MicroSISAK – Improvements of a device for continuous liquid-liquid-extraction on a microliter scale

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Outline

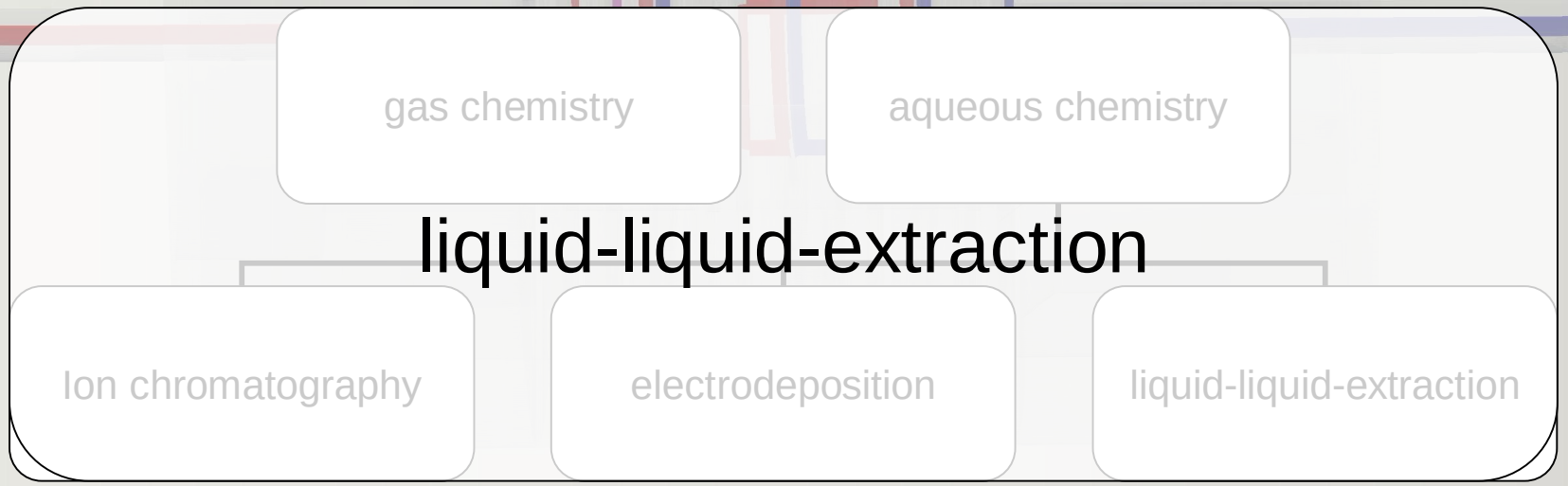
- Motivation
- The MicroSISAK apparatus
- Results of the experiments
- Summary
- Outlook

Motivation

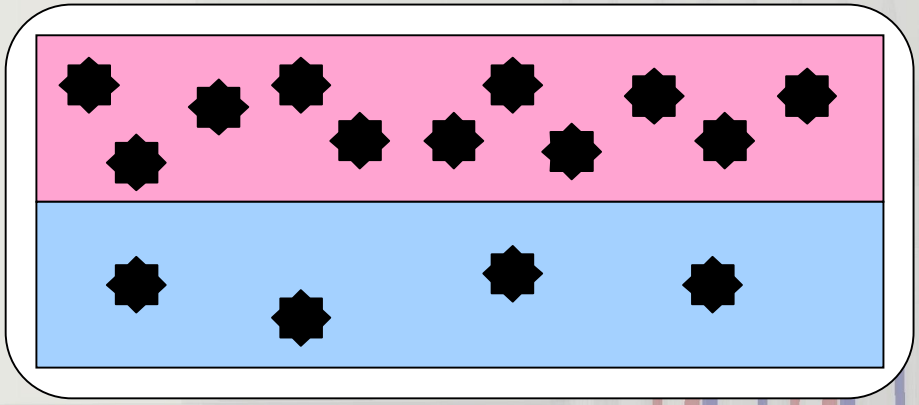
Superheavy elements = Transactinides: Z > 103

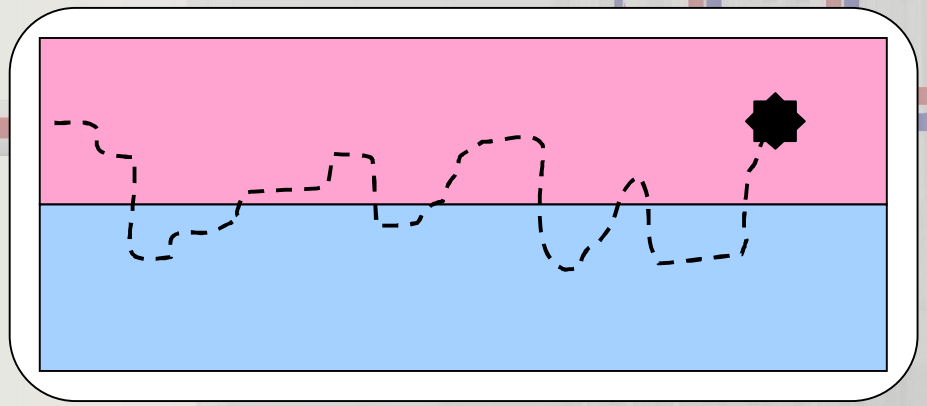
- low production rates from atoms per minute (for Rf Z=104) to a few atoms per week (for element 112)
- short half-lives in order of seconds
- decay mode: α-decay and spontaneous fission (SF)

→ fast and efficient apparatus for chemical separation and detection



Motivation





Thermodynamics:

