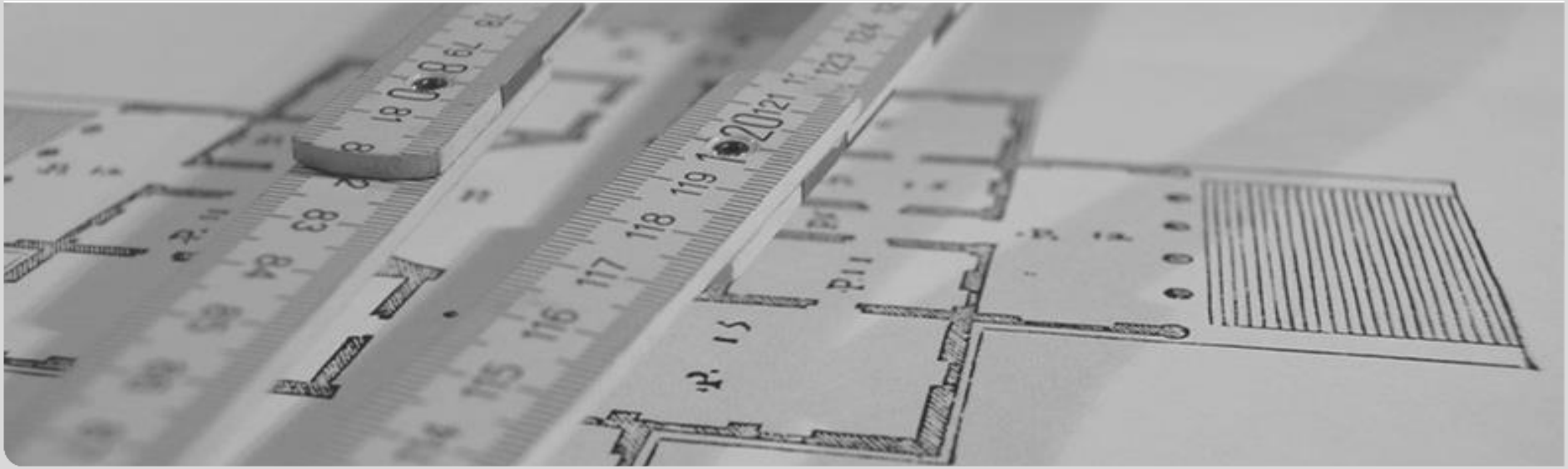
Karlsruhe Institute of Technology

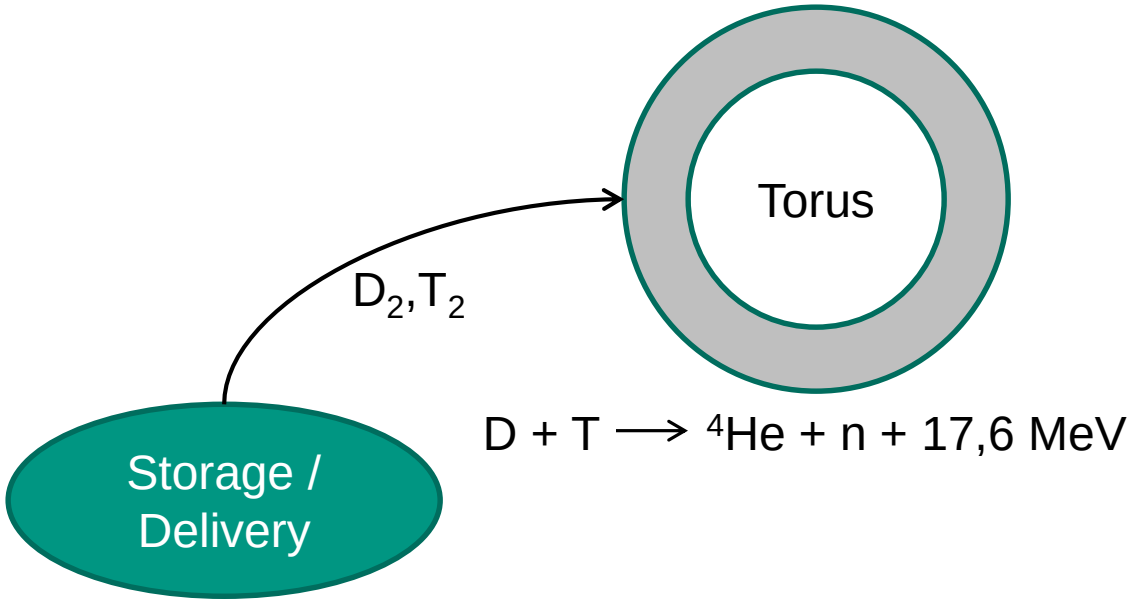
Production and Utilisation of highly concentrated HD for the Validation of the Calibration of Hydrogen Analysis Systems at the Tritium Laboratory Karlsruhe

Presentation of Tim Brunst in the context of his Master‘s Thesis

Institute for Technical Physics – Tritium Laboratory Karlsruhe



Closed tritium cycle of fusion reactors



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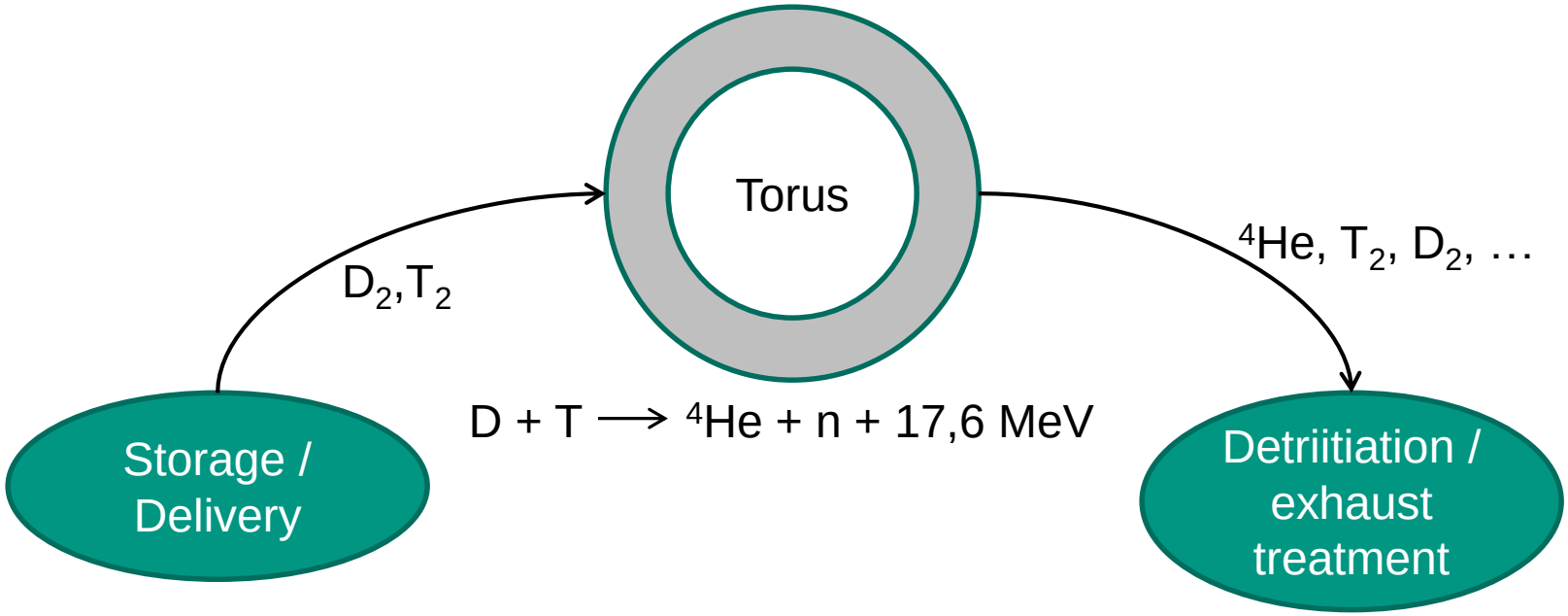

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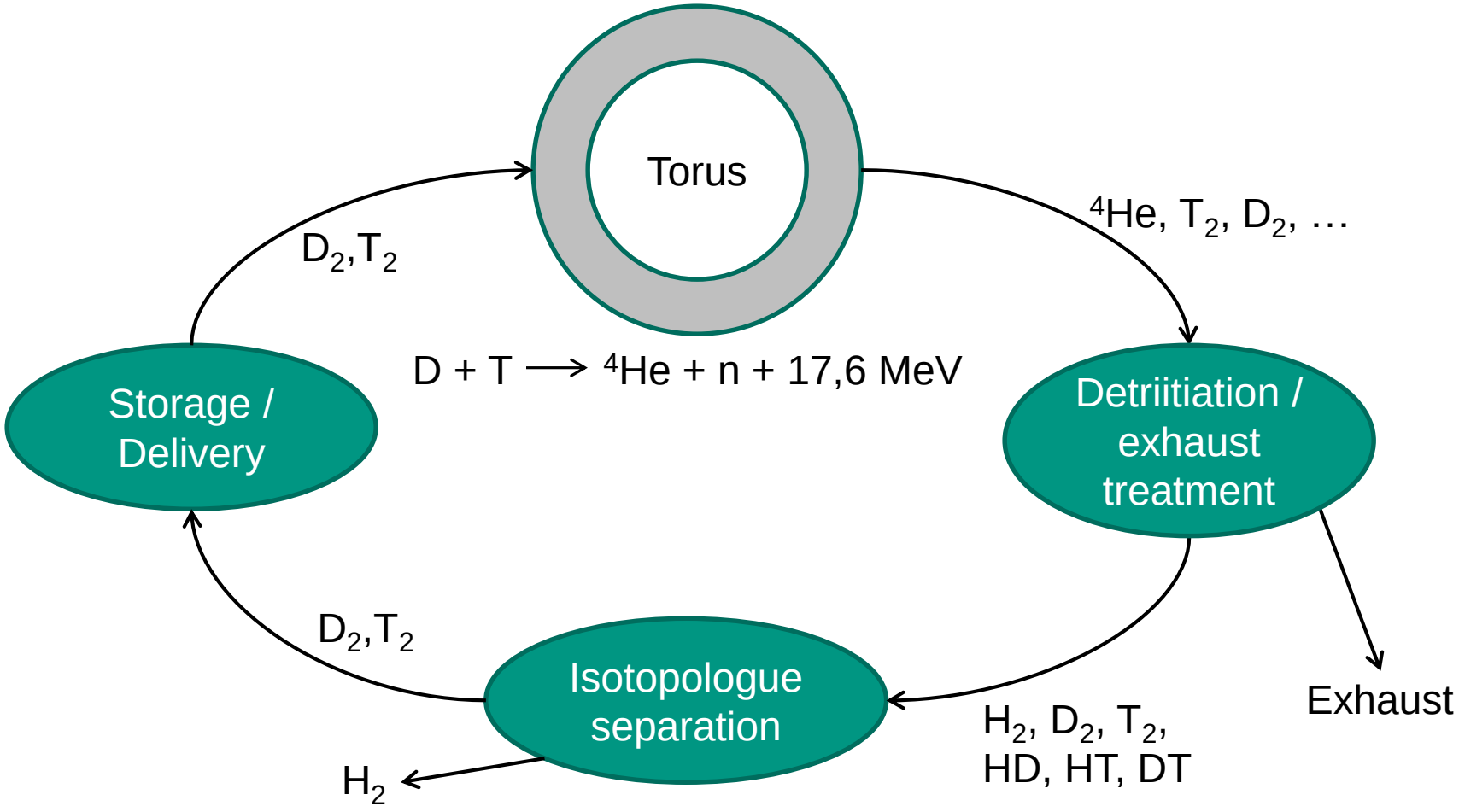

Large amounts (~kg/h) of hydrogen (H, D, T) must be recycled continuously.

How do we separate hydrogen isotopologues?

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Cryogenic distillation.

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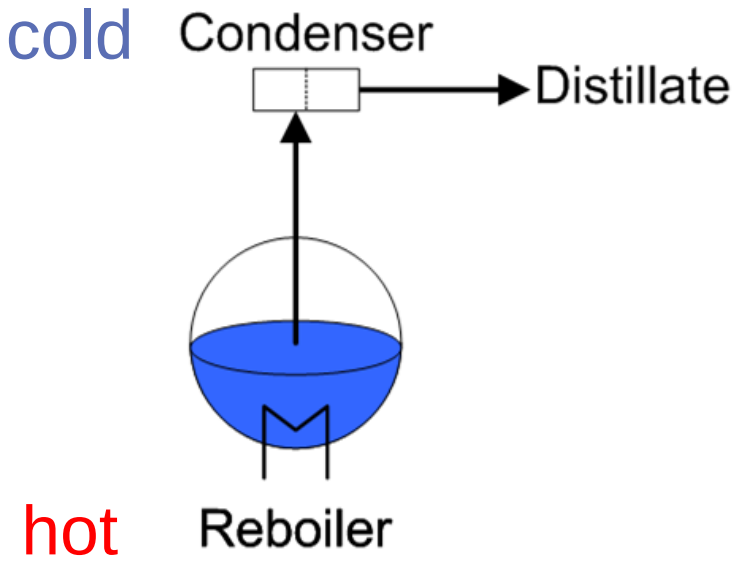
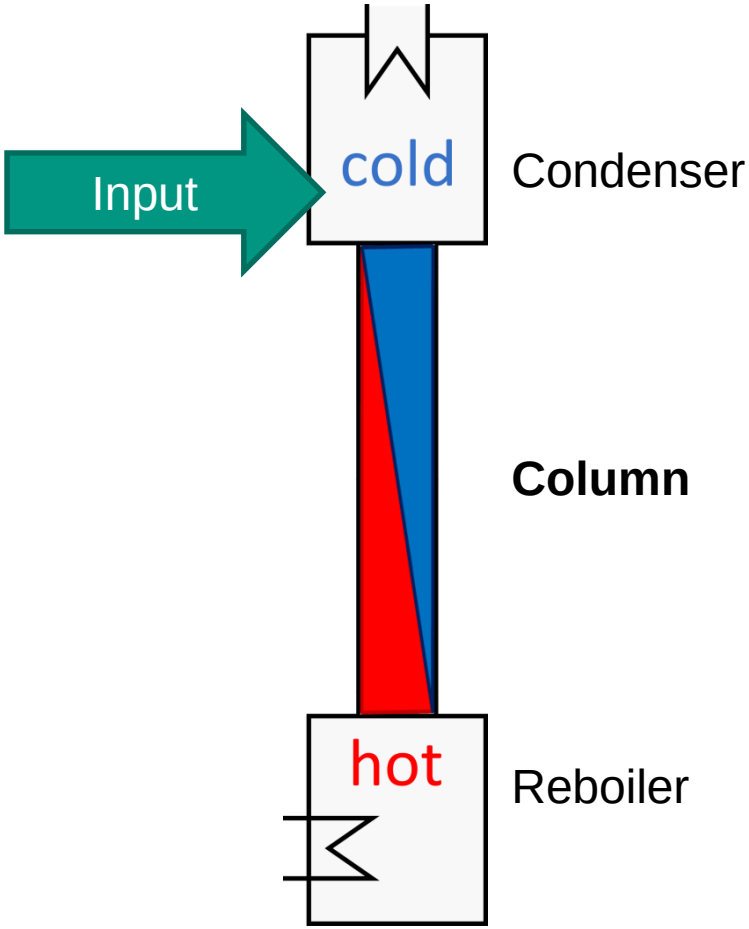
Cryogenic distillation.

