Abstract

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Title of the diploma thesis:	Setup and characterization of an automated method for salt-assisted dispersive liquid-liquid microextraction using a lab-in-syringe system

Sequential Injection Analysis (SIA) is a technique derived from the Flow Injection Analysis technique. The system generally consists of a computer-controlled syringe pump, a selection valve, and a detector, all connected by inert plastic tubing. It is used to automate laboratory procedures. The "Lab-In-Syringe" technique is a modification of SIA used to carry out parts of the experiment inside the used syringe pump's void. Using a PTFE-coated magnetic-propelled stirring bar inside the syringe allows, for example, to mix homogenously the syringe content or to perform liquid extraction protocols such as dispersive liquid-liquid micro-extraction (DLLME).

In this work, the approach to perform salting-out assisted in-syringe DLLME was explored and evaluated for the first time. Starting with a one-phase system, the analyte was extracted from water into n-propanol. For this, a highly-concentrated solution of magnesium sulfate was used to increase the polarity of the aqueous phase. The high polarity causes the separation of the two normally fully miscible liquids.

Astraphloxin and riboflavin were used as model analytes and various conditions, i.e. salt concentration and water/solvent ratio were tested. Measuring the absorbance in the organic phase was done both in-syringe and at the syringe outlet to evaluate the volume of organic phase, time required for phase separation, and for precise analysis of the extracted analytes. The method

performance in dependence of the former parameters was studied, evaluated, and improved to achieve a compromise between a high preconcentration factor and fast phase separation.

The highest achieved preconcentration factor was 6.43. The fastest phase separation took less than 5 s. The reproducibility of 3 repetitive extractions was generally below 1 % RSD.

Using n-propanol, even compounds of moderate polarity can be extracted with high-efficiency. Furthermore, n-propanol is a HPLC compatible solvent, so the extract can be optionally analyzed on-line in modern HPLC systems.

In conclusion, the salting-out assisted DLLME presents an interesting approach to perform a fast, precise, and automated extraction in small scale for the analyte preconcentration using an environment-friendly and HPLC compatible solvent.