Syncore®
Application Guide

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This application guide describes important aspects of the evaporation of solvents and the concentration of a sample to a predefined volume using the Syncore®. Guidelines for the evaporation of your specific solvent and solvent mixtures are presented. They will help you to streamline your own process with sample applications, checklists, hints, rules, tables and tests. Further information about applications can be found on our website, www.buchi.com, in BUCHI Application Notes and best@buchi publications. You can also contact your local BUCHI representative for additional information on a particular application.

BUCHI Labortechnik AG, 2012, Dr D.M. Meier, M. Heierli, Dr D.P. Rütti and J.B. Reid

1 Introduction

The BUCHI Syncore® is a vacuum vortex evaporator for parallel evaporation of up to 96 samples with a volume of 0.5 to 500 ml per sample. The sample can either be evaporated to dryness or concentrated to a predefined volume of 0.3, 1 or 3 ml. In addition, purification of the samples to be evaporated or concentrated can be performed with a solid phase extraction (SPE) module using common SPE cartridges.

Today, the Syncore® is employed in a variety of environmental laboratories, in the chemical- and pharmaceutical industries, as well as in research-, food- and other analytic laboratories to prepare samples for further analysis.[1]

The aim of this Application Guide is to provide tips and tricks for optimizing existing evaporation processes and to assist the Syncore® user in developing new applications.

[1] See for example: A. Kaufmann et. al Journal of Chromatography A, 1194, 66-79, 2008.; T.A. Bucheli, Chemosphere 56, 1061–1076, 2004.

2 Syncore® - Configurations

Common to all Syncore® configurations is the platform (Figure 1), the core component. The platform performs an orbital movement with a maximum speed of maximal 600 rpm at programmable temperatures (up to 150 °C), producing a strong vortex in the sample vessel. This is ideal for fast solvent evaporation.

Figure 1. Syncore® platform.

Depending on the configuration, i.e. the attachment placed onto the platform, the solvent is either fully evaporated (Polyvap configuration) or concentrated to a predefined volume (Analyst configuration). A special cover plate can be used for solid phase extraction, SPE, a method often applied in the preparation of environmental samples.

In this chapter, the different configurations and their applications are described in detail. The available Syncore® configurations and the corresponding working sample volumes are listed in Table 1.

Table 1. Available Polyvap, Analyst and SPE configurations

Sample positions	Working volume [ml]	Polyvap	Analyst	SPE*
4	50-500	✓	✓	
6	25-250	✓	✓	✓
12	5-120	✓	✓	✓
24	2-30	✓		✓
48	1-20	✓		
96	0.5-10	✓		

* SPE basic module available for the R-12 and R-24, SPE advanced module available for the R-6 and R-12.

2.1 Syncore® Polyvap

When using the Polyvap configuration, the samples are fully evaporated by means of gentle heating under vacuum. Exchangeable sample racks allow parallel evaporation of 4, 6, 12, 24, 48, and 96 samples with a volume of 0.5-500 ml.

Figure 2. Exchangeable Polyvap rack with 24 sample glasses and a vacuum cover.

To maintain a reduced pressure, i.e. a vacuum, during operation, a vacuum cover is fitted onto the sample vessels. In Figure 2, a Polyvap sample rack with 24 sample vessels and the corresponding vacuum cover is shown. Condensation of evaporated solvent inside the cover is prevented by heating the vacuum cover itself (up to 70 °C). The vapors are directed to the condenser yielding a complete solvent recovery. Cross-contamination is avoided by an individual sample sealing system and separate vacuum channels to each sample. The vacuum cover is made of chemically inert materials (see Appendix).

Typical applications of the Polyvap configuration are the evaporation of chromatography samples, evaporation after parallel extraction, or reactions and gravimetric analyses in quality control.

2.2 Syncore® Analyst

In contrast to the Polyvap configuration, the Analyst configuration is designed to concentrate – in parallel – up to 12 samples with working volumes of 0.5-500 ml down to predefined residual volumes of 0.3, 1, or 3 ml. A key feature of the Analyst is an integrated cooling zone that collects the concentrated sample in a cooled environment (Figure 3).[2]

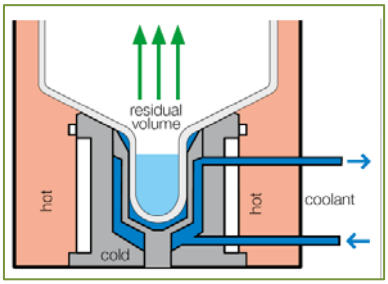


Figure 3. Analyst rack cools the appendix of the sample vessel avoiding evaporation to dryness.

The cooling zone is cooled by water from a recirculation chiller or tap water and helps to efficiently retain a defined volume of sample in the analyst glass appendix. The cooling temperature selected for the glass appendix and the vacuum applied must be coordinated. The Analyst vacuum cover is identical to the Polyvap cover.

The Analyst configuration is predominantly applied for environmental analyses, food-processing and quality control where the concentration of an extract is required.

2.3 Syncore® Solid Phase Extraction (SPE)

In SPE, a liquid sample is passed over a bed of so-called stationary phase. Depending on the affinity of the substances for the stationary phase, they either pass over or are retained on the stationary phase. Depending on whether the fraction that passes the stationary phase contains the desired substance it is collected or discarded. If the analytes adhere to stationary phase, they can then be eluted from the stationary phase for collection in an additional step, in which the stationary phase is rinsed with an appropriate eluent.

Figure 4. Above: SPE advanced module. Below: SPE basic module with equipped with 12 cartridges.

SPE can easily be performed with the Syncore® by installing an SPE-module having 6, 12, or 24 cartridge ports compatible with the corresponding Polyvap and Analyst racks (Figure 4). All essential sample SPE work-up steps can be performed without any in-between handling.

Unique feature of this setup is a three-way stopcock, which allows liquid separation into either a waste vessel or a collection vessel after passing through a SPE cartridge (Figure 6).[2] This makes it possible to first transfer the liquids of the conditioning, adsorption and washing steps into the waste vessel, and then to elute directly into the evaporation vessel. No exchange of glassware or aeration whatsoever of the vacuum manifold is required. Moreover by turning the stopcock into the stop position, the elute can directly be evaporated either to dryness or to a pre-defined residual volume. Typical SPE applications comprise environmental and foodstuffs analysis.[3]

Figure 5. BUCHI vacuum controller V-855 (professional).

[2] C. Blum, R. Hartmann, Speed Extractor, Application Booklet, BUCHI Labortechnik AG, 2009.

[3] P. Kölbener, A. Wernli and R. Hartmann, best@buchi 43, 2006.

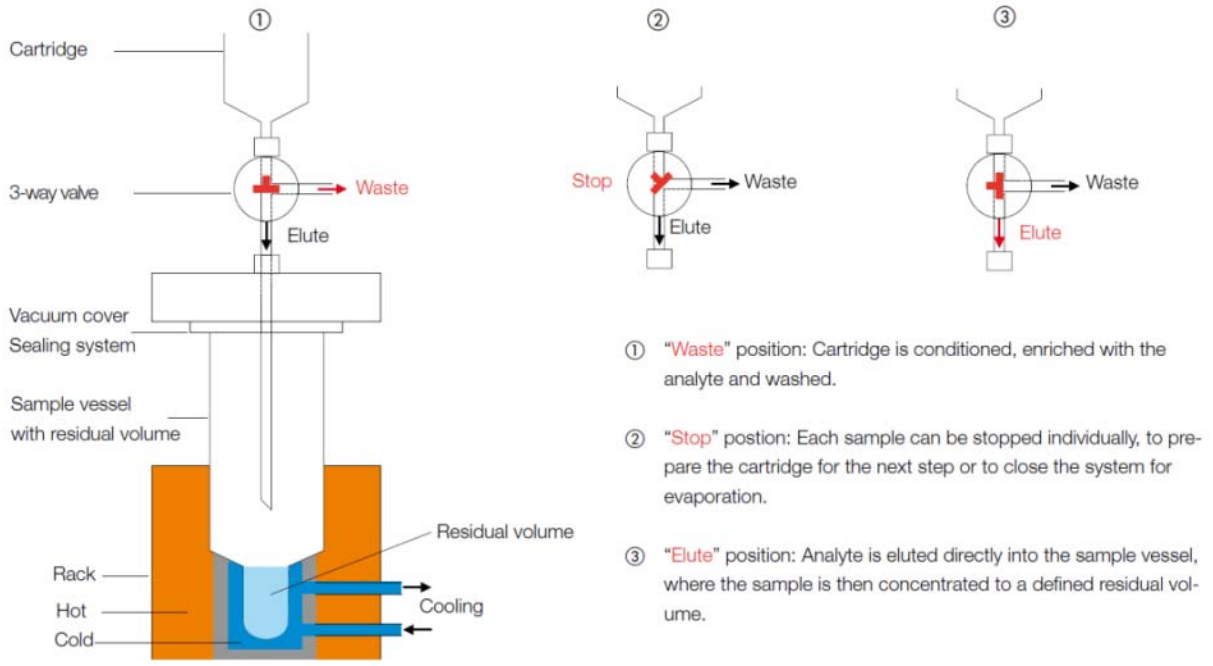


Figure 6. Operation principle of the three-way stopcock of the advanced Syncore® SPE module.