$c_{phase1} / c_{phase2} = K$

with $K = \exp [(\mu^\circ_1 - \mu^\circ_2) / RT]$

„one-atom-at-a-time“:

concentrations were removed by probabilities

$K = \varphi_{phase1} / \varphi_{phase2}$

=> this concept works with liquid-liquid-extraction as shown with SISAK

Motivation

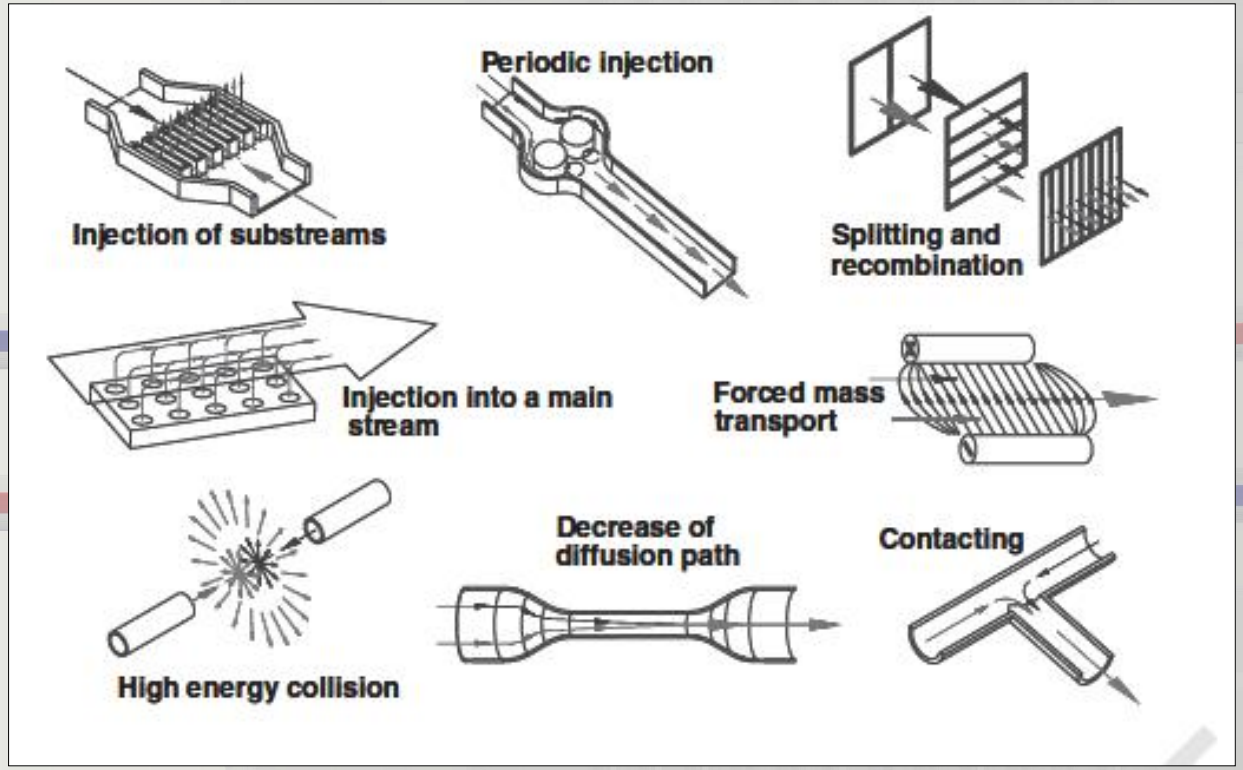
SISAK - Short-lived Isotops Studied by the AKUFVE technique

Pro:	Con:
centrifuges made of Ti/Pd or PEEK	minimum flow rate ~ 0,3 ml/s
small inner volume: 0,3 ml	high consumption of chemicals
extraction efficiency: 80-90 %	detection of transactinides restricted to liquid scintillation counting (LSC) in a fast flowing liquid
nuclides with half-lifes < 1 s can be studied	
successfully applied for chemical studies on the properties of Rf	

