

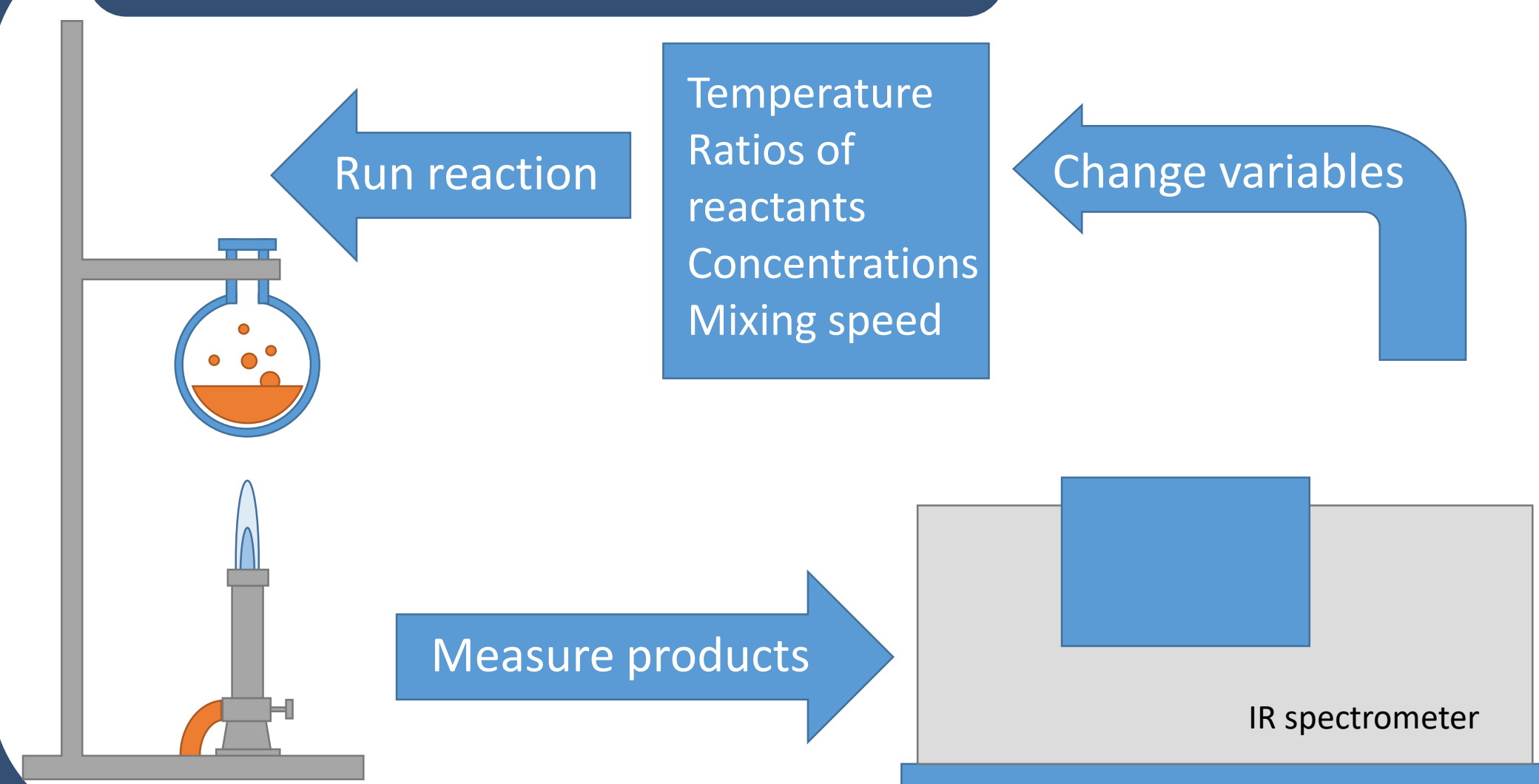
# MODULAR ATR FT-IR MICROREACTOR CHIP FOR OPTIMIZING REACTION CONDITIONS

Jasper J.A. Lozeman, Jeroen C. Vollenbroek, Johan G. Bomer, Hans L. de Boer, Albert van den Berg, Mathieu Odijk

BIOS Lab-on-a-Chip Group, MESA+ Institute for Nanotechnology, Technical Medical Centre, Max Planck Center for Complex Fluid Dynamics, University of Twente, Enschede, The Netherlands.