Tritium Enrichment Test Assembly (TRENTA)

TRENTA and cryogenic distillation



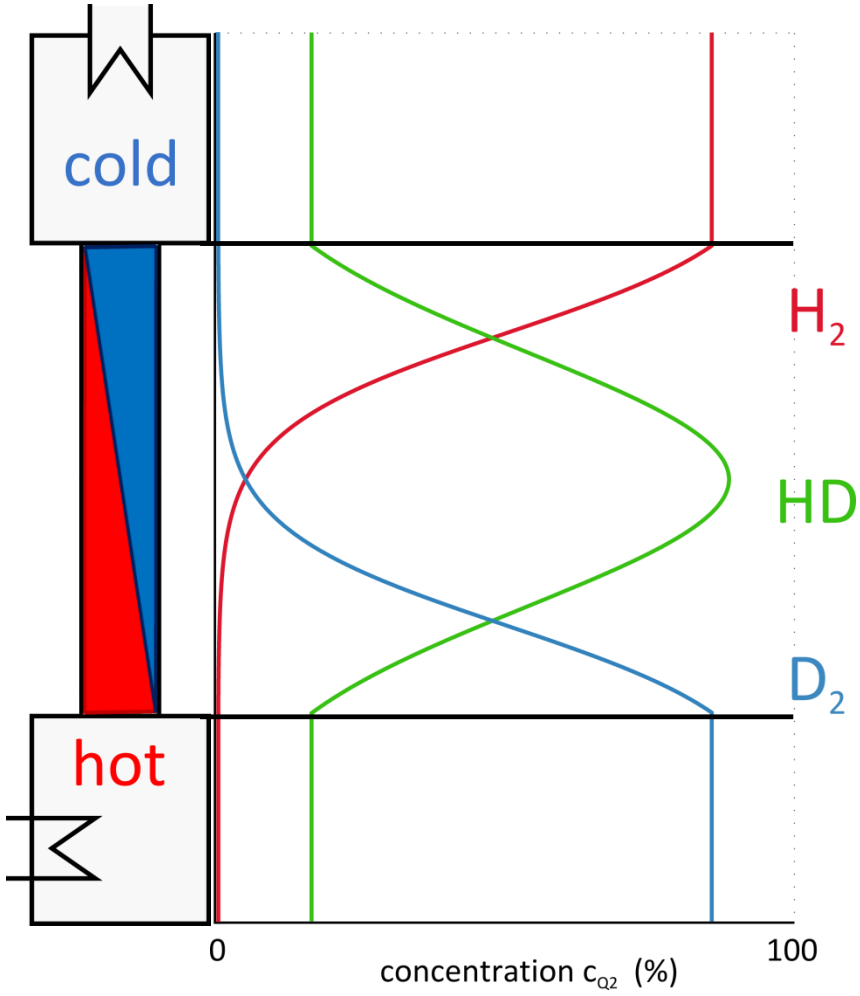
TRENTA and cryogenic distillation

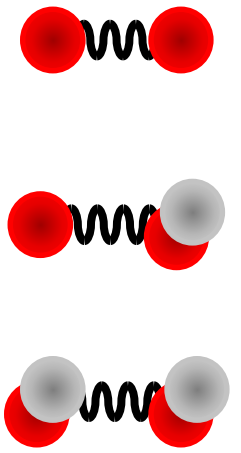


Improved separation through several distillation steps

TRENTA and cryogenic distillation

Isot.	T_B /K (1 bar)
H_2	20.3
HD	22.1
HT	22.9
D_2	23.6
DT	24.4
T_2	25.0





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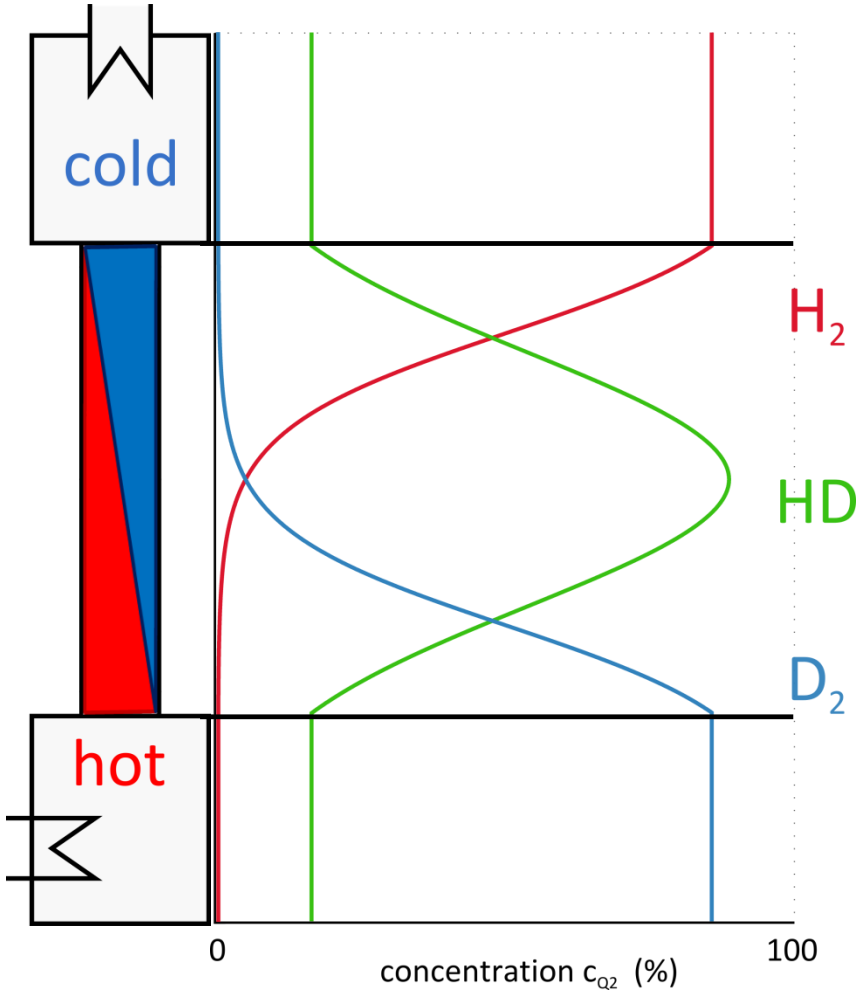

Every isotopologue can occur highly concentrated

Continuous, real-time monitoring of column content necessary

How do we monitor the column content?

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Infrared spectroscopy.

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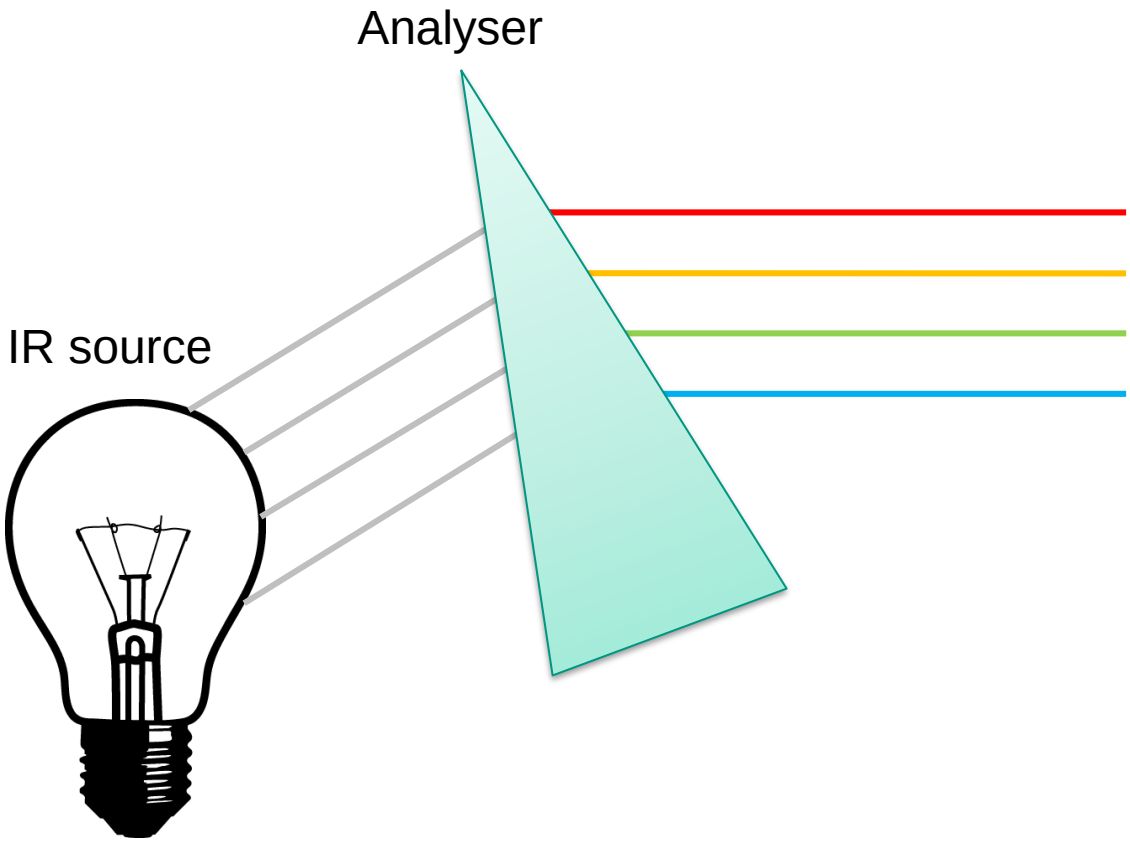
Infrared spectroscopy.

Tritium Absorption Infrared Experiment (TApIR)

