

MultivaporTM Application Booklet



048858 en

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Pure Solvents

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Solvent mixtures

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| | | | | |
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Tips for evaporation under vacuum

There are distinct ways to evaporate multiple samples in parallel with the Multivapor™ using the Büchi vacuum controller V-855. The main functions are briefly described in the following.

Manual distillation

Ideal evaporation conditions such as the temperature of the cooling water, the heating medium and the pressure must be tuned to one another. As a rule of thumb, to optimize these parameters, a temperature difference of 25 °C between the set temperature (heating medium) and the vapor temperature, and 20 °C between the vapor temperature and the condenser is optimal. The corresponding vacuum setting is available from the Solvent Table provided.

Example: Evaporation of ethanol at 55 °C

Set the temperature of the instrument to 55°C. Set the vacuum pressure to 97 mbar. Start rotation and evacuation. According to the 25/20 °C rule the temperature of the vapor is then approx. 30 °C. To sustain an efficient dissipation of the heat of evaporation in the condenser, the temperature of the cooling should not exceed 10 °C.

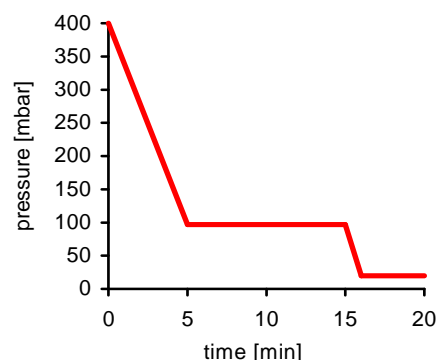


Pressure gradients

Setting the pressure manually and immediately to the corresponding boiling point, accommodates the risk of bumping. To minimize this risk, it is strongly recommended to program a pressure gradient yielding a slow convergence to the optimized conditions.

Example: Evaporation of ethanol at 55 °C

Set the initial pressure to 400 mbar. The first ramp goes down to 97 mbar (see Solvent Table) over a 5 minute period. Depending on the solvent volume, the pressure is kept constant for approx. 10 - 15 min. An optional second ramp can be implemented as an additional drying step. However, this procedure requires an efficient cooling of the distillate (ice-bath or refrigerated receiver) to prevent back-evaporation.



Automatic distillation – the EasyVac mode

The EasyVac algorithm measures pressure differences over time and adjusts the pressure automatically depending on the efficiency of the condenser. This “press-and-go” procedure requires no supervision nor any knowledge about temperature-pressure relations. Moreover, the user is alerted as soon as the procedure is finished.

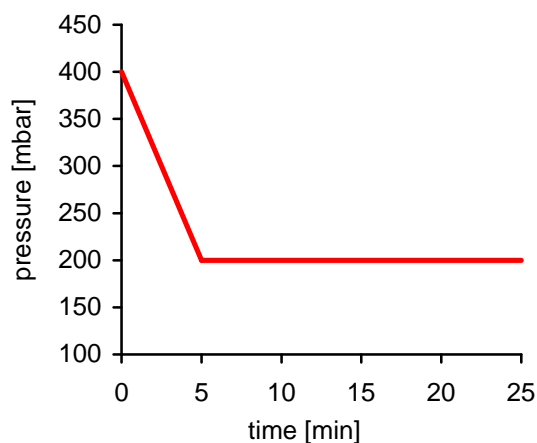
Description of the parameters

System Configuration

Multivapor™ configuration: P R A
 number of positions format: 6 12
 optional accessories vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 frits SPE module
 Vacuum solution pump: V-700 V-710 condenser
 operation modes V-855 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
 Cooling medium tab water: yes use of tab water instead of chiller
 chiller: B-740/8 B-740/14 B-741
 Comments