2.4 Recommended Accessories

2.4.1 Vacuum Pump, Vacuum Controller, and Recirculation Chiller

A prerequisite for a smooth evaporation process is the control of the vacuum. The combination of a BUCHI V-700/V-710 pump and a BUCHI V-855 vacuum controller (Figure 5) is designed to be used with the Syncore®. With this combination, pressure gradients can be programmed to ensure efficient, smooth, and reproducible evaporation.

Constant cooling of the Syncore® condenser is achieved with a BUCHI recirculation chiller (Figure 7). Low temperature cooling allows gentle distillation at low temperatures with high solvent recovery yielding an environmentally benign process, with no solvent loss to the atmosphere. Furthermore, the V-855 vacuum controller can also control the recirculation chiller.

Figure 7. BUCHI recirculation chiller.

2.4.2 Appendix Glasses for Analyst Configurations

Special glasses with an appendix volume of 0.3, 1, or 3 ml, shown in Figure 8, are available for all Analyst configurations (R-4, R-6, and R-12). These glasses are available with (Figure 8) or without graduation. Using these glasses, the sample can be concentrated to the predefined volume. Due to the cooled appendix, the residual volume remains stable for hours.

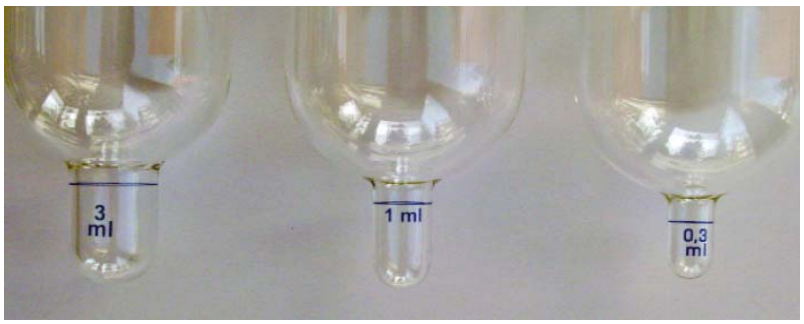


Figure 8. Analyst glasses with different appendix sizes.

2.4.3 O-Ring Appendix Insulation

To protect the fluid remaining in the appendix from heating up and evaporation, an insulation is placed around the appendix (Figure 9). With this appendix insulation the sample remains in the appendix even when the platform continues to heat.

This insulation is optionally available for the appendix sizes of 0.3 ml (transparent) and 1 ml (red). For the 3 ml appendix there is no need for an additional insulation since it perfectly fits into the sample holder.

Figure 9. O-Ring Appendix insulation for Analyst sample vessels. Left: for 1ml Appendix; Right: for 0.3 ml Appendix.

2.4.4 Amber Glass

Many chemical compounds are light sensitive - they undergo molecular transformation upon interaction with UV or visible light. These compounds have to be treated and stored in lightproof glass vessels. BUCHI offers amber glasses for all Syncore® configurations that prevent the compound from interaction with light and possible degradation. Important is that the amber coating is applied on the outside and not on the inside of the glass vessel. This assures that the coating does not leach into the sample.

Figure 10. Flushback Module for an R-6 configuration.

2.4.5 Flushback Module

Both the Polyvap and Analyst configurations (R-6 and R-12), are optionally equipped with a so-called Flushback Module (shown in Figure 10). The module is placed on the rack and connected to a cooling source. With this unique feature, the top of each vessel is cooled; there the vaporized solvent partially condenses as it leaves the sample vessel, causing a gentle continuous rinse of the glass wall during the entire evaporation process. This ensures that the dissolved sample remains at the bottom of the vessel or in the cooled appendix and does not stick to the glass wall. It has been demonstrated (see Figure 11) that the Flushback Module significantly enhances the analyte recovery rate in particular for analytes with a high affinity for glass.

We suggest to cool the Flushback Module before starting the evaporation process. Usually, the Flushback Module is cooled by the same cooling source as the appendix and the condenser.

Figure 11. Effect of the Flushback Module. Left: When using the Flushback Module the analyte is collected in the appendix. Right: Without using the Flushback Module some analyte sticks to the glass wall of the vessels.

Clearly, the drawback of the Flushback effect is a decrease in the evaporation speed.[4] The better the Flushback effect, the slower the evaporation process.

2.4.6 High-Boiling Insulation

Solvents with a boiling point above 150 °C are so called high-boiling solvents. Examples of high-boiling solvents are dimethylformamide (DMF, bp. 153°C) and dimethylsulfoxide (DMSO, bp. 189 °C). Due to their high boiling points, evaporation of these solvents requires high vacuum and elevated temperatures.

To speed-up the evaporation of high-boiling solvents, an optional insulation kit is available, shown in Figure 12. With this insulation, the energy is kept in the system, resulting in a more efficient evaporation (see section 6.6 for application examples of applications).

Figure 12. Insulation-kit for high boiling solvents.

2.4.7 PE-Frits

Porous polyethylene (PE) frits can be installed to close the vapor duct and to protect the vacuum cover from splashes and foaming samples can be installed. Two frit sizes, available as optional parts, are compatible with the respective vacuum cover formats.

2.5 Installation Qualification (IQ) and Operation Qualification (OQ)

Installation- and Operation Qualifications (IQ/OQ) are mandatory when using good manufacturing practice (GMP), required in the pharmaceutical and chemical industry. For the Syncore®, both protocols (IQ and OQ) are available.

The Installation Qualification ensures that the Syncore® instrument is correctly set up and complies with the specifications. The environment of the instrument is checked according to the specific installation requirements for the subsequent Operational Qualification.

The Operational Qualification is intended to ensure and record that the Syncore® meets predefined specifications within a specific environment. The Operational Qualification is a procedure that must be repeated periodically. The Operational Qualification protocols allow the test conditions and test results to be fully inspected and documented.

[4] For example, evaporation of 12 times 60 ml ethyl acetate in an R-12 was 6 % slower when employing the Flushback module and otherwise identical conditions.

2.6 Example Set-up

The schematic set up of a Syncore® Analyst System, with a Flushback module and a V-700 Professional vacuum system, a secondary condenser and a BUCHI recirculation chiller is illustrated in Figure 13. Pathways of the cooling water are shown in blue.

