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## Computer Controlled Chemical Micro-Reactor

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**Abstract.** Chemical reactions or chemical equilibria can be influenced and controlled by several parameters. The ratio of two liquid ingredients, the so called reactants or educts, plays an important role in determining the end product and its yield. The reactants must be weighed and accordingly mixed with the conventional batch mode. If the reaction is done in a micro-reactor or in several parallel working micro-reactors, units for allotting the educts in appropriate quantities are required.

In this report we present a novel micro-reactor that allows the constant monitoring of the chemical reaction via Raman spectroscopy. Such monitoring enables an appropriate feedback on the steering parameters for the PC controlled micro-pumps for the appropriate educt flow rate of both liquids to get optimised ratios of ingredients at an optimised total flow rate. The micro-reactors are the core pieces of the design and are easily removable and can therefore be changed at any time to adapt the requirements of the chemical reaction. One type of reactor consists of a stainless steel base containing small scale milled channels covered with anodically bonded Pyrex glass. Another type of reactor has a base of anisotropically etched silicon, and is also covered with anodically bonded Pyrex glass. The glass window allows visual observation of the initial phase interface of the two educts in the reaction channels by optical microscopy and does not affect, in contrast to infrared spectroscopy, the Raman spectroscopic signal for detection of the reaction kinetics.

On the basis of a test reaction, we present non-invasive and spatially highly resolved in-situ reaction analysis using Raman spectroscopy measured along the reaction channel at different locations.

### 1. Introduction

The authors manufactured a chemical micro-reactor with a glass window. Chemical reaction inside the system can be monitored using optical techniques, such as Raman spectroscopy. The information collected could give real-time feedback to optimize the reaction. The micro-reactor may be a new contribution to this research field.