Parameters

Solvent type: xx
 volume: xx ml, xx ml/position
 Heating medium temperature: xx °C
 volume: xx ml H₂O/position
 Cooling medium type: xx
 temperature: xx °C
 flow: xx ml/min
 Rotational speed position x
 Vacuum profile manual: xx mbar automatic
 vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 400 | 200 | 5 |
| 2 | 200 | 200 | 20 |

Solvent: Acetonitrile

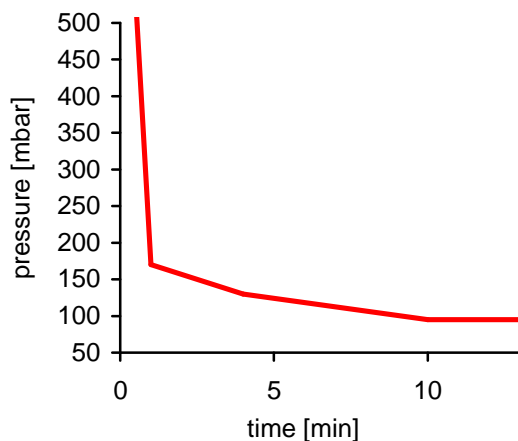
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: Acetonitrile
 volume: 360 ml, 30 ml/position
- Heating medium temperature: 60 °C
 volume: 30 ml H₂O/position
- Cooling medium type: water
 temperature: 15 °C
 flow: 600 ml/min
- Rotational speed position 9
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 900 | 170 | 1 |
| 2 | 170 | 130 | 3 |
| 3 | 130 | 95 | 7 |
| 4 | 95 | 95 | 3 |

Results

- | | | |
|---|--|----------------|
| - | Time for evaporation process | 13 min |
| - | Solvent recovery after primary condenser | 350 ml, 97% |
| - | Solvent recovery after secondary condenser | not determined |
| - | Total solvent yield | 350 ml, 97% |

Remarks

- The pressure is automatically adjusted using the EasyVac mode. However, the gradient depicted in the table reflects the EasyVac adjustments.
- The process stops automatically using EasyVac.

Solvent: Chlorobenzene

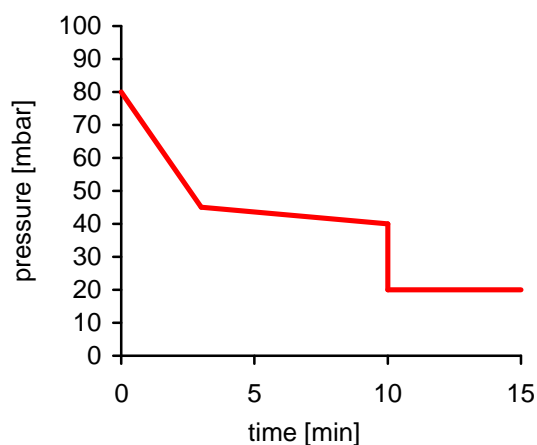
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments instrument equipped with Woulff bottle and FKM (Viton®) O-rings

Parameters

- Solvent type: Chlorobenzene
 volume: 120 ml, 10 ml/position
- Heating medium temperature: 70 °C
 volume: 35 ml H₂O/position
- Cooling medium type: Water/glycol
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 80 | 45 | 3 |
| 2 | 45 | 40 | 7 |
| 3 | 20 | 20 | 5 |

Results

- | | | |
|---|--|----------------|
| - | Time for evaporation process | 15 min |
| - | Solvent recovery before primary condenser | 115, 96 % |
| - | Solvent recovery after secondary condenser | not determined |
| - | Total solvent yield | 115 ml, 96 % |

Remarks

- To avoid swelling of the EPDM O-rings of the vacuum cover, it is strongly recommended to use the FKM (048827) or FFKM alternative (48849).
- There is considerable condensation of the vapor in the ribbed vacuum tubing observed. Use of the Woulff bottle (48857) traps all solvent condensed on the way to the condenser and prevents back rinsing into the vacuum cover when the experiment is finished.
- Evaporation of chlorobenzene was finished after 10 min. However, an additional drying step at 20 mbar was programmed to dry the vacuum cover. With this measure the vacuum cover got completely dry (not droplets, no wet parts whatsoever). Depending on the environmental temperature it is recommended to cool the condensate in the receiving flask with an ice bath.

