Multivapor™
Application Guide

BÜCHI
SWITZERLAND

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This document describes all aspects which are important for the evaporation of various solvents using the Multivapor. This handbook is a guideline for the evaporation of your specific solvent and mixtures at high performance. Some standard applications are described in detailed Application Notes. best@buchi are found on the internet under www.buchi.com, then Applications and best@buchi. Please contact your local BUCHI representative for additional information on a particular application.

1   Introduction

1.1   Multivapor and Configurations

The Multivapor is a compact 6 or 12 position parallel vortex evaporator. Since the efficiency of evaporation depends on a variety of parameters, careful optimization for each type of sample/evaporation task is important. This handbook provides practical guidelines to successfully develop new applications with the goal that you could enjoy your Multivapor.

1.1.1   Standard configuration

If not indicated otherwise, the following Multivapor configuration was used in this booklet (**Figure 1**).

Component	Configuration
Multivapor	P-12 (MP11001S12) 12 positions PETP Cover and Adapters
Condenser	Type S condenser for tap water or a recirculating chiller
Vacuum controller	V-855, Software V 3.03
Vacuum pump	V-700, Woulff bottle, Secondary condenser
Chiller	F-105, Set to 5°C Cooling power at 15°C: 500 W Flow rate (no accessories): 1.8 l/min
Vessel	BUCHI SpeedExtractor tube 60 ml 30 ml max working volume (49535)
Options	None (no Woulff bottle, no Protective shield, no frits)

Figure 1: BUCHI Multivapor P-12 with type S condenser, vacuum pump V-700 and vacuum controller V-855

1.1.2 Variations and Versions

Component	Variants
Tube adapter	PETP (white, blind adapter: black)
	PEEK (ecru)
Seals for tube adapters	PTFE
Vacuum cover	PETP (black)
	PEEK (ecru)
O-ring for the vacuum cover adapter	EPDM (black)
	FKM (green)
	FFKM (black)
Vacuum cover seal	EPDM
Vacuum tube	Ribbed PFA with PTFE seals

The PETP vacuum cover is equipped with EPDM O-rings. A set of 12 FKM O-rings is enclosed. Optionally, there are highly resistant FFKM O-rings available. The PEEK vacuum cover is equipped with FFKM O-rings by default.

For order codes please consider the operation manual or contact your local dealer.

1.1.3 Recommended accessories

The most convenient way to achieve the right setting is to use the BUCHI vacuum controller V-855 (**Figure 2**). With this instrument, the pressure is either determined automatically using the EasyVac function (P-12 only), or it can be easily determined by means of an integrated temperature/pressure converter (the so-called Solvent Library) which is available for a variety of solvents. Moreover, pressure gradients can be programmed to ensure smooth and reproducible evaporation with the highest possible yields.[1]

Figure 2: BUCHI vacuum controller V-855 (professional)

The use of a chiller (**Figure 3**) is highly recommended. A chiller enables a constant low temperature without tap water input. A low condenser temperature is needed to distill at low temperatures without loss of solvents into the environment. BUCHI chillers can be controlled by the V-855 vacuum controller.

1.1.4 Limits of the Multivapor

The limit is given by the lowest sensible working pressure of approximately 25-30 mbar (V-700) and the unheated cover leading to condensation in the cover of certain solvents. For high-boiling solvents, the Syncore Polyvap with heated cover is recommended.

Figure 3: BUCHI recirculation chiller

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[1] Optimizing the evaporation using the Syncore® Analyst, recovery of 94±3% was achieved for naphthalene in PAH determination of soil samples. See: J. Cavegn, B. Haag, R. Hartmann, Trace analysis of PAHs: evaluation of two extraction methods (EPA3541 and EPA3545) and optimization of subsequent concentration, best@buchi no. 51/2008.

The Multivapor is limited when the task is to concentrate to a defined volume instead to dryness. For this application, the Syncore Analyst with cooled appendix was developed.

1.2 The process of distillation

Vacuum evaporation is the process of causing the pressure in a liquid-filled container to be reduced, causing the liquid to evaporate at a lower temperature than normal. A distillation consists of vaporization and subsequent condensation. When these two processes are well parted, separation of substances is possible based on differences in vapor pressure. The transport of the vapor is driven by a thermal pump, a dynamic difference in pressure between places of evaporation and condensation.

The evaporation speed is positively correlated with the amount of heat supplied by the heating plate. However, there is no correlation between the set temperature on the instrument (T_i) and the temperature of the sample (T_s). Liquids cannot assume a temperature higher than their boiling point (T_{bp}), which is solely determined by the applied vacuum. The only critical point for the sample is at the end of an evaporation to dryness process (**Figure 4**).

In the condenser, the heat of evaporation is then withdrawn from the vapor; the vapor condenses.[2] For best performance and highest recovery of the solvent, it is important to maintain a balanced evaporation-condensation process. Whenever more substance is vaporized than is condensed in the cooler, the pressure within the apparatus rises. To maintain the set pressure, the vacuum pump draws continually pulling the vaporized solvent into the environment. This must be avoided.[3] Pollution is prevented by assembling a secondary condenser behind the pump, or preferred, adjusting the evaporation to the cooling capacity of the condenser. Ideal conditions are achieved when the condensate covers approximately the half of the condenser (**Figure 5**).

Figure 4: Change of the sample temperature during the evaporation process with a sudden rise in temperature at the end of the process.

1.3 General guidelines for evaporation of solvents

To achieve the best evaporation conditions, five parameters, the temperature of the heating plate, the boiling point, the cooling water temperature, and the rotational speed of the orbital movement, must be attuned to one another. A tight system enables best distillation performance. Therefore, the apparatus should be checked for leaks without the solvent in it. A well-sealed system keeps the vacuum losses within bounds (< 10 mbar / 5 min at vacuum of 20 mbar).

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[2] This phenomenon is referred to as a thermal pump. See: The laboratory assistant, BÜCHI Labortechnik AG, second edition, p. 24.

[3] It is estimated that worldwide approx. 160'000 liters of solvent per day are discharged into the air due to unbalanced evaporation and condensation speeds. See: The laboratory assistant, BÜCHI Labortechnik AG, second edition, p. 47.

1.3.1 Correct load of the condenser

Evaporated solvent is condensed in the condenser and evaporation heat is dissipated. Best performance and highest recovery of the solvent are generally achieved when the condensate covers approximately the half of the height of the condenser (**Figure 5**).

1.3.2 Manual distillation and the Δt 25/20°C rule

A simple rule of thumb can be used as a starting point to find good settings for the heating medium, the vapor temperature, and the cooling temperature: the Δt 25/20°C rule. This rule specifies the temperature difference between the three zones. For instance, when the temperature on the instrument is set to 55°C, the vacuum should be set that the boiling point is 30°C, which is 25°C lower. In order to achieve sufficient condensing, cooling temperature of another 20°C lower also 10°C is recommended (**Figure 6**).

The following steps are performed in practice:

1. Set the heating medium to the desired temperature according to the stability of the product and the solvent

2. Determine the pressure using the Solvent Library so that a boiling point of 25°C lower results

3. Set the cooling temperature another 20°C lower

If the distillation performs too slowly, either increase vacuum or heating temperature. Aerate respectively decrease vacuum when sample is foaming or bumping. Distillation of problematic solutions can be distilled without supervision by using the gradient function (see Section 1.3.3). Further troubleshooting is found in detail in Section 1.4.9.

Figure 5: The vacuum and cooling conditions are ideal when the condensate covers approximately half of the height of the condenser.

1.3.3 Distillation with gradients

Distillation of problematic solutions can be handled without supervision by using the gradient function. Setting the pressure manually and immediately to the corresponding boiling point accommodates the risk of bumping. To avoid boiling retardation or loss of analyte, program a pressure gradient. Gradients also allow convenient integration of "drying" steps by applying a very low pressure during some minutes after the effective evaporation. To prevent reevaporation of solvent from the receiving flask during "drying", cool the distillate with ice bath or refrigerated receiver.

Optimization of the conditions for your specific problem is usually started with manual distillation. Define the desired boiling point according to the sensitivity of the product and the Δt 25/20°C rule. Start 500 mbar above the calculated pressure. Decrease the pressure quickly by 300-400 mbar, and then approximate slowly the desired
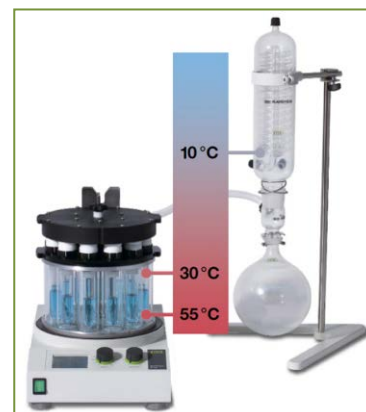

Figure 6: Illustration of the Δt 25/20°C rule.

boiling point. Keep the pressure constant until the evaporation is finished. For highly volatile compounds slow aeration over 1-2 min is recommended.

