Improved analyte recovery with keeper solvents

Syncore Analyst: Evaporation to a defined volume with appendix chiller and keeper solvent.

Polychlorinated biphenyls are suspected endocrine disruptors. They are enriched in the food chain which can result in elevated concentrations in fish and meat [1]. The concentration of some PCBs is the subject of regulations and must be analyzed by reliable methods [2]. Often, PCBs are extracted from a matrix. The obtained extract must be concentrated before analysis. It is then crucial to maintain a high analyte recovery in the concentration procedure.

1. Introduction

The aim of this application note was to demonstrate how the use of a keeper solvent can significantly improve the recovery of analytes during evaporation to a defined volume. The Syncore Analyst can be used to concentrate analyte solutions to a defined volume through the use of a chilled appendix. However, if aggressive distillation conditions are necessary to obtain the desired final volume, analytes may be lost. The addition of a small volume of a second, higher-boiling solvent helps retain the analytes in the chilled appendix [3].

2. Experimental

Table 1: Pressure program during evaporation.

Step No.	Initial pressure (mbar)	Final pressure (mbar)	Step time (min)
1	1000	165	1
2	165	165	35
3	165	1200	3
Total time			39 min

The evaporation experiments were carried out in a Syncore® Analyst with 6 glass tubes with a 0.3 mL appendix. The Syncore® Analyst was equipped with a flusback module, a V-300 vacuum pump with secondary condenser and a recirculating chiller.

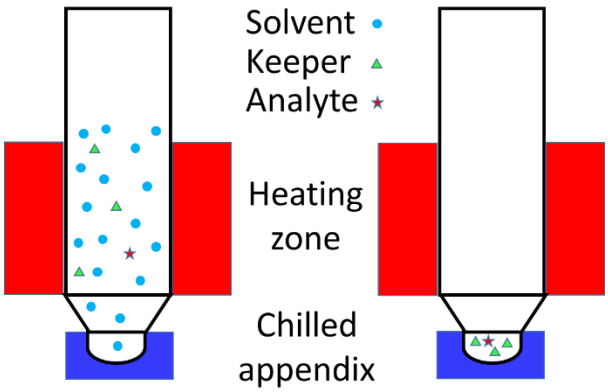

Figure 1: Functional principle of a keeper solvent: while the main solvent is evaporated, the analyte and the keeper are retained in the chilled appendix.

100 mL of hexane and 20 µL of PCB standard were added to each vial. For the experiments with the keeper solvent, an additional 180 µL of nonane were added to the vial. The heating plate was heated to 50 °C and the vaccuum cover to 30 °C. The flushback module was cooled to -5 °C, appendix chiller and condenser to 5 °C. The vacuum was set according to the program in Table 1. The analyte recovery was measured by GC-MS.

3. Results

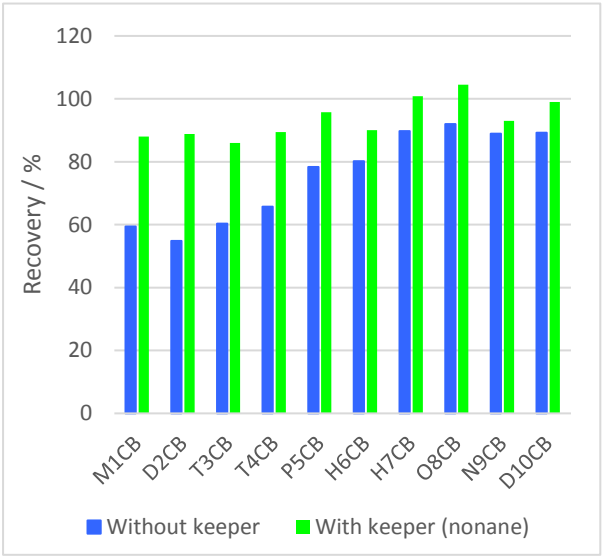

Figure 2: Recoveries for PCBs without (blue) and with nonane keeper.

The analyte recoveries obtained with and without addition of the nonane keeper solvent are shown in Figure 2. The addition of the nonane keeper improves the solvent recovery in all tested cases. The effect is especially pronounced for the less chlorinated PCBs. Without the nonane keeper, poor recoveries of around 60 % are obtained for some of the PCBs. With the use of a nonane keeper, all PCBs are recovered at a level of over 80 %.

4. Conclusion

The use of a nonane keeper significantly improves the recovery of all PCBs, especially the ones with fewer chlorine atoms. Keeper solvents in combination with appendix chilling are a powerful tool for obtaining high recoveries when evaporating solutions of volatile analytes to a predefined volume.

5. References

[1] Leo Nollet (Ed.), Analysis of Endocrine Disrupting Compounds in Food, Blackwell Publishing, Ltd, 2011.

[2] Commission regulation (EC) No 1883/2006.

[3] Łukasz Dąbrowski, Review of use of keepers in solvent evaporation procedure during the environmental sample analysis of some organic pollutants, Trends in Analytical Chemistry, 80 (2016) 507-516.