→ MicroSISAK approach: fast extraction on a µl scale

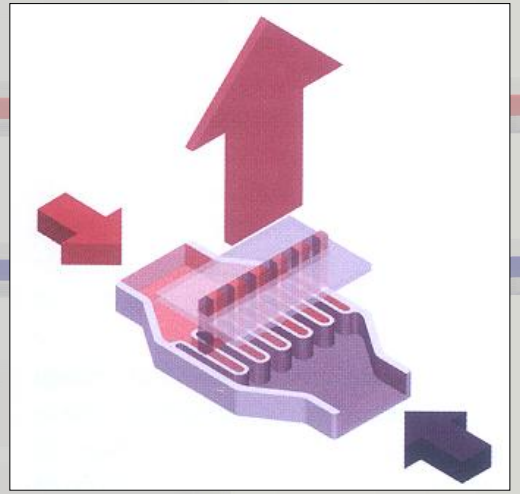
Motivation

use of microreaction technology for efficient mixing of the two phases



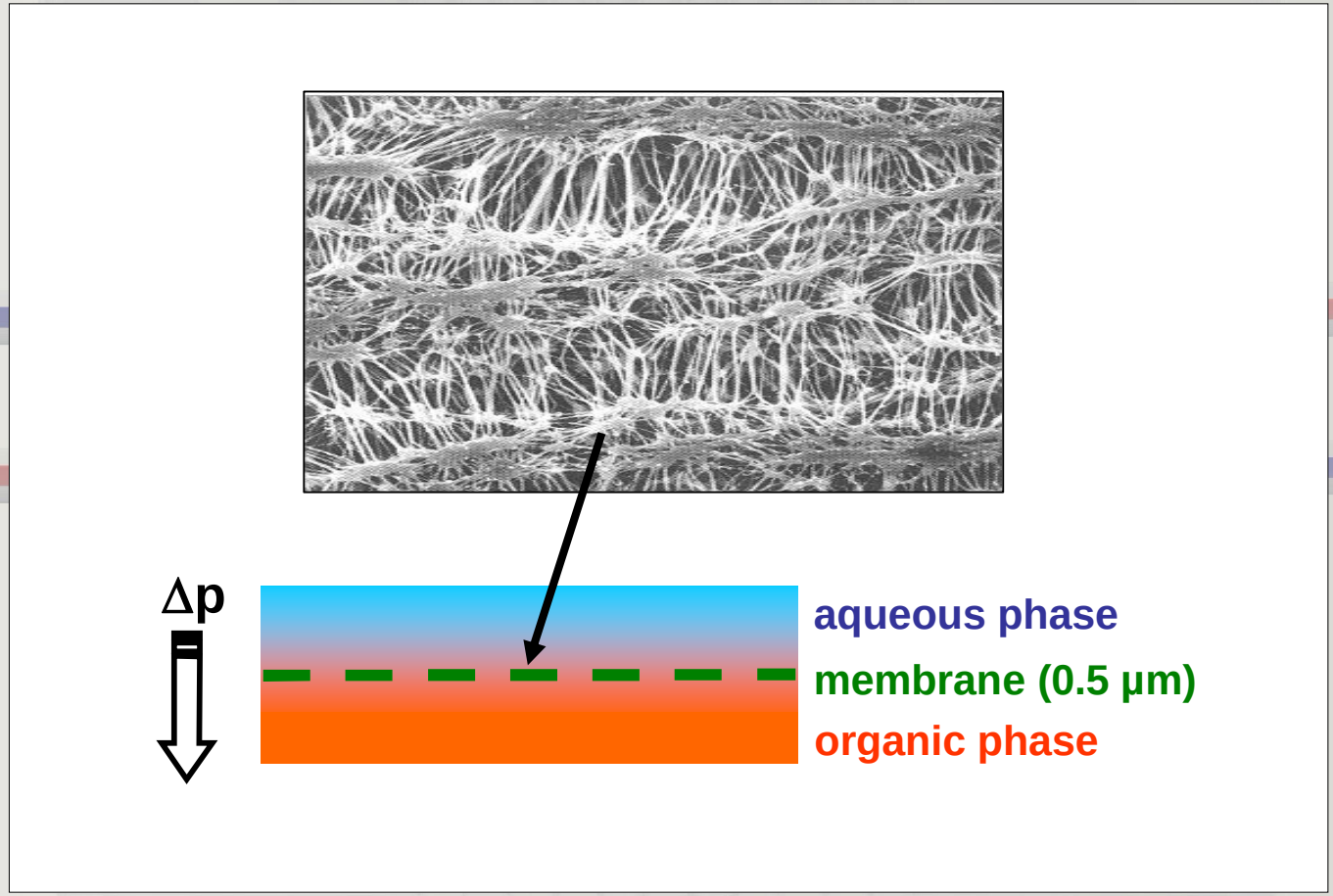
schematic drawings of selected passive and active micromixing principles, source: Löwe et al., 2000