Solvent: Dimethylformamide, DMF

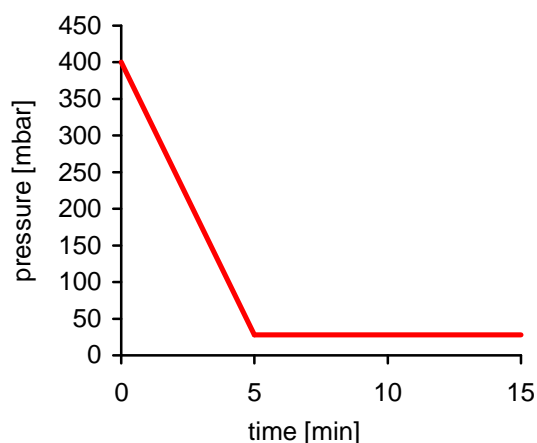
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: DMF
 volume: 300 ml, 25 ml/position
- Heating medium temperature: 80 °C
 volume: 50 ml H₂O/position
- Cooling medium type: Ethanol/Water
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 400 | 28 | 5 |
| 2 | 28 | 28 | 10 |

Results

- Time for evaporation process 15 min
- Solvent recovery after primary condenser 280 ml, 93%
- Solvent recovery after secondary condenser not determined
- Total solvent yield 280 ml, 93%

Solvent: Ethylacetate

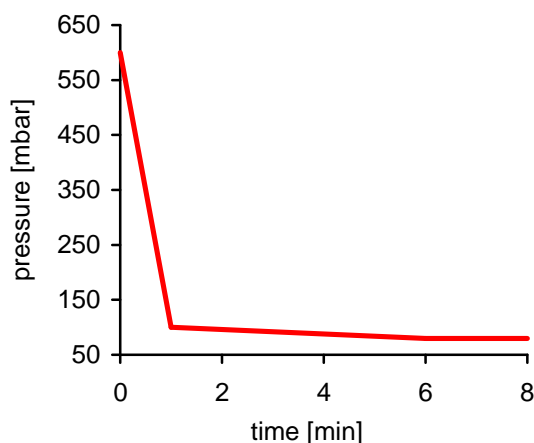
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: ethylacetate
 volume: 240 ml, 20 ml/position
- Heating medium temperature: 55 °C
 volume: 30 ml H₂O/position (min position)
- Cooling medium type: water/glycol
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 600 | 100 | 1 |
| 2 | 100 | 80 | 5 |
| 3 | 80 | 80 | 2 |

Results

- Time for evaporation process 8 min
- Solvent recovery after primary condenser not determined
- Solvent recovery after secondary condenser not determined
- Total solvent yield not determined

Remarks

- Evaporation time using EasyVac: 9 min
- Evaporation time using EasyVac and a cooled double jacket receiving flask: 7 min

