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for Environmental Analysis

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Syncore® Analyst allows for efficient and parallel concentration of liquid samples in less than one hour and minimizes the environmental impact by solvent recovery. These advantages make the Syncore® Analyst the system of choice for concentration in trace analysis. A best practice guideline is presented below.

Introduction

Harmful substances are often very difficult to detect since they are present in concentrations within the trace and ultra-trace range. In conclusion a sensitive detection is essential. Lower detection limits are achieved by a pre-concentration of the sample.

The automated parallel evaporation system Syncore® Analyst is designed for gentle concentration of 4, 6 or 12 samples with working volumes of 10 to 500 ml down to pre-defined residual volumes of 0.3, 1.0, or 3.0 ml by means of heating under vacuum. The key feature of the Syncore® Analyst is an integrated cooling zone which stores the concentrated sample in a cooled environment. High recovery rates are achieved with the ultimate Flushback Module that gently rinses the glass wall during operation with the solvent being evaporated. A variable orbital movement prevents evaporation retardation. The cover is constructed from inert materials to avoid the leaching of contaminants. The risk of cross-contamination is eliminated by an individual sample sealing system and separated vacuum channels. The vacuum cover's integrated heater makes it possible to evaporate even high boiling point solvents. The evaporated solvents are recovered by a condenser.

Programmable conditions (vacuum, temperature, orbital movement) allow a reproducible concentration without supervision and with high precision. Loss of volatile compounds during

concentration can be almost eliminated by the clever combination of a vacuum controller, a Flushback Module and parameter adjustments of temperature and orbital movement.

This guideline serves to enhance the performance of the Syncore® Analyst. Conditions for best operation are shown and the impact of the parameters is discussed.

Installation

Proper installation of the Syncore® Analyst is essential for efficient and reproducible performance. Both a



Fig. 1: Parallel evaporation system to a pre-defined volume: Syncore® Analyst R-12 from Buchi for 12 samples equipped with Flushback Module and a condenser.



Fig.2: Buchi Vacuum Pump V-700 with Vacuum Controller V-855 and a secondary condenser.

tight system and uniform cooling are necessary.

Instrumentation

Buchi's parallel evaporation system consists of the Syncore® Analyst, the Vacuum Pump V 700, the Vacuum Controller V-855, and the Recirculating Chiller B-740/14 cooled with ethanol+water (40+60).

Requirements at the installation site

The Syncore® Analyst system can be placed outside the fume hood due to the fact that the solvent is recovered by a condenser with receiving flask.

Connection to vacuum pump and controller

Each sample glass is individual sealed by the vacuum cover of the Syncore® Analyst which is connected to the primary condenser, which collects the distillate in a receiving flask (Fig. 3). The condenser is connected to both the controller and the pump by means of a T-piece or a Wouff bottle (47170).

Connection to cooling medium

To ensure efficient and uniform cooling, it is recommended to attach a distribution piece (three-way stopcock, Buchi 37742) to the outlet of the Recirculating Chiller B 740/14. One outlet is connected sequentially to the secondary condenser of the vacuum pump followed by the primary condenser of the Syncore® Analyst. The other outlet is connected parallel to the residual cooling zone of the sample rack and the Flushback Module by using T-pieces. An installation diagram is shown in Figure 3.

Operation

After an installation, the system should be tested on tightness, homogeneous temperature transfer between the heating block and the rack and on cooling efficiency.

