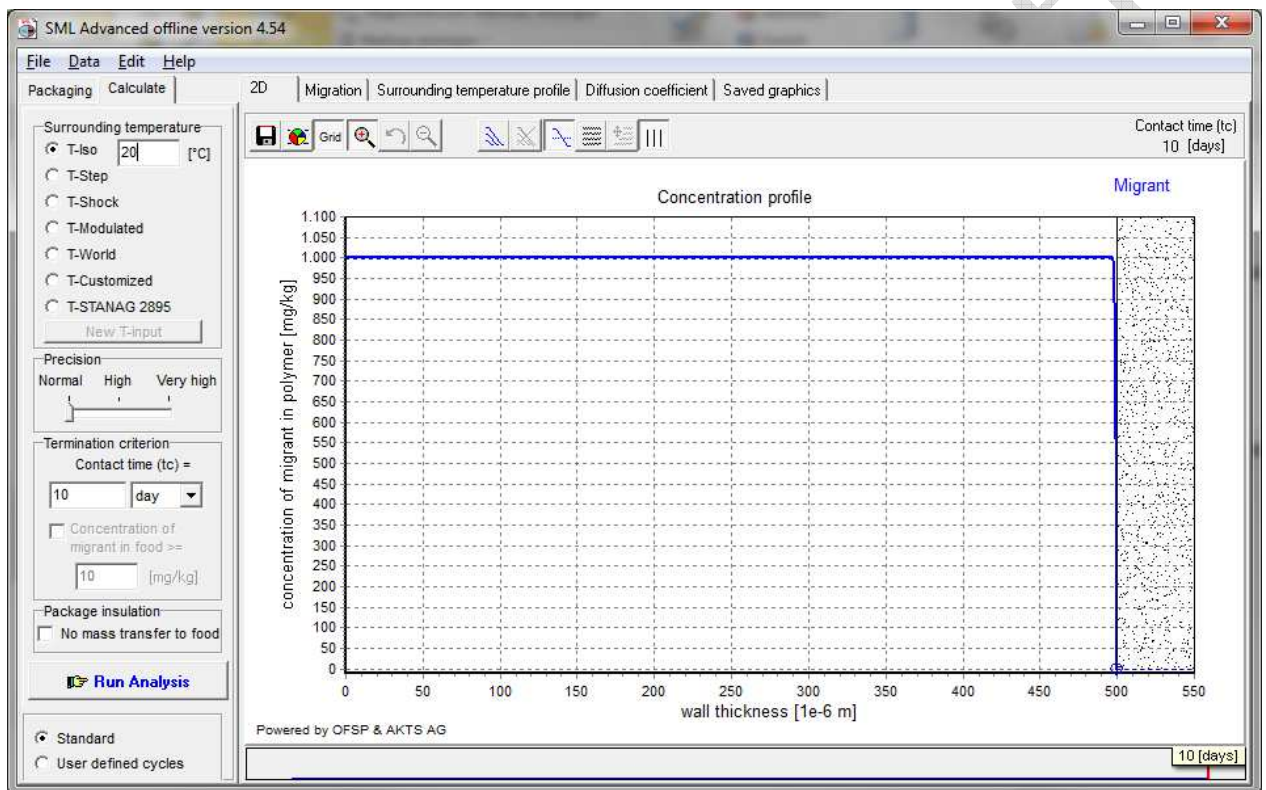
1992 => 1/2 x 99% layer thickness = 1.6 μm

1993 to be used for worst case calculation of specific migration under assumption of total transfer

1994 => 2 x 99% layer thickness = 6.4 μm

1995 above 6.4 μm two sides to be considered for calculation of migration if full immersion testing applied

1997



1998

1999

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2001

2002

2003 **2h @ 100°C**

2004 => 100% layer thickness = 15.6 μm

2005 no absolute barrier at thicknesses below 15.6 μm

2006 => 99% layer thickness = 11.6 μm

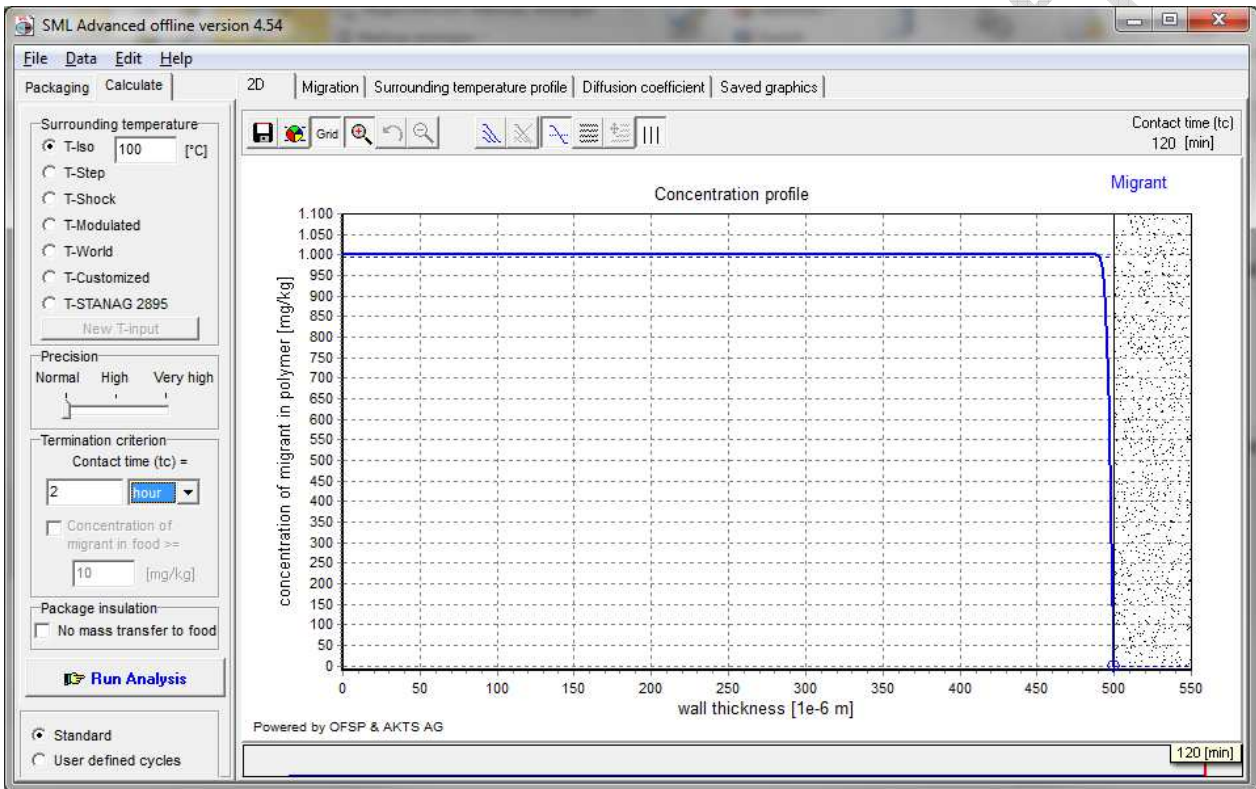
2007 => 1/2 x 99% layer thickness = 5.8 μm

2008 to be used for worst case calculation of specific migration under assumption of total transfer

2009 => 2 x 99% layer thickness = 23.2 μm

2010 above 23.2 μm two sides to be considered for calculation of migration if full immersion testing applied

2012



2013

2014

2015

2016

2017

2018 **PA12**

2019 *(not swollen: e.g. contact with simulant D2, iso-octane; or any simulant not in direct*
2020 *contact, e.g. plastic multilayer)*

2021 ► **molecular mass 100 - 250 g/mol**

2022 **10d @ 60°C**

2023 => 100% layer thickness = 810 µm

2024 no absolute barrier at thicknesses below 810 µm

2025 => 99% layer thickness = 660 µm

2026 => 1/2 x 99% layer thickness = 330 µm

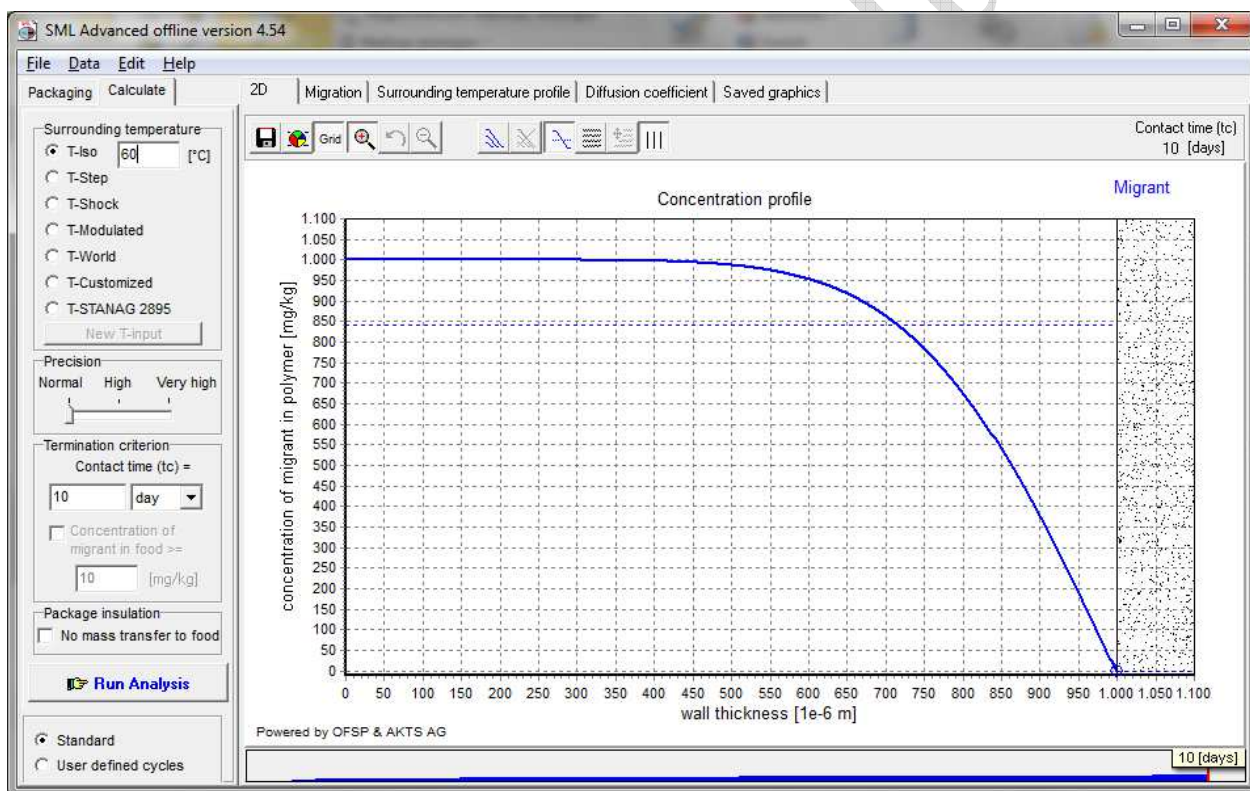
2027 to be used for worst case calculation of specific migration under assumption of total transfer

2028 => 2 x 99% layer thickness = 1320 µm

2029 above 1320 µm two sides to be considered for calculation of migration if full immersion

2030 testing applied

2031



2032

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2037 **10d @ 40°C**

2038 => 100% layer thickness = 420 µm
2039 no absolute barrier at thicknesses below 420 µm

2040 => 99% layer thickness = 250 µm

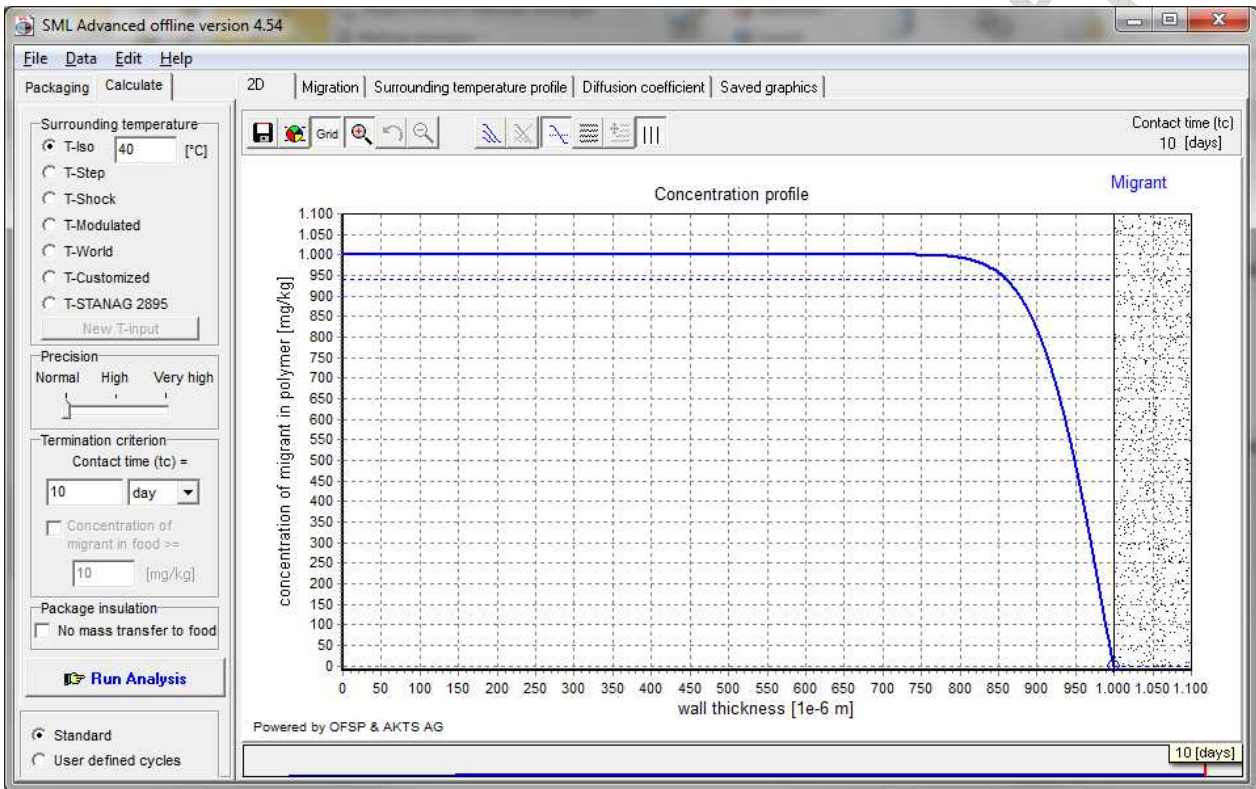
2041 => 1/2 x 99% layer thickness = 125 µm

2042 to be used for worst case calculation of specific migration under assumption of total transfer

2043 => 2 x 99% layer thickness = 500 µm

2044 above 500 µm two sides to be considered for calculation of migration if full immersion testing
2045 applied

2046



2047

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2052 **10d @ 20°C**

2053 => 100% layer thickness = 100 µm

2054 no absolute barrier at thicknesses below 100 µm

2055 => 99% layer thickness = 80 µm

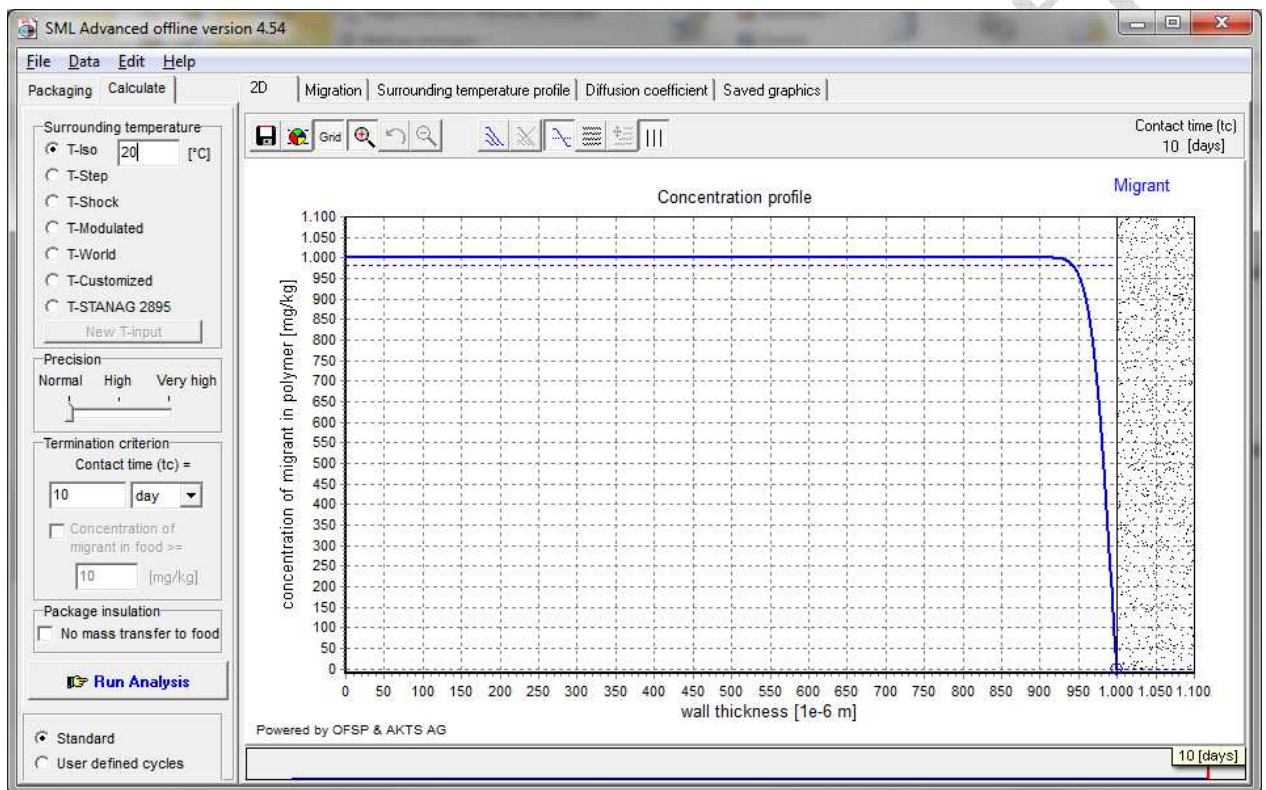
2056 => 1/2 x 99% layer thickness = 40 µm

2057 to be used for worst case calculation of specific migration under assumption of total transfer

2058 => 2 x 99% layer thickness = 160 µm

2059 above 160 µm two sides to be considered for calculation of migration if full immersion testing applied

2061



2062

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2067 **2h @ 100°C**

2068 => 100% layer thickness = 400 µm

2069 no absolute barrier at thicknesses below 400 µm

2070 => 99% layer thickness = 330 µm

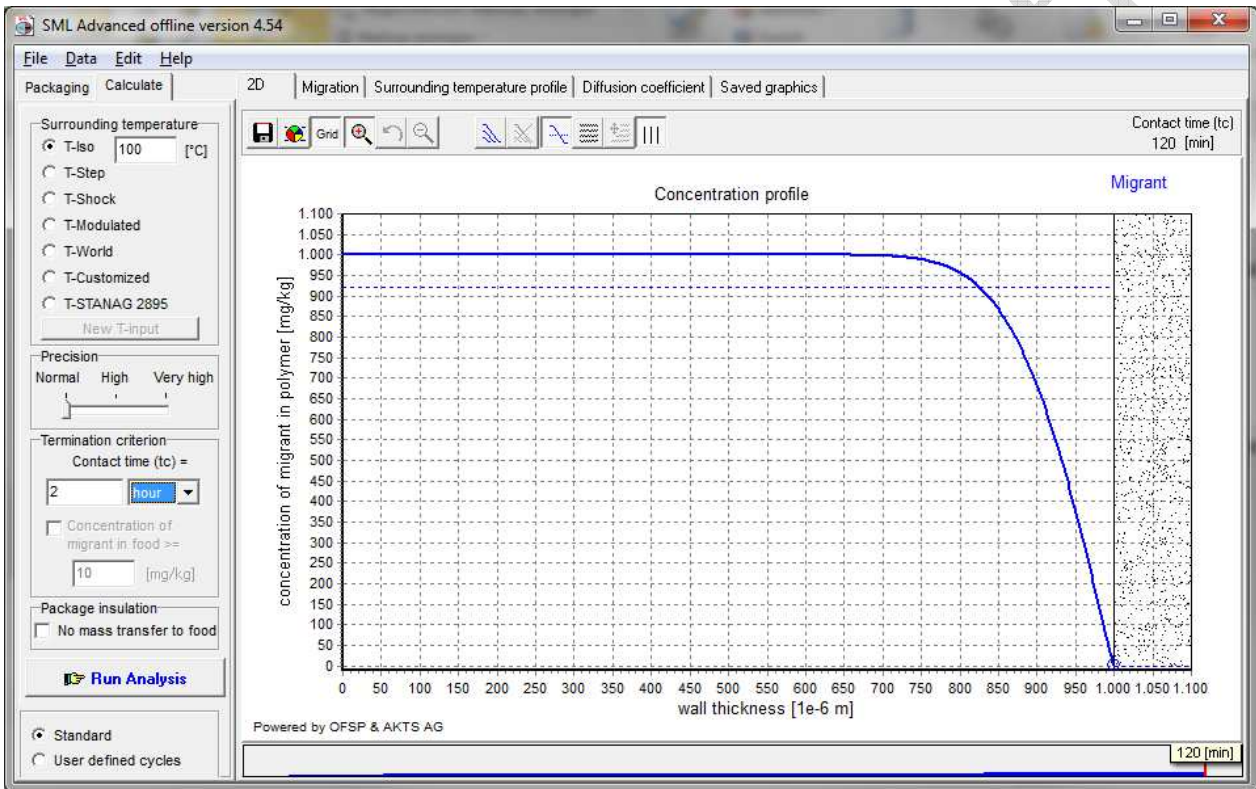
2071 => 1/2 x 99% layer thickness = 165 µm

2072 to be used for worst case calculation of specific migration under assumption of total transfer

2073 => 2 x 99% layer thickness = 660 µm

2074 above 660 µm two sides to be considered for calculation of migration if full immersion testing applied

2076



2077

2078

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2081

2082 ► molecular mass 251 - 500 g/mol

2083 10d @ 60°C

2084 => 100% layer thickness = 300 µm

2085 no absolute barrier at thicknesses below 300 µm

2086 => 99% layer thickness = 250 µm

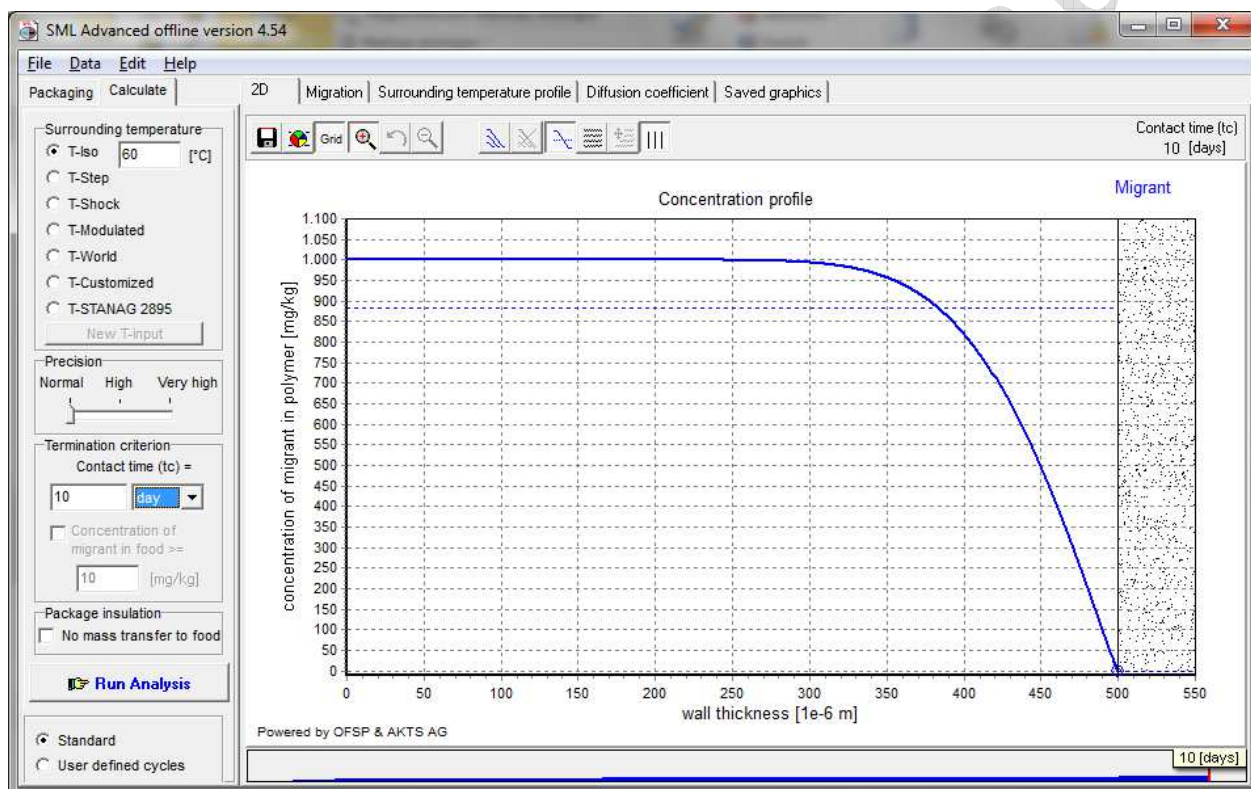
2087 => 1/2 x 99% layer thickness = 125 µm

2088 to be used for worst case calculation of specific migration under assumption of total transfer

2089 => 2 x 99% layer thickness = 500 µm

2090 above 500 µm two sides to be considered for calculation of migration if full immersion testing
2091 applied

2092



2093

2094

2095

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2097

2098 **10d @ 40°C**

2099 => 100% layer thickness = 114 µm

2100 no absolute barrier at thicknesses below 114 µm

2101 => 99% layer thickness = 90 µm

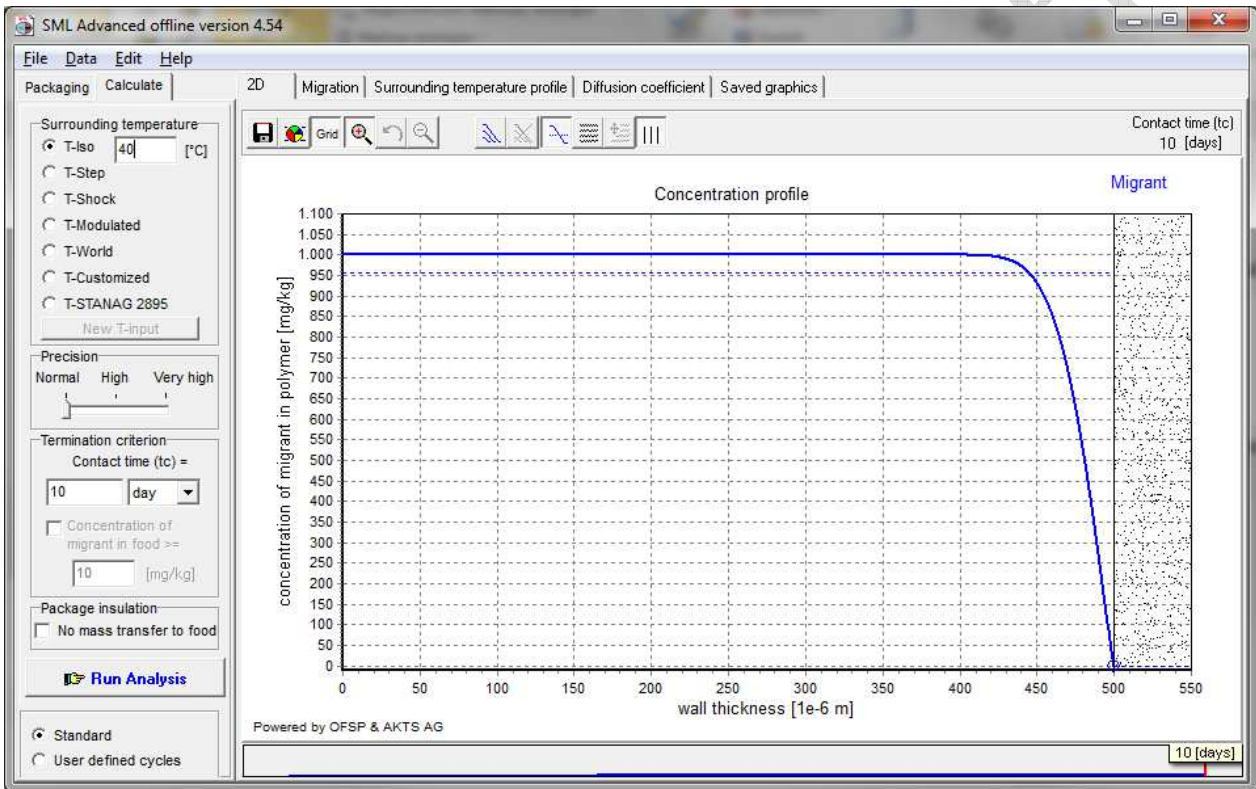
2102 => 1/2 x 99% layer thickness = 45 µm

2103 to be used for worst case calculation of specific migration under assumption of total transfer

2104 => 2 x 99% layer thickness = 180 µm

2105 above 180 µm two sides to be considered for calculation of migration if full immersion testing
2106 applied

2107



2108

2109

2110

2111

2112

2113 **10d @ 20°C**

2114 => 100% layer thickness = 44 µm

2115 no absolute barrier at thicknesses below 44 µm

2116 => 99% layer thickness = 30 µm

2117 => 1/2 x 99% layer thickness = 15 µm

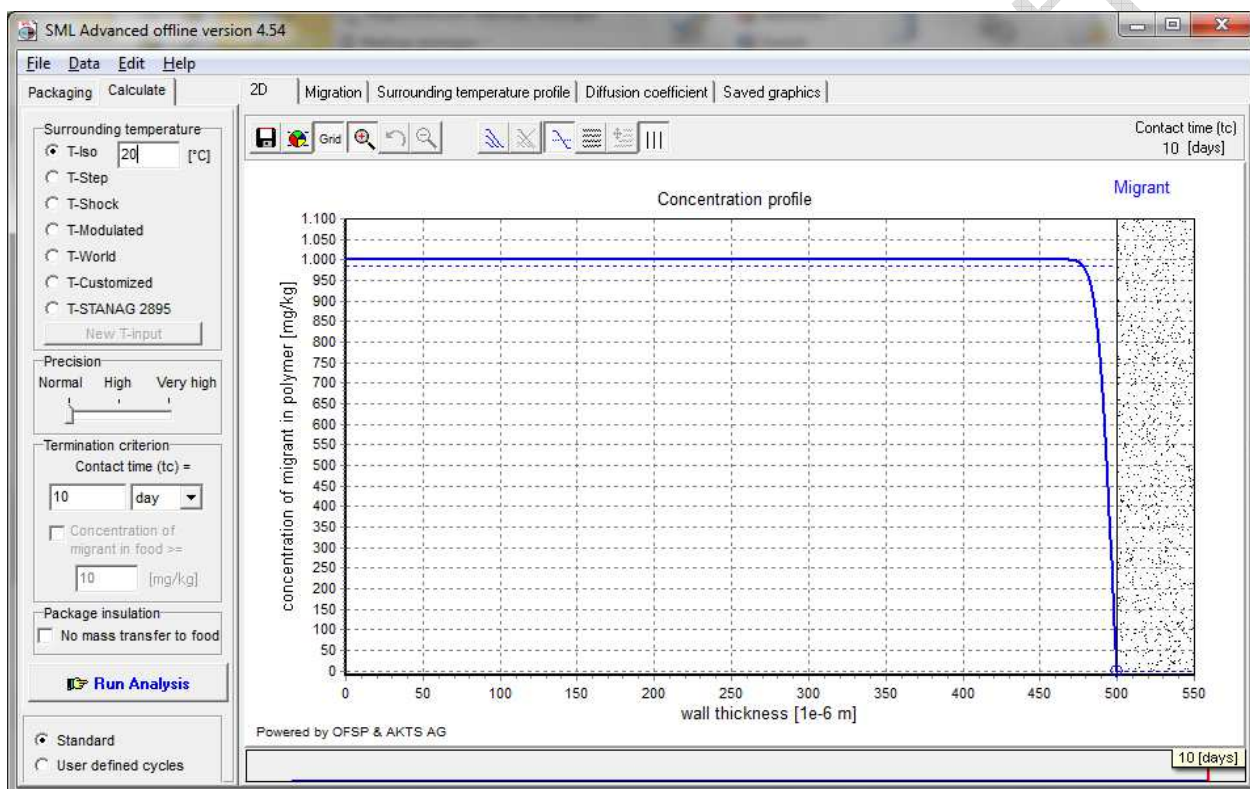
2118 to be used for worst case calculation of specific migration under assumption of total transfer

2119 => 2 x 99% layer thickness = 60 µm

2120 above 60 µm two sides to be considered for calculation of migration if full immersion testing

2121 applied

2122



2123

2124

2125

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2127

2128 **2h @ 100°C**

2129 => 100% layer thickness = 147 µm

2130 no absolute barrier at thicknesses below 147 µm

2131 => 99% layer thickness = 120 µm

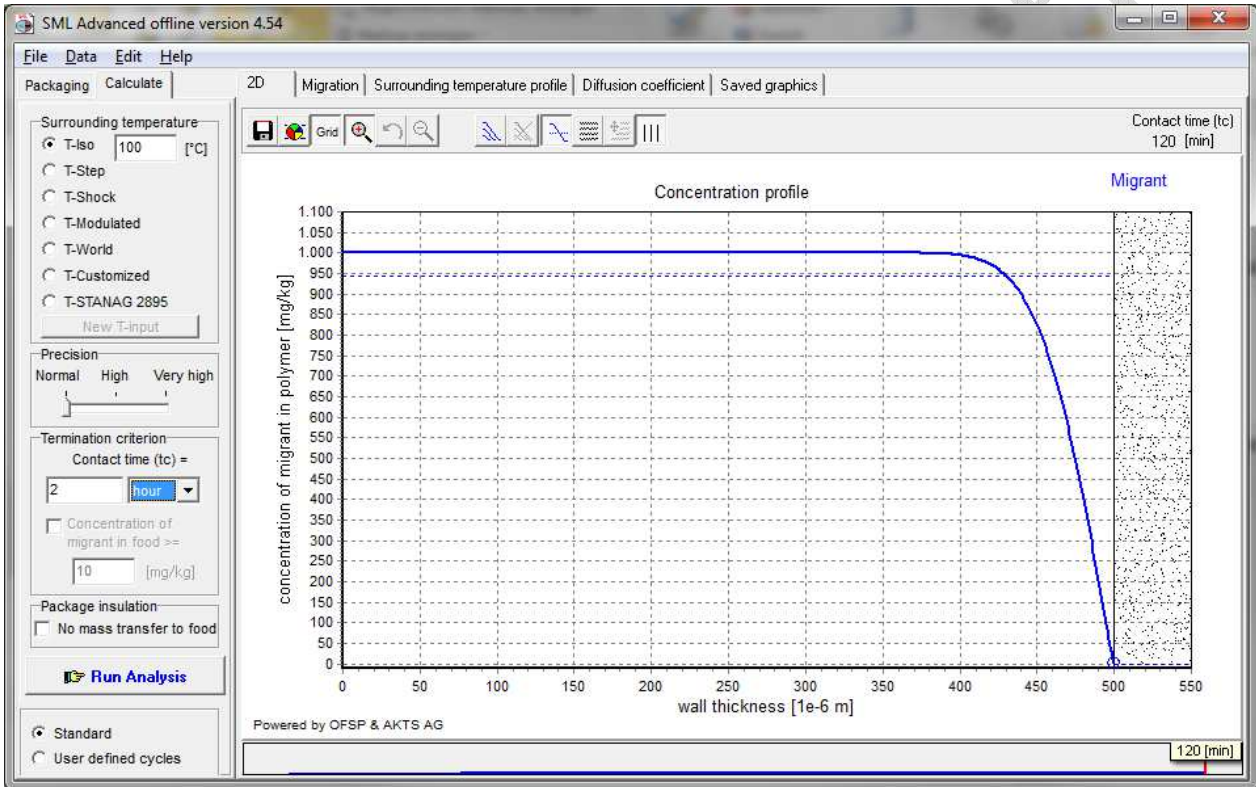
2132 => 1/2 x 99% layer thickness = 60 µm

2133 to be used for worst case calculation of specific migration under assumption of total transfer

2134 => 2 x 99% layer thickness = 240 µm

2135 above 240 µm two sides to be considered for calculation of migration if full immersion testing applied

2137



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2143 ► molecular mass 501 - 750 g/mol

2144 10d @ 60°C

2145 => 100% layer thickness = 91 µm

2146 no absolute barrier at thicknesses below 91 µm

2147 => 99% layer thickness = 74 µm

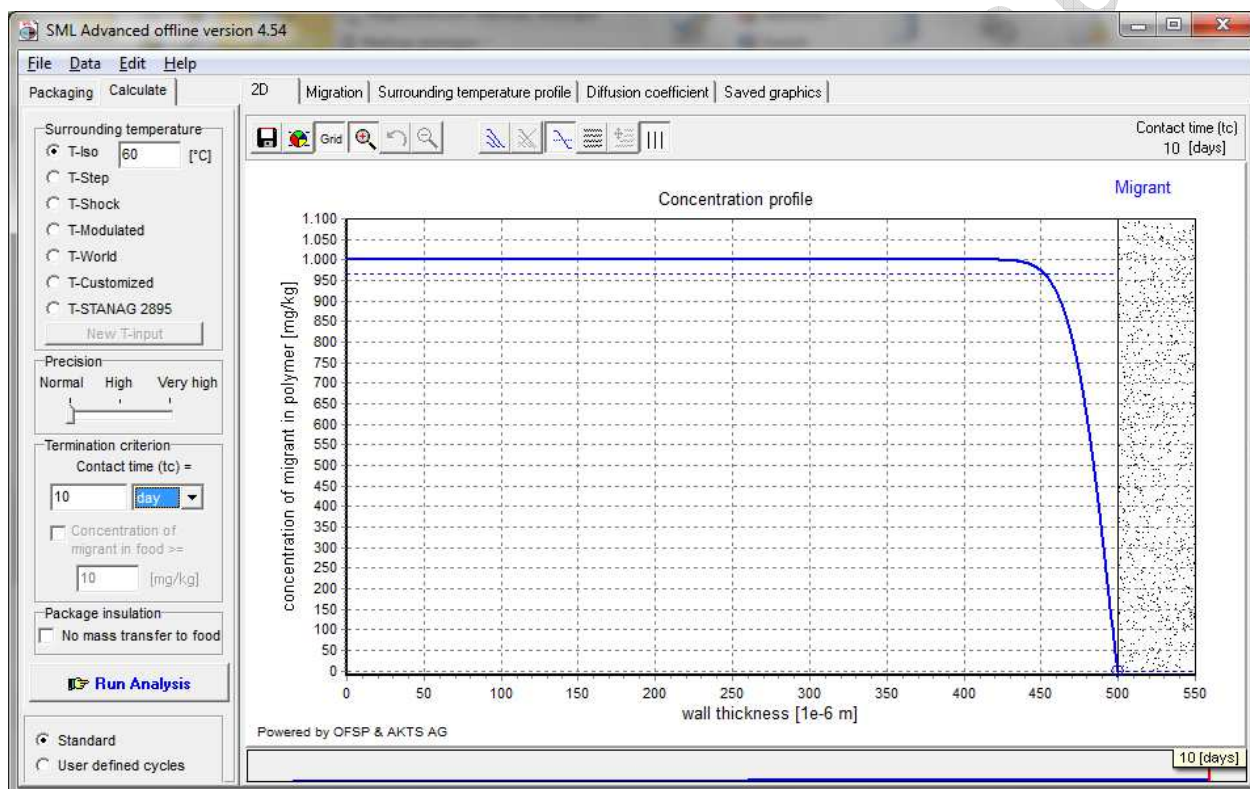
2148 => 1/2 x 99% layer thickness = 37 µm

2149 to be used for worst case calculation of specific migration under assumption of total transfer

2150 => 2 x 99% layer thickness = 148 µm

2151 above 148 µm two sides to be considered for calculation of migration if full immersion testing
2152 applied

2153



2154

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2159 **10d @ 40°C**

2160 => 100% layer thickness = 34 µm

2161 no absolute barrier at thicknesses below 34 µm

2162 => 99% layer thickness = 28 µm

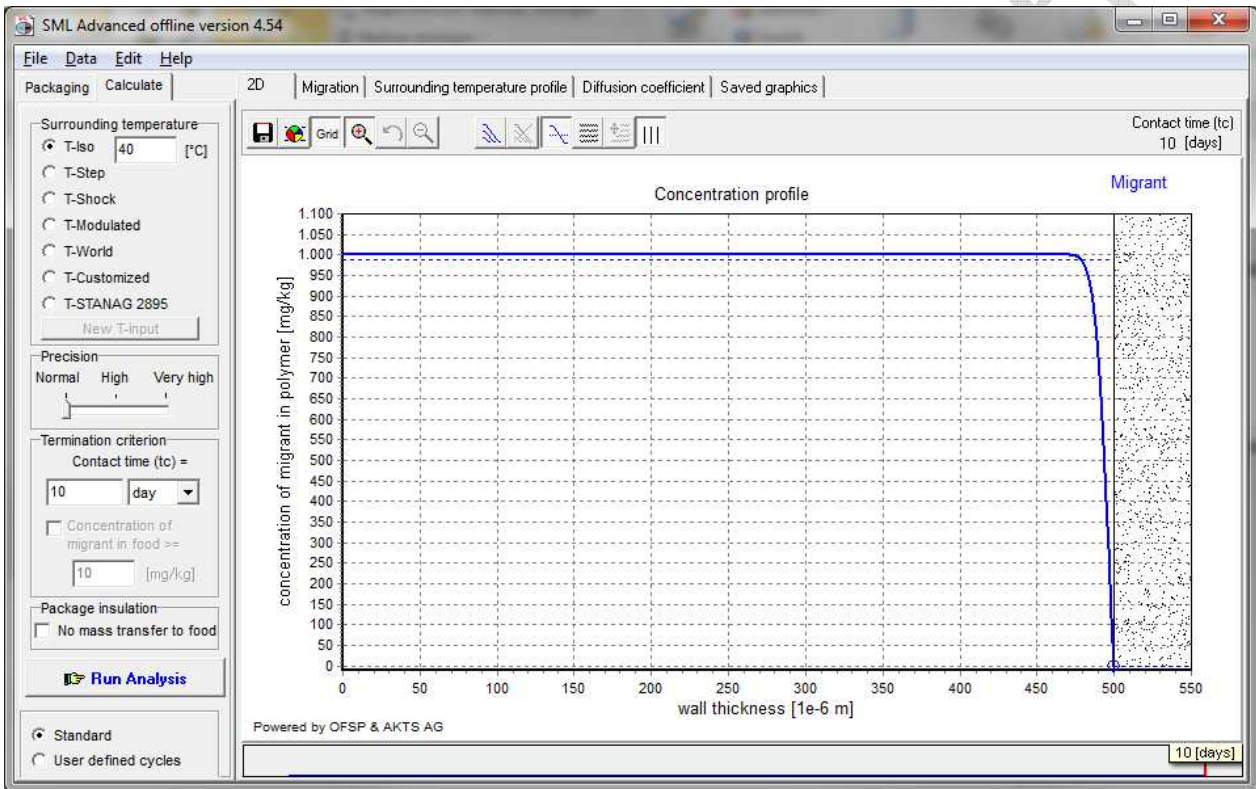
2163 => 1/2 x 99% layer thickness = 14 µm

2164 to be used for worst case calculation of specific migration under assumption of total transfer

2165 => 2 x 99% layer thickness = 56 µm

2166 above 56 µm two sides to be considered for calculation of migration if full immersion testing applied

2168



2169

2170

2171

2172

2173

2174 **10d @ 20°C**

2175 => 100% layer thickness = 13 µm

2176 no absolute barrier at thicknesses below 13 µm

2177 => 99% layer thickness = 10 µm

2178 => 1/2 x 99% layer thickness = 5 µm

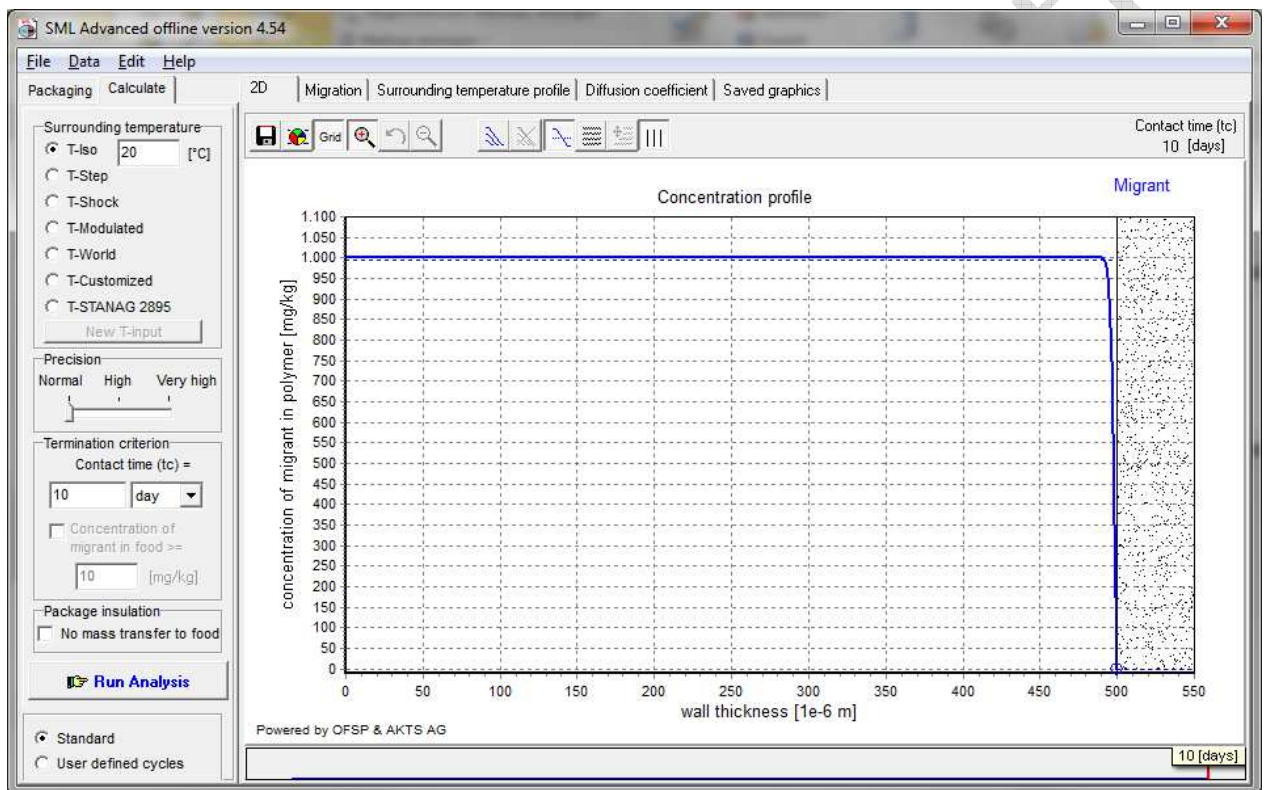
2179 to be used for worst case calculation of specific migration under assumption of total transfer