Solvent: Hexane

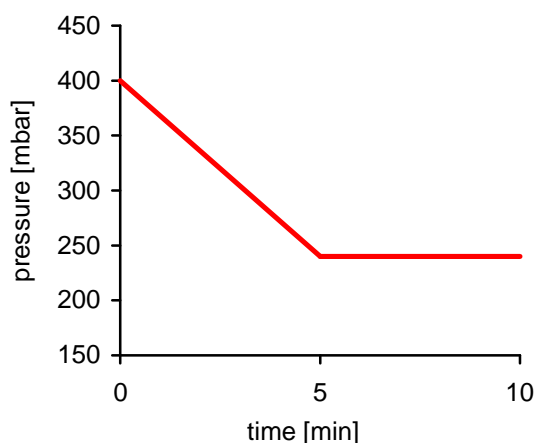
System Configuration

- Multivapor™
 - configuration: P R A
 - format: 6 12
 - vessel type: Büchi PSE/ASE Radley's
 - flange 10 ml flange 20 ml
 - options: frits SPE module
- Vacuum solution
 - pump: V-700 V-710 condenser
 - controller: V-850 V-855
 - mode: manual gradient library
 - AutoDest EasyVac
- Cooling medium
 - tap water: yes
 - chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent
 - type: Hexane
 - volume: 300 ml, 25 ml/position
- Heating medium
 - temperature: 50 °C
 - volume: 50 ml H₂O/position
- Cooling medium
 - type: Ethanol/Water
 - temperature: 5 °C
 - flow: 800 ml/min
- Rotational speed
 - position 9
- Vacuum profile
 - manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 400 | 240 | 5 |
| 2 | 240 | 240 | 5 |

Results

- Time for evaporation process 8 min
- Solvent recovery after primary condenser 280 ml, 93%
- Solvent recovery after secondary condenser not determined
- Total solvent yield 280 ml, 93%

Solvent: Toluene

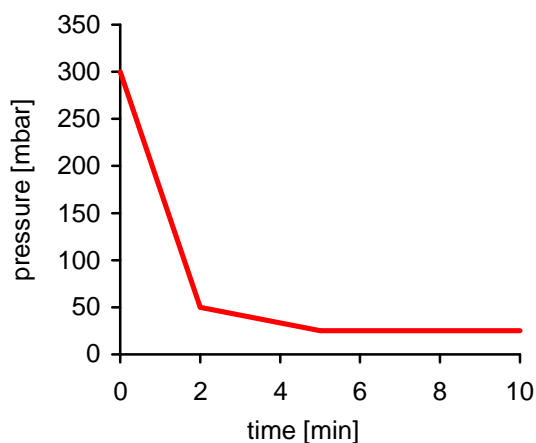
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: toluene
 volume: 240 ml, 20 ml/position
- Heating medium temperature: 55 °C
 volume: 30 ml H₂O/position (min position)
- Cooling medium type: water/glycol
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 300 | 50 | 2 |
| 2 | 50 | 25 | 3 |
| 3 | 25 | 25 | 5 |

Results

- Time for evaporation process 10 min
- Solvent recovery after primary condenser not determined
- Solvent recovery after secondary condenser not determined
- Total solvent yield not determined

Remarks

- Evaporation time using EasyVac: 11 min
- Evaporation time using EasyVac and a cooled double jacket receiving flask: 9 min

Solvent: Water

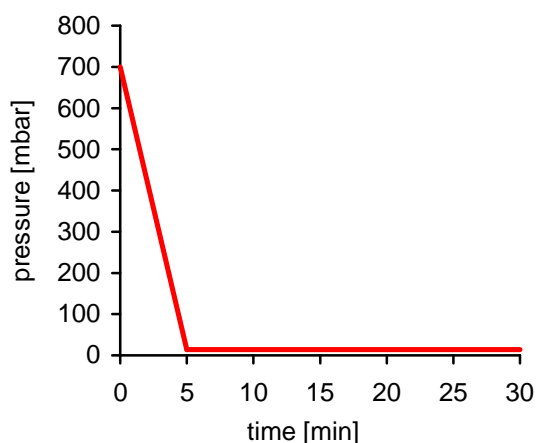
System Configuration

- Multivapor™
 - configuration: P R A
 - format: 6 12
 - vessel type: Büchi PSE/ASE Radley's
 - flange 10 ml flange 20 ml
 - options: frits SPE module
- Vacuum solution
 - pump: V-700 V-710 condenser
 - controller: V-850 V-855
 - mode: manual gradient library
 - AutoDest EasyVac
- Cooling medium
 - tap water: yes
 - chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent
 - type: Water
 - volume: 300 ml, 25 ml/position
- Heating medium
 - temperature: 60 °C
 - volume: 50 ml H₂O/position
- Cooling medium
 - type: Ethanol/Water
 - temperature: 5 °C
 - flow: 800 ml/min
- Rotational speed
 - position 10
- Vacuum profile
 - manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 700 | 14 | 5 |
| 2 | 14 | 14 | 25 |