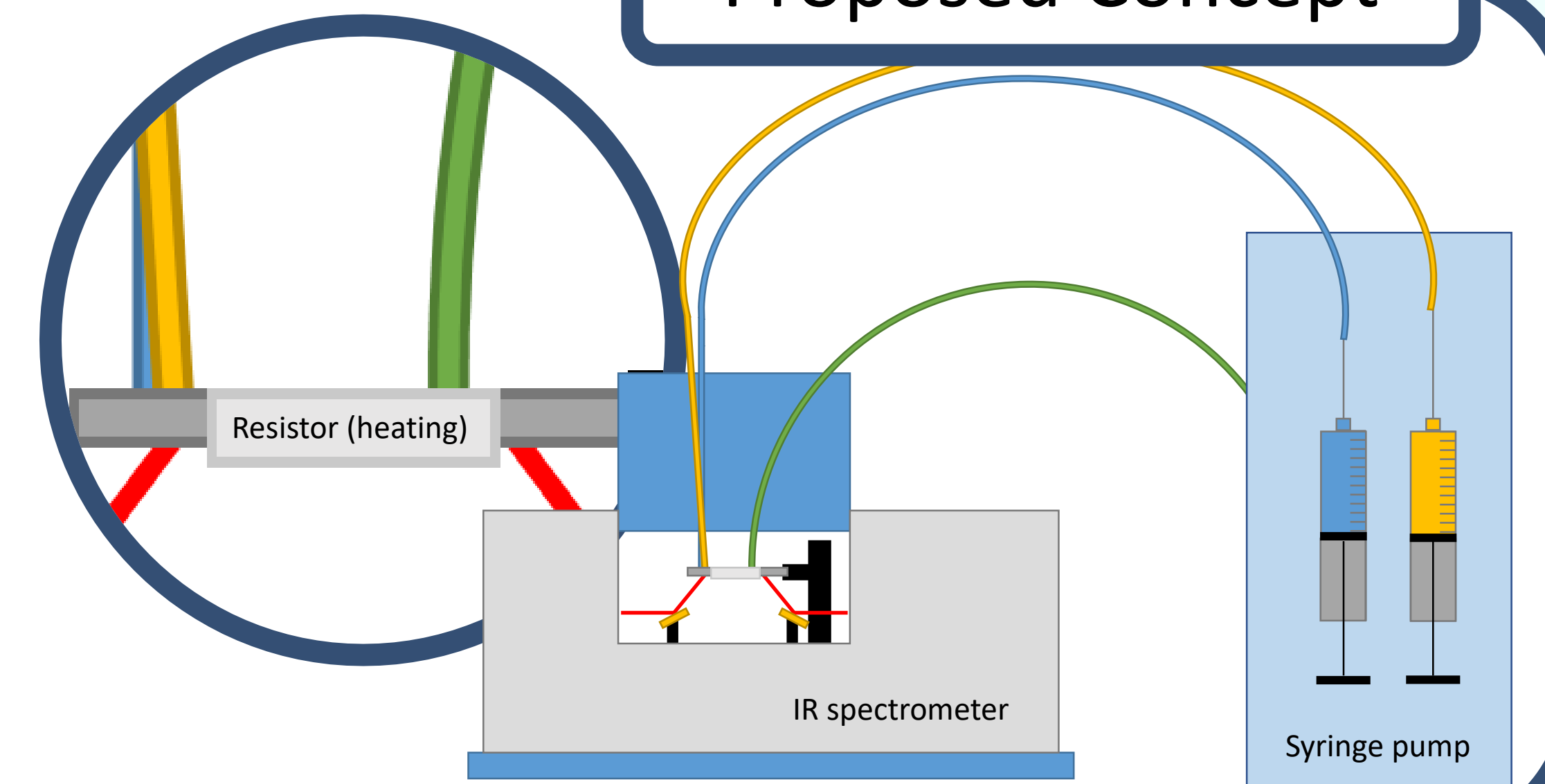
A silicon chip for attenuated total reflection (ATR) Fourier transform infrared (FT-IR) spectroscopy in combination with a modular herringbone mixer<sup>1</sup> and a microreactor has been successfully fabricated and tested. The modular design allows the chip to be used for a variety of reactions. A model synthesis of 1-butyl-2,5-dimethyl-1H-pyrrole from hexane-2,5-dione with 1-butylamine has been performed on chip. When plotting the natural logarithm of the peak area corresponding to the ketone stretch vibration at  $1710\text{cm}^{-1}$  against the residence time, a linear curve can be fitted, suggesting this step to be a first order reaction.