2180 => 2 x 99% layer thickness = 20 µm

2181 above 20 µm two sides to be considered for calculation of migration if full immersion testing

2182 applied

2183



2184

2185

2186

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2188

2189 **2h @ 100°C**

2190 => 100% layer thickness = 46 µm

2191 no absolute barrier at thicknesses below 46 µm

2192 => 99% layer thickness = 37 µm

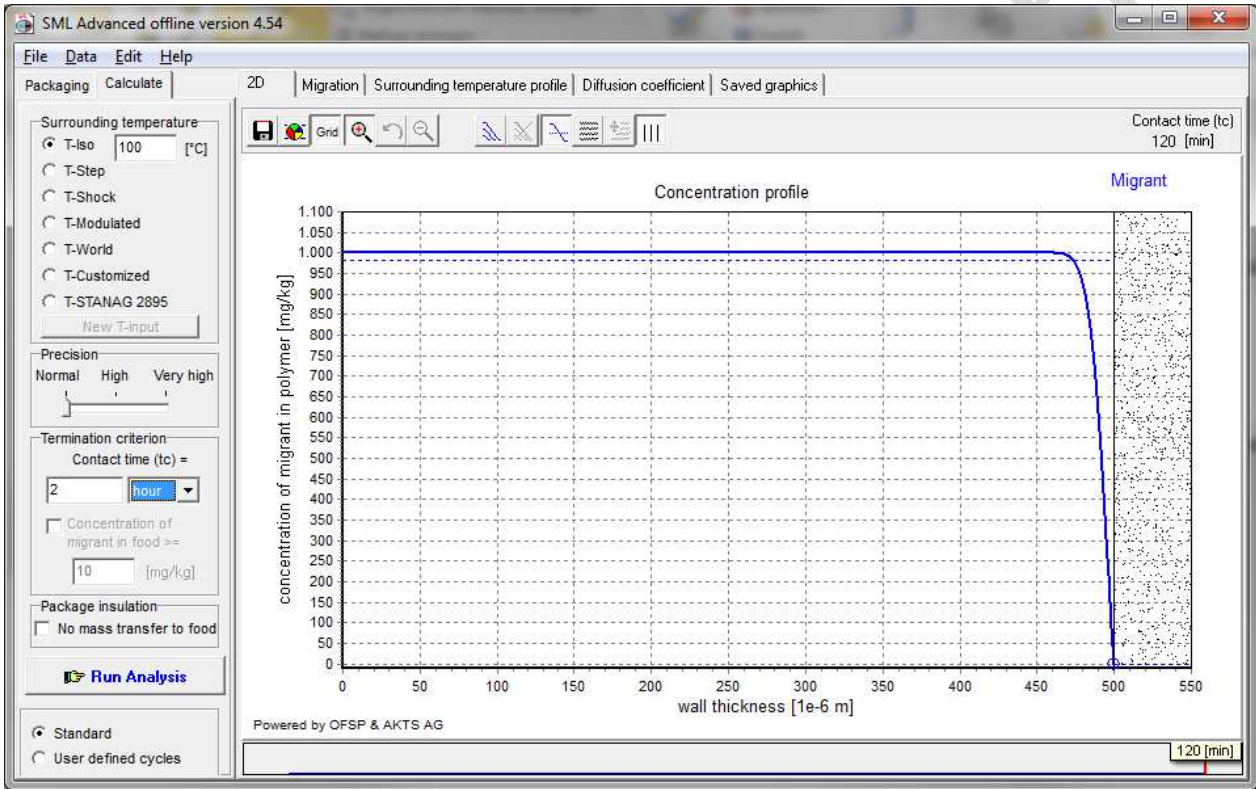
2193 => 1/2 x 99% layer thickness = 18.5 µm

2194 to be used for worst case calculation of specific migration under assumption of total transfer

2195 => 2 x 99% layer thickness = 74 µm

2196 above 74 µm two sides to be considered for calculation of migration if full immersion testing applied

2198



2199

2200

2201

2202

2203

2204 ► molecular mass 751 - 1000 g/mol

2205 10d @ 60°C

2206 => 100% layer thickness = 37 µm

2207 no absolute barrier at thicknesses below 28 µm

2208 => 99% layer thickness = 30 µm

2209 => 1/2 x 99% layer thickness = 15 µm

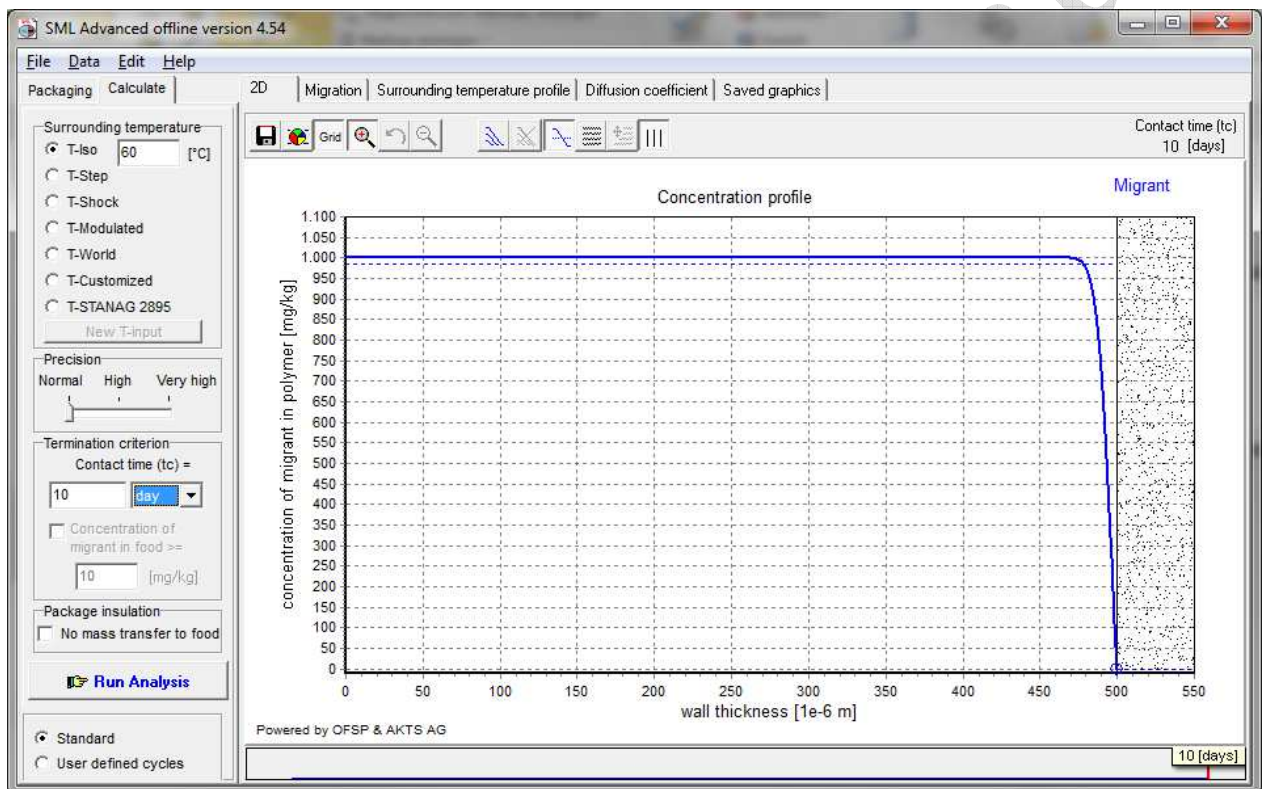
2210 to be used for worst case calculation of specific migration under assumption of total transfer

2211 => 2 x 99% layer thickness = 60 µm

2212 above 60 µm two sides to be considered for calculation of migration if full immersion testing

2213 applied

2214



2215

2216

2217

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2219

2220 **10d @ 40°C**

2221 => 100% layer thickness = 15 µm

2222 no absolute barrier at thicknesses below 15 µm

2223 => 99% layer thickness = 11.6 µm

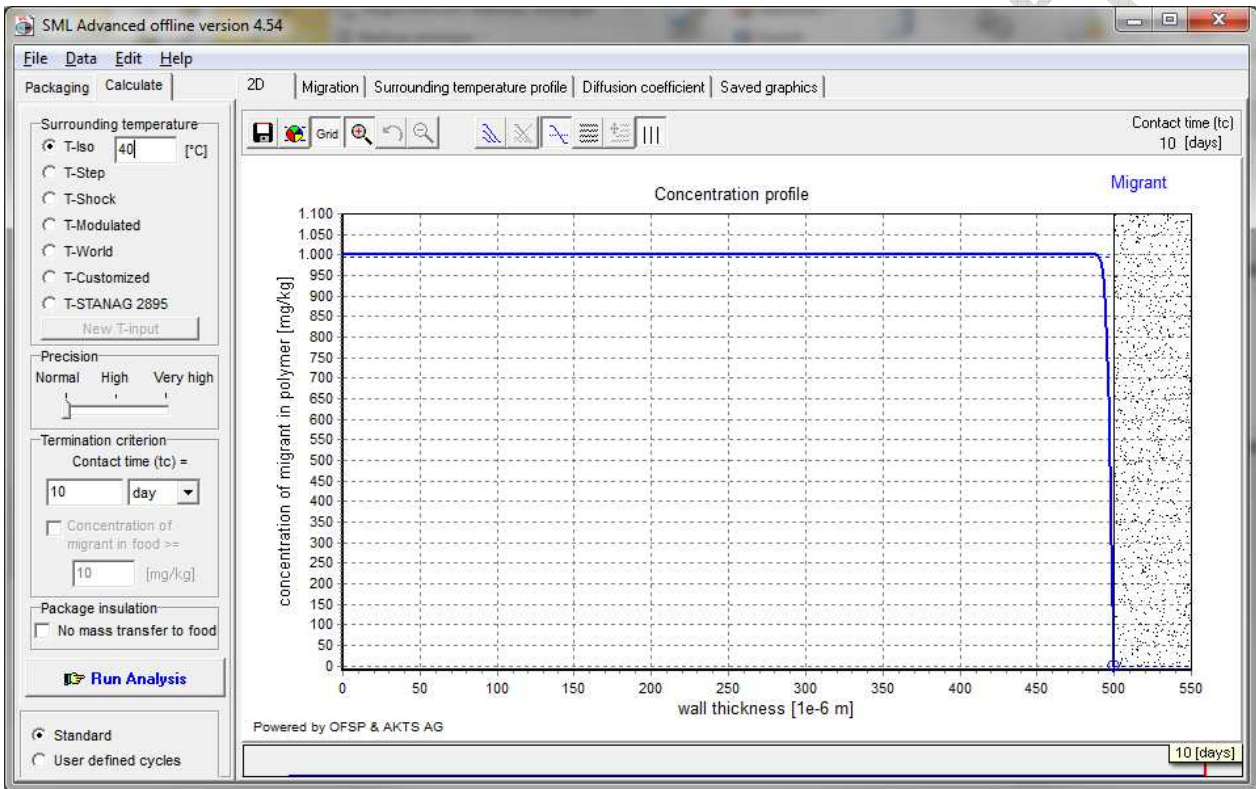
2224 => 1/2 x 99% layer thickness = 4.6 µm

2225 to be used for worst case calculation of specific migration under assumption of total transfer

2226 => 2 x 99% layer thickness = 18.4 µm

2227 above 18.4 µm two sides to be considered for calculation of migration if full immersion testing applied

2229



2230

2231

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2234

2235 **10d @ 20°C**

2236 => 100% layer thickness = 6 µm

2237 no absolute barrier at thicknesses below 6 µm

2238 => 99% layer thickness = 4.4 µm

2239 => 1/2 x 99% layer thickness = 2.2 µm

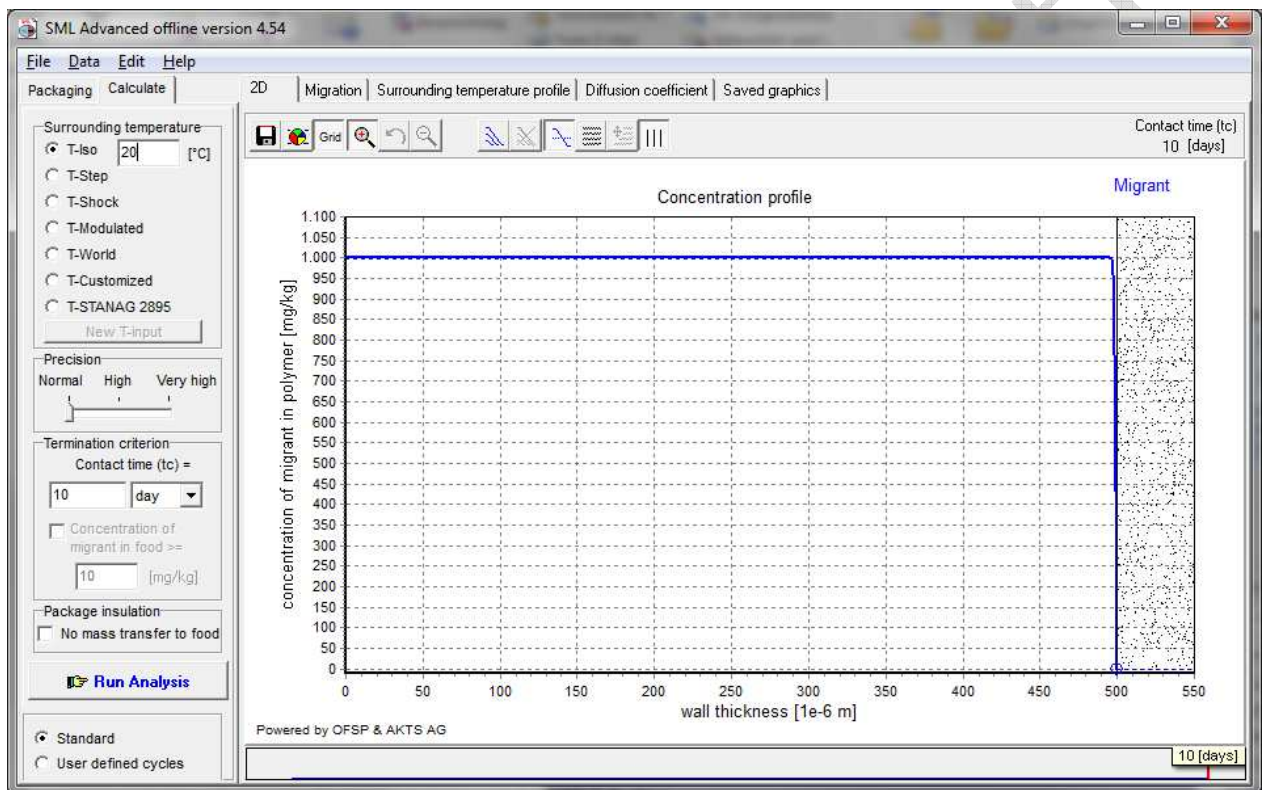
2240 to be used for worst case calculation of specific migration under assumption of total transfer

2241 => 2 x 99% layer thickness = 8.8 µm

2242 above 8.8 µm two sides to be considered for calculation of migration if full immersion testing

2243 applied

2244



2245

2246

2247

2248

2249

2250 **2h @ 100°C**

2251 => 100% layer thickness = 19 µm

2252 no absolute barrier at thicknesses below 19 µm

2253 => 99% layer thickness = 15 µm

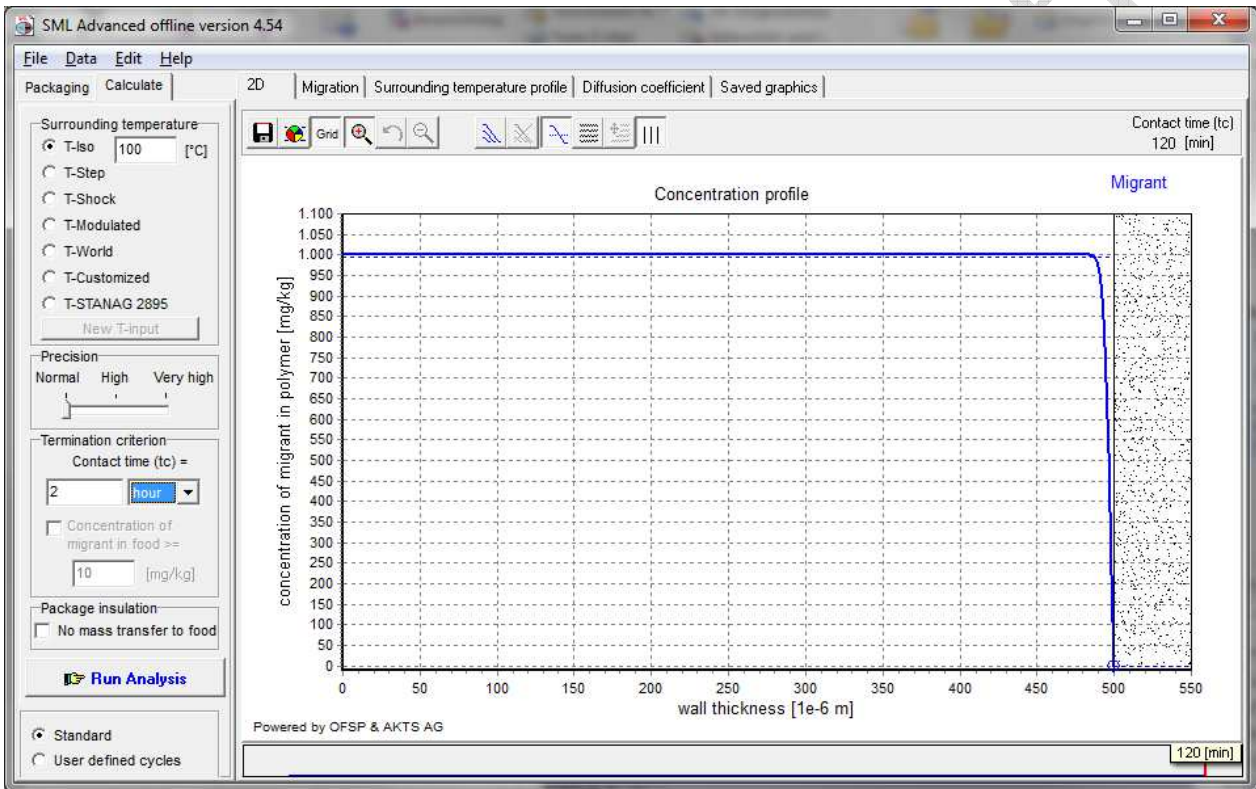
2254 => 1/2 x 99% layer thickness = 7.5 µm

2255 to be used for worst case calculation of specific migration under assumption of total transfer

2256 => 2 x 99% layer thickness = 30 µm

2257 above 30 µm two sides to be considered for calculation of migration if full immersion testing
2258 applied

2259



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2265 **PVC, rigid**

2266 **(not plasticized; not swollen: e.g. contact with simulant D2, iso-octane)**

2267 **► molecular mass 100 - 250 g/mol**

2268 **10d @ 60°C**

2269 => 100% layer thickness = 127

2270 no absolute barrier at thicknesses below 127 µm

2271 => 99% layer thickness = 110 µm

2272 => 1/2 x 99% layer thickness = 55 µm

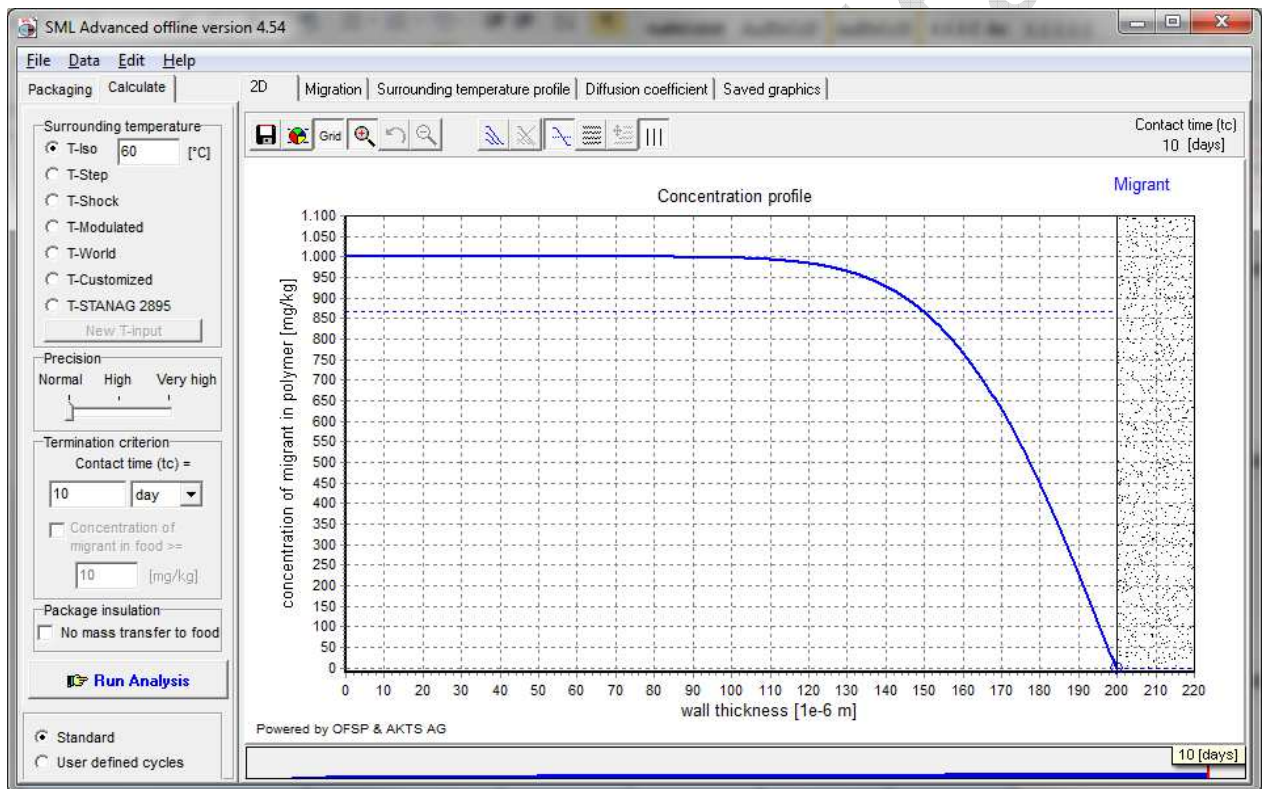
2273 to be used for worst case calculation of specific migration under assumption of total transfer

2274 => 2 x 99% layer thickness = 220 µm

2275 above 220 µm two sides to be considered for calculation of migration if full immersion testing

2276 applied

2277



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2283 **10d @ 40°C**

2284 => 100% layer thickness = 46

2285 no absolute barrier at thicknesses below 46 µm

2286 => 99% layer thickness = 40 µm

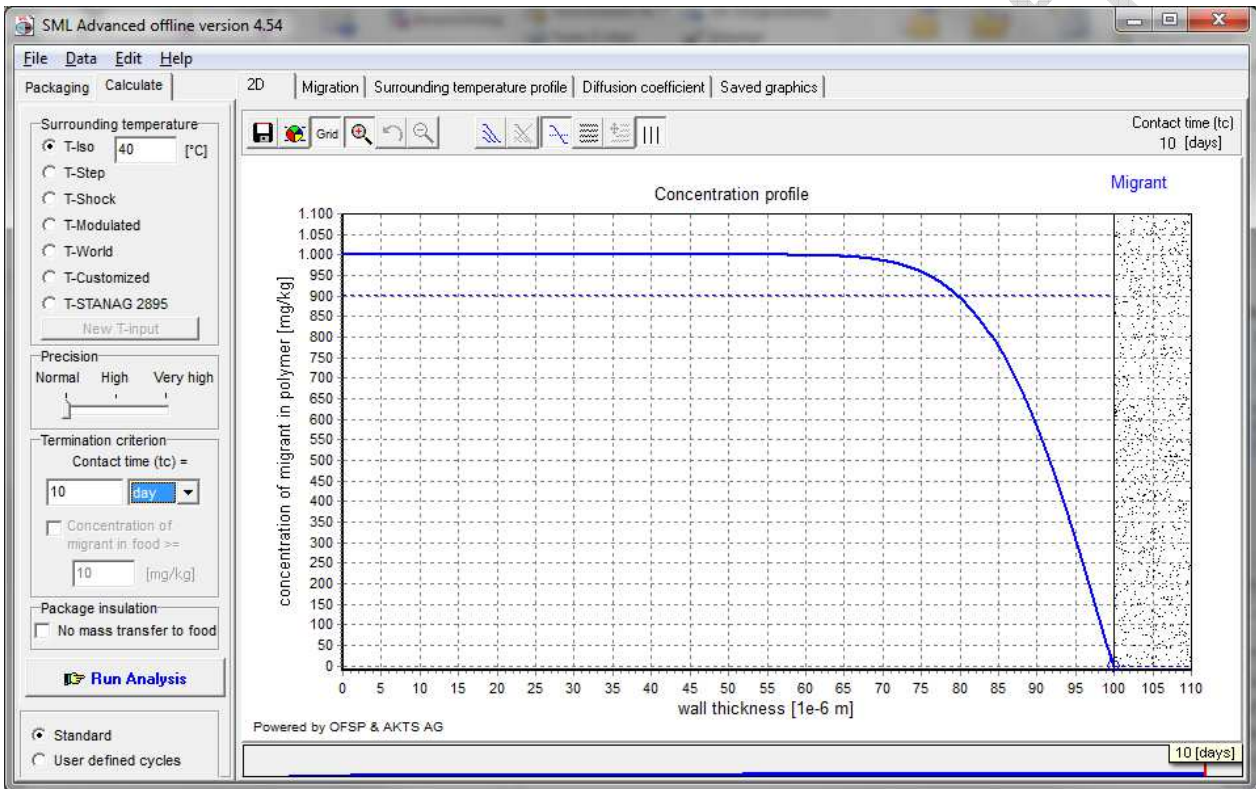
2287 => 1/2 x 99% layer thickness = 20 µm

2288 to be used for worst case calculation of specific migration under assumption of total transfer

2289 => 2 x 99% layer thickness = 80

2290 above 80 µm two sides to be considered for calculation of migration if full immersion testing applied

2292



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2298 **10d @ 20°C**

2299 => 100% layer thickness = 17 µm

2300 no absolute barrier at thicknesses below 17 µm

2301 => 99% layer thickness = 13 µm

2302 => 1/2 x 99% layer thickness = 7 µm

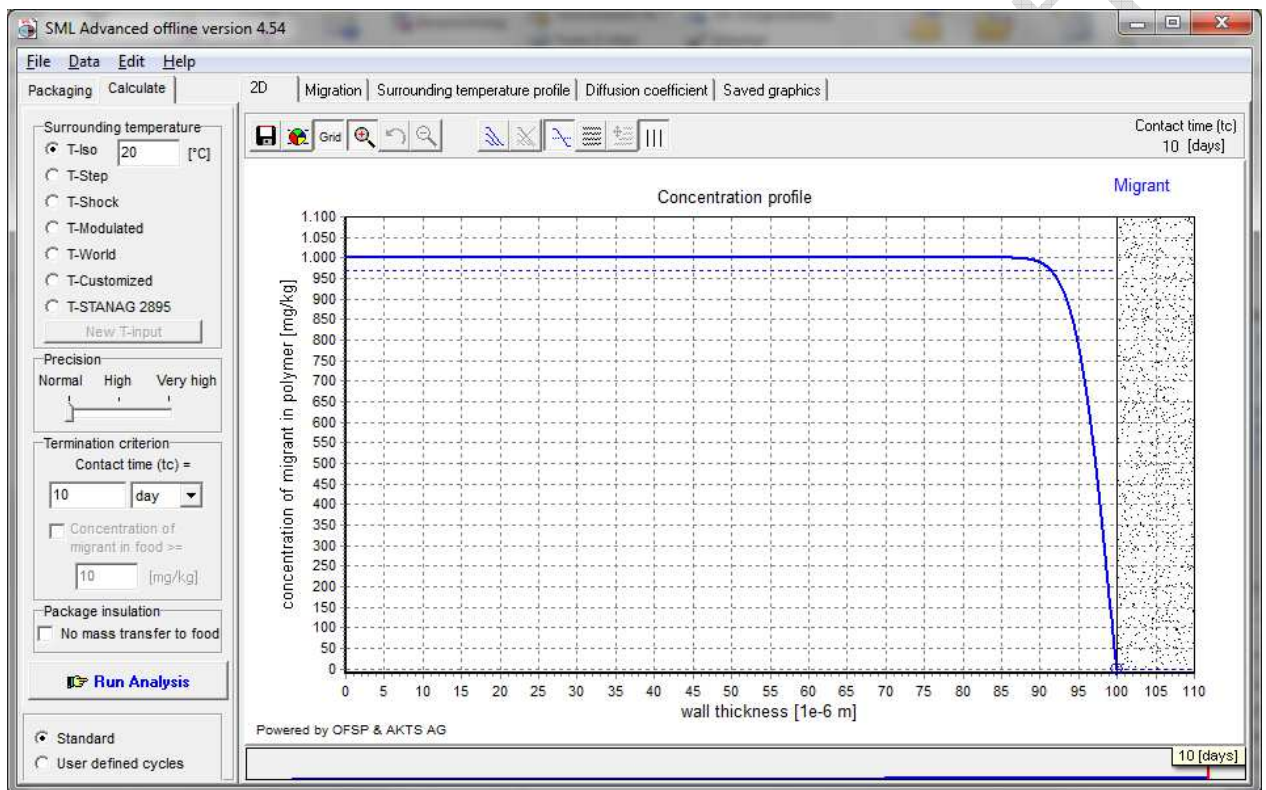
2303 to be used for worst case calculation of specific migration under assumption of total transfer

2304 => 2 x 99% layer thickness = 26 µm

2305 above 26 µm two sides to be considered for calculation of migration if full immersion testing

2306 applied

2307



2308

2309

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2313 **2h @ 100°C**

2314 => 100% layer thickness = 65 µm

2315 no absolute barrier at thicknesses below 65 µm

2316 => 99% layer thickness = 54 µm

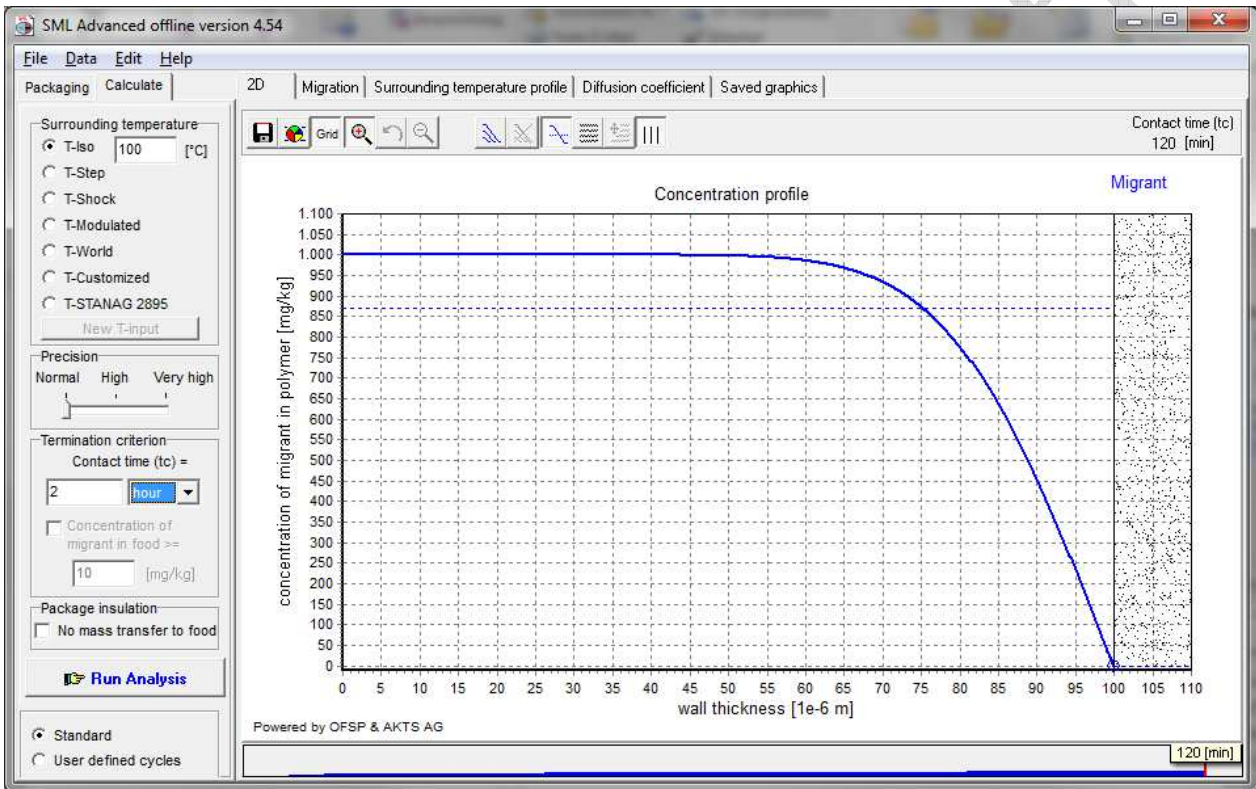
2317 => 1/2 x 99% layer thickness = 27 µm

2318 to be used for worst case calculation of specific migration under assumption of total transfer

2319 => 2 x 99% layer thickness = 108 µm

2320 above 108 µm two sides to be considered for calculation of migration if full immersion testing applied

2322



2323

2324

2325

2326

2327

2328 ► molecular mass 251 - 500 g/mol

2329 10d @ 60°C

2330 => 100% layer thickness = 49 µm

2331 no absolute barrier at thicknesses below 49 µm

2332 => 99% layer thickness = 41 µm

2333 => 1/2 x 99% layer thickness = 20.5 µm

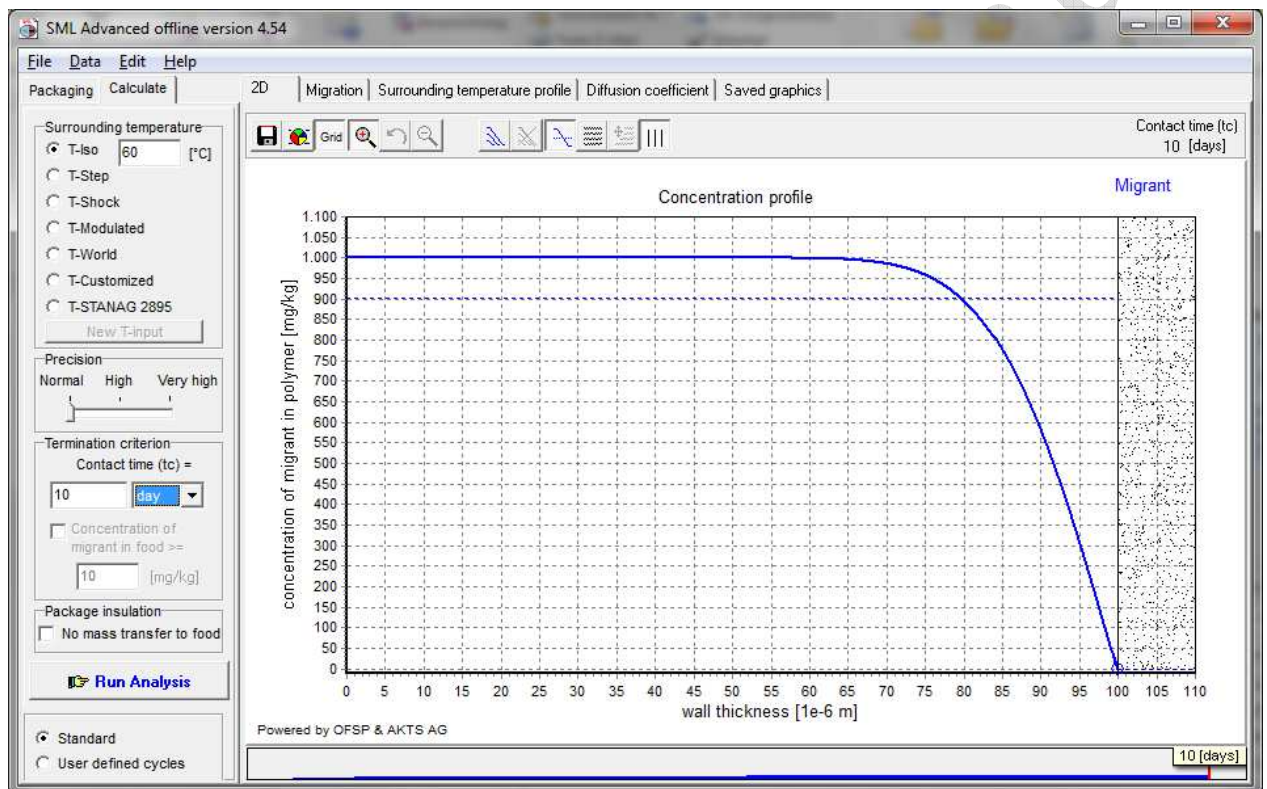
2334 to be used for worst case calculation of specific migration under assumption of total transfer

2335 => 2 x 99% layer thickness = 82 µm

2336 above 82 µm two sides to be considered for calculation of migration if full immersion testing

2337 applied

2338



2339

2340

2341

2342

2343

2344 **10d @ 40°C**

2345 => 100% layer thickness = 18 µm

2346 no absolute barrier at thicknesses below 18 µm

2347 => 99% layer thickness = 15 µm

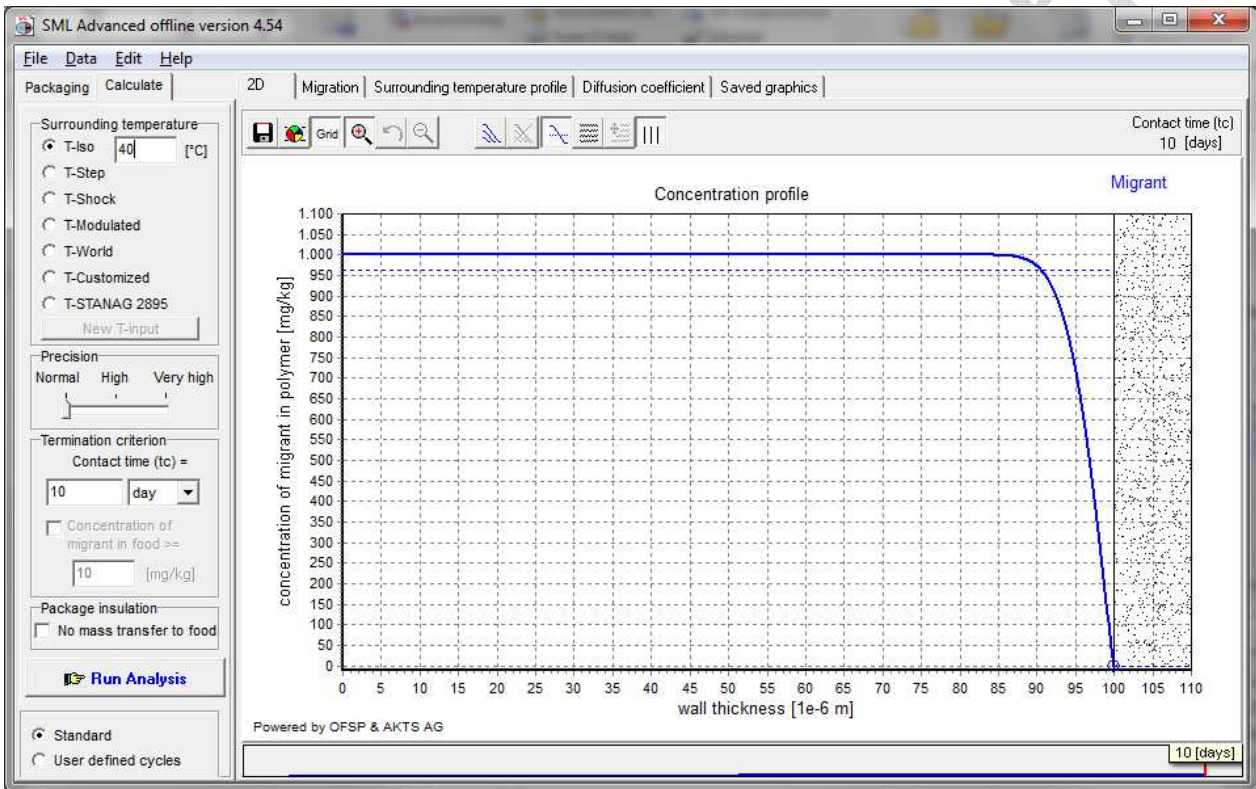
2348 => 1/2 x 99% layer thickness = 7.5 µm

2349 to be used for worst case calculation of specific migration under assumption of total transfer

2350 => 2 x 99% layer thickness = 30 µm

2351 above 30 µm two sides to be considered for calculation of migration if full immersion testing
2352 applied

2353



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2359 **10d @ 20°C**

2360 => 100% layer thickness = 6.2 μm

2361 no absolute barrier at thicknesses below 6.2 μm

2362 => 99% layer thickness = 5 μm

2363 => 1/2 x 99% layer thickness = 2.5 μm

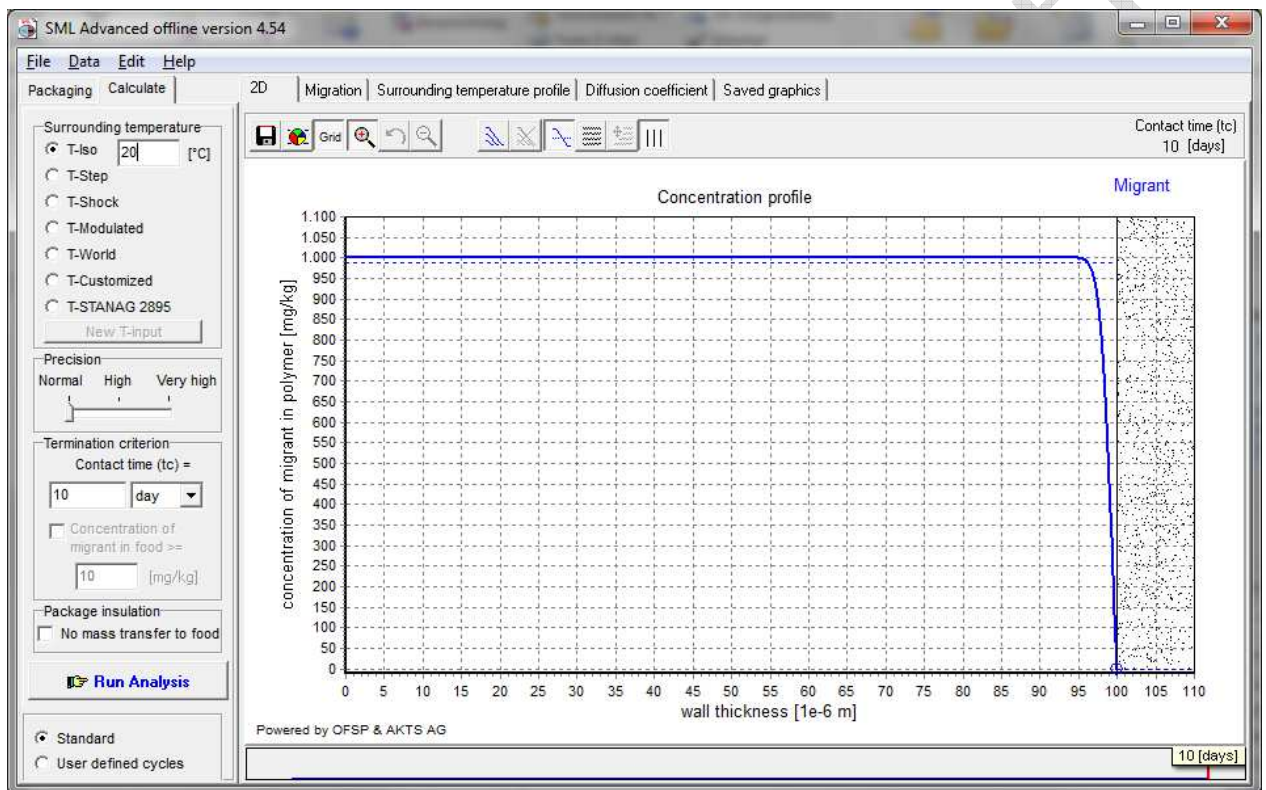
2364 to be used for worst case calculation of specific migration under assumption of total transfer

2365 => 2 x 99% layer thickness = 10 μm

2366 above 10 μm two sides to be considered for calculation of migration if full immersion testing

2367 applied

2368



2369

2370

2371

2372

2373

2374 **2h @ 100°C**

2375 => 100% layer thickness = 26 µm

2376 no absolute barrier at thicknesses below 26 µm

2377 => 99% layer thickness = 20 µm

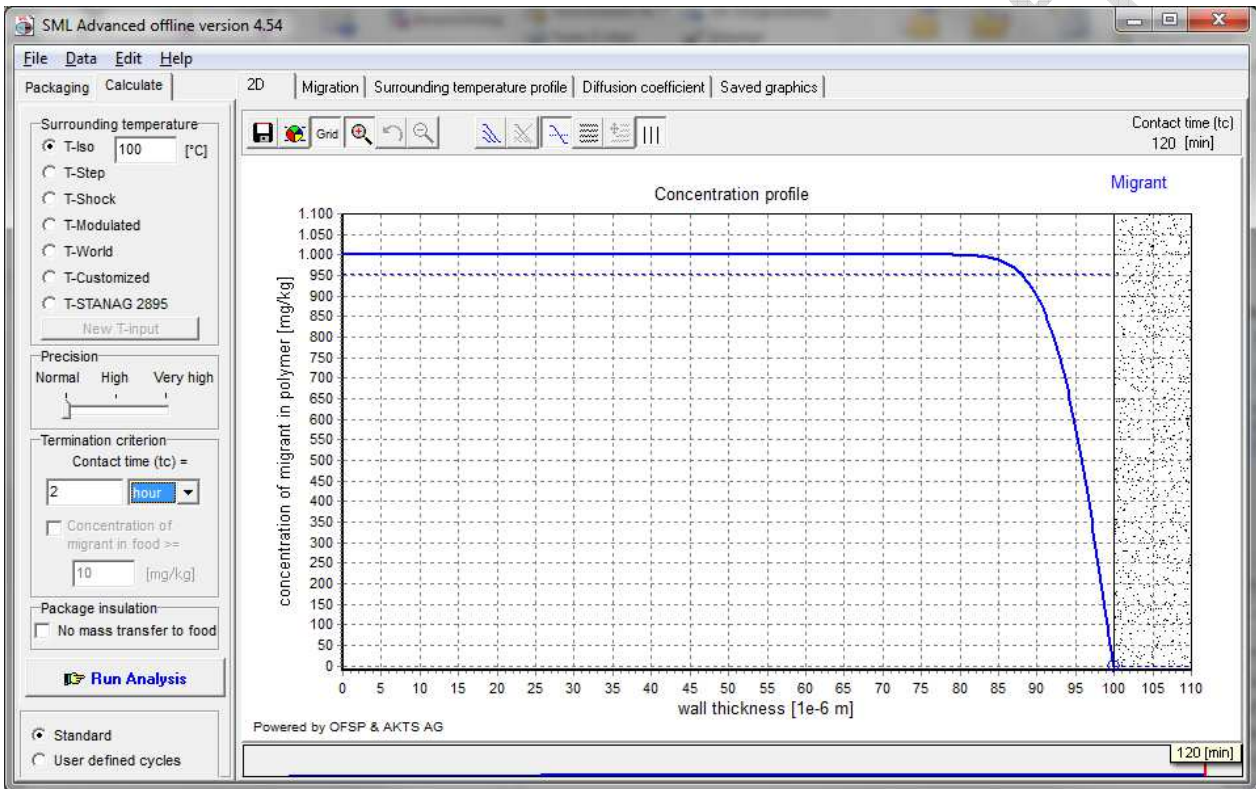
2378 => 1/2 x 99% layer thickness = 10 µm