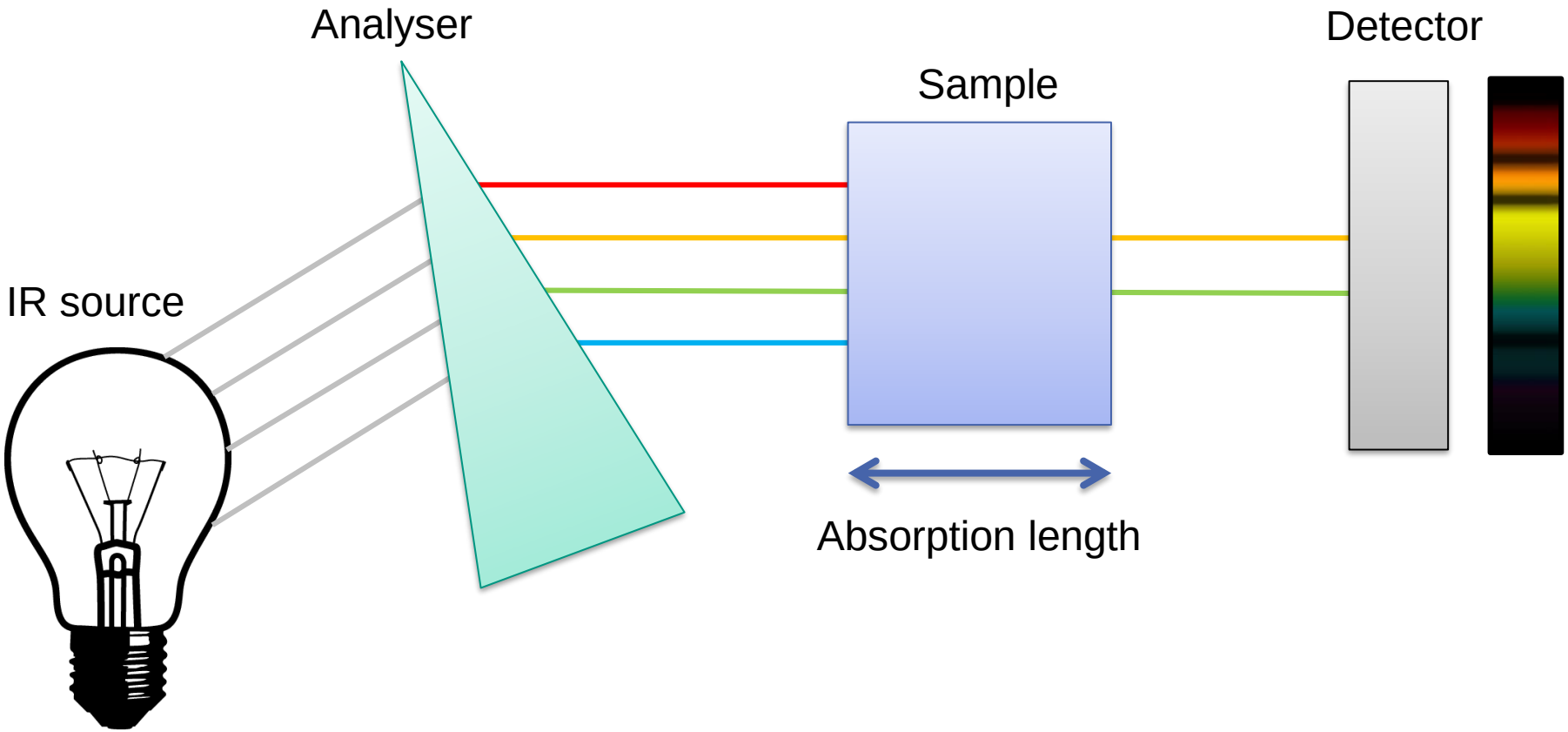
Monitoring with TApIR



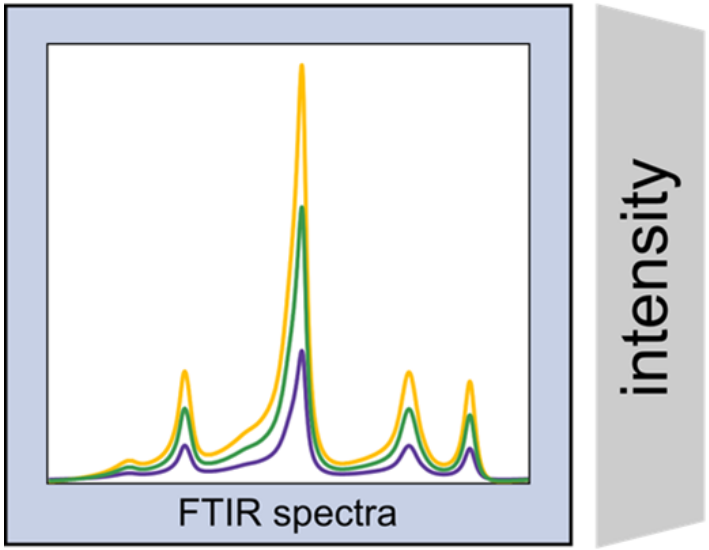
„white“ light

Total spectrum

Monitoring with TAplR



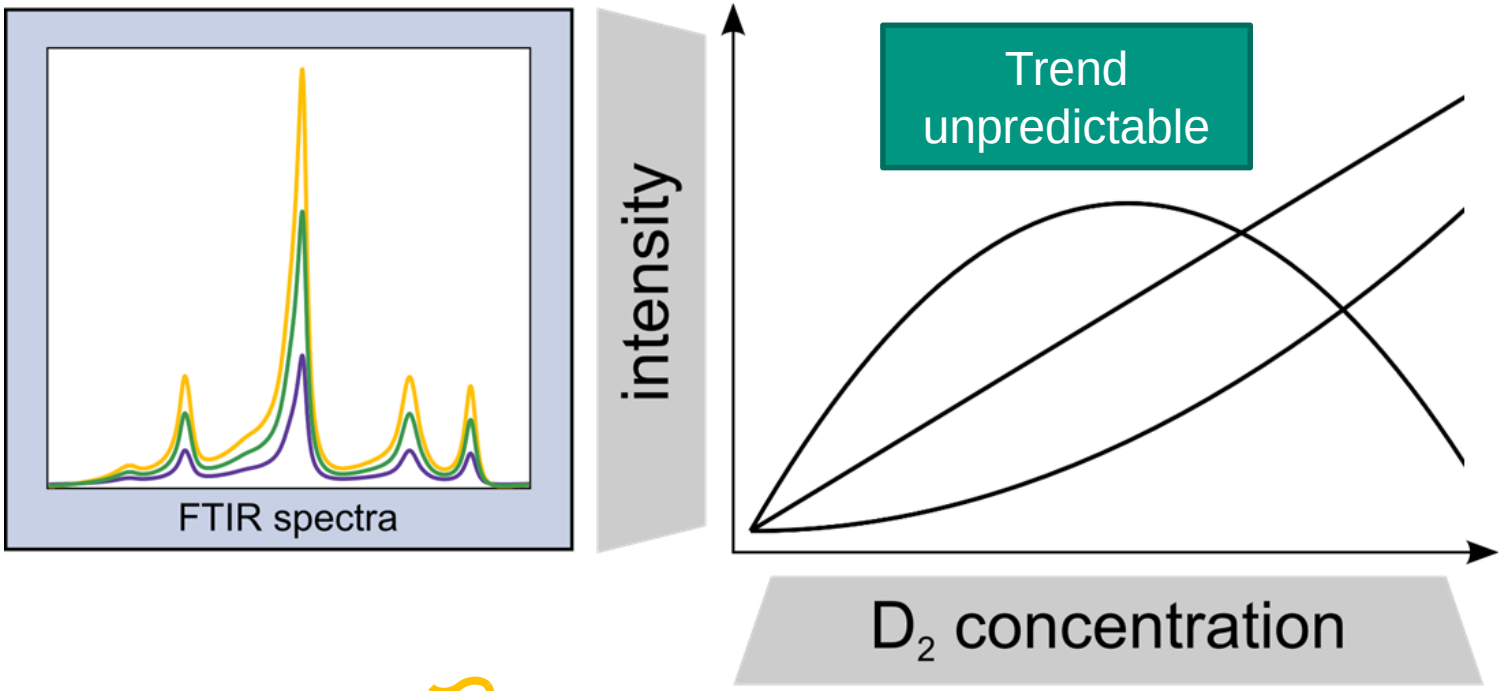
Monitoring with TAplR

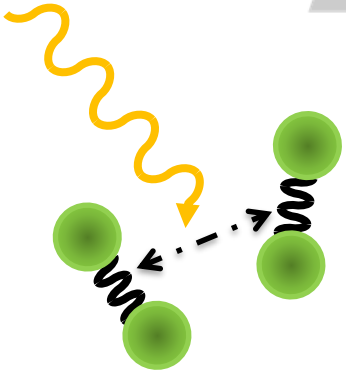


concentration of isotopologues

type of isotopologues

Non-linear calibration functions





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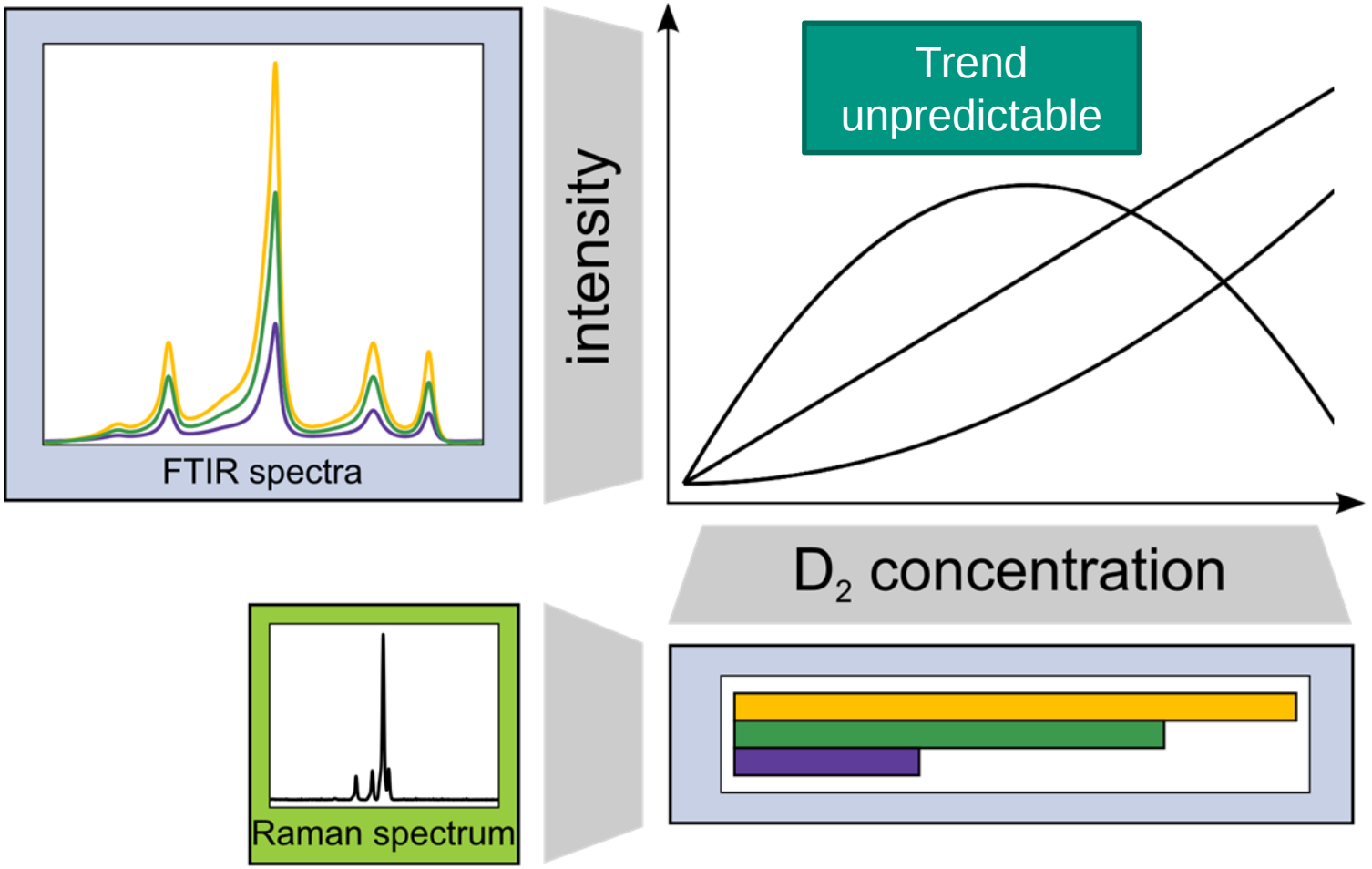
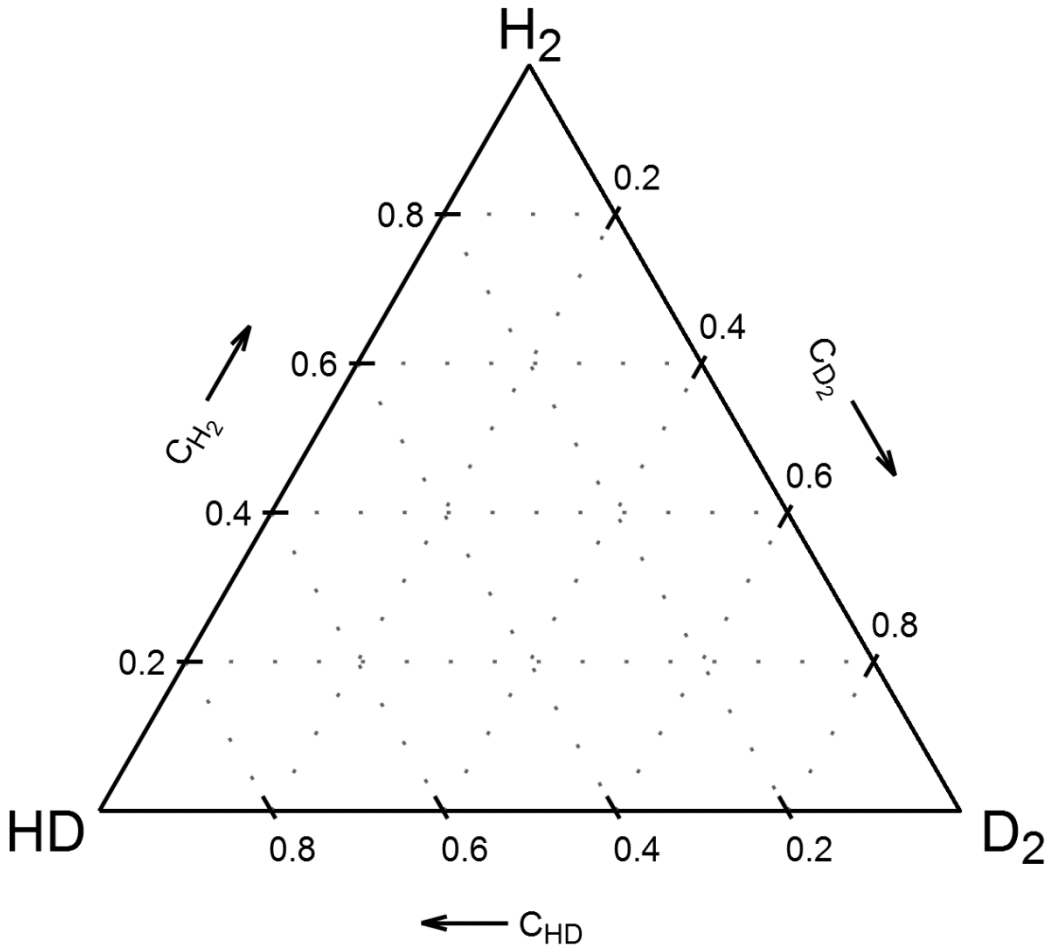


Calibration spectra of the entire phase space of concentrations

Calibration with reference samples



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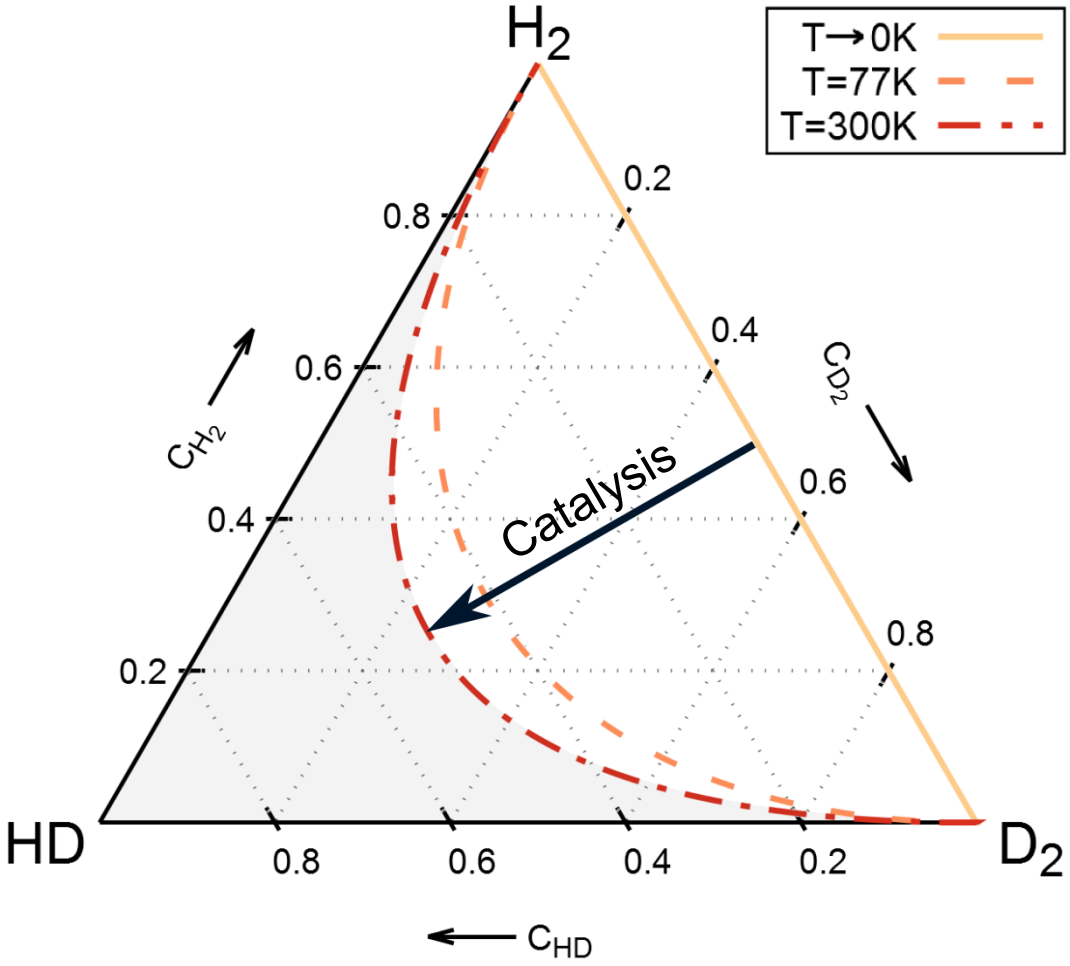