Example: Pressure settings for evaporation of 12 x 30 ml *n*-hexane using P-12.

Solvent		*n*-hexane
Desired boiling point, T_{bp}		30°C
Pressure derived from the solvent list		241 mbar
Instrument temperature, T_i		55°C
Pressure gradient		
Starting point	241 + 500 = 741 mbar	
Ramp	741-291 mbar	2 min
	291-237 mbar	2 min
	237-230 mbar	2 min
	230-202 mbar	3 min
Constant	202 mbar	1 min
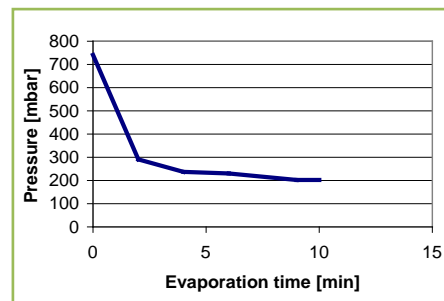
A first rapid decrease in pressure prevents heating up the sample. The following gradient decreases bumping, retardation and foaming of the product. After a satisfying manual distillation, the repeat function (V-855 only) displays the last distillation for easy documentation and repetition.

1.3.4 EasyVac function

The EasyVac mode (Vacuum controller V-855, software version 3.03 or higher) serves to carry out an automatic and gentle distillation based on the vapor pressure of the solvent. The process measures pressure differences over time and adjusts the vacuum automatically depending on the efficiency of the condenser. This mode is suited for distilling single solvents with the Multivapor under certain prerequirements.

Prerequirement	
Rack temperature	≥ 50°C
Solvents with boiling point	< 100°C
Total evaporated volume	≥ 80 ml
Only weak foaming products	
Glassware diameter	< 12 mm

The operator may interact at any time during automatic distillation and the process continues according to the manual set parameters.

EasyVac Multivapor P-12	Detection start point	Distillation	Detection end point
Glassware diameter 13 < x < 19 mm	☑	☑	
Glassware diameter > 19 mm	☑	☑	☑

EasyVac Multivapor P-6	Detection start point	Distillation	Detection end point
Low-boiling solvents	☑	(☑)	Often too early

Please note: A tight system is crucial for correct determination. The EasyVac mode only works with a speed controlled vacuum pump (V-700 or V-710). The distillation time can be increased by 30 to 100% compared to manual distillation.

1.4   Parameters and impact on distillation

1.4.1   Heat medium

Supplying more energy to the solvent by elevated heat medium temperature will accelerate the distillation process. This additional energy will dissipate in the condenser leading to possible losses of solvent to the environment due to an overburdened condenser. Leaking solvent might condense in the pump leading to decreased pump performance. Acceleration of the distillation increases risk of foam and bumping sample. Another danger is the loss of analytes due to increased temperature at the end of an evaporation process.

Water in the crystal rack ensures good heat transfer to the sample. Best evaporation performance is achieved when the water level reaches at least the solvent level. However, do not overfill or spill of water by the vortex is expected.

Increase distillation speed
Increase temperature of heat medium
Risks
Loss of analytes Heat degradation of sample Foam and bumping of sample

1.4.2   Vacuum

Similarly, decreasing of boiling point of the solvent by reduction of pressure will accelerate the distillation process. This additional energy will dissipate in the condenser leading to possible loss of solvent in the environment due to an overburden of the condenser capacity. If the system is insufficiently tight for the applied vacuum, the pump continually draws solvent, leading to known problems. Analytes might be lost due to boiling. Acceleration of the distillation increases risk of foam and bumping sample.

Increase distillation speed
Increase vacuum
Risks
Loss of analytes Foam and bumping of sample

1.4.3   Temperature of condenser

For efficient condensation of the vapor, the temperature of the condenser should be at least 15-20°C less of the vapor temperature. If the temperature of the cooling medium is fixed e.g. by the temperature of tap water, adjust the pressure and vapor temperature accordingly higher. Otherwise, exhaust of vapor into the environment might occur. Ideal conditions are achieved when the condensate covers approximately the half of the condenser.

1.4.4 Rotation speed

The surface area of the sample influences the evaporation. The larger the surface, the faster is the evaporation. The surface area is determined by the rotational speed of the heating plate. A faster rotation increases the surface area and hence accelerates the process. However, increasing the rotational speed also increases the vortex of the heating medium water. This might result in a reduced heat transfer surface.

Generally speaking, start the optimization with a pronounced vortex. For the Multivapor, this is typically the case at position 8. Then increase rotational speed until an optimal position for evaporation speed and handling of the sample (foam, bumping) is found.

Increase distillation speed
Rotation speed 8-10
Risks
Spill of heat medium Large vortex decreases heat transfer Foam and bumping of sample

1.4.5 Vessel type

The vessel shape (**Figure 7**) influences greatly the distillation performance. Especially the length and diameter are important characteristics. Distillation speed of ethanol and acetone were decreased by approx. 50% in a 50 ml Falcon tube (29 x 114 mm, conical end) and reaction tube (16 x 130 mm) compared to the SpeedExtractor tube (28 x 140 mm). Applied pressures could be transferred but the gradient time will be extended accordingly.

The vessel for quickest distillation is as long and as wide in diameter as possible without conical parts.

Figure 7: Selection of compatible P-12 tubes. *From left*: BUCHI SpeedExtractor tube, 50 ml Falcon and test tube.

1.4.6 Solvent volume

The volume of evaporated ethanol showed no effect on the speed using SpeedExtractor tubes. However, highly volatile solvents and large volumes might lead to starvation of the heat medium, thus elongating the evaporation. A further increase of the vacuum is advisable.

P-12 compatible tubes are preferably filled to only 50% of the total volume with sample. Standard P-6 tubes and SpeedExtractor vessels have working volumes of 150 and 180 ml, respectively.

Applied pressures could be transferred but the gradient time will be extended accordingly.

1.4.7 Solvent mixtures

Mixtures are evaporated by removing one solvent after the other in sequence according to the increasing boiling points. The purpose of this, in general, is not to achieve a sharp separation of the individual components, but rather to concentrate on the dry residue. A number of problems such as boiling delays or undesirable solvent emissions can occur in the area of transitioning from one component to the next.[4] In addition to this, a time or concentration dependent foam build-up can occur.

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[4] A. Talamona, G. Schönenberger, Vacuum control during vaporization of problematic solutions, best@buchi no. 06/2001.

These dangers are avoided if the system pressure is reduced slowly using the gradient function. Start with optimal conditions for lower-boiling solvent according to the gradient rule.

Some mixtures of a certain composition, however, cannot be separated by distillation. They are called azeotrope. When an azeotrope is boiled, the resulting vapor has the same ratio of constituents as the original mixture.

Do not go under lowest condensation temp with boiling point of lowest-boiling solvent, or use a well-cooled receiving vessel. Otherwise, re-evaporation from receiving flask might occur reducing evaporation performance.

1.4.8 Multivapor P-6 and P-12

Using SpeedExtractor vessels for P-6 and P-12, distillation speed increased by 20% with P-6 compared to P-12 (tested with *n*-hexane). Applied pressures could be transferred but the gradient time will be extended accordingly.

1.4.9 Troubleshooting

Problem	Action	Reference
Foam	1. Reduce vacuum	Section 1.4.2
	2. Program ramp	Section 1.3.3
	3. Install frit in the adapter	See Multivapor manual
Overheating, Bumping up	1. Reduce vacuum	Section 1.4.2
	2. Program ramp	Section 1.3.3
	3. Reduce heat	Section 1.4.1
	4. Use vessel with larger diameter	Section 1.4.5
	5. Reduce time span from immersing the vessels into the heat medium to start vacuum and rotation	
Condensation in cover	1. Reduce boiling temperature (increase vacuum) and adjust heat medium as well as condenser temperature accordingly	Sections 1.3.2, 1.4.1, 1.4.2, 1.4.3
	2. Reduce length of vacuum tubing to condenser	
Slow distillation speed	1. Improve heat transfer	Sections 1.4.1, 1.4.4, 1.4.8, 1.4.5
	2. Optimize rotational speed	Section 1.4.4
	3. Increase vacuum	Section 1.4.2
	4. Increase temperature	Section 1.4.1

1.5   Sample applications

The Multivapor was designed to fit excellently in your analytical work flow without transferring of vessels. Samples are often concentrated after extraction (BUCHI Extraction units, SpeedExtractor) or reaction processes as well as after separation by flash chromatography (BUCHI Sepacore).