Results

- | | | |
|---|--|----------------|
| - | Time for evaporation process | 27 min |
| - | Solvent recovery after primary condenser | 265 ml, 86% |
| - | Solvent recovery after secondary condenser | not determined |
| - | Total solvent yield | 265 ml, 86% |

Remarks

Water is due to its tremendous heat capacity difficult to evaporate with the Multivapor. Heat is continuously transferred from the heating medium to sample and eventually to the condenser assembly. This leads to a considerable drop in the temperature of the heating medium of approx. 15 °C.

Solvent: MeCN / Water 70:30

System Configuration

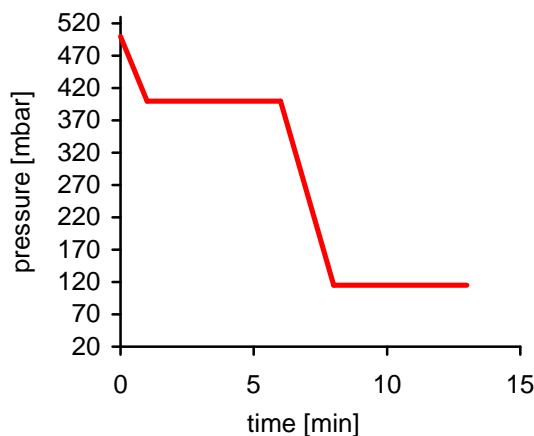
- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 Multistat40
- Comments



Parameters

- Solvent type: acetonitrile / water 70:30
 volume: 120 ml, 10 ml/position
- Heating medium temperature: 70 °C
 volume: 30 ml H₂O/position
- Cooling medium type: Ethanol / Water
 temperature: 10 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 500 | 400 | 1 |
| 2 | 400 | 400 | 5 |
| 3 | 400 | 115 | 2 |
| 3 | 115 | 115 | 5 |

Results

- Time for evaporation process 13 min
- Solvent recovery after primary condenser 113 ml, 94%
- Solvent recovery after secondary condenser not determined
- Total solvent yield 113 ml, 94%

Solvent: MeCN / Water 90:10

System Configuration

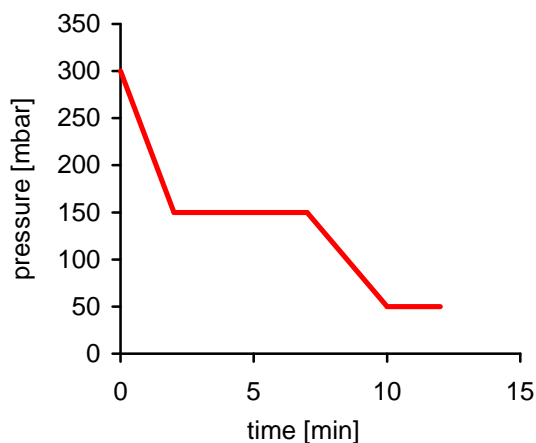
- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 Multistat40
- Comments



Parameters

- Solvent type: acetonitrile / water 90:10
 volume: 120 ml, 10 ml/position
- Heating medium temperature: 50 °C
 volume: 30 ml H₂O/position
- Cooling medium type: ethyleneglycol/water
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 9
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 300 | 150 | 2 |
| 2 | 150 | 150 | 5 |
| 3 | 150 | 50 | 3 |
| 4 | 50 | 50 | 2 |

Results

- Time for evaporation process 12 min
- Solvent recovery after primary condenser 112 ml, 93%
- Solvent recovery after secondary condenser not determined
- Total solvent yield 112 ml, 93%

Remarks

- Pre-heating time is 15 min.
- To prevent the back evaporation of the solvent, the receiving flask have to be cooled down with the ice/water mixture or with the refrigerated receiver (048640).