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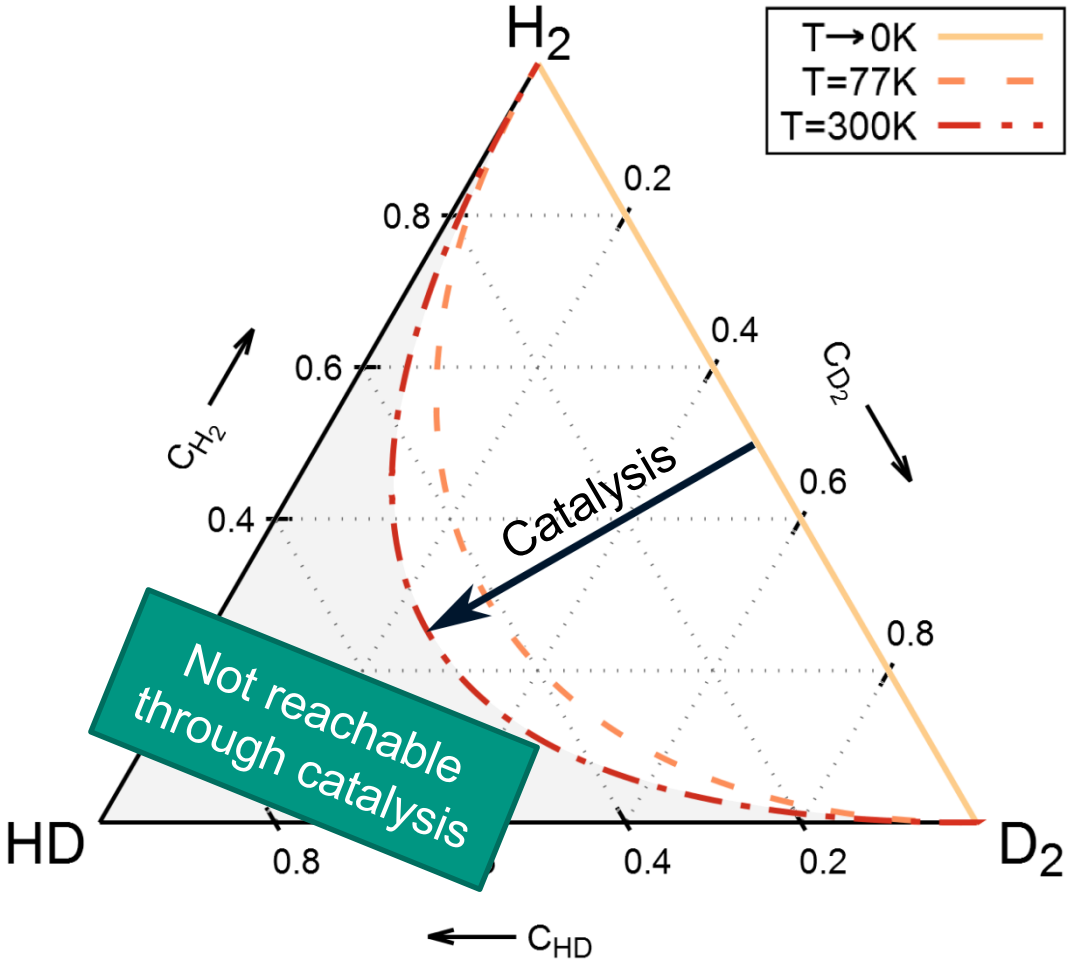

Calibration through extrapolation to highly concentrated HD

Objectives

- Production of highly concentrated HD (>50%)
  - ➡ Cryogenic distillation with TRENTA

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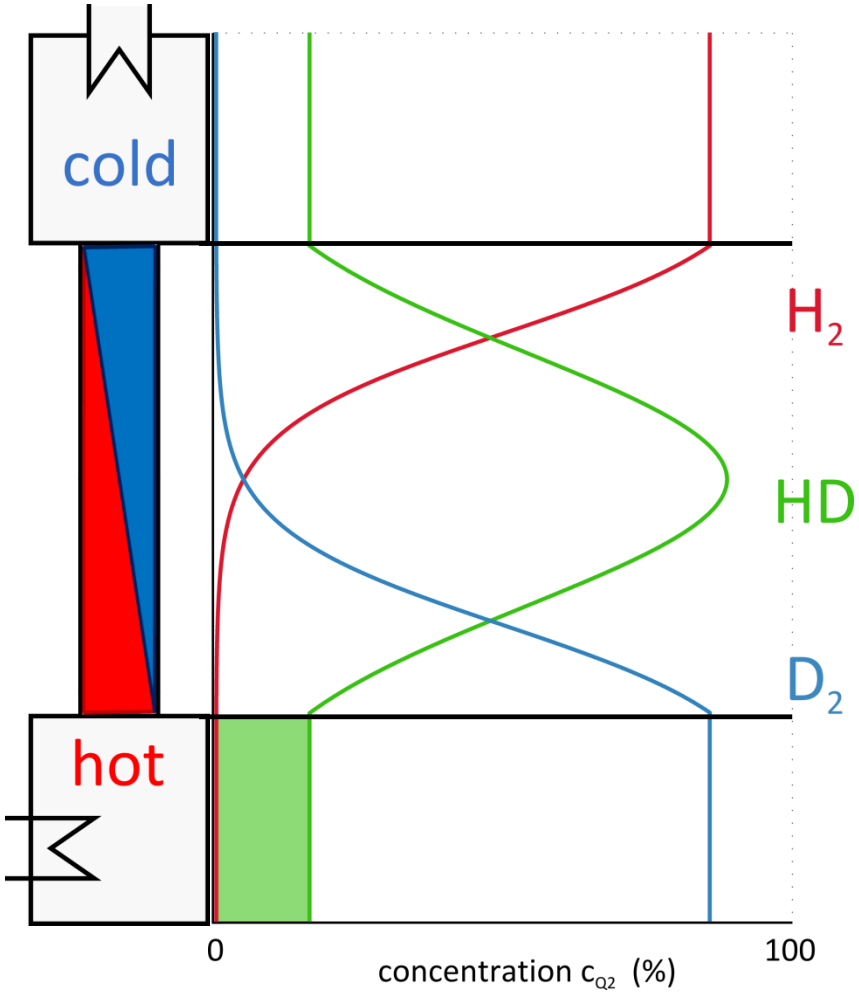
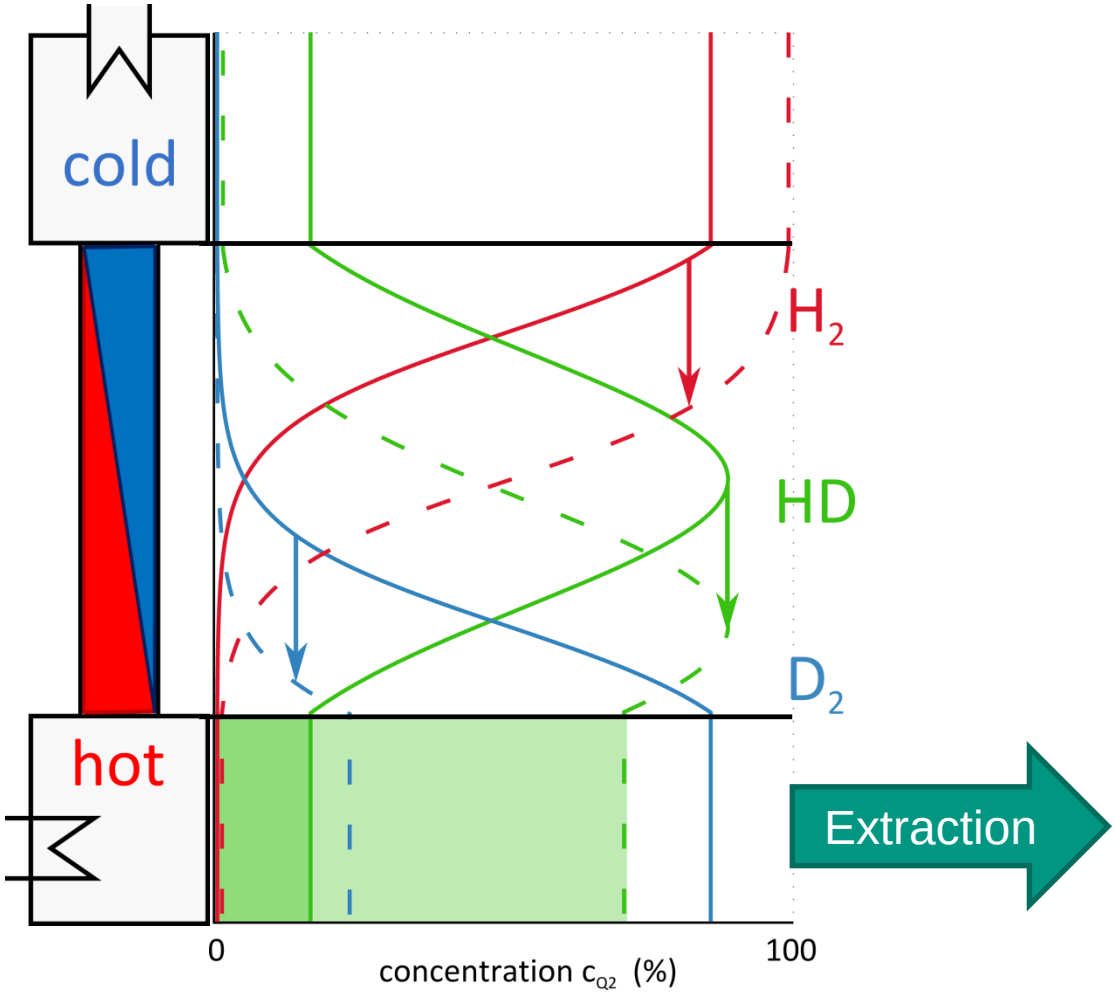
- Stability analysis of highly concentrated HD
  - ➡ Long-term measurements in gaseous and liquid phase

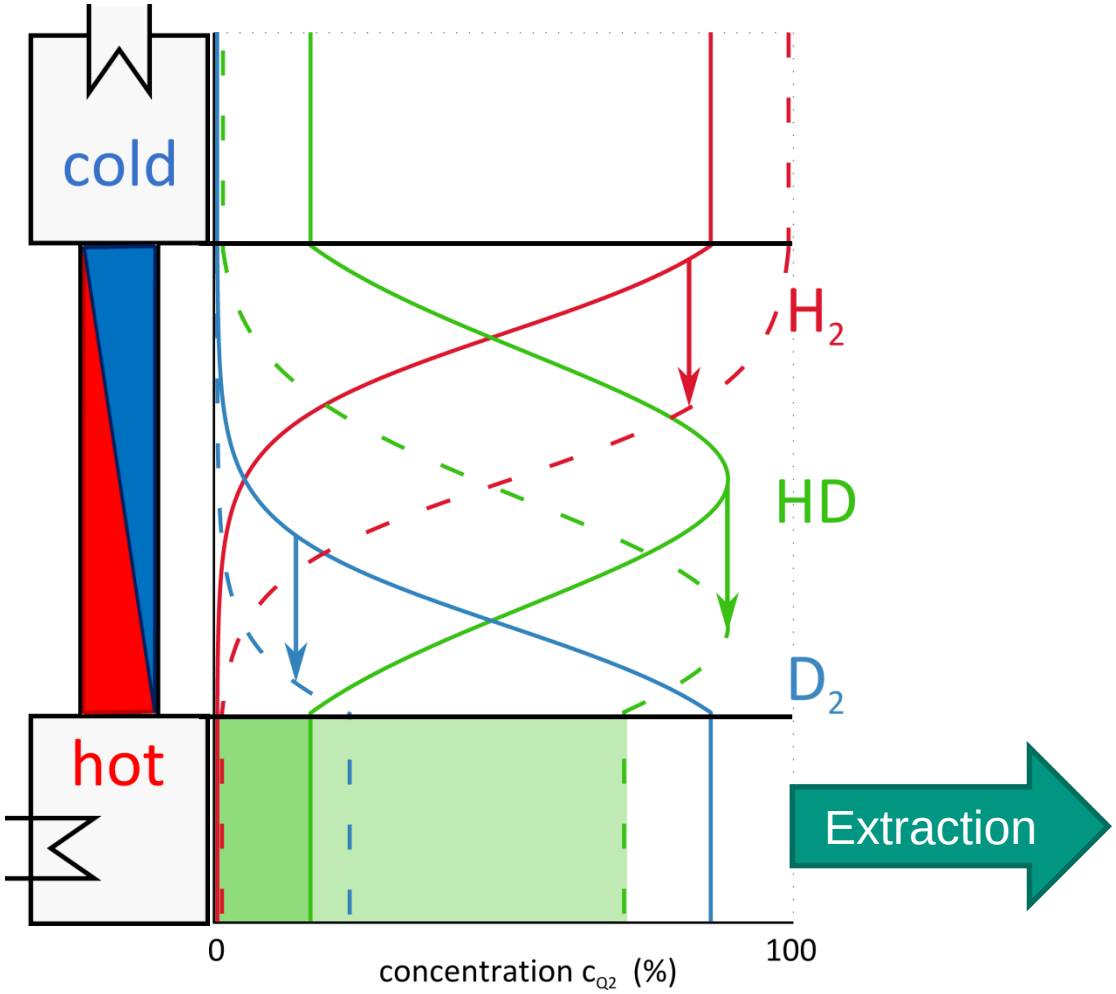
Production of highly concentrated HD



Production of highly concentrated HD



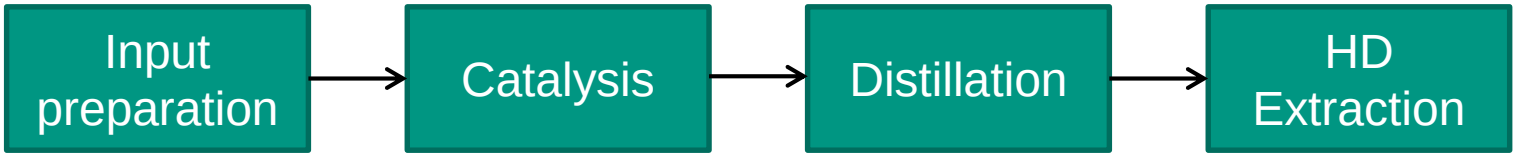
Production of highly concentrated HD



Simulation to determine operating parameters

Production of highly concentrated HD

RUN A



Production of highly concentrated HD

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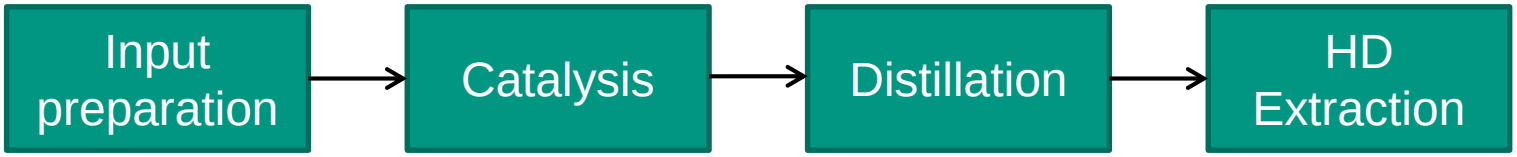