2379 to be used for worst case calculation of specific migration under assumption of total transfer

2380 => 2 x 99% layer thickness = 40 µm

2381 above 40 µm two sides to be considered for calculation of migration if full immersion testing
2382 applied

2383



2384

2385

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2388

2389 ► molecular mass 501 - 750 g/mol

2390 10d @ 60°C

2391 => 100% layer thickness = 15.2 µm

2392 no absolute barrier at thicknesses below 15.2 µm

2393 => 99% layer thickness = 12.4 µm

2394 => 1/2 x 99% layer thickness = 6.2 µm

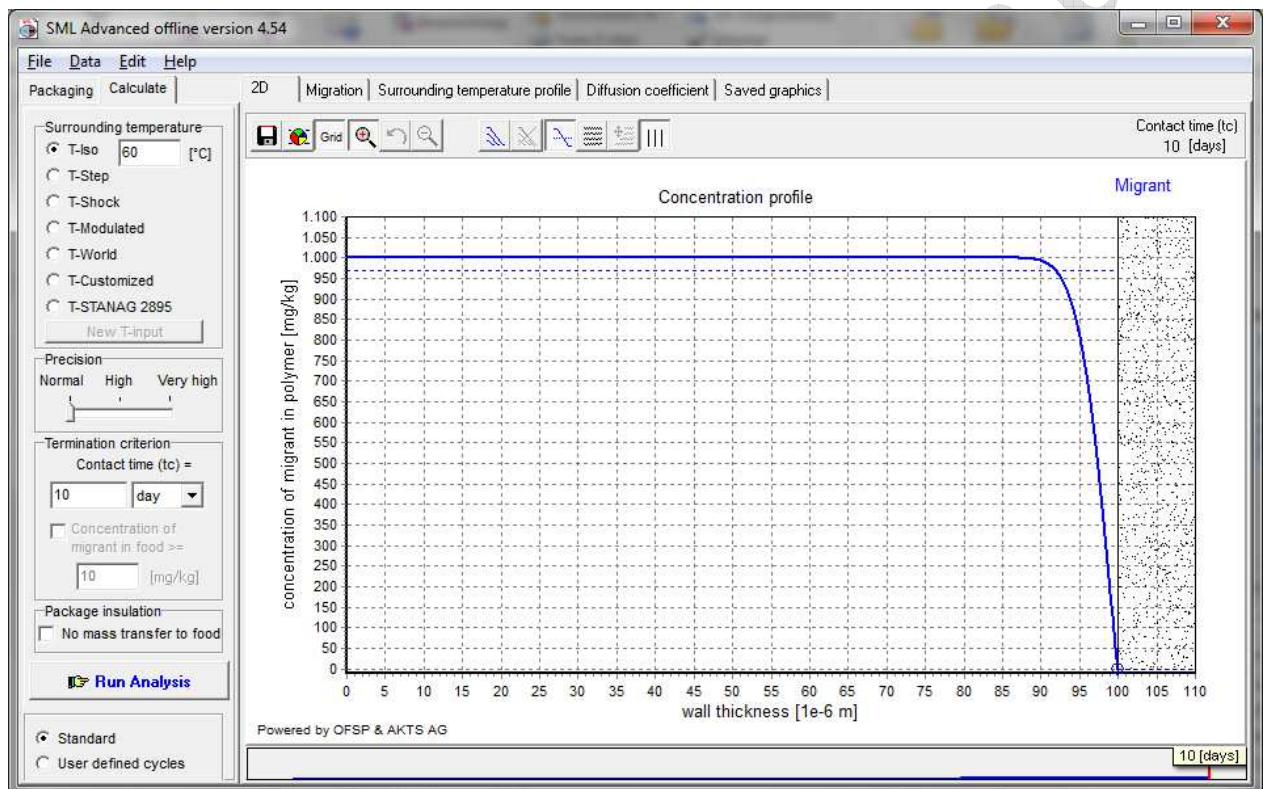
2395 to be used for worst case calculation of specific migration under assumption of total transfer

2396 => 2 x 99% layer thickness = 24.8 µm

2397 above 24.8 µm two sides to be considered for calculation of migration if full immersion testing

2398 applied

2399



2400

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2405 **10d @ 40°C**

2406 => 100% layer thickness = 6 µm

2407 no absolute barrier at thicknesses below 6 µm

2408 => 99% layer thickness = 5 µm

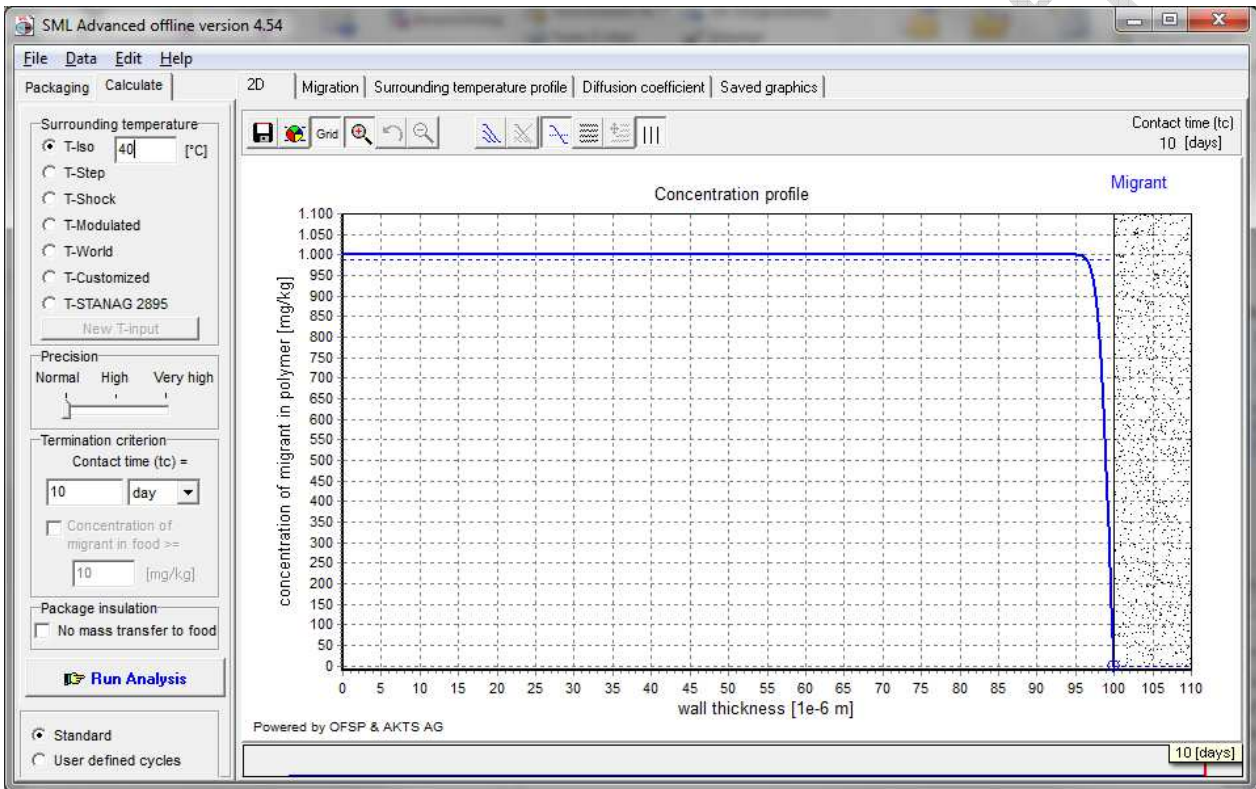
2409 => 1/2 x 99% layer thickness = 2.5 µm

2410 to be used for worst case calculation of specific migration under assumption of total transfer

2411 => 2 x 99% layer thickness = 10 µm

2412 above 10 µm two sides to be considered for calculation of migration if full immersion testing
2413 applied

2414



2415

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2420 **10d @ 20°C**

2421 => 100% layer thickness = 2.2 µm

2422 no absolute barrier at thicknesses below 2.2 µm

2423 => 99% layer thickness = 1.6 µm

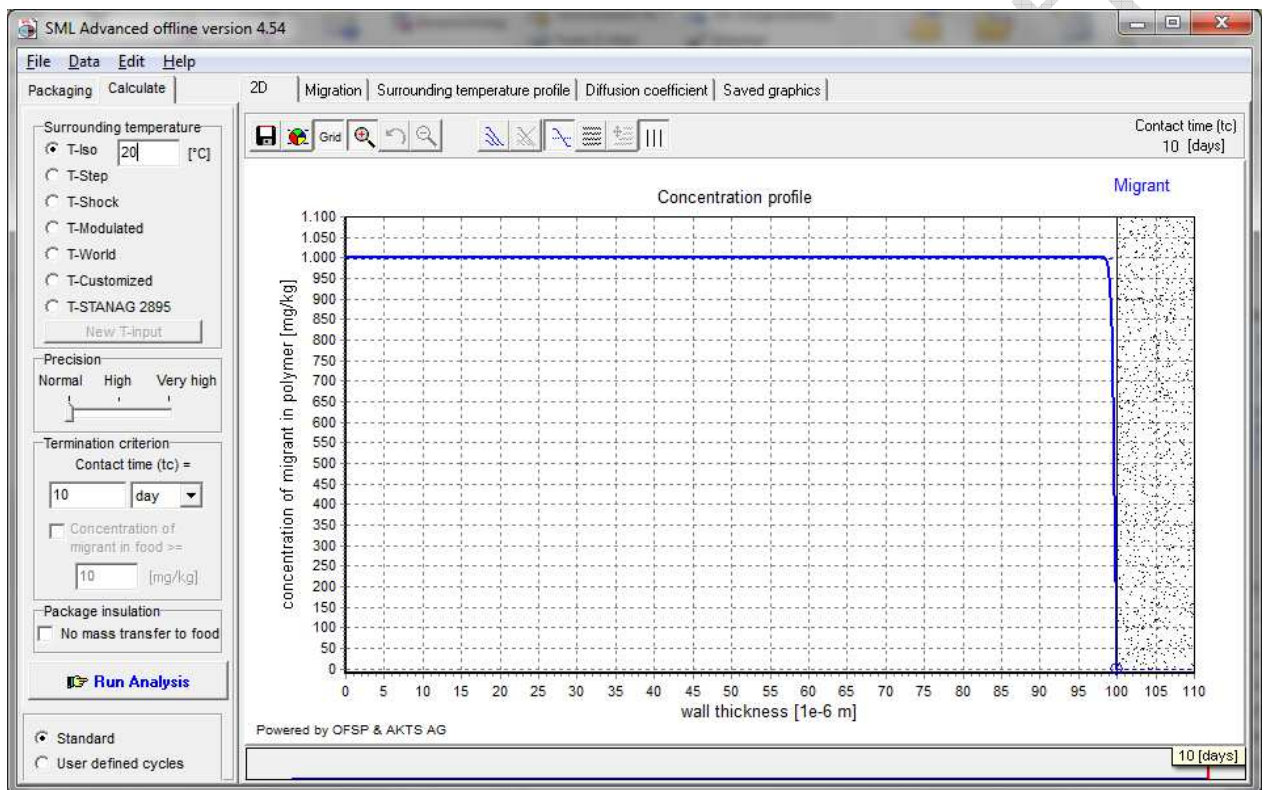
2424 => 1/2 x 99% layer thickness = 0.8 µm

2425 to be used for worst case calculation of specific migration under assumption of total transfer

2426 => 2 x 99% layer thickness = 3.2 µm

2427 above 3.2 µm two sides to be considered for calculation of migration if full immersion testing applied

2429



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2435 **2h @ 100°C**

2436 => 100% layer thickness = 8 µm

2437 no absolute barrier at thicknesses below 8 µm

2438 => 99% layer thickness = 6.2 µm

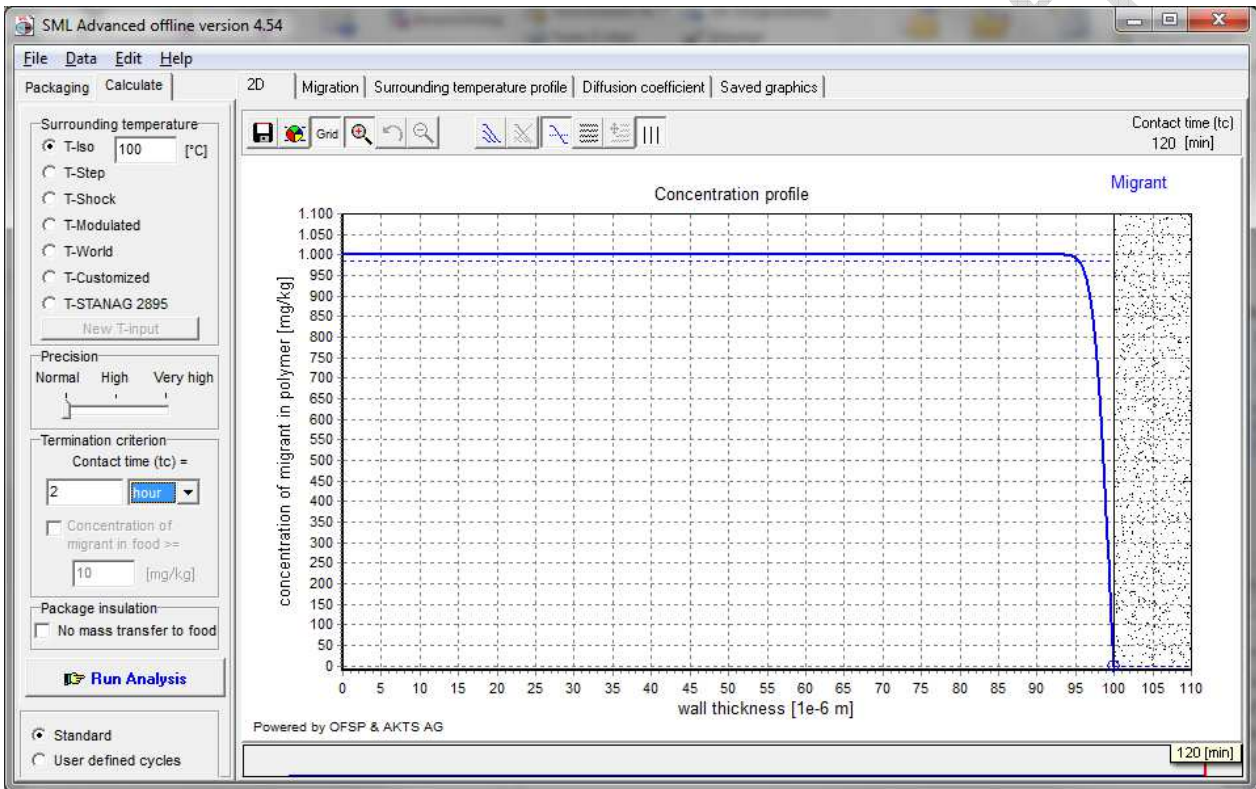
2439 => 1/2 x 99% layer thickness = 3.1 µm

2440 to be used for worst case calculation of specific migration under assumption of total transfer

2441 => 2 x 99% layer thickness = 12.4 µm

2442 above 12.4 µm two sides to be considered for calculation of migration if full immersion testing applied

2444



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2450 ► molecular mass 751 - 1000 g/mol

2451 10d @ 60°C

2452 => 100% layer thickness = 6 µm

2453 no absolute barrier at thicknesses below 6 µm

2454 => 99% layer thickness = 4.8 µm

2455 => 1/2 x 99% layer thickness = 2.4 µm

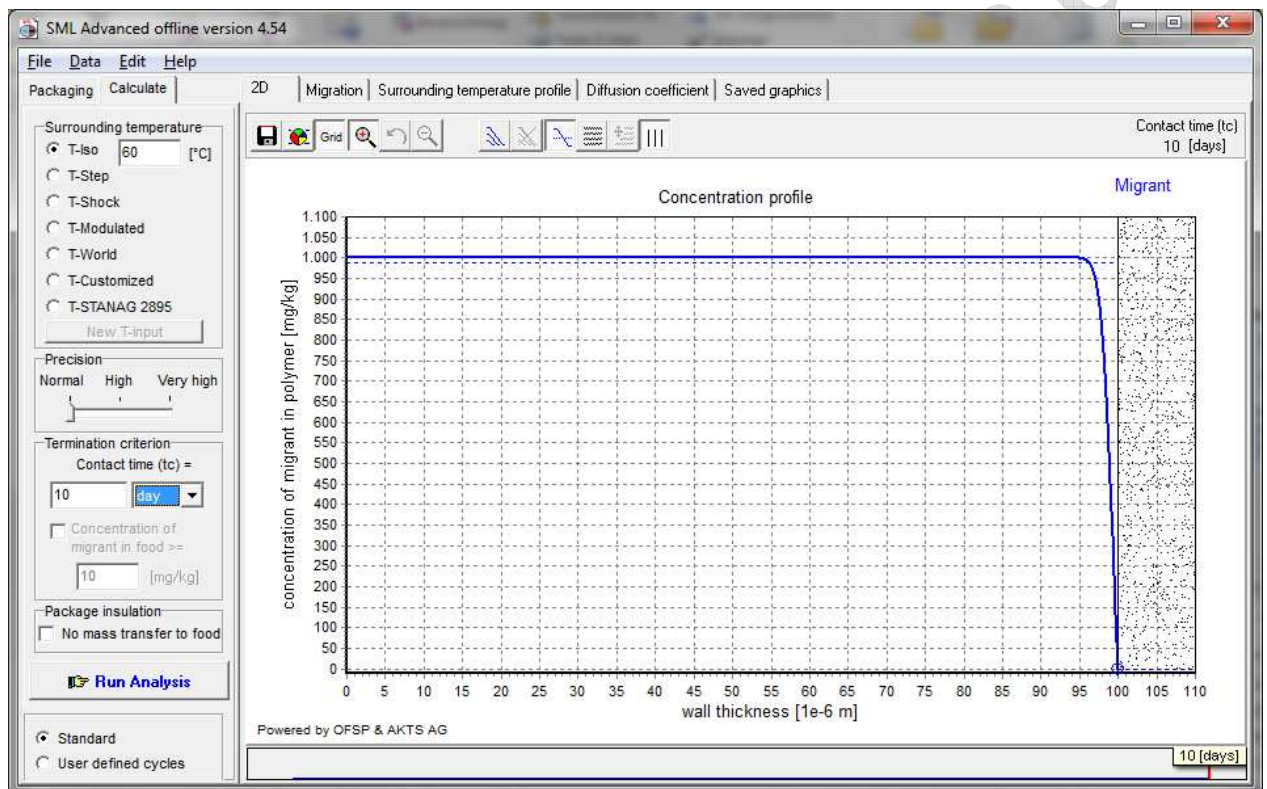
2456 to be used for worst case calculation of specific migration under assumption of total transfer

2457 => 2 x 99% layer thickness = 9.2 µm

2458 above 9.2 µm two sides to be considered for calculation of migration if full immersion testing

2459 applied

2460



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2466 **10d @ 40°C**

2467 => 100% layer thickness = 2.8 µm

2468 no absolute barrier at thicknesses below 2.8 µm

2469 => 99% layer thickness = 2 µm

2470 => 1/2 x 99% layer thickness = 1 µm

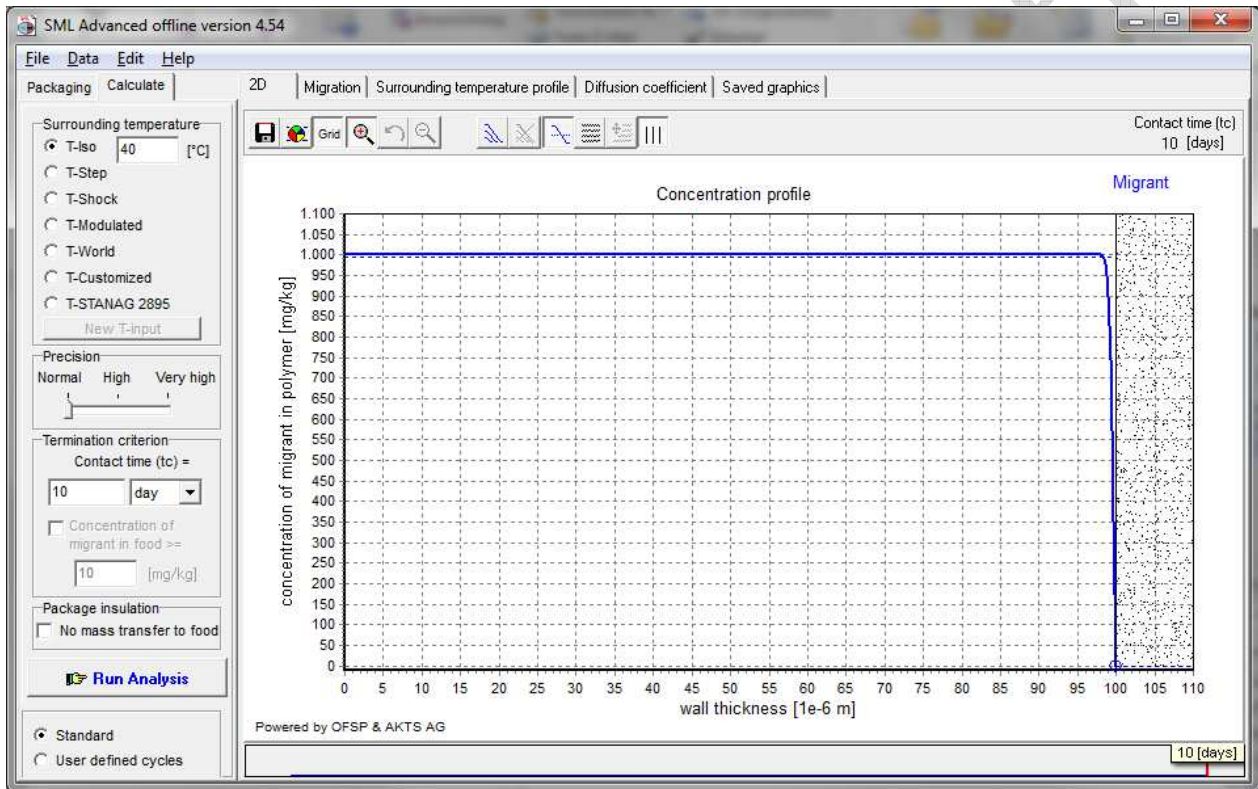
2471 to be used for worst case calculation of specific migration under assumption of total transfer

2472 => 2 x 99% layer thickness = 4 µm

2473 above 4 µm two sides to be considered for calculation of migration if full immersion testing

2474 applied

2475



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2481 **10d @ 20°C**

2482 => 100% layer thickness = 0.8 μm

2483 no absolute barrier at thicknesses below 0.8 μm

2484 => 99% layer thickness = 0.6 μm

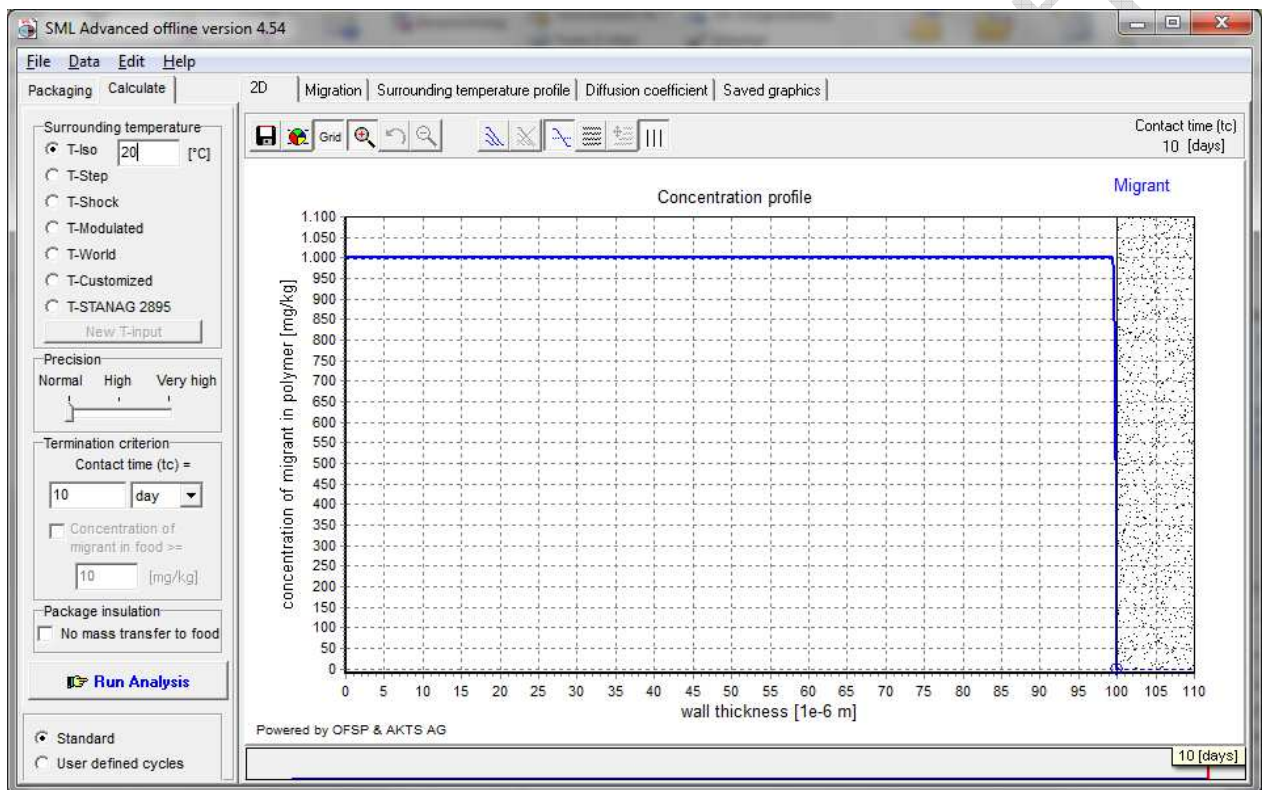
2485 => 1/2 x 99% layer thickness = 0.3 μm

2486 to be used for worst case calculation of specific migration under assumption of total transfer

2487 => 2 x 99% layer thickness = 1.2 μm

2488 above 1.2 μm two sides to be considered for calculation of migration if full immersion testing applied

2490



2491

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2493

2494

2495

2496 **2h @ 100°C**

2497 => 100% layer thickness = 3.3 µm

2498 no absolute barrier at thicknesses below 3.3 µm

2499 => 99% layer thickness = 2.6 µm

2500 => 1/2 x 99% layer thickness = 1.3 µm

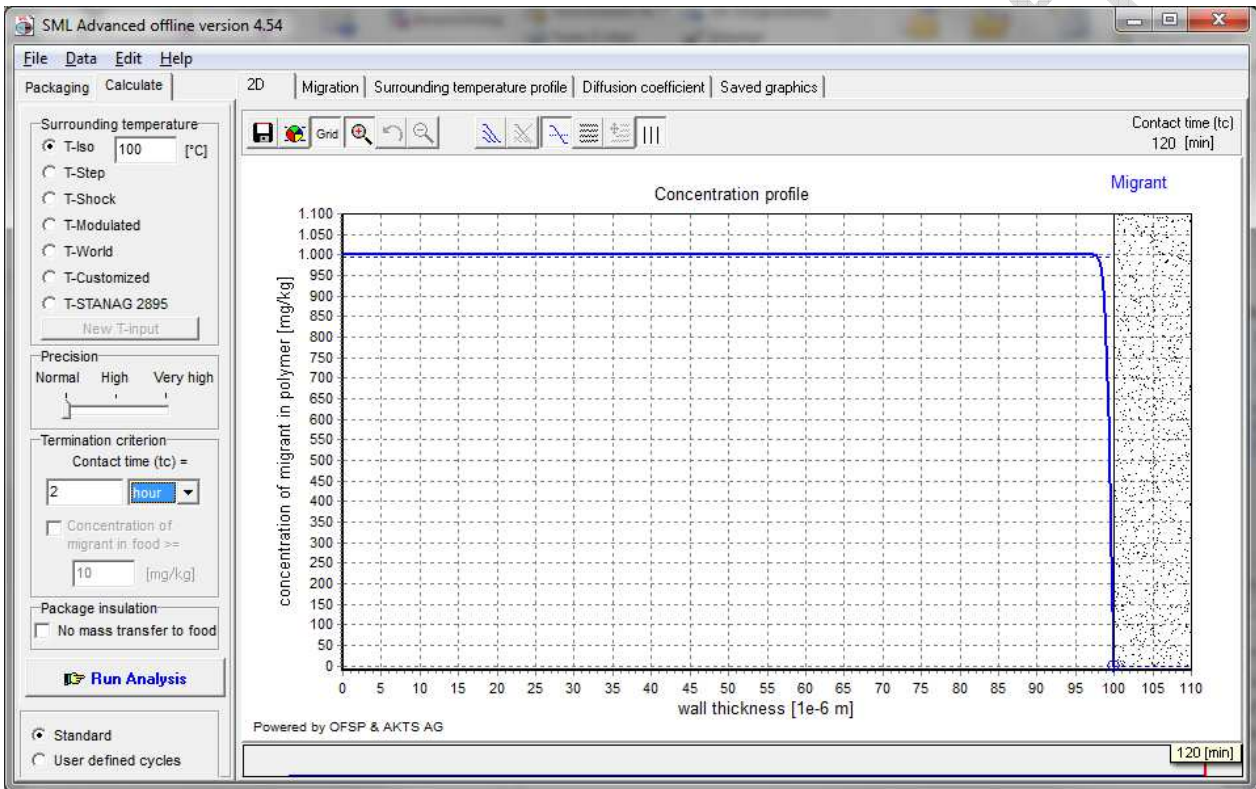
2501 to be used for worst case calculation of specific migration under assumption of total transfer

2502 => 2 x 99% layer thickness = 5.2 µm

2503 above 5.2 µm two sides to be considered for calculation of migration if full immersion testing

2504 applied

2505



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Draft for consultation

2512 Annex 5 Section 5.2.1

2513

2514 EXAMPLE 1 – Determination of compliance with an SML using the OM result

2515 SM of 2,4,6-triamino-1,3,5-triazine (CAS No. 108-78-1) into simulant D2

2516 (a) Taking into account the analytical tolerance

2517 Overall migration of a film was determined after 10 days at 40°C in food simulant D2. The
2518 average overall migration value was 1 mg/dm². The conventional surface-to-volume ratio of 6
2519 dm²/kg of food leads to an equivalent to 6 mg/kg food simulant. Taking into account the
2520 analytical tolerance for the determination of OM into food simulant D2 (18 mg/kg) then the
2521 maximum overall migration value (measured value + analytical tolerance) is 24 mg/kg food
2522 simulant. The SML for 2,4,6-triamino-1,3,5-triazine given in Regulation (EU) No 10/2011 is 30
2523 mg/kg. Therefore the overall migration value including analytical tolerance is below the SML.
2524 Therefore it can be concluded that as long as the migrant is stable in the food simulant during
2525 the contact phase and that it is not lost by volatilisation then for all SM conditions equal to or
2526 less severe than 10 days at 40°C the overall migration result can be used to confirm that the
2527 migration of 2,4,6-triamino-1,3,5-triazine into fatty food is in compliance with the SML.

2528 (b) Taking into account the stability of the analyte

2529 Considering the structure of 2,4,6-triamino-s-triazine it is not expected that it will breakdown
2530 during contact but it is expected that it will react with food simulant D2. However the reaction
2531 products formed are not volatile and therefore it can be concluded that as long as the migrant
2532 and its reaction products is not lost by volatilisation then for all SM conditions equal to or less
2533 severe than 10 days at 40°C the overall migration result can be used to confirm that the
2534 migration of 2,4,6-triamino-1,3,5-triazine into fatty food is in compliance with the SML.

2535 (c) Taking into account the volatility of the analyte

2536 From the literature the melting point of 345°C is known, i.e. the migrant is non-volatile.
2537 Therefore it can be concluded that for all SM conditions equal to or less severe than 10 days at
2538 40°C the overall migration result can be used to confirm that the migration of 2,4,6-triamino-
2539 1,3,5-triazine into fatty food is in compliance with the SML.

2540

2541 NOTE: This example is dealing with food simulant D2 however when compliance is
2542 determined with food simulant B then the OM may exceed the SML where as the SM of
2543 melamine would comply with the SML. It should be noticed that the melamine will react with
2544 the acetic acid to form the salt. The molecular mass of melamine is 126 g/mol and the
2545 molecular mass of the melamine triacetate is 306 g/mol. So the OM will increase by a factor of
2546 $(306/126) = 2.4$. A SM of 30 mg melamine/kg will result into an OM of 73 mg melamine
2547 triacetate /kg. The SM is compliant if the analytical tolerance is included but the OM is non-
2548 compliant if the analytical tolerance of 12 mg/kg is included.

2549

2550 EXAMPLE 2 – Determination of compliance with an SML using the OM result

2551 SM of diethylene glycol (CAS No. 111-46-6) into simulant A

2552 (a) Taking into account the analytical tolerance

2553 Overall migration of a film was determined after 10 days at 40°C in food simulant A. The average
2554 overall migration value was 1 mg/dm². The conventional surface-to-volume ratio of 6 dm²/kg of
2555 food leads to an equivalent to 6 mg/kg food simulant. Taking into account the analytical
2556 tolerance for the determination of OM into food simulant A (12 mg/kg) then the maximum
2557 overall migration value (measured value + analytical tolerance) is 18 mg/kg food simulant. The
2558 SML(T) for diethylene glycol given in Regulation (EU) No 10/2011 is 30 mg/kg. Therefore it can
2559 be concluded that as long as the migrant is stable in the food simulant during the contact phase

2560 and that it is not lost by volatilisation then for all SM conditions equal to or less severe than 10
2561 days at 40°C the overall migration result can be used to confirm that the migration of diethylene
2562 glycol into food simulant A is in compliance with the SML.

2563 (b) Taking into account the stability of the analyte

2564 Considering the structure of diethylene glycol it is not expected that it will breakdown during
2565 the contact time and nor is it expected that it will react with simulant A and therefore it can be
2566 concluded that as long as the migrant is not lost by volatilisation then for all SM conditions equal
2567 to or less severe than 10 days at 40°C the overall migration result can be used to confirm that
2568 the migration of diethylene glycol into simulant A is in compliance with the SML.

2569 (c) Taking into account the volatility of the analyte

2570 From the literature the boiling point of 244°C is known, i.e. the analyte is semi-volatile. It has
2571 previously been demonstrated Bradley et al. (2009) that the recovery of diethylene glycol
2572 following evaporation of aqueous simulants is 0%. Therefore for this substance the volatility is
2573 such that the compliance with the SML cannot be demonstrated by the determination of the OM.

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-butylphenyl)-4,4'-biphenylene 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@10T orr		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									
-----	--	--	--	--	--	--	--	--	--

2575

2576 **Explanations:**

- 2577 YES (1): all food simulants, if recovery is ok
- 2578 YES (2): for volatile food simulants, if recovery is ok
- 2579 borderline for oils as simulant, if recovery is ok
- 2580 YES (3): for volatile food simulants, if recovery is ok
- 2581 NOT for oils as food simulant
- 2582 YES (4): borderline for volatile food simulants, if recovery is ok
- 2583 NOT for oils as food simulants
- 2584 NO: all food simulants, because SML too low or due to poor recovery

2585

2586

Draft for consultation

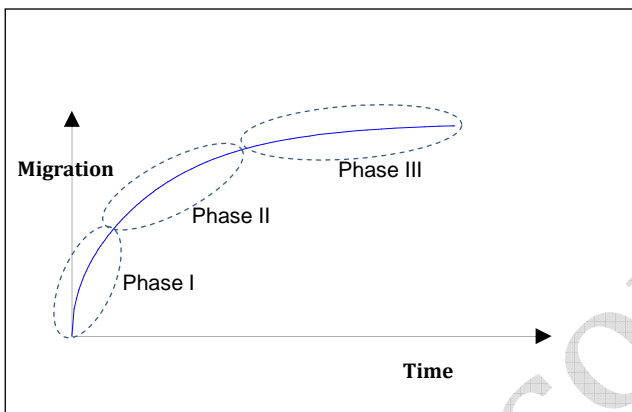
2587 **Annex 6 Section 5.2.5 background document**

2588 **Scientific reasoning for use of screening food simulants instead of vegetable oils and**
2589 **related testing conditions.**

2590

2591 The following two principal contributions to the migration behaviour in the system food contact
2592 material in contact with food simulants will determine the result of a migration test:
2593 1) thermodynamic contribution, i.e. relative solubility of the migrant between plastic and
2594 simulant, described by the partition coefficient of the migrant between both matrices
2595 2) kinetic contribution, i.e. diffusion rate of the migrant in and from the plastic into the simulant.
2596 Typically a migration curve (understood as migration as a function of time) for polymers in
2597 contact with liquids consists of 3 phases: an initial phase (phase I) where the migration is
2598 controlled only or largely by the kinetic contribution and final phase (phase III) where the
2599 migration is controlled by the thermodynamic contribution. In between of both phase the
2600 migration is influenced by both elements (Figure 1).

2601 Figure 1: Typical migration curve showing three basic phases of migration



2602

2603 A specific migration test for a plastic with an screening food simulant will be more severe
2604 compared to the conventional migration test if: (a) both of the two contributions are more
2605 severe, or (b) one contribution dominates the other, i.e. the more severe contribution
2606 compensates the less severe contribution, resulting overall again in a more severe migration
2607 test.

2608 To prevent testing under to severe conditions resulting in extremely overestimating and totally
2609 unrealistic results testing migration with an screening simulant may not employ only an
2610 screening food simulant but also appropriately different time/temperature testing conditions.

2611 Deviation from food simulants and conventional time - temperature conditions for migration
2612 testing according to PIM is possible based on scientific evidence. However, currently not enough
2613 data and knowledge exists to establish a general rule covering all possible migration test cases.
2614 Especially, the increase of migration rates due to swelling effects in the polymer strongly
2615 depends on the nature of the polymer and the type of simulant and is not predictable based on
2616 today's knowledge.

2617

2618 **Thermodynamic considerations**

2619 Thermodynamic considerations should focus in the first place on the solubility of the migrant in
2620 the screening food simulant. The relative solubility of the migrant, understood as the ratio of
2621 concentrations of the migrant in the FC polymer and the food simulant at equilibrium (which is
2622 the partition coefficient) determines in which amounts the migrant will migrate in phases II and
2623 III of the migration process.

2624 From a purely thermodynamic point of view the requirement that the migration tests with the
2625 screening food simulants must be more severe compared to the test with vegetable oil implies

2626 that the solubility of the migrants in the screening food simulant is equal to or higher than in
2627 vegetable oils. As a consequence, when applying the same conventional time and temperature
2628 conditions as applicable for vegetable oil, at least the same or higher migration test result should
2629 be obtained.

2630

2631 **Solubility of migrants in screening food simulants**

2632 There is a well-known chemical rule of the thumb stating "similar dissolves similar". For
2633 screening food simulants this means that they need to have a similar polarity as vegetable oils,
2634 which ensures that the solubility of the migrants in the screening food simulant is comparable to
2635 or even higher than in vegetable oils.

2636 With introduction of the solubility parameter an important scientific approach which makes the
2637 quantification of polarities for liquid media like food simulants or typical solvents and polymers
2638 possible was published by Hildebrandt in 1950 [1]. The solubility parameter δ is defined as the
2639 square root of the cohesive energy density at 20°C. There are three main contributions to the
2640 cohesive energy: (1) contribution of dispersion forces, (2) contribution of polar forces and (3)
2641 contribution of hydrogen bonding. The solubility parameter for a medium (solvent or polymer)
2642 is calculated $\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$. For a mixture of two solvents the solubility parameter of the
2643 mixture is calculated based on the volume fraction of the two solvents.

2644 A substance is soluble in a medium (solvent or polymer) if the solubility parameters are equal or
2645 close to each other. According to Yalkowsky [2], any two liquids will be completely miscible at
2646 room temperature if their solubility parameters do not differ by more of three units.

2647 Regarding interaction between a polymer and the contact medium the extent of swelling of the
2648 polymer by the solvent will be more pronounced if their solubility parameters are close to each
2649 other. Solubility parameters can be estimated by the group contribution method introduced by
2650 van Krevelen [3].

2651 Based on the above considerations an appropriate screening food simulant (FS) may be selected
2652 for a given polymer – migrant system.

2653 In this context it is important to note that due to the lower molecular weight of screening food
2654 simulants the entropic contribution of the screening food simulants to the solubility is higher
2655 compared to vegetable oils. It can therefore be expected that at the same polarity of an screening
2656 FS versus vegetable oil, migrants are better soluble in the screening food simulants compared to
2657 vegetable oil or, in other word, the solubility of a migrant in an screening FS is sufficiently high
2658 even if the polarity of this FS deviates from that of the edible oil. .

2659

2660 **Selection of screening food simulants**

2661 Based on the above considerations the selection of an screening food simulant can be made. The
2662 requirement is that the result of the migration tests with the screening food simulants must be
2663 at least as severe as the test with vegetable oil. This implies that the solubility of the migrants in
2664 the screening food simulant is at a minimum as high as in vegetable oils. In combination with the
2665 use of conventional time and temperature conditions as applicable for vegetable oil, in general a
2666 more severe migration test results will be obtained.

2667 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

2668 According to the scientific considerations [4] esters built from C2 to C4 acids with C2 to C4
2669 alcohols and mixtures of these with aliphatic hydrocarbons with C6 to C8 carbon atoms can be
2670 recommended as screening food simulants for migration testing (specific and overall), which
2671 most likely will satisfy the requirement that the solubility of the migrants in the screening food
2672 simulant is as high as in vegetable oils, due to similar polarity of the screening food simulant
2673 with vegetable oil.

2674 For the above general recommendation regarding nature of screening food simulants it was
2675 considered that the screening food simulants should exhibit the same chemical functionalities,
2676 i.e. ester functionality (dipole-dipole interaction) and segments of hydrocarbons (Van der Waals or
2677 dispersion interaction) as vegetable oils.

2678 Because vegetable oils are not capable to undergo hydrogen bonding screening food simulants
2679 capable to undergo hydrogen bonding like alcohols are taken into consideration as screening
2680 food simulants for vegetable oils only for specific cases, i.e. dedicated polymers. This means that
2681 alcohols like for example ethanol are eligible in specific situations, but from generic point of
2682 view due to the hydroxyl functionality they show a different solubilizing mechanism compared
2683 to fatty acid esters, i.e. vegetable oils. Similar limitation is valid for use of pure hydrocarbons like
2684 iso-octane which are eligible in specific situations, but from general point of view due to lack of
2685 functional groups they show a different solubilizing mechanism compared to fatty acid esters,
2686 i.e. vegetable oils.

2687 In the further considerations distinction between specific migration testing and overall
2688 migration testing is required.

2689 Screening food simulants selection for specific migration testing

2690 The recommendation on screening food simulants selection is based on the rule "similar solves
2691 similar", i.e. the closer the polarity of the migrant and the simulant is, the better the solubility of
2692 the migrant will be in the simulant. As a measure of polarity the octanol to water partition
2693 coefficient ($K_{O/W}$) is used because plenty of scientific literature is in place and numerous
2694 estimation procedures including software tools exist.

2695 A migration experiment from thermodynamic point of view involves three parties: (A) the
2696 simulant, (B) the migrant and (C) the polymer. Each of these exhibits a polarity which can be
2697 expressed by an octanol to water partition coefficient. Establishing octanol to water partition
2698 coefficient for polymers is unlikely, but based on some conventional assumption an estimate can
2699 be made.

2700 Starting point is a specific migration experiment for a migrant from a plastic with vegetable oil
2701 as required by legislation. The migrant will exhibit an octanol to water partition coefficient of
2702 $K_{O/W}(\text{mig})$ and the vegetable oil will exhibit an octanol to water partition coefficient of $K_{O/W}(\text{oil})$.
2703 Octanol to water partition coefficients for vegetable oils are in the range of 20 to 30 [5], e.g. for
2704 Tripalmitoylglycerol $K_{O/W}(\text{oil}) = 21.9$ and Tristearoylglycerol $K_{O/W}(\text{oil}) = 25.11$.

2705 When substituting the vegetable oil with a screening food simulant this would exhibit an
2706 octanol to water partition coefficient of $K_{O/W}(\text{sim})$. The following systematic approach is based
2707 on the screening food simulants ethanol and iso-octane, because they represent the two
2708 extremes from polarity point of view and substantial experience has been gained in the past
2709 published in the scientific literature.

2710
2711 $K_{O/W}(\text{ethanol}) \sim 0$ (estimated values are scattering around 0 depending on the estimation tool
2712 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-

2713 $K_{O/W}(\text{iso-octane}) = 4.1$ (estimated with EPI suite)
 2714 Because of the significantly lower molecular weight of screening food simulants compared to
 2715 vegetable oil the octanol to water partition coefficient $K_{O/W}(\text{sim})$ is corrected by the ratio of the
 2716 molecular weight between vegetable oil and the screening food simulant.
 2717 As generic vegetable oil the triglyceride with one stearic acid, one linoleic acid and one palmitic
 2718 acid was considered showing a molecular weight of 861.44 g/mol and an estimated octanol to
 2719 water partition coefficient of 22.74 (EPI suite).

Log Kow(version 1.68 estimate): 22.74

SMILES : O=C(CCCCCC=CCCCCCCC)OC(COC(=O)CCCCCCCCCCCC)COC(=O)CCCCCCCCCCCC
 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< [olefinic 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

2720 Because of the significantly lower molecular weight of screening food simulants compared to
 2721 vegetable oil the octanol to water partition coefficient $K_{O/W}(\text{sim})$ is corrected by the ratio of the
 2722 molecular weight between vegetable oil and the screening food simulant.
 2723

2724 This procedure is reasonable because the most estimation procedures for the octanol to water
 2725 partition coefficient (e.g. EPI Suite from US EPA) are based on fragment increments. As a
 2726 consequence bigger molecules with higher molecular weight like vegetable oil exhibit higher
 2727 octanol to water partition coefficients compared to smaller molecules with lower molecular
 2728 weight like ethanol or iso-octane, even if they are of similar polarity.

2729 As an example iso-octane with molecular weight of 114 g/mol exhibits an octanol to water
 2730 partition coefficient of $K_{O/W}(\text{C8}) = 4.1$. A saturated hydrocarbon with molecular weight of 254
 2731 g/mol exhibits an octanol to water partition coefficient of $K_{O/W}(\text{C18}) = 9.2$. A saturated
 2732 hydrocarbon with molecular weight of 858 g/mol exhibits an octanol to water partition
 2733 coefficient of $K_{O/W}(\text{C61}) = 30.3$.

2734 Correcting the octanol to water partition coefficient for iso-octane by the ratio of the molecular
 2735 weight between vegetable oil and the screening food simulant means calculating the octanol to
 2736 water partition coefficient for a saturated hydrocarbon with the same molecular weight as
 2737 vegetable oil.