Extraction: Fat Determination in Shortbread

The determination of fat in food and feed is a routine procedure for quality assurance and labeling. Fat was determined in butter shortbread after homogenization of the sample and extraction with the SpeedExtractor E-916. The solvent was evaporated in parallel using the Multivapor P-6. The total fat content was determined gravimetrically after the extract has been dried to a constant weight.[5]

Figure 8: Shortbread

Chromatography: Separation of plant extracts

Flash chromatography systems can be used not only for rapid and easy purification of synthetic products, but also for the separation of complex natural product mixtures such as plant extracts.

Sepacore cartridges enabled a good separation of compounds with a broad range of polarity, as typically found in plant extracts. The chromatographic resolution remained, however, lower than that achieved by medium pressure liquid chromatography (MPLC) on columns packed with material of smaller particle size. For poorly soluble extracts, solid introduction gave better results than liquid injection.[6]

To recover these extracts in pure form, the mobile phase of the selected fractions will be evaporated. This is preferably performed using Multivapor, as the collection vessel can be used directly for evaporation without sample transfer.

Figure 9: Separation of plant extracts

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[5] Fat in Shortbread using SpeedExtractor E-916 and Multivapor P-6. BUCHI Application Note no. 005/2009.

[6] Weber P, Schafroth N, Hamburger M, Potterat O. 2010. Flash chromatography on cartridges for the separation of plant extracts – Rules for the selection of chromatographic conditions, and comparison with MPLC. Poster in collaboration with University of Basel. http://www.buchi.com/Applications.19910.0.html.

2 Evaporation of pure solvents

2.1 List of solvents and classification

Solvent	Boiling point	Enthalpy of vaporization [J/ml]	[J/g]	Density [g/ml]	Classification	Constant b
Acetic acid	118°C	729	695	1.049	Polar protic	0.183
Acetone	56°C	437	553	0.79	Low boiling	0.196
Acetonitrile (MeCN)	82°C	570	725	0.786	Low boiling	0.195
Benzene	80°C	481	548	0.877	Low boiling	0.202
n-Butanol	118°C	502	620	0.81	Polar protic	0.155
Chlorbenzene	132°C	417	377	1.106	Medium boiling	0.202
Chloroform	61°C	392	264	1.483	Low boiling	0.203
Cyclohexane	81°C	303	389	0.779	Low boiling	0.206
Cyclopentane	40°C	313	417	0.751	Low boiling	0.207
Dichloromethane (DCM)	40°C	439	330	1.33	Low boiling	0.194
Diethyl ether	35°C	278	389	0.714	Low boiling	0.200
*Dimethylformamide (DMF)**	*153°C*	*549*	*578*	*0.949*	*High boiling*	*0.180*
1,4-Dioxane	101°C	400	388	1.033	Medium boiling	0.200
Ethanol	78°C	694	879	0.789	Polar protic	0.159
Ethyl acetate	77°C	355	394	0.9	Low boiling	0.189
Formic acid	101°C	601	493	1.22	Polar protic	0.200
n-Hexane	69°C	243	368	0.66	Low boiling	0.206
Isopropanol (IPA)	82°C	549	699	0.786	Polar protic	0.154
Methanol	65°C	971	1227	0.791	Polar protic	0.167
Pentane	36°C	239	381	0.626	Low boiling	0.214
n-Propanol	97°C	633	787	0.804	Polar protic	0.154
Tetrahydrofuran (THF)	66°C	395	444	0.889	Low boiling	0.192
Toluene	111°C	370	427	0.867	Medium boiling	0.202
Water	100°C	2261	2266	0.998	Polar protic	0.167
Xylene (mixture of isomers)	138.5°C	338	389	0.87	Medium boiling	0.199

*) Distillation of high-boiling solvents is not recommended with the Multivapor.

2.2 Calculation of boiling point from applied pressure

$$T_p = \frac{T_{bp}}{(3.006 - \log p) \cdot b + 1} \quad \text{(Equation 1)}$$

T_p [K] is the temperature of the boiling point at the pressure p [mbar], T_{bp} [K] the boiling point under standard conditions, and b the solvent specific constant.

## 2.3 Evaporation of low-boiling solvents

2.3.1 Solvent: Acetone (≥99.8%)

System Configuration	
Multivapor	P-12
Cover	PETP (black)
Cover sealing	EPDM (black)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters		
Solvent	Acetone	≥99.8%
	Evaporated volume	240 ml, 20 ml/position
	Category	Low-boiling
	Boiling point	56°C
	Desired boiling point	30°C
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient		
Starting point	850 mbar	
Ramp	850-518 mbar	1 min
	518-361 mbar	2 min
	361-333 mbar	2 min
Constant	333 mbar	6 min

Evaporation performance	
Positions	12
Total volume	240 ml
Time	11 min
Rate overall	1.3 l/h
Rate per position	109 ml/h
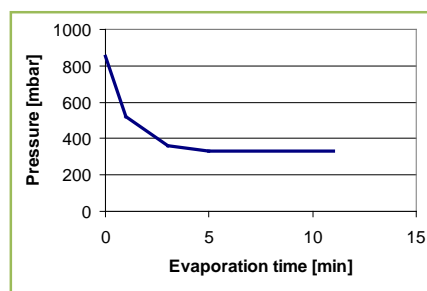
Results	
Total process time	11 min
Solvent recovery after main condenser	235 ml (98%)
Solvent recovery after secondary condenser	< 1 ml

EasyVac	Detection start point	Distillation	Detection end point
Acetone	☑	☑	☑
Limitations:			
Glassware diameter < 13 mm			
Glassware diameter 13 < x < 19 mm	☑	☑	
Glassware diameter > 19 mm	☑	☑	☑

2.3.2 Solvent: Ethyl acetate (Analytical grade)

System Configuration

Multivapor	P-12
Cover	PETP (black)
Cover sealing	FFKM (black)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent	Ethyl acetate	Analytical grade
	Evaporated volume	240 ml, 20 ml/position
	Category	Low-boiling
	Boiling point	77°C
	Desired boiling point	30°C
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient

Starting point	653 mbar	
Ramp	653-242 mbar	2 min
	242 mbar	1 min
	242-140 mbar	2 min
Constant	140 mbar	5 min

Evaporation performance

Positions	12
Total volume	240 ml
Time	10 min
Rate overall	1.4 l/h
Rate per position	120 ml/h

Results

Total process time	10 min
Solvent recovery after main condenser	237 ml (98.8%)
Solvent recovery after secondary condenser	< 1 ml

EasyVac

	Detection start point	Distillation	Detection end point
Ethyl acetate	☑	☑	☑
Limitations:			
Glassware diameter < 13 mm			
Glassware diameter 13 < x < 19 mm	☑	☑	
Glassware diameter > 19 mm	☑	☑	☑

2.3.3   Solvent: *n*-Hexane (purum ≥95.0%)

System Configuration

|	
Multivapor	P-12
Cover	PETP (black)
Cover sealing	FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent	*n*-Hexane	Purum ≥95.0%
	Evaporated volume	360 ml, 30 ml/position
	Category	Low-boiling
	Boiling point	69°C
	Desired boiling point	30°C
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient

Starting point	741 mbar	
Ramp	741-291 mbar	2 min
	291-237 mbar	2 min
	237-230 mbar	2 min
	230-202 mbar	3 min
Constant	202 mbar	1 min

Evaporation performance

Positions	12
Total volume	360 ml
Time	10 min
Rate overall	2.2 l/h
Rate per position	180 ml/h
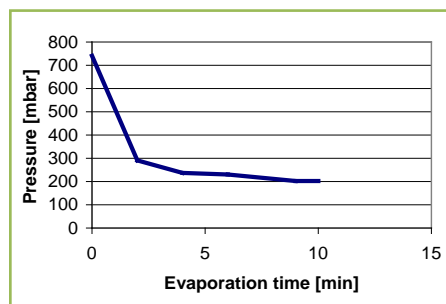
Results

Total process time	10 min
Solvent recovery after main condenser	360 ml (100%)
Solvent recovery after secondary condenser	< 1 ml

EasyVac

	Detection start point	Distillation	Detection end point
n-Hexane	☑	☑	☑
Limitations:			
Glassware diameter < 13 mm			
Glassware diameter 13 < x < 19 mm	☑	☑	
Glassware diameter > 19 mm	☑	☑	☑

2.3.4 Solvent: *n*-Hexane – P-6 (purum ≥95.0%)

System Configuration

|	
Multivapor	P-6
Cover	PETP (black)
Cover sealing	FKM (green)
Vessel type	BUCHI SpeedExtractor vessel (52672)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent	*n*-Hexane	Purum ≥95.0%
	Evaporated volume	600 ml, 100 ml/position
	Category	Low-boiling
	Boiling point	69°C
	Desired boiling point	30°C
Heating medium	Temperature	55°C
	Volume	145 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Evaporation performance