RUN	Amount (l_s)	HD concentration (%)
A	138	80.1
B	150	85.8

Goal achieved: HD concentration >50%

Objectives

- Production of highly concentrated HD (>50%)
  - ➡ Cryogenic distillation with TRENTA

- Verification of extrapolated calibration of TApIR
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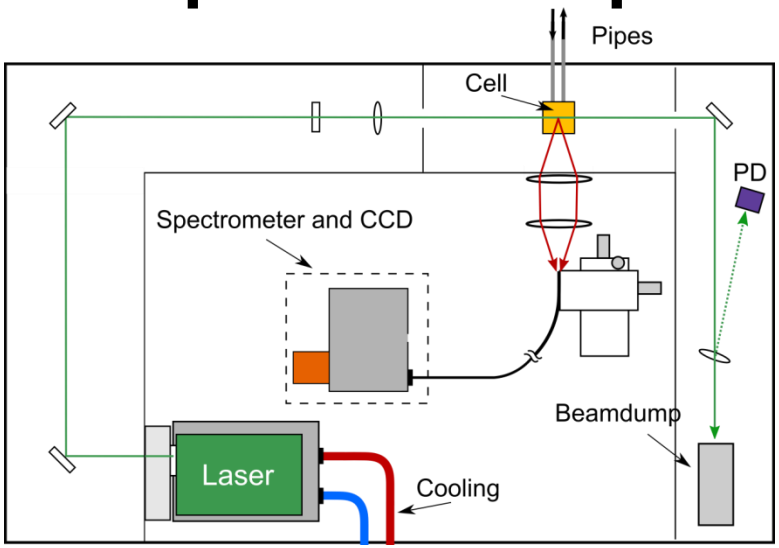
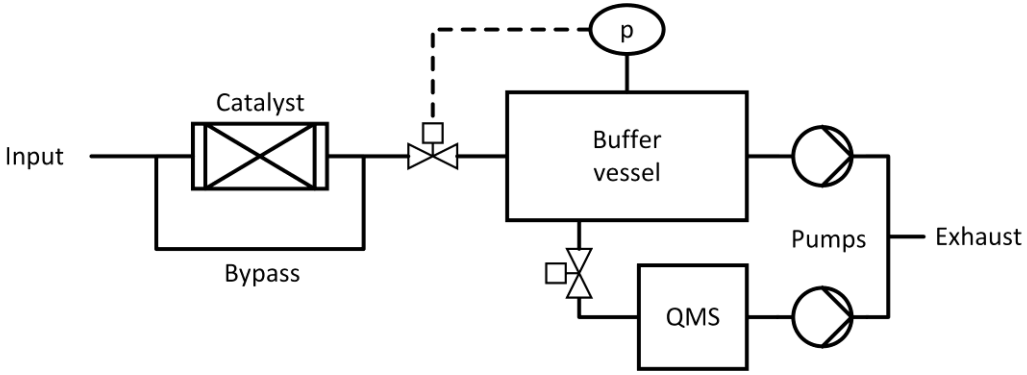
- Stability analysis of highly concentrated HD
  - ➡ Long-term measurements in gaseous and liquid phase

Comparison of spectroscopic methods





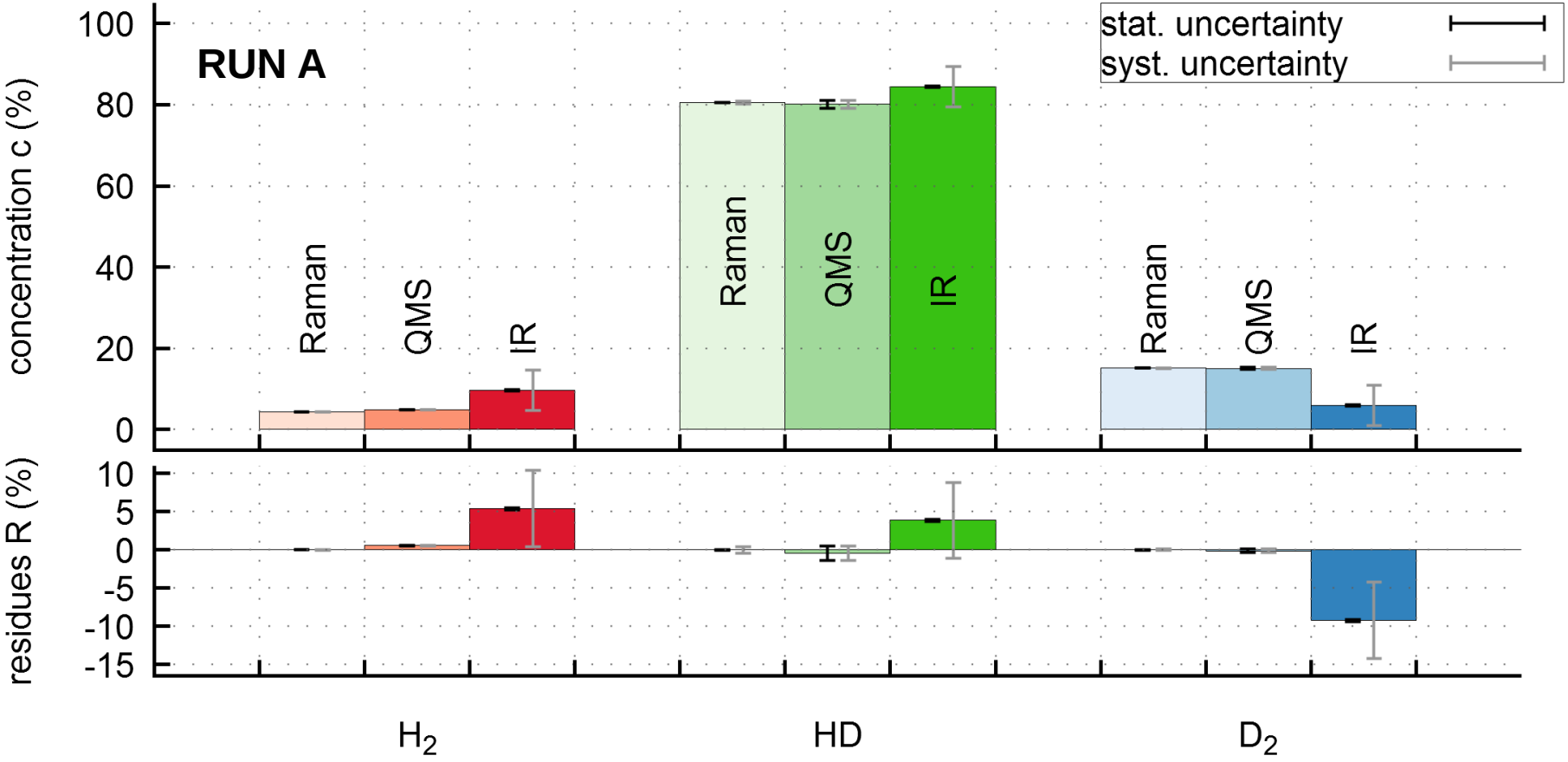
Raman System

- Gaseous sample in Raman cell
- Raman scattering of the laser photons
- Spectroscopic investigation of scattered light

Quadrupole Mass Spectrometer

- Extraction of 3 l/h from the sample
- Ionisation of isotopologues
- Mass-to-charge ratio selection in an electrical field

Comparison of spectroscopic methods



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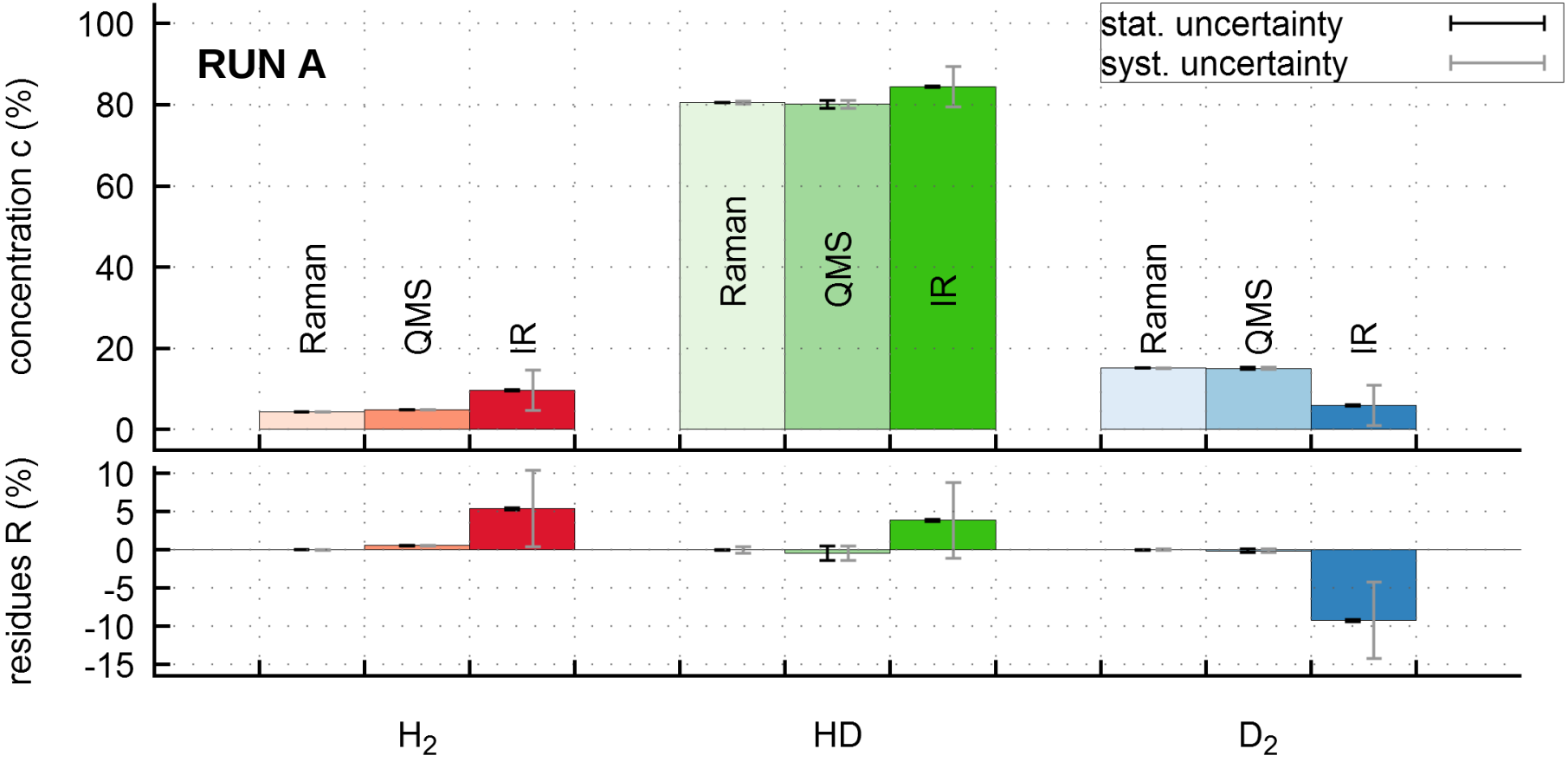

Usage of extrapolated calibration leads to deviations.
IR calibration has to be expanded for highly concentrated HD.