principle of interdigital mixing



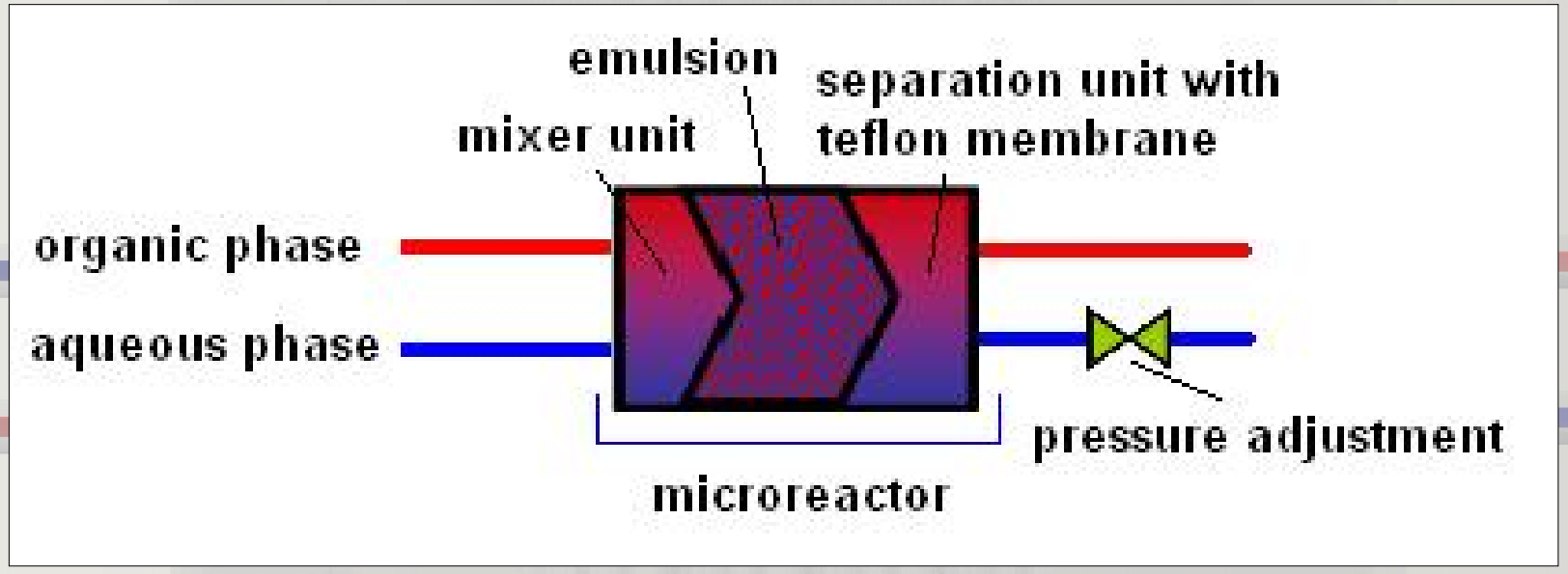
Motivation

separation of the emulsion by a hydrophobe membrane of Teflon



The MicroSISAK apparatus

combination of both steps in one apparatus

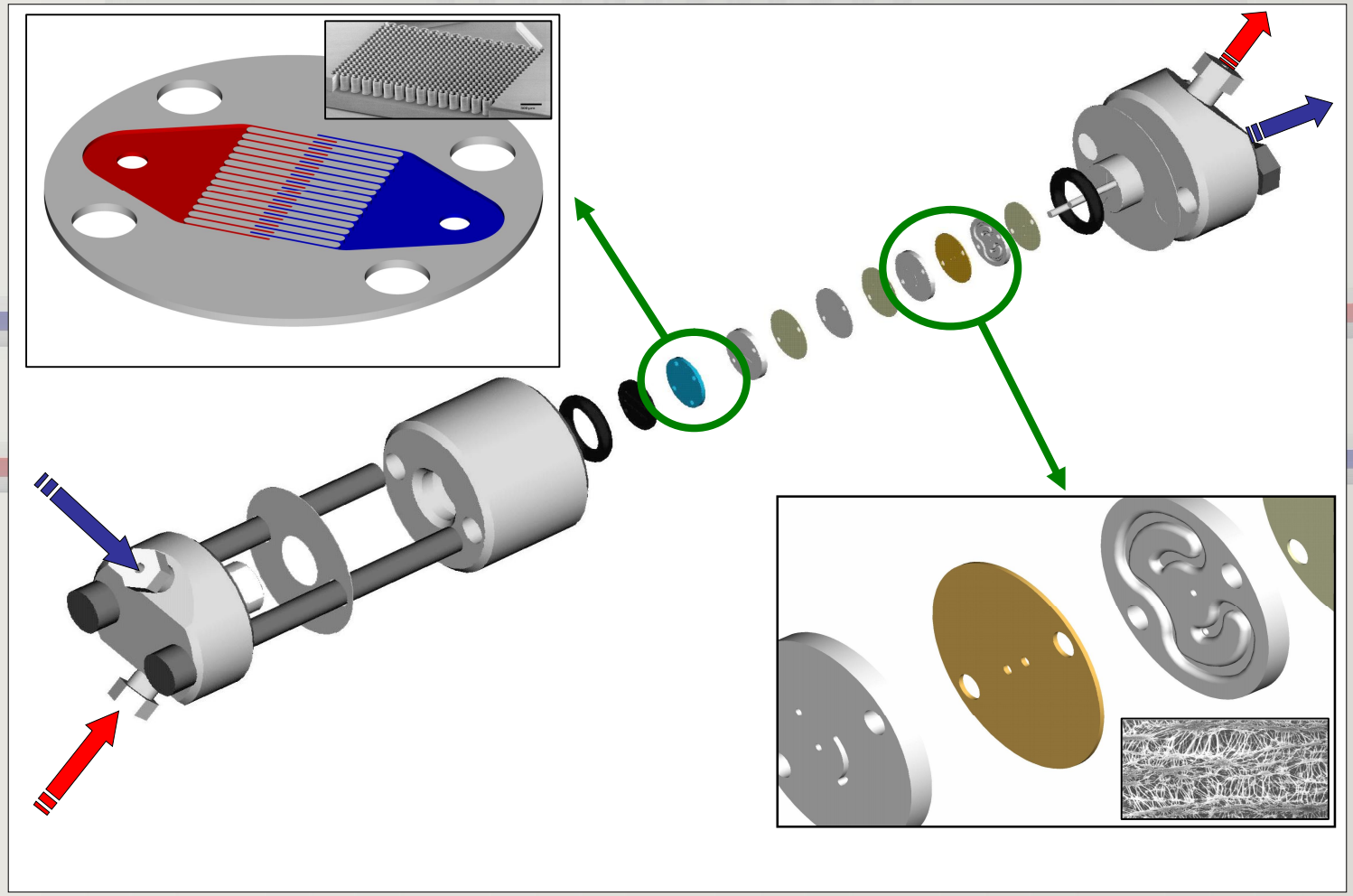


schematic view of the MicroSISAK – system

development and construction of the microreactorby the
Institut für Mikrotechnik Mainz IMM