## Conventional



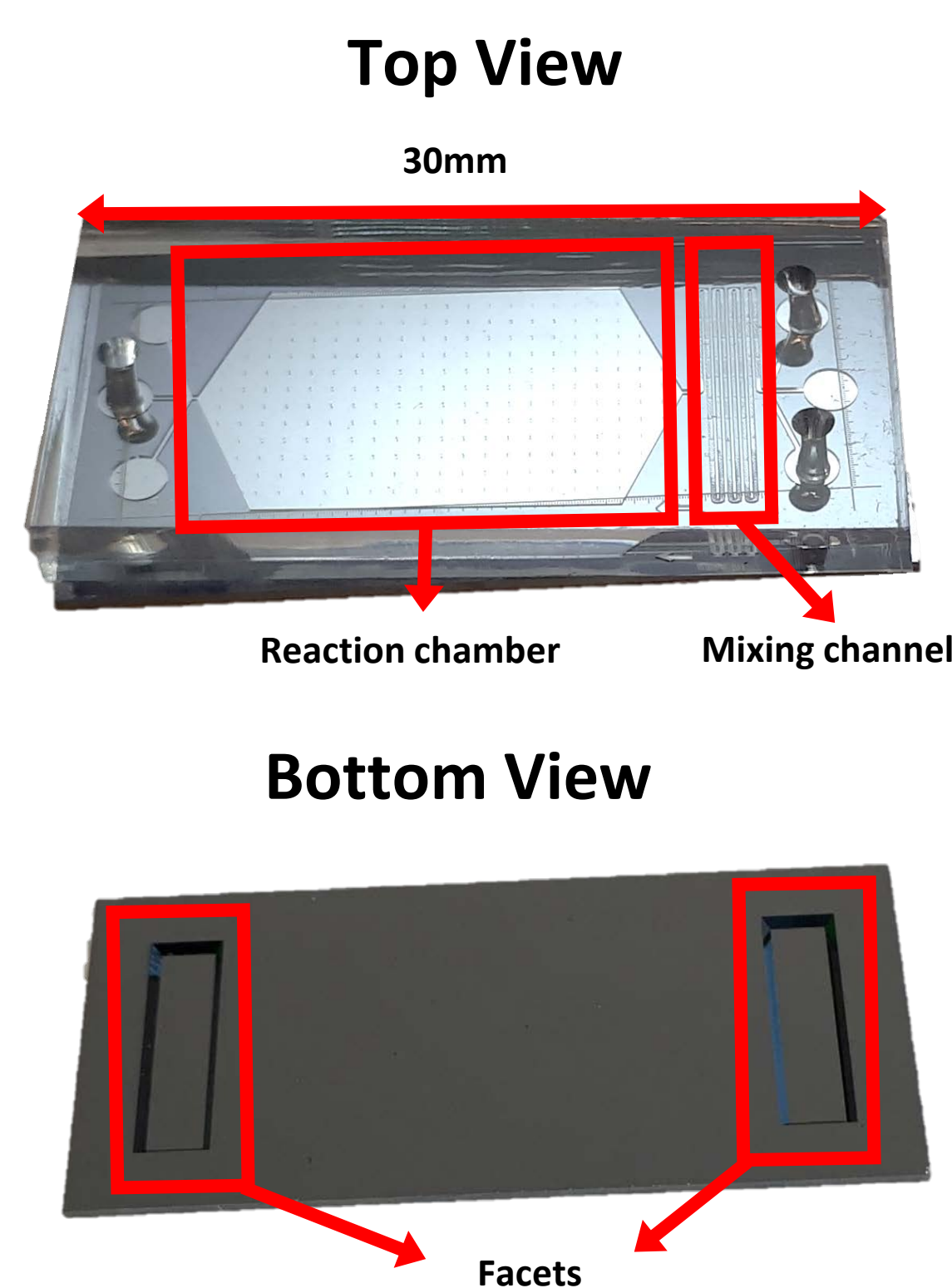
A setup is proposed, using microfluidic flow chemistry (MFC), in which the reactions conditions can be changed in real-time, while measuring the formed products online. MFC allows for better control over temperature, concentrations and mass transport than conventional laboratory setups<sup>2</sup>. Additional benefit of online analysis is further insight in reaction kinetics and intermediate products.