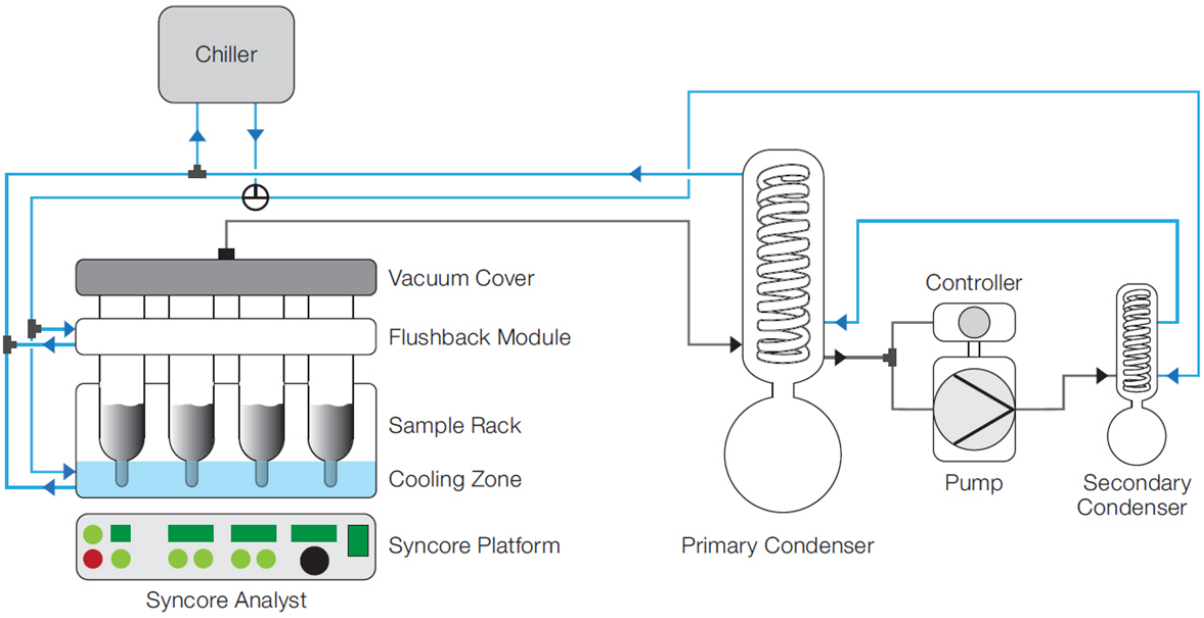


Figure 13. Schematic set-up of a Syncore® Analyst System including a pump and a chiller. Blue lines represent the cooling water loop.

3 Process Checklist

In Table 2 a process checklist guiding the Syncore® user through the evaporation process is presented. The user can follow the checklist point by point. Checkpoints printed in grey are optional but recommended for an efficient process. All other checkpoints are mandatory.

Table 2. Syncore® Process Checklist. Checkpoints printed in **black** are mandatory checkpoints printed in grey are optional but recommended.

Process step	Checkpoints	Reference
Installation	☑ *Install Syncore® platform according to manual*	
	☐ *Check water and electricity connections*	
	☐ *IQ/OQ*	2.5
Platform and Cover Preparation	☐ *Choose configuration – Polyvap, Analyst, SPE*	2
	☐ *Choose sample glasses*	2
	☐ *Check balance and eccentricity*	4.3
	☐ *Insert PE frits*	2.4.7
	☐ *Install high boiling insulation*	2.4.6
	☐ *For Analyst configuration: use appendix insulation*	2.4.3
	☐ *Fill-in heat transfer medium*	4.8
	☐ *Tightness check*	5.2
	☐ *Set evaporation temperatures (the Δ25/20 rule) and press start*	4.4, 4.5
	☐ *Preheat the sample block and cover (30 min)*	4.4
Set Evaporation Parameters	☐ *Set pressure gradient*	4.6.2
	☐ *Place Flushback module*	
	☐ *Place the samples*	
	☐ *Close vacuum cover*	
	☐ *Set vortex speed*	4.9
Evaporation Process	☐ *Control load of the condenser*	4.7
	☐ *Determine end of process*	4.11
	☐ *Stop procedure*	5.6
Cleaning	☐ *Cleaning procedure*	5.7

4 Parameters, Settings and their Impact on Distillation

4.1 Instrument Configuration

Evaporation rates greatly differ for the Polyvap and Analyst configurations. Using these two configurations and employing the same conditions (see 6.3) yielded an increase of the evaporation efficiency when switching from the Analyst to the Polyvap configuration. As a rule of thumb, using similar conditions the evaporation rate is about twice as fast with the Polyvap than with the Analyst set-up.

A change in the rack size can also affect the evaporation efficiency. Maximization of the surface to volume ratio by adjusting the vortex speed is required in this case (see section 4.7).

4.2 Initial Solvent Volume

For optimum evaporation it is advised to fill the vessel to no more than the maximum working volume (Table 1).

In a test series the effect of different starting volumes on the evaporation efficiency was evaluated using an R-4 Polyvap configuration. A pressure gradient was applied.[5]

It was found that the evaporation rate slows down when the starting volume is small compared to the total volume of the tubes. In this case the gradient time, i.e. the time required for reaching the final vacuum, is long compared to the total evaporation time, hence, the evaporation efficiency is reduced.

4.3 Platform Eccentricity and Balance

Depending on the rack to be installed, the eccentricity of the platform has to be adjusted according to Table 3. The correct eccentricity helps to obtain a vortex movement such that a sample becomes thoroughly mixed, i.e. is brought into a swirling movement at a minimum shaking speed. For example, a sample in an R-6 glass (using a Polyvap R-6 configuration) is brought to a swirling movement very efficiently at 250 rpm with an eccentricity of 4.0 mm. With an eccentricity of 2.5 mm, significantly higher speeds of 400 rpm are needed for the same sample to swirl.

Table 3. Rack dependent optimum eccentricities.

Rack Type	Eccentricity
R-4	4-5 mm
R-6	4-5 mm
R-12	4-5 mm
R-24	2.5-4.5 mm
R-96	2-4 mm

[5] Evaporation conditions: Crystal Rack R-4 Polyvap, Vacuum controller V-855 (firmware 3.03), Vacuum pump V-700, Recirculation chiller with set temperature 10°C, Platform temperature 55 °C, 160 rpm, pressure gradient: 500-200 mbar in 3 min, 200-150 mbar in 3 min, hold at 150 mbar until process end, solvent: ethyl acetate (Merck 99.5 %).

After changing the eccentricity, the platform has to be balanced again. The balancing weight compensates for the moving masses of the base plate and the rack fastened to it. Such compensation is necessary to ensure smooth and safe operation. The greater the weight of the base plate, with the accessories and samples on it, and the greater the eccentricity of the vortex movement, the farther the balancing weight must be from the axis as described in the Syncore® platform manual.

4.4 Temperatures - The ΔT 25/20 °C Rule

For optimum performance, the Syncore® platform is preheated to the required temperature 30 min prior to starting the evaporation. The maximum temperature is 150 °C.

Simultaneously the vacuum cover is preheated. The maximum temperature to which the vacuum cover can be heated is 70 °C.

As a starting point to find the optimum temperatures for the rack, the boiling point, and the condensation temperature, a rule of thumb can be applied – the ΔT 25/20 °C rule. This rule specifies the temperature difference between the three different zones, i.e., the heating plate, the vapor temperature[6], and the cooling temperature, as illustrated in Figure 14. When for example the temperature of the platform is set to 65 °C, the vacuum should be set such that a boiling point of 40 °C results, i.e., a ΔT of 25 °C. In order to achieve sufficient condensation, the cooling temperature should be lower by at least another ΔT of 20 °C. Hence, in this example ≤ 20 °C.

Figure 14. Illustration of the Δt 25/20 °C rule.

When using the analyst configuration the cooling medium for the condenser is also used to cool the appendix. When applying the ΔT 25/20 °C rule the heating and cooling temperatures differ by 45 °C, which assures that the analyst residual volume remains in the appendix.

4.5 Vacuum Cover Temperature

Set the temperature of the vacuum cover to at least 5 °C higher than the temperature of the solvent vapor (boiling point). A temperature of the vacuum cover lower than that of the solvent fumes causes the fumes to condense in the vacuum cover. Maximum cover temperature is 70 °C.

4.6 Vacuum and Boiling Point

Boiling is referred to as the state where the vapor pressure equals the pressure acting on the liquid's surface. Logically, reducing this

[6] The temperature of the vapor above a boiling (evaporating) liquid phase is identical to the temperature of the boiling point.

pressure, by applying a vacuum, lowers the boiling point of the solution.

In Figure 15, the pressure-dependent boiling point of pure water is illustrated. A decrease of the pressure from ambient to 120 mbar reduces the boiling point from 100 °C to 50 °C. A further pressure decrease to 42 mbar reduces the boiling point of water to 30 °C. From Figure 15 is obvious that at low pressures the boiling point may vary greatly with small pressure changes. Therefore at low pressures distillation has to be performed carefully to avoid boiling, bumping and foaming. To avoid bumping and foaming, the vacuum is reduced, or the system is shortly aerated at constant temperature.

