MICROSISAK – A NEW DEVICE FOR FAST AND CONTINUOUS LIQUID-LIQUID EXTRACTIONS ON A MICROLITER SCALE

K. Eberhardt¹, S. Andersson², C. Ekberg², B. Horn³, J.V. Kratz¹, A. Müller³, M. Nilsson², G. Skarnemark², N. Trautmann¹

¹ Institut für Kernchemie, Universität Mainz, D-55099 Mainz, Germany; ² Chalmers University of Technology, S-41296 Göteborg, Sweden; ³ Institut für Mikrotechnik Mainz, D-55133 Mainz

The fast liquid-liquid extraction system SISAK-3 is based on small centrifuges with continuous feed and output of the liquid phases at flow rates ranging from 0.5 up to 3.0 ml/s [1]. With SISAK-3, the investigation of nuclides with half-lives down to about 1 s is possible. Recently, SISAK-3 combined with an on-line detection system for α -particles and SF-events [2] based on liquid scintillation counting (LSC) has been applied to study 4.3-s ²⁵⁷Rf, produced in the reaction ²⁰⁸Pb(⁵⁰Ti,1n) [3].

From the high flow rates inherent in the application of SISAK-3 various drawbacks result:

- High consumption of organic solvents and extracting agents. Thus, continuous recycling of chemicals during a long-term experiment is irrevocable
- LSC suffers from poor energy resolution and is also sensitive to β-particles and γ-rays interfering with the detection of α-particles.

In order to overcome these problems, a new device for continuous liquid-liquid extraction on a microliter scale has been developed in a cooperation between the Chalmers University of Technology, the Institut für Mikrotechnik Mainz (IMM) and the Institut für Kernchemie. MicroSISAK consists of a stack of microstructured discs with an overall diameter of 8 mm sealed in a Ti-housing. For mixing the aqueous phase with the organic phase, a micromixer unit fabricated at IMM is used, where the phases are conducted as two counter-flows through 2 mm long and 20-50 µm broad interdigital channels (see figure 1a). The laminated flow leaves the device perpendicular to the direction of the feed flows and - due to the small thickness of the lamellae - fast mixing takes place through diffusion [4]. The mixer can be made of titanium, stainless steel or SiO₂. The mixed phases are then fed into a filter unit (see figure 1b) for instant phase separation using a teflon filter with a pore size of 0.5-1 µm. Here, the aqueous phase is completely retained from the hydrophobous filter, while the organic phase penetrates the filter. A small differential pressure (10-50 mbar) must be applied across the membrane [5]. The volume of the mixer and the filter unit, respectively, is in the order of 2-5 µl. The system is designed to provide a hold-up time of about 1 s for the mixer-filter combination at a flow rate of 0.02 ml/s.

Phase separation has been checked with an aqueous $NaCO_3$ -solution and toluene as organic phase. Aliquots of the outgoing phases were irradiated at the research reactor TRIGA Mainz. The ²⁴Na-activity in the two phases delivers the phase purity. It could be shown that at flow rates of 0.002-0.04 ml/s less then 0.5% aqueous phase contamination is in the organic phase.

Next, the D-values of Gd extracted into toluene with 2-ethyl-hexyl-orthophosporic acid (HDEHP) from 0.050 M HNO₃ and of Hf into dibutyl-phosphate (DPB)/toluene from 6 M HNO₃ will be determined. Further experiments are planned to measure the total hold-up time of the system and to optimize the extraction yield for flow rates below 0.002 ml/s. Under these conditions a new detection system can be applied where the outgoing organic phase is evaporated rapidly to dryness and assayed for activity by silicon detectors.



Figure 1: Schematic view of the new MicroSISAK-device with a microstructured unit for intense mixing of phases (1a) and a filter unit for subsequent phase separation (1b).

References

- [1] H. Persson et al., Radiochim. Acta **48**, 177 (1989)
- [2] B. Wierczinski et al., Nucl. Instr. Meth. A370, 532 (1996)
- [3] J.P. Omtvedt et al., J. Nucl. Radiochem. Sci.3, 143 (2002)
- [4] W. Ehrfeld et al., Microreactors, Wiley-VCM Weinheim (2000)
- [5] K. Eberhardt et al., Institut für Kernchemie Annual Report (2003)