Positions	6
Total volume	600 ml
Time	14 min
Rate overall	2.6 l/h
Rate per position	429 ml/h

Pressure gradient

Starting point	741 mbar	
Ramp	741-308 mbar	2 min
	308-241 mbar	2 min
	241 mbar	3 min
	241-213 mbar	1 min
Constant	213 mbar	6 min
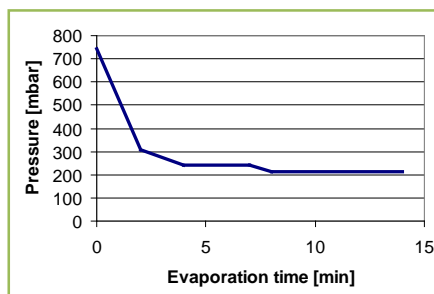
Results

Total process time	14 min
Solvent recovery after main condenser	583 ml (97.2%)
Solvent recovery after secondary condenser	2 ml

EasyVac

	Detection start point	Distillation	Detection end point
n-Hexane	☑	(☑)	Too early

2.4 Evaporation of medium-boiling solvents

2.4.1 Solvent: Xylene (Mixture of isomers, purum ≥97.0%)

System Configuration	
Multivapor	P-12
Cover	PETP (black)
Cover sealing	FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700
	Woulff flask (48857)

Parameters		
Solvent	Xylene	Purum ≥97.0% (isomers)
	Evaporated volume	240 ml, 20 ml/position
	Category	Medium-boiling
	Boiling point	139°C
	Desired boiling point	50°C
Heating medium	Temperature	80°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient		
Starting point	200 mbar	
Ramp	200-30 mbar	3 min
Constant	30 mbar	4 min

Evaporation performance	
Positions	12
Total volume	240 ml
Time	7 min
Rate overall	2.1 l/h
Rate per position	171 ml/h
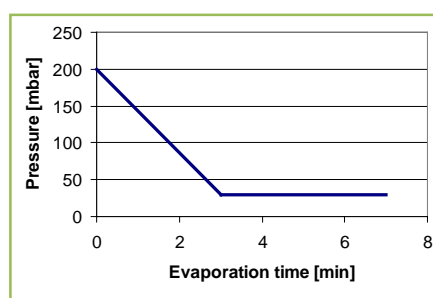
Results	
Total process time	7 min
Solvent recovery after main condenser	187 ml (77.9%)
Solvent recovery in Woulff flask	52 ml (21.7%)
Solvent recovery after secondary condenser	1 ml

EasyVac	Detection start point	Distillation	Detection end point
Xylene			

2.5 Evaporation of high-boiling solvents

2.5.1 Solvent: Dimethylformamide, DMF (purum, ≥98.0%)

System Configuration

Multivapor	P-12
Cover	PETP (black)
Cover sealing	EPDM (black)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent	N,N-Dimethyl-formamide (DMF)	Purum ≥98.0%
	Evaporated volume	240 ml, 20 ml/position
	Category	High-boiling
	Boiling point	153°C
	Desired boiling point	60°C
Heating medium	Temperature	80°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	20°C
	Measured flow	1200 ml/min
Rotational speed	Position 10	

Pressure gradient

Starting point	400 mbar	
Ramp	400-28 mbar	2 min
Constant	28 mbar	23 min

Evaporation performance

Positions	12
Total volume	240 ml
Time	25 min
Rate overall	0.6 l/h
Rate per position	48 ml/h
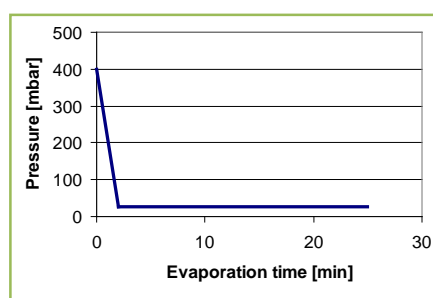
Results

Total process time	25 min
Solvent recovery after main condenser	236 ml (98.3%)
Solvent recovery after secondary condenser	1 ml

EasyVac

	Detection start point	Distillation	Detection end point
N,N-Dimethylformamide			

2.6 Evaporation of polar protic solvents

2.6.1 Solvent: Ethanol (4.8% methanol, purum, ≥99.8%)

System Configuration

Multivapor	P-12
Cover	PETP (black)
Cover sealing	EPDM (black) / FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700 PE frit (44856)

Parameters

Solvent	Ethanol (4.8% methanol) Evaporated volume Category Boiling point Desired boiling point	Purum, ≥99.8% 240 ml, 20 ml/position Polar protic 78°C 30°C
Heating medium	Temperature Volume	55°C 45 ml H_2O/position
Cooling medium (F-105)	Water/glycol Temperature Measured flow	70:30% (v/v) 10°C 1200 ml/min
Rotational speed	Position 9	

Pressure gradient

Starting point	602 mbar	
Ramp	602-173 mbar	2 min
	173-96 mbar	3 min
	96-86 mbar	4 min
	86 mbar	4 min
	86-78 mbar	6 min

Evaporation performance

Positions	12
Total volume	240 ml
Time	19 min
Rate overall	0.8 l/h
Rate per position	63 ml/h
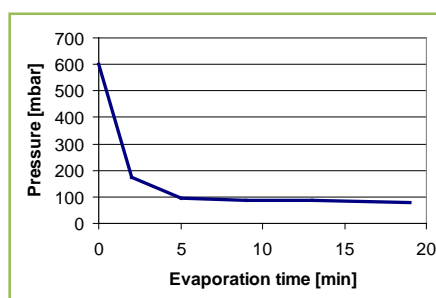
Results

Total process time	19 min
Solvent recovery after main condenser	239 ml (99.6%)
Solvent recovery after secondary condenser	< 1 ml

EasyVac	Detection start point	Distillation	Detection end point
Ethanol	☑	☑	☑
Limitations:			
Glassware diameter 13 < x < 19 mm	☑	☑	
Glassware diameter > 19 mm	☑	☑	☑

2.6.2 Solvent: Distilled water, H₂O

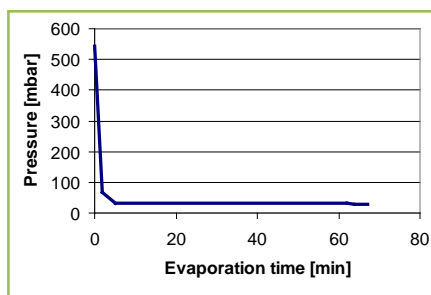**System Configuration**

Multivapor	P-12
Cover	PETP (black)
Cover sealing	EPDM (black) / FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700
	Woulff flask (48857)

Parameters

Solvent	Distilled water	BUCHI Fontavapor 210
	Evaporated volume	240 ml, 20 ml/position
	Category	Polar protic
	Boiling point	100°C
	Desired boiling point	30°C
Heating medium	Temperature	55°C
	Volume	45 ml H₂O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Evaporation performance

Positions	12
Total volume	240 ml
Time	67 min
Rate overall	0.2 l/h
Rate per position	18 ml/h

Pressure gradient

Starting point	542 mbar	
Ramp	542-69 mbar	2 min
	69-33 mbar	3 min
Constant	33 mbar	57 min
	33-27 mbar	2 min
Constant	27 mbar	3 min

Results

Total process time	67 min
Solvent recovery after main condenser	235 ml (97.9%)
Solvent recovery in Woulff flask	3 ml (1.3%)
Solvent recovery after secondary condenser	2 ml

EasyVac

	Detection start point	Distillation	Detection end point
Water	☑		
Limitations:			
Glassware diameter < 13 mm			
Glassware diameter 13 < x < 19 mm	☑		
Glassware diameter > 19 mm	☑		

3 Evaporation of solvent mixtures

Evaporation of the solvent mixtures hexane / dichlormethane and ethanol / water has been discussed extensively in the best@buchi 06/2001.[7] Custom mixtures are addressed similarly. Solvent mixtures are usually distilled by evaporation of one component after the other. In the presented examples, a boiling temperature of 30 to 40°C is sought in order to protect the products.

Optimal conditions are determined in a boiling diagram. Theoretical useful pressures are determined by the heat medium temperature and the lowest possible condensation temperature. The boiling curve is calculated using Equation 1 (Section 2.2).

3.1 Hexane / dichloromethane 7:3 (e.g. flash chromatography)

The problem lies in the fact that the dichloromethane has to be distilled off at a relatively high system pressure of from 750 to 800 mbar, while hexane requires a system pressure of about 300 mbar (**Figure 10**). If dichloromethane is distilled off at too low pressure, then the condenser will soon be overburdened and solvent emissions into the environment become unavoidable.