2738 When considering ethanol and iso-octane as screening food simulants to vegetable oil and
 2739 correcting their octanol to water partition coefficients by the molecular weight ratio a polarity
 2740 scale between 0 and 31 results:

2741 $K_{O/W}^{\text{corr}}(\text{ethanol}) = 861.44 / 46 * K_{O/W}(\text{ethanol}) \sim 0$

2742 $K_{O/W}^{\text{corr}}(\text{iso-octane}) = 861.44 / 114 * K_{O/W}(\text{iso-octane}) = 31$

2743 The condition for the selection of the screening food simulant is now:
 2744

2745 $\text{ratio}^K = [K_{O/W}(\text{sim}) - K_{O/W}(\text{mig})] / [K_{O/W}(\text{oli}) - K_{O/W}(\text{mig})]$

2746 $-1 < \text{ratio}^K < 1$

2747

2748 If the above ratio^K is above -1 and below 1 the screening food simulant can be considered to be
 2749 an screening for vegetable oil. If the above ratio^K is between -1.5 and -1 respectively 1 and 1.5
 2750 the food simulant may be a reasonable screening for vegetable oil, but a certain risk of
 2751 underestimation exists.

2752 The condition " $-1 < \text{ratio}^K < 1$ " is a strong requirement if migration comes close to equilibrium
2753 concentration. For $-1.5 < \text{ratio}^K < -1$ or $1 < \text{ratio}^K < 1.5$ no underestimation is expected if:

2754 a) the migrating amount of the substance will be significantly lower than its equilibrium
2755 concentration, or

2756 b) the migrant is sparingly soluble in vegetable oil and as sparingly soluble in the screening food
2757 simulant.

2758 This is the case for example because release of the substance from the polymer under testing
2759 conditions is diffusion controlled [6] or simply because the total amount of substance present in
2760 the material is significantly lower than the equilibrium concentration.

2761 It should be noted that selection of an screening food simulant were the solubility for the
2762 migrant is less than in vegetable oil will have an impact on partitioning of the substance between
2763 polymer and simulant, which will lower the equilibrium concentration in the simulant.

2764 The suitability of the screening food simulant ethanol 95% and iso-octane for some typical
2765 examples (monomers or additives) is considered below including reference to existing literature
2766 data.

2767	1) Lauro lactam	$K_{O/W}$	=	3.6	(estimated	with	EPI	Suite)
2768	2) Tinuvin 1577	$K_{O/W}$	=	6.2	(estimated	with	EPI	Suite)
2769	2) Erucamide	$K_{O/W}$	=	8.4	(estimated	with	EPI	Suite)
2770	3) Irganox 1076	$K_{O/W}$	=	13.4	(estimated	with	EPI	Suite)
2771	4) Irgaphos 168	$K_{O/W}$	=	18.1	(estimated	with	EPI	Suite)
2772								

2773 For testing the specific migration of lauro lactam ethanol is a suitable screening food simulant for
2774 vegetable oil because lauro lactam is better soluble in ethanol than in vegetable oil due to the fact
2775 that $K_{O/W}$ (lauro lactam) is closer to $K_{O/W}$ (ethanol) than to $K_{O/W}$ (oil).

2776 The corresponding calculation is:

$$2777 \quad \text{ratio}^K = [0 - 3.6] / [22.74 - 3.6] = -0.19$$

$$2778 \quad \mathbf{-1 < \text{ratio}^K < 1}$$

2779 For testing the specific migration of lauro lactam iso-octane may be a suitable screening food
2780 simulant for vegetable oil because lauro lactam is less soluble in iso-octane than in vegetable oil
2781 due to the fact that $K_{O/W}$ (lauro lactam) is closer to $K_{O/W}$ (oil) than to $K_{O/W}$ (iso-octane).

2782 The corresponding calculation is:

$$2783 \quad \text{ratio}^K = [31 - 3.6] / [22.74 - 3.6] = 1.43$$

$$2784 \quad \mathbf{< 1 \text{ ratio}^K < 1.5}$$

2785 For lauro lactam a systematic migration study from Polyamide 12 in olive oil compared to is-
2786 octane exists which demonstrates that iso-octane is a suitable screening simulant compared to
2787 olive oil [7]. In this case the suitability of iso-octane vs. olive oil is based on the fact that
2788 lauro lactam exhibits limited solubility in both simulants.

2789 For testing the specific migration of Tinuvin 1577 ethanol is a suitable screening food simulant
2790 for vegetable oil because Tinuvin 1577 is better soluble in ethanol than in vegetable oil due to
2791 the fact that $K_{O/W}$ (Tinuvin 1577) is closer to $K_{O/W}$ (ethanol) than to $K_{O/W}$ (oil).

2792 The corresponding calculation is:

$$2793 \quad \text{ratio}^K = [0 - 6.2] / [22.74 - 6.2] = -0.37$$

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

- 2794 **-1 < ratio^K < 1**
- 2795 For testing the specific migration of Tinuvin 1577 iso-octane may be a suitable screening food
 2796 simulant for vegetable oil because Tinuvin 1577 is less soluble in iso-octane than in vegetable oil
 2797 due to the fact that $K_{O/W}$ (Tinuvin 1577) is closer to $K_{O/W}$ (oil) than to $K_{O/W}$ (iso-octane).
- 2798 The corresponding calculation is:
- 2799 $\text{ratio}^K = [31 - 6.2] / [22.74 - 6.2] = 1.5$
- 2800 **1 < ratio^K = 1.5**
- 2801 For Tinuvin 1577 no comparative systematic migration investigation exists. Instead for Tinuvin
 2802 234 (log $K_{O/W} = 7.67$; calculated with EPI Suite) a systematic migration study from PET into
 2803 ethanol 95%, iso-octane and Miglyol (a fractionated coconut oil) exists [8].
- 2804 $\text{ratio}^K = [31 - 7.67] / [22.74 - 7.67] = 1.55$; $\text{ratio}^K > 1.5$
- 2805 Migration results obtained with iso-octane lightly overestimate those obtained with Miglyol.
- 2806 For testing the specific migration of Erucamide ethanol is a suitable screening food simulant
 2807 because Erucamide is better soluble in ethanol than in vegetable oil due to the fact that
 2808 $K_{O/W}$ (Erucamide) is closer to $K_{O/W}$ (ethanol) than to $K_{O/W}$ (oil).
- 2809 The corresponding calculation is:
- 2810 $\text{ratio}^K = [0 - 8.4] / [22.74 - 8.4] = -0.59$
- 2811 **-1 < ratio^K < 1**
- 2812 For testing the specific migration of Erucamide iso-octane is not expected to be a suitable
 2813 screening food simulant because Erucamide is less soluble in iso-octane than in vegetable oil due
 2814 to the fact that $K_{O/W}$ (Erucamide) is closer to $K_{O/W}$ (oil) than to $K_{O/W}$ (iso-octane).
- 2815 The corresponding calculation is:
- 2816 $\text{ratio}^K = [31 - 8.4] / [22.74 - 8.4] = 1.58$
- 2817 **ratio^K > 1.5**
- 2818 For testing the specific migration of Irganox 1076 ethanol may be a suitable screening food
 2819 simulant for vegetable oil because Irganox 1076 is less soluble in ethanol than in vegetable oil
 2820 due to the fact that $K_{O/W}$ (Irganox 1076) is closer to $K_{O/W}$ (oil) than to $K_{O/W}$ (ethanol).
- 2821 The corresponding calculation is:
- 2822 $\text{ratio}^K = [0 - 13.4] / [22.74 - 13.4] = -1.43$
- 2823 **-1.5 < ratio^K < -1**
- 2824 For testing the specific migration of Irganox 1076 iso-octane is not expected to be a suitable
 2825 screening food simulant for vegetable oil because Irganox 1076 is much less soluble in iso-
 2826 octane than in vegetable oil due to the fact that $K_{O/W}$ (Irganox 1076) is much closer to $K_{O/W}$ (oil)
 2827 than to $K_{O/W}$ (iso-octane).
- 2828 The corresponding calculation is:
- 2829 $\text{ratio}^K = [31 - 13.4] / [22.74 - 13.4] = 1.88$
- 2830 **ratio^K > 1.5**
- 2831 For Irganox 1076 a systematic migration study from LDPE in various foods and olive oil
 2832 compared to ethanol 95% and iso-octane exists which demonstrates that ethanol 95% is still a
 2833 suitable screening simulant compared to olive oil [9].

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

2834 The systematic migration study also indicates that iso-octane might be a suitable screening food
2835 simulant as well. Due to strong swelling of the LDPE film by iso-octane and sufficient solubility
2836 for the amount of Irganox 1076 present in the LDPE film in iso-octane the migration into iso-
2837 octane is higher compared to olive oil.

2838 For testing the specific migration of Irgafos 168 ethanol is not expected to be a suitable
2839 screening food simulant for vegetable oil because Irgafos 168 is much less soluble in ethanol
2840 than in vegetable oil due to the fact that $K_{O/W}$ (Irgafos 168) is much closer to $K_{O/W}$ (oil) than to
2841 $K_{O/W}$ (ethanol).

2842 The corresponding calculation is:

$$2843 \quad \text{ratio}^K = [0 - 18.1] / [22.74 - 18.1] = -3.9$$

$$2844 \quad \text{ratio}^K < -1.5$$

2845 This consideration is substantiated by comparative experimental results were migration values
2846 obtained for Irgafos 168 with ethanol 95% are significantly lower compared to olive oil [10].

2847 For testing the specific migration of Irgafos 168 iso-octane is not expected to be a suitable
2848 screening food simulant for vegetable oil because Irgafos 168 is much less soluble in iso-octane
2849 than in vegetable oil due to the fact that $K_{O/W}$ (Irgafos 168) is much closer to $K_{O/W}$ (oil) than to
2850 $K_{O/W}$ (iso-octane).

2851 The corresponding calculation is:

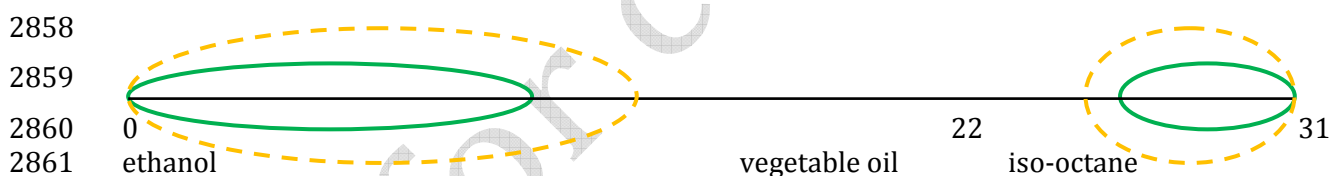
$$2852 \quad \text{ratio}^K = [31 - 18.1] / [22.74 - 18.1] = 2.78$$

$$2853 \quad \text{ratio}^K > 1.5$$

2854 However the same considerations made for Irganox 1076 may apply for Irgafos 168 but no
2855 published comparative experimental data exist.

2856

2857 Polarity scale based on octanol to water partition coefficients:



2862

2863 In most of the cases specific migration testing can be performed with the screening food
2864 simulants ethanol 95% for substances with an octanol to water partition coefficient $\log K_{O/W} <$
2865 14 and with iso-octane for substances with an octanol to water partition coefficient $\log K_{O/W} >$
2866 26 . There is a gap for substances exhibiting a polarity similar to vegetable oil which is too far
2867 from that of ethanol 95% but not close enough to that of iso-octane. In these cases it is
2868 considered that iso-octane is the suitable screening food simulant, provided the residual content
2869 of the substance in the material is below 100 mg/dm^2 of plastic material investigated, because in
2870 this case solubility of the migrant in iso-octane is expected to be sufficiently high.

2871 Finally solubility of substances in liquid media strongly depends on temperature. Because most
2872 of the literature data cited are at or below 60°C it is considered that the recommendations made

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

2873 above are valid up to a maximum test temperature of 70°C. Above 70°C the use of screening food
2874 simulants in many cases will induce physical changes of the materials investigated and from
2875 laboratory point of view their use above 70°C is dangerous due to their flammability. At higher
2876 temperatures (above 100°C) it is considered that food simulant E might be the more appropriate
2877 screening food simulant for vegetable oils.

2878 The closer the polarity of the screening food simulant to the polarity of the plastic is, the higher
2879 is the risk of interaction between polymer and simulant, i.e. swelling of the polymer by uptake of
2880 screening food simulant. Depending on (a) the amount of simulant taken up by the polymer and
2881 (b) the extent of the plasticising effect related to the amount of simulant taken up, an increase of
2882 the migration rate compared to testing with vegetable oil can be expected.

2883 The swelling effect may open the possibility for deviations from the conventional time and
2884 temperature testing conditions for vegetable oil when testing with screening food simulants.
2885 This option will be discussed below in the kinetic considerations section in more detail.

2886

2887 Screening food simulants selection for overall migration testing

2888 Under the assumption, that the nature of the migrating substances contributing to the overall
2889 migration from the plastic is known, the considerations for screening simulant selection made
2890 for specific migration testing are applicable as well.

2891 Regarding screening food simulants for overall migration testing and in the view of the fact that
2892 overall migration testing is an inertness test without toxicological relevance it is considered that
2893 the testing scheme based on use of ethanol 95% and iso-octane established with Directive
2894 82/711/EEC including amendments is still feasible due to its long term use and existing
2895 experience.

2896 The two solvents ethanol 95% and iso-octane span the polarity range of migrants from plastics
2897 encountered in practice. Substituting the overall migration test with vegetable oil requires
2898 testing with both solvents under consideration of the highest result for compliance evaluation.
2899 Regarding ethanol 95% its polarity is much higher compared to vegetable oil, reason for which
2900 solubility of non-polar migrants in ethanol 95% is expected to be lower compared to olive oil and
2901 as a consequence contribution of non-polar migrants (e.g. polyolefine oligomers or typical
2902 antioxidants) to overall migration may be underestimated. Regarding isooctane its polarity is
2903 lower compared to olive oil, reason for which solubility of polar migrants in isooctane is
2904 expected to be lower compared to vegetable oil and as a consequence contribution of polar
2905 migrants (e.g. polyamide oligomers or residual monomers) to overall migration may be
2906 underestimated.

2907 To minimize the risk of underestimation testing with both screening food simulants is requested
2908 and the highest migration result needs to be considered for compliance evaluation.

2909 However the use of ethanol 95% and isooctane as screening food simulants was historically
2910 associated with time/temperature conditions which deviate from those for vegetable oil. This
2911 turned out to be a serious source of underestimation especially for testing temperatures above
2912 40°C.

2913 Possible deviations from the conventional time and temperature testing conditions for vegetable
2914 oil when testing with screening food simulants will be discussed below in the kinetic
2915 considerations section.

2916

2917 **Kinetic considerations (time/temperature conditions)**

2918 Food contact materials most likely interact with foods to a given extent if they come into contact.
2919 Interaction means that materials may release components to the contact medium (migration)
2920 and/or may take up components from the contact medium or part of the contact medium itself.
2921 The migration rate of components from the material in most of the cases is diffusion controlled
2922 and may depend on the uptake rate and amount related to the contact medium.

2923 In most food contact material applications the extent of polymer swelling by the packaged food
2924 is low, but there are also examples where the extent of swelling is very high like swelling of
2925 polyamide based sausage casings by water. Increase of migration rates due to swelling strongly
2926 depends on the nature of the polymer and the nature of the food or food simulant.

2927 Due to the fact that screening food simulants most likely are volatile media they may interact
2928 with the plastic material during the migration test, i.e. take up simulant in the material to a given
2929 extent. Depending on polarity match between plastic and contact medium the uptake of simulant
2930 in the material is higher due to similar polarity and consequently the migration rates of
2931 components out of the material will increase due to swelling, i.e. the plasticising effect on the
2932 plastic material caused by the contact medium.

2933 Because screening food simulants are volatile media they may not be used for migration testing
2934 at high temperatures. Instead time and temperature conditions for migration testing (both
2935 overall and specific) should be selected according to the formula given in Annex V under 2.1.4,
2936 i.e. lower testing temperature at longer testing time.

2937 As a consequence one might tend to lower test time and/or test temperature to account for the
2938 swelling effect. However from today's knowledge point of view there are two main effects during
2939 swelling: (a) the amount of simulant taken up by the material and (b) the extent of the
2940 plasticising effect related to the amount of simulant taken up.

2941 At the moment there is limited scientific background available to make a general
2942 recommendation regarding selection of time and temperature conditions for migration testing
2943 with respect to the possible combinations of plastic materials and screening food simulants.

2944

2945 Interaction of plastics with screening food simulants

2946 Working with screening food simulants instead of vegetable oil most likely involves working
2947 with volatile media (solvents). Depending on the polarity of the plastic and the polarity of the
2948 solvent interaction between plastic and solvent may occur. The closer the polarity of plastic and
2949 solvent is the higher the extent of interaction, i.e. swelling will be. Due to swelling the diffusion
2950 controlled release of components from the plastic will increase. This effect is best described for
2951 PVC where the polymer specific constant (A_p -value) will change when adding a highly effective
2952 plasticiser to rigid PVC. An upper limit A_p -value of -1 is observed for rigid PVC and an upper limit
2953 A_p -value of 14.5 is observed for PVC plasticised with 30% Dioctylphthalate. Due to that diffusion
2954 rates of component from plasticised PVC will be more than ten orders of magnitude faster
2955 compared to rigid PVC.

2956 During the migration test of a plastic with a solvent under swelling conditions in fact two
2957 parallel processes will take place, i.e. the diffusion controlled uptake of the solvent under non-
2958 swollen conditions which will induce the diffusion controlled release of the component under
2959 swollen conditions. Because solvents are small molecules in most of the cases they will diffuse
2960 faster into the plastic than the bigger component out of the plastic. As a consequence overall a
2961 diffusion like release of the component from the plastic under swollen conditions will be
2962 observed. In terms of polymer specific constants the A_p -value for the non-swollen polymer and
2963 the A_p -value for the swollen polymer will differ significantly. The described behavior will be
2964 shown for the example polyamide 6 (PA6) when tested with aqueous food simulants, where
2965 water, acetic acid 3% and ethanol 10% are swelling the polymer during testing.

2966 • Example: polyamide, PA6

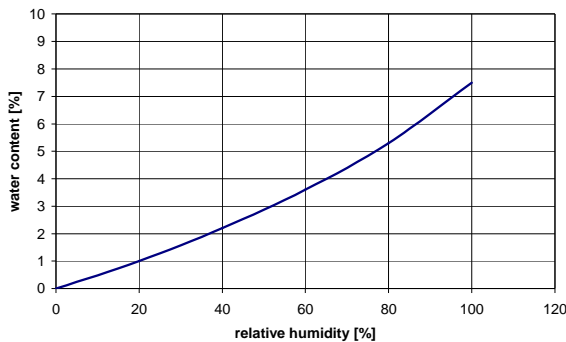
2967 Polyamides are plastic materials widely used for food contact applications. They are very polar
2968 compared to other plastics like polyolefines, polystyrene or polyethyleneterephthalate and
2969 hence prone to interact with foods. Prone to interaction with foods mean uptake of water from
2970 the food which on one hand will change the mechanic properties of the material and on the
2971 other hand will change its migration properties. From mechanical point of view water uptake
2972 will increase the flexibility of polyamides because water exhibits a plasticising effect.

2973 Because of the complex migration behaviour of the PA6 film additional migration investigations
 2974 with the aqueous food simulants water, 10% ethanol and 3% acetic acid were performed to
 2975 establish a systematic view on the migration properties of polyamide in support of migration
 2976 modelling. The work is focused on Caprolactam migration from a PA6 film but more general
 2977 conclusions on the migration behaviour of PA6 can be drawn.

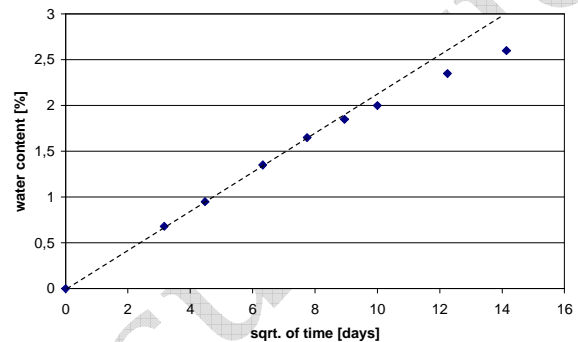
2978 >> Water uptake of PA6

2979 Due to its high polarity PA6 will take up significant amounts of water from the environment or
 2980 contact medium. The water uptake of PA6 depends on the relative humidity of the surrounding
 2981 air. Typical values are reported in the technical documentation of leading PA6 producers and are
 2982 in the range given in Figure 1 [11].

2983 (a)



(b)

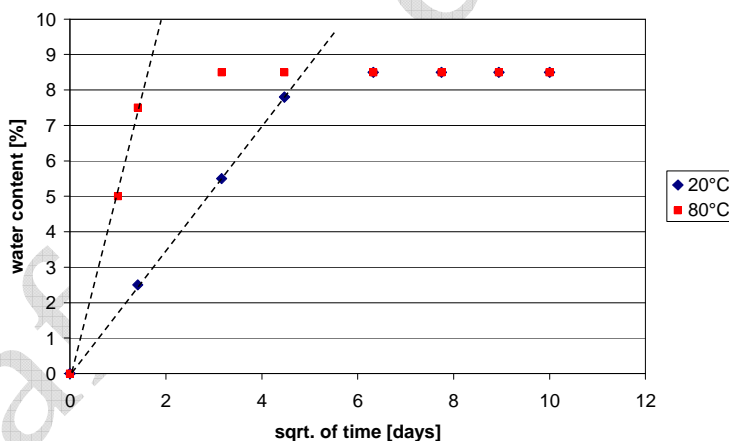


2984

2985 Figure 1 (a) Water content of PA6 at equilibrium in dependence of the relative humidity
 2986 (b) Water uptake of PA6 (2 mm thickness) with time at 23°C and 50% rel. humidity

2987

2988 The water uptake rate of PA6 is strongly time and temperature dependent and much higher
 2989 water uptake rates result if water comes in direct contact with PA6 as shown in Figure 2.



2990

2991 Figure 2 Water uptake of PA6 (2 mm thickness) with time at 20°C and 80°C in direct
 2992 contact with water

2993 The linear dependence of the water content from the square root of time indicates that the water
 2994 uptake is diffusion controlled. PA6 will interact with aqueous foods (liquid or semi-solid) similar
 2995 to pure water when used as direct food contact material.

2996

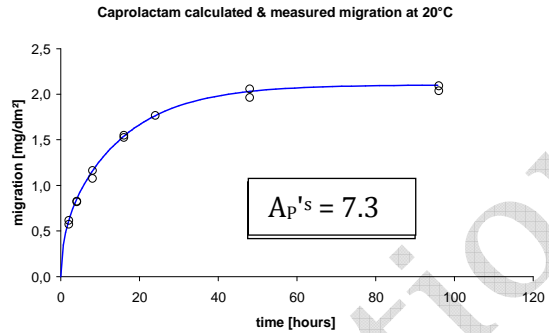
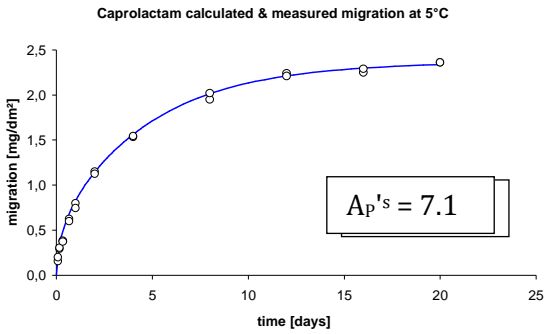
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¹¹ BASF Schrift KTEM 0401 BD Ultramid /Capron www.basf.de/ultramid

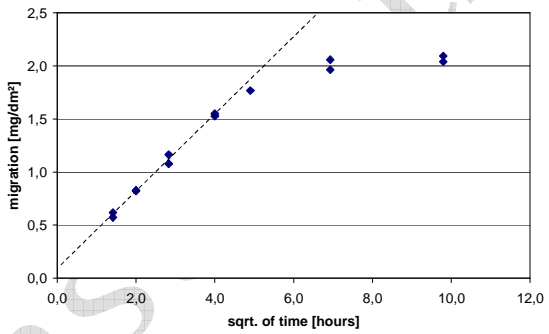
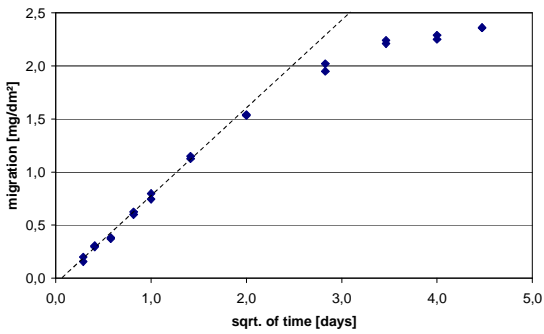
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2999 >> Migration kinetics of caprolactam from PA6 film into aqueous food simulants

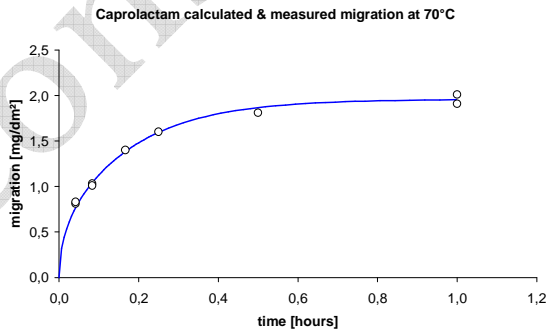
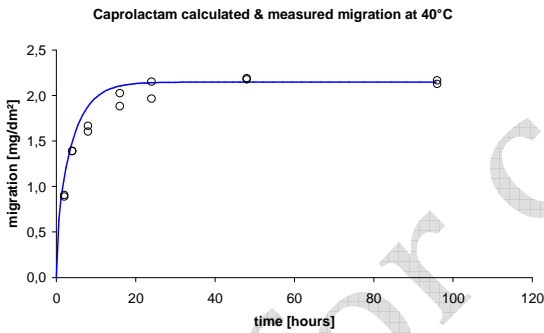
3000 Migration into water at 5°C to 70°C:



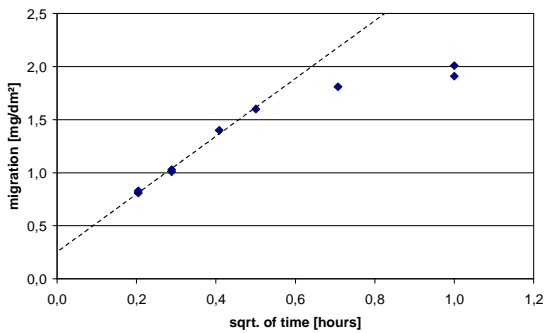
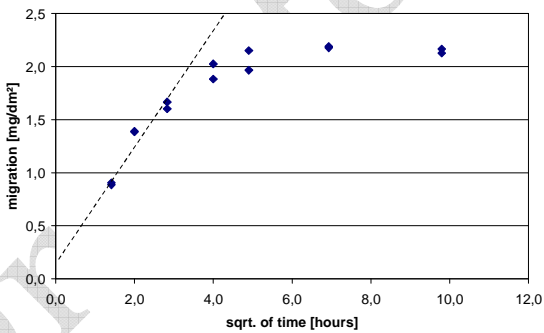
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3002



3003



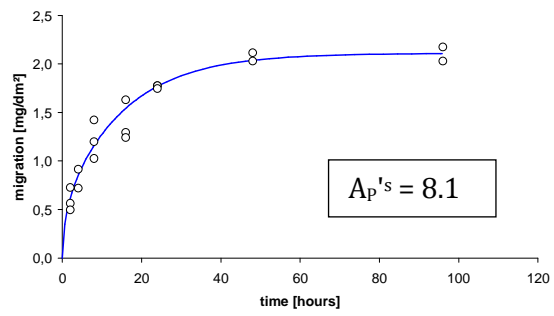
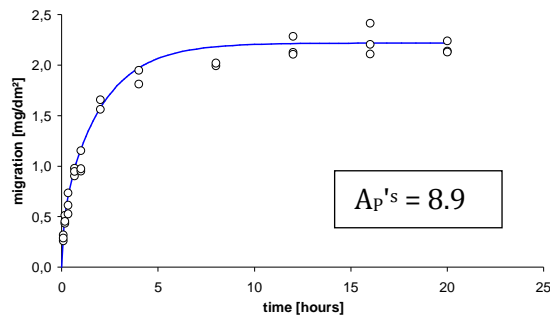
3004

3005 It can be observed that with increasing temperature the linear regression of the migration
3006 kinetic when related to the square root of time (up to approximate 60% of the equilibrium
3007 value) deviates more and more from the origin which is an indication that the rate of the two
3008 processes (a) diffusion controlled sorption of the simulant (manly water) in the non-swollen
3009 PA6 and (b) diffusion controlled release of caprolactam from the swollen PA6 are getting closer.

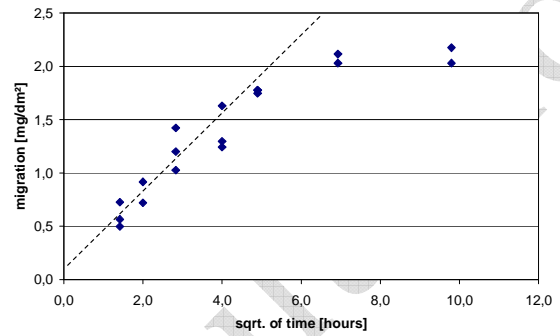
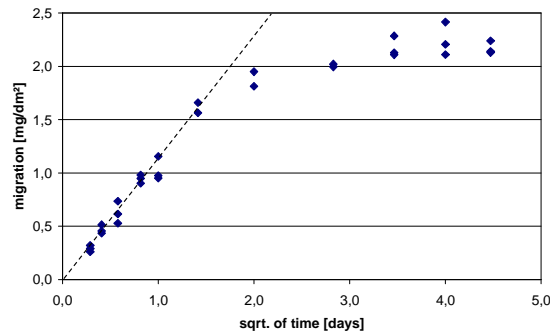
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3011

3012 Migration into acetic acid 3% at 5°C and 20°C:

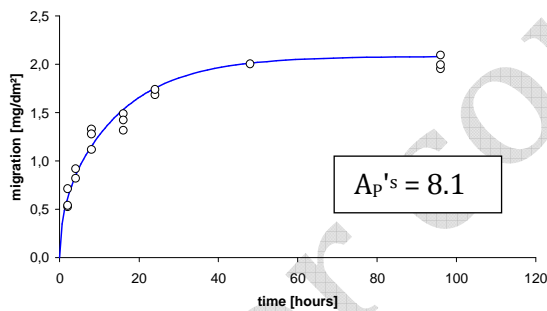


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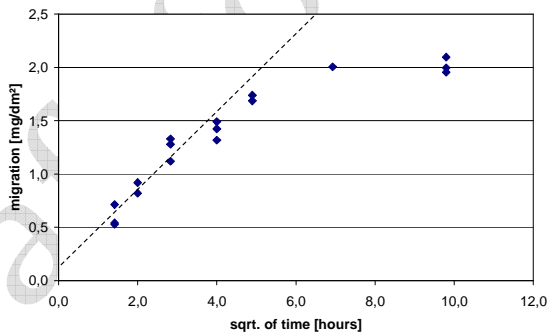


3014
3015

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 were performed for
3024 caprolactame. A summary of the results is given in the table below.

¹² EU-Project "Certified Reference Materials"

3025
3026

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	$C_{p,0}$ mg/kg	SM mg/dm ³	D_p			A_p			K_{FF}		
			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.

3027

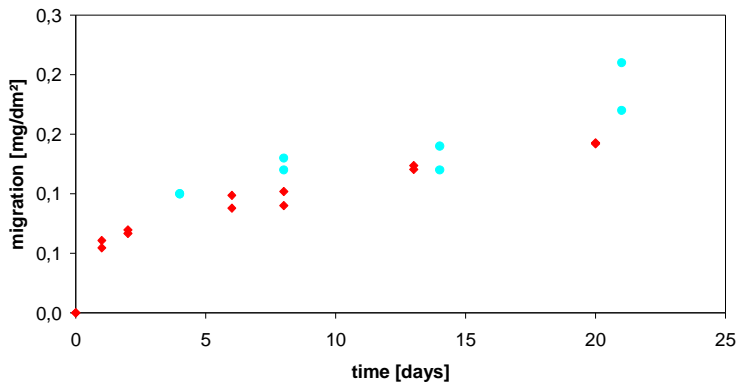
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3029

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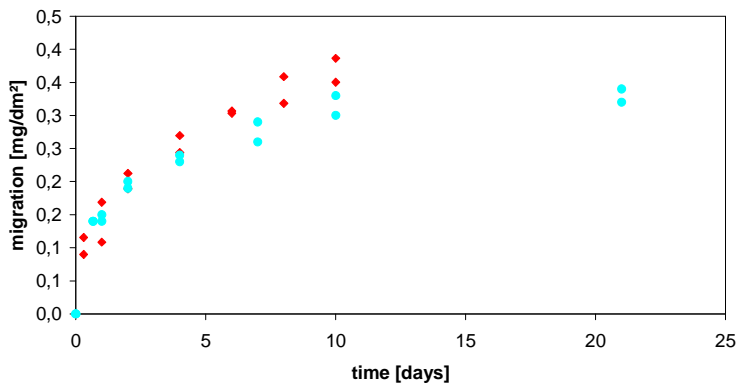
Some representative migration kinetics for caprolactam from PA6 into iso-octane at different temperatures (40°C, 60°C and 80°C) are given in the figures below.

PA6 diffusion caprolactam (40°C)

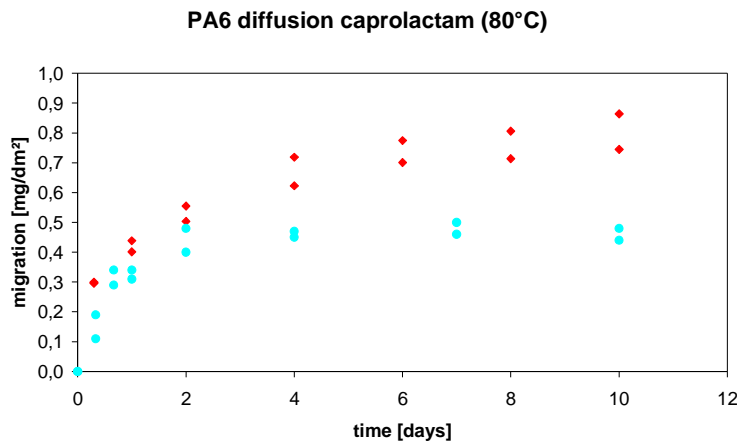


3031

PA6 diffusion caprolactam (60°C)



3032



3033

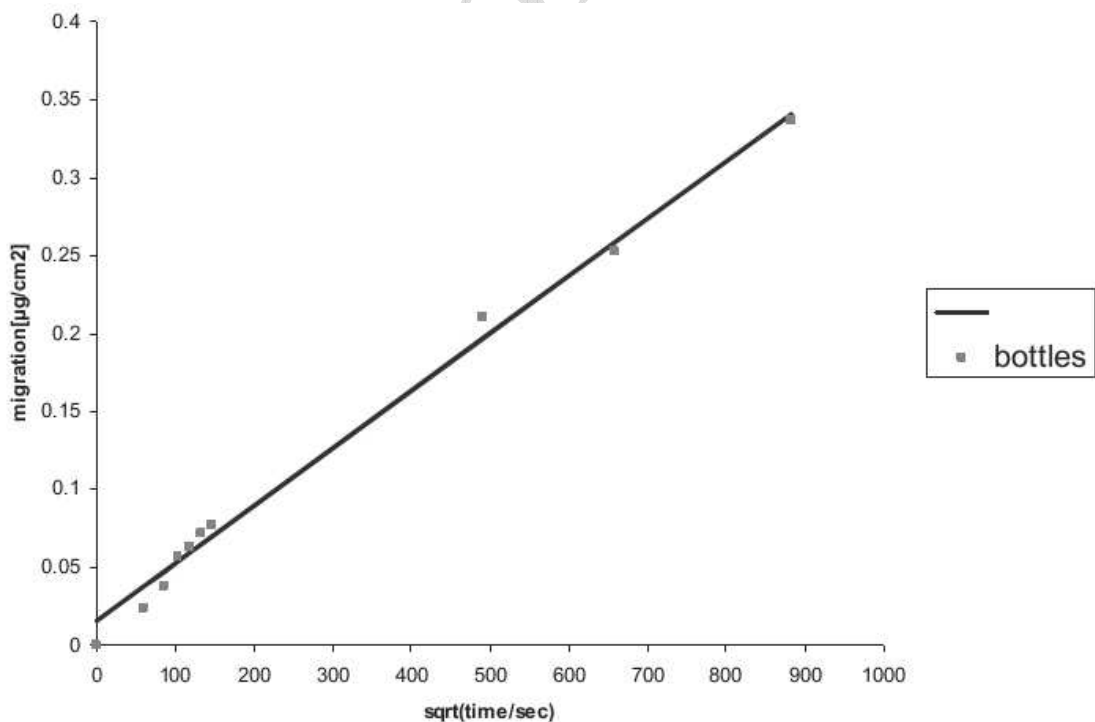
3034 Comparing the A_p' -value at 40°C for non-swollen PA6 and PA6 swollen by water an increase of
 3035 up to 9 units (from $A_p' = -1$ to $A_p'^s = 8.1$) is observed. A similar swelling behavior can be expected
 3036 for ethanol 95% and ethanol/water mixtures.

3037

3038 • Example Polyethylene terephthalate (PET)

3039 The migration characteristics of the UV stabilizer Tinuvin 234 (2-(2H-benzotriazol-2-yl)-4,6-bis
 3040 (1-methyl-1-phenylethyl)phenol) into food simulants has been measured from polyethylene
 3041 terephthalate (PET) using HPLC with UV detection. Ethanol/water, isooctane and a fractionated
 3042 coconut oil simulant (Miglyol) were used as food simulating solvents. The migration
 3043 characteristics were measured at temperatures in the range of 40–70C [13].

3044



3045 *Figure 3. Migration of Tinuvin 234 into 95% ethanol at 60°C from PET bottles.*

3046

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

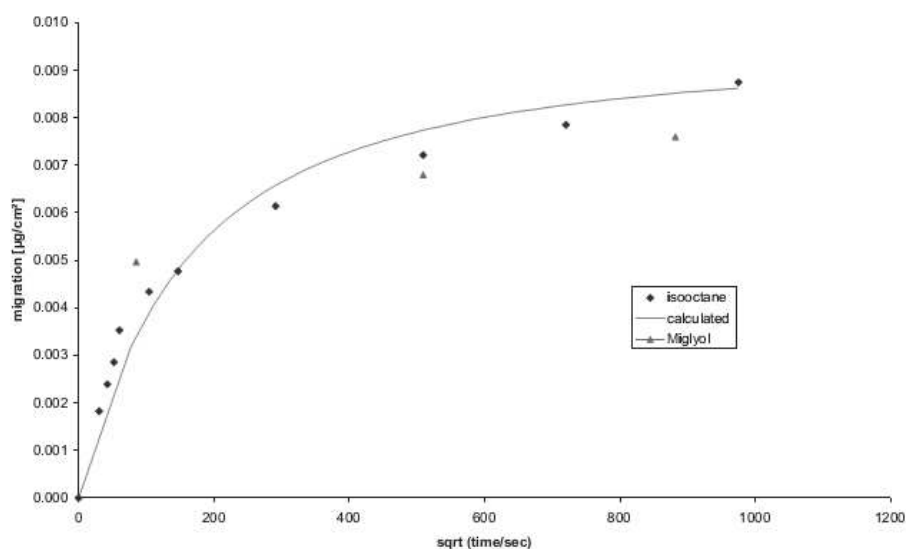


Figure 4. Migration of Tinuvin 234 into isooctane at 60°C from rectangular PET strips. Data represented by triangles (▲) are for migration experiments conducted from rectangular PET strips into Miglyol. The solid line is a calculated migration result according to equation (1).

3047

3048

3049 The authors conclude that: "Using the short-time isooctane migration data, the magnitude of the
 3050 diffusion of T234 in PET is determined to be $\sim 3 \times 10^{-16} \text{ cm}^2/\text{s}$. This diffusion value is
 3051 significantly less than the $1 \times 10^{-14} \text{ cm}^2/\text{s}$ value determined at 60°C with ethanol, indicating that
 3052 ethanol has an interaction with the PET causing diffusion to be faster. The apparent diffusion of
 3053 T234 in PET in the presence of 95% ethanol is ~ 33 times faster than to isooctane."

3054 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

3055

3056 It can be concluded that swelling of PET by ethanol 95% will increase the polymer specific value
 3057 for PET by 3.5 units (from $A_p' = -4.1$ to $A_p'^s = -0.6$)

3058

3059 • Example Polyolefines (LDPE, HDPE, PP)

3060 For polyolefines in the swollen state no systematic kinetic data are available. However if for
 3061 LDPE testing conditions 2 days at 20°C with iso-octane were considered to be equivalent to

3062 testing conditions 10 days at 40°C with olive oil [14] it can be concluded that swelling of LDPE by
 3063 iso-octane will increase the polymer specific value for LDPE by 5.5 units (from $A_{P'} = 10$ to $A_{P's} =$
 3064 15.5). A similar swelling behavior for iso-octane can be expected for HDPE and PP, however the
 3065 impact of their crystallinity on swelling is not known.

3066 **Conclusion**

3067 If for a plastic material both (a) the polymer specific constant in the non-swollen ($A_{P'}$) state
 3068 assumed to be applicable when in contact with vegetable oil and (b) the polymer specific
 3069 constant in the swollen state ($A_{P's}$) applicable when in contact with the screening food simulant
 3070 are available, time and temperature conditions to be used for testing with vegetable oil can be
 3071 translated into time and temperature conditions to be used for testing with the screening food
 3072 simulant.

3073

3074 **Testing specific migration with screening food simulants**

3075 Selection of screening food simulants for specific migration testing should follow the criteria
 3076 defined for ratio^K described above. As a first recommendation the same time/temperature
 3077 conditions for specific migration testing with screening food simulants should be used as those
 3078 to be used with vegetable oil.

3079 If the screening food simulants to be used are ethanol 95% or iso-octane the following
 3080 time/temperature conditions are considered to be suitable under consideration of polymer
 3081 swelling:

3082 **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

				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°	

3083

3084 Time-temperature conditions recommended for screening food simulants deviate from the
3085 conventional ones used for vegetable oil due to swelling.

3086

3087 **Testing overall migration with screening food simulants**

3088 Based on the above conclusion the following time/temperature conditions for overall migration
3089 testing with screening food simulants are recommended:

3090

3091 **Table 3 overall migration test conditions for screening food simulants**

plastic	vegetable oil	ethanol 95%	iso-octane
LDPE, LLDPE	OM2	OM2	2d @ 20°C
PP random			1d @ 40°C
PP rubbery	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	OM2
		1d @ 50°C	
	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

3092

3093 Time / temperature conditions recommended for screening food simulants deviate from the
 3094 conventional ones used for vegetable oil due to swelling.

3095

3096

3097

3098

3099 **Annex 7 Test method for overall migration into vegetable**
 3100 **oil**

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3138

3139 This annex describes the test method for overall migration into vegetable oil. It consists of four
3140 main sections:

- 3141 • Test method for overall migration into vegetable oil in the temperature range of 20-100°C
- 3142 • Test method for overall migration into vegetable oil in the temperature range of 5-20°C
- 3143 • Test method for overall migration into vegetable oil in the temperature range of 100-175°C
- 3144 • Test method for overall migration into vegetable oil in case of incomplete extraction of
3145 vegetable oil in the temperature range of 5-175°C

3146 Testing at low temperatures

3147 Testing with oil at 5°C may lead to technical problems if the oil partially solidifies.

3148 Sunflower oil, which is free of components which solidify at the temperature of test (i.e. a “de-
3149 waxed” oil), may be used.

3150 However, with olive oil and sunflower oil the test is usually without this problem at 10°C. If the
3151 overall migration does not exceed the limit when tested at 10°C this indicates that it would not
3152 have exceeded the limit at 5°C.

3153 Testing by total immersion or in a cell or in a pouch is practicable at low temperatures, although
3154 if a cell or pouch is used for the vegetable oil where a visual check on solidification is difficult, a
3155 de-waxed vegetable oil shall be used.

3156 The method of test for the determination of overall migration at low temperatures in the range
3157 of 5-20°C is given in section 7.2.

3158 Testing at high temperature

3159 In practice, severe difficulties have been found in obtaining consistent and comparable results in
3160 inter-laboratory trials with the test conditions for simulating contact at temperatures of use in
3161 excess of 121°C. The main source of inconsistency appears to be due to variation in the time
3162 required to achieve the test contact temperature with olive oil and other fatty food simulants.
3163 Various options such as heating of sample tubes in electrically heated cells, etc. are under
3164 investigation as possible solutions to the problem. These have been incorporated into the
3165 methods described in section 7.3.

3166 **7.1 Test method for overall migration into vegetable oil in the temperature** 3167 **range of 20-100°C**

3168 **7.1.1 Scope**

3169 This Annex describes a test method for the determination of the overall migration into vegetable
3170 oil from plastics materials and articles. The test method is applicable for temperatures above
3171 20°C and up to, but not including, 100°C. The method describes four different ways to perform
3172 the migration, i.e. immersion, filling, pouch forming and filling and cell for one-sided contact.

3173 The immersion method is most suitable for plastics in the form of films and sheets, but can be
3174 applied to a wide range of articles or containers, from which test pieces of a suitable size can be
3175 cut.

3176 The cell method is most suitable for plastics in the form of films and sheets, but is particularly
3177 applicable to those materials consisting of more than one layer or surfaces that differ in their
3178 migration characteristics and that have to be tested with food simulant in contact with the
3179 surface which is intended to come into contact with foodstuffs.

3180 The pouch method is most suitable for plastics in the form of films and sheets, which are
3181 sealable by heat or pressure, but it is particularly applicable to those materials consisting of

3182 more than one layer, which has to be tested with the food simulant in contact only with the
3183 surface which is intended to come into contact with the foodstuffs.

3184 The filling method is most suitable for plastics in the form of containers and articles that can be
3185 filled. Testing samples by this method enables testing of non-homogenous articles provided they
3186 are not too large.

3187 NOTE – This test method has been written for use with rectified olive oil. The test method can
3188 also be used with appropriate modifications with other vegetable oils as defined in Regulation
3189 (EU) No 10/2011. These other vegetable oils will produce different chromatograms for the
3190 simulant methyl esters to those of the methyl esters of rectified olive oil. Select suitable
3191 chromatogram peaks of the methyl esters of the other vegetable oils for the quantitative
3192 determination of the simulant extracted from the test specimens.

3193 The test method described is applicable to most types of plastics, although there are some
3194 plastics for which it is known not to be applicable.

3195

3196 **7.1.2 Principe**

3197 The overall migration from a sample of the plastics is determined as the loss in mass per unit of
3198 surface area intended to come into contact with foodstuffs.

3199 The selection of the test conditions will be determined by the conditions of use as described in
3200 Regulation (EU) No 10/2011.

3201 Test specimens of known mass are immersed in, filled with or put in contact to rectified olive oil
3202 at temperatures above 20°C and below 100°C for a relevant contact time during which
3203 substances may migrate into the olive oil. Next, the oil is removed. The test specimen, are blotted
3204 to remove oil adhering to the surface, and reweighed. Pouches are cut open before blotting.