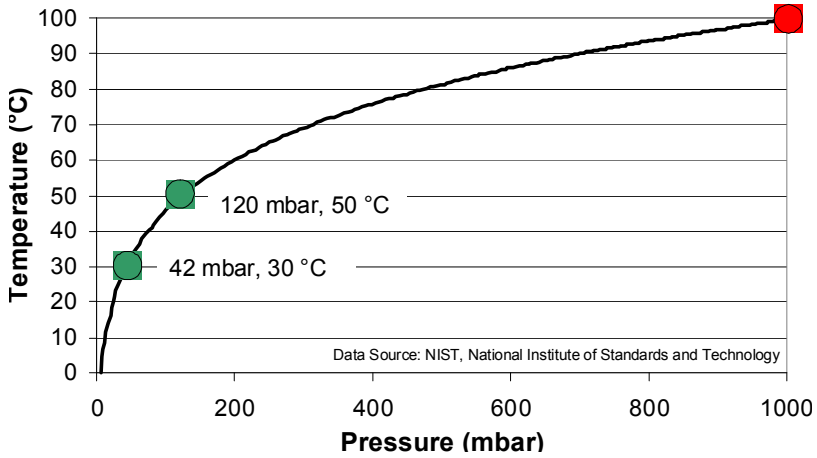


Figure 15. Pressure-dependent boiling point of water. Red point indicates the boiling point (bp.) in ambient conditions.

4.6.1 How to Determine the Vacuum for given Boiling Point by Manual Distillation

The most convenient way to control the applied vacuum is to use a BUCHI vacuum pump in combination with the BUCHI vacuum controller V-850 or V-855. To determine the vacuum that has to be applied for given evaporation conditions follow these points:

1. Set the platform temperature, e.g. to 65 °C
2. Determine the pressure using the Solvent List so that a boiling point of ΔT 25 °C below, i.e. 40 °C, results
3. Set the vacuum cover temperature at least 5 °C higher than the boiling point
4. Set the cooling temperature another 20 °C lower, i.e. ≤ 20 °C
5. Optimize the as-found process

4.6.2 How to Determine a Vacuum Gradient for Distillation

To avoid boiling retardation, foaming, bumping-up or loss of analyte, use of a pressure gradient is recommended. Gradients also allow convenient integration of “drying” steps by applying a very low pressure after the actual evaporation.

To prevent re-evaporation of solvent from the receiving flask during “drying”, cool the distillate with an ice bath or a refrigerated receiver. As a first approach for setting a gradient, the following procedure can be taken as a guideline:

1. Start 500 mbar above the calculated pressure according to the ΔT 25/20 °C rule
2. Decrease the pressure by 350 mbar in 4 min
3. Decrease pressure further by 100 mbar in 5 min and another 60 mbar in 10 min
4. Keep the pressure constant until the evaporation is finished
5. For volatile compounds, slow aeration over 5 min is recommended

After a first assessment, optimization of the gradient is recommended to shorten the process time.

4.7 Condenser Load

For efficient condensation of the vapor, the temperature of the condenser should be at least 20 °C (Δ25/20 rule) lower than the vapor temperature, i.e. the boiling point. When evaporated at relatively low temperatures, i.e. instrument settings of approx. 50 °C, a recirculation chiller is required to maintain the temperature. When tap water is used as a cooling agent, seasonal temperature differences have to be considered when adjusting the vacuum and heating settings.

Figure 16. Illustration of the optimum condenser load.

Whenever the condensate covers approximately half the height of the condenser, the evaporation is optimum (Figure 16). Higher condenser loads usually have a negative impact on solvent recovery. To avoid possible emissions of solvent vapor into the environment, the use of a post-pump secondary condenser is highly recommended.

When no more condensation is observed in the condenser, the evaporation process is finished.

4.8 Heat and Heating Medium

Energy is needed to evaporate the solvents in the sample vessels. Energy is provided to the sample by heating the platform. For an efficient evaporation, the applied heat has to be transported from the platform to the rack, from the rack to the glass vessel, and from the glass to the solvent.

Small gaps, filled with air, between the rack and the sample vessel may significantly slow down the heat transfer to the sample. This is because air has a very low heat transfer coefficient compared to water and aluminum. Thus gaps between the rack and the glass vessels have to be filled with water for an efficient heat transfer.

When using the crystal racks (R-4, or R-6 configuration), the amount of water to be added is indicated. For all other racks, the gap between the heating block and the glass should be completely filled.

Heat supplied to evaporate the solvent has to be removed by the condenser to liquefy the solvent again. When supplying more energy than can be dissipated by the condenser, the solvent is lost to the environment due to an overburdened condenser. Escaping solvent vapor may also condensate in the pump and reduce its efficiency.

Furthermore, acceleration of the distillation through excess heat increases the risk of foaming and bumping as well as the loss of analyte with the vapor stream. Bumping and foaming can be avoided by reducing the heat supply and further measures (see section 5).

4.9 Vortex Speed and Vacuum Hose

Another important factor that influences the speed and the recovery is the surface area of the sample. The larger the surface, the faster the evaporation. The surface area is determined by the rotational speed of the circularly moving heating plate and the size of the glass vessels. Faster rotation increases the surface area and hence accelerates the process. However, as shown in Figure 17, it also distributes the sample on a larger glass area, which usually reduces the recovery.

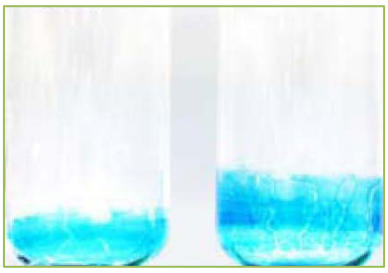


Figure 17. Grease sticking to the glass. Left after evaporation at 170 rpm (left) and 300 rpm (right).

Generally, better results in terms of recovery are achieved by generating a smooth vortex with the lowest possible rotation. Such conditions are obtained by optimizing the eccentricity and balance according to chapter 4.1.

Starting the vortex movement and applying the vacuum should be done immediately after placing the samples in the rack and closing the vacuum cover.

With decreasing sample volume in the vessel during evaporation the optimum vortex speed may change and can be adjusted accordingly.

The vortex shaking movement of the rack sets the vacuum hose into a rotating movement together with the vacuum connection. The vacuum hose must not come into contact with or rub against other parts of the unit. To avoid back mixing, there should be a slight drop of the vacuum hose connecting the vacuum cover and the adapter to the condenser unit.

4.10 Solvent Mixtures

Parallel evaporation of solvent mixtures is often needed when working with solvent fractions collected during chromatographic separation using a solvent gradient.

Interestingly, mixtures of solvents may have evaporation properties different than their pure constituents. Evaporation tables of pure solvents can only give a first hint on the evaporation conditions of solvent mixtures. Two solvents with a boiling point difference of more than 80 °C can be separated by a single distillation.[7] Solvents with similar boiling points tend to form azeotropes. An azeotrope is a solvent mixture whose composition cannot be changed by distillation. You can check whether your solvent mixture forms an azeotrope using public databases.[8]

When evaporating solvents with high boiling point differences, it is important to cool the receiver flask, in order to avoid re-evaporation of the lower-boiling solvent.

Figure 18 illustrates an evaporation process of an ideal mixture of components X and Y. Starting from point 1 the solvent mixture of 50 % X and 50 % Y is heated up. At point 2a, the solution starts to evaporate. Interestingly, the composition of the vapor phase, 2b, is different from that of the liquid phase, 2a. In fractional distillation, different fractions of re-condensed vapor phases are collected. When using the Syncore®, the recondensed vapor is collected in the collection vessel. With the progress of the evaporation, the compositions of the vapor and liquid phases change. In the liquid phase more and more X is found while the vapor phase contains less Y (point 3a and 3b). Finally, when everything is evaporated (indicated by points 4 and 5) the vapor composition equals the initial composition.

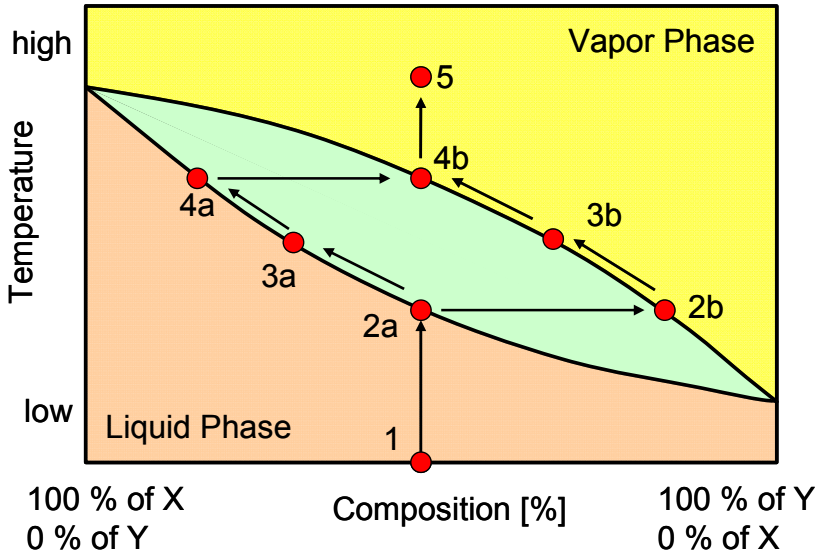


Figure 18. Evaporation of an ideal solvent mixture of 50 % X and 50 % Y. The progress and the composition of the mixture are indicated by red points (see text).