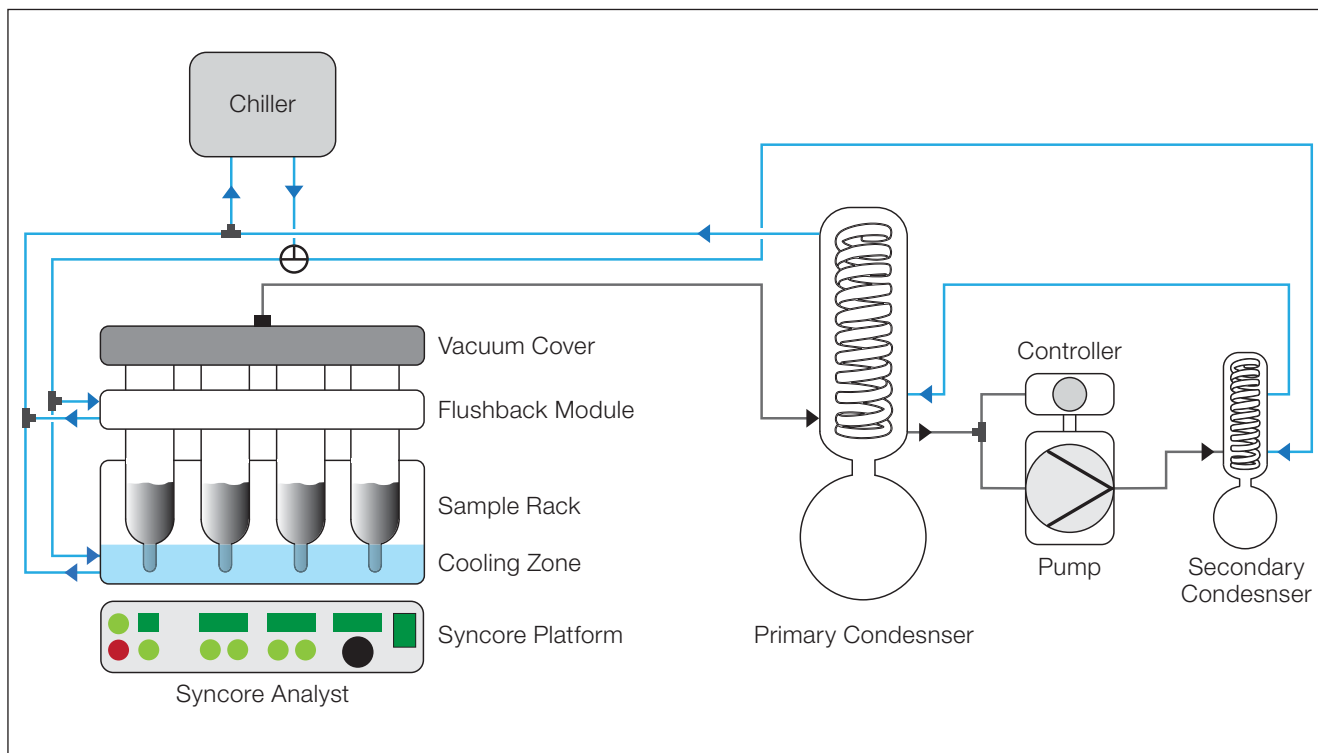


Fig.3: Installation of Syncore® Analyst System with Recirculating Chiller B 740/14.

Tightness test procedure

The tightness of the Syncore® Analyst System is tested in a closed, empty and dry system by stopping evacuation when the set vacuum is reached. If the values do not comply with Table 1, the tightness of the system should be verified.

Tab.1: Expected values for the tightness of a dry system

Syncore Analyst System	Set vacuum	≤ 30 mbar
	Pressure increase	≤ 3 mbar/min
Pump	End vacuum	< 15 mbar

Temperature test procedure

Heating: To ensure uniform heat transfer, propylene glycol is applied between the heating plate and the rack. The rack positions are filled with water (dead volume between rack and vessel) and the water temperature should be measured for each position. The temperature should be reached within 10 minutes and the maximum deviation between defined and measured temperature should be 5°C . The desired temperature is reached faster by applying an orbital movement (e.g. 100 RPM).

Cooling: The evaporation efficiency of the Syncore® Analyst System can be improved by using a powerful recirculation chiller. The recommended chiller for the Syncore® Analyst System is the Recirculating Chiller B-740/14. It is important that the cooling temperature is reached within a certain time and does not increase significantly during concentration. Thanks to the temperature display of the chiller B-740/14, the time to pre-cool and the increase of temperature during concentration can easily be monitored.