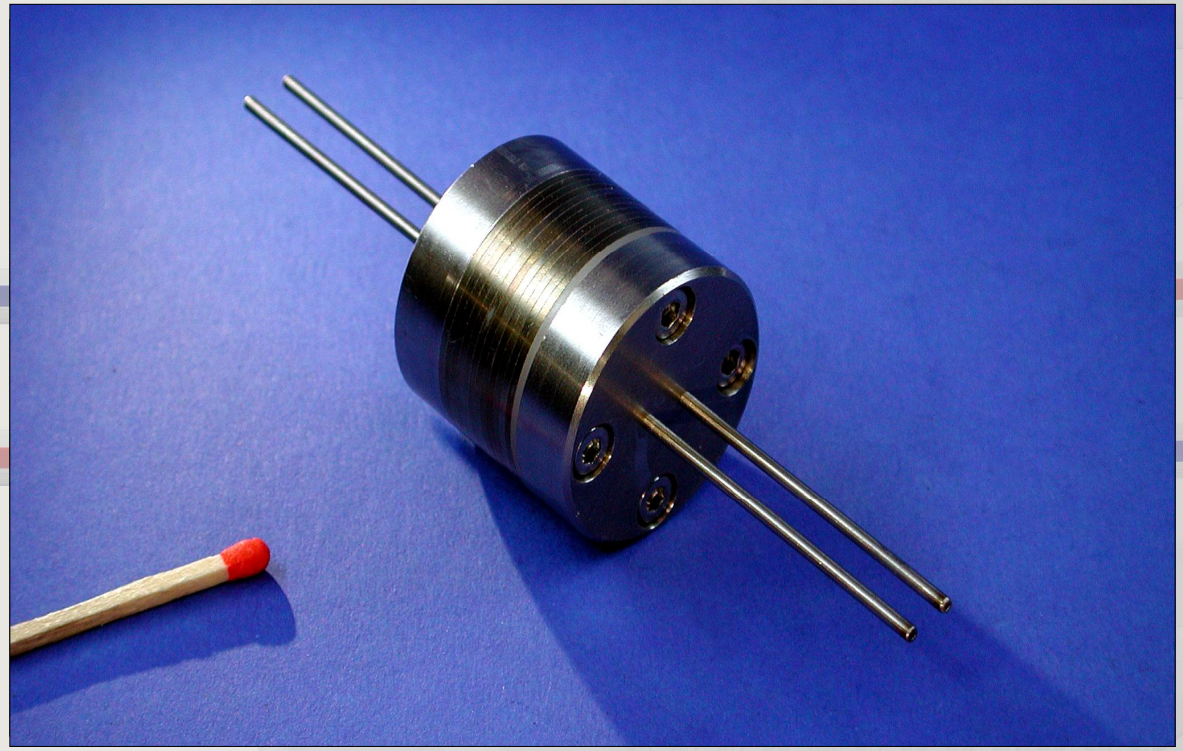
The MicroSISAK apparatus

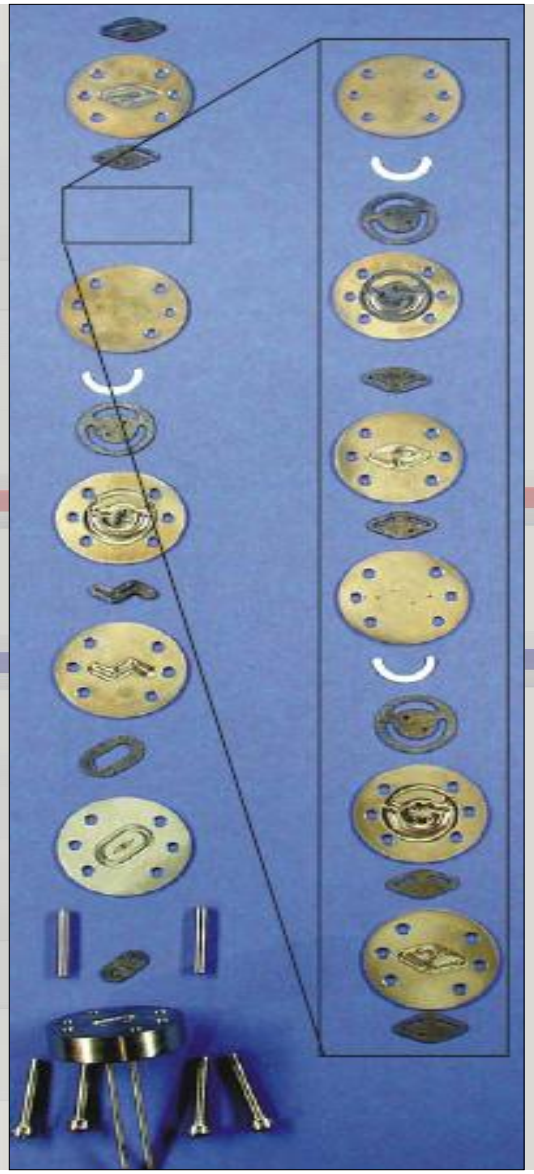
first prototype, that combine a mixer and a separation unit



The MicroSISAK apparatus

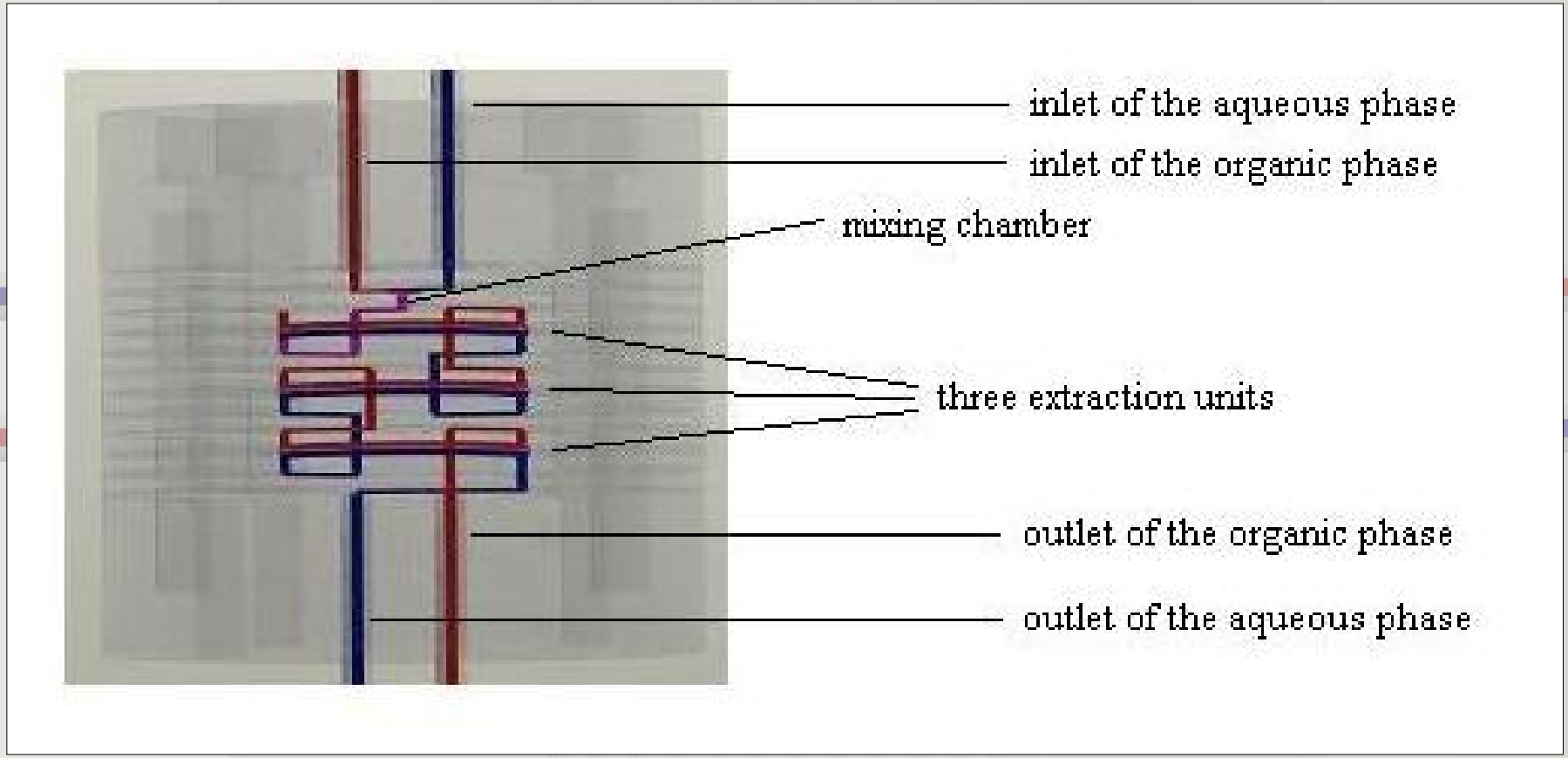
the setup of the actuell MicroSISAK apparatus