Attention: When employing the Analyst configuration for solvent mixtures the higher boiling solvent is collected in the appendix while the lower boiling solvent escapes from the sample vessel. If

[7] BUCHI -The Laboratory Assistant, ISBN 98-3-033-01315-5, Flawil 2007.

[8] http://eweb.chemeng.ed.ac.uk/chem_eng/azeotrope_bank.html

the dissolved components are not soluble in the higher boiling solvent, they will precipitate in the appendix.

4.11 End of Process

In general, evaporation is finished when no more condensation is observed at the condenser. In most of the Polyvap configurations, the end of the process can also be determined by checking the solvent level in the sample vessel.

However, when using the Analyst configuration it is not possible to visually check the solvent level in the appendix. Therefore the user has to optimize his process.

In this Application guide, experiments were terminated when the condensation rate was lower than one drop per minute. In this way, the appendix volume, when using the Analyst configuration, was never lower than indicated, i.e. 0.3, 1 or 3 ml.

5 Troubleshooting

5.1 Overview

On the one hand, an economic evaporation process should be as fast as possible. However, a too fast evaporation process could lead to foaming, bumping, condensation of the solvent in the cover as well as to lower analyte recoveries.

On the other hand, a too slow evaporation process that avoids the above problems can be very costly. Hence, there is a trade-off between “fast and efficient”, and “slow and inefficient” operation, which asks for an optimum solution.

Problems and their solutions encountered in optimizing the evaporation process are listed in Table 4.

Table 4. Solutions to the most frequent problems.

Problem	Action	Reference
Foam	*1. Reduce vacuum*	4.6
	2. Program gradient	4.6.2
	3. Install frits	2.4.7
Overheating, bumping up	*4. Reduce vacuum*	4.6
	5. Program gradient	4.6.2
	6. Reduce heat	4.8
	7. Reduce time span from immersing the vessels into the heat medium to starting the vacuum and vortex movements	
Condensation in cover	*8. Increase cover temperature*	4.5
	9. Reduce boiling temperature (increase vacuum) and adjust heating medium as well as condenser temperature accordingly	4.4, 4.5
	10. Ensure that there is a slight drop between the vacuum connection and the adapter on the condenser unit	4.9
	11. Use high boiling insulation	2.4.6
Slow distillation speed	*12. Improve heat transfer*	4.8
	13. Optimize rotational speed	4.9
	14. Increase vacuum	4.6
	15. Increase platform/rack temperature	4.4
Inhomoge-neous evaporation	*16. Control heat transfer medium level*	5.3
	17. Preheat the sample block and cover (30 min)	4.4
	18. Clean interface between rack and platform	5.3
	19. Make a tightness check	5.2

5.2 Tightness Check

A prerequisite for an efficient evaporation is a tight system. In order to measure the leak rate, close the vacuum line to the vacuum pump with a clamp. This measure ensures that the leak rate of the vacuum pump does not contribute to the tightness of the Syncore® system.

The tightness of the Syncore® system is tested in a closed, empty and dry system by stopping evacuation when the set vacuum of 50 mbar is reached. Then the vacuum is monitored for over a period of 2 minutes.

For the formats, 4, 6, 12 and 24 the leak rate must not exceed 15 mbar/min. For the formats, 48 and 96 the leak rate must not exceed 35 mbar/min. If the results of the test do not comply with these values, the tightness of the system has to be checked for leaks. Typically, the observed leak rates are lower, as listed in Table 5.

Table 5. Expected typical values for the tightness of a dry Syncore® system.

Syncore® System	Set vacuum	≤ 50 mbar
	Pressure increase	≤ 3 mbar/min
Pump	End vacuum	< 15 mbar

5.3 Inhomogeneous Heating

If inhomogeneous evaporation is observed make sure that evaporation does not start before a heat-up time of 30 minutes. In addition, the Syncore® platform and the rack should be checked visually for scratches, chemical contamination, dust or mechanical damage. A smooth and even surface is a prerequisite for efficient and uniform heat transfer. Furthermore, check the level of the heat transfer medium between rack and sample vessel. Finally, perform a tightness check to make sure that there are no leaks.

5.4 Insufficient Solvent Recovery Rate

Evaporated solvent that is not recovered in the receiving flask escapes into the environment. Since evaporated solvents may be harmful for people working in the proximity of the device and the environment, solvent loss must be prevented. Unwanted release of solvent vapor is avoided by sealing possible leaks and optimizing the load of the condenser as well as by installing a secondary condenser after the pump.

After placing the samples in the rack, the vacuum cover has to be closed immediately and the evaporation process can be started. When closing the preheated vacuum cover, make sure to avoid skin burns. If necessary, use protective gloves.

5.5 Cross-Contamination and Low Analyte Recovery Rates

Cross-contamination is the transport of analyte from one sample to another by an evaporation condensation process. Cross-contamination is most likely for highly volatile compounds that can move from one glass to the next. Here we refer to a best@buchi publication showing that with the Syncore®, parallel evaporation can be performed without cross-contamination.[9]

In addition it is important to avoid condensation of the analytes in the vacuum cover that might drop into another glass vessel by the rotation of the vacuum cover.

5.6 Stop Procedure

After the evaporation process has finished, it is crucial to stop the procedure immediately, especially if the sample has been evaporated to dryness. After evaporation to dryness, the sample is no longer cooled by the loss of evaporation energy. The sample sticks to the glass wall of the sample vessel and is directly exposed to the temperature of the heating plate. This results in an immediate increase of the sample temperature at the end of the evaporation process and a possible denaturation of the sample. The Syncore® Analyst overcomes this problem by concentrating the sample in a cooled appendix (see section 2.4.2).

Caution: An abrupt release of the vacuum can lead to re-condensation of the solvent in the sample vessel and in the worst case to cross-contamination. Gentle venting is recommended. As a guideline, the vacuum should be released by programming a pressure gradient from the final pressure to ambient pressure taking 1-5 minutes.

5.7 Cleaning Procedure

After each evaporation run, the glassware has to be scrupulously cleaned. This guarantees good recoveries and measuring precision without cross-contamination from earlier experiments. Because analytes tend to adsorb onto glass surfaces contaminated with organic impurities, the cleaning effect is considerably improved by employing alkaline cleaners. For environmental analyses it is moreover recommended to deactivate the glassware in the oven at 450 °C.[9]

To clean the glass plate of the vacuum cover, it has to be removed by releasing the two clamping screws. Use a sponge dampened in alcohol to clean the coated plate of the vacuum cover. Caution: The EMATAL coating must never be damaged with hard brushes

[9] J. Cavegn, S. Cleres, R. Hartmann, N. Schafroth, best@buchi 54, 2009.

or other hard parts. After cleaning, it is essential to dry the vacuum cover and its heating system to prevent any short circuit. The sealing discs can be cleaned with a mild detergent in water or in alcohol. If they are severely contaminated, we recommend replacing the sealing discs with new ones.

6 Applications – Evaporation of Pure Solvents

In this chapter, applications and tables are presented that will help the user to optimize his own process. The presented settings, as for example gradients to evaporate pure solvents, can be taken as a good starting point for your own application. Depending on the user's conditions and the solutions to be concentrated or evaporated, the specified parameters may be further optimized and adapted. For example, to lower the volume that is collected in the appendix of a Syncore® Analyst it is recommended to lower the final pressure. In addition, the evaporation efficiency will change when solutes are dissolved in the solvents.

6.1 Solvent Table and Classification

Table 6. Boiling point, enthalpy of vaporization, density, classification and constant b of common solvents.

