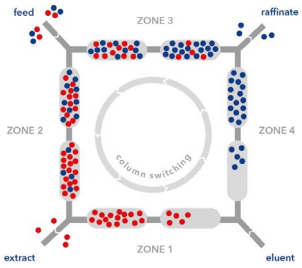
Science Together

KNAUER

Simultaneous sampling of two product streams



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SUMMARY

During preparative HPLC method development, frequent sampling is essential to evaluate the process and the efficiency of the parameter adaptations. This gets even more crucial during the development of complex simulated moving bed (SMB) methods. During the optimisation of an SMB method to separate caffeine and paracetamol, an 8-port two-position valve was implemented to collect simultaneously samples from extract and raffinate. This improves the sampling process considerably.

INTRODUCTION

In the development process of an SMB method, it is necessary to determine the concentration of the target substances in extract and raffinate outlets. The development of an SMB process requires extensive demands on process design and control due to the higher complexity compared to a classical batch separation. One possibility to monitor the SMB process is the collection of the product fractions for one switch or a whole cycle to determine the target substance distribution. The easiest and most frequent way is to take these samples by removing the outlet tubing from the corresponding bottles and switching them to the sample vessels. Characterized by a complicated handling, this method causes a delayed sampling by the large dead volumes of the outlet tubing. To avoid this disadvantage, the classical solution for sampling of two product streams is the usage of two 3-way valves or of two 6-port two-position valves. A new and smarter way to collect the sample offers the KNAUER 8-port two-position valve.

Additional Information

Simultaneous sampling of two product streams

RESULTS

The 8-port 2-position sampling valve was applicated for the separation of caffeine and paracetamol with the AZURA Lab SMB in a 2:2:2:2 configuration **(Fig. 1)**.

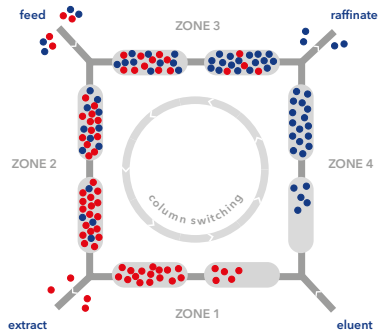

Fig. 1 Operation mode of the SMB.

The integration in the flow path of the sampling is depicted in **Fig. 2** and **Fig. 3**.

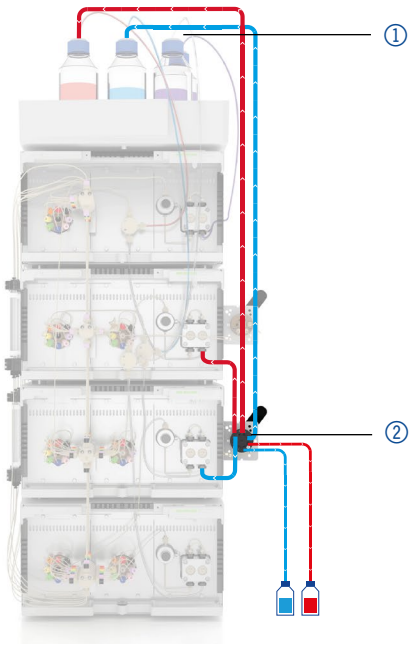

Fig. 2 System setup with the old (1) and new (2) sampling point. The sample extract is shown in red, the sample raffinate in blue.

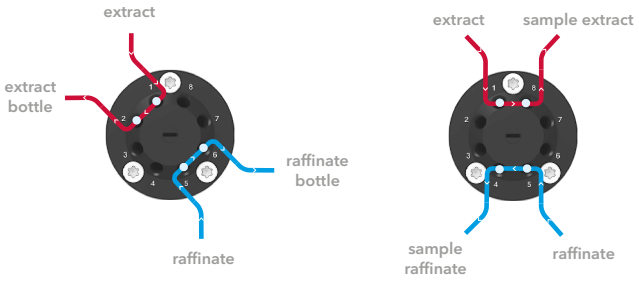

Fig. 3 Operation mode of the sampling valve (8-port 2-position).