Summary

- Simulation of TRENTA cryogenic distillation performance with non-radioactive isotopologues developed

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- Three relevant spectroscopic methods at TLK compared and necessity for expansion of IR calibration identified

Summary

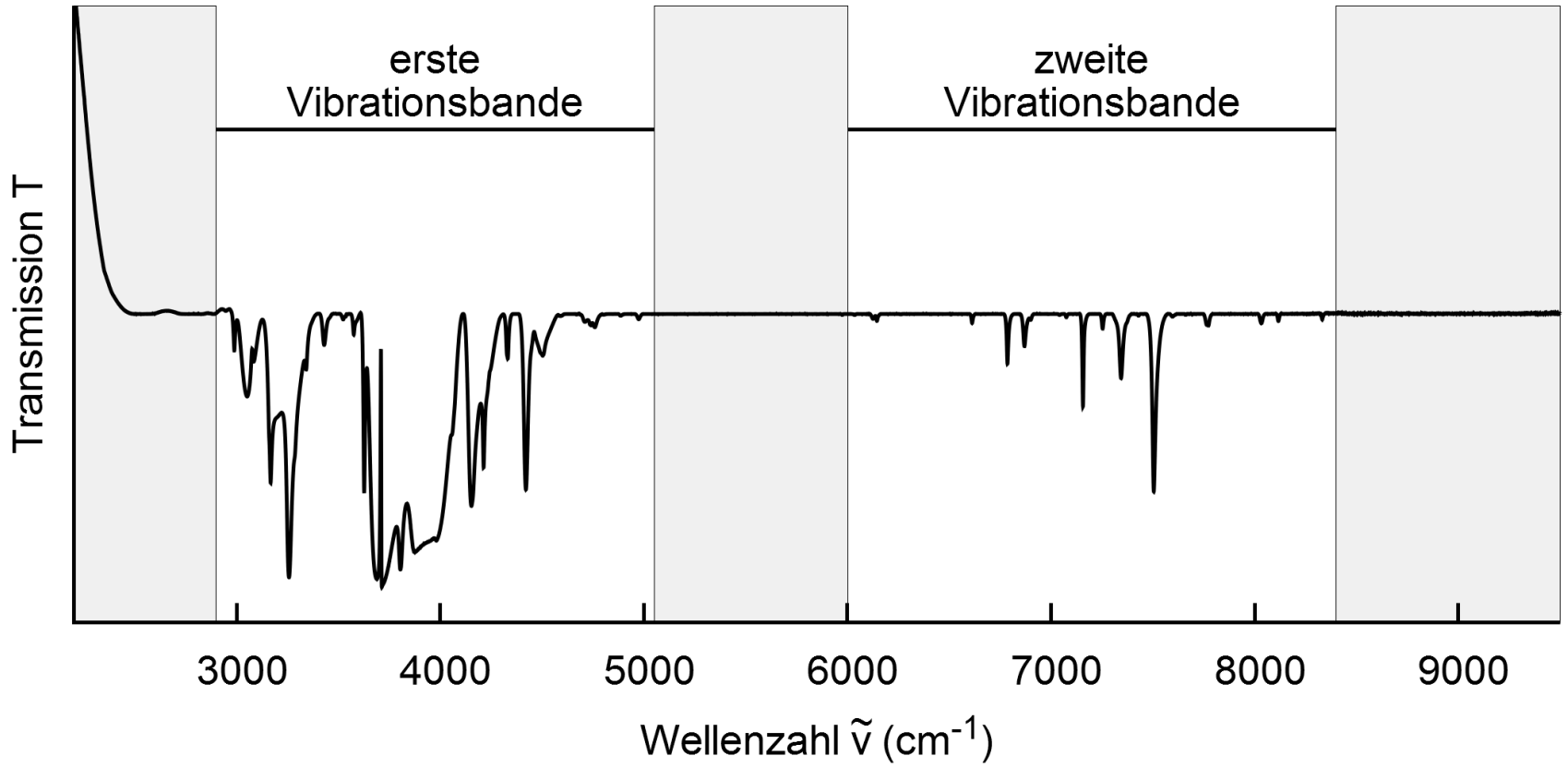
- Simulation of TRENTA cryogenic distillation performance with non-radioactive isotopologues developed
- Method for the production of large amounts of highly concentrated HD with TRENTA developed
- Three relevant spectroscopic methods at TLK compared and necessity for expansion of IR calibration identified
- Worldwide first two data points of highly concentrated HD in liquid phase for IR calibration measured

Thank you for your attention.

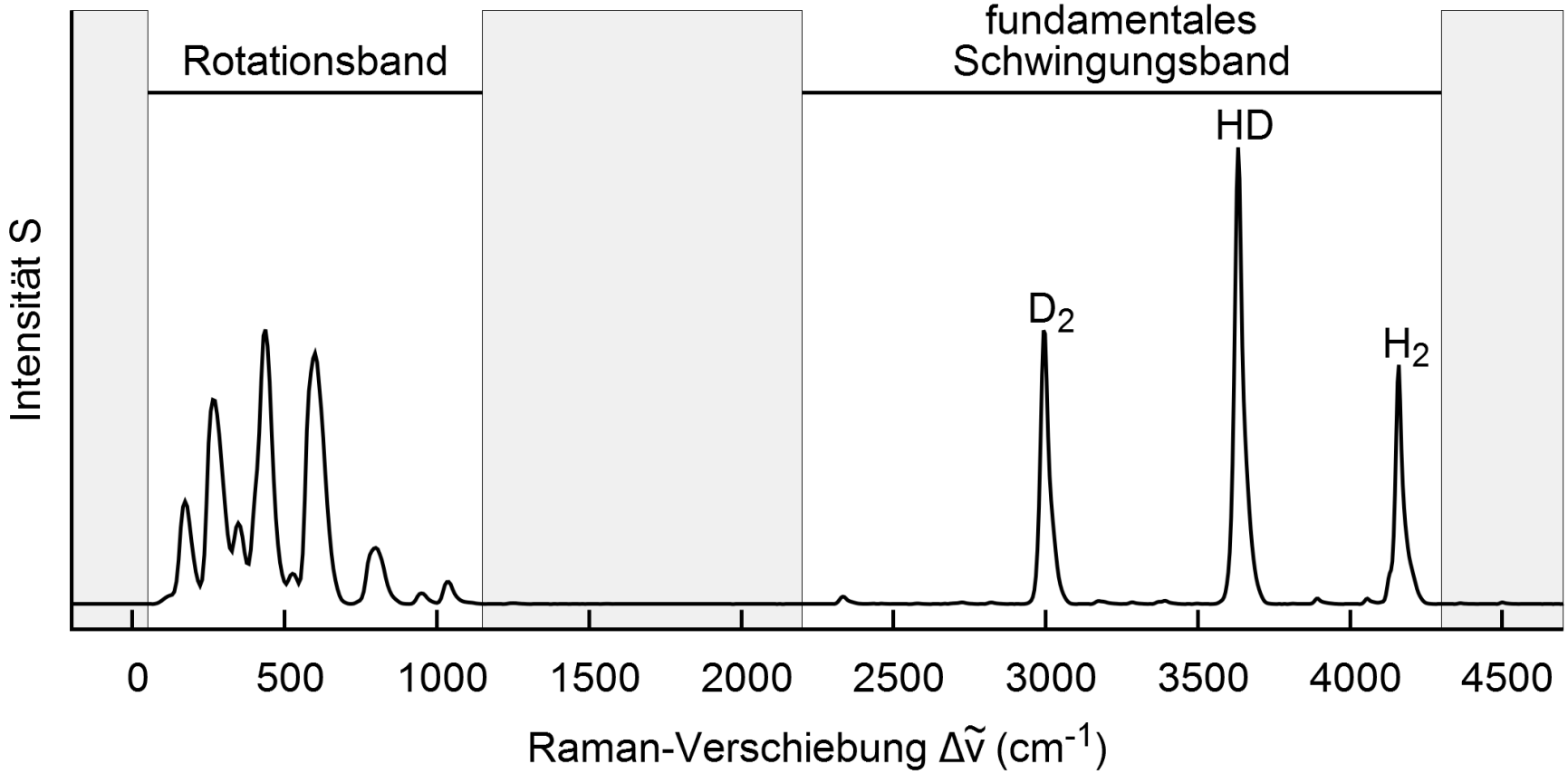
Questions?

Appendix

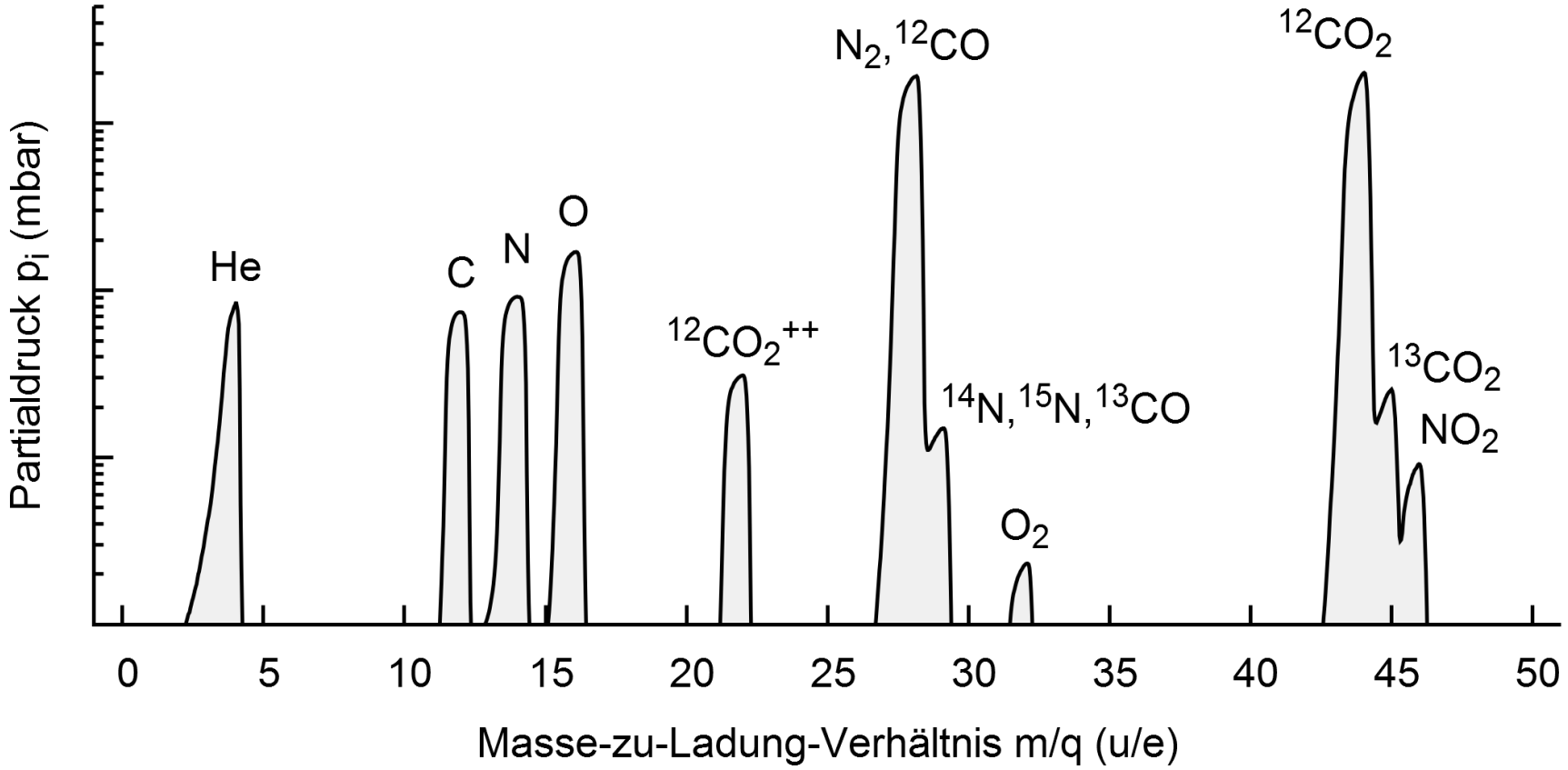
Exemplary IR spectrum



Exemplary Raman spectrum



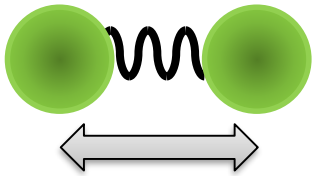
Exemplary QMS spectrum



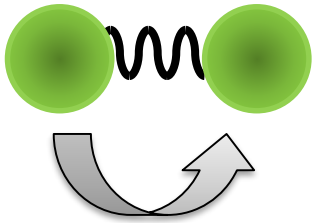
Rein, A. 2015.
Abschätzung und Analyse von Verunreinigungen in Kalibriergasmischungen für das Tritium-Hydrogen-Deuterium-Experiment (TRIHYDE),
Masterarbeit, Karlsruher Institut für Technologie