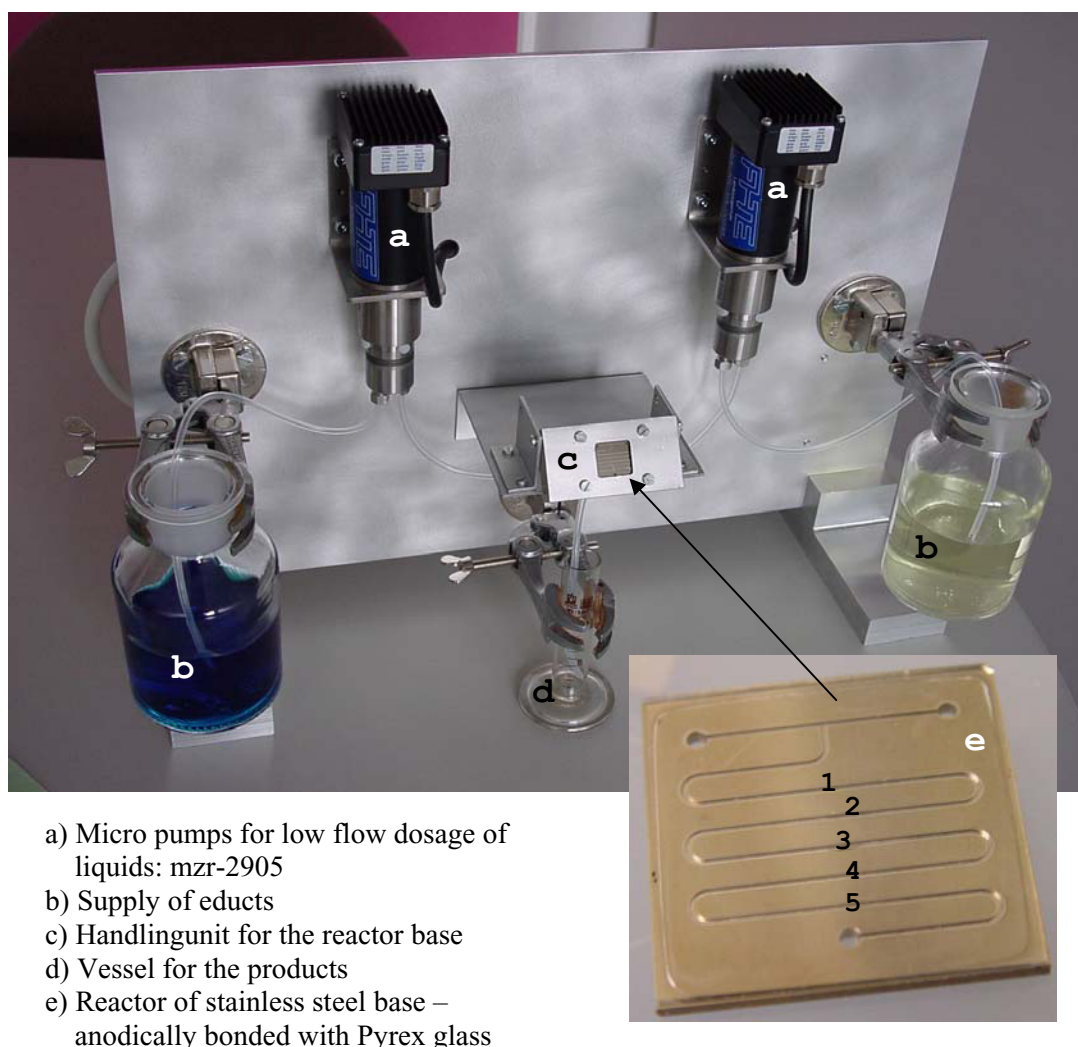
## 2. Results and Discussion

The total micro-reactor is shown in Figure 1. The reactor cores where the chemical reactions take place consist of either micro milled stainless steel Invar or wet-etched Silicon and can be exchanged easily enabling for a broad variety of chemical reactions. All reactor cores are covered with anodically bonded Pyrex glass for easy optical observation as well as spectroscopic inspection of the chemical reactions taking place.

The total size of the reactor cores is  $20 \times 20 \text{ mm}^2$ , the dimensions of the fluidic canals are:

- 200 to 400  $\mu\text{m}$  width and 200  $\mu\text{m}$  depth for the stainless steel based cores
- 50 to 100  $\mu\text{m}$  width and 50 to 70  $\mu\text{m}$  depth for the Silicon based cores

The total length of the fluidic canals varies with the base material, the abovementioned dimensions as well as the additionally integrated mixing elements and ranged in between 5 cm and 50 cm.



- a) Micro pumps for low flow dosage of liquids: mzs-2905
- b) Supply of educts
- c) Handlingunit for the reactor base
- d) Vessel for the products
- e) Reactor of stainless steel base – anodically bonded with Pyrex glass

Fig. 1: Assembly of the micro-reactor arrangement with extension of the stainless steel reactor base.

The total micro-reactor system utilises two external computer controlled micro-pumps (manufactured: HNP Mikrosysteme GmbH) ensuring the optimized mixing ratio of the two educts as



Fig. 2: Lab-VIEW 7.0 programmed „Front Panel“ for triggering the micro-pumps.

well as optimized mean residence times for the chemical constituents within the reactor cores. The user interface controlling the micro-pumps is programmed in LAB-VIEW<sup>®</sup> 7.0 [1]. The appropriate Front Panel is shown in Figure 2.

The Pyrex glass cover allows the spectroscopic investigation of the chemical reaction along the total length of the reactor cores, mappings e.g. across the width of the fluidic canals and even chemical imaging of the total reactor core and thus of the chemical reaction taking place. In case of a constant flow each data point along the length of the fluidic canal refers to a specific reaction time of the chemical reaction. This allows tracking the sequence of a chemical reaction and such it's chronology with very high resolution in time and minimized signal to noise ratios, since each data point can be inspected for an arbitrary time.

The ideal spectroscopic method is Raman spectroscopy, since the Raman signal is not absorbed or otherwise influenced by the glass cover [2] [3]. As a test reaction a Paal-Knorr-Synthesis was chosen, using the educts acetylacetone and ethanolamine whereby the product is a substituted pyrrole. The Raman spectra of both educts and that of the product mixture are shown in Figure 3.