Solvent	Boiling point	Enthalpy of vaporization		Density [g/ml]	Classification*	Constant b
		[J/ml]	[J/g]			
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
Chlorobenzene	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
Dimethylsulfoxide (DMSO)	189 °C	759	690	1.100	High-boiling	0.200
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

* Low-boiling solvents have boiling points below 100 °C. Medium-boiling solvents boil between 100-150 °C and high-boiling solvents above 150 °C

6.2 Calculation of Boiling Point as a Function of the Applied Pressure

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

Tp [K] is the temperature of the boiling point at the pressure *p* [mbar], *Tbp* [K] the boiling point under standard conditions, and *b* the solvent specific constant.

The above equation holds only for pure solvents; solutes and impurities alter the boiling point of a pure solvent.

6.3 Pressure – Boiling Point and Temperature Table

Table 7. Pressure requirements for a given boiling point for common solvents.

Solvent	bp. 20 °C	bp. 30 °C	bp. 40 °C	bp. 50 °C	bp. 60 °C
Acetic acid	15	26	44	71	113
Acetone	239	370	556	815	atmospheric
Acetonitrile (MeCN)	83	133	208	315	465
Benzene	98	155	236	352	511
n-Butanol	7	14	25	44	76
Chlorbenzene	13	22	36	56	86
Chloroform	207	318	474	689	atmospheric
Cyclohexane	99	154	234	347	501
Cyclopentane	445	702	atmospheric	atmospheric	atmospheric
Dichloromethane (DCM)	451	685	atmospheric	atmospheric	atmospheric
Diethyl ether	562	838	atmospheric	atmospheric	atmospheric
Dimethylformamide (DMF)*	5	3	14	23	37
1,4-Dioxane	42	68	108	165	246
Ethanol	58	102	167	289	463
Ethyl acetate	95	153	240	366	544
Formic acid	42	68	108	165	246
n-Hexane	145	223	360	490	701
Isopropanol (IPA)	43	78	136	231	378
Methanol	122	206	385	534	824
Pentane	563	819	atmospheric	atmospheric	atmospheric
n-Propanol	20	33	53	83	126
Tetrahydrofuran (THF)	154	244	374	560	817
Toluene	29	48	76	118	177
Water	23	42	72	120	194
Xylene (mixture of isomers)	9	15	25	40	63

6.4 Evaporation of Low-Boiling Solvents

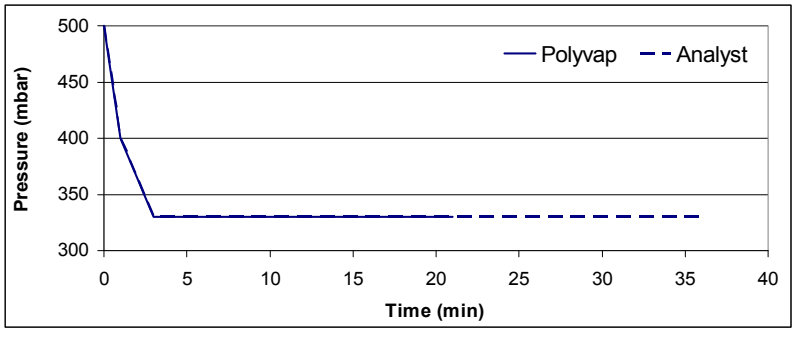
6.4.1 Solvent: Acetone

Solvent Information		
Solvent	Acetone	≥99.0 %, Fluka
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	56 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm
Collection vessel	Cooled with ice bath	Cooled with ice bath

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	500 mbar	500 mbar
Ramp	500-400 mbar in 1 min 400-330 mbar in 2 min	500-400 mbar in 1 min 400-330 mbar in 2 min
Constant	330 mbar for 33 min	330 mbar for 18 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	36 min
Rate overall	1.2 l/h
Rate per position	98 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	21 min
Rate overall	2.1 l/h
Rate per position	171 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	685 ml (97 %)	694 ml (96 %)
Solvent in Appendix	1.2 ml	-
Solvent recovery after secondary condenser	2 ml	2 ml

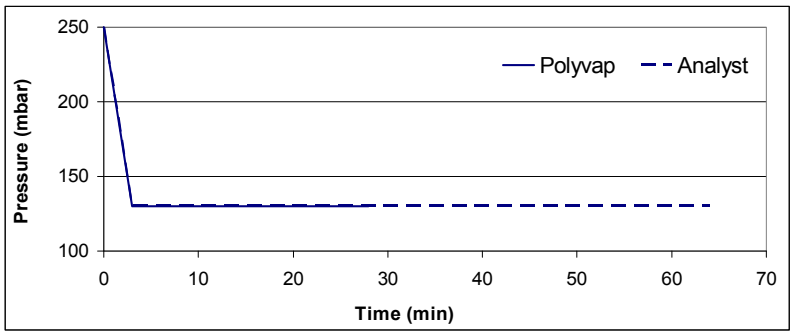
6.4.2 Solvent: Acetonitrile (MeCN)

Solvent Information		
Solvent	Acetonitrile	≥99.9 %, Sigma-Aldrich
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	82 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm
Collection vessel	Cooled with ice bath	Cooled with ice bath

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	250 mbar	250 mbar
Ramp	250-130 mbar in 3 min	250-130 mbar in 3 min
Constant	130 mbar for 61 min	130 mbar in 25 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	64 min
Rate overall	0.7 l/h
Rate per position	55 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	28 min
Rate overall	1.5 l/h
Rate per position	128 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	688 ml (97 %)	718 ml (>99 %)
Solvent in Appendix	1.1 ml	
Solvent recovery after secondary condenser	<1 ml	<1 ml

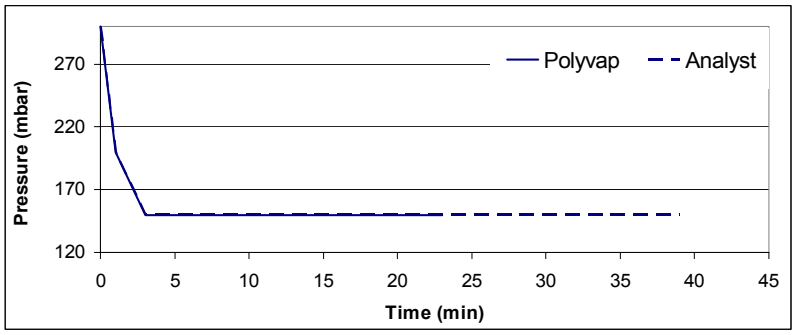
6.4.3 Solvent: Cyclohexane

Solvent Information		
Solvent	Cyclohexane	≥99.9 %, Scharlau
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	81 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm
Collection vessel	Cooled with ice bath	Cooled with ice bath

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	300 mbar	300 mbar
Ramp	300-200mbar in 1 min 200-150 mbar in 2 min	300-200 mbar in 1 min 200-150 mbar in 2 min
Constant	150 mbar in 36 min	150 mbar in 20 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	39 min
Rate overall	1.1 l/h
Rate per position	90 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	23 min
Rate overall	1.9 l/h
Rate per position	156 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	692 ml (98 %)	718 ml (>99 %)
Remaining in appendix	1.4 ml	-
Solvent recovery after secondary condenser	<1 ml	<1 ml

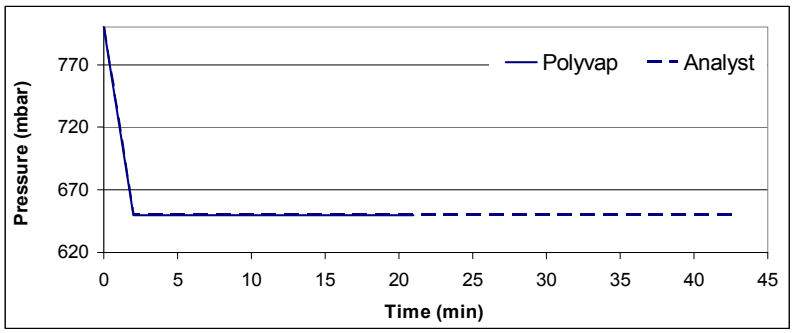
6.4.4 Solvent: Dichloromethane

Solvent Information		
Solvent	Dichloromethane	≥99.0 %, Sigma-Aldrich
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	40 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm
Collection vessel	Cooled with ice bath	Cooled with ice bath

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	800 mbar	800 mbar
Ramp	800-650 mbar in 2 min	800-650 mbar in 2 min
Constant	650 mbar 41 min	650 mbar 19 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	43 min
Rate overall	1.0 l/h
Rate per position	82 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	21 min
Rate overall	2.1 l/h
Rate per position	171 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	690 ml (98 %)	702 ml (98 %)
Remaining in appendix	1.25 ml	-
Solvent recovery after secondary condenser	< 1 ml	< 1 ml