3205 The test specimens will usually retain absorbed oil that is extracted and determined
3206 quantitatively by means of gas chromatography after conversion to methyl esters. Methylation is
3207 carried out by hydrolysing the oil with potassium hydroxide followed by methylation with a
3208 boron trifluoride/methanol complex. An internal standard, triheptadecanoin, is added prior to
3209 the extraction of the absorbed oil from the test specimens. This ensures that any active or
3210 extractable component of the plastic that reacts with the extracted oil also reacts with the
3211 internal standard. The internal standard is also subjected to hydrolysis and methylation,
3212 providing compensation for any inefficiency in the hydrolysis and methylation processes.

3213 Migration into the oil is calculated by subtracting the mass of oil retained by the test specimen
3214 from the mass of the test specimen after removal from the oil, then subtracting this mass from
3215 the initial mass of the test specimen.

3216 The total loss in mass is expressed in milligrams per square decimetre of surface area of the test
3217 specimen.

3218 To allow for inaccuracies which may arise during the procedure and which may be difficult to
3219 detect, due for example to contamination or loss of oil during the sample handling stages, four
3220 determinations are carried out on the sample allowing for one failing result.

3221 This method includes variations that are applicable to certain plastics.

3222 NOTE – Before starting a migration exercise, the test sample should be examined for the
3223 presence of substances interfering in the determination of the amount of extracted oil (see
3224 Annex 0). If an unacceptable amount of interference is present then suitability of one of the
3225 other vegetable oils should be examined. If an interference is present which would interfere
3226 with the triheptadecanoin internal standard an alternative internal standard should be used.

3227

3228 **7.1.3 Reagents**

3229 NOTE – All reagents should be of recognized analytical quality, unless otherwise specified.

3230 NOTE – Specifications for solid reagents, used as such in discrete quantities, may not address
3231 suitability for use in methods of analysis in this Annex. Solid reagents might not be
3232 homogeneous with respect to contaminants not addressed by specifications, therefore it may be
3233 necessary to demonstrate that such reagents are suitable for use.

3234 **7.1.3.1 Rectified olive oil (see section 4.2.3)**

3235 Some substances which may migrate from plastics are capable of interfering with the gas
3236 chromatographic method for the determination of olive oil, e.g. glyceryl oleates. When testing
3237 articles containing these substances they may be tested with other vegetable oils, such as
3238 sunflower oil, corn oil, coconut oil, palm kernel oil.

3239 NOTE: The refining or rectifying process will remove interfering substances and unsaponifiable
3240 matter and free fatty acids. In general, oil containing less than 1% of unsaponifiable
3241 matter and/or free fatty acids will be suitable for all migration experiments provided the
3242 oil is stored in the dark at refrigerated or frozen conditions until it is used.

3243 **7.1.3.2 Extraction solvent**

3244 For non-polar plastics such as polyethylene and polypropylene:

3245 - Pentane (98 %; boiling point 36°C)

3246 For polar plastics, such as polyamide and polyacetal:

3247 - 95/5 v/v azeotropic mixture of pentane (98 %) and ethanol (99 %).

3248 NOTE 1 – Pentane is a very volatile and highly flammable solvent. Care should therefore be
3249 taken when handling this solvent to prevent contact with ignition sources. Ethanol is also a
3250 flammable solvent. It is not recommended that extractions with either pentane or
3251 pentane/ethanol mixture be left unattended, particularly overnight.

3252 NOTE 2 – Due to the low boiling points of the solvents, cooled condenser water may be required
3253 to prevent undue loss of solvent from the condenser.

3254 NOTE 3 – Some solvents can contain non-volatile substances which, after hydrolysis and
3255 methylation processes, produce gas chromatography peaks with retention times similar to the
3256 retention times oil methyl esters and methyl heptadecanoate from the internal standard.
3257 Solvents found to contain such substances should be redistilled before use.

3258 **7.1.3.3 Internal standard**

3259 Triheptadecanoin (glyceryl trimargarate; CAS No, 2438-40-6) of a quality such that the products
3260 from hydrolysis and methylation processes do not contain substances giving detectable gas
3261 chromatography peaks with similar retention times to the oil methyl ester peaks. Prepare a
3262 solution containing 2.0 mg/ml in cyclohexane.

3263 Other migrating substances may give rise to peaks in the gas chromatogram which interfere
3264 with the internal standard peak. Alternative internal standards such as hydrocinnamic acid,
3265 ethyl ester or trionadecanoin may be used in such cases.

3266 **7.1.3.4 Potassium hydroxide solution**

3267 Prepare a solution of 11.0 g/L in methanol

3268 **7.1.3.5 Boron trifluoride/methanol complex**

3269 Prepare a solution of approximately 150 g/L BF₃ in methanol.

- 3270 **7.1.3.6 n-Heptane**
- 3271 **7.1.3.7 Sodium sulphate**
- 3272 7.1.3.7.1 Sodium sulphate, anhydrous, Na₂SO₄
- 3273 7.1.3.7.2 Sodium sulphate, saturated solution
- 3274 **7.1.3.8 Diethyl ether**
- 3275
- 3276 **7.1.4 Apparatus**
- 3277 **7.1.4.1 Cutting slab**
- 3278 Clean smooth glass, metal or plastics slab of sufficient area to prepare test specimens, 250 mm x
3279 250 mm is suitable.
- 3280 **7.1.4.2 Tweezers, stainless steel, blunt nosed.**
- 3281 **7.1.4.3 Cutting implement, scalpel, scissors, sharp knife or other suitable device.**
- 3282 **7.1.4.4 Metal templates**
- 3283 Size 100 ± 0.2 mm x 10 ± 0.2 mm (square).
- 3284 **7.1.4.5 Rule or template**
- 3285 Size 25 ± 1 mm wide.
- 3286 **7.1.4.6 Rule, graduated in millimetres**
- 3287 with an accuracy of 0.1 mm.
- 3288 **7.1.4.7 Analytical balance**
- 3289 capable of determining a change in mass of 0.1 mg.
- 3290 **7.1.4.8 Test specimen supports**
- 3291 Constructed of stainless steel with cross arms attached by welding or silver soldering. Stainless
3292 steel X4 CrNi 18 10 according to EN 10088 or of composition, chromium 17%, nickel 9%, carbon
3293 0.04%, is suitable. Before initial use thoroughly clean the steel supports. The use of a degreasing
3294 solvent and then diluted nitric acid has been found to be suitable.
- 3295 NOTE – The method has been written for the supports shown in Figure 3 that have been found
3296 to be suitable for holding thin film and sheet test pieces. However other supports can be used
3297 providing they are capable of holding and keeping the test pieces apart and at the same time
3298 ensuring complete contact with the food simulant. For rigid samples, supports with a single
3299 cross arm can be used.
- 3300 **7.1.4.9 Gauze**
- 3301 Pieces of fine stainless steel gauze, with a mesh size of 1 mm have been found to be suitable,
3302 approximately 25 mm x 100 mm for insertion between the test pieces on the supports. Before
3303 initial use thoroughly clean the gauze, first with a degreasing solvent and then with diluted nitric
3304 acid.
- 3305 **7.1.4.10 Conditioning containers**
- 3306 For conditioning test specimens at 50% ± 5% relative humidity and 80% ± 5% relative humidity
3307 at 20°C ± 5°C.
- 3308 NOTE – For 50% relative humidity, 43% w/v sulphuric acid solution in water is suitable and for
3309 80% relative humidity, 27% w/v sulphuric acid solution is suitable. The solutions should be

3310 freshly prepared by adding a weighed amount of acid to a suitable volume of water, cooling to
3311 room temperature and making up to the required volume. It is recommended that relative
3312 humidity and temperature be maintained during the conditioning period. Therefore the
3313 containers should be placed in a thermostatically controlled room or oven, at a temperature of
3314 approximately 20°C, the set temperature should not vary by more than ± 1°C.

3315 **7.1.4.11 Glass tubes**

3316 With ground neck and stoppers, for retaining the oil and test specimens. Tubes with an internal
3317 diameter of approximately 35 mm and length in the range of 100 mm to 200 mm, excluding the
3318 ground neck have been found to be satisfactory.

3319 Note – the samples are tested at a fixed ratio of surface area of test specimen to food simulant
3320 volume. In order to ensure that all parts of the test specimen are in contact with the food
3321 simulant, glass tubes of the appropriate diameter are used. Minor adjustments to the level of the
3322 simulant in the tubes may be made by adding glass rods or glass beads sufficient to ensure
3323 complete immersion of all of the surfaces of the test specimen. Again the dimensions of suitable
3324 glass rods and glass beads are specified in the individual methods.

3325 **7.1.4.12 Thermostatically controlled oven or incubator**

3326 capable of maintaining the set temperature within the tolerances specified in Table 5.

3327 NOTE: An oven with forced air circulation (e.g. GC oven) has been found very suitable and
3328 capable to maintain the required temperature very accurate. In addition heat transfer is
3329 increased.

3330 **7.1.4.13 Filter paper, lint-free**

3331 **7.1.4.14 Anti-bumping beads**

3332 **7.1.4.15 Soxhlet type extractors**

3333 capable of holding test specimens on the supports, with 250 ml or 500 ml round bottom flasks
3334 10 fit.

3335 NOTE – Alternative extractors capable of satisfactorily extracting absorbed olive oil from the
3336 test specimens can be used.

3337 **7.1.4.16 Water bath**

3338 capable of holding the flasks of soxhlet type extractors (7.1.4.15)

3339 **7.1.4.17 Rotary evaporator or distillation apparatus**

3340 for evaporation and collection of the extraction solvent.

3341 NOTE Artificially cooled water can be necessary for efficient condensation of a low boiling point
3342 solvent.

3343 **7.1.4.18 Steam bath or water bath.**

3344 **7.1.4.19 Flasks**

3345 50 ml, long neck with condensers to fit, for methyl ester preparations.

3346 **7.1.4.20 Measuring cylinders**

3347 complying with the minimum requirements of ISO 4788, 500 ml, 250 ml, 100 ml, 25 ml, and 10
3348 ml. A 10 ml graduated syringe may be used in place of the 10 ml measuring cylinder.

3349 **7.1.4.21 Pipettes**

3350 complying with the minimum requirements of ISO 648, 5 ml and 10 ml.

3351 **7.1.4.22 Glass beads or rods**

3352 Beads: 2-3 mm in diameter; rods: 2-3 mm in diameter and approximately 100 mm long (see note
3353 7.1.4.11).

3354 **7.1.4.23 Gas chromatograph**

3355 equipped with flame ionisation detector and an appropriate column. When using a polar column,
3356 the major peaks of olive oil, such as C16:0, methyl hexadecanoate (methyl palmitate), C16:1,
3357 methyl 9-hexadecenoate (methyl palmitoate), C18:0, methyl octadecanoate (methyl stearate),
3358 C18:1, methyl 9- octadecenoate (methyl oleate), C18:2, methyl 9,12-octadecadienoate (methyl
3359 linoleate) and the internal standard C17:0, methyl heptadecanoate (methyl margarate) shall
3360 demonstrate baseline separation. Optionally, a non-polar column can be used which shall give
3361 baseline separation of the methyl esters with 16 and 18 carbon numbers and the internal
3362 standard with 17 carbon number.

3363 NOTE The following columns have been found to be suitable:

3364 - Column 1, polar column, WCOT fused silica column, length 50 m, internal diameter 0.25 mm,
3365 coated with a 0.21 µm film of cyanopropyl silicone; Column 1 is a column with a polar stationary
3366 phase that allows separation of the individual methyl esters of fatty acids according to their
3367 carbon number as well as their number of double bonds in the chain, e.g. the methyl esters of
3368 stearic acid is separated from the methyl esters of oleic acid and this is separated from the
3369 methyl esters of linoleic acid.

3370 - Column 2, non-polar column, 100% dimethyl polysiloxane, length 25 m, internal diameter 0.32
3371 mm, with a 1 µm film thickness; Column 2 is a column with a non-polar coating which allows
3372 only separation of the carbon number, e.g. no separation is obtained between the methyl esters
3373 of oleic acid and the methyl esters of stearic acid.

3374 Both types of columns have their own specific advantages and disadvantages. A gas
3375 chromatogram obtained with column 1 will reveal more information on the distribution of fatty
3376 acid in the olive oil extracted from the test specimen than with column 2. To determine the total
3377 area of the fatty acids using column 1, the area of at least 5 peaks may be measured and
3378 summed. With column 2 only 2 peaks have to be measured. On the other hand the determination
3379 will be more sensitive to interferences when using column 2. In the case where interferences
3380 occur on one of the minor peaks, when using column 1, it is possible to exclude that peak and to
3381 adapt the calibration graph for the excluded peak. It is even possible to measure only the major
3382 peak of oleic acid to quantify the total amount of oil, provided the calibration graph is
3383 constructed in the same way.

3384 NOTE A polar column is the preferred one

3385 **7.1.4.24 Glass tubes**

3386 with ground glass necks and stoppers, of a volume of approximately 10 ml, for storing the
3387 heptane layer if necessary.

3388 **7.1.4.25 Vacuum oven or vacuum desiccator**

3389 capable of maintaining a temperature of 60°C ± 2°C. The vacuum oven or vacuum desiccator
3390 shall be equipped with or connected to a vacuum pump capable of achieving a vacuum of 1,3 kPa
3391 or less. The vacuum pump shall be provided with a time controller to switch on the vacuum
3392 pump every hour for 15 min.

3393 NOTE If a vacuum oven is not available, a vacuum desiccator placed in an oven at 60°C can be
3394 used.

3395 **7.1.4.26 Desiccator**

3396 containing self-indicating silica gel or anhydrous calcium chloride,

- 3397 **7.1.4.27 Balance**
- 3398 capable of determining a change of mass of 10 mg.
- 3399 **7.1.4.28 Disposable plastic syringes**
- 3400 with luer fitting. 1 ml or 10 ml size.
- 3401 **7.1.4.29 Wide gauge luer needles**
- 3402 80 mm x 1.2 mm.
- 3403 **7.1.4.30 Cell**
- 3404 Migration cell as shown in Figure 5-Figure 10
- 3405 **7.1.4.31 Pouch holder**
- 3406 Figure 11 shows a pouch holder that is suitable. It is constructed from aluminium or other
3407 suitable material or an equivalent holder, plus clips to secure corners of pouches.
- 3408 **7.1.4.32 Heat or pressure sealing device**
- 3409 For use in forming pouches
- 3410 **7.1.4.33 Chromatography tank**
- 3411 Or any other airtight container for test sample storage.
- 3412 **7.1.4.34 Glass rods or metal gauze**
- 3413 For use as spacers between test pieces during solvent extraction.
- 3414 **7.1.4.35 Lint-free cloth**
- 3415
- 3416 **7.1.5 Preparation of test specimens**
- 3417 **7.1.5.1 General**
- 3418 The sample taken for testing is the final article, in its ready-for-use state. In some cases this may
3419 be impracticable and test specimens can be taken from the material, article or, where
3420 appropriate, test specimens representative of this material or article can be used.
- 3421 An example is where an article is filled with food at the time it is formed. In this case the test
3422 may be carried out on a test article prepared especially for testing purposes. This article shall be
3423 as representative as possible of the article in actual use.
- 3424 A further example is where the sample to be tested is of inhomogeneous construction and is too
3425 large to be tested by filling and no flat surfaces can be cut from the sample for testing in a cell. In
3426 this case the test may be carried out on a test article prepared especially for testing purposes.
3427 This article shall as representative as possible of the article in actual use.
- 3428 Where samples are taken at random from a production batch this shall be indicated when
3429 reporting the result. The samples shall be representative of normal production material.
3430 Similarly if the sample was not a random sample, and it was selected according to some other
3431 parameter, e.g. thickness variation, this shall also be reported.
- 3432 Samples may be inhomogeneous, e.g. varying in crystallinity or in molecular orientation, or of
3433 irregular shape or thickness, e.g. sections cut from bottles, trays, work surfaces, cutlery etc., or
3434 so small that several samples are required to constitute a test specimen. Replicate samples as
3435 similar as possible to each other and proportionally representing the sample article shall be
3436 tested and the sampling details shall be included in the final report.
- 3437 It is essential that test specimens are clean and free from surface contamination (many plastics
3438 can readily attract dust due to static charges). Before preparing test specimens, remove any

3439 surface contamination from the sample by gently wiping it with a lint-free cloth, or by brushing
3440 with a soft brush. Under no circumstances wash the sample with water or solvent. Minimise
3441 handling of the samples and, where necessary, wear cotton gloves.

3442 If articles are accompanied with an instruction that they should be cleaned before use then this
3443 instruction should be followed before testing. If, however, the instruction prescribes rubbing of
3444 the article with e.g. an oil, then this instruction should not be followed as the oil will contribute
3445 to the overall migration.

3446 To ensure that test pieces are well separated and that their surfaces are freely exposed to oil
3447 during the period of the test, for thin films insert a piece of fine stainless steel gauze (7.1.4.9)
3448 between the test pieces or for thick samples not placed on the supports, insert glass rods
3449 between the test pieces after immersion in the oil. Where test specimen supports are used, label
3450 the supports with a tag bearing the test specimen identification.

3451 7.1.5.1.1 Surface to volume ratio

3452 Where the surface to volume ratio to be used in contact with food is known this is used in the
3453 migration testing. An example of this is where a bottle or other container is intended to contain a
3454 specified volume of contents even if this does not completely fill the article. In this case the
3455 article is tested with the specified volume of simulant.

3456 Where the surface to volume ratio to be used in contact with food is not known conventional
3457 conditions are used, in the following sections.

3458 7.1.5.1.1.1 *Single surface versus double surface testing by total immersion*

3459 For verification of compliance section 4.4.3 of this document needs to be taken into account.
3460 Overall migration tests shall be performed in such a way that only those parts of the sample
3461 intended to come into contact with foodstuffs in actual use will be in contact with the foodstuff
3462 or simulant. In cases where the overall migration limit is exceeded when testing by total
3463 immersion, the test shall be repeated using a method applying single sided contact.

3464 However, it is permissible to demonstrate compliance with an overall migration limit by the use
3465 of a more severe test (screening). In the total immersion test, both the surface which is intended
3466 to come into contact with the foodstuff and the outside surface are in contact with the food
3467 simulant. No allowance is made for this in the calculation of migration per unit of surface area.
3468 Although the total surface exposed is 2 dm², only 1 dm², i.e. the food contact surface, is taken into
3469 account in the calculation. It is therefore a more severe test than testing in a pouch or in a cell or
3470 by filling. However, if it is possible to demonstrate experimentally that the value obtained in a
3471 total immersion test is double that obtained in a single surface test, the value obtained in the
3472 total immersion test shall be divided by the total surface area exposed.

3473 Test specimens with cut edges tend to give higher results than those without. In use, the plastics
3474 material or article would not normally have cut edges in contact with the foodstuff. The process
3475 of cutting may have an irreversible effect on the morphology of the edges of the sample. As a
3476 result, the obtained overall migration value is not a true reflection of the real migration under
3477 actual conditions of use. Therefore the number of cut edges shall be limited, where possible, and
3478 in the case that the overall migration limit is exceeded the test shall be repeated using a method
3479 applying single sided contact.

3480 If the area of the cut edges of the test specimen exceeds 10% of the measured area of the sample
3481 then this area has to be included in the calculation of the surface area used in the calculation of
3482 overall migration.

3483 Testing samples with the test specimens prepared by cutting sections from the plastic and
3484 totally immersing in the food simulant, is a more severe test.

3485 The surface to volume ratio in the total immersion test is conventionally 1 dm² of food contact
3486 area to 100 ml of food simulant.

3487 *7.1.5.1.1.2 Single surface testing using a cell*

3488 Where single surface testing is the preferred procedure, particularly important for multi-layer
3489 articles, this may be carried out in a cell. For samples that may be obtained in flat form, e.g. film
3490 or sheet, testing in the cell has the advantage of readily reproducible sample geometry.

3491 The surface to volume ratio in the type A cell as used in this method, is conventionally 2.5 dm² of
3492 food contact area to 125 ml of food simulant. Inter-laboratory trials carried out by experienced
3493 laboratories have shown that consistent overall migration results can be obtained using cell type
3494 A. Comparative studies carried out on the performance of cells type A, B, C, D, E and F revealed
3495 that these cells gave similar results. Therefore the cells referred to in Figure 5-**Figure 10** are
3496 considered equivalent.

3497 *7.1.5.1.1.3 Single surface testing by pouch*

3498 For flat articles which have sufficient seal strength to form durable pouches, single surface
3499 testing in a pouch may be preferred as this does not require specialized apparatus and allows
3500 more efficient use of oven space. Inter-laboratory collaborative testing studies using pouches of
3501 precisely specified dimensions have shown that variations in pouch geometry (particularly
3502 varying areas outside the seals) can lead to significant variability in the final result.

3503 The surface to volume ratio in the pouch is conventionally 2 dm² of food contact area to 100 ml
3504 of food simulant.

3505 NOTE Generally pouches are filled with preheated simulant. However for test temperatures
3506 above 40°C the pouches may be filled with food simulant at ambient temperature and then the
3507 test specimens preheated in a microwave oven to reach the test temperature. A procedure that
3508 has been found to be suitable is to insert into the simulant of one of the test specimens a fibre
3509 optic probe or to check the temperature after heating by a thermometer. The filled pouches are
3510 placed in a microwave oven and heated until the simulant has attained the test temperature. The
3511 test specimens are removed to a thermostatically controlled oven or incubator that is preheated
3512 to the test temperature. This part of the operation should be carried out in the minimum time to
3513 prevent undue heat loss. The pouches are left for the selected test period.

3514 *7.1.5.1.1.4 Single surface testing using a reverse pouch*

3515 As an alternative to using a pouch, a reverse pouch may be used. In this case the surface
3516 intended to come into contact with the foodstuff is the outer surface and the pouch is exposed to
3517 the food simulant by total immersion.

3518 The use of a reverse pouch offers advantages over the pouch. Since pouches are filled with food
3519 simulant, the sealed edges have to be capable of bearing the mass of that simulant; if they are not
3520 the seals give way and the pouches are prone to leakage. With the reverse pouch the seals do not
3521 have to withstand the pressure of the food simulant and consequently are less likely to leak and
3522 the sealed area can be reduced. The use of a reverse pouch permits a more accurate
3523 determination of the area exposed to food simulant. However, it is possible that food simulant
3524 may leak into the reverse pouch thus increasing the area exposed to food simulant. A way of
3525 checking if leaks have occurred, is to seal into the reverse pouch a piece of filter paper which is
3526 of similar dimensions to the pouch. If the pouch leaks the paper will absorb the food simulant
3527 and this will be visible. This method may not be applicable for overall migration into fatty food
3528 simulants, as the mass of the inserted paper may change during storage due to loss of water. Any
3529 pouch that leaks shall be discarded and the test shall be repeated.

3530 Where the surface to volume ratio to be used in contact with food is not known, the conventional
3531 conditions are used, i.e. 2 dm² of surface in contact with 100 ml of simulant.

3532 *7.1.5.1.1.5 Single surface testing by filling*

3533 For articles in container form, e.g. bottles and trays, it is often most convenient to test them by
3534 filling with food simulant. For very large containers testing by filling may not be practicable and
3535 it may be necessary to fabricate smaller test specimens representing the article to be tested.

- 3536 *7.1.5.1.1.6 Articles intended for repeated use*
- 3537 It is accepted that where a material or article is intended to come into repeated contact with
3538 foodstuffs, the migration tests are carried out three times on the same test sample in accordance
3539 with the conditions laid down, using a fresh sample of the food simulant on each occasion.
3540 However, if there is conclusive proof that the level of migration does not increase in the second
3541 and third test and if the migration limit is not exceeded on the first test, no further test is
3542 necessary.
- 3543 *7.1.5.1.1.7 Caps, closures and other sealing devices*
- 3544 Caps, sealing gaskets and other sealing devices shall be tested under conditions that, as far as
3545 possible, simulate actual conditions of use.
- 3546 The test is carried out on closures in the state and form in which they are intended to be used.
- 3547 The simulants are placed in jars, known to give only consistently low migration, and the jars are
3548 closed with the test closures. The jars are then inverted and subjected to the test conditions
3549 appropriate for the actual conditions of use. The surface to volume ratio used shall be the same
3550 as that intended for use.
- 3551 In many cases lids and closures may be expected to come into contact with foodstuffs and are
3552 tested under similar conditions to the rest of the container.
- 3553 *7.1.5.1.1.8 Large containers*
- 3554 Large containers, where filling is not practicable, may be tested by cutting test specimens from
3555 them and testing these by total immersion or by the cell method or using an equivalent cell.
3556 Alternatively, smaller test samples representing the large container may be fabricated and
3557 tested by filling.
- 3558 *7.1.5.1.1.9 Tubing, taps, valves and filters*
- 3559 Articles such as tubing, taps, valves etc. may be in contact with flowing foodstuff, this may be
3560 considered to be repeated brief contact for the purposes of migration testing. Such articles may
3561 be tested by repeated total immersion or by repeated filling; tubing may be stoppered with an
3562 inert stopper.
- 3563 *7.1.5.1.1.10 Fibres and cloths*
- 3564 Polymeric fibres and cloths are used to make such articles as sacks, filters, conveyor belting and
3565 bags for the infusion of beverages. In these circumstances it is not practicable to determine the
3566 surface area of the individual fibres in contact with the foodstuffs. Where limits of overall
3567 migration are expressed in milligrams per square decimetre of surface area the surface area may
3568 be taken as the superficial or projected area of the article.
- 3569 *7.1.5.1.1.11 Articles of irregular shape*
- 3570 Many articles that are required to be tested are of irregular shape or dimensions, e.g. thickness.
3571 Examples of these are sinks and work surfaces, eating and cooking utensils, shaped bottles and
3572 containers. When portions of these samples are taken for test by total immersion or in a cell care
3573 has to be exercised to ensure that the test specimens selected are representative of the whole of
3574 those parts of the article intended to come into contact with food. Also, care shall be taken to
3575 ensure that replicate test specimens are sufficiently dimensionally similar, one to another, to
3576 allow valid replication of results.
- 3577 *7.1.5.2 Number of test specimens*
- 3578 Five test specimens are required for samples, in the form of fillable article, thin films, sheets, cut
3579 sections from containers or similar articles. Seven test specimens, similar dimensionally one to
3580 another, are required for samples of articles of irregular shape.
- 3581 These test specimens are utilized as follows:

- 3582 a) four test specimens for the migration test;
- 3583 b) one test specimen to determine the suitability of oil as the fatty food simulant and
3584 triheptadecanoin as the internal standard (see Annex 0);
- 3585 c) two test specimens for determination of the surface area, in the case of samples of irregular
3586 shape (see 7.1.5.5).

3587 If previous testing has established that interference in the gas chromatography procedure is
3588 unlikely and section 0 is omitted, one test specimen less will be required.

3589 Testing in triplicate is allowed but in this case if one test result is invalid repeat the entire
3590 procedure.

3591 **7.1.5.3 Cutting films, sheets and other flat materials or articles**

3592 7.1.5.3.1 Immersion method

3593 Lay the sample on the cutting slab (7.1.4.1) and cut test specimens of 1 dm² (7.1.5.1.1.1) using
3594 the 100 mm x 100 mm template (7.1.4.4). Check, using the rule (7.1.4.6), that the dimensions of
3595 the test specimen are within the specified deviation (± 1 mm).

3596 Cut each test specimen into four test pieces 25 mm x 100 mm using the rule (7.1.4.5). Assemble
3597 one test specimen onto the support by piercing suitable holes in the test pieces and placing two
3598 test pieces on each side of the cross arms of the support. Repeat this procedure for all remaining
3599 test specimens.

3600 7.1.5.3.2 Cell method

3601 Lay the sample with its non-food contact surface on the cutting slab (7.1.4.1). Take the ring from
3602 the cell type A (7.1.4.30) and place it on the food contact surface of the test sample. Cut out the
3603 test specimen by cutting round the outer edge of the ring, using the cutting implement (7.1.4.3)

3604 7.1.5.3.3 Pouch method

3605 Lay the sample with its non-food contact surface on the cutting slab (7.1.4.1) and cut test pieces
3606 using the 120 mm x 120 mm template (7.1.4.4). Two test pieces are required for each test
3607 specimen.

3608 Place pairs of the test pieces together with the surfaces to be in contact with the olive oil facing.
3609 Use the heat or pressure sealer (7.1.4.32) to form pouches with four seals parallel to all four
3610 edges, 10 mm from the edge. Measure the distances between the inner edges of the seals to the
3611 nearest 1 mm and calculate the total surface area of the test specimen which will be exposed to
3612 olive oil, to the nearest 0.01 dm². This shall be approximately 2 dm². Using the cutting
3613 implement, remove excess film from the sealed area (to reduce the area of film not directly
3614 exposed to olive oil) whilst leaving enough to withstand the test conditions without leaking.

3615 Measure and record the surface area of the pouch which will be in contact with the simulant and
3616 the total external area of the pouch after trimming excess material.

3617 Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently
3618 large to insert a 100 ml pipette.

3619 **7.1.5.4 Cutting containers and other articles**

3620 7.1.5.4.1 Immersion method

3621 Cut sections from the walls of the container or article to give test specimens each of area
3622 approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles
3623 to provide each test specimen.

3624 Measure the dimensions of each test specimen to the nearest 1 mm, using the rule (see
3625 7.1.5.1.1.1).

3626 Calculate the area of each test specimen to the nearest 0.01 dm² and record. If necessary, cut
3627 each test specimen into smaller pieces to enable them to fit into the glass tubes (7.1.4.11). The
3628 test specimens or pieces are placed on the specimen supports if these are appropriate or, if the
3629 test specimens or pieces are sufficiently rigid, they can be tested unsupported.

3630 NOTE Cutting the test specimens into smaller pieces will increase the area of cut edges, so that
3631 the area of cut edges exceeds 10% of the test specimen area (see 7.1.5.1.1.1).

3632 7.1.5.4.2 Filling method

3633 If the article is large, to avoid handling and weighing problems or using excessive amounts of
3634 olive oil it may be preferable to cut it so that the surface of the test specimen in contact with the
3635 olive oil does not exceed 3 dm².

3636 If this is done, take care that olive oil does not come into contact with the cut edges of the test
3637 specimen. It is important that the area in contact with the oil is determined as it will be
3638 incorporated into the calculation later.

3639 Scratch lightly an identification code on the external surface of each test specimen.

3640 NOTE If only part of a test specimen is tested, this part should be representative of the whole in
3641 terms of composition and wall or layer thickness.

3642 **7.1.5.5 Cutting articles of irregular shape for immersion method**

3643 Select representative portions of the article, or multiples of the article for small articles, to give
3644 nine dimensionally similar test specimens each with a known total surface area of at least 1 dm².
3645 Measure only the surface area intended to come into contact with foodstuffs of two of these test
3646 specimens to the nearest 0.05 dm² using the Schlegel Method, as described in annex B of EN ISO
3647 8442-2:1997, or any other suitable method. Record the surface area of each test specimen.

3648

3649 **7.1.6 Procedure**

3650 The procedure consists of nine sections:

- 3651 1. Check whether the vegetable oil and the internal standard are suitable for use in the
3652 procedure
- 3653 2. Check the presence of volatile migrants in the test specimen
- 3654 3. Check the sensitivity of the test specimen for humidity
- 3655 4. Initial weighing of the test specimen
- 3656 5. Contact of the test specimen to the vegetable oil for single use
- 3657 6. Contact of the test specimen to the vegetable oil for repeated use
- 3658 7. Final weighing of the test specimen
- 3659 8. Extraction of the absorbed vegetable oil from the test specimen
- 3660 9. Quantification of the amount of the adsorbed vegetable oil

3661 **7.1.6.1 Determination of the suitability of vegetable oil for overall migration testing**

3662 This procedure is carried out to verify that rectified olive oil is suitable as the fatty food
3663 simulant, and that triheptadecanoin is suitable for use as an internal standard (7.1.3.3) for the
3664 gas chromatographic determination of oil as its methyl esters.

3665 7.1.6.1.1 Chromatogram of vegetable oil and internal standard

3666 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
3667 of the cyclohexane solution of triheptadecanoin (7.1.3.3) by pipette (7.1.4.21). Remove the
3668 cyclohexane using a rotary evaporator or water bath (7.1.4.17 or 7.1.4.18) and add, by

3669 measuring cylinder or graduated syringe (7.1.4.20), 10 ml ± 0.2 ml of n-heptane (7.1.3.6). Ensure
3670 the residue of rectified olive oil is well dispersed by shaking, warming or by ultrasonic
3671 treatment.

3672 7.1.6.1.1 Methyl ester preparation procedure

3673 Add by measuring cylinder or graduated syringe (7.1.4.20), 10 ml ± 0.2 ml of the potassium
3674 hydroxide solution (7.1.3.4) and a few anti-bumping beads (7.1.4.14). Connect a condenser to
3675 the flask and boil the mixture under reflux for 10 min ± 0.5 min.

3676 Add through the condenser by measuring cylinder, or graduated syringe (7.1.4.20), 5 ml ± 0.2 ml
3677 of the methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2
3678 min ± 0.25 min.

3679 Cool to room temperature and add, by measuring cylinder (7.1.4.20), 15 ml to 20 ml of saturated
3680 sodium sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulphate solution
3681 until the liquid level reaches the neck of the flask. Allow to stand until the phases have
3682 separated.

3683 If there will be a delay of more than seven days in using a methyl ester solution for the gas
3684 chromatographic determinations, transfer the n-heptane layer to a small stoppered tube
3685 (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

3686 NOTE The methyl esters for the subsequent gas chromatographic determination are in the
3687 upper, n-heptane, layer.

3688 Inject the methyl ester solution into the gas chromatograph (7.1.4.23).

3689 NOTE A volume of 1-3 µl has been found suitable for the columns described in the note to
3690 7.1.4.23.

3691 Retain the chromatogram for comparison.

3692 7.1.6.1.2 Chromatogram of test specimen extract

3693 Take one of the test specimens, as prepared in section 7.1.5, and place it in a soxhlet type
3694 extractor (7.1.4.15). Take a 250 ml or 500 ml flask (7.1.4.15) and add 10 ml of cyclohexane
3695 without the internal standard and sufficient extraction solvent (7.1.3.2) to allow cycling of the
3696 soxhlet type extractor (approximately 200 ml or 400 ml, according to the size of the flask) with
3697 anti-bumping beads (7.1.4.14) to control boiling. Using either a water bath or steam bath
3698 (7.1.4.18) extract for a period of 7 h (0/+1 h), with not less than six extraction cycles per hour,
3699 ensuring that the test pieces are totally submerged in the solvent during each soxhlet cycle, and
3700 that they remain separated from each other.

3701 Drain all of the solvent from the soxhlet type extractor into the flask, remove the flask from the
3702 soxhlet type extractor and evaporate the solvent to a volume of approximately 10 ml using a
3703 rotary evaporator or simple distillation apparatus (7.1.4.17). Transfer the solution of the test
3704 specimen extract to a separate 50 ml flask (7.1.4.19) and wash the flask with three portions of 5
3705 ml of solvent. Add the washings to the 50 ml flask. Evaporate to dryness using a rotary
3706 evaporator or water bath (7.1.4.17 or 7.1.4.18).

3707 Add 10.0 ml ± 0.2 ml of the n-heptane to the 50 ml flask. Ensure that the test specimen extract is
3708 well dispersed by shaking, warming or by ultrasonic treatment. Then subject the contents of the
3709 flask to the methyl ester preparation procedure, described in 7.1.6.1.1 and inject the same
3710 volume of the resulting solution into the gas chromatograph (7.1.4.23). Retain the
3711 chromatogram.

3712 7.1.6.1.3 Comparison of chromatograms

3713 Compare the chromatogram of the methyl esters produced from the rectified olive oil and
3714 internal standard in the procedure in 7.1.6.1.1 with the chromatogram of the preparation from
3715 the test specimen extract produced in procedure 7.1.6.1.2. If peaks are present in the
3716 chromatogram of the extract with similar retention times to those of the peaks of rectified olive

3717 oil methyl esters, and equate to 2 mg or more of olive oil, then the method is unsuitable for the
3718 material under examination. If a polar column has been used and interferences are observed on
3719 the peaks of the C18:0 and/or C18:2 peak, but not on other olive oil methyl ester peaks, then
3720 olive oil may be considered to be a suitable fat simulant, following method 3 given in 7.1.6.9.2.2.

3721 If a peak is present in the chromatogram of the extract with similar retention time to that of the
3722 peak for methyl heptadecanoate, originating from triheptadecanoin, the internal standard, and is
3723 more than 1 % of the height or area of that peak, then consider an alternative internal standard.

3724 NOTE A suitable alternative internal standard is trionadecanoin or hydrocinnamic acid, ethyl
3725 ester.

3726 NOTE **Figure 12** and **Figure 13** show typical chromatograms of the methyl esters of olive oil and
3727 triheptadecanoin using columns 1 and 2, respectively.

3728 **7.1.6.2 Determination of the presence of volatile substances**

3729 Determine the need for removing volatile substances of the test specimens by carrying out the
3730 procedure described in this section. If prior tests have established that sample conditioning for
3731 removing volatile substances is not required then follow Annex 7.1.6.4.1.

3732 Take one test specimen, as prepared in 7.1.5 and determine the mass to the nearest milligram.
3733 Place the test specimen in a vacuum oven (7.1.4.25) at $60 \pm 5^\circ\text{C}$. Reduce the pressure in the oven
3734 to 1.3 kPa or less. Leave the test specimen in the oven for 60 ± 10 min. Release the pressure and
3735 transfer the test specimen from the vacuum oven to a desiccator (7.1.4.26) containing self-
3736 indicating silica gel or anhydrous calcium chloride. Determine, after cooling for 60 ± 10 min the
3737 mass of the test specimen. Calculate the difference between the mass of the test specimen before
3738 and after the one hour vacuum conditioning. Discard the test specimen.

3739 If the difference between the masses of the test specimen is greater than 2 mg/dm^2 , then
3740 conditioning of the test specimens to be used in the test will be necessary before each weighing
3741 operation in the test procedure (7.1.6.4.2). If the difference between the masses of the test
3742 specimen is less than 2 mg/dm^2 , then conditioning of the test specimens to be used in the test
3743 will not be necessary before each weighing operation in the test procedure.

3744 **7.1.6.3 Determination of the moisture sensitivity of the test specimen**

3745 This procedure determines whether the conditioning of test specimens with respect to moisture
3746 content will be required.

3747 Take one test specimen, as prepared in 7.1.5 and place in a container (7.1.4.10) maintained at
3748 80% relative humidity for 24 ± 4 h. Remove the test specimen and weigh as quickly as possible
3749 after its removal from the controlled environment, to minimise loss of moisture and change in
3750 mass.

3751 Place the same test specimen in a container (7.1.4.10) maintained at 50% relative humidity for
3752 24 ± 4 h. Remove the test specimen and weigh, taking the same precautions as above.

3753 If the difference between the masses of the test specimen is greater than 2 mg/dm^2 , then
3754 conditioning of the test specimens will be necessary before each weighing operation in the test
3755 procedure. If the difference between the masses of the test specimen is less than 2 mg/dm^2 , then
3756 conditioning of the test specimens will not be necessary before each weighing operation in the
3757 test procedure.

3758 **7.1.6.4 Initial weighing of test specimens**

3759 Before weighing, discharge any build-up of static electricity with an antistatic gun or other
3760 suitable means.

3761 7.1.6.4.1 Determination of initial weight of non-moisture sensitive test specimen in absence of
3762 volatiles

3763 If the test in section 7.1.6.2, shows that there is no substantial amount of volatile substances
3764 present in the test specimen and if the test in section 7.1.6.3 shows that the test specimen is not
3765 moisture sensitive, determine and record the mass of each test specimen (m_a).

3766 7.1.6.4.2 Determination of initial weight of non-moisture sensitive test specimen in presence of
3767 volatiles

3768 If the test in section 7.1.6.2 shows that there is a substantial amount of volatile substances
3769 present and if the test in section 7.1.6.3 shows that the test specimen is not moisture sensitive,
3770 follow the directions in this section.

3771 *7.1.6.4.2.1 Vacuum conditioning of non-moisture sensitive test specimen in presence of volatiles*

3772 Weigh the four test specimens (w_a), as prepared in 7.1.5. Then transfer to a vacuum oven at
3773 $60 \pm 5^\circ\text{C}$ and reduce the pressure to approximately 1.3 kPa using a high vacuum pump. The
3774 vacuum pump can be turned off provided the pressure is maintained. Turn on the vacuum pump
3775 every hour for a period of 10-15 min to remove moisture from the oven and to refresh the
3776 vacuum. Leave the test specimens under this condition in the vacuum oven for a period of 24 ± 2
3777 h. Transfer the test specimens from the vacuum oven to a desiccator containing self-indicating
3778 silica gel or anhydrous calcium chloride. Determine, after cooling for 60 ± 10 min, the mass of the
3779 test specimen. Repeat the conditioning procedure until the change in mass between two
3780 consecutive weighing's is less than 2 mg/dm². Record the final mass of each test specimen (w_b).

3781 *7.1.6.4.2.2 Reconditioning of the test specimen after vacuum conditioning*

3782 Place the test specimens at ambient humidity (not less than 60%) and determine, after cooling
3783 for 60 ± 10 min, the mass of the test specimen. Repeat the conditioning procedure until the
3784 change in mass between two consecutive weighing's is less than 2 mg/dm². Record the final
3785 mass of each test specimen (m_a).

3786 NOTE: Reconditioning is only required for test specimen that have lost water and that may have
3787 influenced the physical properties of the test specimen. E.g. polyamide samples need
3788 reconditioning to regain their initial properties. If it is known that the loss of mass is due to
3789 volatile substances, other than water, then reconditioning is not needed.

3790 NOTE The difference of the mass before and after the reconditioning, w_b and m_a , respectively,
3791 indicates the amount of volatiles, including water, that is present in the test specimen.

3792 Usually 70% of the mass lost will be regained. However if significantly less mass is regained then
3793 this is an indication for the loss of organic volatiles.

3794 7.1.6.4.3 Determination of initial weight of moisture sensitive test specimen in absence of
3795 volatiles

3796 If the test in section 7.1.6.2 shows that there is no substantial amount of volatile substances
3797 present and if the test in section 7.1.6.3 shows that the test specimen is moisture sensitive,
3798 follow the directions in this section.

3799 Place the test specimen in the container maintained at 50% relative humidity, weigh at intervals
3800 of about 24 h, until the change in mass between consecutive weighings of each test specimen is
3801 less than 2 mg/dm² and record the final mass of each test specimen (m_a).

3802 NOTE: This determination is also applied in case the sample cannot be conditioned using the
3803 vacuum method in section 7.1.6.4.2.1 due to decomposition or irreversible changes of the test
3804 specimen. However volatiles may then be included.

3805 NOTE: This determination may be used in the screening procedure in which the migration of
3806 volatiles is included to demonstrate compliance with generic SML's of volatile organic
3807 substances.

3808 7.1.6.4.4 Determination of initial weight of moisture sensitive test specimen in presence of
3809 volatiles

3810 If the test in section 7.1.6.2 shows that there is a substantial amount of volatile substances
3811 present and if the test in section 7.1.6.3 shows that the test specimen is moisture sensitive,
3812 follow the directions in section 7.1.6.4.2.1 followed by section 7.1.6.4.3.

3813 **7.1.6.5 Contact with food simulant for single use**

3814 7.1.6.5.1 Immersion method

3815 Take six of the glass tubes (7.1.4.11), mark them for identification purposes. Measure 100 ml \pm 5
3816 ml of olive oil (7.1.3.1) into each tube by measuring cylinder and stopper the tube.

3817 Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place into
3818 one of the tubes a thermometer or thermocouple and stopper the tubes. Place the six tubes in
3819 the thermostatically controlled oven or incubator (7.1.4.12) set at the test contact temperature.
3820 Leave until the olive oil has attained the test contact temperature, using the thermometer or
3821 thermocouple to monitor the temperature. Take all tubes from the oven and place into five of the
3822 tubes containing olive oil, weighed test specimens prepared as in section 7.1.5 and conditioned if
3823 necessary as in section 7.1.6.4. Place into one of the tubes a thermometer or thermocouple and
3824 stopper the tubes. Ensure that the test specimens are totally immersed in olive oil; if they are
3825 not, add then either glass beads or glass rods (7.1.4.22) preheated at the test contact
3826 temperature to raise the level of the olive oil until total immersion is achieved.

3827 NOTE 2 The olive oil in the sixth tube is used as a reference standard in constructing the
3828 calibration graph (see 7.1.6.9.2.2).

3829 NOTE - To ensure that test pieces are well separated and that their surfaces are freely
3830 exposed to oil during the period of the test, insert a piece of fine stainless steel gauze
3831 (10.2.4.9) between the test pieces for thin films or insert glass rods between the test pieces
3832 after immersion in the oil for thick samples not placed on the supports. Where test specimen
3833 supports are used, label the supports with a tag bearing the test specimen identification.

3834 NOTE the tube with the thermometer or thermocouple is only used for monitoring the
3835 temperature

3836 Replace all six tubes in the thermostatically controlled oven or incubator set at the test
3837 temperature. This part of the operation should be carried out in the minimum time possible to
3838 prevent undue heat loss. Observe the temperature of the thermostatically controlled oven or
3839 incubator or the olive oil (see NOTE 5) in the sixth tube and leave the tubes for the selected test
3840 period, taking into account the tolerances specified in Table 4, after the olive oil in the sixth tube
3841 has reached a temperature within the tolerance specified in Table 5.

3842 NOTE 4 Table 4 and Table 5 include tolerances on a wide range of contact times and contact
3843 temperatures. All of these contact times and contact temperatures are not necessarily
3844 relevant to this standard.

3845 NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the
3846 air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
3847 temperature of the simulant.

3848 NOTE 5a - in some cases it is difficult to keep the test contact temperature as soon as the test
3849 specimen is immersed in the food simulant and put in the oven at the test contact
3850 temperature, taking into account the tolerances specified in Table 4. It is very important that
3851 all materials except the test specimen used in the test are at the test contact temperature. An
3852 extra measure can be to put a water bath with circulation in the oven in which the tubes are
3853 put. If the test contact temperature cannot be maintained it is allowed to ensure that the test
3854 contact temperature is correct by setting the oven temperature higher.

3855 Take the tubes from the oven or incubator and immediately remove the test specimens from the
3856 tubes. For those specimens that have been in olive oil, allow the oil to drain. Remove any

3857 adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the pressing
3858 procedure until the filter paper shows no spots of olive oil. For test specimens on supports,
3859 remove the individual test pieces from the supports to carry out this operation. Clean the
3860 supports of oil by washing with the extraction solvent and replace the test pieces on them.

3861 7.1.6.5.2 Cell method

3862 Take five type A cells (7.1.4.30), mark them for identification purposes. Place in the
3863 thermostatically controlled oven or incubator (7.1.4.12), which is set at the test contact
3864 temperature and leave until the test contact temperature has been attained.

3865 Take six glass tubes (7.1.4.11), measure 125 ± 5 ml of olive oil (7.1.3.1) into each tube by
3866 measuring cylinder and stopper the tubes.

3867 Alternatively mark the tubes for a volume of 125 ml and fill with olive oil to the mark. Place into
3868 one of the tubes a thermometer or thermocouple and stopper the tubes. Place the six tubes in
3869 the thermostatically controlled oven or incubator (7.1.4.12) set at the test temperature. Leave
3870 until the olive oil has attained the test temperature, using the thermometer or thermocouple to
3871 monitor the temperature.

