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Process-suited Raman Spectroscopy with High Optic Collection Efficiency for In-line Analytics

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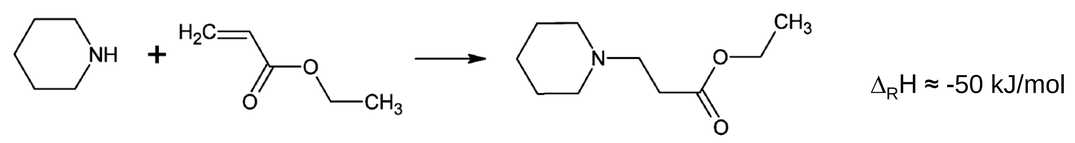
INTRODUCTION

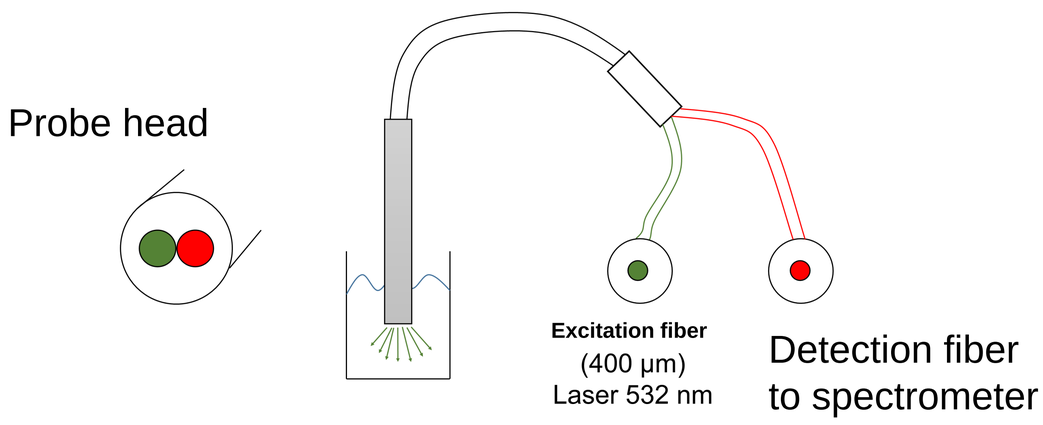
- **Established off-line methods for industrial monitoring of chemical reactions increasingly replaced by optical in-line methods**
  - Application of in-line Raman spectroscopy with fiber-optic probe
  - Significant time and material savings compared to off-line analytics (eliminating additional work steps)
  - No safety or contamination risks during handling
- **Spectrometer optics optimized for maximum photon flux**
  - Rapid sampling rates despite reduced laser output
- **Michael addition as a quick sample reaction with known kinetics [1]**
  - Rapid recording of time conversion curves in the batch reactor
  - Low excitation power (7 mW)