The outlet tubing for extract and raffinate are connected to positions 1 respectively 5. By switching the valve, the outlet can be shifted from the bottles (position 2 for extract, position 6 for raffinate) on top of the system to the sample vessels (position 8 for extract, position 4 for raffinate). Extract and raffinate were sampled over the whole process. Samples were taken for each of the first 12 cycles and a whole cycle was collected. An overlay of the chromatograms is shown in **Fig. 4** (raffinate) and **Fig. 5** (extract).

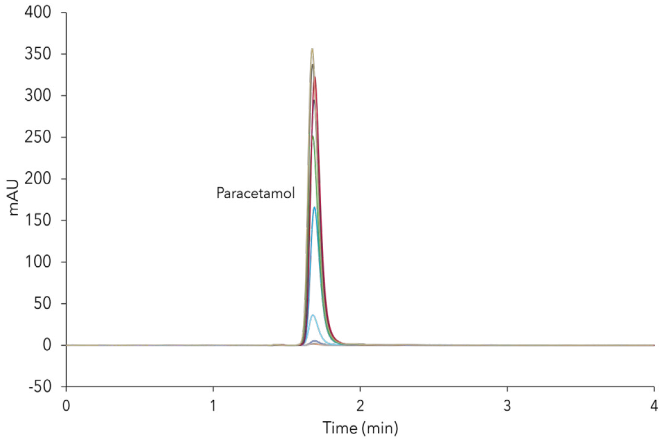

Fig. 4 Increasing concentrations of paracetamol in raffinate during process start-up, samples taken from cycle 1 - 8, 10 and 12. Whole cycles were collected.

RESULTS

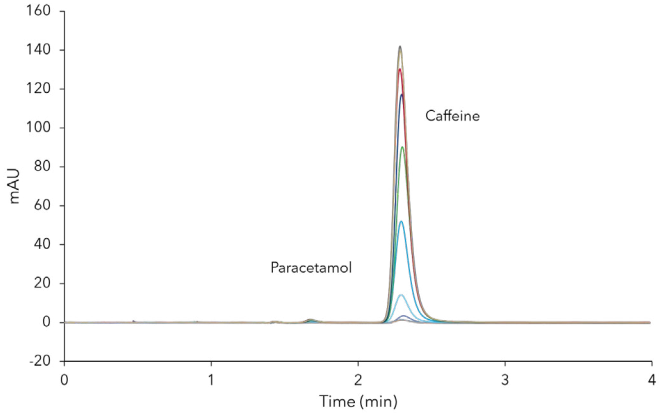

Fig. 5 Increasing concentrations of caffeine in extract during process start-up, samples taken from cycle 1 - 8, 10 and 12. Whole cycles were collected.

The handling is much easier and safer as no tubing must be moved manually from the product vessels. The implementation of the sampling valve minimizes the delay volume and reduces product losses during sampling. Especially in the lab scale, where low flow rates are applicated, a delayed sampling impedes a fast and accurate method development. Therefore, the optimisation of the SMB process can take place significantly more accurate, safer and faster using the sampling valve.

CONCLUSION

The product sampling in the SMB is one smart application for the 8-port two-position valve. The simultaneous sampling of two product streams increases the accuracy and speed of the results. Furthermore, compared to the classical sampling with two valves, a simultaneous sampling of two product streams can be performed with only one valve, reducing cost and space.

MATERIALS AND METHODS

Tab. 1 Method parameters

	Feed (ml/min)	Eluent (ml/min)
In	0.2	4
Column temperature	ambient	
Cycle time	18.8 min	

Tab. 2 System configuration

Instrument	Description	Article No.
AZURA Lab SMB system	SMB, biocompatible system, lab scale	A29100
Manual injection valve	AZURA V 4.1, 8-port two-position valve	AVJ36AE
Mounting bracket	Mounting bracket for AZURA L	A9853
Flow meter	3x M13	A29800
	1x M12	A5394
Column	8x Eurospher II C8 150x8 mm, 100-15, 15 µm	15GE081E2Q
Software	PurityChrom MCC	Included in A29100

RELATED KNAUER APPLICATIONS

VTN0011 – Simulated Moving Bed (SMB) Inline Sampling