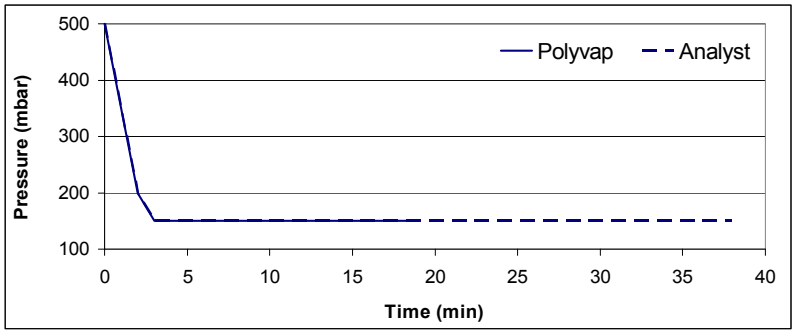
6.4.5 Solvent: Ethyl Acetate

Solvent Information		
Solvent	Ethyl Acetate	≥99.5 %, Merck
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	77 °C
	Desired boiling point	30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	500 mbar	500 mbar
Ramp	500-200 mbar in 2 min	500-200 mbar in 2 min
	200-150 mbar in 1 min	200-150 mbar in 1 min
Constant	150 mbar 35 min	150 mbar 16 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	38 min
Rate overall	1.1 l/h
Rate per position	93 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	19 min
Rate overall	2.3 l/h
Rate per position	189 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	680 ml (96 %)	709 ml (98 %)
Remaining in appendix	1.25 ml	-
Solvent recovery after secondary condenser	< 1 ml	< 1 ml

6.5 Evaporation of Medium-Boiling Solvents

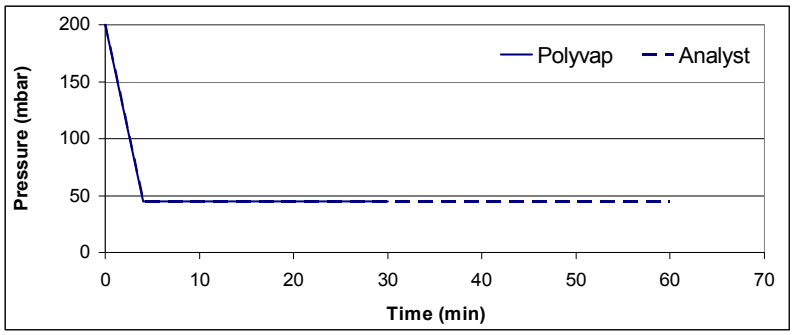
6.5.1 Solvent: Toluene

Solvent Information		
Solvent	Toluene	≥99.8 %, VWR
	Evaporated volume	720 ml, 60 ml/position
	Category	Medium-boiling
	Boiling point	111 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)	Water/glycol 70:30 % (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	200 mbar	200 mbar
Ramp	200-45 mbar in 4 min	200-45 mbar in 4 min
Constant	45 mbar in 56 min	45 mbar in 26 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	60 min
Rate overall	0.7 l/h
Rate per position	58 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	30 min
Rate overall	1.4 l/h
Rate per position	120 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	696 ml (> 99 %)	716 ml (> 99 %)
Remaining in appendix	1.6 ml	-
Solvent recovery after secondary condenser	<1 ml	<1 ml

6.6 Evaporation of High-Boiling Solvents

6.6.1 Solvent: Dimethylsulfoxide, DMSO

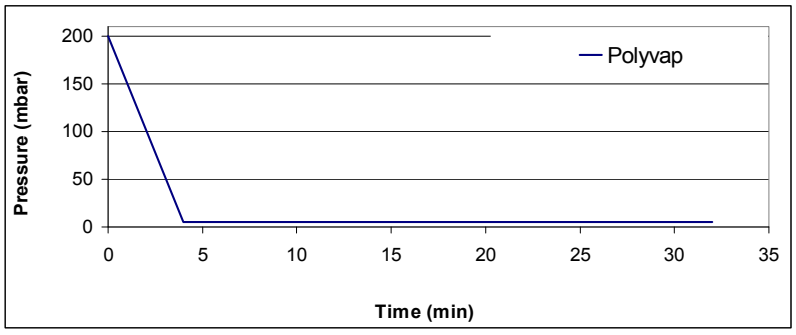
Solvent Information		
Solvent	Dimethylsulfoxide	≥99.5 %, Sigma-Aldrich
	Evaporated volume	600 ml, 50 ml/position
	Category	High-Boiling
	Boiling point	189 °C
	Desired boiling point	~40 °C

Syncore® System Configuration	
Parameter	**Polyvap**
Configuration	R-12 Rack and Cover
Vessel Type	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-710, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30 % (v/v)
Heat transfer medium	No heat transfer medium
Eccentricity	5 mm
Balance	25 mm

Settings	
Platform temp.	140 °C
Cover temp.	70 °C
Cooling temp.	20 °C
Orbital movement	300 rpm

Pressure Gradient	
	Polyvap
Starting point	200 mbar
Ramp	200-5 mbar in 4 min
Constant	5 mbar for 28 min

Evaporation performance	
Polyvap	
Positions	12
Total volume	600 ml
Time	32 min
Rate overall	1.1 l/h
Rate per position	94 ml/h



Results	Polyvap
Solvent recovery after main condenser	575 ml (96 %)
Solvent recovery after secondary condenser	< 1 ml, solvent droplets remain in vacuum cover
Solvent recovery after main condenser	575 ml (96 %)

6.7 Evaporation of Polar Protic Solvents

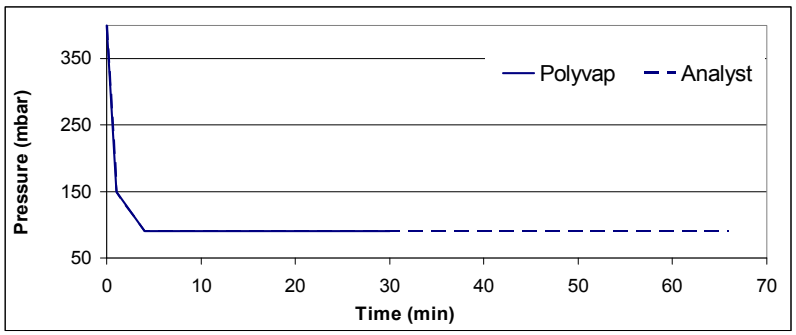
6.7.1 Solvent: Ethanol

Solvent Information		
Solvent	Ethanol	≥99.9 %, Merck
	Evaporated volume	720 ml, 60 ml/position
	Category	Low-boiling
	Boiling point	78 °C
	Desired boiling point	~30 °C

Syncore® System Configuration		
Parameter	**Analyst**	**Polyvap**
Configuration	R-12 Rack and Cover	R-12 Rack and Cover
Vessel Type	Order no. 046071 1 ml Appendix	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30% (v/v)	Water/glycol 70:30% (v/v)
Heat transfer medium	25 ml H_2O/position	7 ml H_2O/position
Eccentricity	4 mm	4 mm
Balance	25 mm	25 mm

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient		
	Analyst	**Polyvap**
Starting point	400 mbar	400 mbar
Ramp	400-150 mbar in 1 min 150-90 mbar in 3 min	400-150 mbar in 1 min 150-90 mbar in 3 min
Constant	90 mbar 62 min	90 mbar 26 min



Evaporation performance	
Analyst	
Positions	12
Total volume	720 ml
Time	66 min
Rate overall	0.6 l/h
Rate per position	53 ml/h
Polyvap	
Positions	12
Total volume	720 ml
Time	30 min
Rate overall	1.4 l/h
Rate per position	120 ml/h

Results	Analyst	Polyvap
Solvent recovery after main condenser	670 ml (95 %)	713 ml (>99 %)
Remaining in appendix	1.2 ml	
Solvent recovery after secondary condenser	< 1 ml	< 1 ml