3872 NOTE the tube with the thermometer or thermocouple is only used for monitoring the
3873 temperature. This tube is also used for filling the cell used for temperature monitoring.

3874 Remove the cells from the thermostatically controlled oven or incubator, dismantle the cells and
3875 place on the base of each cell one of the test specimens. Reassemble the cells, ensuring that the
3876 clamping screw wheel is well tightened down.

3877 Remove all tubes from the thermostatically controlled oven or incubator or refrigerator and
3878 transfer the olive oil from each tube to each of the cells through the filler hole. Remove the
3879 thermometer or thermocouple from the tube and insert, if applicable see NOTE 5, in one of the
3880 cells and replace the filler plugs.

3881 NOTE 2 The olive oil in the sixth tube is used as a reference standard in constructing the
3882 calibration graph (see 7.1.6.9.2.2).

3883 Replace the five cells and the remaining tube in the thermostatically controlled oven or
3884 incubator set at the test temperature. This part of the operation should be carried out in the
3885 minimum time to prevent undue heat loss from the cells and olive oil. Observe the temperature
3886 of the thermostatically controlled oven or incubator or the olive oil (see NOTE 5) in the one of
3887 the cells and leave the cells and tubes for the selected test period, taking into account the
3888 tolerances specified in Table 4, after the olive oil in the cell has reached a temperature within the
3889 tolerance specified in Table 5.

3890 NOTE 3 The above procedure is typically for cell A (7.1.4.30). The procedure may deviate if
3891 another type of cell is used. In all cases attention shall be given to reaching the intended test
3892 contact temperature of the oil in the cell to establish the start of the migration period.

3893 NOTE 4 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
3894 temperatures. All of these contact times and contact temperatures are not necessarily
3895 relevant to this method.

3896 NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the
3897 air-bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
3898 temperature of the simulant.

3899 Take the cells and tube from the oven or incubator and immediately remove the test specimens
3900 from the cells and allow the oil to drain. Remove any adhering olive oil by gently pressing
3901 between filter papers (7.1.4.13). Repeat the pressing procedure until the filter paper shows no
3902 spots of olive oil.

3903 7.1.6.5.3 Pouch method

3904 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
3905 measuring cylinder (7.1.4.20) and stopper the tube.

3906 NOTE 1 The pouch holder (7.1.4.31) should be cleaned before use, if necessary, using
3907 solvents, such as acetone and or detergents. For olive oil that is difficult to remove, use
3908 proprietary solvent mixtures.

3909 WARNING Proprietary solvent mixtures usually contain caustic substances and also volatile
3910 solvents. Handle with care, using protective gloves and eye protection, in a fume cupboard.
3911 Information regarding sources of the proprietary mixtures specified in this test method is
3912 available from National Standards Bodies.

3913 Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place the
3914 six tubes and the pouch holder, in the thermostatically controlled oven or incubator (7.1.4.12)
3915 set at the test temperature.

3916 NOTE 3 Leakage can occur from the pouches and it is advisable to have a drip tray in the
3917 oven.

3918 NOTE the tube with the thermometer or thermocouple is only used for monitoring the
3919 temperature. This tube is also used for filling the pouch used for temperature monitoring.

3920 Leave until the olive oil has attained the test temperature, using the thermometer or
3921 thermocouple to monitor the temperature.

3922 Remove the pouch holder from the thermostatically controlled oven or incubator and place the
3923 test specimens between the spacers.

3924 Remove all tubes containing olive oil from the oven and pipette sufficient olive oil into four
3925 pouches. This shall be approximately 100 ml, but for thick/semi-rigid materials the quantity will
3926 be less. Place a thermocouple in one pouch and close the open corners with a clip.

3927 NOTE 4 The olive oil in the sixth tube is used as a reference standard for constructing the
3928 calibration graph (see 7.1.6.9.2.2).

3929 Replace the pouch holder, containing the four pouches, and the tube in the thermostatically
3930 controlled oven or incubator set at the test temperature. This part of the operation should be
3931 carried out in the minimum time possible to prevent undue heat loss. Observe the temperature
3932 of the thermostatically controlled oven or incubator or the olive oil (see NOTE 7) in the pouch
3933 and leave the pouches and tubes for the selected test period, taking into account the tolerances
3934 specified in Table 4, after the olive oil in the pouch has reached a temperature within the
3935 tolerance specified in Table 5.

3936 NOTE 6 Table 4 and Table 5 include tolerances on a wide range of contact times and contact
3937 temperatures. All of these contact times and contact temperatures are not necessarily
3938 relevant to this part of the test method.

3939 NOTE 7 For contact times of 24 h or more it is acceptable to monitor the temperature of the
3940 air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
3941 temperature of the simulant.

3942 Take the pouch holder and the tubes containing olive oil from the thermostatically controlled
3943 oven or incubator.

3944 If an evident leak has occurred with more than one pouch the test is invalid and shall be
3945 repealed.

3946 If no evident leaks have occurred in at least three pouches, then remove the pouches from the
3947 holder.

3948 Pour the olive oil from each pouch and wipe any excess from the outside with filter paper
3949 (7.1.4.13). Take each of the four pouches in turn, lay them on the cutting slab (7.1.4.1) and open

3950 them carefully by cutting through one layer along the inner edges of the seals using the cutting
3951 implement (10.2.4.3).

3952 Take the two portions of each pouch and remove adhering olive oil by gently pressing between
3953 filter papers. Repeat the pressing procedure until the filter paper shows no spots of olive oil.

3954 7.1.6.5.4 Filling method

3955 Place a sufficient volume of olive oil in a beaker in the thermostatically controlled oven or
3956 incubator (7.1.4.12) which is set at the test temperature and leave until the test temperature has
3957 been attained.

3958 Place a sufficient volume of olive oil in five beakers, to fill each of five test specimens to the
3959 nominal volume or to 5 mm from the top if the nominal volume is not known. Insert a
3960 thermometer or thermocouple in one of the beakers containing the olive oil for a test specimen.

3961 NOTE the beaker with the thermometer or thermocouple is only used for monitoring the
3962 temperature. This beaker is also used for filling the test specimen used for temperature
3963 monitoring.

3964 Take one glass tube (10.1.4.11), measure 100 ± 5 ml of olive oil (10.1.3.1) into it by measuring
3965 cylinder (10.1.4.20) and stopper the tube. This tube is used as reference standard in
3966 constructing the calibration graph (see 7.1.6.9.2.2).

3967 Place the beakers and the tube in the thermostatically controlled oven or incubator or
3968 refrigerator set at the test temperature and leave until the olive oil has attained the test
3969 temperature.

3970 Place each test specimen on a clean, oil free surface and fill five test specimens with olive oil to
3971 within 0.5 cm of the top. If the container has a specified nominal volume of contents, see section
3972 7.1.5.1.1. Place into one of the filled test specimens a thermometer or thermocouple.

3973 NOTE 2 Care should be taken not to spill any oil on the external surfaces.

3974 Place the five filled test specimens and the tube in the thermostatically controlled oven or
3975 incubator set at the test contact temperature. This part of the operation should be carried out in
3976 the minimum time possible to prevent undue heat loss.

3977 Observe the temperature of the thermostatically controlled oven or incubator or the olive oil
3978 (see NOTE 5) in the filled test specimen and leave the test specimens for the selected test period,
3979 taking into account the tolerances specified in Table 4, after the olive oil in the test specimen has
3980 reached a temperature within the tolerance specified in Table 5.

3981 NOTE 4 Table 4 and Table 5 include tolerances on a wide range of contact times and contact
3982 temperatures. All of these contact times and contact temperatures are not necessarily
3983 relevant to this part of the test method.

3984 NOTE 5 For contact times of 24 h or more it is acceptable to monitor the temperature of the
3985 air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
3986 temperature of the simulant.

3987 Remove the test specimens and the tube from the thermostatically controlled oven or incubator
3988 and immediately empty the test specimens that contained olive oil and allow the oil to drain.
3989 Remove any adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the
3990 pressing procedure until the filter paper shows no spots of olive oil.

3991 **7.1.6.6 Contact with food simulant for repeated use**

3992 With vegetable oil, the repeated contact of the same test specimen to fresh portions of food
3993 simulant is not a feasible procedure, since the procedure requires solvent extraction to remove
3994 the oil. Therefore, the test is carried out on three sets of five test specimens from the same
3995 sample of the material or article. One set is subjected to the test appropriate for articles intended
3996 for single use by the standard procedure and the mean result calculated (M1) (see section
3997 7.1.6.5). The other two sets for the second and third migration are exposed in a manner identical

3998 in every respect to those of the first migration except for the period of contact. The test
3999 specimens of the second migration (M2) are exposed for a period of twice that of sample one
4000 and the test specimens of the third migration (M3) are exposed for a period three times that of
4001 those of the first migration.

4002 NOTE: the sets dedicated for the second and third migration may have four test specimens if
4003 the migration of all test specimens is carried out in one oven. Then the fifth test specimen of
4004 the first migration containing the thermocouple serves for all sets.

4005 **7.1.6.7 Final weighing of test specimens**

4006 7.1.6.7.1 Determination of final weight of non-moisture sensitive test specimen

4007 Weigh all four test specimens and record their mass (m_b).

4008 7.1.6.7.2 Determination of final weight of moisture sensitive test specimen

4009 Place the four test specimen in the container maintained at 50% relative humidity, weigh at
4010 intervals of about 24 h, until the change in mass between consecutive weighings of each test
4011 specimen is less than 2 mg/dm² and record the final mass of each test specimen (m_b).

4012 **7.1.6.8 Extraction of absorbed vegetable oil**

4013 Take four flasks, 250 ml or 500 ml as appropriate to the size of the soxhlet type extractor
4014 (7.1.4.15) to be used for the extraction, and place in each flask 10.0 ml of the internal standard
4015 cyclohexane solution of triheptadecanoin (7.1.3.3), using a pipette (7.1.4.21), or an alternative
4016 higher quantity if more than 100 mg of olive oil is present.

4017 NOTE 1 if the test specimens have retained more than 100 mg of olive oil, 10.0 ml of the
4018 internal standard solution will be insufficient for optimum precision in the gas
4019 chromatography determination after extraction. Before commencing the operations in this
4020 section an estimation of the quantity of olive oil retained in the test specimens should be
4021 obtained by comparing the final masses of the test specimens with their initial masses. If
4022 considered necessary the quantity of internal standard solution can be increased from 10 ml
4023 although it is essential that the same quantity is used for each test specimen, and that this
4024 quantity is also used with the olive oil standards for the calibration graph (see 7.1.6.9.2.2). As
4025 a guide, approximately 0.5 mg of the internal standard is required for every mg of extracted
4026 olive oil.

4027 Add sufficient extraction solvent (7.1.3.2) to allow cycling of the soxhlet type extractor
4028 (approximately 200 ml or 400 ml, according to the size of the flask) with anti-bumping beads
4029 (7.1.4.14) to control boiling.

4030 Cut the test specimens into suitable sized strips, not wider than 30 mm and of correct length
4031 such that the strips shall be totally immersed during the soxhlet cycle.

4032 NOTE 1a Care should be taken when carrying out the cutting operations to ensure that slivers
4033 are not produced and lost.

4034 Place the four test specimens that have been in contact with olive oil into four soxhlet type
4035 extractors. Couple each soxhlet to a flask containing the internal standard prepared as above.
4036 Using either a water bath or steam bath (7.1.4.16), extract for a period of 7 h (0/+1h), with a
4037 minimum of six cycles per hour, ensuring that the test pieces are totally submerged in the
4038 solvent during each soxhlet cycle, and that they remain separated from each other.

4039 Drain all of the solvent from the soxhlet type extractors, remove the flasks from the soxhlet type
4040 extractors and evaporate the solvent to approximately 10 ml using a rotary evaporator, or
4041 simple distillation apparatus (7.1.4.17). Transfer the solutions containing the extracted olive oil
4042 and internal standard to separate 50 ml flasks (7.1.4.19), and wash each flask with three
4043 portions of 5 ml of solvent. Add the three washings to the respective individual 50 ml flasks.
4044 Evaporate to dryness using a rotary evaporator or a water bath.

4045 NOTE 2 Oxidation of the olive oil should be avoided where possible. Therefore evaporation of
4046 the solvent to dryness should be carried out under mild conditions of temperature. In
4047 addition contact of the olive oil to oxygen should be limited.

4048 NOTE 3 Some types of plastic are known to retain some of the absorbed olive oil despite
4049 prolonged soxhlet extraction with pentane. In these cases extraction of the olive oil is
4050 incomplete. This is known to give falsely low results in the test procedure. This difficulty may
4051 be overcome by subjecting the test specimens to a second extraction, this time with diethyl
4052 ether, or to the dissolution/precipitation method set out in section 7.4. The amount of oil
4053 obtained in the diethyl ether extract or in the solution after precipitation of the polymer is
4054 added to the amount of oil obtained in the pentane extract. To obtain reliable results the
4055 migration test shall be repeated using the dissolution/precipitation method.

4056 Repeat the extraction of the test specimens for an additional 7 h (0/+1h), with diethyl ether
4057 (7.1.3.8), adding a further quantity of the internal standard solution.

4058 NOTE 4 The same quantity of internal standard solution is used as for the first 7 h extraction.
4059 This quantity may not be the optimum if the quantity of olive oil in the first 7 h extraction is
4060 high. Good precision is not required for the second 7 h determinations since they are
4061 intended primarily as a check on the efficiency of the first 7 h extraction and using the same
4062 quantity of internal standard enables one calibration graph to be used.

4063 If previous testing has established that all of the olive oil will be extracted from the test
4064 specimens during the first 7 h extraction then the second 7 h extraction may be omitted.

4065 Isolate the residues in 50 ml flasks, using the procedure described above.

4066 Determine the extracted olive oil in both the first 7 h and the second 7 h extraction by the
4067 procedure described in 7.1.6.8, but retain the test specimens in the soxhlet type extractors until
4068 the extracted olive oil has been determined for the second extraction.

4069 **7.1.6.9 Determination of extracted olive oil**

4070 7.1.6.9.1 Preparation of fatty acid methyl esters

4071 Add 10 ± 0.2 ml of n-heptane to each of the 50 ml flasks containing the first 7 h extraction
4072 residue, by measuring cylinder (7.1.4.20), ensuring that the residues of olive oil and plastics
4073 extractables dissolve or are well dispersed by shaking, warming or by ultrasonic treatment.

4074 NOTE 1 Unless the residues in the flasks are dissolved or well dispersed in the n-heptane,
4075 quantitative hydrolysis or methylation of the olive oil and of the internal standard might not be
4076 obtained under the conditions described particularly when these residues contain extractables
4077 from plastics in excess of 50 mg. The internal standard might not react with the plastics
4078 extractables to the same degree as does the olive oil and correct results for olive oil might not be
4079 obtained.

4080 Add by measuring cylinder or graduated syringe (7.1.4.20), 10 ± 0.2 ml of the potassium
4081 hydroxide solution (7.1.3.4) and a few anti-bumping beads (7.1.4.14). Connect a condenser to
4082 the flask and boil the mixture under reflux for 10 ± 1.0 min.

4083 Add through the condenser by measuring cylinder, or graduated syringe, 5.0 ± 0.2 ml of the
4084 methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2 ± 0.25
4085 min.

4086 Cool to room temperature and add, by measuring cylinder, 15-20 ml of saturated sodium
4087 sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulfate solution until the
4088 liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

4089 NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the
4090 upper layer of n-heptane.

4091 Treat the residues from the second 7 h extraction as described above.

4092 If there will be a delay of more than 7 days in using a methyl ester solution for the gas
4093 chromatographic determinations, transfer the n-heptane layer to a small stoppered tube
4094 (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

4095 7.1.6.9.2 Determination of fatty acid methyl esters

4096 7.1.6.9.2.1 *Instrument*

4097 Determine the methyl esters of the olive oil fatty acids using a gas chromatograph (7.1.4.23).

4098 NOTE 1 For column 1 described in the note to 7.1.4.23 the following operating conditions
4099 have been found to be suitable:

4100 carrier gas helium at 2 ml/min

4101 injector spit (ratio 40:1)

4102 detector flame ionisation

4103 temperature programme initially 1 min at 140°C then ramped at 5°C/min to 190°C and
4104 maintained at 190°C for 8 min.

4105 injector temperature 220°C

4106 detector temperature 240°C

4107 For column 2 described in the note to 7.1.4.23 the following operating conditions have been
4108 found to be suitable:

4109 carrier gas helium

4110 oven temperature 250°C isothermal

4111 injector temperature 320 °C

4112 detector temperature 320 °C

4113 Use an integrator to measure the area of each of the olive oil peaks and the internal standard.

4114 NOTE 2 The use of an integrator and measurement of the peak area is the preferred method.

4115 7.1.6.9.2.2 *Calibration graph*

4116 Weigh a range of quantities of the blank reference olive oil which has been subjected to the same
4117 test conditions as the test specimens into 50 ml flasks (7.1.4.19). Weigh a range of olive oil
4118 quantities spanning the quantities of olive oil in the first 7 h extractions, taking no fewer than
4119 four standards.

4120 Add 10.0 ml of the internal standard cyclohexane solution of triheptadecanoin (7.1.3.3) to each
4121 flask using a pipette (7.1.4.21), or the alternative quantity which has been added to the
4122 extraction flasks in section 7.1.6.8. Remove the cyclohexane using a rotary evaporator or water
4123 bath (7.1.4.18 or 7.1.4.17). Subject the olive oil quantities, with the added internal standard, to
4124 the methyl ester preparation procedure described in section 7.1.6.9.1.

4125 Inject each of the n-heptane methyl ester solutions in duplicate, as a minimum, into the gas
4126 chromatographic column.

4127 NOTE 1 Typical chromatograms generated using columns 1 and 2 are shown respectively in
4128 Figure 12 and Figure 13,

4129 Construct a calibration graph, plotting the ratios of olive oil methyl esters to the internal
4130 standard peak on the y-axis and against the weighed quantities of olive oil on the x-axis.

4131 Various methods for the construction of a calibration graph are suitable and the choice of
4132 method depends on the equipment and chromatographic column used. The following methods
4133 are acceptable:

4134 Method 1 Peak height method

4135 Measure the peak height of the internal standard peak and of the methyl oleate (C18:1) peak,
4136 when a polar column has been employed. In the case where a non-polar column has been used
4137 for the separation of the methyl ester, then measure the internal standard peak and the C18
4138 peak of the olive oil. Calculate the ratio of the measured C18 peaks to the internal standard peak
4139 and plot the ratios versus the weighed quantities of olive oil.

4140 Method 2 Peak area method

4141 Measure the peak area of the internal standard peak and of each of the methyl esters originating
4142 from the olive oil. Add together the peak areas of the C16 and C18 peaks if a non-polar column
4143 was employed. If a polar column was used, sum the areas of all the peaks (C16:0, C16:1, C18:0,
4144 C18:1 and C18:2) originating from the olive oil. Calculate the ratio of the combined areas of the
4145 measured peaks to the area of the internal standard peak and plot the ratio versus the weighed
4146 quantities of olive oil.

4147 Method 3 Peak area method in the case of interference from the test sample.

4148 In the event that the analysis of a blank test sample, see Annex 0, has revealed an interference
4149 with one or more of the olive oil methyl esters, but not all of the peaks, then this peak or peaks
4150 shall be excluded from the calculation of the total area of the olive oil methyl esters. Calculate
4151 the ratio of the total area of the methyl esters originating from olive oil and which are free from
4152 interference and the area of the internal standard and plot the ratios versus the weighed
4153 quantities of oil.

4154 NOTE 2 A typical calibration graph is shown in Figure 14.

4155 Calculate from each calibration standard chromatogram the C18:1/C16:0 ratio if a polar column
4156 was used or C18/C16 ratio in the case of a non-polar column. Determine the mean ratio value
4157 from the duplicate or multiple injections for comparison with the same ratio obtained from the
4158 test specimen extracts, see 7.1.6.9.2.3.

4159 NOTE 3 If another vegetable oil than olive oil is used then the ratio of the main peaks in the
4160 chromatogram shall be calculated

4161 *7.1.6.9.2.3 Determination of olive oil absorbed by test specimens*

4162 Inject into the gas chromatograph (7.1.4.23) a suitable quantity from each of the n-heptane
4163 methyl ester solutions prepared from the residues containing the extracted olive oil (see
4164 7.1.6.9.1). Inject in duplicate, as a minimum.

4165 For each chromatogram, measure the height or area of the olive oil methyl ester peak or peaks
4166 and the internal standard peak using the same peaks and method as used in the construction of
4167 the calibration graph, see 7.1.6.9.2. Calculate the ratio of the relevant peaks to the internal
4168 standard peak for each chromatogram and for each solution determine the mean ratio value
4169 from the duplicate or multiple injections.

4170 Calculate the amount of olive oil extracted from the test specimen from the regression
4171 parameters

4172 If the regression line equation is

4173
$$y = ax + b \quad (1)$$

4174 then:

4175
$$m_{\infty} = \frac{(y-b)}{a} \quad (2)$$

4176 where

4177 m_{∞} is the mass of olive oil extracted from the sample, in milligrams;

4178 a is the slope of the calibration graph;

4179 b is the intercept of the calibration graph;

4180 x is the mass of olive oil in the standard, in milligrams;

4181 y is the ratio of olive oil methyl esters to internal standard.

4182 The procedure yields directly the amount of olive oil extracted from the test specimen, in
4183 milligrams.

4184 NOTE 1 The method applying calculation from the regression parameters is the preferred
4185 method.

4186 If olive oil is found in the second extract from more than one of the test specimens and the
4187 amount is less than 10 mg, but measurable, add this to the amount determined from the first 7 h
4188 extraction and record the total mass of extracted olive oil for each test specimen in grams.

4189 If more than 10 mg of olive oil is found in the second extract, see NOTE 3 of Annex 7.1.6.8

4190 If the ratio C18 to the C16 peaks has changed, read NOTE 1a

4191 NOTE 1a A difference in C18:1/C16:0 ratio (using column 1) between the olive oil extracted
4192 from the test specimen and the olive oil applied as the fatty food simulant in the migration
4193 test indicates that the composition of the extracted oil for some reason is different from the
4194 composition of the oil that has not been in contact with a test specimen. Possible causes for
4195 the changes of the composition are:

4196

- 4197 • reaction of olive oil constituents with plastics constituents;
- 4198 • oxidation of unsaturated constituents of the olive oil. This has been observed to occur
4199 when rather long periods for conditioning the test specimen after contact with the oil are
4200 necessary;
- 4201 • incomplete methylation of fatty acids in the trans-esterification procedure, such
4202 difficulties arise with some types of high impact polystyrene (HIPS) and acrylonitrile-
4203 butadiene-styrene (ABS);
- 4204 • selective absorption of oil constituents by test specimens. Polyolefins for example do
4205 absorb selectively mono- and diglycerides of saturated free fatty acids in some cases,
4206 whereas HIPS, ABS and nitrilebutadiene rubber (NBR) often selectively absorb
4207 diglycerides, and to a lesser extent also monoglycerides of unsaturated fatty acids;
- 4208 • interference by plastics constituents having the same retention time as C16:0 or C18:1
4209 methyl ester or forming those esters in the trans-esterification stage.

4209 Whether a change in the C18:1/C16:0 ratio acts upon the final result of the overall migration
4210 determination to an extent which is not acceptable depends mainly on the magnitude of the
4211 change and on the amount of oil recovered from the test specimen, e.g. a 25 % change in the
4212 C18:1/C16:0 ratio may result in a 25 % lower result in the amount of fat extracted, which
4213 would mean 2.5 mg when only 10 mg fat is absorbed by the test specimen but 25 mg when
4214 100 mg of fat is absorbed. So a proportional change in C18:1/C16:0 ratio will result in an
4215 absolute difference in the amount of fat calculated, and consequently in an absolute
4216 difference in the overall migration values. Whilst an absolute difference of 2.5 mg is
4217 acceptable, because it is within the accepted analytical tolerance, one of 25 mg is not.

4218 Whether there might be a possibility of obtaining false results because of a change in the
4219 C18:1/C16:0 ratio, can easily be established by measuring the amount of oil extracted from
4220 the test specimen using two different calibration graphs. In one graph the ratio C16:0/C17:0
4221 is plotted versus the amount of olive oil and in the other one the ratio C18:1/C17:0. The
4222 amount of oil calculated using the C16:0/C17:0 graph shall differ from the amount calculated
4223 using the C18:1/C17:0 graph by no more than 2 mg/dm². In case a larger difference is
4224 observed the cause of it has to be identified and an appropriate action be taken. Remedies for
4225 problems could be:

4226

- 4227 • if reaction of oil constituents with plastics constituents is suspected a less reactive oil, e.g.
4228 coconut oil or palmkernel oil, can be used;
- 4229 • if oxidation of unsaturated fatty acids is suspected a less vulnerable fatty food simulant, e.g.
4230 coconut oil or palmkernel oil, can be used;

- 4230 • if incomplete methylation of fatty acids during trans-esterification is suspected the
4231 heptane layer obtained in the normal trans-esterification procedure is subjected to an
4232 additional trans-esterification treatment;
- 4233 • if selective absorption of fatty simulant constituents by the test specimen is suspected,
4234 which can be ascertained by thin layer chromatography comparison of the composition of
4235 extracted and olive oil a fatty food simulant low in free fatty acids and mono- and
4236 diglycerides can be used;
- 4237 • if interference of oleic acid (C18:1) or heptadecanoic acid (C17:0) peak area measurement
4238 by plastic constituents is suspected which can be ascertained by running a blank
4239 experiment with a sample of the final article in question, the palmitic acid (C16:0) peak
4240 area of olive oil can be used as a reference. It is preferable however to use, if possible,
4241 sunflower, coconut or palmkernel oil as the food simulant instead.

4242

4243 For each chromatogram from the first 7 h extractions, calculate the ratio of the height or area of
4244 the C18 peak to the height or area of the C16 peak. Determine the mean value of these ratios and
4245 compare to the similar ratio determined in 7.1.6.9.2.2 from the olive oil calibration
4246 chromatograms. Establish whether the difference between the two ratios values is acceptable.

4247 NOTE 2 A change in the C18/C16 ratio for extracted olive oil samples compared with the
4248 same ratio for olive oil used for the calibration graph indicates that some reaction or
4249 fractionation of the olive oil has occurred, either during the test period or during extraction of
4250 the test specimens. Such changes will have an adverse effect on the overall migration result.

4251 7.1.7 Expression of results

4252 7.1.7.1 Method of calculation for single use

4253 Express the overall migration as milligrams lost per square decimetre of surface of the sample
4254 which is intended to come into contact with foodstuffs, calculated for each test specimen using
4255 the following formula:

$$4256 \quad M = \frac{(m_a - (m_b - m_c)) \cdot 1000}{S} \quad (3)$$

4257 where

4258 M is the overall migration into olive oil, in milligrams per square decimetre of the surface area of
4259 sample intended to come into contact with the foodstuff;

4260 m_a is the initial mass of the test specimen, before contact with the olive oil, in grams (see section
4261 7.1.6.4);

4262 m_b is the mass of the test specimen after contact with olive oil, in grams (see section 7.1.6.5.2);

4263 m_c is the mass of olive oil absorbed by test specimen, in grams (see section 7.1.6.9.2.3);

4264 S is the surface area of the test specimen in contact with food simulant in square decimetres (see
4265 7.1.5.1.1). Table 4 in section 4.4.3 gives guidelines for selection of the surface area in case of the
4266 immersion test.

4267 Calculate the result for each test specimen to the nearest 0.1 mg/dm².

4268 7.1.7.2 Method of calculation for repeated use

4269 The mean result for the test specimens of the second migration is calculated (M2) as is that for
4270 the test specimens of the third migration (M3).

4271 The migration as a result of the second or third period is calculated as follows:

4272 - migration caused by first period = M1

4273 - migration caused by the second period = M2 - M1

4274 - migration caused by the third period = $M3 - M2$.

4275 No increase in migration into fatty food simulant is deemed to have occurred if the results ($M3 -$
4276 $M2$) and ($M2 - M1$) do not exceed $M1$ by more than the analytical tolerance.

4277 The true values for $M1$, $M2$ or $M3$ are subject to uncertainty owing to the lack of precision
4278 inherent in the method. Systematic errors in the determination of the overall migration are
4279 likely to occur equally to the determination of $M1$, $M2$ or $M3$ and therefore need not be allowed
4280 for. Random errors do need to be recognized and allowed for.

4281 When repeated testing is used to determine the overall migration into a vegetable oil the
4282 individual results for each set of the determinations ($M1$, $M2$ or $M3$) shall be deemed valid if at
4283 least three results are obtained in each set which do not differ from the mean for that set by
4284 more than 30% for results above 10 mg/dm² or by more than 3 mg/dm² for results below 10
4285 mg/dm². Results which exceed this tolerance shall be discarded according to the procedure
4286 given in 11.1.7.2.

4287 **7.1.7.3 Validity of individual results**

4288 The following analytical tolerances are allowed:

4289 20 mg/kg or 3 mg/dm² for all vegetable oils.

4290 If a reduction factor does not apply, results above 10 mg/dm² shall not differ by more than 30%
4291 from the mean of the set of results.

4292 The determination of overall migration into the fatty food simulant is normally carried out in
4293 quadruplicate to allow three valid results to be obtained even if one determination is discarded.

4294 Where four results have been obtained from four determinations, i.e. no single determination
4295 has been rejected because of an obvious manipulative error, all four results are valid when each
4296 individual result differs from the mean of the four results by not more than the analytical
4297 tolerance. However:

4298 • If one of the four results is greater or less than the mean by an amount more than the
4299 tolerance, then this result can be rejected and the mean recalculated on the remaining three
4300 results.

4301 • If two results are greater or less than the mean by amounts more than the tolerance, the
4302 result with the largest difference from the mean can be rejected and a new mean calculated
4303 from the remaining three results. The remaining three test results are valid if they are within
4304 the analytical tolerance.

4305 If a minimum of three results do not meet the above criteria of being within the analytical
4306 tolerance, then the test shall be repeated using fresh test specimens from the sample.

4307 **7.1.7.4 Precision**

4308 Evaluation of the results of a collaborative trial with a plastic film having a mean overall
4309 migration of 6.6 mg/dm², determined by the total immersion method, has given the following
4310 values for repeatability (r) and reproducibility (R):

4311 $r = 2.0 \text{ mg/dm}^2$

4312 $R = 2.9 \text{ mg/dm}^2$

4313 The precision data were determined from an experiment conducted in 1996 involving 11
4314 laboratories and six replicates.

4315 Evaluation of the results of a further collaborative trial with a plastic film having a mean overall
4316 migration of 8.3 mg/dm² and determined by the total immersion method, has given the
4317 following values for repeatability and reproducibility:

4318 $r = 1.8 \text{ mg/dm}^2$

4319 $R = 3.7 \text{ mg/dm}^2$

4320 The precision data were determined from an experiment conducted in 1997 involving eight
4321 laboratories and six replicates.

4322 7.1.8 Test report

4323 The test report shall include the following:

- 4324 • reference to this annex of the guidance document;
- 4325 • all information necessary for complete identification of the sample such as chemical type,
4326 supplier, trade mark, grade, batch number(s), thickness;
- 4327 • conditions of contact time and temperature to food simulants;
- 4328 • Details of the options in this test method used for the determination
- 4329 • deviations from the specified procedure and reasons for these;
- 4330 • individual test results including measurement uncertainty;
- 4331 • relevant comments on the test results;

4332

4333 **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

4334

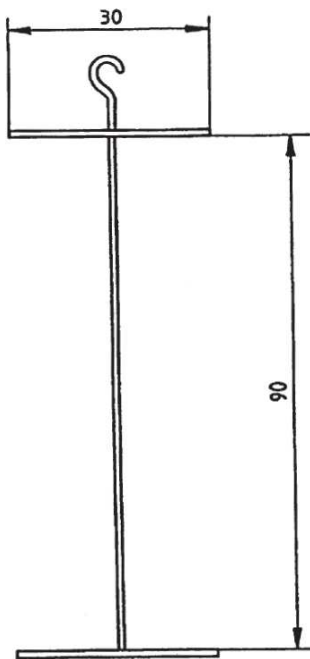
4335

4336 **Table 5 tolerances related to test contact temperature**

Temperature	Tolerance
°C	°C
5	±1
20	±1
30	±1

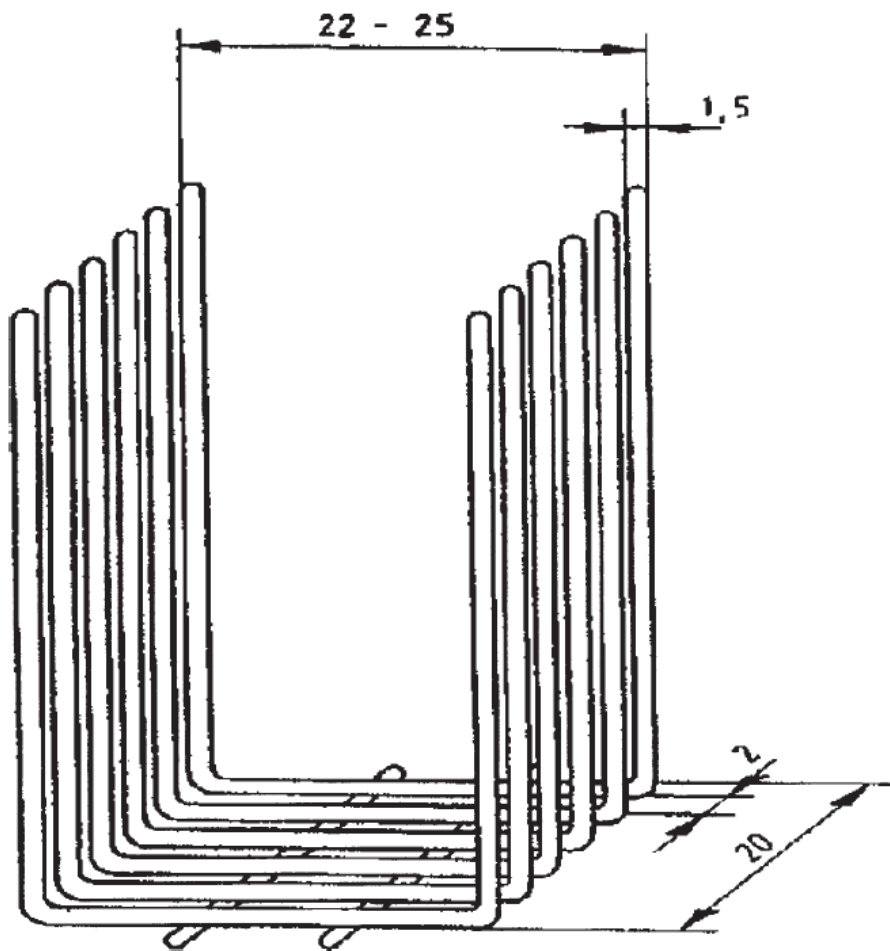
40	±1
50	±2
60	±2
70	±2
80	±3
90	±3
100	±3
121	±3
130	±5
140	±5
150	±5
160	±5
170	±5
175	±5

4337

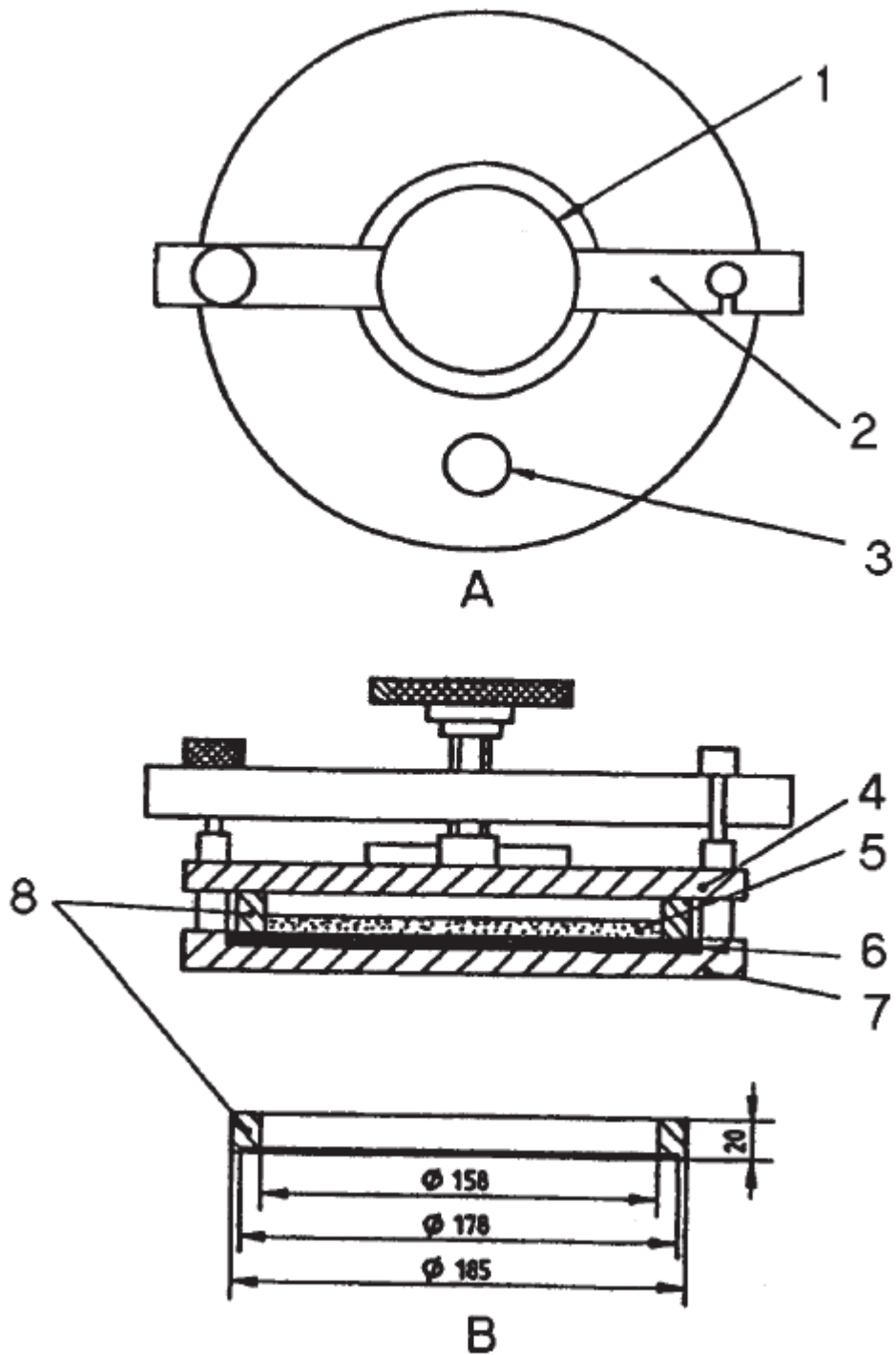


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4339 **Figure 3 support; dimensions in mm**

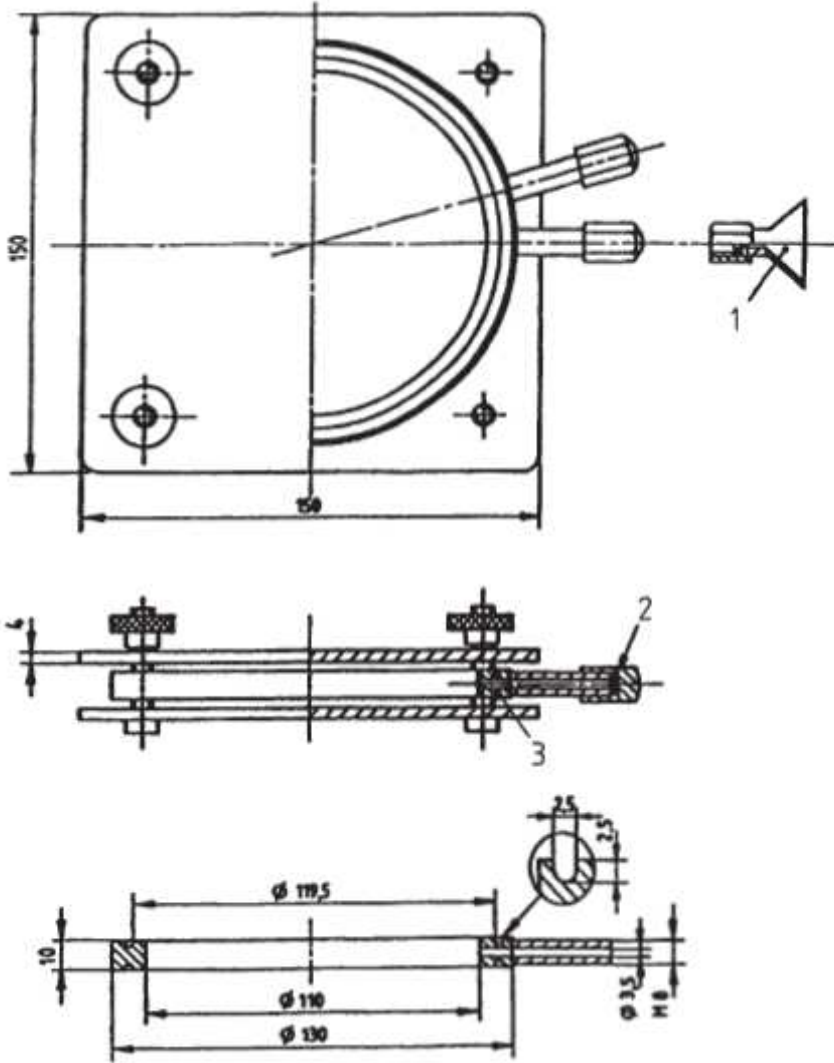


4340
4341 Figure 4 support; dimensions in mm



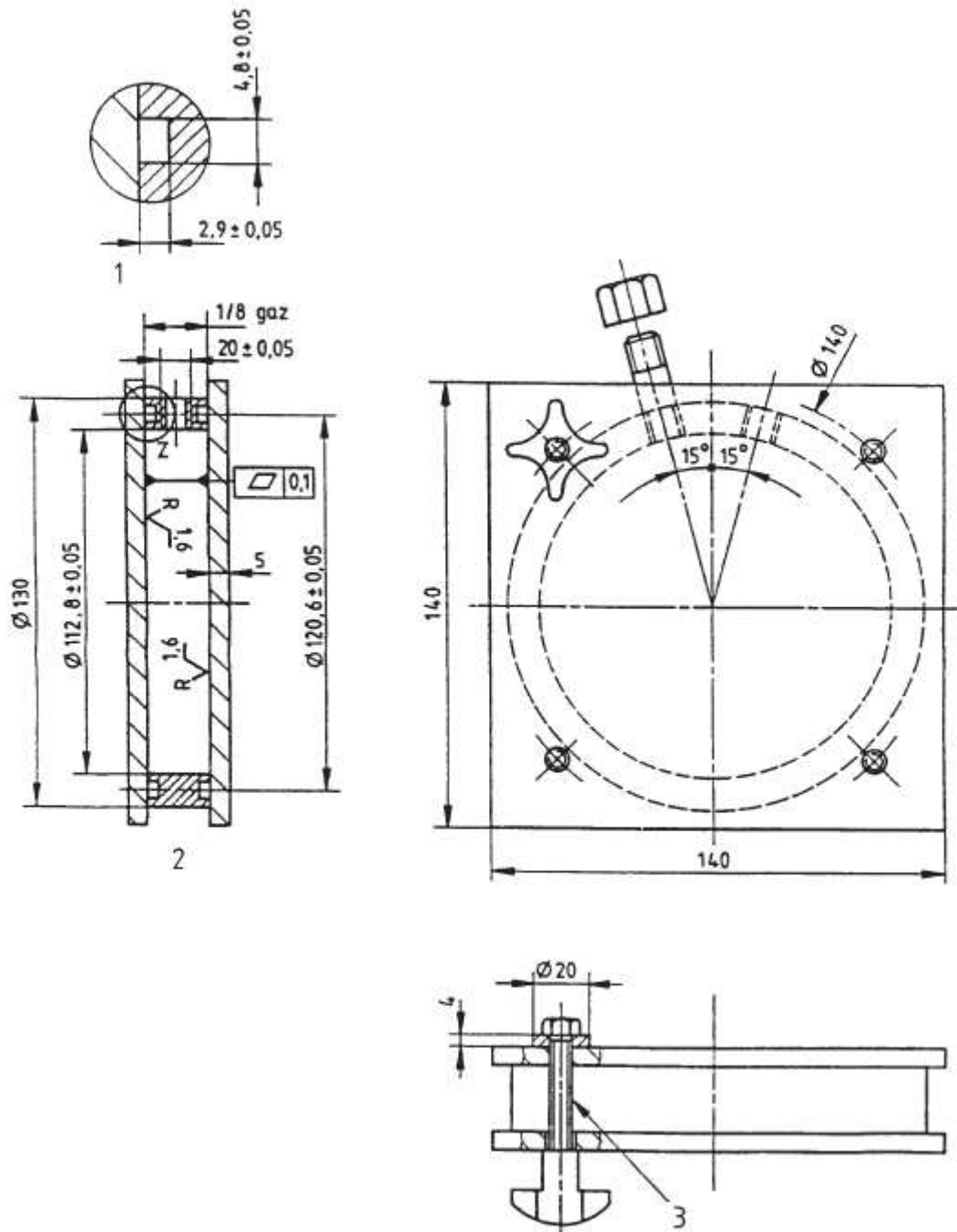
4342

4343 **Figure 5** Cell type A. Dimensions in mm. A, plan elevation; B, side elevation; 1, clamp screw;
 4344 2, clamp bar; 3, filler plug; 4, lid; 5, food simulant; 6, rubber mat; 7, base plate; 8,
 4345 sealing ring



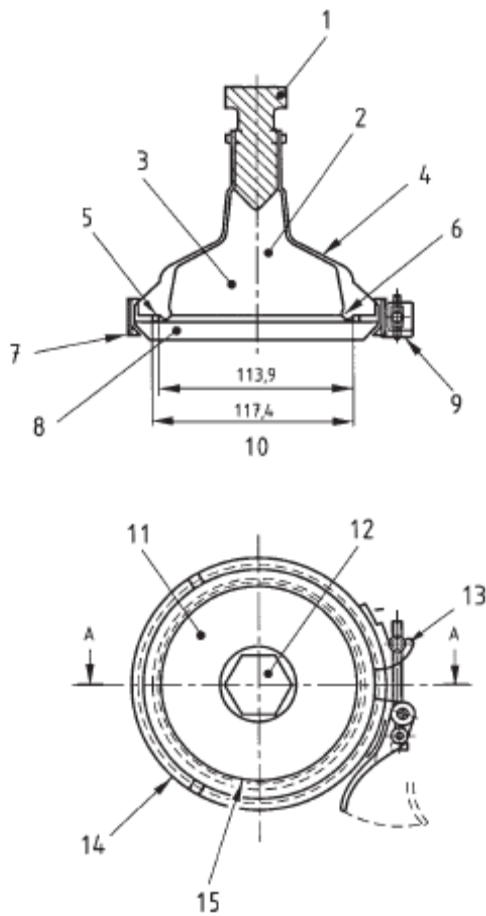
4346
4347
4348

Figure 6 Cell type B. Dimensions in mm. 1, funnel for filling; 2, PTFE disk; 3, PTFE O-ring (119.5 x \varnothing 3)