## Proposed Concept

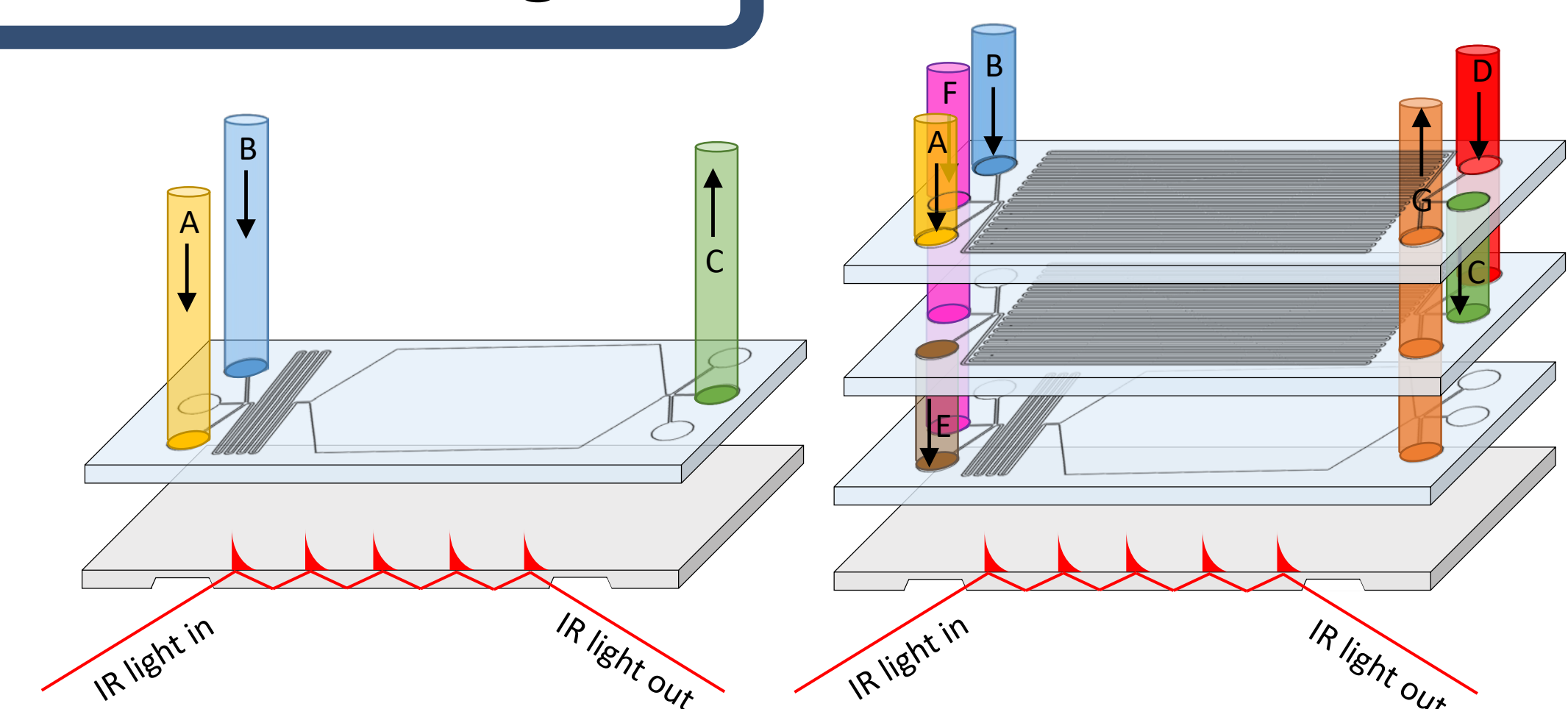


## Chip Design

The ATR crystal, an adaptation of earlier designs<sup>3,4</sup>, is made of silicon. This crystal, the bottom layer of the device, is used for IR-ATR sensing. To couple light into the silicon, facets are fabricated using KOH etching. The reaction chamber and mixing channel, fabricated from PDMS, PMMA, Teflon or COC, are modular in the sense that they can be stacked. The mixing channels mix by using herringbone mixers<sup>1</sup>. Channel dimensions:  $H=100\mu\text{m}$   $W=200\mu\text{m}$ .