7 Evaporation of Solvent Mixtures

7.1 Mixture of Acetonitrile and Water (50:50 % Vol.)

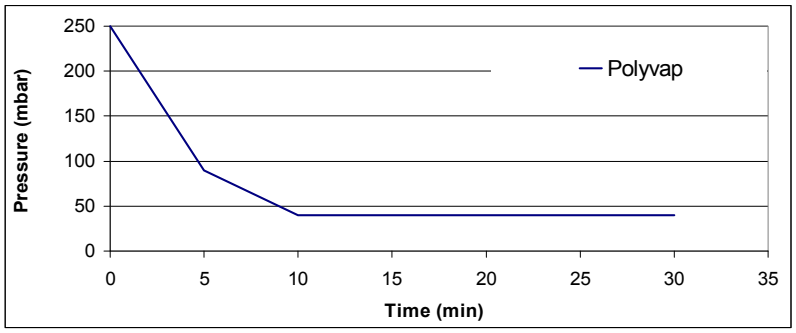
Solvent Information		
Solvent	Acetonitrile	≥99.9 %, Sigma-Aldrich
	Water	Distilled water
	Evaporated volume	240 ml, 20 ml/position
	Category	Solvent Mixture
	Desired boiling point	~30°C

Syncore® System Configuration	
Parameter	**Polyvap**
Configuration	R-12 Rack and Cover
Vessel Type	Order no. 040907
Options	Vacuum controller V-855 (firmware 3.03) Vacuum pump V-700, Recirculation chiller
Cooling medium Cooling capacity at 15°C: 1400 W	Water/glycol 70:30% (v/v)
Heat transfer medium	7 ml H_2O/position
Eccentricity	5 mm
Balance	25 mm
Collection vessel	Cooled with ice-bath

Settings	
Platform temp.	55 °C
Cover temp.	50 °C
Cooling temp.	10 °C
Orbital movement	300 rpm

Pressure Gradient	
	Polyvap
Starting point	250 mbar
Ramp	250-90 mbar in 5 min 90-40 mbar in 5 min
Constant	40 mbar for 20 min

Evaporation performance	
Polyvap	
Positions	12
Total volume	240 ml
Time	30 min
Rate overall	0.5 l/h
Rate per position	40 ml/h



Results	Polyvap
Solvent recovery after main condenser	204 ml (85 %)
Solvent recovery after secondary condenser	18 ml

8 Application SPE Module

Fundamentals of solid-phase extraction were introduced in chapter 2.3. Here an application using SPE is presented, revealing the composition of the red color of the well known Campari drink.[10]

8.1 Isolation of Food Dyes using Syncore with SPE Module

Equipment

- Syncore® Analyst R-12 equipped with SPE Advanced Module
- Glassware: BUCHI 12 Analyst tube, 1.0 ml residual volume
- Vacuum: V-700 vacuum pump with V-850/V-855 controller

If subsequent concentration to a defined residual volume is to be done, an appropriate cooling medium (temperature < 20 °C) is required. This could be taken from tap water, an in-house cooling installation or a chiller (e.g. BUCHI F-108 Chiller).

Materials

- Silicycle Siliaprep C18 SPE cartridge (SPE-R30130B-06P)
- Drink colored with Azorubine (E122), Tartrazine (E102) and Brilliant Blue FCF (E133) e.g. Campari
- Pasteur pipettes

Chemical structure of colorants Azorubine, Tartrazine and Brilliant Blue FCF are displayed in Figure 19.

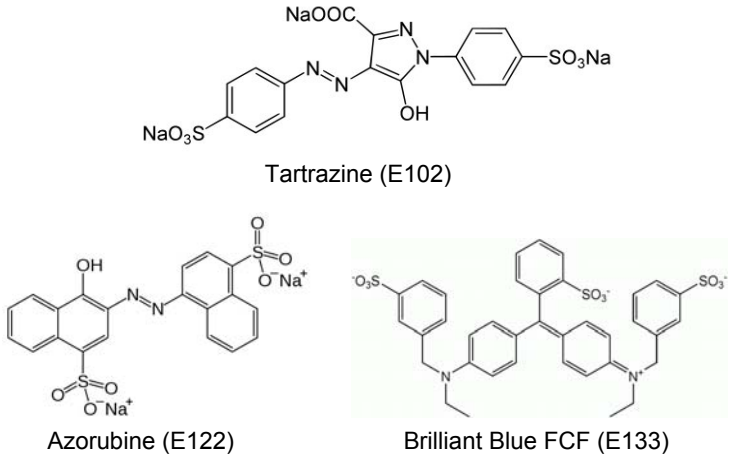


Figure 19. Colorants Azorubine, Tartrazine and Brilliant Blue FCF

[10] D. Rütti, Büchi Demo Application, Isolation of food dyes using Syncore® with SPE module, 2011.

Procedure

Fill approx. 15 ml water in each position of the rack and install 12 empty Syncore® Analyst R-12 vessels. Close cover and tighten with screws.

Mixture of colorants

Sample preparation: Not necessary

SPE procedure:

- Equip SPE module with C18 cartridges, turn all stopcocks to the middle position, apply 800-mbar vacuum (Figure 20, left)
- Equilibrate the cartridge with 5 ml distilled water containing 10 drops of ethanol (5 ml is the reservoir volume of the cartridge)
- Turn stopcock to the right position and transfer the liquid into the waste vessel, turn back to stop
- Pipette 5 ml of the drink into the cartridge. If a non-alcoholic drink is used, add 10 drops of ethanol and mix with pipette (Figure 20,middle)
- Transfer the liquid to the waste vessel (Figure 20, right)
- Wash with 5 ml distilled water containing 10 drops of ethanol to the waste
- Elute with 5 ml ethanol to Syncore® vessel by turning the stopcock left

Figure 20. C18 cartridge (left); Sample (middle); loading the sample onto solid phase (right)

Campari and Vodka

Sample preparation: not necessary

SPE procedure:

- Equip SPE module with C18 cartridges, turn all stopcocks to the middle position, apply vacuum of 800 mbar
- Equilibrate the cartridge with 5 ml water containing 10 drops of vodka (5 ml is the reservoir volume of the cartridge)
- Turn stopcock to the right position and transfer the liquid into the waste vessel, turn back to stop
- Pipette 5 ml of Campari into the cartridge
- Transfer the liquid into the waste vessel
- Wash twice with 5 ml tap water containing 10 drops of vodka to the waste
- Elute with 5 ml vodka to Syncore® vessel by turning the stopcock to the left
- Evaporate to the defined residue

Directly after the SPE procedure, the eluted green-yellow fraction can be concentrated to a defined residual volume with the Syncore® Analyst without sample transfer using the parameters shown in Table 8.

Table 8. Parameters for the evaporation of after SPE procedure.

Vacum	90 mbar
Platform	65 °C
Cover	50 °C
Vortex speed	200 rpm
Chiller (if used)	5 °C
Time:	10-15 min

Results

Both methods yield the same result for separation of dyes (Figure 21). Red azorubine was not adsorbed by the cartridge and was washed to the waste vessel (right). The other two colors that were eluted to the receiving vessel stained the collected fraction green-yellow (left).

After evaporation the green-yellow fraction is concentrated into the appendix.

Figure 21. Separation of colors using SPE module of Syncore® .

9 Appendix

9.1 Chemical Resistance of Materials in Contact with Solvents to be Evaporated

Table 9. Polymer material in contact with vapor from solvents.

EPDM	Ethylenepropylenedimonomer	O-Ring
PE	Polyethylene	Sealing discs
PEEK	Polyetheretherketone	Screw caps
PFA	Perfluoroalkoxy	Vacuum hose and vacuum cover coating
PTFE	Polytetrafluoroethylene	Sealing discs
EMATAL	Al/Ti-Oxide Coating	Vacuum cover

Table 10. Chemical resistance of polymers in contact with various solvents.

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

[11] Operation Manual, Syncore® Accessories
[12] Semadeni, Chemical resistance table for polymers, www.semadeni.com.
[13] Eugen Seitz AG, Technical Information

	EPDM*[11]	PE*[11]	PFA**[12]	PEEK*[11]	PTFE**[12]	EMATAL*[13]
Triethylamine	C	A	-	A	-	-
Trichloroethane	D	C	-	A	-	A
Trichloroacetic acid	B	A	A	A	A	-
Vinylidene chloride	D	D	-	A	-	-
Aq. HBr, sat.	B	C	A	C	A	D
Aq. HCl, sat.	A	A	A	B	A	D
Aq. ammonia solution	A	A	A	A	A	A
Aqueous caustic soda	A	A	A	A	A	D
Aqueous nitric acid	B	B	A	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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