4349

4350 Figure 7 Cell type C. Dimensions in mm. 1, detail of Z; 2, O-ring ($\text{Ø } 117.07 \times 124.13 \times 3.53$); 3,
 4351 screw HM8-50



4352

4353

4354

4355

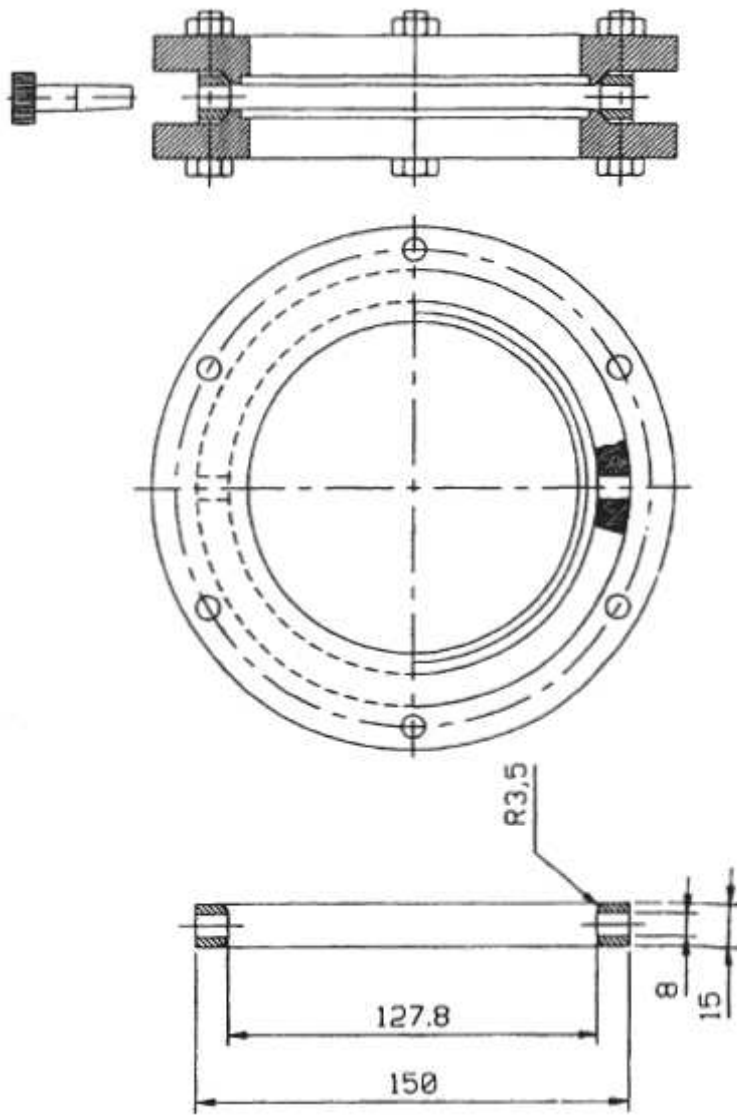
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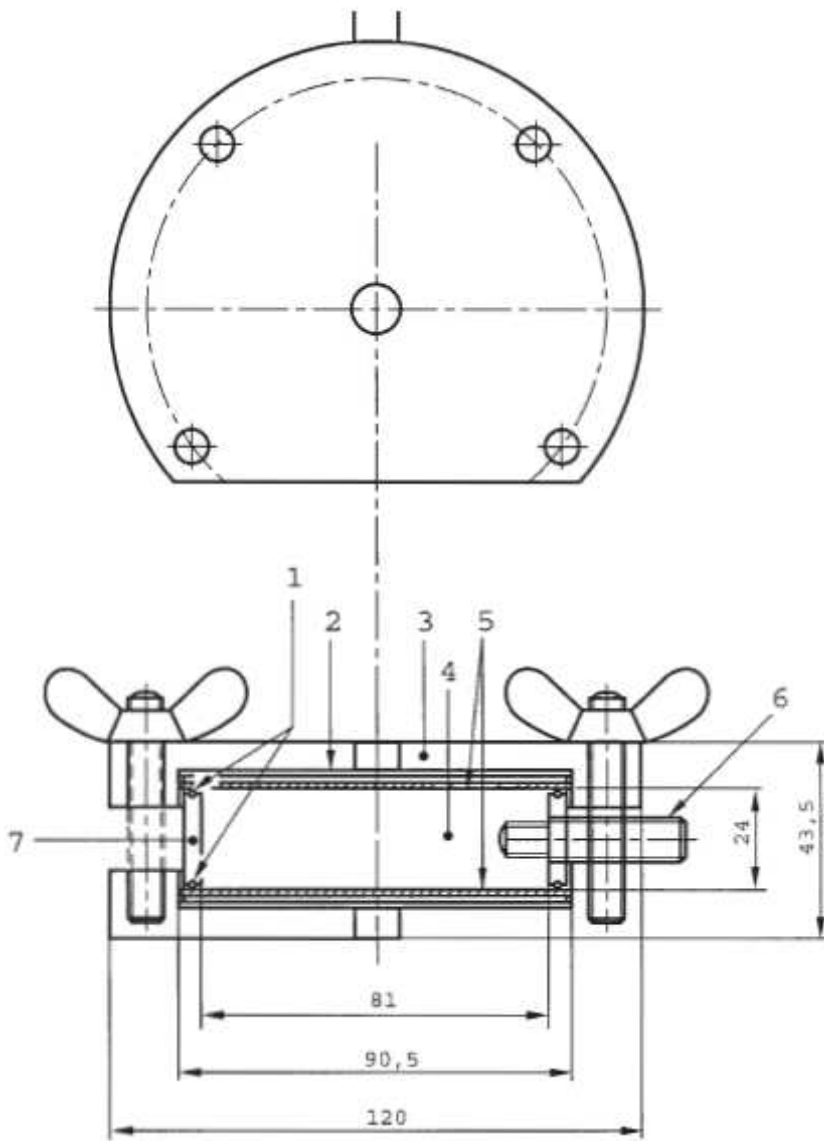
4359

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 dm²; 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



4360

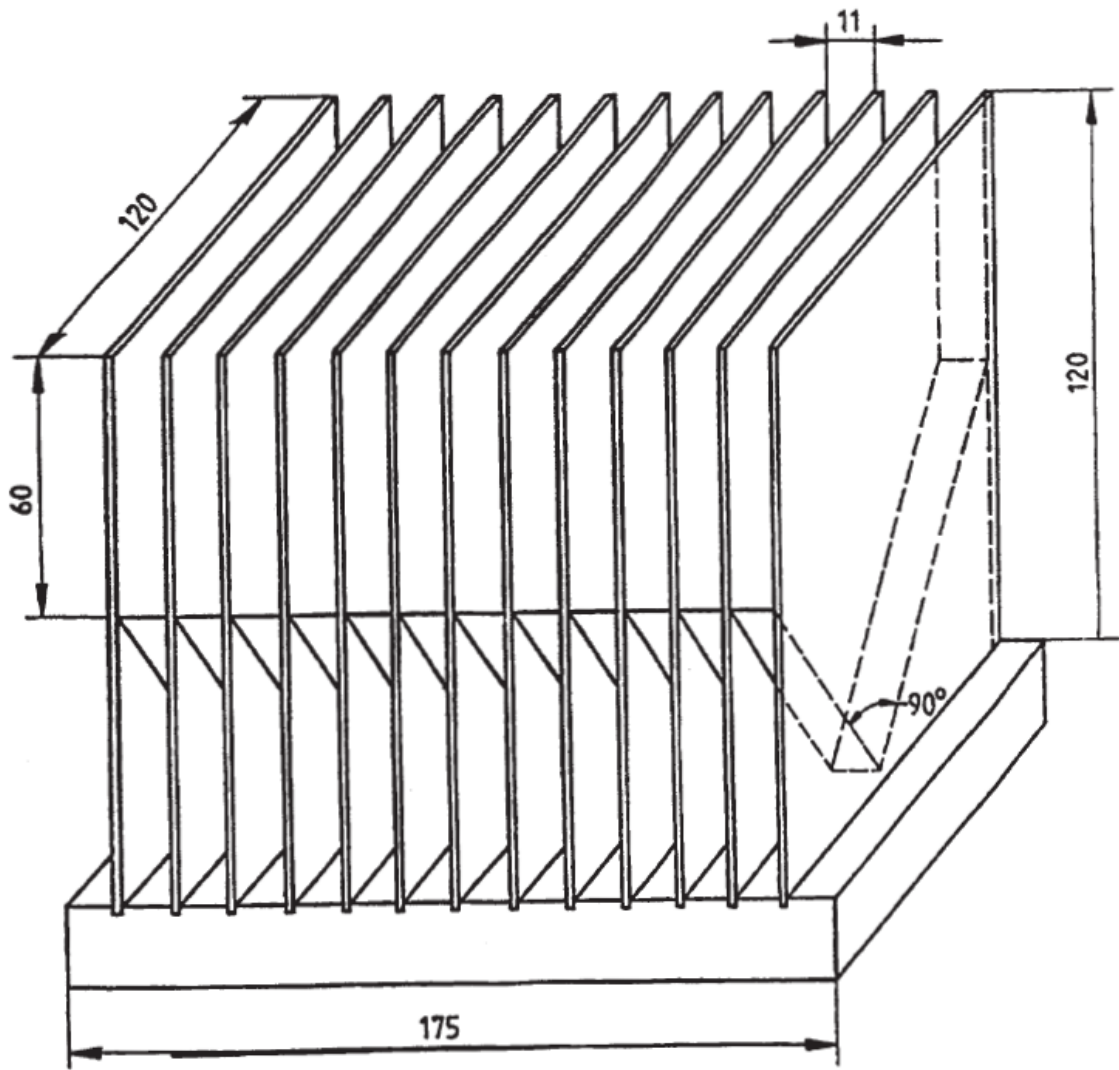
4361 **Figure 9** Cell type E. Dimensions in mm.



4362

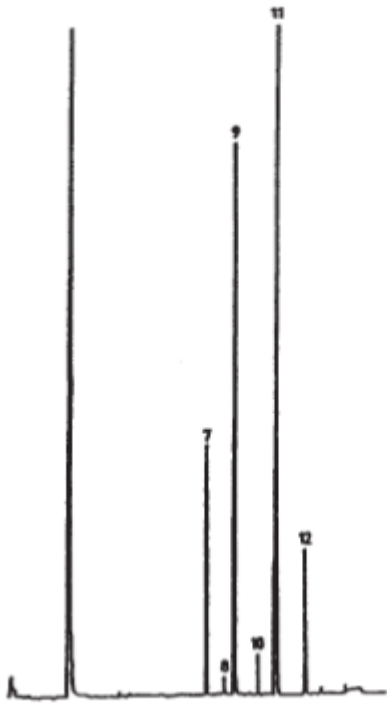
4363 **Figure 10 Cell type F. Dimensions in mm. 1, sealing ring; 2, lid (stainless steel); 3, body**
 4364 **(aluminium); 4, food simulant; 5, test sample; 6, stopper (PTFE); 7, ring (stainless steel)**

4365



4366

4367 Figure 11 Pouch holder. Dimensions in mm

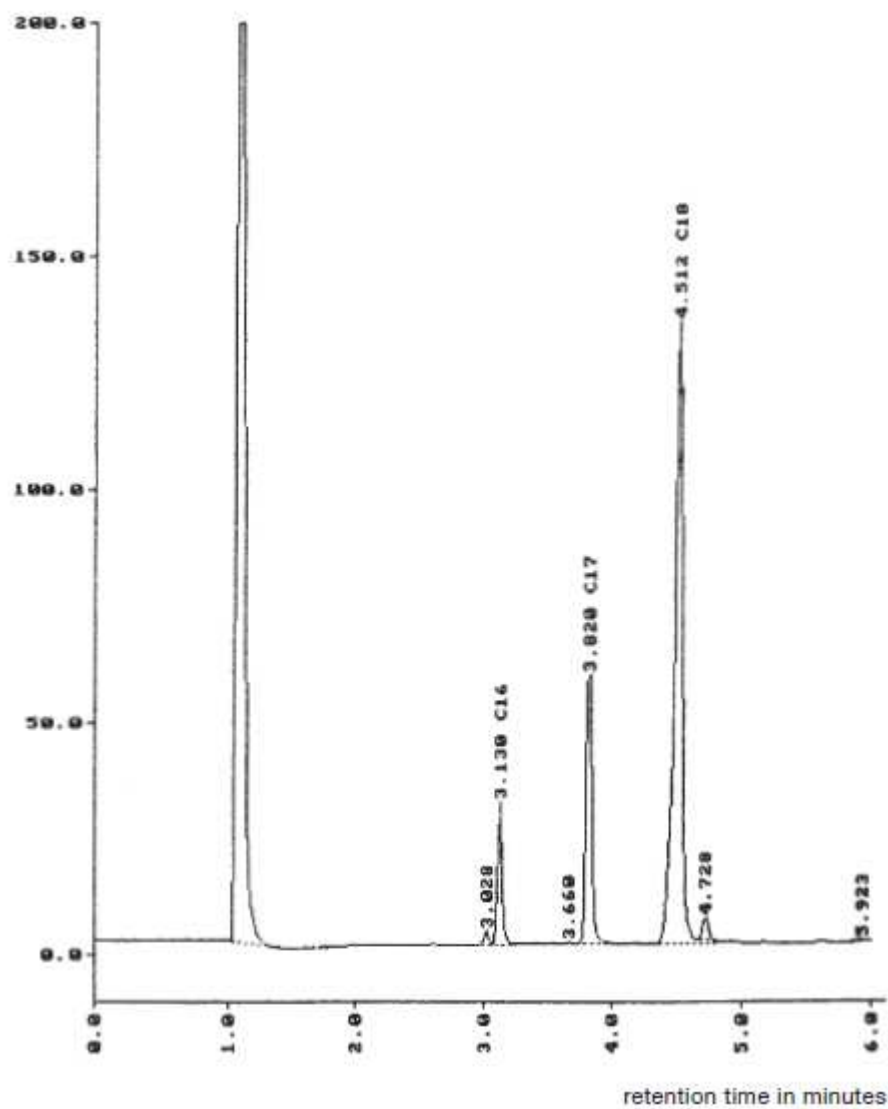


7 = C16:0 8 = C16:1 9 = C17:0 10 = C18:0 11 = C18:1 12 = C18:2

4368

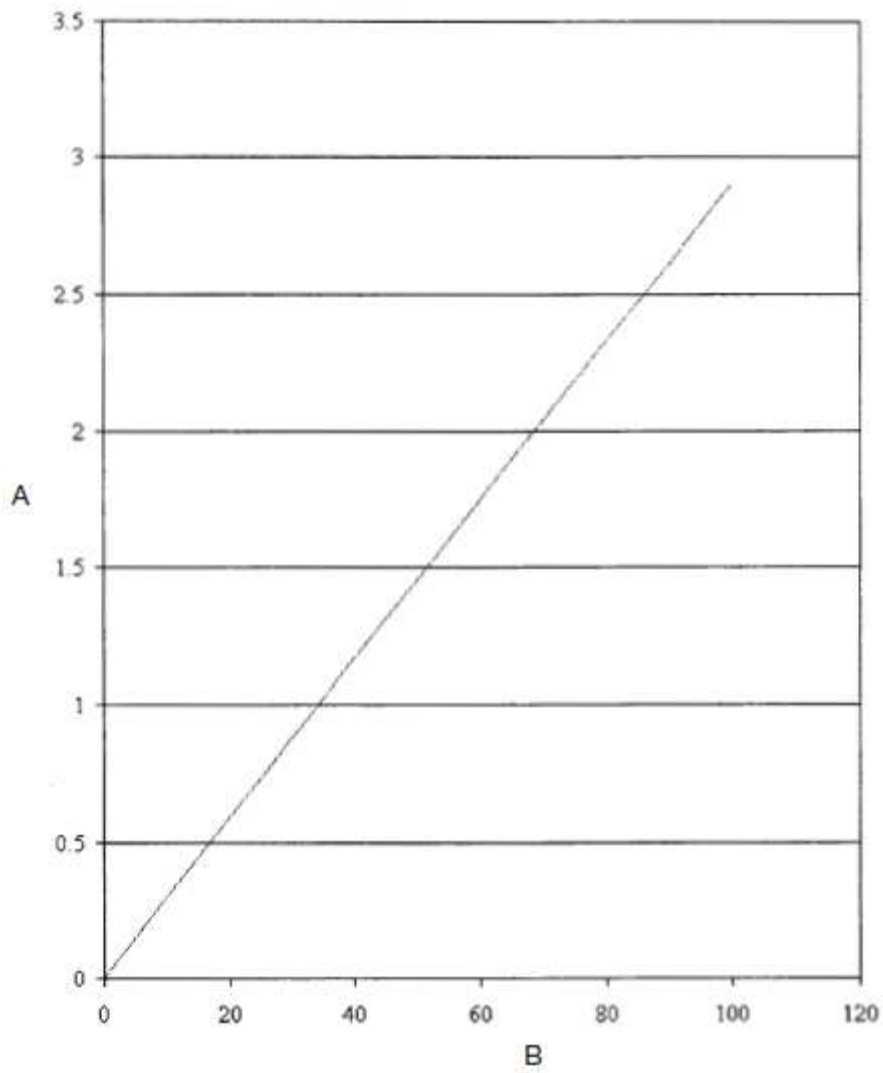
4369

Figure 12 Typical chromatogram of column 1 (7.1.4.23)



4370

4371 Figure 13 Typical chromatogram of column 2 (7.1.4.23)



4372

4373 **Figure 14** Typical calibration graph. A, peak area ratio (C18 + C18)/C17; B, mg of olive oil

4374

4375

4376 **7.2 Test method for overall migration into vegetable oil in the temperature**
4377 **range of 5-20°C**

4378 **7.2.1 Scope**

4379 The scope of this method is described in Annex 7.1.1. Only the deviations are mentioned here.

4380 This method specifies test methods for the determination of the overall migration into vegetable
4381 oil from plastics materials and articles, at contact temperatures from 5°C up to and including
4382 20°C.

4383 The oil used in this test method is de-waxed sunflower oil since, unlike olive oil, this remains
4384 liquid at the lower test temperature.

4385 **7.2.2 Principle**

4386 The principle of this method is described in Annex 7.1.2. Only the deviations are mentioned here.

4387 Test specimens of known mass are immersed in, filled with or put into contact to de-waxed
4388 sunflower oil for the contact time, at contact temperatures in the range of 5-20°C.

4389 **7.2.3 Reagents**

4390 The reagents shall be as described in Annex 7.1.3, except that olive oil is replaced by de-waxed
4391 sunflower oil.

4392 **7.2.4 Apparatus**

4393 The apparatus shall be as described in Annex 7.1.4.

4394 **7.2.5 Preparation of test specimens**

4395 Test specimens shall be prepared as described in Annex 7.1.5.

4396 **7.2.6 Procedure**

4397 Perform sections 7.1.6.1 to 7.1.6.4

4398 Perform the contact with the de-waxed sunflower oil as described in section 7.1.6.5 or 7.1.6.6
4399 followed by sections 7.1.6.7 and 7.1.6.8.

4400 Determine the extracted de-waxed sunflower oil in accordance with Annex 7.1.6.9.

4401 NOTE: The fatty acid pattern of de-waxed sunflower oil will show C18:2 as the major peak. In
4402 addition C18:1 will be present in significant amounts. C16:0 and C18:0 may be present in minor
4403 amounts. For quantification of the extracted amount of oil the peaks obtained for C18:1 and
4404 C18:2 may be used.

4405 **7.2.7 Expression of results**

4406 Follow section 7.1.7

4407 **7.2.8 Test report**

4408 Prepare the test report in accordance with Annex 7.1.8.

4409

4410

4411

4412

4413 **7.3 Test method for overall migration into vegetable oil in the temperature** 4414 **range of 100-175°C**

4415

4416 **7.3.1 Introduction**

4417 Migration testing with olive oil at high temperatures introduces a number of analytical
4418 difficulties. Experience has shown that it is difficult to achieve reproducible results owing to
4419 different laboratories having different equipment which give rise to variations in the time taken
4420 to reach the contact temperature. A method is described for determining overall migration by
4421 total immersion using an aluminium block with a consistent thermal capacity. Other analytical
4422 difficulties with olive oil include possible oxidation of oil at elevated temperatures and the
4423 hazard to personnel working with hot oil.

4424

4425 **7.3.2 Scope**

4426 This method specifies a test method for the determination of the overall migration into fatty
4427 food simulants from plastics materials and articles, by total immersion of test specimens in a
4428 fatty food simulant at temperatures from 100°C up to and including, 175 °C for selected contact
4429 times. Another procedure, to be applied at temperatures >175°C, uses food simulant E, i.e.
4430 poly(2,6-diphenyl-p-phenylene oxide). In this procedure the mass of substances adsorbed on
4431 food simulant E is taken as a measure for the assessment of the overall migration into olive oil.

4432 NOTE 1 The total immersion test method has been written for use with olive oil. The test
4433 method can also be used with appropriate modifications with other vegetable oils. These
4434 other vegetable oils produce different chromatograms for the simulant methyl esters to those
4435 of the methyl esters of olive oil. Suitable chromatogram peaks of the methyl esters of the
4436 other vegetable oils should be selected for the quantitative determination of the oil extracted
4437 from the test specimens.

4438 NOTE 2: A comparative migration test carried out with polypropylene and polyethylene
4439 terephthalate high temperature application containers as test samples at conditions of 2 h at
4440 100 °C and 2 h at 175 °C, respectively, in contact with ¹⁴C-labelled synthetic triglyceride and
4441 food simulant E provided test results comparable within the analytical tolerance of the
4442 methods.

4443 NOTE 3: To obtain reproducible and repeatable results using the food simulant E method, it
4444 may be necessary to measure the temperature of the test specimen before starting the
4445 migration period. An appropriate method for measuring the temperature of the test specimen
4446 needs to be established.

4447 NOTE 4: The described method is written to determine the overall migration by total
4448 immersion using an aluminium block for consistent contact temperature and preheating of
4449 the simulant. However, when using an oven with forced air circulation (e.g. GC oven), samples
4450 may be tested by filling (article, pouch) or in a cell with oil at room temperature, or slightly
4451 preheated oil (e.g. 70°C). After bringing the test specimens in contact with the oil, they are
4452 placed in the oven and the temperature of the oil of one of the test specimen is recorded. As
4453 soon as the oil reaches the intended temperature then the contact time is started. At the end
4454 of the contact time the oil is removed immediately or allowed to cool down to a more safe
4455 temperature to handle the oil.

4456 The described methods are most suitable for food contact articles in the form of sheets and
4457 films, but can also be applied to a wide range of articles and containers.

4458

4459 **7.3.3 Method of total immersion in olive oil**

4460 **7.3.3.1 Principle**

4461 The scope of this method is described in Annex 7.1.1. Only the deviations are mentioned here.

4462 Test specimens of known mass are immersed in olive oil for the contact time, at contact
4463 temperatures from 100°C up to 175 °C.

4464 **7.3.3.2 Reagents**

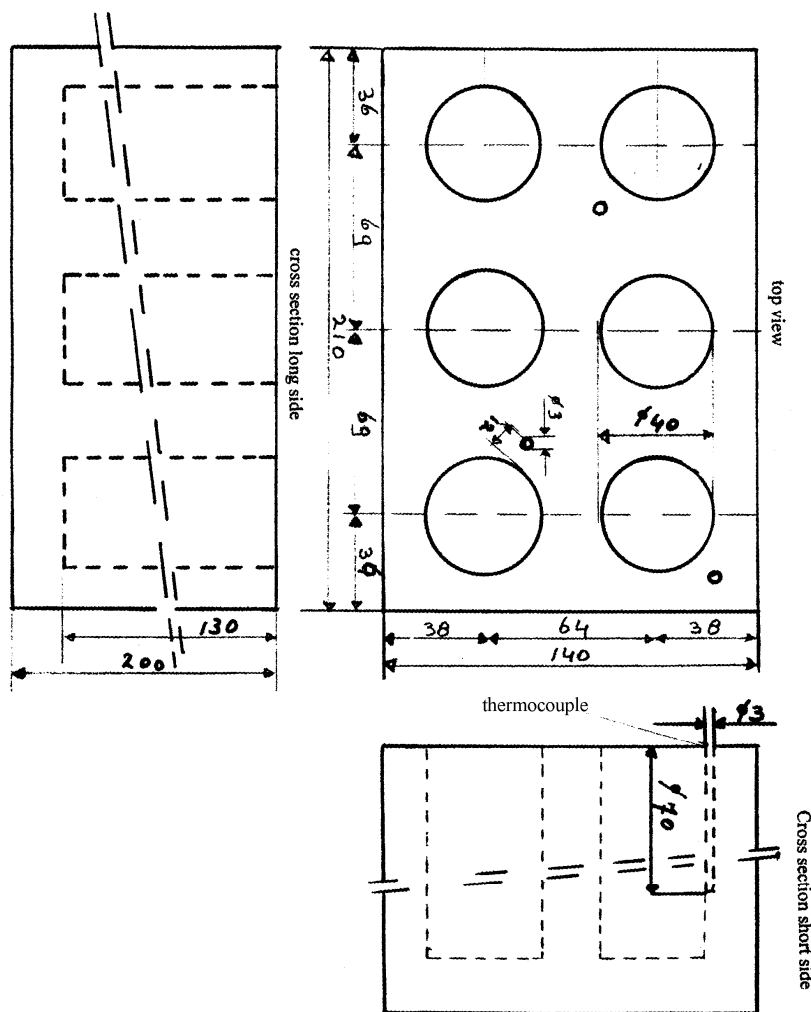
4465 The reagents shall be as described in Annex 7.1.3

4466 **7.3.3.3 Apparatus**

4467 The apparatus shall be as described in Annex 7.1.4, with the addition of:

4468 Aluminium block or blocks with wells for holding up to ten glass tubes (Annex 7.1.4.11) during
4469 the contact time period in the oven or incubator.

4470 NOTE A diagram of a suitable block is shown in Figure 15. The wells in the block should hold
4471 the tubes so that there is close contact between the tubes and the block. The block should be
4472 of sufficient depth that when the test specimen is placed in the oil in the tubes, the level of the
4473 oil is lower than, or equal to the height of the block.



UN

4474

4475 **Figure 15 Aluminium block (dimensions in mm). 1) cross section long side, 2) top view, 3)**
 4476 **thermocouple and 4) cross section short side.**

4477 **7.3.3.4 Preparation of test specimens**

4478 Test specimens shall be prepared as described in Annex 7.1.5, except that an additional test
 4479 specimen is required. This test specimen shall be placed in the tube in which the temperature is
 4480 monitored.

4481 **7.3.3.5 Procedure**

4482 Perform sections 7.1.6.1 to 7.1.6.4

4483 **7.3.3.5.1 Contact with food simulant for single use**

4484 Insert a thermocouple in the metal block (Annex 7.3.3.3). Place the metal block and
 4485 thermocouple in the thermostatically controlled oven or incubator, set at the test temperature
 4486 and leave for 24 h.

4487 Observe the temperature of the metal block. Confirm that the temperature of the metal block
 4488 reaches the contact temperature, taking into account the tolerances specified in Table 5.

4489 NOTE 1 If the oven temperature is at the contact temperature, but the temperature of the
4490 metal block is not, taking into account the tolerances specified in Table 5, the accuracy of the
4491 thermocouple and the temperature control of the oven should be checked. The oven
4492 temperature should be adjusted, if necessary, either by increasing or decreasing the oven set
4493 temperature, until the temperature of the metal block reaches the contact temperature.

4494 Take six of the glass tubes (7.1.4.11), mark them for identification purposes. Measure 100 ml \pm 5
4495 ml of olive oil (7.1.3.1) into each tube by measuring cylinder and stopper the tube.

4496 Alternatively mark the tubes for a volume of 100 ml and fill with olive oil to the mark. Place into
4497 one of the tubes a thermometer or thermocouple and stopper the tubes. Place the tubes in the
4498 thermostatically controlled oven or incubator (7.1.4.12) set at the test contact temperature.
4499 Leave until the olive oil has attained the test contact temperature, using the thermometer or
4500 thermocouple to monitor the temperature.

4501 NOTE 3 More rapid temperature equilibration of the oil can be established by lifting the tubes
4502 from the block periodically and rotating gently before replacing rapidly or by stirring the oil
4503 in the tubes with a metal or glass rod, without removing from the block.

4504 Place into five of the tubes containing olive oil, weighed test specimens prepared as in 7.3.3.4
4505 and condition if necessary. Place into one of the tubes a thermometer or thermocouple and
4506 stopper the tubes. Ensure that the test specimens are totally immersed in olive oil; if they are
4507 not, then add either glass beads or glass rods (Annex 7.1.4.22) to raise the level of the olive oil
4508 until total immersion is achieved.

4509 WARNING 1 Take care when handling the hot metal block or blocks during removal from and
4510 replacement into the oven or incubator, to prevent skin burns. Take particular care when
4511 placing the test specimens in the hot olive oil to prevent splashing or spillage of the olive oil
4512 on to the skin. It is recommended that heat protective gloves and a face shield are worn.

4513 NOTE 4 The olive oil in the sixth tube is used as a reference standard in constructing the
4514 calibration graph.

4515 Place one test specimen in the sixth tube where the temperature of the oil is being monitored.

4516 Replace the block or blocks containing all tubes in the thermostatically controlled oven or
4517 incubator set at the contact temperature. This part of the operation should be carried out in the
4518 minimum time to prevent undue heat loss.

4519 NOTE 5 Where the contact time during the migration test is in the range of 0,5-4 h, it is
4520 particularly important that the time period between placing the test specimens in the olive oil
4521 and the olive oil reaching the contact temperature, be kept as short as possible. This time
4522 period can be minimized by placing the test specimens in the tubes without removing the
4523 tubes from the block and without removing the block from the oven or incubator. It can be
4524 necessary to raise the tubes from the block to check that the oil is above the level of the
4525 sample and that the test pieces remain separated. The tubes should be raised the minimum
4526 amount necessary to carry out these checks and then replaced as quickly as possible. All
4527 tubes should be treated in a similar manner.

4528 Using a higher food simulant temperature is a risk for compliance testing but it can be
4529 applied for high test contact temperatures of $\geq 175^{\circ}\text{C}$, because the temperature drops very
4530 quickly below the 175°C after contact with the test specimen. Be aware that in such cases the
4531 temperature profile should be measured and the results should be added to your raw data.

4532 Observe the temperature of the thermostatically controlled oven or incubator or the olive oil in
4533 the tube and leave the tubes for the selected test period, taking into account the tolerances
4534 specified in Table 4, after the olive oil in the tube has reached a temperature within the tolerance
4535 specified in Table 5. For test temperatures in the range of $100\text{-}150^{\circ}\text{C}$, the time between the
4536 immersion of the test specimens and the test temperature being regained shall be 10 min or less.
4537 For test temperatures in the range of $151\text{-}175^{\circ}\text{C}$ the time shall be 15 min or less.

- 4538 NOTE 7 It has been found that these times can be achieved by using silicone oil in the wells to
4539 increase thermal conductivity between the block and the tube.
- 4540 NOTE 8 Table 4 and Table 5 include tolerances on a wide range of contact times and contact
4541 temperatures. All of these contact times and contact temperatures are not necessarily
4542 relevant to this standard.
- 4543 Take the metal block or blocks containing the tubes from the oven or incubator and immediately
4544 remove the test specimens from the tubes. Discard the test specimen that was in the tube in
4545 which the temperature had been monitored. For the remaining test specimens, which have been
4546 in olive oil, allow the oil to drain.
- 4547 WARNING 2 Take care when handling the metal block or blocks and removing the test
4548 specimens from the olive oil at the end of the contact period, to prevent skin burns. Use the
4549 protective wear recommended in WARNING 1.
- 4550 Remove any adhering olive oil by gently pressing between filter papers (7.1.4.13). Repeat the
4551 pressing procedure until the filter paper shows no spots of olive oil. For test specimens on
4552 supports, remove the individual test pieces from the supports to carry out this operation. Clean
4553 the supports of oil by washing with the extraction solvent and replace the test pieces on them.
- 4554 7.3.3.5.2 Contact with food simulant for repeated use
- 4555 Perform the procedure as described in section 7.1.6.6 using section 7.3.3.5.1 in stead of section
4556 7.1.6.5.
- 4557 7.3.3.5.3
- 4558 Perform sections 7.1.6.7. to 7.1.6.9.
- 4559 **7.3.3.6 Expression of results**
- 4560 7.3.3.6.1 Method of calculation
- 4561 Perform sections 7.1.7.1 to 7.1.7.4.
- 4562 **7.3.3.7 Test report**
- 4563 Prepare the test report in accordance with Annex 7.1.8.
- 4564
- 4565 **7.3.4 Test method of adsorption by poly(2,6-diphenyl-p-phenylene oxide)**
- 4566 **7.3.4.1 Principle**
- 4567 The surface of the article to be tested is covered with poly(2,6-diphenyl-p-phenylene oxide) and
4568 is held at the selected contact time-temperature test conditions where the maximum
4569 temperature applicable is 225°C. If the material is used in a microwave oven the material can be
4570 tested in a conventional oven using EN 14233 (see footnote 1 on page 24 of main text). The
4571 migration is followed by extraction of the adsorbent using diethyl ether. Finally, the extract is
4572 evaporated to dryness using a nitrogen stream and the residue remaining is determined
4573 gravimetrically. poly(2,6-diphenyl-p-phenylene oxide) is a porous polymer with a high
4574 molecular mass in the range of 500,000-1,000,000 amu, a very high temperature stability ($T_m =$
4575 350°C), a high specific surface area of 35 m²/gram and a low specific mass (0.25 g/cm³). Its pore
4576 volume is 2.4 cm³/g and the average pore size is 200 nm.
- 4577 **7.3.4.2 Reagents**
- 4578 All reagents shall be of recognized analytical quality, unless otherwise specified.
- 4579 7.3.4.2.1 Diethylether
- 4580 99.8% purity and stabilized with 1.5-2,5% of ethanol.

4581 7.3.4.2.2 Poly(2,6-diphenyl-p-phenylene oxide)
4582 60-80 mesh; New poly(2,6-diphenyl-p-phenylene oxide) shall be cleaned following one of the
4583 procedures in Annex 3.

4584 7.3.4.2.3 Nitrogen
4585 purity 99.999%.

4586 **7.3.4.3 Apparatus**

4587 7.3.4.3.1 Cutting slab (Annex 7.1.4.1)

4588 7.3.4.3.2 Cutting implement, scalpel, scissors, sharp knife or other suitable device (Annex
4589 7.1.4.3), .

4590 7.3.4.3.3 Rule (Annex 7.1.4.6)

4591 7.3.4.3.4 Analytical balance (Annex 7.1.4.7)

4592 7.3.4.3.5 Oven or incubator (Annex 7.1.4.12)

4593 NOTE In case of an oven with a ventilating system the ventilation rate should be switched to low.

4594 7.3.4.3.6 Petri dishes
4595 Made of glass with an internal diameter of 140 mm; heat resistant.

4596 7.3.4.3.7 Rings
4597 Made of glass with an internal diameter of 125 mm and an external diameter of approximately
4598 130 mm.

4599 7.3.4.3.8 Glass-stoppered Erlenmeyer flasks
4600 capacity of 300 ml.
4601 NOTE The size of the flask that is used to wash poly(2,6-diphenyl-p-phenylene oxide) depends
4602 on the mass that is used (see Table 6).

4603 7.3.4.3.9 Glass filter funnels,.

4604 7.3.4.3.10 Folded filter
4605 with a diameter of 125 mm.

4606 7.3.4.3.11 Glass vials
4607 with capacities of 10 ml and 100 ml.

4608 7.3.4.3.12 Dropping pipettes with dropper teats.

4609 7.3.4.3.13 Lint-free cloth (Annex 7.1.4.35)

4610 7.3.4.3.14 Glass plates
4611 to cover the dishes or trays.

4612 7.3.4.3.15 Apparatus to blow off solvent with nitrogen

4613 **7.3.4.4 Preparation of test specimens**

4614 7.3.4.4.1 General
4615 It is essential that test specimens are clean and free from surface contamination (many plastics
4616 can readily attract dust due to static charges). Before preparing test specimens, remove any

4617 surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing
4618 with a soft brush. Under no circumstances wash the sample with water or solvent. If it is
4619 specified in the instructions for use of the article that it should be washed or cleaned before use
4620 (7.1.5.1). Minimize handling of the samples and where necessary, wear cotton gloves.

4621 7.3.4.4.2 Number of test specimens

4622 Three test specimens are required for the test.

4623 7.3.4.4.3 Films and sheets

4624 Lay the sample on a cutting slab (Annex 7.1.4.1). Take the glass ring (Annex 7.3.4.3.7) and place
4625 on the surface of the sample. Cut out the test specimen by cutting round the outside of the glass
4626 ring, using the cutting implement (Annex 7.1.4.3).

4627 NOTE Taking the inner diameter of the glass ring into account the effective contact area
4628 obtained in this way is 1.22 dm².

4629 7.3.4.4.4 Containers and other articles

4630 Articles do not have to be cut if it is possible to cover these samples with pieces of glass. In this
4631 case, determine the flat bottom area of the article to find the required mass of adsorbent (see
4632 Annex 7.3.4.4.5).

4633 7.3.4.4.5 Preparation of poly(2,6-diphenyl-p-phenylene oxide)

4634 To cover the food contact surface sufficiently, 4 g of poly(2,6-diphenyl-p-phenylene oxide) per
4635 square decimetre of surface area of the test specimen is required.

4636 **7.3.4.5 Procedure**

4637 7.3.4.5.1 Contact with poly(2,6-diphenyl-p-phenylene oxide)

4638 For flexible thin film and sheet materials (Annex 7.3.4.4.3), take four Petri dishes (Annex
4639 7.3.4.3.6) and place a prepared test specimen into each dish. Stabilize the test specimen with a
4640 glass ring (Annex 7.3.4.3.7) and place 4.8 g poly(2,6-diphenyl-p-phenylene oxide) evenly on the
4641 surface of each test specimen, inside the glass ring. Close the Petri dishes.

4642 For rigid containers and other articles (Annex 7.3.4.4.4) which are to be tested as a whole, cover
4643 the flat bottom of the article with the required amount of poly(2,6-diphenyl-p-phenylene oxide).
4644 Calculate the required amount of poly(2,6-diphenyl-p-phenylene oxide) according to the flat
4645 bottom food contact surface area which can be covered (Annex 7.3.4.4.5). Weigh the appropriate
4646 mass of poly(2,6-diphenyl-p-phenylene oxide) with an accuracy of ± 0.1 g and place it on the flat
4647 bottom area of the test specimen. Cover each of the three articles with a glass plate (Annex
4648 7.3.4.3.14).

4649 NOTE 1 When the test sample, prepared as described, is placed in the oven the time required
4650 to reach the intended contact temperature can be significant when compared to the intended
4651 contact time and the allowed tolerances on contact time and contact temperature. Therefore
4652 it can be necessary to include a procedure for the control of the time and temperature of the
4653 contact of the test specimens with poly(2,6-diphenyl-p-phenylene oxide), in order to achieve
4654 reproducible and repeatable results.

4655 NOTE 2 In the case of articles of irregular geometry and with no flat areas, a corresponding
4656 way needs to be found to expose the food contact surface to poly(2,6-diphenyl-p-phenylene
4657 oxide). Possible solutions are to cut appropriate parts from the article and cover or mix them
4658 with poly(2,6-diphenyl-p-phenylene oxide), using the conventional mass of poly(2,6-
4659 diphenyl-p-phenylene oxide) to food contact area ratio (Annex 7.3.4.4.5). If necessary, higher
4660 amounts of poly(2,6-diphenyl-p-phenylene oxide) should be used to ensure complete contact
4661 between the test specimen and poly(2,6-diphenyl-p-phenylene oxide).

4662 For the blank determination, take an empty Petri dish and put in the same mass of poly(2,6-
4663 diphenyl-p-phenylene oxide) as was put on each test specimen, and cover the dish, Set the oven

4664 (section 7.3.4.3.5) at the required test temperature and observe the temperature. When the oven
4665 has reached the test temperature place the blank and the four Petri dishes prepared with test
4666 specimens in the oven.

4667 Observe the temperature and leave the test specimens for the selected period of contact time
4668 after the temperature of the oven has reached a temperature within the permitted tolerance for
4669 the test temperature, see Table 4 and Table 5 for tolerances on time and temperature.

4670 Remove the test specimens from the oven and allow them to cool to room temperature without
4671 removing the glass covers.

4672 NOTE 3 Cooling to room temperature takes approximately half an hour.

4673 7.3.4.5.2 Determination of the migrating substances

4674 Transfer the poly(2,6-diphenyl-p-phenylene oxide) into the Erlenmeyer flask (Annex 7.3.4.3.8)
4675 with the aid of a funnel (7.3.4.3.9). If necessary use a brush for complete transfer of the poly(2,6-
4676 diphenyl-p-phenylene oxide).

4677 Calculate, by reference to Table 6, the volume of diethylether needed for extraction of the
4678 poly(2,6-diphenyl-p-phenylene oxide) and add this volume to the poly(2,6-diphenyl-p-
4679 phenylene oxide) in the Erlenmeyer flask.

4680 **Table 6 Volumes of diethylether needed for the extraction of poly(2,6-diphenyl-p-phenylene**
4681 **oxide) (PPPO)**

Mass of PPPO	Extraction volume of diethylether		
	1 st	2 nd	3 rd
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

4682

4683 Pour the diethylether through the funnel (Annex 7.3.4.3.9) into the Erlenmeyer flask and shake
4684 it manually for 1 min. Allow the Erlenmeyer flask and its contents to stand for 1 min, without
4685 shaking.

4686 Place a folded filter (Annex 7.3.4.3.10) into the funnel and insert the funnel into the 100 ml vial
4687 (Annex 7.3.4.3.11). Decant the diethylether from the Erlenmeyer flask through the filter into the
4688 vial.

4689 Repeat this extraction procedure twice, using the diethylether volumes given in Table 1.

4690 NOTE During the extraction of poly(2,6-diphenyl-p-phenylene oxide) the solvent is decanted
4691 from the extract at each washing step. Considerable amounts of solvent remain on the

4692 poly(2,6-diphenyl-p-phenylene oxide) after each washing step which allows the use of a 100
4693 ml volume capacity vial. If necessary, a second 100 ml vial should be used.

4694 Rinse the filter with 10 ml diethylether and concentrate the combined diethylether solutions to
4695 approximately 5 ml, first by using a rotary evaporator and finally with the aid of a gentle
4696 nitrogen flow (Annex 7.3.4.3.15).

4697 Weigh, with an accuracy of ± 0.1 mg, a 10 ml vial (Annex 7.3.4.3.11) for each test specimen and
4698 for the blank determination.

4699 Transfer each of the concentrated extracts, quantitatively, into the prepared 10 ml vials using a
4700 dropping pipette (Annex 7.3.4.3.12) and include a rinsing step using 5 ml of diethylether.

4701 Evaporate the concentrates to dryness, using a stream of nitrogen, until constant mass has been
4702 achieved in the following way; evaporate to dryness (which takes approximately 30 min.) and
4703 remove the condensed water formed at the outside of the glass vial. Under a stream of nitrogen
4704 monitor the mass of the residue remaining in the glass vial every 5 minutes. Constant mass has
4705 been achieved when, after the second weighing, the mass difference is equal to or smaller than
4706 0.5 mg.

4707 Determine the mass of residue by subtracting the original mass of the vial from the stable mass
4708 of the vial and residue.

4709 **7.3.4.6 Expression of results**

4710 7.3.4.6.1 Method of calculation

4711 Express the amount of material adsorbed onto poly(2,6-diphenyl-p-phenylene oxide) as
4712 milligrams lost per square decimetre of the test specimen taking only that area into account
4713 which was covered by the poly(2,6-diphenyl-p-phenylene oxide). Calculate the adsorbed
4714 amount, M , for each specimen according to the following formula:

$$M = \frac{m_a - m_b}{S}$$

4715

4716 where

4717 M is the mass of migrated substances adsorbed onto poly(2,6-diphenyl-p-phenylene oxide)
4718 from the test specimen, in milligrams per square decimetre;

4719 m_a is the mass of residue from the poly(2,6-diphenyl-p-phenylene oxide) that had been in
4720 contact with the test specimen, in milligrams;

4721 m_b is the mass of residue from the poly(2,6-diphenyl-p-phenylene oxide) that had not been
4722 in contact with the test specimen, in milligrams;

4723 S is the surface area of the test specimen that was in contact with the poly(2,6-diphenyl-p-
4724 phenylene oxide), in square decimetres.

4725 Calculate the result for each test specimen to the nearest 0.1 mg/dm².

4726 7.3.4.6.2 Validity of results

4727 See section 7.1.7.3.

4728 7.3.4.6.3 Precision

4729 No data available.

4730 From a large number of tests, the described procedure provided a maximum repeatability value
4731 $r = 1$ mg/dm².

4732 **7.3.4.7 Test report**

4733 Prepare the test report in accordance with Annex 7.1.8.

4734

4735

4736

4737 **7.4 Test method for overall migration into vegetable oil in case of**
4738 **incomplete extraction of vegetable oil in the temperature range of 5-**
4739 **175°C**

4740

4741 **7.4.1 Scope**

4742 This method specifies tests for the determination of the overall migration into fatty food
4743 simulants from plastics materials and articles, by total immersion of test specimens in a fatty
4744 food simulant at any temperatures above 5°C up to and including 175°C for selected contact
4745 times.

4746 When some plastics are tested by the methods in Annex 1, the soxhlet extraction process does
4747 not achieve complete recovery of the absorbed olive oil from the test specimens. In this method,
4748 the olive oil is released from the plastic test specimens by dissolving them in chloroform,
4749 toluene, xylene or tetrahydrofuran.

4750 This method is suitable for plastics that come in contact with olive oil by total immersion as
4751 described in Annex 1, 7.2 and 7.3.

4752 This is provided the plastics are soluble in chloroform, toluene, xylene or tetrahydrofuran and
4753 insoluble in methanol.

4754 The method can also be suitable for plastics which are only partially soluble in chloroform,
4755 toluene, xylene or tetrahydrofuran and insoluble in methanol.

4756 NOTE 1 This test method has been written for use with olive oil. The test method can also be
4757 used with appropriate modifications with other vegetable oils, i.e. sunflower oil and corn oil.
4758 These other vegetable oils will produce different chromatograms for the methyl esters of the
4759 relevant oil compared to those of the methyl esters of olive oil. Select suitable chromatogram
4760 peaks of the methyl esters of the other vegetable oils for the quantitative determination of the oil
4761 extracted from the test specimens.

4762 NOTE 2 If it has been established that the overall migration into olive oil from the plastics cannot
4763 be determined by use of either this method or the methods described in Annex 1 then the use of
4764 tests in section 4.2.5 should be considered.

4765

4766 **7.4.2 Principle**

4767 The principle is described in Annex 7.1.2. Only the deviations are mentioned here.

4768 Test specimens of known mass are exposed to olive oil for the contact time, at contact
4769 temperatures varying from 5°C to 175°C, and then taken from the olive oil, blotted to remove oil
4770 adhering to the surface, and reweighed.

4771 The absorbed olive oil is extracted by a dissolution and precipitation procedure.

4772 In case the plastic does not dissolve completely, swelling of the plastic in the solvent should be
4773 such that the olive oil absorbed can be released from the plastic.

4774 Depending on the type of plastic an appropriate organic solvent is selected in order to dissolve
4775 or swell the plastic.