The MicroSISAK apparatus

flow scheme inside of the microreactor

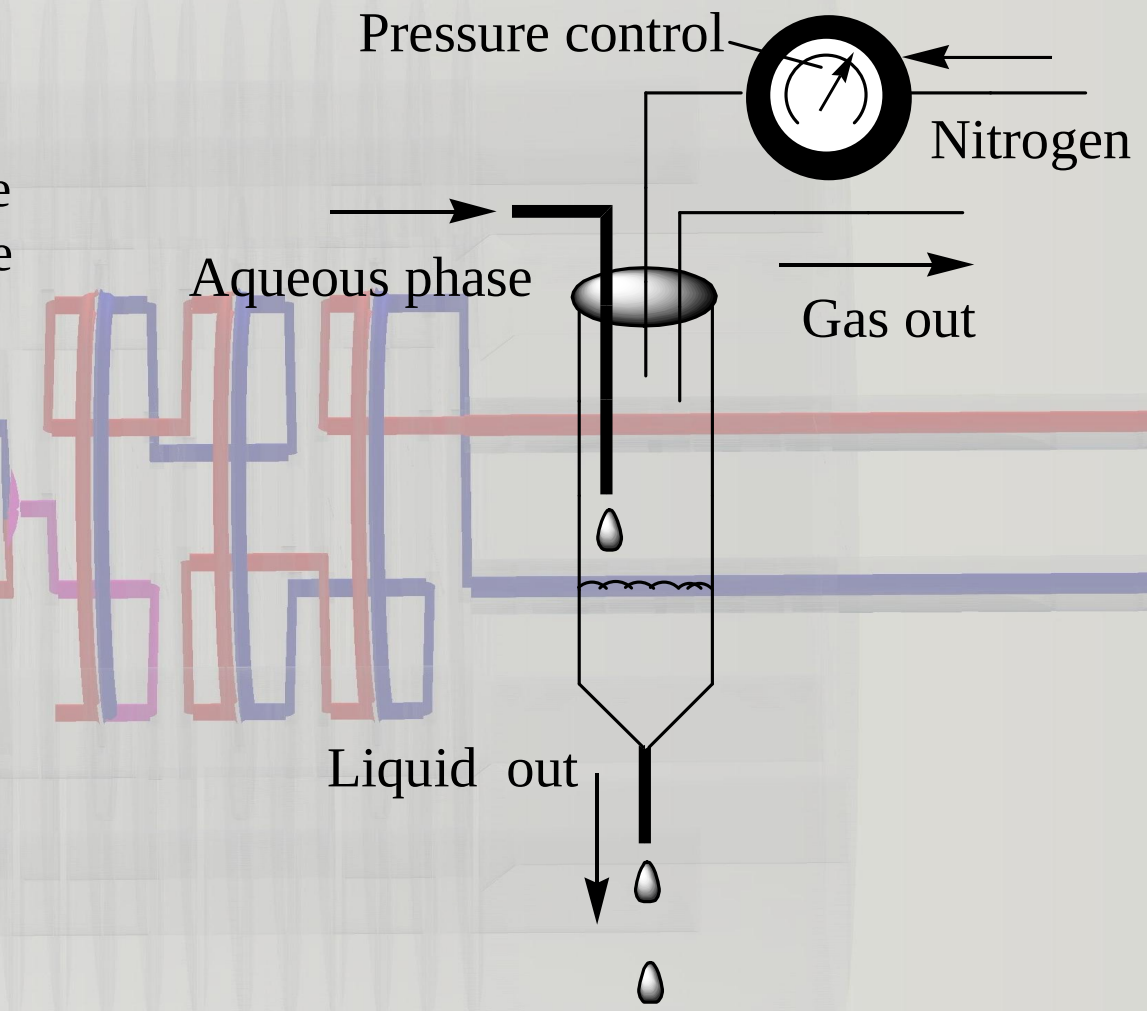


The MicroSISAK apparatus

- the organic solutions of the different phase systems have an unequal behavior at the Teflon membrane