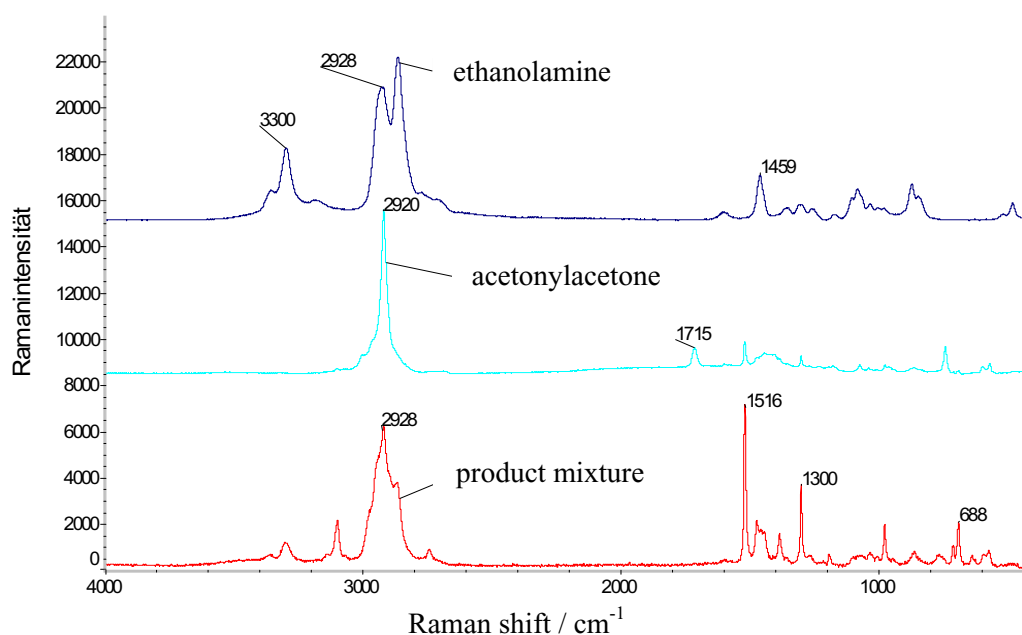


Fig. 3: Raman spectra of both educts and of the product mixture.

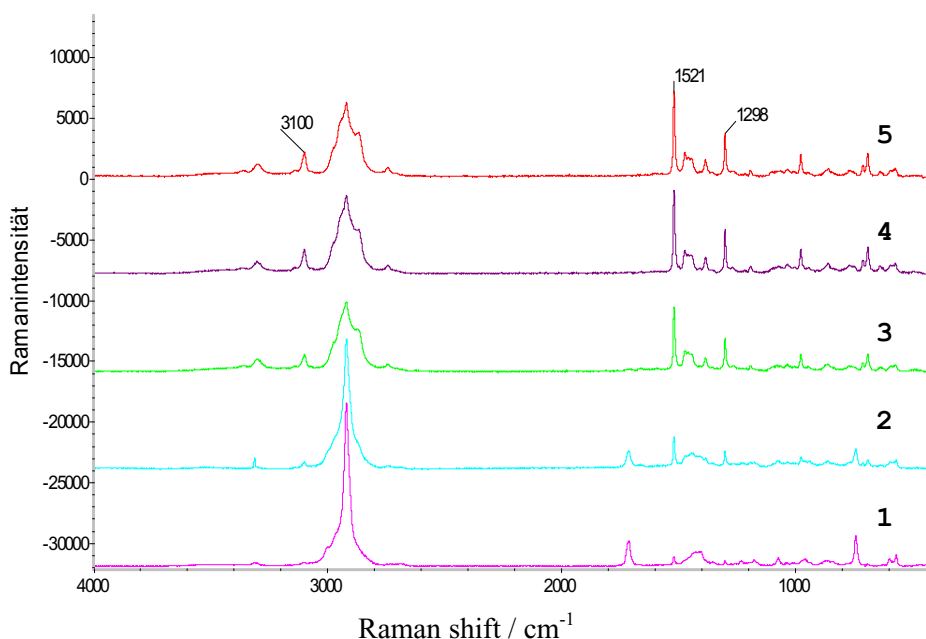


Fig. 4: Increasing peak-intensity of the products with increasing reaction time. The numbers 1-5 refer to the appropriate ones in Fig. 1 (enlarged section).

Figure 4 shows Raman-spectra taken online along the reaction channel whereby the numbers 1-5 refer to the appropriate ones in Figure 1. Clearly the decrease of the signals for the educts and the increase of the signals for the products can be verified.

### 3. Conclusions

We manufactured a chemical micro-reactor with a glass window covering the entire system of fluidic canals. Such design with a glass cover allows the monitoring of the chemical reaction via optical spectroscopic techniques like Raman spectroscopy. Additionally tracking the sequence of the chemical reaction is possible by taking data points along the micro-fluidic canal. The spectroscopic information is used as a feedback for the steering parameters of the PC controlled micro-pumps.

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