4776 For chloroform soluble plastics like polystyrene and polycarbonate, chloroform is used to
4777 release the olive oil absorbed.

4778 For polyolefins, toluene and xylene are used. Low density polyethylene shows good solubility in
4779 toluene and high density polyethylene and polypropylene dissolve or swell sufficiently in xylene.

4780 For polyvinylchloride or polyvinylidene chloride, tetrahydrofuran can be applied.

4781 **7.4.3 Reagents**

4782 The reagents shall be as described in Annex 7.1.3, except that the extraction solvent (Annex
4783 7.1.3.2) is not required and the following reagents are added to the list:

4784 a) chloroform

4785 b) methanol

4786 c) tetrahydrofuran

4787 d) toluene

4788 e) xylene

4789

4790 **7.4.4 Apparatus**

4791 The apparatus shall be as described in Annex 7.1.4, with the exception of the soxhlet extractors
4792 (Annex 7.1.4.15) which are not required and the addition of the following:

4793 a) centrifuge

4794 b) centrifuge tubes 150 ml

4795 c) conical funnels, 100 mm diameter

4796 d) filter papers, 185 mm diameter

4797

4798 **7.4.5 Preparation of test specimens**

4799 Prepare the test specimens in accordance with Annex 7.1.5.

4800

4801 **7.4.6 Procedure**

4802 **7.4.6.1 Determination of the suitability of vegetable oil for overall migration**

4803 Follow section 7.1.6.1.1

4804 7.4.6.1.1 Chromatogram of test specimen extract

4805 Take one of the test specimens, prepared in Annex 7.4.5, and place it in a 250 ml round bottom
4806 flask and add 10 ml of cyclohexane without the internal standard. Add to the flask by measuring
4807 cylinder 50-60 ml of chloroform (7.4.3.a), tetrahydrofuran (7.4.3.c), toluene (7.4.3.d) or xylene
4808 (7.4.3.e) and a few anti-bumping beads to control boiling.

4809 Couple the flask to a condenser. Place on either a water bath or steam bath and reflux for 30 min
4810 (0/+5 min). Slowly add by measuring cylinder or syringe, 10 ± 0.2 ml of the potassium hydroxide
4811 down the condenser and continue refluxing for 15-20 min. Add by measuring cylinder at least
4812 50 ± 2 ml of methanol and continue refluxing for 5-6 min.

4813 Remove the flask from the water bath and allow cooling. Transfer the solution from the flask to a
4814 150 ml centrifuge tube (7.4.4.b), washing out the flask with 5-10 ml of methanol into the tube.
4815 Centrifuge the solution for 20 min (0/+5 min), at 2000-2500 rpm. Filter the supernatant
4816 solution through a filter paper into a 250 ml flask.

4817 Evaporate the solution to 15-20 ml, either using a rotary evaporator or simple distillation
4818 apparatus. Transfer the solution to a 50 ml round bottom flask, washing out with 5-7 ml of
4819 methanol, and add a few anti-bumping beads. Evaporate the solution to dryness on a water bath.

4820 Subject the extracted material to the methyl ester preparation procedure, described in Annex
4821 7.1.6.1.1.1, but substituting the 10 ± 0.2 ml of the potassium hydroxide solution with 10 ± 0.2 ml of
4822 methanol (4.b).

4823 Inject the same volume of resulting solution as used in section 7.1.6.1.1 into the gas
4824 chromatograph. Retain the chromatogram,

4825 7.4.6.1.2 Comparison of chromatograms

4826 Draw the conclusions in accordance with section 7.1.6.1.3.

4827 **7.4.6.2**

4828 Follow sections 7.1.6.2 to 7.1.6.7

4829 **7.4.6.3 Extraction of absorbed olive oil**

4830 Take four flasks, 250 ml, to be used for the extraction, and place in each flask 10.0 ml of the
4831 internal standard cyclohexane solution of triheptadecanoin (7.1.3.3), using a pipette (7.1.4.21),
4832 or an alternative higher quantity if more than 100 mg of olive oil is present.

4833 NOTE 1 If the test specimens have retained more than 100 mg of olive oil, 10.0 ml of the
4834 internal standard solution is not sufficient for optimum precision in the gas chromatography
4835 determination after extraction. Before commencing the operations in this clause an
4836 estimation of the quantity of olive oil retained in the test specimens should be obtained by
4837 comparing the final masses of the test specimens with their initial masses. If considered
4838 necessary the quantity of internal standard solution can be increased from 10 ml although it
4839 is essential that the same quantity is used for each test specimen and that this quantity is also
4840 used with the olive oil standards for the calibration graph. As a guide, approximately 0.5 mg
4841 of the internal standard is required for every milligram of extracted olive oil.

4842 Place the four test specimens in the flasks and add to each flask, by measuring cylinder, 50-60 ml
4843 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
4844 anti-bump beads to control boiling. Carefully remove the test specimens from the supports using
4845 tweezers. If necessary, carefully cut the test specimens in pieces of approximately 2*2 cm. Wash
4846 the tweezers and supports with 50-60 ml of the relevant solvent and transfer the washings to
4847 the flask. Couple each flask to a condenser, place on either a water bath or a steam bath and
4848 reflux for 30 min. Add slowly down the condenser, by measuring cylinder or syringe, 10±0.2 ml
4849 of the potassium hydroxide solution (7.1.3.4) and continue refluxing for 15-20 min. Add by
4850 measuring cylinder at least 50±2 ml of methanol slowly down the condenser and continue
4851 refluxing for 5-6 min.

4852 NOTE 2 In such cases that the mass of the test sample is relatively high then the amount of
4853 the dissolving and precipitating solvent might need to be adapted to the mass of the test
4854 sample. In all cases a solution with reasonable viscosity should be obtained.

4855 Remove the flasks from the water bath and allow to cool. Transfer the solution from each flask to
4856 individual 150 ml centrifuge tubes (7.4.4.b), washing out each flask with 5-10 ml of methanol
4857 into the tube. Centrifuge each solution until a clear supernatant liquid is obtained. Filter the
4858 supernatant solutions through a filter paper into 250 ml round bottom flasks. Evaporate the
4859 solutions to 15-20 ml, either using a rotary evaporator or a simple distillation apparatus.
4860 Transfer the solutions to individual 50 ml round bottom flasks, washing out with 5-7 ml of
4861 methanol, add a few anti-bumping beads. Evaporate each solution to dryness on a water bath.

4862 NOTE 3 Oxidation of the olive oil should be avoided where possible. Therefore evaporation of
4863 the solvent to dryness should be carried out under mild conditions of temperature. In
4864 addition contact of the olive oil with oxygen should be limited.

4865 **7.4.6.4 Determination of extracted olive oil**

4866 7.4.6.4.1 Preparation of fatty acid methyl esters

4867 Add 10±0.2 ml of n-heptane to each of the 50 ml flasks containing the dry residue as obtained in
4868 the previous section 7.4.6.3 by measuring cylinder (7.1.4.20), ensuring that the residues of olive
4869 oil and plastic extractables dissolve or are well dispersed by shaking, warming or by ultrasonic
4870 treatment.

4871 NOTE 1 Unless the residues in the flasks are dissolved or well dispersed in the n-heptane,
4872 quantitative hydrolysis or methylation of the olive oil and of the internal standard might not
4873 be obtained under the conditions described particularly where these residues contain
4874 extractables from plastics in excess of 50 mg. The internal standard might not react with the
4875 plastic extractables to the same degree as does the olive oil and correct results for olive oil
4876 might not be obtained.

4877 Add by measuring cylinder or graduated syringe, 10 ± 0.2 ml of methanol (7.4.3.b) and a few anti-
4878 bumping beads (7.1.4.14). Connect a condenser to the flask and boil the mixture under reflux for
4879 10 ± 1.0 min.

4880 Add through the condenser by measuring cylinder, or graduated syringe, 5.0 ± 0.2 ml of the
4881 methanol solution of boron trifluoride (7.1.3.5) and boil the mixture under reflux for 2 ± 0.25
4882 min.

4883 Cool to room temperature and add, by measuring cylinder, 15-20 ml of saturated sodium
4884 sulphate solution (7.1.3.7.2) and shake well. Then add further sodium sulphate solution until the
4885 liquid level reaches the neck of the flask. Allow to stand until the phases have separated.

4886 NOTE 2 The methyl esters for the subsequent gas chromatographic determination are in the
4887 upper, n-heptane, layer.

4888 If there is a delay of more than 7 days in using a methyl ester solution for the gas
4889 chromatographic determinations, transfer the n-heptane layer to a small stoppered tube
4890 (7.1.4.24) containing solid anhydrous sodium sulphate (7.1.3.7.1) and store in a refrigerator.

4891 7.4.6.4.2 Determination of fatty acid methyl esters

4892 Determine the fatty acid methyl esters in accordance with Annex 7.1.6.9.2.

4893 7.4.7 Expression of results

4894 Perform section 7.1.7.1 to 7.1.7.3.

4895 7.4.7.1 Precision

4896 No data

4897 7.4.8 Test report

4898 Prepare the test report in accordance to Annex 7.1.8.

4899

4900 **Annex 8 Test method for overall migration into water,**
 4901 **aqueous food simulants, isooctane and ethanol 95%**

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4952

4953 **8.1 Scope**

4954 This section describes test methods for the determination of the overall migration into water,
 4955 aqueous based food simulants, i.e. A, B, C and D1, and the organic solvents isooctane and ethanol
 4956 95% from plastics which are intended to come into contact with foodstuffs. The method
 4957 describes four different ways to perform the migration, i.e. immersion, filling, pouch forming
 4958 and filling and cell for one-sided contact.

4959 The immersion method is most suitable for plastics in the form of films and sheets, but can be
 4960 applied to a wide range of articles or containers from which test pieces of suitable size can be
 4961 cut. The test contact temperature can go up to reflux conditions for water and aqueous food
 4962 simulants.

4963 The cell method is most suitable for plastics in the form of films and sheets, but is particularly
 4964 applicable to those materials consisting of more than one layer or for surfaces that differ in their
 4965 migration characteristics, which should be tested with the food simulant in contact only with
 4966 the surface which is intended to come into contact with foodstuffs. The test contact temperature
 4967 can go up to and including 60°C for organic solvents and 70°C for water and aqueous food
 4968 simulants.

4969 The pouch method is most suitable for plastics in the form of films and sheets which are
 4970 sealable by heat or pressure. The test is particularly applicable to those materials consisting of
 4971 more than one layer, which are tested with the food simulant in contact only with the surface
 4972 which is intended to be in contact with the foodstuffs. The test contact temperature can go up to
 4973 and including 60°C for organic solvents and 70°C for water and aqueous food simulants.

4974 This filling method is most suitable for plastics in the form of containers and articles that can be
 4975 filled. The test contact temperature can go up to and including 60°C for organic solvents and
 4976 70°C for water and aqueous food simulants.

4977 **8.2 Principle**

4978 The overall migration of non-volatile substances from a sample of the plastics is determined as
4979 the mass of non-volatile residue after evaporation of the solvent, i.e. aqueous food stimulant or
4980 organic solvent, following immersion.

4981 The selection of the test conditions will be determined by the worst foreseeable conditions of
4982 use as described in Regulation (EU) No 10/2011.

4983 Test specimens are exposed to the food simulant for the contact time at the test contact
4984 temperature. At the end of the test period, each test specimen is removed from the food
4985 simulant. The food simulant from each test specimen is evaporated to dryness, the mass of the
4986 non-volatile residue is determined gravimetrically and expressed as milligrams per square
4987 decimetre of surface area of test specimen.

4988 **8.3 Reagents**

4989 Each of the food simulants below shall give a non-volatile residue of less than 5 mg/l, when
4990 evaporated to dryness and dried to constant mass at 105-110°C.

4991 **8.3.1 Ethanol 10% (v/v) in aqueous solution (food simulant A)**

4992 A solution is prepared by diluting 100 ml of acetic acid with distilled water to a volume of 1 l.

4993 **8.3.2 Acetic acid 3% (w/v) in aqueous solution (food simulant B)**

4994 A solution is prepared by diluting 30 g of acetic acid with distilled water to a volume of 1 l.

4995 **8.3.3 Ethanol 20% (v/v) in aqueous solution (food simulant C)**

4996 A solution is prepared by diluting 200 ml of acetic acid with distilled water to a volume of 1 l.

4997 **8.3.4 Ethanol 50% (v/v) in aqueous solution (food simulant D1)**

4998 A solution is prepared by diluting 500 ml of acetic acid with distilled water to a volume of 1 l.

4999 **8.3.5 Ethanol**

5000 purity 96 % (v/v) or greater. A 95 % (v/v) aqueous solution is prepared.

5001 **8.3.6 Iso-octane, (2,2,4-trimethyl pentane)**

5002 purity 98.5 % (v/v) or greater, CAS No. 540-84-1.

5003 WARNING — isooctane and ethanol are flammable. Take care at all times when handling
5004 these organic solvents to prevent contact with sources of ignition.

5005 **8.4 Apparatus**

5006 **8.4.1 Cutting slab**

5007 Clean smooth glass, metal or plastics slab with a suitable area to prepare test specimens, 250
5008 mm x 250 mm is suitable.

5009 **8.4.2 Tweezers**

5010 Stainless steel, blunt nosed.

5011 **8.4.3 Cutting implement**

5012 Scalpel, scissors or sharp knife or other suitable device

- 5013 **8.4.4 Metal template**
5014 (100 ± 0.2 mm) x (100 ± 0.2 mm) (square) or (120 ± 1 mm) x (120 ± 1 mm) (square)
- 5015 **8.4.5 Rule or template**
5016 25 ± 1 mm wide
- 5017 **8.4.6 Rule**
5018 Graduated in mm, and with an accuracy of 0.1 mm.
- 5019 **8.4.7 Analytical balance**
5020 Capable determining a change in mass of 0.1 mg.
- 5021 **8.4.8 Specimen supports**
5022 Constructed of stainless steel with cross arms attached by welding or silver soldering, or of
5023 glass. Stainless steel X4 CrNi 18 10 according to EN 10088-1:2014 or stainless steel having a
5024 composition of 17% chromium, 9% nickel and 0.04% carbon is suitable. Before initial use
5025 thoroughly clean the stainless steel supports. The use of a degreasing solvent and then dilute
5026 nitric acid has been found to be suitable. For the aqueous acetic acid food simulant, use supports
5027 constructed out of glass, as there is a tendency for the acetic acid to corrode stainless steel
5028 supports, particularly if the joints are silver soldered.
- 5029 NOTE 1 However stainless steel supports can be used for acetic acid if it can be
5030 demonstrated that when immersed on their own in simulant, for the test period, at the test
5031 temperature, the residue after evaporating the simulant to dryness and drying in an oven or
5032 incubator or refrigerator to constant mass at 105-110°C is less than 5 mg/l.
- 5033 NOTE 2 The method has been written for the supports shown in Figure 3 on page 223 which
5034 have been found to be suitable for holding thin film and sheet test pieces. However other
5035 supports can be used provided that they are capable of holding and keeping the test pieces
5036 apart and ensuring complete contact with the food simulant at the same time. For rigid
5037 samples, supports with a single cross arm may be used.
- 5038 **8.4.9 Gauze**
5039 Pieces of fine stainless steel gauze with a mesh size of 1 mm have been found to be suitable,
5040 approximately 25 mm x 100 mm or, glass rods, 2-3 mm in diameter and approximately 100 mm
5041 long to be used with the acetic acid food simulant, for insertion between the test pieces. Before
5042 initial use thoroughly clean the gauze, first with a degreasing solvent and then with dilute nitric
5043 acid.
- 5044 **8.4.10 Glass tubes**
5045 Ground neck, for retaining the food simulant and test specimens. Tubes with an internal
5046 diameter of approximately 35 mm and length of between 100 mm and 200 mm, excluding the
5047 ground neck have been found to be satisfactory.
- 5048 **8.4.11 Glass beads**
5049 2-3 mm diameter or glass rods of 2-3 mm in diameter and approximately 100 mm long.
- 5050 **8.4.12 Thermostatically controlled oven or incubator or refrigerator**
5051 Capable of maintaining the set temperature within the tolerances specified in Table 5 on page
5052 222.

5053 WARNING — The interior/sample space of the oven, incubator or refrigerator should not
5054 have any exposed heating elements, to minimise safety hazards arising from any loss of
5055 flammable test media from the tubes during the test period.

5056 **8.4.13 Dishes**

5057 made of stainless steel, nickel, platinum, platinum alloy or gold, 50-90 mm diameter and a
5058 maximum mass of 100 g, for evaporation of food simulants and weighing of residues. Glass,
5059 glass ceramic or ceramic dishes may be used provided that the surface characteristics are such
5060 that the masses of the dishes after evaporation of any specified food simulants followed by
5061 conditioning in the desiccator used achieves a constancy of ± 0.5 mg. Stainless steel and nickel
5062 dishes are suitable only for aqueous ethanol solutions. Glass, glass ceramic, glazed ceramic,
5063 platinum or, platinum alloy or gold dishes are suitable for all food simulants.

5064 **8.4.14 Steam bath, hot plate, distillation apparatus or rotary
5065 evaporator**

5066 For evaporation of food simulant at the end of test period.

5067 **8.4.15 Desiccator**

5068 Filled with anhydrous calcium chloride or self indicating silica gel.

5069 **8.4.16 Measuring cylinder**

5070 100 ml, complying with the minimum requirements of ISO 4788:2005.

5071 **8.4.17 flasks, 250 ml**

5072 Suitable for attaching to reflux condensers

5073 **8.4.18 condensers to fit the flasks**

5074 **8.4.19 heating mantle**

5075 for maintaining the food simulants at reflux temperature during contact

5076 **8.4.20 glass filter of porosity G1**

5077

5078 **8.4.21 Cells**

5079 type A as shown in Figure 5 on page 225, either the all aluminium (anodized) cells or the cells
5080 with the stainless steel (316 grade) lids and rings, are suitable for water and aqueous ethanol
5081 food simulants. For the aqueous acetic acid food simulant the cells with the stainless steel lids
5082 and rings are used. The internal diameter of the rim of the sealing ring shall be 178.4 ± 0.1 mm,
5083 to give an area of the test specimen exposed to the food simulant of 2.5 dm².

5084 NOTE The cells, type A, are constructed with a rubber mat in the base plate. When using the
5085 cells with either the water or aqueous ethanol food simulants it is advised that a disc of
5086 aluminium foil is placed on the mat before inserting the test specimen. For the aqueous
5087 acetic acid food simulant a disc of polytetrafluoroethylene or other suitable material which is
5088 inert to acetic acid can be used. The use of these discs will prevent any substances from the
5089 mat influencing the migration result.

5090 For details of equivalent cells see Figure 6 to Figure 10.

- 5091 **8.4.22 Pipettes**
- 5092 Having volumes of 50, 100 and 200 ml, complying with the minimum requirements of ISO
5093 648:2008.
- 5094 **8.4.23 Measuring cylinders**
- 5095 250 ml, complying with the minimum requirements of ISO 4788:2005.
- 5096 **8.4.24 Metal template**
- 5097 $(120 \pm 1 \text{ mm}) \times (120 \pm 1 \text{ mm})$ (square).
- 5098 **8.4.25 Pouch holder**
- 5099 the example shown in Figure 11 has been shown to be suitable, constructed from aluminium or
5100 other suitable material or an equivalent holder, plus clips to secure corners of pouches.
- 5101 **8.4.26 Heat or pressure sealing device**
- 5102 for use in forming pouches.
- 5103 **8.4.27 Lint-free cloth or soft brush.**
- 5104 **8.4.28 Beaker**
- 5105 250 ml and 2 l.
- 5106
- 5107 **8.5 Preparation of test specimens**
- 5108 **8.5.1 General**
- 5109 It is essential that test specimens are clean and free from surface contamination, because many
5110 plastics can readily attract dust due to static charges. Before preparing test specimens, remove
5111 any surface contamination from the sample by gently wiping it with a lint free cloth, or by
5112 brushing with a soft brush (8.4.27). Under no circumstances wash the sample with water or
5113 solvent. If it is specified in the instructions for use of the article that it should be washed or
5114 cleaned before use see (7.1.5.1). Minimise handling of the samples and where necessary, wear
5115 cotton gloves.
- 5116 To ensure that test pieces are well separated and that the surfaces are freely exposed to the
5117 food simulant during the period of the test, for thin films, insert a piece of fine stainless steel
5118 gauze, or glass rods with the acetic acid simulant, between the test pieces or for thick samples
5119 not placed on the supports, insert glass rods between the test pieces after immersion in the food
5120 simulant. Where specimen supports are used, label the supports with a tag bearing the test
5121 specimen identification.
- 5122 When preparing test specimens measure the surface area according to 7.1.5.1.1.
- 5123 **8.5.2 Number of test specimens**
- 5124 Three test specimens are required for samples, in the form of thin films, sheets, cut sections
5125 from containers or similar articles. Five test specimens, similar dimensionally one to another,
5126 are required for fillable samples or samples of articles of irregular shape. These test specimens
5127 are utilized as follows:
- 5128 a) three test specimens for the migration test;

5129 b) two test specimens for determination of the surface area, in the case of samples of irregular
5130 shape (8.5.5).

5131 For articles with a nominal volume of more than 200 ml five articles are required to provide five
5132 test specimens. These test specimens are utilized as follows:

5133 a) three test specimens for the migration test;

5134 b) two test specimens for the determination of surface area.

5135 For articles with a nominal volume of less than 200 ml one test specimen shall be made up of
5136 sufficient articles to contain a minimum of 200 ml of the food simulant. Record the number of
5137 articles used to provide one test specimen.

5138 Five test specimens are required. These test specimens are utilized as follows:

5139 a) three test specimens for the migration test;

5140 b) two test specimens for the determination of surface area.

5141

5142 8.5.3 Films and sheets

5143 8.5.3.1 Immersion

5144 Lay the sample on the cutting slab (8.4.1) and cut the test specimens of 1 dm² (), using the 100
5145 mm x 100 mm template (8.4.4). Check, using the rule (8.4.6), that the dimensions of the test
5146 specimen are within the specified tolerance (± 1 mm).

5147 8.5.3.1.1 Immersion method using oven

5148 Cut each test specimen into four test pieces 25 mm x 100 mm using the rule (8.4.5). Assemble
5149 one test specimen onto the support (8.4.8) by piercing suitable holes in the test pieces and
5150 placing two test pieces on each side of the cross arms of the support. Repeat this procedure for
5151 all remaining test specimens.

5152 8.5.3.1.2 Immersion method using reflux

5153 Cut each test specimen into sixteen test pieces 25 mm x 25 mm using the rule. Repeat this
5154 procedure for all remaining test specimens.

5155 8.5.3.2 Cell

5156 Lay the sample on the cutting slab (8.4.1) with the surface to be in contact with the food
5157 simulant uppermost. Take the ring from the standard cell (8.4.21) and place on the surface of
5158 the sample. Cut out the test specimen by cutting round the outer edge of the ring, using the
5159 cutting implement (8.4.3).

5160 8.5.3.3 Pouch

5161 Lay the sample on the cutting slab (8.4.1) with the surface to be in contact with the food
5162 simulant uppermost and cut the test specimens using the 120 mm x 120 mm template (8.4.24).

5163 Place pairs of the test pieces together with the surfaces to be in contact with the food simulant
5164 uppermost. Using the heat or pressure sealer (8.4.26), join all four edges 10 mm from the edge
5165 to form pouches with four seals. Measure the distances between the inner edges of the seals to
5166 the nearest 1 mm and calculate the total surface area of the test specimen which will be exposed
5167 to the food simulant, to the nearest 0.01 dm². This shall be approximately 2 dm². Using the
5168 cutting implement (8.4.3), remove excess film from the sealed area (to reduce area of film not
5169 directly exposed to food simulant whilst leaving enough to withstand the test conditions
5170 without leaking.

5171 Measure and record the surface area of the pouch which will be in contact with the food
5172 simulant and the total external area of the pouch after trimming excess material.

5173 Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently
5174 large to insert a 100 ml pipette.

5175 NOTE Pouches of dimensions other than 100 mm x 100 mm can be used for testing. These
5176 pouches should be prepared where possible so that the total surface area exposed is not less
5177 than 1 dm².

5178 8.5.4 Containers and other articles

5179 8.5.4.1 Immersion

5180 Cut sections from the walls of the container or article to give test specimens each of area
5181 approximately 1 dm². For articles with individual areas less than 1 dm², use a number of articles
5182 to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1
5183 mm, using the rule. Calculate only the surface area of the sample which is intended to come into
5184 contact with foodstuffs. Calculate the area of each test specimen to the nearest 0.01 dm² and
5185 record.

5186 8.5.4.1.1 Immersion method using oven

5187 If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes
5188 (8.4.10). The test specimens or pieces are placed on the specimen supports if these are
5189 appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested
5190 unsupported.

5191 8.5.4.1.2 Immersion method using reflux

5192 Cut each test specimen into test pieces 25 mm x 25 mm.

5193 8.5.4.2 Filling

5194 Determine and record the volume of food simulant required to fill an article to its nominal
5195 foodstuff volume. If the nominal volume of foodstuff to fill the article is not known, determine
5196 the surface area that will be in contact with the food simulant when filled to 5 mm from the top
5197 of the test specimen.

5198 Next determine and record the surface area of the test specimen which is intended to come into
5199 contact with its nominal volume of foodstuff.

5200 NOTE 2 In the case of articles with a volume of less than 200 ml this will be the surface area
5201 of one article multiplied by the number of articles used to provide one test specimen.

5202 8.5.5 Articles of irregular shape

5203 Select representative portions of the article, or multiples of the article for small articles, to give
5204 five dimensionally similar test specimens each with a known total surface area of at least 1 dm².

5205 Measure only the surface area intended to come into contact with foodstuffs of two of these test
5206 specimens to the nearest 0.05 dm² using e.g. the Schlegel Method, as described in Annex B of EN
5207 ISO 8442-2:1997, the methods described in Mieth and Hoekstra (2014) or any other suitable
5208 method. Record the surface area of each test specimen.

5209 8.6 Procedure

5210 8.6.1 Contact with food simulant

5211 NOTE When testing some samples by single surface testing the small amounts of food
5212 simulant may permeate through the sample. For example, there may be a small loss of

5213 ethanol when testing with high strength ethanol/water food simulants by filling. In this case,
5214 as the loss in ethanol from the food simulant may be expected to reflect what would happen
5215 under actual conditions of use of the alcoholic beverage, this loss may be disregarded.
5216 However, if permeation occurs when testing in a cell, care has to be taken to ensure
5217 contamination does not arise from contact of the food simulant with components of the cell.

5218 **8.6.1.1 Immersion method using oven**

5219 Take three glass tubes (8.4.10) for the test specimens and a further two to provide blanks,
5220 measure by measuring cylinder 100 ± 2 ml of the food simulant into each tube and stopper the
5221 tube. Insert a thermometer or thermocouple, if applicable see NOTE 2, in one of the test
5222 specimen tubes, and stopper all five tubes. Place the five tubes in the thermostatically controlled
5223 oven, incubator or refrigerator, set at the test temperature, and leave until the food simulant has
5224 attained the test temperature.

5225 NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so,
5226 then use a separate tube for measuring the temperature and an additional test specimen

5227 Place a test specimen into three tubes, re-insert the thermometer or thermocouple, and stopper
5228 the tubes. Mark the tubes for identification. Ensure that the test specimens are totally immersed
5229 in the food simulant; if they are not then add either glass beads or rods to raise the level of the
5230 food simulant until total immersion is achieved. This part of the operation should be carried out
5231 in the minimum time to prevent undue heat loss from the food simulant.

5232 Mark the liquid level on the outside of each tube with a suitable marker.

5233 Replace all of the tubes in the thermostatically controlled oven, incubator or refrigerator, set at
5234 the test temperature.

5235 Observe the temperature and leave the tubes for the selected period of contact time after the
5236 temperature of the food simulant has reached a temperature within the permitted tolerance for
5237 the test temperature, see Table 4 and Table 5 for permitted tolerances on test times and
5238 temperature.

5239 NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
5240 temperatures. All of these contact times and contact temperatures are not necessarily
5241 relevant.

5242 WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take
5243 care to ensure that the tubes are well stoppered to prevent solvent volatilizing into the
5244 interior of the oven, incubator or refrigerator and generating an explosive mixture.

5245 WARNING 2 — If possible place the tubes in a drip container capable of holding the total
5246 volume of organic solvent in case of accident.

5247 WARNING 3 — To minimise hazards arising due to the volatile and flammable nature of the
5248 organic solvents the maximum test temperature is 60°C . Do not conduct the tests at
5249 temperatures above 60°C .

5250 Take the tubes from the oven or incubator or refrigerator and check the level of food simulant
5251 If this level has fallen by more than 10 mm below the mark, or has exposed any part of the test
5252 pieces, repeat the test using fresh test specimens.

5253 If the level of the food simulant in a tube is less than 10 mm below the mark, remove the test
5254 specimen from the tube, and allow the simulant adhering to the test specimen and support to
5255 drain back into the tube. Recover at least 90% of the original volume of simulant or repeat the
5256 test.

5257 NOTE 2 For contact times of 24 h or more it is acceptable to monitor the temperature of the
5258 air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
5259 temperature of the food simulant.

5260 NOTE 3 For short test contact times (≤ 6 h) the use of hot stirring plate heating controlled by
5261 a thermocouple may be a good alternative for an oven.

5262 **8.6.1.2 Immersion method using reflux**

5263 Take three flasks (8.4.17) for the test specimens and a further two to provide blanks, measure
5264 by measuring cylinder 100 ± 2 ml of food simulant into each flask.

5265 Place the flasks in the heating mantle, connect the condensers.

5266 Turn on the water supply to the condensers.

5267 Switch on the heating mantle and heat the food simulant in each of the flasks to boiling. Turn off
5268 the heating, allow the flasks to cool for 2-3 min, remove the condensers from three flasks
5269 containing 100 ml of simulant and place a test specimen in each flask. Ensure that the test
5270 specimens are totally immersed in the food simulant.

5271 WARNING: Hot fumes can emit from the flasks when the lids are removed.

5272 Replace the condensers and switch on the heating mantles, and heat so that reflux is achieved
5273 within 5 min.

5274 Observe the food simulant in the flask, following the onset of reflux, leave for the test time,
5275 taking into account the tolerances in Table 4. Turn off the heating mantle, turn off the water to
5276 the condenser and remove the flask from the mantle.

5277 NOTE Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
5278 temperatures. All of these contact times and contact temperatures are not necessarily
5279 relevant.

5280 To separate the food simulant from the test specimens, pour off the hot food simulant through a
5281 glass filter (8.4.20), collecting the filtrate in a clean container. Rinse each of the flasks and the
5282 test specimens in the flasks with two portions of 10 ± 1 ml of unused simulant and pour these
5283 washings through the filter.

5284 WARNING: Danger hot flasks and contents.

5285 **8.6.1.3 Cell**

5286 Take three cells (8.4.21), mark these for identification purposes. Place in the thermostatically
5287 controlled oven or incubator or refrigerator (8.4.12), which is set at the selected test
5288 temperature and leave until the test temperature has been attained.

5289 Take three glass tubes (8.4.10), for the food simulant for contact with the test specimens and a
5290 further two to provide blanks, measure by measuring cylinder 125 ± 2 ml of the food simulant
5291 into each tube. Insert a thermometer or thermocouple, if applicable, see NOTE 2, in one of the
5292 tubes and stopper the tubes. Mark the liquid level on the outside of each tube with a suitable
5293 marker.

5294 NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so,
5295 then use a separate tube for measuring the temperature and use an additional test specimen
5296 and cell.

5297 Place the tubes in the thermostatically controlled oven or incubator or refrigerator, set at the
5298 test temperature and leave until the food simulant has attained the test temperature.

5299 Remove the cells from the thermostatically controlled oven or incubator or refrigerator,
5300 dismantle and place on the base of each cell one of the test specimens. Reassemble the cells,
5301 ensuring that the clamping screw wheel is well tightened down.

5302 Remove three tubes containing 125 ml of the food simulant from the thermostatically controlled
5303 oven or incubator or refrigerator and transfer the food simulant from each tube to the cells
5304 through the filler hole. Remove the thermometer or thermocouple from the tube and, insert, if

5305 applicable see NOTE 2, in one of the cells and replace the filler plugs. This part of the operation
5306 should be carried out in the minimum time to prevent undue heat loss from the cells and food
5307 simulants. If any leakage is observed reject that cell from further tests.

5308 WARNING 1 — Never place a leaking cell in the oven.

5309 Replace the test cells in the thermostatically controlled oven or incubator or refrigerator, set at
5310 the test temperature.

5311 Observe the temperature, leave the cells and the blank tubes for the selected period of time
5312 after the temperature of the food simulant in the cell has reached a temperature within the
5313 permitted tolerance for the test temperature, see Table 4 and Table 5 for permitted tolerances
5314 on test times and temperature.

5315 NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
5316 temperatures. All of these contact times and contact temperatures are not necessarily
5317 relevant.

5318 WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take
5319 care to ensure that the tubes are well stoppered and pouches are carefully closed at the
5320 corner to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator
5321 and generating an explosive mixture.

5322 WARNING 2 — For safety reasons do not load an oven with more than the test specimens of
5323 one test sample.

5324 WARNING 3 — Place the pouches and tubes in a drip container capable of holding the total
5325 volume of organic solvent in case of an accident.

5326 WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the
5327 organic solvents, the maximum test temperature is 60°C. Do not conduct the tests at
5328 temperatures above 60°C.

5329 Take the cells and the two tubes containing the blank food simulant from the thermostatically
5330 controlled oven or incubator or refrigerator.

5331 Transfer the solvent from each of the cells into the tubes, check the level of solvent in each, if
5332 this has fallen by more than 10 mm below the mark, repeat the test with fresh test pieces.
5333 Recover at least 90% of the original volume of food simulant or repeat the test.

5334 Rinse each cell twice with 20 ± 2 ml of unused food simulant, add these rinses to the respective
5335 tubes. Add twice 20 ± 2 ml of food simulant to each of the two tubes containing blank simulant.

5336 NOTE 2 For contact times of 24 h or more it is acceptable to monitor the temperature of the
5337 air bath of the thermostatically controlled oven or incubator or refrigerator, instead of the
5338 temperature of the simulant.

5339 **8.6.1.4 Pouch**

5340 Take three glass tubes (8.4.10) for the food simulant for filling the pouches and a further two to
5341 provide the blanks, measure by measuring cylinder 100 ± 2 ml of the food simulant into each
5342 tube. Insert a thermometer or thermocouple, if applicable, see NOTE 3, in one of the tubes and
5343 stopper the tubes.

5344 NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so,
5345 then use a separate tube for measuring the temperature and use an additional pouch.

5346 Place the tubes and the pouch holder in the thermostatically controlled oven or incubator or
5347 refrigerator, set at the test temperature and leave until the test temperature has been attained.

5348 Remove the pouch holder from the thermostatically controlled oven or incubator or refrigerator
5349 and place between the spacers the test specimen pouches.

5350 Remove the tubes containing the 100 ml of food simulant from the thermostatically controlled
5351 oven or incubator or refrigerator. Pipette sufficient food simulant into three test specimen to
5352 just fill the pouch. This shall be about 100 ml, but for thick/semi-rigid materials the quantity
5353 will be less. Remove the thermometer or thermocouple from the tube and insert, if applicable,
5354 see NOTE 3, in one of the filled pouches. Secure the open corner of each pouch with a clip.

5355 NOTE After filling, the corner of the pouch can be closed by heat sealing for aqueous food
5356 simulants.

5357 If not all food simulant is used to fill the pouch, retain the tube and residual contents. Measure
5358 and record the volume of the residual food simulant. This part of the operation should be
5359 carried out in the minimum time to prevent undue heat loss. If any leakage is observed reject
5360 that pouch from further tests.

5361 WARNING 1 — Never place a leaking pouch in the oven.

5362 Replace the pouch holder, containing the test specimen pouches, in the thermostatically
5363 controlled oven or incubator or refrigerator, set at the test temperature. Observe the
5364 temperature and leave the pouches and blank tubes for the selected contact time after the
5365 temperature of the simulant has reached a temperature within the permitted tolerance for the
5366 test temperature, see Table 4 and Table 5 for permitted tolerances on test times and
5367 temperature.

5368 NOTE 1 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
5369 temperatures. All of these contact times and contact temperatures are not necessarily
5370 relevant.

5371 Take the pouch holder and the tubes containing the blank food simulant from the
5372 thermostatically controlled oven or incubator or refrigerator.

5373 Examine the pouches for leaks, if at least 90% of original volume of food simulant is not
5374 recovered from each pouch, the test is invalid and shall be repeated using fresh pouches.

5375 NOTE 2 For plastics that loose food simulant during the test period due to permeation
5376 through the plastic see .

5377 NOTE 3 For contact times 24 h or more it is acceptable to monitor the temperature of the air
5378 bath of the thermostatically controlled oven or incubator or refrigerator or refrigerator,
5379 instead of the temperature of the simulant.

5380 WARNING 1 — Both iso-octane and ethanol are volatile flammable organic solvents. Take
5381 care to ensure that the tubes are well stoppered and pouches are carefully closed at the
5382 corner to prevent solvent volatilizing into the interior of the oven, incubator or refrigerator
5383 and generating an explosive mixture.

5384 WARNING 2 — For safety reasons do not load an oven with more than the test specimens of
5385 one test sample.

5386 WARNING 3 — Place the pouches and tubes in a drip container capable of holding the total
5387 volume of organic solvent in case of an accident.

5388 WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the
5389 two solvents, the maximum test temperature is 60°C. Do not conduct the tests at
5390 temperatures above 60°C.

5391 **8.6.1.5 Filling**

5392 Mark three test specimen for identification, and where more than one article has been used for a
5393 test specimen, also mark individually.

5394 Place, in a beaker, a sufficient volume of the food simulant to fill the three test specimens to the
5395 nominal volume or to 5 mm from the top if the nominal volume is not known. and to provide

5396 two 200 ml blanks. Insert a thermometer or thermocouple, if applicable see NOTE 3, in the food
5397 simulant. Place the beaker in the thermostatically controlled oven or incubator or refrigerator
5398 set at the test temperature and leave until the food simulant has attained the test temperature.

5399 NOTE Check whether the thermometer or thermocouple contributes to the OM result. If so,
5400 then use separate beakers filled with food stimulant for each test specimen and one
5401 additional for measuring the temperature. Use an additional test specimen for measuring the
5402 temperature during the contact time.

5403 Remove the beaker containing the food simulant from the thermostatically controlled oven or
5404 incubator or refrigerator. Fill the three test specimens with food simulant to the nominal
5405 volume of the article or to 0.5 cm from the top. Insert the thermometer or thermocouple in one
5406 of the test specimens containing food simulant, if applicable see NOTE 3. Cover the test
5407 specimens and the remaining food simulant with an inert material to prevent evaporation. This
5408 part of the operation should be carried out in the minimum time to prevent undue heat loss
5409 from the simulant.

5410 WARNING 1 — Covering of articles filled with organic solvents is very important in respect
5411 to safety. Due to the variety of fillable articles it is impossible to prescribe one method for
5412 covering the filling opening. In general aluminium foil has appeared to be suitable in many
5413 cases. Also, a combination of glass plates with aluminium foil can be useful. Containers, like
5414 bottles and cups, are easily closed by carefully wrapping aluminium foil over the filling
5415 opening. Articles with large open areas, such as trays or dishes, should be covered with a
5416 glass plate of an appropriate size. The article and cover should then be placed on a sheet of
5417 aluminium foil, which is then folded around and over the article and glass plate. In this way a
5418 pouch is made that prevents evaporation of the organic solvent or reduces it to an acceptable
5419 level. To insert a thermocouple the glass plate should be provided with a hole that fits the
5420 thermocouple or that is large enough to accept a polytetrafluoroethylene stopper with the
5421 thermocouple.

5422 WARNING 2— For safety reasons do not load an oven with more than the test specimens of
5423 one test sample. In case were the capacity of the article is large, then the test specimens
5424 should be placed in the oven one at a time.

5425 WARNING 3 — If possible place the article in a drip container capable of holding the total
5426 volume of organic solvent in case of an accident.

5427 WARNING 4 — To minimise hazards arising due to the volatile and flammable nature of the
5428 organic solvents, the maximum test temperature is 60°C. Do not conduct the tests at
5429 temperatures above 60°C.

5430 Place the test specimens in the thermostatically controlled oven or incubator or refrigerator set
5431 at the test temperature. Observe the temperature and leave the test specimens and food
5432 simulant for the selected contact time after the temperature of the simulant has reached a
5433 temperature within the permitted tolerance for temperature, see Table 4 and Table 5 for
5434 permitted tolerances on test times and temperature.

5435 NOTE 1 Where the surface of simulant is large, a check should be made to ensure that
5436 excessive loss of simulant by evaporation does not occur.

5437 NOTE 2 Table 4 and Table 5 includes tolerances on a wide range of contact times and contact
5438 temperatures. All of these contact times and contact temperatures are not necessarily
5439 relevant.

5440 Take the marked test specimens and beaker with blank food simulant from the thermostatically
5441 controlled oven or incubator or refrigerator.

5442 NOTE 3 For contact times of 24 h or more it is acceptable to monitor the temperature of the
5443 air bath of the thermostatically controlled oven or incubator or refrigerator or refrigerator,
5444 instead of the temperature of the simulant.

5445 **8.6.2 Determination of migrating substances**

5446 WARNING — Both iso-octane and ethanol are volatile and flammable organic solvents. Take
5447 care when evaporating them to prevent vapours contacting sources of ignition, particularly
5448 when using a hot plate to carry out the evaporation. The evaporation should be carried out in
5449 a fume cupboard.

5450 **8.6.2.1 Preparation of dishes**

5451 Take five dishes (8.4.13), marked for identification, place the dishes in an oven maintained at
5452 105-110 °C, for a period of 30 ± 5 min, to dry.

5453 NOTE three dishes for the exposed food simulant and 2 dishes for the blank

5454 Remove the dishes from the oven, place in a desiccator (8.4.15) and allow cooling to ambient
5455 temperature. Weigh and record the individual masses of each dish.

5456 Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until
5457 individual consecutive masses differ by not more than 0.5 mg, record their final masses.

5458 **8.6.2.2 Evaporation method**

5459 Take the tubes, pouches or filled test specimens containing the food simulant and pour 40-50 ml
5460 from each into separate dishes. By means of a steam bath, hot plate or other form of heating
5461 (8.4.14) evaporate to a low volume, taking care to avoid loss, in particular, by sputtering or
5462 overheating of the residues.

5463 NOTE 1 The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

5464 When most of the food simulant has evaporated, pour the remaining food simulant from each of
5465 the tubes into the respective dishes and continue the evaporation. Wash out each of the tubes,
5466 including the blank tubes with two lots of 10 ± 1 ml of unused simulant and pour these
5467 washings into the respective dishes. Continue the evaporation.

5468 NOTE 2 A stream of nitrogen can be used to facilitate evaporation.

5469 When the simulant has almost completely evaporated, place the dish in an oven maintained at
5470 105-110 °C, for a period of 30 ± 5 min, to complete the evaporation and dry the residue.

5471 Remove the dishes from the oven, place in a desiccator (8.4.15) and allow cooling to ambient
5472 temperature. Weigh and record the individual masses of a dish and residue.

5473 Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until
5474 individual consecutive masses differ by not more than 0.5 mg.

5475 Determine the mass of the residue by subtracting the original stable mass of the dish (8.6.2.1)
5476 from the stable mass of the dish and residue.

5477 **8.6.2.3 Distillation method**

5478 Transfer the food simulants to individual round bottom flasks (250 ml are suitable). Wash out
5479 each of the tubes, including the blank tubes, with two lots of 10 ± 1 ml of unused food simulant,
5480 add these rinses to the respective flasks. Place the flasks in an electric heating mantle and
5481 connect to a side arm distillation arrangement or rotary evaporator. Distil off the food simulant
5482 until approximately 30-50 ml remains in each flask. Transfer the remaining food simulant from
5483 each flask to individual evaporating dishes (8.4.13). Rinse each flask with two lots of 10 ± 1 ml
5484 of fresh simulant and add the rinses to the appropriate dishes. Continue the evaporation of the
5485 food simulant by means of a steam bath, hot plate or other form of heating, proceeding as in
5486 8.6.2.2.

5487 NOTE The evaporation of acetic acid and ethanol should be carried out in a fume cupboard.

5488 **8.6.3 Expression of results**

5489 **8.6.3.1 Method of calculation**

5490 Express the overall migration as milligrams of residue per square decimetre of the surface of
5491 the sample which is intended to come into contact with foodstuffs, calculated for each test
5492 specimen using the following formula:

$$M = \frac{(m_a - m_b) \cdot 1000}{S}$$

5493
5494 where

5495 M is the overall migration into the simulant, in milligrams per square decimetre of surface area
5496 of sample intended to come into contact with foodstuffs;

5497 m_a is the mass of the residue from the test specimen after evaporation of the food simulant in
5498 which it had been immersed, in grams;

5499 m_b is the mass of residue from the blank food simulant, in grams;

5500 S is the surface area of the test specimen intended to come into contact with foodstuff, in square
5501 decimetres, see 7.1.5.1.1. In the case of articles with a nominal volume less than 200 ml, S is the
5502 surface area of one article multiplied by the amount of articles that constitutes the test
5503 specimen.

5504 Calculate the result for each test specimen to the nearest 0.1 mg/dm² and the mean of the
5505 individual test results, to the nearest 0.1 mg/dm².

5506 **8.6.3.2 Validity of results**

5507 The following analytical tolerances are allowed:

5508 12 mg/kg or 2 mg/dm² for all aqueous food simulants.

5509 The test result for each individual test specimen is valid if it differs from the mean of the
5510 triplicate test results by not more than the permitted analytical tolerance. If a minimum of three
5511 results is not within the analytical tolerance, then the test is repeated using fresh test specimens
5512 from the sample.

5513 **8.6.3.3 Precision**

5514 The precision data were determined for a polyamide sample under the test conditions of 24 h at
5515 40 °C with water, 3% acetic acid and .

5516 The precision data for water were determined from the BSI/DTI trial conducted in 1991 (Pira
5517 Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

5518 The precision data for 3% acetic acid were determined from the BSI/DTI trial conducted in
5519 1991 (Pira Report No.SP91/2- January 1992) involving 10 laboratories and one sample.

5520 The precision data for ethanol 10% were determined from the BSI/DTI trial conducted in 1991
5521 (Pira Report No.SP91/2- January 1992) involving 13 laboratories and one sample.

5522 Table 7 Precision data (mg/dm²)

	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

5523

5524 The difference between two single results found on identical test material by one operator using
5525 the same apparatus within the shortest feasible time interval can exceed the repeatability value
5526 r on average not more than once in 20 cases in the normal and correct operation of the method.

5527 Single results on identical test material reported by two laboratories can differ by more than the
5528 reproducibility value R on average not more than once in 20 cases in the normal and correct
5529 operation of the method.

5530 **8.6.4 Test report**

5531 The test report shall include the following:

- 5532 • reference to this method and to the part used for the test procedure;
- 5533 • all information necessary for complete identification of the sample such as chemical type,
5534 supplier, trade mark, grade, batch number, thicknesses;
- 5535 • conditions of time and temperature of contact with food simulants;
- 5536 • departures from the specified procedure, and reasons for these:
- 5537 • individual test results, expressed as milligrams of residue per square decimetre of sample
- 5538 • f) relevant comments on the test results such as test contact area and contact volume.

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