To avoid this, dichloromethane is distilled off at an only slightly reduced pressure and then the system pressure is lowered to distill hexane. Such a lowering of pressure in stages can of course be done manually.

The monitoring time is considerable, however. Due to the fact that the boiling temperature of the hexane residue rises at a certain pressure with decreasing dichloromethane content, there is also always the danger that there could be a boiling delay if a stage is slightly too large.

If, however, the system pressure is continuously lowered after a short starting period, i.e., along a pressure gradient, then this danger is avoided and the distillation process can be carried out without supervision or servicing.
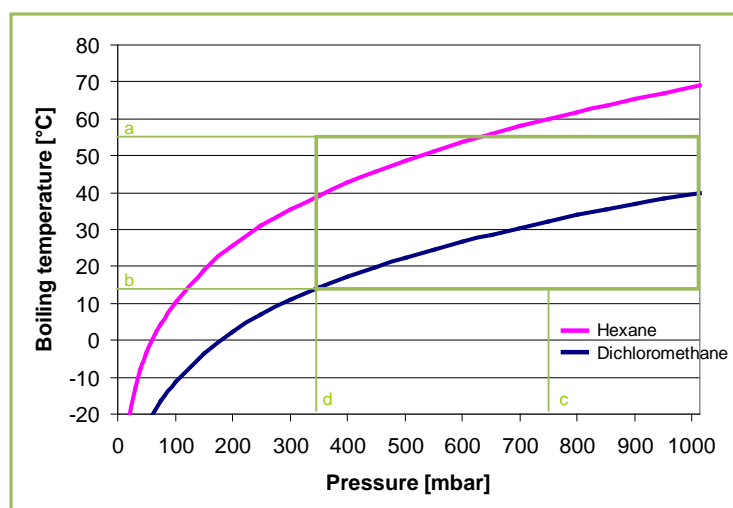

Figure 10: Boiling diagram for dichloromethane/hexane. *a* Heat medium temperature; *b* Lowest possible condensation temperature for a cooling temperature of 10°C. The rectangle area, which is surrounded in green, marks the theoretically useful area; *c* → *d* shows the used pressure range.

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[7] A. Talamona, G. Schönenberger, Vacuum control during vaporization of problematic solutions, best@buchi no. 06/2001.

Solvent mixture: Hexane / dichloromethane 7:3

| System Configuration	
Multivapor	P-12
Cover	PEEK (ecru) / PETP (black)
Cover sealing	FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters		
Solvent mixture	Hexane	≥96%
	Dichloromethane	≥99.8%
	Ratio	7:3 (v/v)
	Evaporated volume	240 ml, 20 ml/position
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient		
Starting point	760 mbar	
Constant	760 mbar	3 min
Ramp	760-350 mbar	7 min
Constant	350 mbar	2 min

Evaporation performance	
Positions	12
Total volume	240 ml
Time	12 min
Rate overall	1.2 l/h
Rate per position	100 ml/h
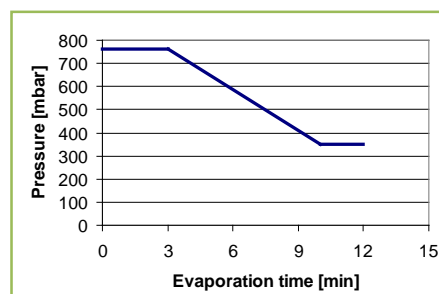
Results	
Total process time	12 min
Solvent recovery after main condenser	237 ml (98.8%)
Solvent recovery after secondary condenser	1 ml

3.2 Ethanol / water 6:4 (e.g. reverse phase-chromatography)

Ethanol/water mixtures tend to bump and foam during the distillation process. If the distillation process is carried out very carefully, then the process can be almost endless. If, on the other hand, the pressure is lowered a bit too sharply, then the solution foams over and the distillation has to be started all over again! Additionally, the distillation is performed at a truly low-pressure level to achieve the desired boiling point (**Figure 11**).

In this application as well, a vacuum control system with programmable pressure gradient is very helpful and results in a significant reduction in laboratory personnel. The distillation record sheet on the next page shows how the pressure during manual operation is reduced rather tediously during the first 20 minutes of operation, always close to the limit of over-foaming, and how the problem can be elegantly solved with the use of a gradient function. True, the distillation process lasts a bit longer, but the danger of over-foaming is clearly eliminated.
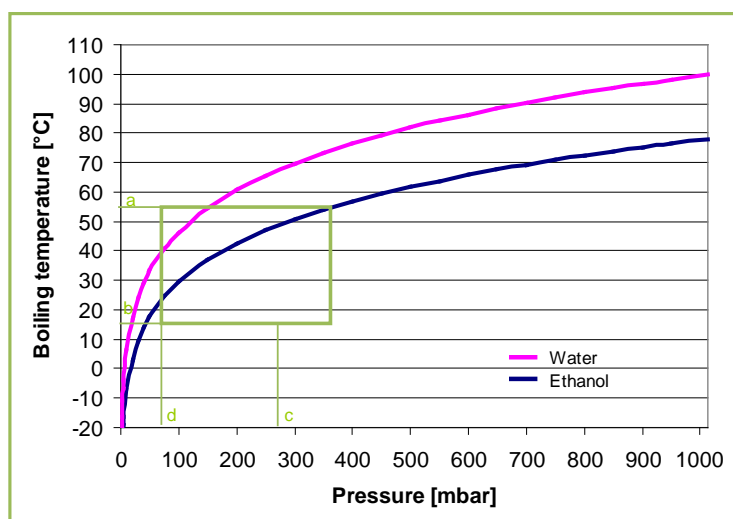

Figure 11: Boiling diagram for ethanol/water. *a* Heat medium temperature; *b* Lowest possible condensation temperature for a cooling temperature of 10°C. The rectangle area, which is surrounded in green, marks the theoretically useful area; *c* → *d* shows the used pressure range.

Solvent mixture: Ethanol / water 6:4

System Configuration

|	
Multivapor	P-12
Cover	PETP (black)
Cover sealing	EPDM (black) / FKM (green)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent mixture	Ethanol (absolute)	purum ≥99.8%
	Distilled water	BUCHI Fontavapor 210
	Ratio	6:4 (v/v)
	Evaporated volume	240 ml, 20 ml/position
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	10°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient

Starting point	602 mbar	
Ramp	602-267 mbar	2 min
	267-107 mbar	4 min
	107-84 mbar	2 min
Constant	84 mbar	49 min

Evaporation performance

Positions	12
Total volume	240 ml
Time	57 min
Rate overall	0.3 l/h
Rate per position	21 ml/h
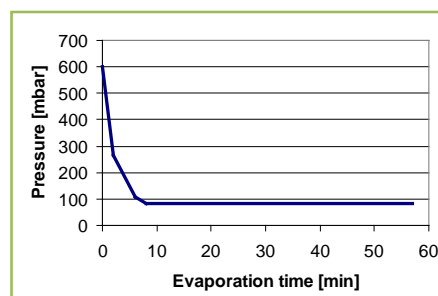
Results

Total process time	57 min
Solvent recovery after main condenser	225 ml (93.8%)
Solvent recovery after secondary condenser	4 ml

3.3 Azeotrope chloroform / methanol 8:1 (e.g. flash chromatography)

An azeotrope a mixture of two or more liquids in such a ratio that its composition cannot be changed by simple distillation. The vapour of boiled azeotrope has the same ratio of constituents as the original mixture

Each azeotrope has a characteristic boiling point. The boiling point temperature of an azeotrope is either less than the boiling point temperatures of any of its constituents (a positive azeotrope), or greater than the boiling point temperatures of any of its constituents (a negative azeotrope). Decrease of boiling point is seen in the phase diagrams of the positive azeotrope chlororform / methanol (**Figure 12**).
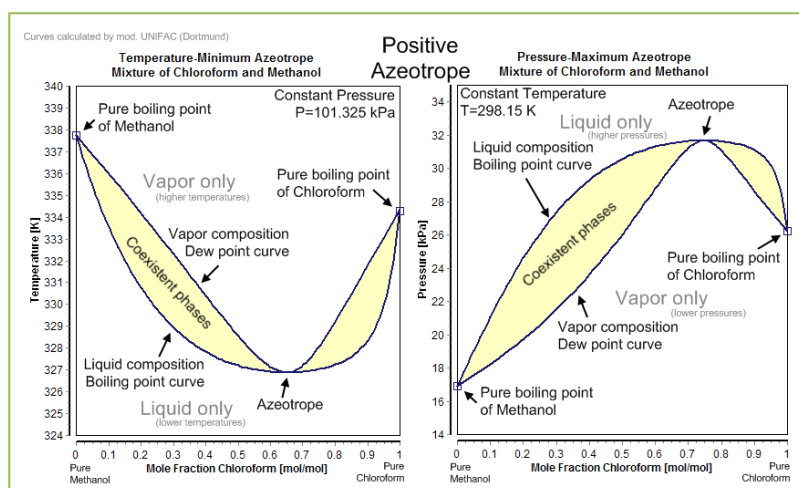

Figure 12: Phase diagrams of the positive azeotrope chlororform / methanol.[8]

If two solvents can form a positive azeotrope, then distillation of any mixture of those constituents will result in the distillate being closer in composition to the azeotrope than the starting mixture. Repeated series distillations will produce mixtures that are progressively closer to the azeotropic ratio. But you cannot concentrate above the azeotropic ratio. Distillation of a mixture above the azeotropic point, the distillate (contrary to intuition) will be poorer in higher concentrated volatile compound than the original but slightly richer than the azeotrope.