Solvent: Hexane / Ethylacetate 4 : 1

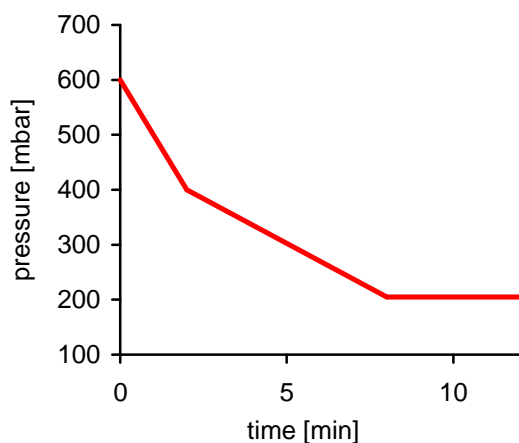
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: hexane / ethylacetate 4 : 1
 volume: 600 ml, 100 ml/position
- Heating medium temperature: 55 °C
 volume: 100 ml H₂O/position
- Cooling medium type: water / glycol
 temperature: 10 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 600 | 400 | 2 |
| 2 | 400 | 205 | 6 |
| 3 | 205 | 205 | 4 |

Results

- Time for evaporation process 12 min
- Solvent recovery after primary condenser 5 ml, xx%
- Solvent recovery after secondary condenser not determined
- Total solvent yield 98%

Remarks

Application was also performed with EasyVac. Total time used 11 min, solvent recovery ~ 95%, no bumping, smooth evaporation, no end-point determination

Solvent: Hexane / Ethylacetate 4 : 1

System Configuration

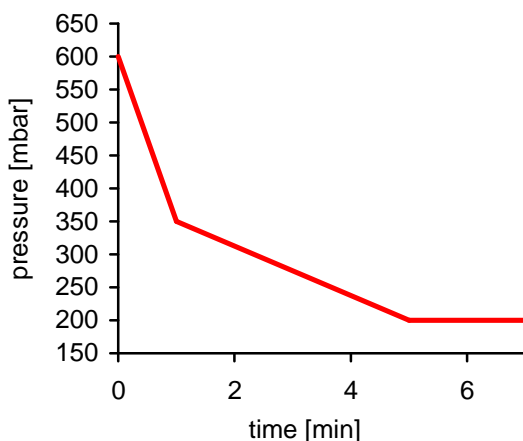
- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments



Parameters

- Solvent type: hexane / ethylacetate 4 : 1
 volume: 240 ml, 20 ml/position
- Heating medium temperature: 55 °C
 volume: 30 ml H₂O/position (min position)
- Cooling medium type: water/glycol
 temperature: 5 °C
 flow: 800 ml/min
- Rotational speed position 10
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 600 | 350 | 1 |
| 2 | 350 | 200 | 4 |
| 3 | 200 | 200 | 3 |

Results

- Time for evaporation process 7 min
- Solvent recovery after primary condenser not determined
- Solvent recovery after secondary condenser not determined
- Total solvent yield not determined

Remarks

- Evaporation time using EasyVac: 6 min
- Evaporation time using EasyVac and a cooled double jacket receiving flask: 6 min

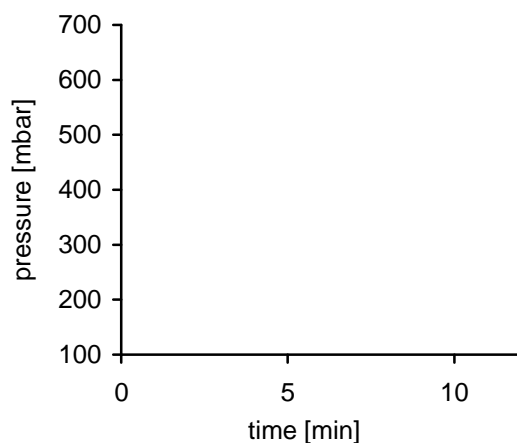
Solvent: Hexane / Ethylacetate / Petrolether 1 : 1 : 1