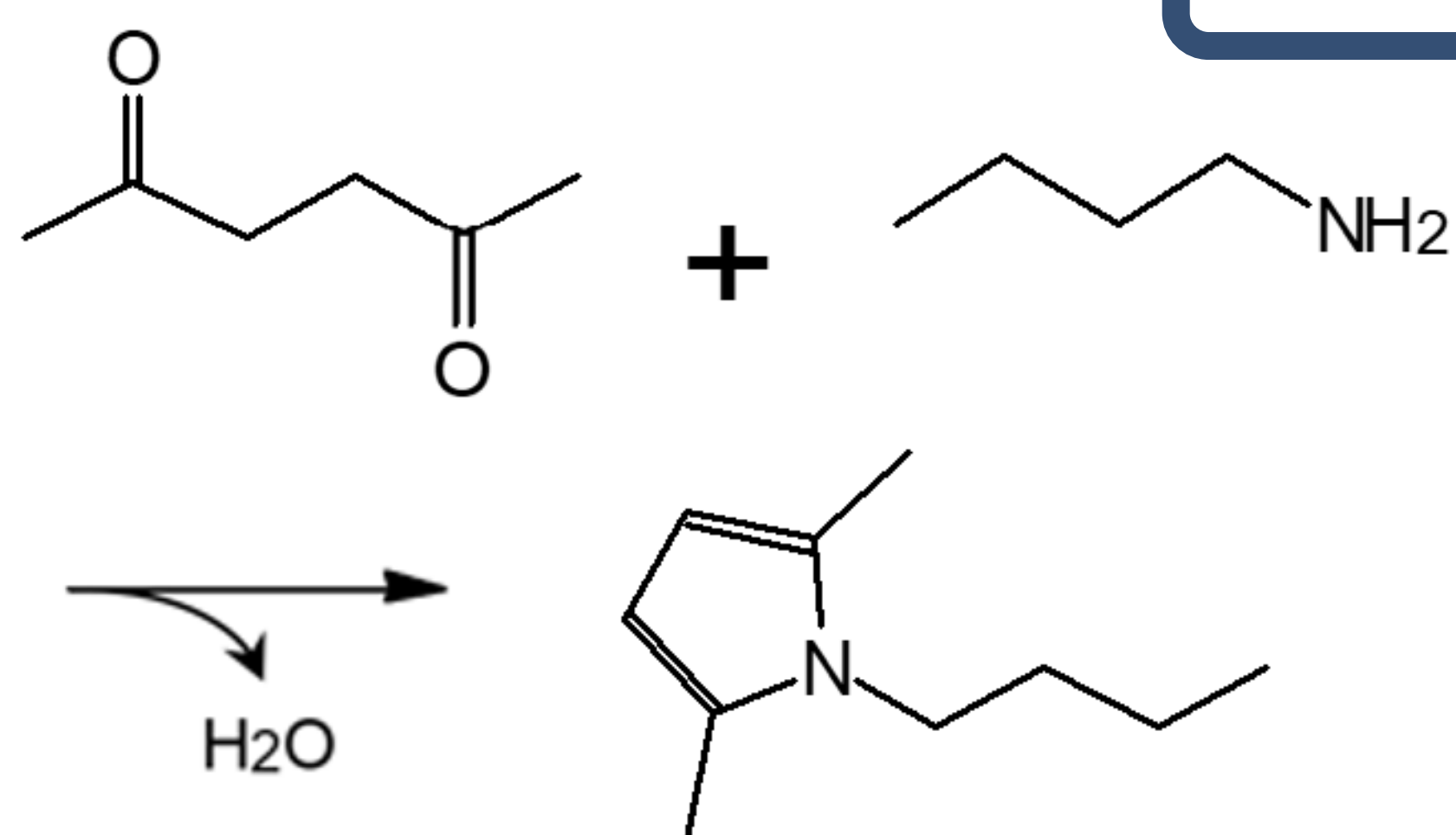


## Modular Design

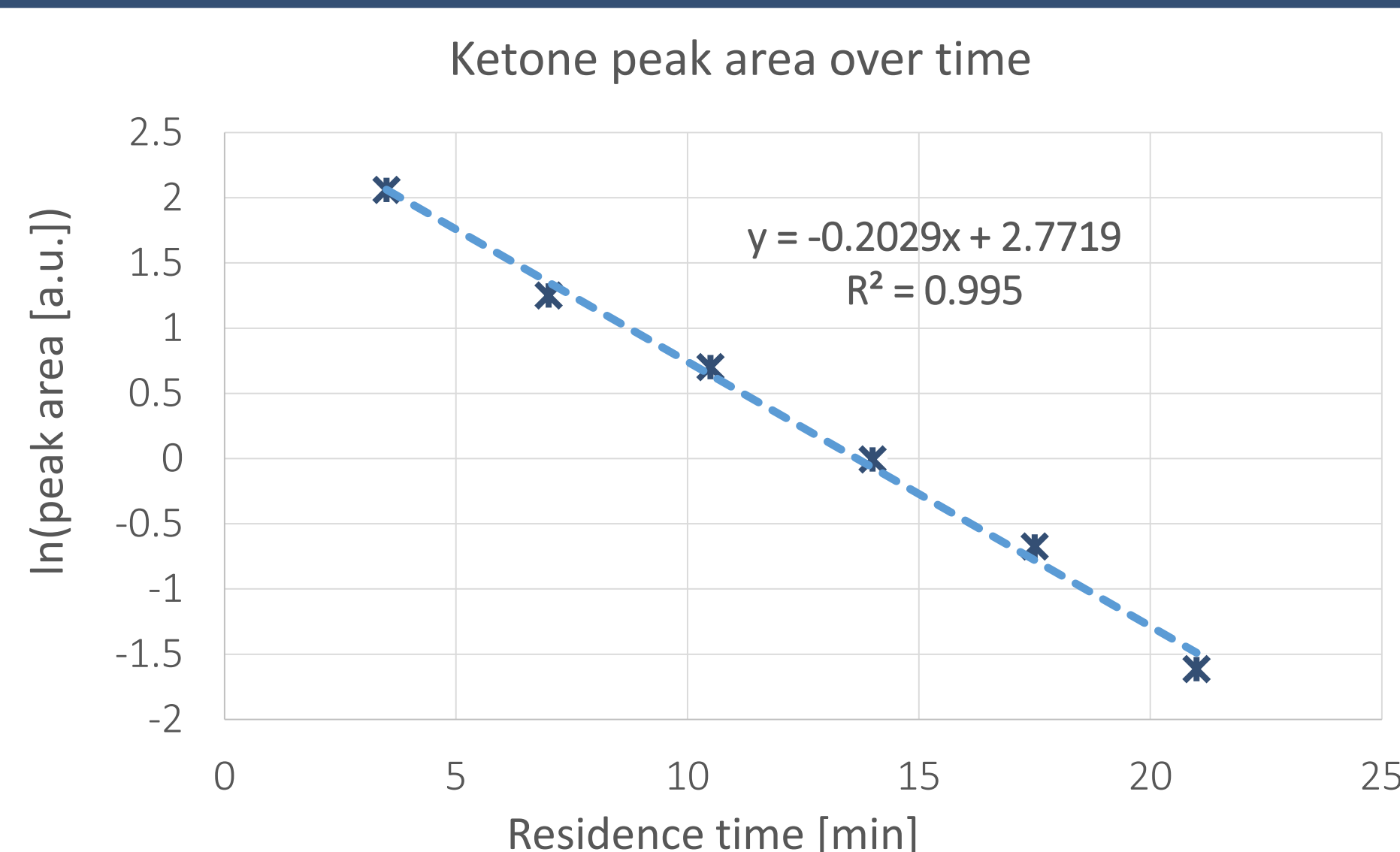


Modular design of the chip allows for a variety of reactions to be monitored. The simplest combination (left) has been used to obtain the results presented in this poster. A more complex design (right), can currently use up to 3 layers. Parallelization with multiple chips, could also increase the amount of reaction components.