Tab.3: Heating and cooling performance for Syncore® Analyst and Recirculating Chiller B-740/14

Heating	Time to set temperature	30-60 min
	Sample to sample uniformity	$\pm 2^{\circ}\text{C}$
	Maximal deviation of sample temperature to set value	10°C
Cooling	Time to set temperature	10 min
	Maximal deviation to set value during concentration	$+ 5^{\circ}\text{C}$

System performance test

A good performance of the Syncore® Analyst System results in uniform residual volumes.

Table 4 shows a method to test the system performance. In-house methods can be used instead. However, the amount of residual volume depends on the conditions. Outlying values often result from a poorly sealed vacuum cover. Other reasons are leaks or an inhomogeneous heat transfer.

Performance

Cleaning of the glass equipment

Analytes tend to adsorb on glass surfaces contaminated by organic impurities. Therefore, proper glass cleaning is crucial to obtain good recoveries and precision. The cleaning procedure can be improved considerably by using alkaline cleaners (e.g. Deconex 20NS, Borer Chemie, Switzerland) instead of organic surface-active substances. For environmental analysis, it is further recommended to deactivate the glass equipment at 450°C .

System Preparation

The rack positions must be filled with water (only dead volume) to ensure good heat transfer between the rack and sample vessels. A uniform and reproducible concentration is further

achieved by preheating the rack and vacuum cover for 30-60 minutes. Pre-cooling is usually started 10 minutes before pre-heating ends. An empty receiving flask of appropriate size is connected to the condenser.

Tab.4: Conditions for system performance test

Rack	R-12		
Vessel	200 ml / 1 ml appendix		
Solvent	<i>n</i> -hexane		
Sample amount	20 ml		
Vacuum gradient (60 min)			
Step	Start [mbar]	End [mbar]	Time [min]
1	700	320	4
2	320	240	5
3	240	180	30
4	180	180	10
5	180	1000	10
Orbital movement	170 RPM		
Heating block temperature	50°C		
Vacuum cover temperature	50°C		
Chiller temperature	5°C		
Residual volume	~1.45 ± 0.15 ml		

Temperature

Both the rack temperature and the appendix temperature have an impact on the sample temperature. At the beginning of the concentration the sample temperature is dominated by the rack temperature. Due to the cooled appendix the sample temperature is lowered during concentration until the cooling temperature, and therewith, the residual volume is reached. In conclusion the heating and cooling temperature should differ by at least 30°C to guarantee that a residual volume remains in the vessel. For instance, if the conditions in Table 4 are applied with 18°C instead of 5°C, the residual

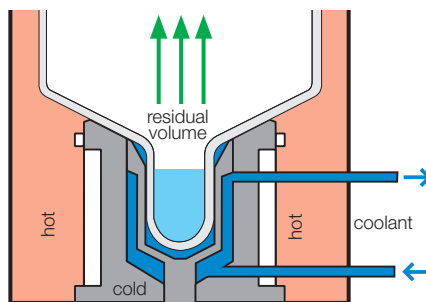


Fig.4: Syncore® Analyst with the locally cooled appendix of the sample vessel preventing evaporation to dryness.

volume is reduced to 0.7 ± 0.15 ml. To avoid decomposition, a heating temperature of 50°C is advisable. This corresponds to a maximum sample temperature of 45°C. Lower temperatures will slow down evaporation.

Vacuum conditions and evaporation rate

A vacuum is applied in order to decrease the boiling point temperature. Optimum vacuum conditions can be found in the vacuum controller's solvent library. Additionally, more theoretical background is published in the Buchi Laboratory Assistant [1].

An efficient concentration is performed in 30-60 minutes. Best results are achieved with a moderate evaporation rate generated by a vacuum gradient. Optimized initial-vacuum conditions show a condensation within the first 10 minutes, respectively 5 minutes for a fast evaporation. Concentration can be accelerated by applying a steep gradient or a lower end pressure.

To prevent a concentration to dryness it is recommended to choose an end vacuum pressure which results in a boiling point of the solvent at least 20°C above the cooling temperature. For example, the end-vacuum for *n*-hexane should be set to at least 195 mbar and the initial-vacuum to 436 mbar, if a cooling temperature of 5°C and a maximum sample temperature of 45°C (corresponds to a set temperature of 50°C) are applied. An end vacuum pressure of 77 mbar would result in a concentration to dryness.