- precise control of Δp over the membrane

→design of a new pressure regulator

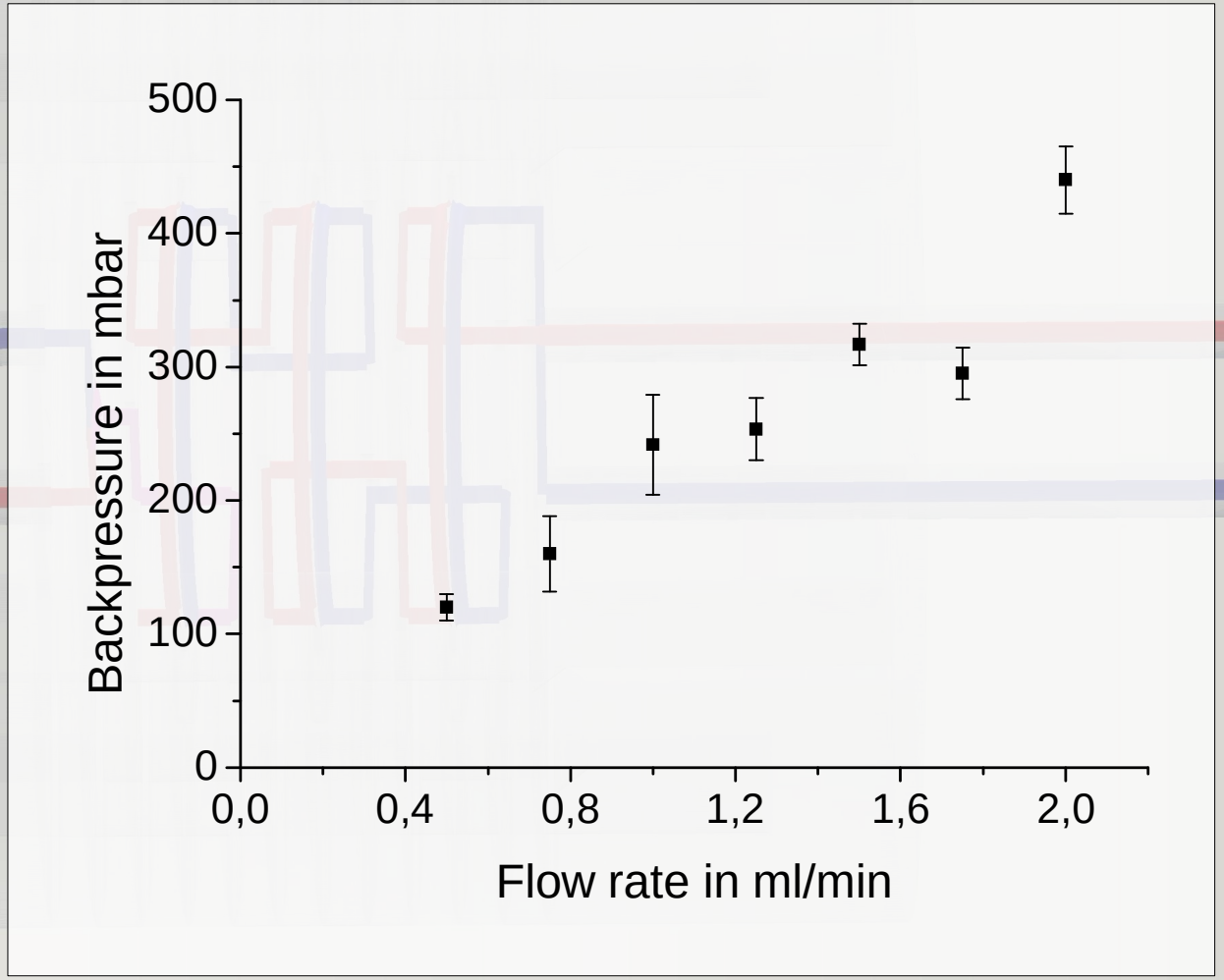


Results of the experiments

separation experiments with a pressure regulator

for the phase system
1 mol/l TOA in toluene
vs. 0.5 mol/l sulfuric acid
(taken from successful SISAK experiments with Zr, Hf and Rf)

for a complete separation at higher flow rates more back pressure at the outlet of the aqueous phase is needed



Results of the experiments

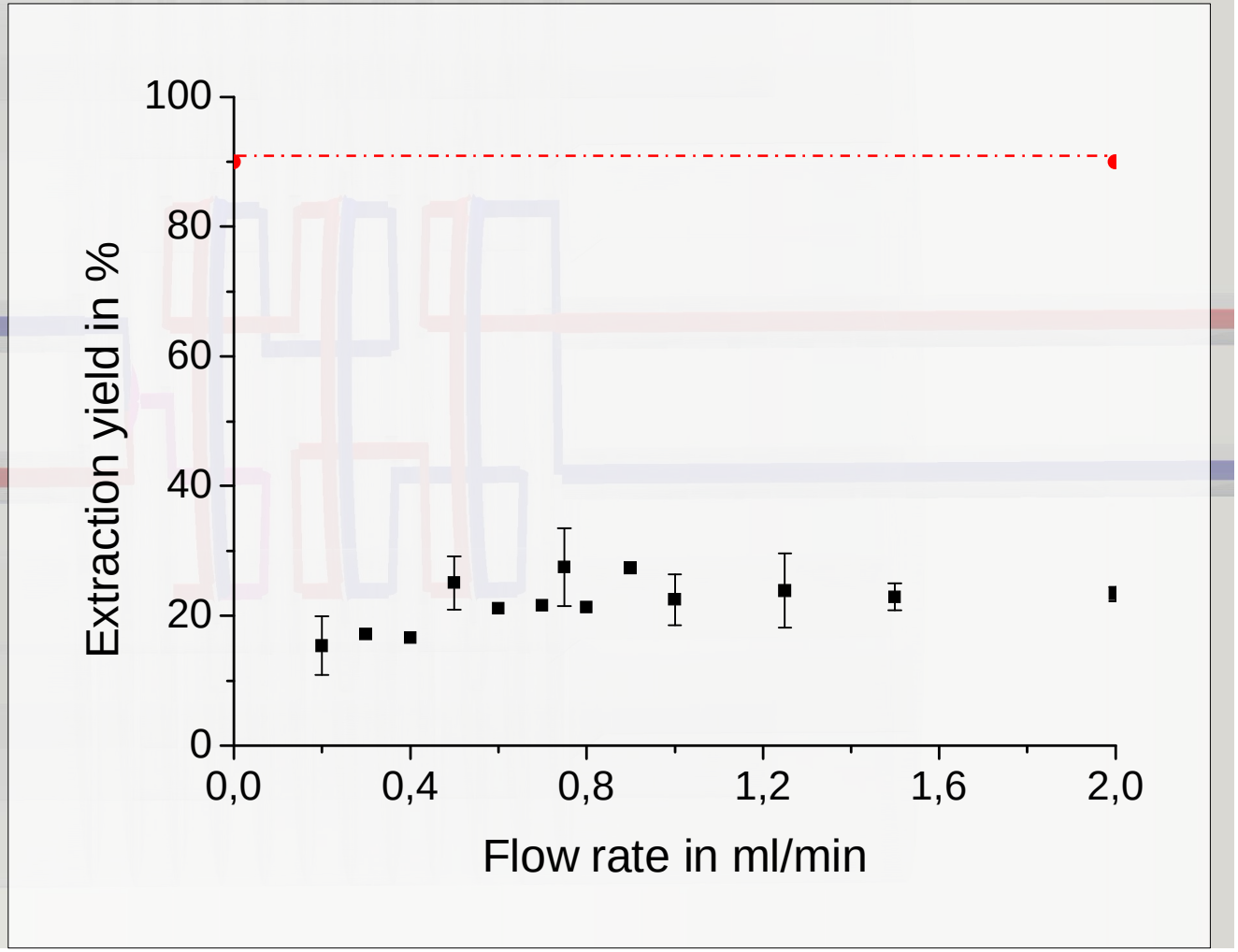
extraction experiments with hafnium (group IV)