## Reaction Monitoring

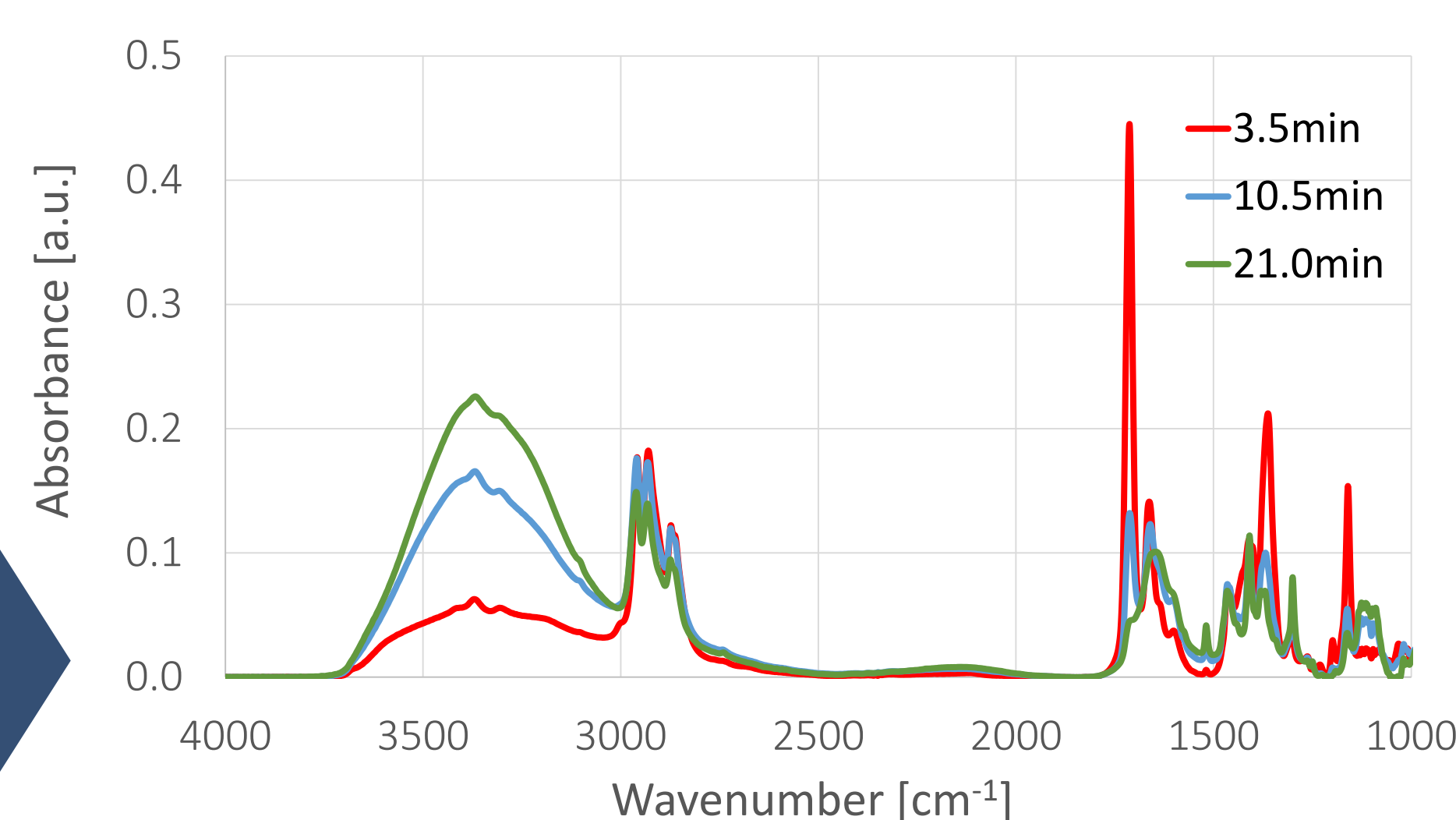


Synthesis of 1-butyl-2,5-dimethyl-1H-pyrrole from hexane-2,5-dione and 1-butylamine. The reactants are mixed by the herringbone mixer, flow is stopped once the reaction chamber is