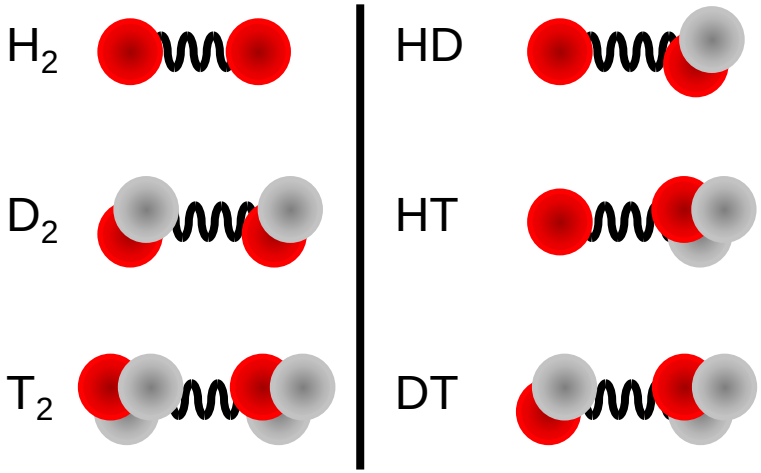
IR absorption measurements

Vibration Δv

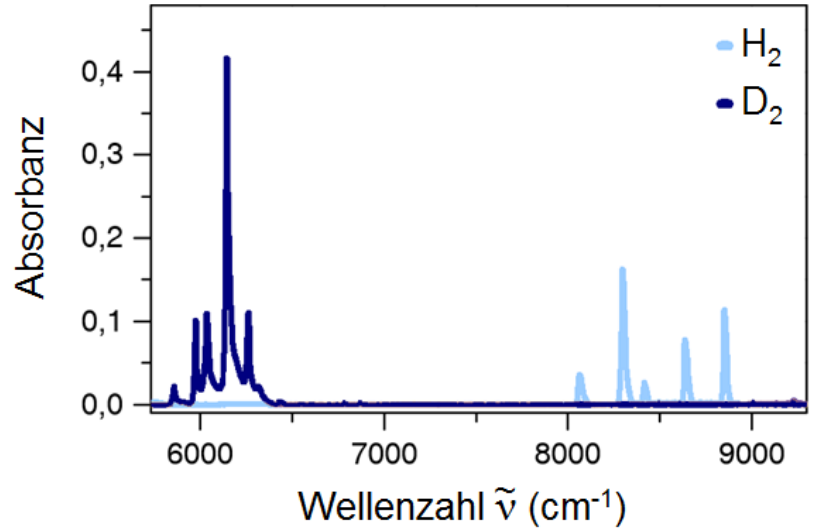


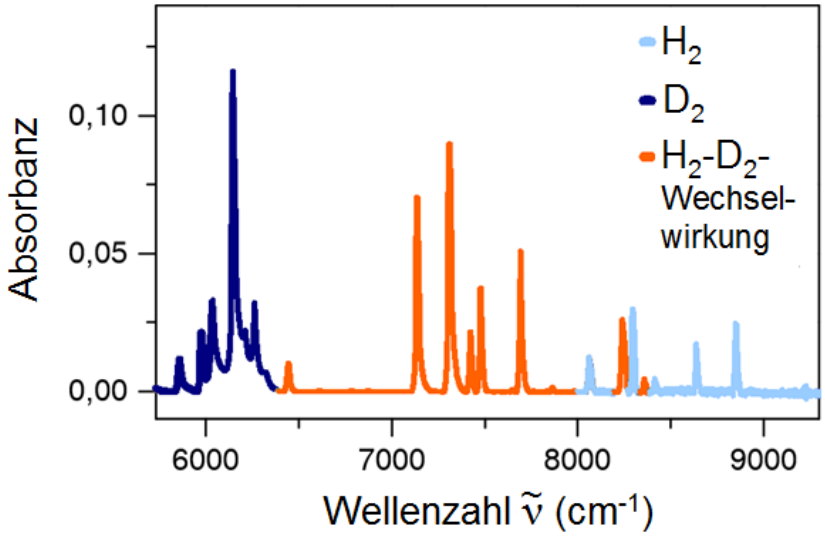
Rotation ΔJ

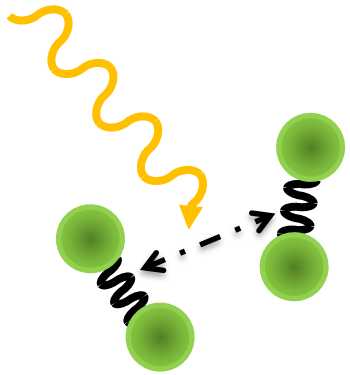




Dimers – double transitions

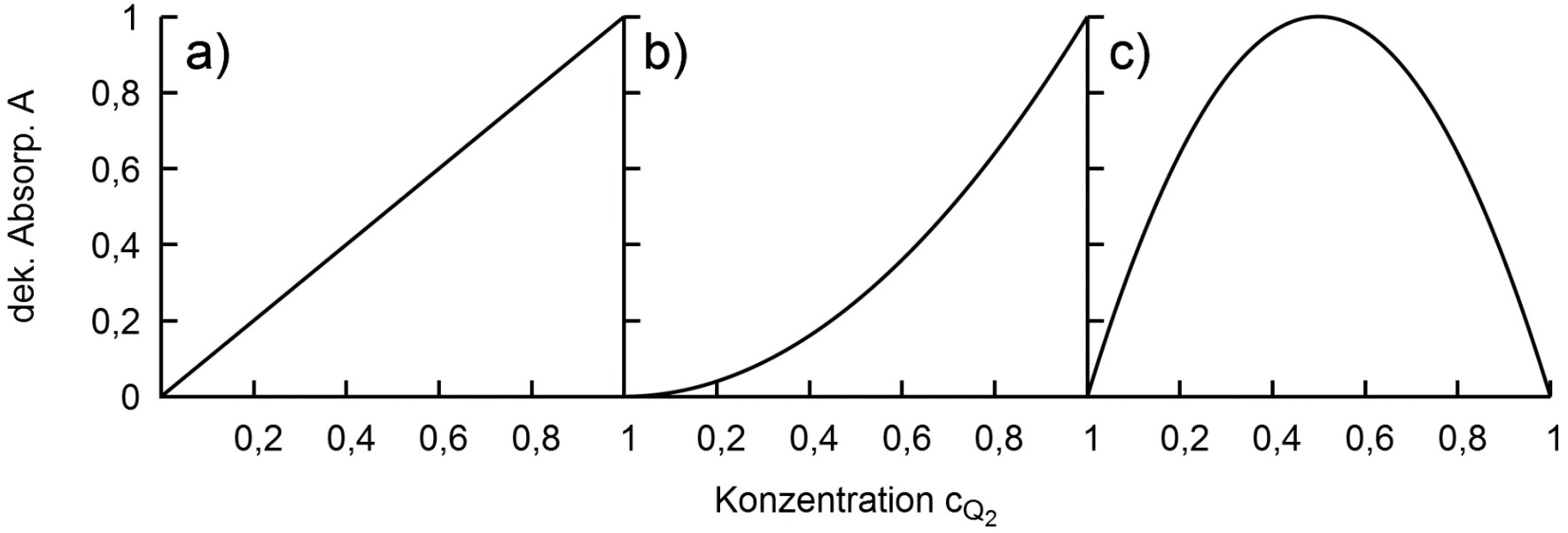






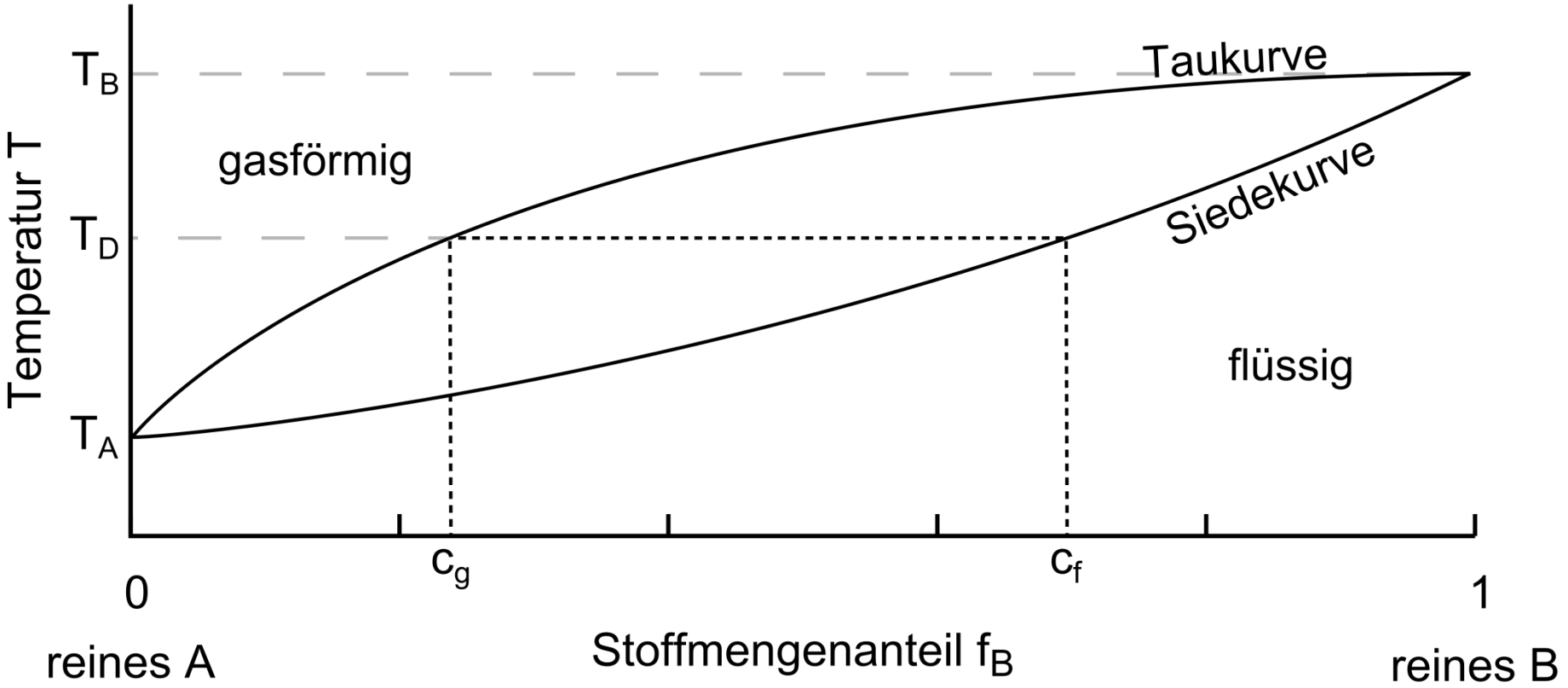
Wozniewski, S. 2014.
Relative Kalibrierung der IR-Absorptionsspektren gegen die H2-, HD- und D2-Konzentrationen,
Bachelorarbeit, Karlsruher Institut für Technologie.

Exemplary Raman spectrum

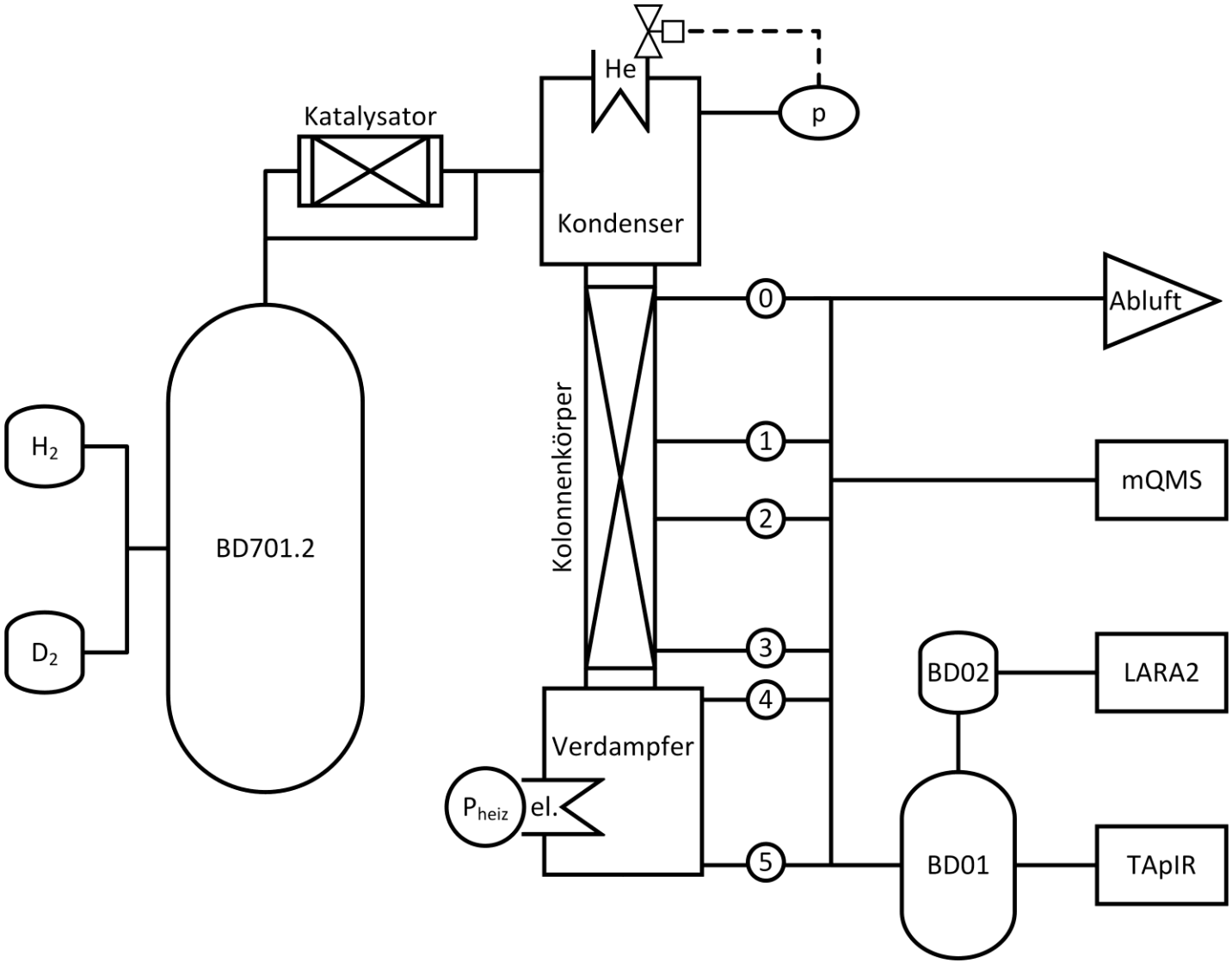


$$A \propto c_X \cdot c_Y = c_X \cdot (1 - c_X - c_Z) = \underbrace{c_X}_{\text{a)}} - \underbrace{c_X^2}_{\text{b)}} - \underbrace{c_X \cdot c_Z}_{\text{c)}}$$