Similarly, if two solvents can form a negative azeotrope, then distillation of any mixture of those constituents will result in the residue being closer in composition to the azeotrope than the original mixture. Boiling of any negative azeotrope solution long enough will cause the solution left behind to approach the azeotropic ratio.

Non-azeotropic solvent mixtures are distilled by evaporation of one component after the other. Assuming chloroform / methanol is non-azeotropic, optimal conditions are displayed in the rectangle area of the boiling diagram (**Figure 13**). In fact, chloroform / methanol at a ratio of

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[8] Azeotrope. Wikipedia.org, the free encyclopedia. http://en.wikipedia.org/wiki/Azeotrope. Last visit: February 17th 2011.

8:1 corresponding to 0.8 mol-% has a boiling point of 54°C (**Figure 12**). Therefore, distillation is performed at a higher vacuum than with non-azeotropic solvent mixtures (*c* → *d*, **Figure 13**).
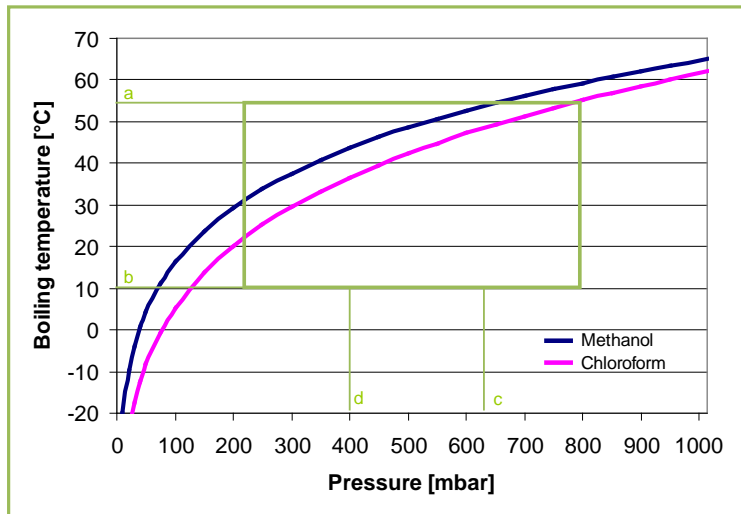

Figure 13: Boiling diagram for chloroform/methanol. *a* Heat medium temperature; *b* Lowest possible condensation temperature for a cooling temperature of 5°C. The rectangle area, which is surrounded in green, marks the theoretically useful area; *c* → *d* shows the used pressure range.

Solvent mixture: Chloroform / methanol 8:1

System Configuration

|	
Multivapor	P-12
Cover	PEEK (ecru) / PETP (black)
Cover sealing	FKM (green) / FFKM (black)
Vessel type	BUCHI SpeedExtractor tube (49535)
Options	Vacuum controller V-855 (firmware 3.03)
	Vacuum pump V-700

Parameters

Solvent mixture	Chloroform	puriss. p.a. ≥99.8%
	Methanol	≥99.8%
	Ratio	8:1 (v/v)
	Evaporated volume	240 ml, 20 ml/position
Heating medium	Temperature	55°C
	Volume	45 ml H_2O/position
Cooling medium (F-105)	Water/glycol	70:30% (v/v)
	Temperature	5°C
	Measured flow	1200 ml/min
Rotational speed	Position 9	

Pressure gradient

Starting point	800 mbar	
Ramp	800-612 mbar	1 min
	612-480 mbar	2 min
	480-400 mbar	5 min
Constant	400 mbar	6 min

Evaporation performance

Positions	12
Total volume	240 ml
Time	14 min
Rate overall	1.0 l/h
Rate per position	86 ml/h
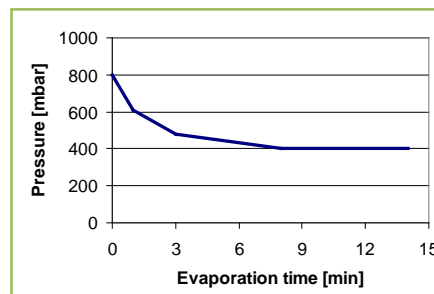
Results

Total process time	14 min
Solvent recovery after main condenser	237 ml (98.8%)
Solvent recovery after secondary condenser	1 ml

4 Appendix A – Reference BUCHI Application Notes

4.1 Fat Determination in Shortbread using SpeedExtractor E-916

The determination of fat in food and feed is a routine procedure for quality assurance and labeling. Fat was determined in butter shortbread after homogenization of the sample and extraction with the SpeedExtractor E-916. The total fat content was determined gravimetrically after the extract has been dried to a constant weight. The fat content of 10.21% corresponds to the value determined with classic Soxhlet.

Introduction

The determination of fat in food and feed is a routine procedure for in quality assurance and labeling. Fat was determined in butter shortbread after homogenization of the sample and extraction with the SpeedExtractor E-916. The solvent was evaporated in parallel using the Multivapor P-6. The total fat content was determined gravimetrically after the extract has been dried to a constant weight.

Experimental

Instrumentation: Mixer B-400, SpeedExtractor E-916, Multivapor P-6 with Vacuum pump V-700 and Controller V-855, drying oven. The homogenized sample was mixed with quartz sand and extracted with the SpeedExtractor using the parameters shown in the adjacent table. The sample was extracted in triplicate.

The total time for the extraction is approx. 60 min and per position, approx. 50 ml solvent are used. The solvent was evaporated in parallel using the Multivapor P-6 (see **Figure 15**). The extracts were then dried to a constant weight in a drying oven (102°C) and the fat content was calculated.

Results

The determined fat content (**Table 1**) of 10.20% corresponds to the content obtained when extracting the same sample with Soxhlet extraction. With a fat content of 10.20% (rsd=0.42%, n=3) was determined.

Table 1: Determined fat contents in short bread

Results	Fat content
Sample 1	10.25 %
Sample 2	10.14 %
Sample 3	10.24 %
Mean value	**10.21 %**
rsd%	0.60

Figure 14: Shortbread

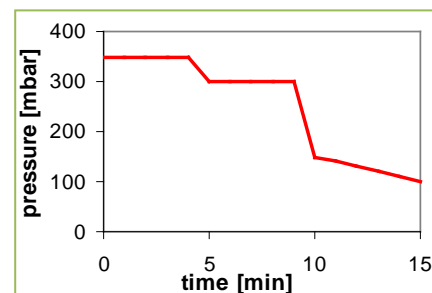| Extraction settings	
Temperature	100°C
Pressure	100 bar
Solvent	*n*-Hexane
Cells	40 ml
Vials	240 ml
Cycles	3
Heat-up	1 min
Hold	5 min
Discharge	4/3/3 min (1st/2nd/3rd cycle)
Flush with solvent	2 min
Flush with gas	4 min

Figure 15: Pressure gradient for evaporating the solvent in the Multivapor P-6

References

SpeedExtractor E-916 operation manual

For more detailed information refer to Application note 005/2009

4.2 Flash chromatography on cartridges for the separation of plant extracts

Flash chromatography on cartridges for the separation of plant extracts - Rules for the selection of chromatographic conditions, and comparison with MPLC

Weber P[1], Schafroth N[2], Hamburger M[1], Potterat O[1]

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[2] Büchi Labortechnik AG, CH-9230 Flawil, Switzerland

Introduction

During the last decade, several systems for rapid preparative chromatography with pre-packed cartridges have been commercialized. Pre-packed cartridges ensure rapid separation cycles and ease of use.

Flash chromatography systems were initially developed for rapid and easy purification of synthetic products, and numerous applications are documented in the experimental section of publications in synthetic chemistry. In contrast, application of such systems in the separation of complex natural product mixtures such as extracts has been neglected.

The aim of this work was to explore the potential and limitations of cartridges for the purification of natural product extracts. Empirical rules have been established for the determination of the separation conditions by preliminary TLC and HPLC analyses. The influence of solid introduction compared to liquid injection has been also investigated. The performance of the cartridges was compared to that of classical self-packed MPLC glass columns for the separation of complex plant extracts of medicinal importance.