filled. Next, every 3.5min a IR spectrum is generated.



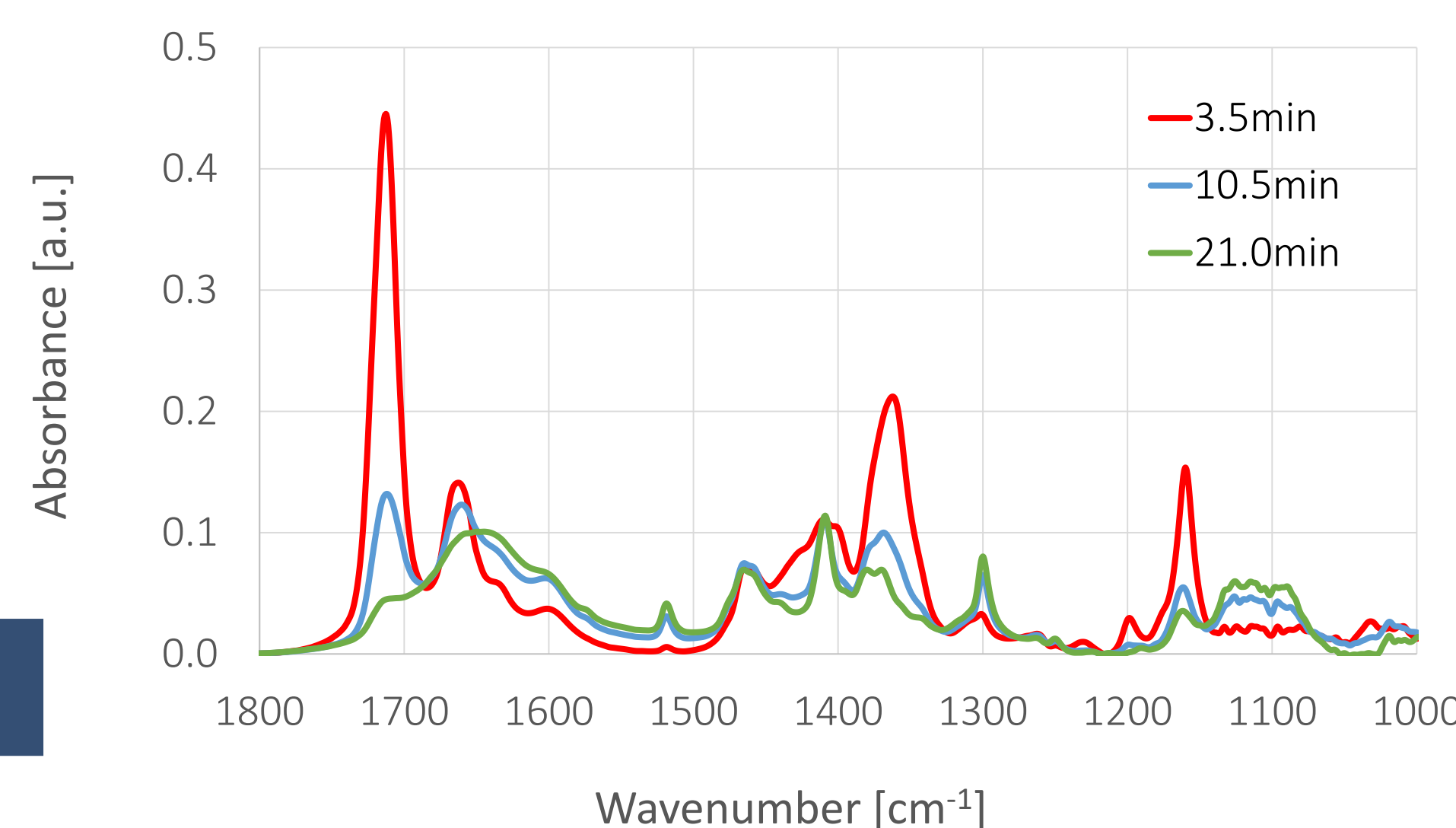
The natural logarithm of the peak area at  $1710\text{cm}^{-1}$ , plotted against the residence time. The linear fit suggests a first order reaction.

