Automated micro liquid-liquid extraction for environmental analysis

Trajan Scientific and Medical

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Overview

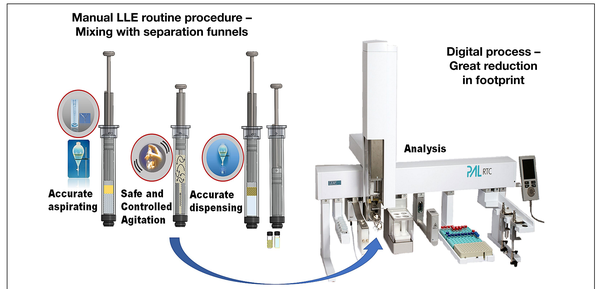

Figure 1. Transferring the LLE routine/standard procedure into a single device for automation.

Introduction

Sample preparation is an essential step in the separation of target analytes from matrix components before analysis. Liquid-liquid extraction (LLE) is of the most straightforward and widely used sample preparation techniques. However, LLE presents certain drawbacks - large volumes of organic solvents, emulsion formation, multiple extraction steps, manual handling, and the lack of analyte-specific workflow optimization. Recently, automated LLE systems have been developed to help overcome some of these problems. However, a high-throughput automated LLE approach for analytical laboratories using a single device has not been presented. Herein, we introduce a fully automated micro liquid-liquid extraction platform to carry out rapid, green and cost-effective LLE sample preparation and injection.

Aim

A fully automated micro LLE integrated into the GCMS workflow that increases laboratory productivity without impacting the measurement quality.

Advantages

- Savings on labour and consumables.
- Saving on instrument for blow down.
- Increases laboratory productivity.
- Decreases organic solvent consumption.
- Increases safety.

LLE Syringe device — Concept

Operation steps

(A) Syringe is ready to aspirate.
(B) Sample and solvent are aspirated accurately.
(C) The sealing device is locked to the flange.
(D) The plunger-agitator moves up and down for mixing and mass transfer while pump control is optimal for performance.
(E) The plunger-agitator is in the highest position.
(F) The sealing device is locked to the stem.
(G) Two phases are dispensed and collected.

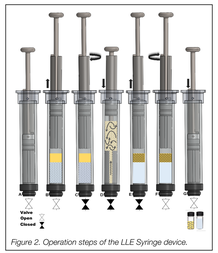

Figure 2. Operation steps of the LLE Syringe device.

PCBc extraction — Method

- A microLLE tool was designed to mount 4 mL LLE micromixer on a PAL system.
- The off-line automated workflow enables accurate aspirating of solutions, mixing, collection of the extract and washing of the mixer.
- The CTC-PAL needle guide was used for driving the LLE agitator. A tab and latch mechanism was used to enable switching between modes of operation and a valve station was added to the PAL system to actuate the micromixer valve.
- Extraction of Polychlorinated Biphenyls (PCBs - 0.1 ng/mL) from spiked tap water samples was performed to evaluate the performance of the automated system.
- The sample and solvent (25% DCM in Hexane) volume were 2.7 mL and 0.3 mL respectively.
- Mixing for 2, 4 and 8 minutes (100, 200 and 400 strokes).
- 2 µL injection - GC x GC µECD analysis.

Figure 3. First iteration of the LLE Syringe/PAL Tool.

Results

- Increasing the mixing time to 4 minutes enhances the extraction recoveries for all PCB congeners. A further mixing time of 8 minutes led to a decrease in recoveries due to emulsion formation.
- 4 minutes mixing results in average PCBc recoveries between 69% and 94% and RSD% below 14%.
- CALA sample results are equivalent to MOECP SPE results for most of the PCB congeners.
- The agitation inside the barrel in the mixing step (3 times) is enough to wash the mixer properly.

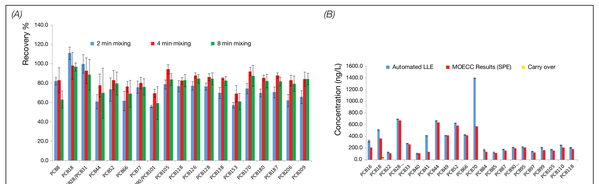

Figure 4. (A) PCBc extraction recoveries for different mixing time, (B) PCBc extraction concentration for CALA (Canadian Association for Laboratories Accreditation) water sample.

PAHs extraction — Method

- A microLLE tool was designed to mount 8.3 mL LLE micromixer on a PAL system.
- The on-line automated workflow enables accurate aspirating of solutions, mixing, collection of the extract, washing of the mixer and injection into GC/MS.
- The CTC-PAL main plunger drive has been used for driving the LLE agitator. A station has been added to the CTC-PAL to actuate the micromixer valve and to enable switching between modes of operation.
- Extraction of Polyaromatic hydrocarbons (0.0187 mg/L and 0.187 mg/L) from spiked water samples (20% NaCl in DI water) was performed to evaluate the performance of the automated system.
- The routine/standard LLE method in environmental laboratories is translated into a fully automated workflow. All the steps from spiking the samples with PAHs and surrogate solutions through adding the internal standards are fully automated.
- In the automated workflow the sample volume is 8.1 mL and the 200 µL respectively.
- Mixing time is 4 minutes and the speed of the mixing was set to 30 mm/s with 60 strokes.
- 1 and 2 µL injection - GC/MS Analysis.

Figure 5. (A) Current version of the microLLE syringe, (B) Current version of the microLLE syringe on the PAL tool, (C) Automated microLLE system installed at Trajan Scientific and Medical.

Results

- 0.187 mg/L PAHs concentration: 4 minutes mixing results in average PAHs recoveries between 73% and 117% with RSD% below 8%.
- 0.0187 mg/L PAHs concentration: 4 minutes mixing results in average PAHs recoveries between 73% and 112% with RSD% below 25%.

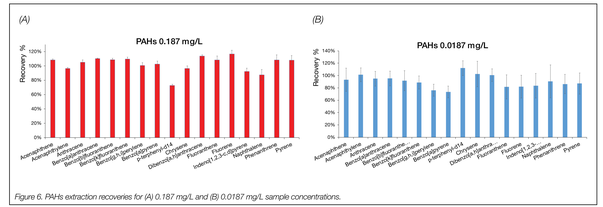

Figure 6. PAHs extraction recoveries for (A) 0.187 mg/L and (B) 0.0187 mg/L sample concentrations.

Conclusion

A fully automated microLLE system with 8.3 mL mixer has been developed. PCBc and PAHs extraction from water samples have been performed to verify the system. For both applications extraction average recoveries are above 70%. PCBc results show that the developed automated µLLE workflow is comparable with accredited MOECP method while the automated method results in great reduction in solvent consumption. The future work is the validation of the automated workflow with PAHs extraction in an Environmental laboratory.

References
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Acknowledgments
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