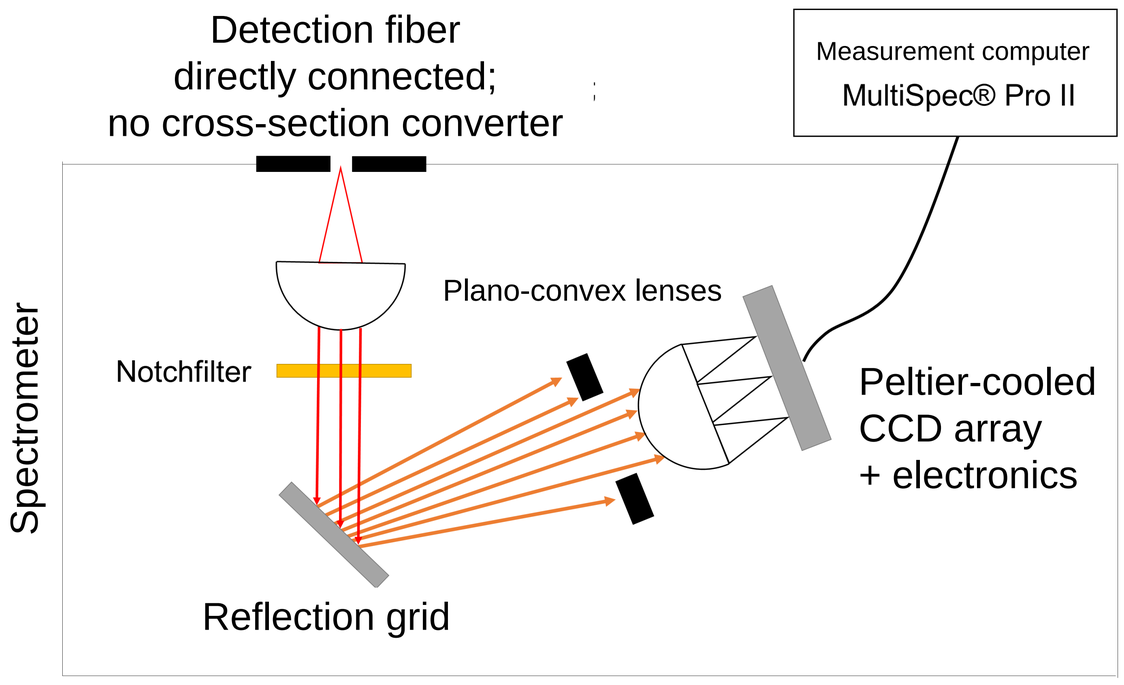
Piperidine | Acrylic acid ethyl ester | 3-Piperidino-propionic acid ethyl ester

MEASUREMENT TECHNOLOGY

- **Backscatter probe, immersed (no focusing optics)**

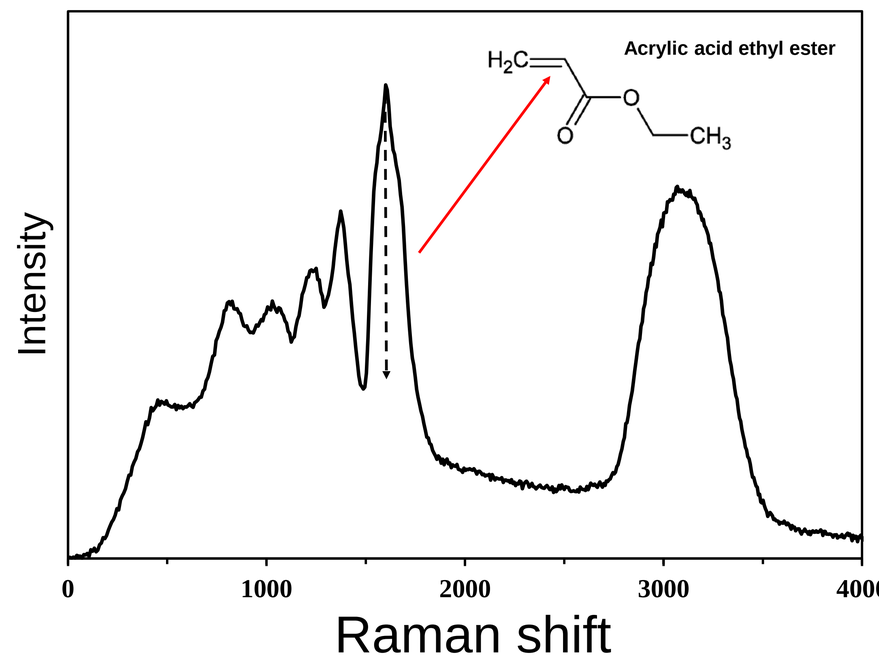


- **Raman spectrometer optics (custom-built) suited to the application**
  - **Laser light source**
    Excitation wavelength: 532 nm -> limitation: fluorescence background
  - **Spectrometer resolution**
    Variable through the use of different detection fibers
  - **Process suitability**
    Robust; spectrometer without movable components and no filters, mirrors, or lenses in the probe