Results

Correlation between HPLC and reversed phase flash chromatography

In preliminary experiments with reference compounds, a correlation was found between the capacity factors k' on the HPLC column and Sepacore cartridges ($k'_{Sepacore} = 1.15 \times k'_{HPLC}$; $R^2 = 0.97$). HPLC separations can be transposed by increasing the gradient time by a factor 2-4. Theses rules have been applied to the separation of dicloromethane extracts of *Curcuma xanthorrhiza* and *Piper nigrum*.
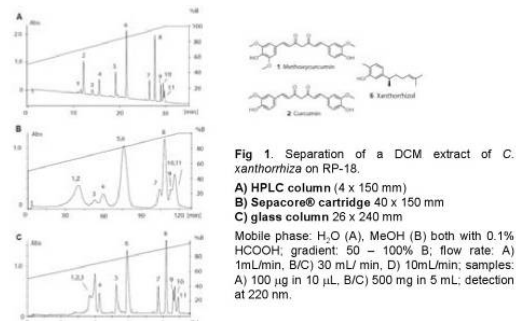
Fig 1. Separation of a DCM extract of *C. xanthorrhiza* on RP-18.
A) HPLC column (4 x 150 mm)
B) Sepacore® cartridge 40 x 150 mm
C) glass column 26 x 240 mm
Mobile phase: H$_2$O (A), MeOH (B) both with 0.1% HCOOH; gradient: 50 – 100% B; flow rate: A) 1mL/min, B/C) 30 mL/ min, D) 10mL/min; samples: A) 100 µg in 10 µL, B/C) 500 mg in 5 mL; detection at 220 nm.
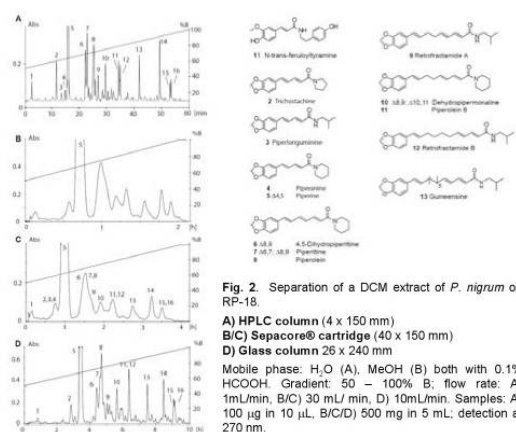
Fig 2. Separation of a DCM extract of *P. nigrum* on RP-18.
A) HPLC column (4 x 150 mm)
B/C) Sepacore® cartridge (40 x 150 mm)
D) Glass column 26 x 240 mm
Mobile phase: H$_2$O (A), MeOH (B) both with 0.1% HCOOH. Gradient: 50 – 100% B; flow rate: A) 1mL/min, B/C) 30 mL/ min, D) 10mL/min. Samples: A) 100 µg in 10 µL, B/C/D) 500 mg in 5 mL; detection at 270 nm.

Transposition from TLC to flash chromatography on silica gel

In preliminary experiments, a correlation was found between $1/R_f$ and k' values (k'= 1.1 x $1/R_f$; R^2 = 0.90). Mobile phase compositions for starting and end point of a gradient should be selected such as to obtain R_f values of 0.15-0.2 for the most lipophilic component and the most hydrophilic constituent, respectively. This principle has been applied to the separation of a dichloromethane extract of *Salvia miltiorrhiza*.
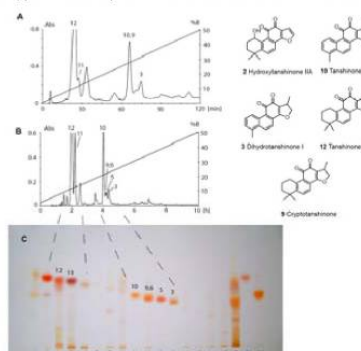
Fig. 3. Separation of a DCM extract of *S. miltiorrhiza* on silica gel.
A) Sepacore® cartridge (40 x 150 mm), B) Glass column 26 x 240 mm. Mobile phase: A/B) n-hexane (A), EtOAc (B); gradient: 2-50% B; slow rate: A) 30 mL/min, B) 10 mL/min; sample: A) 500 mg in 10 mL n-hexane-EtOAc (6:4), B) solid introduction using a precolumn; detection at 265 nm. C) TLC analysis of the extract and the fractions obtained from B with n-hexane-EtOAc-MeOH (25:25:1); R1: tanshinone IIA, R2: cryptotanshinone.

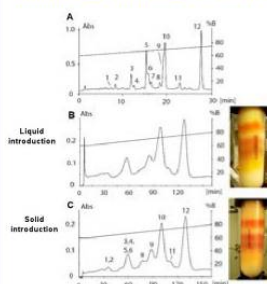### Influence of the mode of sample introduction

Fig. 4. Influence of the sample introduction mode on the separation of a DCM extract of *S. milthiorrhiza*.
A) HPLC column (4 x 150 mm)
B/C) Sepacore® cartridge (40 x 150 mm)
Mobile phase: H$_2$O (A), MeOH (B) both with 0.1% HCOOH; gradient: A) 60 – 75% B in 30 min, B/C) 60% B for 10 min, 60 – 80% B in 110 min; flow rate: A) 1 mL/min, B/C) 30 mL/min; detection at 250 nm; sample: A) 100 µg in 10 µL, B) liquid introduction, 500 mg in 8 mL MeOH, C) solid introduction, 500 mg.

Conclusions

- Reversed phase HPLC separations can be transposed by increasing the gradient time by a factor 2-4.
- For normal phase separations, solvent compositions resulting in R_f values of 0.15-0.2 on TLC for the most lipophilic and the most hydrophilic constituents, respectively, should be selected as gradient endpoints.
- Sepacore® cartridges enabled a good separation of compounds with a broad range of polarity, as typically found in plant extracts. The chromatographic resolution remained, however, lower than that achieved by MPLC on columns packed with material of smaller particle size. For poorly soluble extracts, solid introduction gave better results than liquid injection.
- Despite lower resolution as compared to MPLC, pre-packed cartridges are an attractive alternative for the purification of extracts and crude fractions due to their ease of use and speed of separation.

Instrumentation

Preparative separations were performed on a Sepacore® chromatography system (*Büchi Labortechnik*) consisting of two C 605 pump modules, a C 620 control unit, a C 635 UV detector and a C 660 fraction collector. The system was controlled by the software SepacoreControl 1.0. Flash chromatography separations were performed on pre-packed silica gel (40 – 63 µm) and RP18ec (40 – 63 µm) polypropylene cartridges (12 x 150 or 40 x 150 mm, *Büchi*) at a flow rate of 10 mL (12 x 150 mm cartridges) and 30 mL/min (40 x 150 mm cartridges), resp. Medium pressure liquid chromatography (MPLC) separations were carried out on a glass column (26 x 460 mm) packed with silica gel Si60 (15 – 40 µm) or LiChroprep RP18 (25 – 40 µm) (*Merck*), at a flow rate of 10 mL/min. Liquid injection was carried out through a 6-way valve with a 20 ml loop. Solid introduction was performed by means of a PrepElut cartridge (Flash chromatography) or a glass precolumn (MPLC) connected to the top of the cartridge or the column, resp. The samples were adsorbed to silica gel Si60 or LiChroprep RP-18, resp., prior to introduction.

References

The original Poster is available at:
http://www.buchi.com/Applications.20162.0.html

Publication:
Weber, P., Hamburger, M., Schafroth, N., Potterat, O. 2011. Flash chromatography on cartridges for the separation of plant extracts: Rules for the selection of chromatographic conditions and comparison with medium pressure liquid chromatography. Fitoterapia 82:155–161.