Highly volatile analytes (e.g. naphthalene), in particular, require a rather slow but smooth evaporation, since a fast evaporation rate tends to carry along

volatile analytes. However, this is not the case for non-volatiles (e.g. benzo[a]pyrene).

Tab.6: Analyte and solvent recovery after concentration, measured by GC/FID

Time to concentration	30 min	60 min
Naphthalene	80 ± 6 %	94 ± 3 %
Benzo[a]pyrene	99 ± 3 %	99 ± 2 %

Table 6 shows the recoveries obtained after a concentration of 30 minutes and 60 minutes, respectively, using the conditions shown in Table 4. The vacuum gradient for the 30 minutes was adjusted to a steeper profile (700 to 250 mbar in 3 min, from 250 to 180 mbar in 5 min, hold at 180 mbar for 20 min, from 180 to 1000 mbar in 2 min).

An optimal vacuum gradient shows not only high analyte recoveries but also high solvent recoveries of about 95%, preferably collected in the primary condenser. The faster the evaporation, the more solvent will be collected in the secondary condenser (post-pump). To prevent damaging the pump, the gradient should be optimized in such a way, that the solvent passing through the pump is kept at a minimum. In conclusion, the condenser is operated at a maximum of three-quarter capacity. For the conditions shown in Table 4, a solvent recovery of 95% was obtained (all solvent was collected in the primary condenser receiving flask).

Orbital movement

The orbital movement has a considerable effect on the evaporation rate and the final volume. The residual volume is reduced from 1.45 ml to 1.15 ml by changing the conditions of Table 4 from 170 RPM to 300 RPM, and condensation is completed 10 minutes earlier.

Although concentration is accelerated, a high orbital movement is not only beneficial. An increase of the orbital movement implements an extension of the glass surface being in contact with the sample. Particularly oily and greasy substances tend to stick on the glass wall. Figure 5 shows the difference between the 170 RPM and



Fig.5: Grease sticking to the glass wall obtained from a 170 RPM (left) and a 300 RPM (right) concentration.

the 300 RPM evaporation of 0.2 g hydrocarbon grease (Blasolube 301, Blaser Swissslube) without using a Flushback Module. The 300 RPM evaporation requires a better glass rinsing, respectively a strong need of a Flushback Module.

Flushback Module

The main function of the Flushback Module is flushing the glass wall, which in turn allows a fast and quantitative transfer. Figure 6 shows a concentrated sample of 0.5 g diesel with and without Flushback Module, using the conditions listed in Table 4.



Fig.6: Oil sticking to the glass wall after a concentration with (left) and without (right) Flushback Module.

Cross-contamination

Cross-contamination between samples of high and low concentrations is one of the critical points in trace analysis. This particularly applies to simultaneous sample concentration. Reasons can be sample transfer by vapor phase or contaminated glass surfaces.

Due to the sophisticated design of the vacuum cover, the individual sealing system of each sample glass and the possibility to clean the glass vessels in dish washers, the risk of cross-contamination is mostly insignificant. At a naphthalene sample concentration of up to 800 µg the detected level of

naphthalene in blank positions is below 0.1 % of the sample concentration. In conclusion, samples of high and low concentration can be concentrated simultaneously.

Conclusions

The tightly controlled Syncore® Analyst evaporation process allows for excellent recovery rates and precision. In addition, adjustable parameters (vacuum profile, orbital movement, Flushback Module, cooling and heating temperature) facilitate to match requirements within a reasonable time (30 – 60 minutes). The loss of volatile analytes during extract concentration can be almost completely eliminated when a smooth concentration is performed. On the other hand, a fast concentration is recommended for non-volatile analytes.

Thanks to the ultimate Flushback Module, labor intensive glass wall rinsing after the concentration step is kept at a minimum. Individual sample sealing and separated vapor channels eliminate the danger of cross-contamination. Consequently, samples of high and low concentration can be concentrated simultaneously.

Due to its high solvent recovery ($\geq 95\%$), the Syncore® Analyst can be operated outside a fume hood. Furthermore, laboratory contamination is minimized because of low emissions.

References

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