nuclide: Hf – 181
molar amounts

system:

TOA 1 mol/l in toluene
vs. H_2SO_4 0.5 mol/l

batch experiments:
extraction yield of
90 %



Results of the experiments

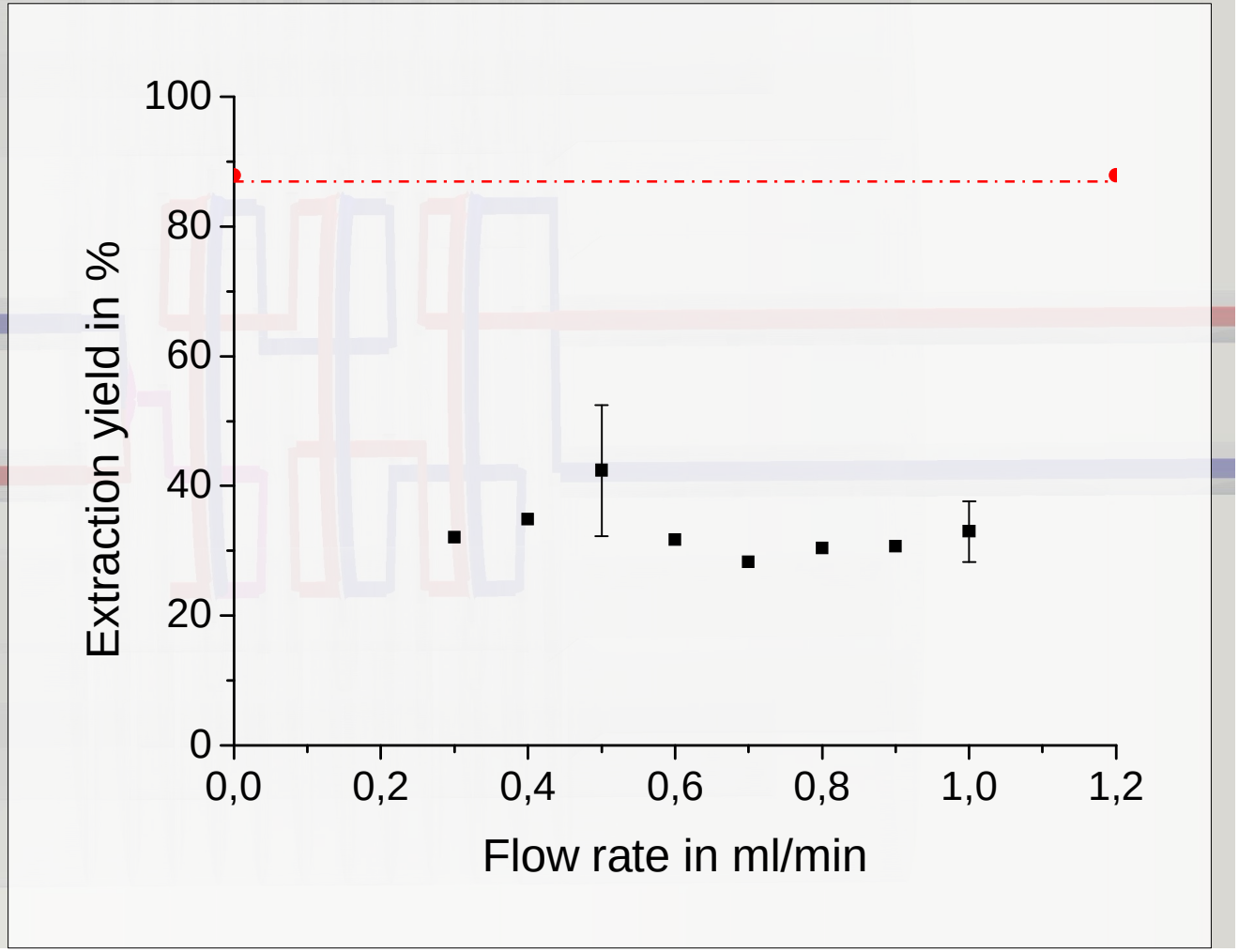
extraction experiments with technetium (group VII)

nuclide: Tc – 99m, carrier-free

system:

TPAC 10^{-4} mol/l in chloroform vs. HNO_3 0.01 mol/l

batch experiments: extraction yield of 88 %



Summary and Outlook

Summary:

- with the aid of a pressure regulator, complete phase separation achieved (0.1 ml/min – 2.0 ml/min)
- design of the mixer unit is still a crucial factor

Outlook:

- tests with further extraction systems for the elements Hf and Zr as lighter homologues for Rf, plus Tc and Re as the lighter homologues for Bh
- searching for an appropiate detection system (LSC/SBD)
- experiments with Rf behind TASCA at the GSI Darmstadt

JOHANNES GUTENBERG UNIVERSITÄT MAINZ

Thank you!