

Liquid – liquid – extraction with the MicroSISAK – system

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In previous experiments it could be demonstrated that continuous liquid – liquid – extractions on a μl scale are possible with the MicroSISAK device [1]. Recently the IMM developed an improved version of this so called microreactor with two different mixer designs and up to three consecutive separation stages. A scheme of the setup for the experiments is shown in figure 1.

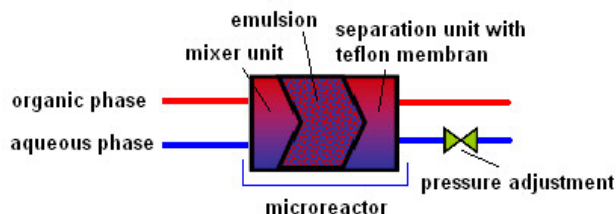


Figure 1: Scheme of the MicroSISAK setup

A static interdigital mixer is used for the formation of small droplets [2]. The formed emulsion is then separated by using a hydrophilic teflon membrane. A backpressure at the outlet of the aqueous phase corresponds to the internal pressure in the MicroSISAK device. This has an effect on the separation at the membrane. The inner volume of the used so far needle valve is much larger than the volume of the mixer, thus it is not possible to adjust a definite and reproducible backpressure in the separation unit.

Therefore a new apparatus was designed to set backpressure on the aqueous outlet of the microreactor by a gas volume. A standard vial made of polyethylene-terephthalate is equipped with three holes in the upper sealing cap: one as inlet for the aqueous phase coming from the reactor, and two for gas in and out. One hole in the bottom serves as liquid outlet (Fig. 2).

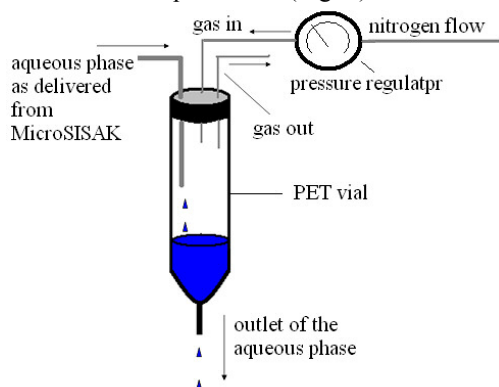


Figure 2: Scheme of the new pressure regulation apparatus.

The pressure in the vial is controlled with a high precision pressure regulator. It is reproducible with an accuracy of 5 mbar.

For studies on the extraction yield for hafnium – the lighter homologue of rutherfordium (Rf) $Z = 104$ – we have chosen an extraction system, that has successfully been applied in chemical investigations of Rf with SISAK [3]: Extraction of hafnium (Hf) with trioctylamine (TOA)

in toluene from dilute sulphuric acid. While keeping the TOA concentration constant, the concentration of the acid was varied from 0.05 mol/l to 2 mol/ml. In order to measure the extraction yield Hf-181 is used as a tracer. Its activity in the organic phase is compared to the total activity in both phases. From this one can calculate the extraction efficiency. The main results of the batch experiments are shown in the upper line of figure 3. The results of the extraction experiments with the microreactor (lower part of figure 3) compare to the one in the batch experiments relating to the concentration of the two solutions.

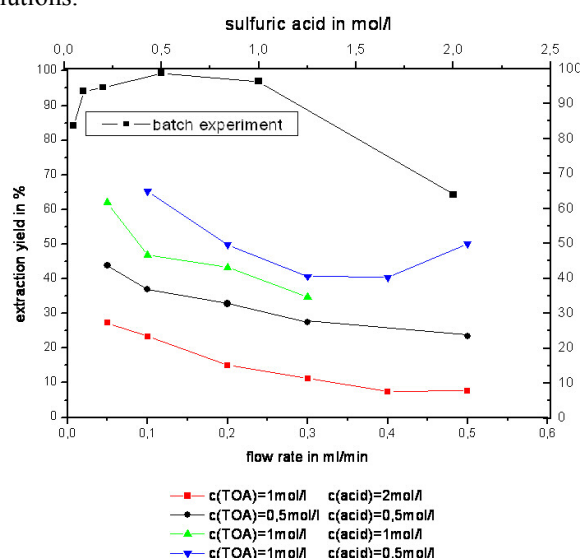


Figure 3: Extraction of Hf-181 with TOA: The upper line shows the extraction yield by varying the concentration of the sulphuric acid and a constant concentration of TOA (1 mol/l). The lower part: the extraction yield depends on the flow rate for different concentrations of the TOA and sulphuric acid.

One can observe a decrease of the extraction yield with increasing flow rates. At flow rates below 1 ml/min phase mixing is inefficient, thus extraction yield is low and mainly depends on the hold-up time in the mixer. At flow rates higher than 1 ml/min, smaller droplets are formed in the mixer. Thereby an emulsion is created and a much higher extraction yield can be expected. Corresponding experiments are currently carried out.

Acknowledgement:

I would like to thank the mechanical workshop of our institute for the realisation of the new pressure regulation apparatus.

References:

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