5 Appendix B – Recommended Multivapor version for a specific solvent

5.1 Abbreviations

EPDM	Ethylenepropylenedimonomer	O-ring
FKM	Fluoric caoutchouc	O-ring
FFKM	Perfluoro caoutchouc	O-ring
PEEK	Polyetheretherketone	Vacuum cover and adapter
PET(P)	Polyethyletherphtalate	Vacuum cover and adapter
PFA	Perfluoroalkoxy	Vacuum hose
PTFE	Polytetrafluoroethylene	Sealing discs

5.2 Chemical resistance table

	EPDM*[9]	FKM*[10]	FKKM*[11]	PEEK*[9]	PET(P)*[13]	PFA**[12]	PTFE**[12]
Acetaldehyde	B	D	A	A	A	A	A
Acetic Acid	A	B	A	A	A	A	A
Acetic acid anhydride	B	D	A	A	A	A	A
Acetone	A	D	A	A	B	A	A
Benzene	D	A	A	A	A	A	A
Butanol	B	A	A	A	B	A	A
Chloroform	D	A	A	A	B	A	A
Diethyl ether	C	C	A	A	A	A	A
Dimethylformamide	A	-	A	A	B	A	A
Dimethylbenzene (Xylol)	D	A	A	A	A	A	A
Dioxane	B	-	A	A	A	A	A
Ethanol	A	A	A	A	A	A	A
Ethyl acetate	B	D	A	A	-	A	A
Hexane	C	A	A	A	A	A	A
Isobutanol	A	A	A	A	A	A	A
Isopropanol	A	A	A	A	A	A	A
Methanol	A	B	A	A	A	A	A
Methylene chloride	D	A	A	A	D	A	A
Nitrobenzene	C	B	A	B	D	A	A
Phenol	B	A	A	B	C	A	A
Propanol	A	A	A	A	A	A	A
Sulphuric acid, fuming	C	A	A	C	C	A	A
Carbon tetrachloride	D	A	A	A	A	A	A

[9] Operation Manual, Syncore® Accessories

[10] Connectors, Verbindungstechnik AG, Chem. Beständigkeitsliste

[11] ExpotechUSA, www.expotechusa.com

[12] Semadeni, Chemical resistance table for polymers, www.semadeni.com

[13] Prelon GmbH Dichtungstechnik, Chem. Beständigkeit von Kunststoffen, www.prelon.de

	EPDM	FKM	FKKM	PEEK	PET(P)	PFA	PTFE
Tetrahydrofurane	B	D	A	A	A	A	A
Toluene	D	A	A	A	A	A	A
Triethylamine	C	A	A	A	-	-	-
Trichloroethane	D	A	A	A	A	-	-
Trichloroacetic acid	B	-	-	A	-	A	A
Vinylidene chloride	D	-	A	A	B	-	-
Aq. HBr, sat.	B	A	A	C	-	A	A
Aq. HCl, sat.	A	A	A	B	A	A	A
Aq. ammonia solution	A	D	A	A	A	A	A
Aqueous caustic soda	A	B	A	A	B	A	A
Aqueous nitric acid	B	A	A	B	B	A	A

*A: Very good resistance, B: Moderate resistance, C: poor resistance, D: very poor resistance

** A: Very good resistance, B: Moderate resistance, C: poor resistance

Please note: The resistance against the corresponding vapors is significantly better. Tabled values may vary by changing temperature and pressure.

Notes

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For optimal
distillation
consider the BUCHI rule

10 °C

30 °C

55 °C
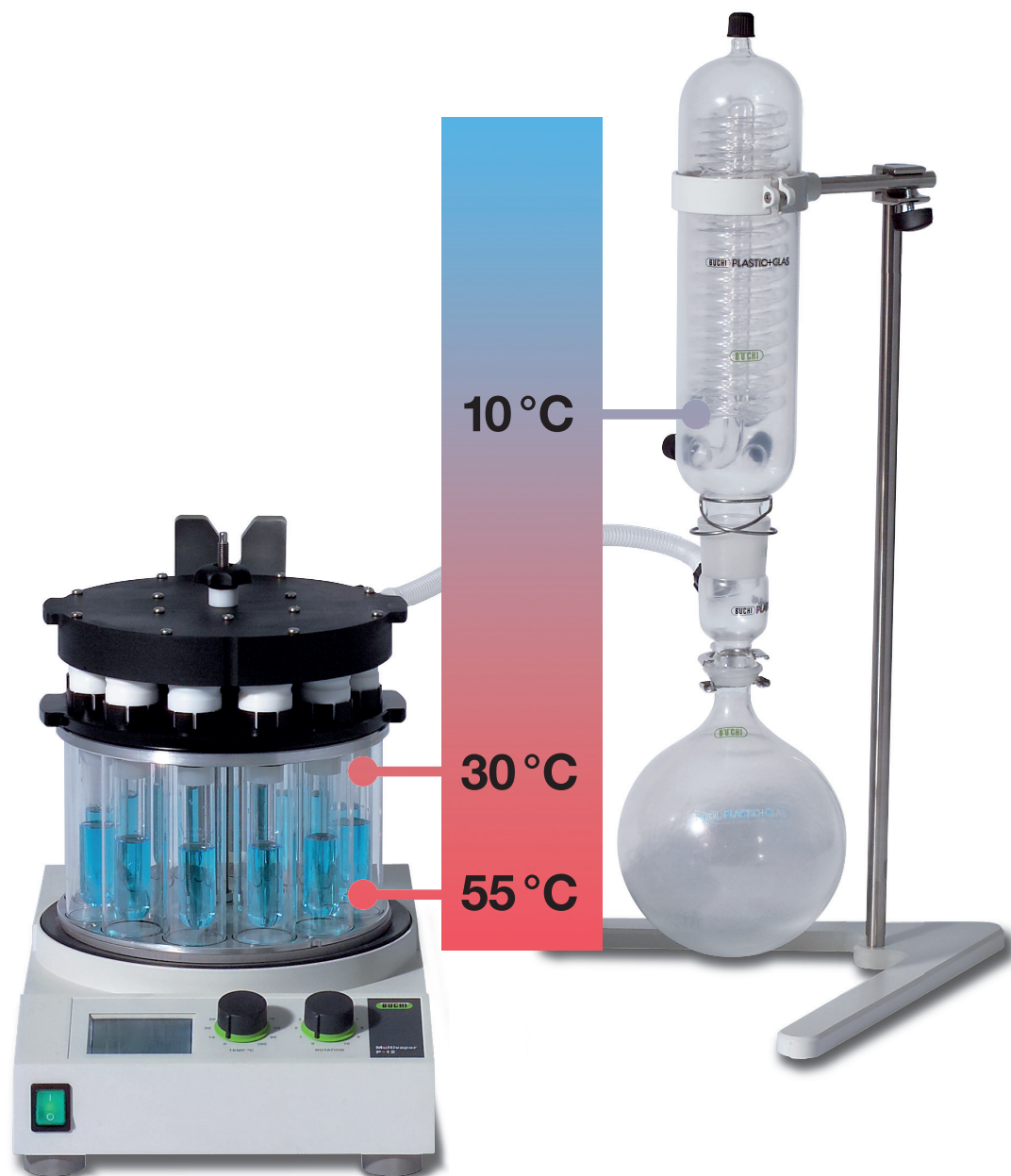
For evaporation at 30 °C (boiling point)

1. instrument setting: 55 °C
2. cooling medium: 10 °C,   500 ml/min
3. recommended rotation setting: position 8–9
4. vacuum setting according to the Solvent List column 30 °C

Solvent List

Solvent	Formula	Vacuum [mbar] for bp at:	
		30 °C	50 °C
Acetic acid	$C_2H_4O_2$	26	72
Acetone	C_3H_6O	370	815
Acetonitrile	C_2H_3N	134	315
n-Amylalcohol, n-Pentanol	$C_5H_{12}O$	6	20
n-Butanol	C_4H_{10}	14	44
tert-Butanol	$C_4H_{10}O$	78	231
Chlorobenzene	C_6H_5Cl	22	56
Chloroform	$CHCl_3$	306	665
Cyclohexane	C_6H_{12}	154	347
1,2-Dichloroethane	$C_2H_4Cl_2$	137	315
Dichloromethane	CH_2Cl_2	685	atm. press.
Diethylether	$C_4H_{10}O$	838	atm. press.
trans-1,2-Dichloroethylene	$C_2H_2Cl_2$	317	705
Diisopropylether	$C_6H_{14}O$	251	545
Dioxane	$C_4H_8O_2$	68	165
Dimethylformamide (DMF)	C_3H_7NO	6	17
Ethanol	C_2H_6O	97	276
Ethylacetate	$C_4H_8O_2$	153	366
Heptane	C_7H_{16}	77	183
Hexane	C_6H_{14}	241	525
Isopropylalcohol	C_3H_8O	78	231
Isoamylalcohol	$C_5H_{12}O$	9	29
Methyl tert-buthyl ether (MTBE)	$C_5H_{12}O$	413	835
Methyl ethyl ketone (MEK)	C_4H_8O	160	359
Methanol	CH_4O	236	607
Pentane	C_5H_{12}	819	atm. press.
n-Propanol	C_3H_8O	37	115
Pentachloroethane	C_2HCl_5	8	21
Tetrachloromethane	CCl_4	179	398
Tetrahydrofurane (THF)	C_4H_8O	234	539
Toluene	C_7H_8	48	118
Trichloroethylene	C_2HCl_3	119	275
Water	H_2O	42	120
Xylene	C_8H_{10}	15	40

Quality in your hands

Quality in your hands