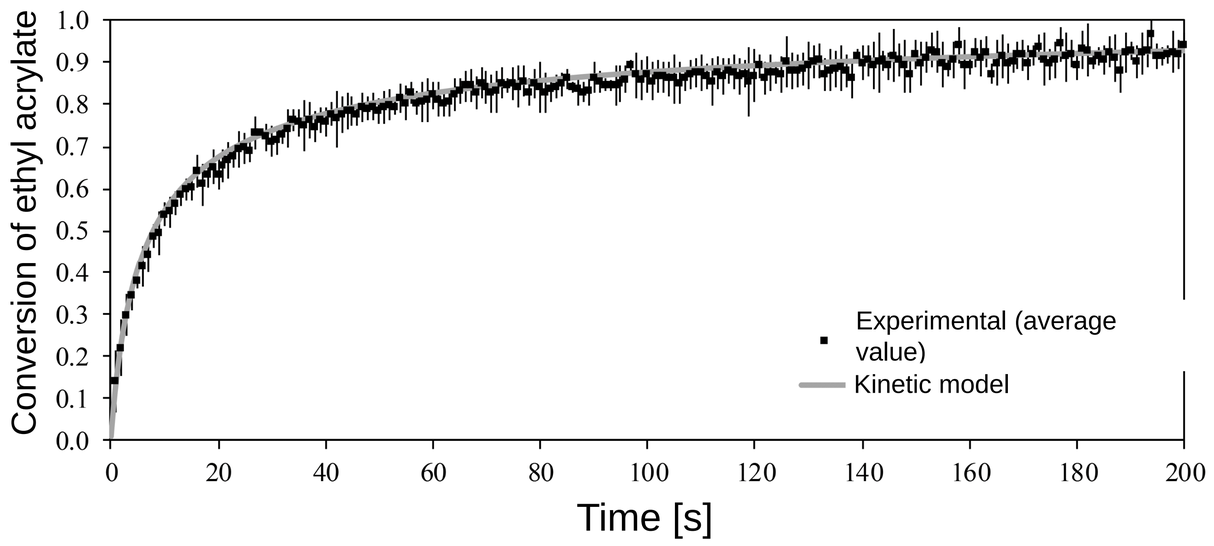


RESULTS

- **Measurements in the batch reactor under backscattering**
  - Excitation power: 7 mW; integration time: 1000 ms; accumulation: 1
  - Evaluation by vinyl band at 1637 cm^{-1}



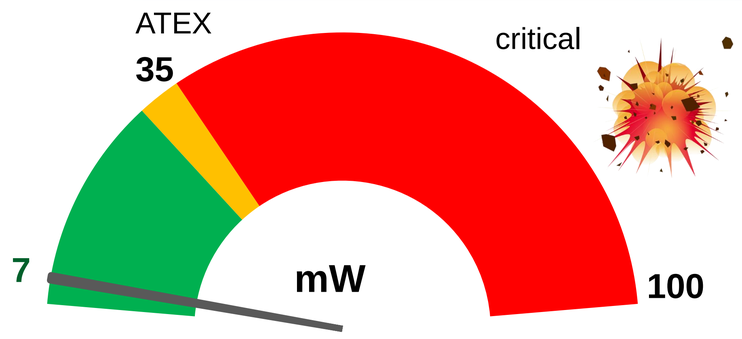
- **Monitoring a Michael addition reaction (n=5)**
  - Raman measurements and comparison with kinetic model



  - Good agreement with kinetic model based on tests with GC off-line analytics [2]

CONCLUSION

- **Realization of spectrometer optics optimized for maximum photon flux**
- **Efficient methods for rapid measurement of kinetics through in-line reaction monitoring**
- **Kinetics of model reaction monitoring confirmed by conventional Raman measurement technique [1] and GC analysis [2]**
- **Allowable continuous power output for laser light sources in explosion-protected areas as per ATEX (DIN EN 60079-28) of 7 mW is below limit by a factor of 5 -> great potential in operational practice**
- **Range of applications can markedly expand in conjunction with chemometric methods**



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Literature

[1] Schwolow, S., Braun, F., Rädle, M., Kockmann, N., Röder, T., 2015. Fast and Efficient Acquisition of Kinetic Data in Microreactors Using In-Line Raman Analysis. Org. Process Res. Dev..
[2] Schwolow, S., Heikenwälder, B., Abahmane, L., Kockmann, N., Röder, T., 2014 . Kinetic and Scale-up Investigations of a Michael Addition in Microreactors. Org. Process Res. Dev.