IR spectra over time



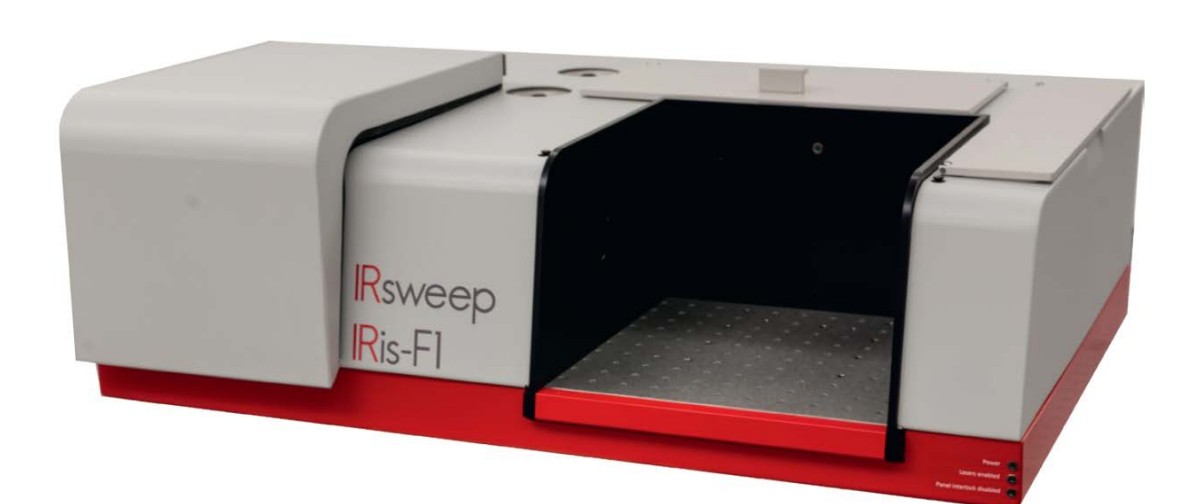
FT-IR spectra over time. The broad peak at  $3200\text{cm}^{-1}$ , corresponding to the formation of water, increases over time.

Fingerprint region



FT-IR spectra at  $1800\text{--}1000\text{cm}^{-1}$ . The ketone peak at  $1710\text{cm}^{-1}$  decreases over time, while the peaks at  $1525\text{cm}^{-1}$  (skeleton vibration) and  $1300\text{cm}^{-1}$  (C-N stretch) increase overtime<sup>5,6</sup>.

## Outlook



The current setup is lacking in analysis speed. Therefore a new IR spectrometer, using a tunable quantum cascade laser has been purchased from IRSweep. The laser, which not only has a time resolution in the microsecond range, also has a higher power output, increasing signal to noise.

## Acknowledgement

We would like to thank Max Krakers for the design of the herringbone mixer mask.

We would like to thank Tobias Elsbecker for fabricating the PMMA, Teflon and COC reaction chamber and mixer channels, and his to continuing contribution to the project.

## References

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