System Configuration

- Multivapor™
 - configuration: P R A
 - format: 6 12
 - vessel type: Büchi PSE/ASE Radley's
 - flange 10 ml flange 20 ml
 - options: frits SPE module
- Vacuum solution
 - pump: V-700 V-710 condenser
 - controller: V-850 V-855
 - mode: manual gradient library
 - AutoDest EasyVac
- Cooling medium
 - tap water: yes
 - chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent
 - type: hexane / ethylacetate / petrolether 1 : 1 : 1
 - volume: 600 ml, 100 ml/position
- Heating medium
 - temperature: 55 °C
 - volume: 100 ml H₂O/position
- Cooling medium
 - type: water / glycol
 - temperature: 10 °C
 - flow: 800 ml/min
- Rotational speed
 - position 10
- Vacuum profile
 - manual: xx mbar automatic
 - vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|-----------------|---------------|---------------|
| | | | |

Results

- Time for evaporation process 16 min
- Solvent recovery after primary condenser not determined
- Solvent recovery after secondary condenser not determined
- Total solvent yield not determined

Remarks

Application was only performed with EasyVac. Smooth evaporation without bumping but no automatic end-point determination.

Solvent: Chloroform / Methanol 2:1

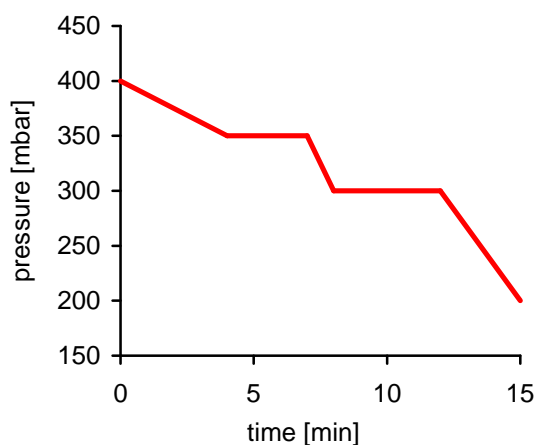
System Configuration

- Multivapor™ configuration: P R A
 format: 6 12
 vessel type: Büchi PSE/ASE Radley's
 flange 10 ml flange 20 ml
 options: frits SPE module
- Vacuum solution pump: V-700 V-710 condenser
 controller: V-850 V-855
 mode: manual gradient library
 AutoDest EasyVac
- Cooling medium tab water: yes
 chiller: B-740/8 B-740/14 B-741
- Comments

Parameters

- Solvent type: CHCl₃ / MeOH 2:1
 volume: 300 ml, 25 ml/position
- Heating medium temperature: 50 °C
 volume: 60 ml H₂O/position
- Cooling medium type: tab water
 temperature: 20 °C
 flow: 500 ml/min
- Rotational speed position 9
- Vacuum profile manual: xx mbar automatic

vacuum gradient:



| step | start [mbar] | end [mbar] | time [min] |
|------|--------------|------------|------------|
| 1 | 400 | 350 | 4 |
| 2 | 350 | 350 | 7 |
| 3 | 350 | 300 | 8 |
| 4 | 300 | 300 | 12 |
| 5 | 300 | 200 | 15 |

Results

- Time for evaporation process 15 min
- Solvent recovery after primary condenser 300 ml, 100%
- Solvent recovery after secondary condenser 0 ml, 0%
- Total solvent yield 300 ml, 100%

Remarks

The gradient depicted on the opposite page reflects the automatic vacuum adjustment by the EasyVac mode.

No automatic endpoint detection possible due to the high environmental temperature and low boiling point of chloroform.