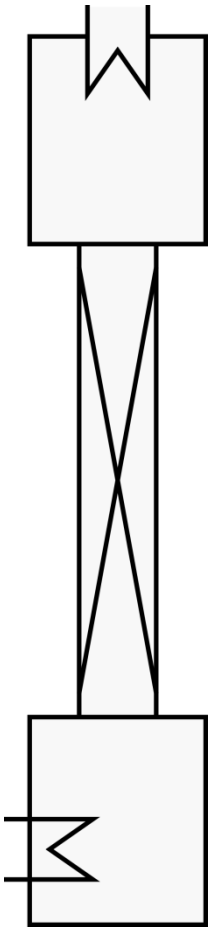
Boiling-point diagram

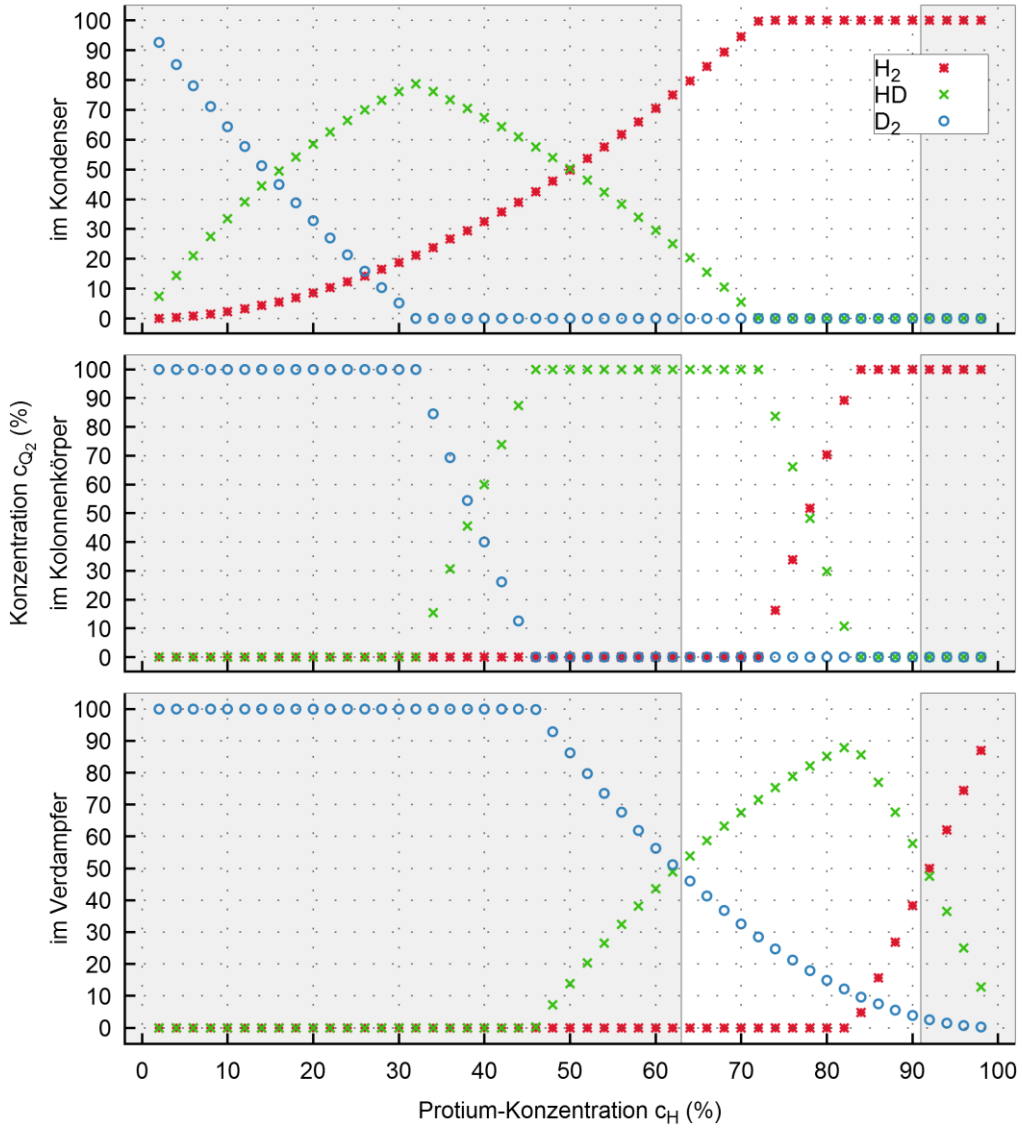


Distillation set-up



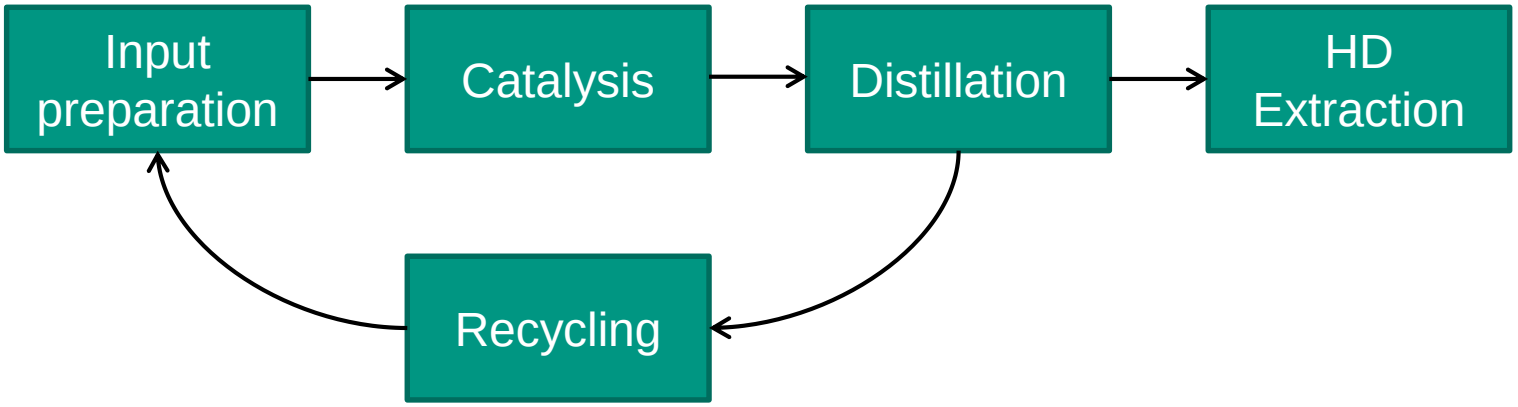
Simulated column content



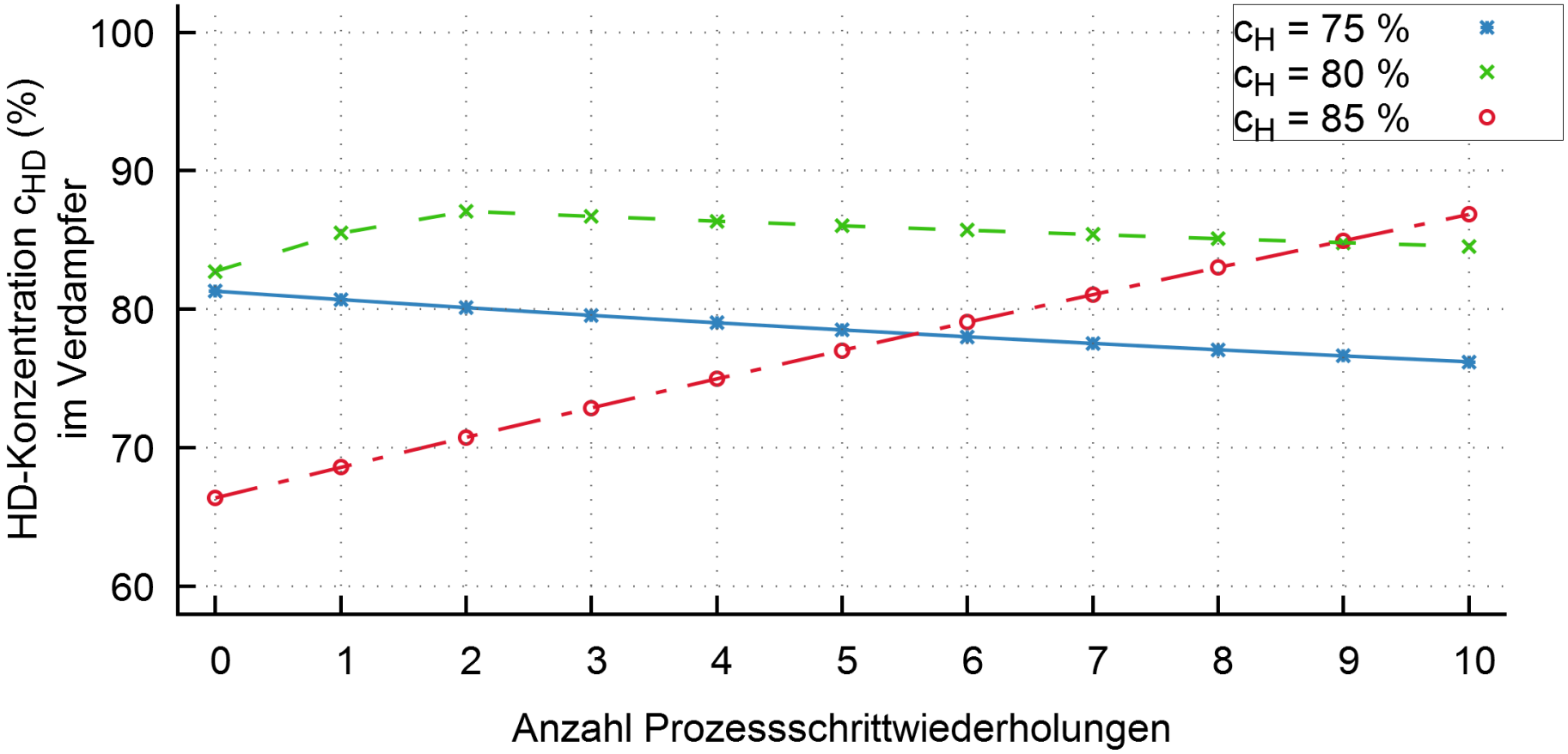


Production of highly concentrated HD

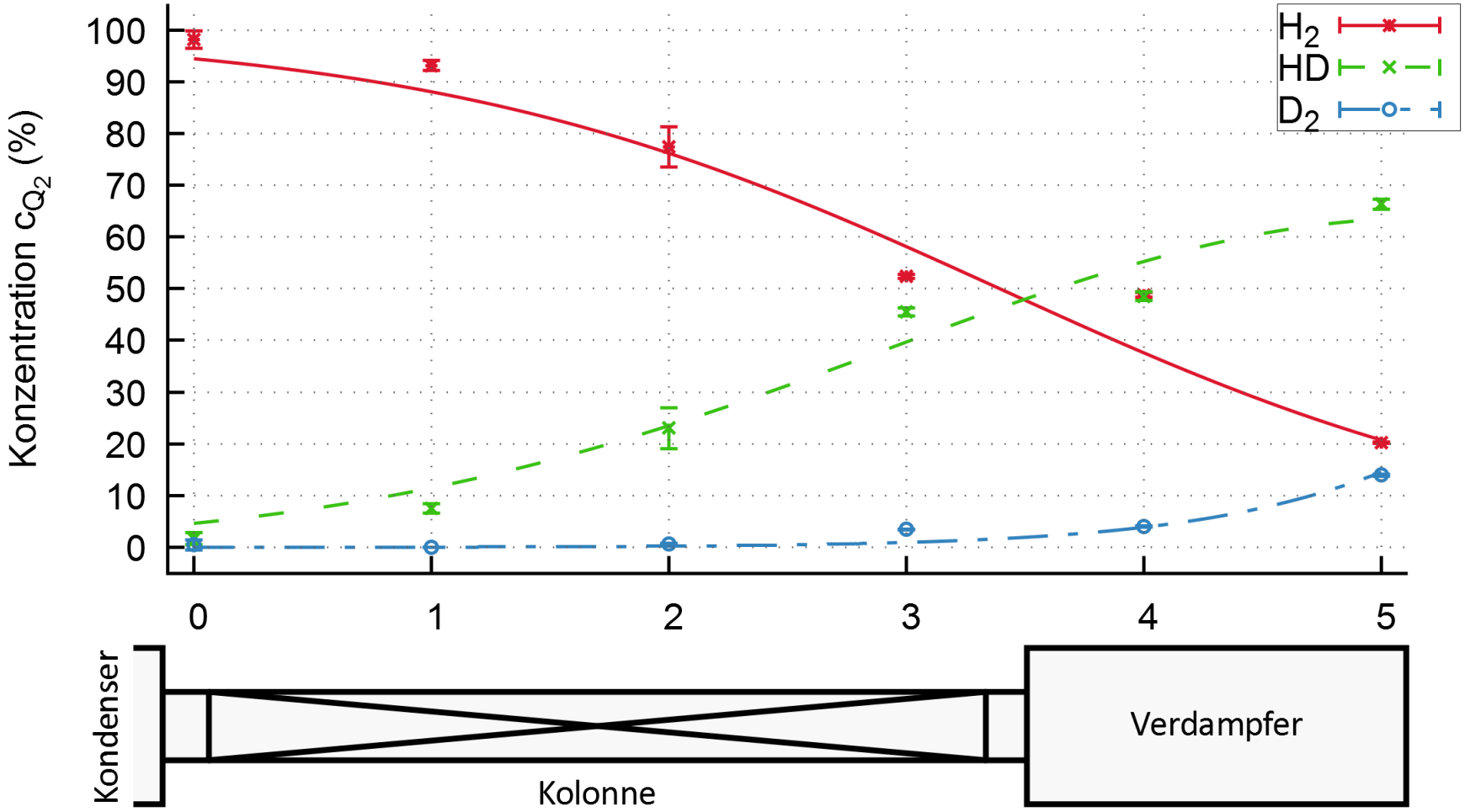
RUN B



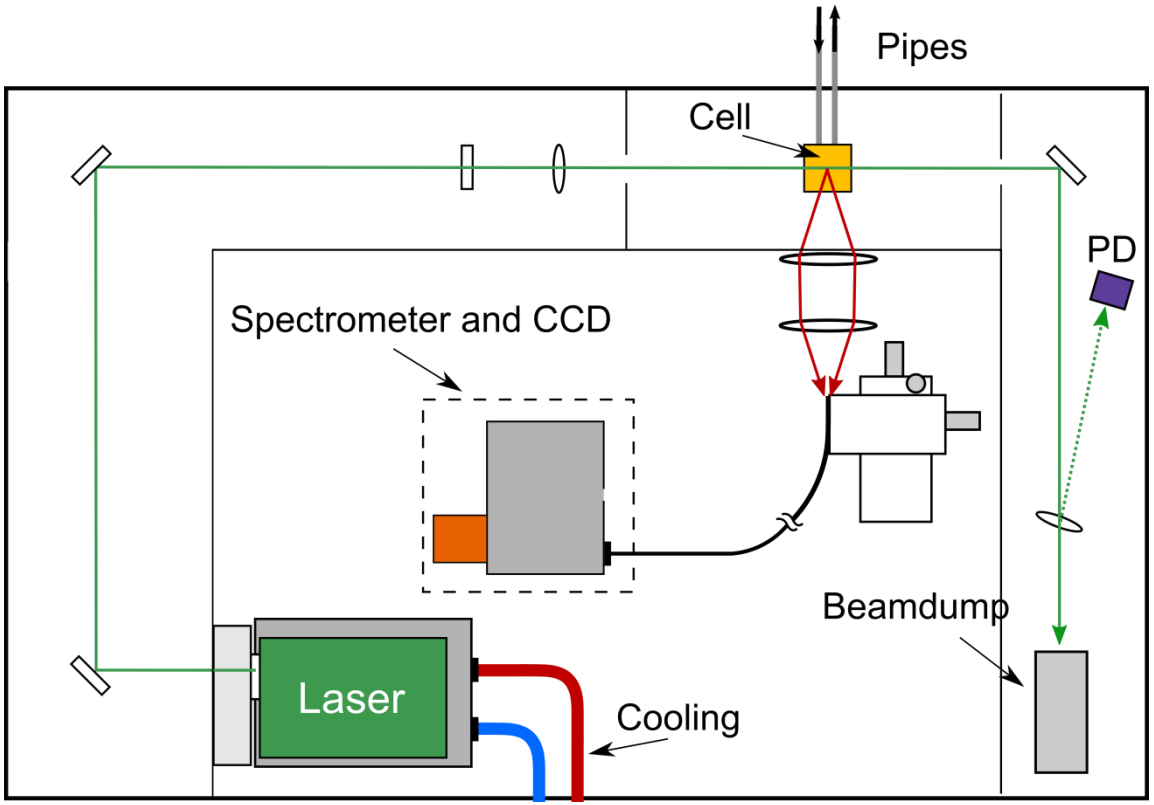
Distillation cycles



Column profile



LARA (Laser Raman System)



- Gaseous sample in Raman cell
- Raman scattering of the laser photons
- Spectroscopic investigation of scattered light

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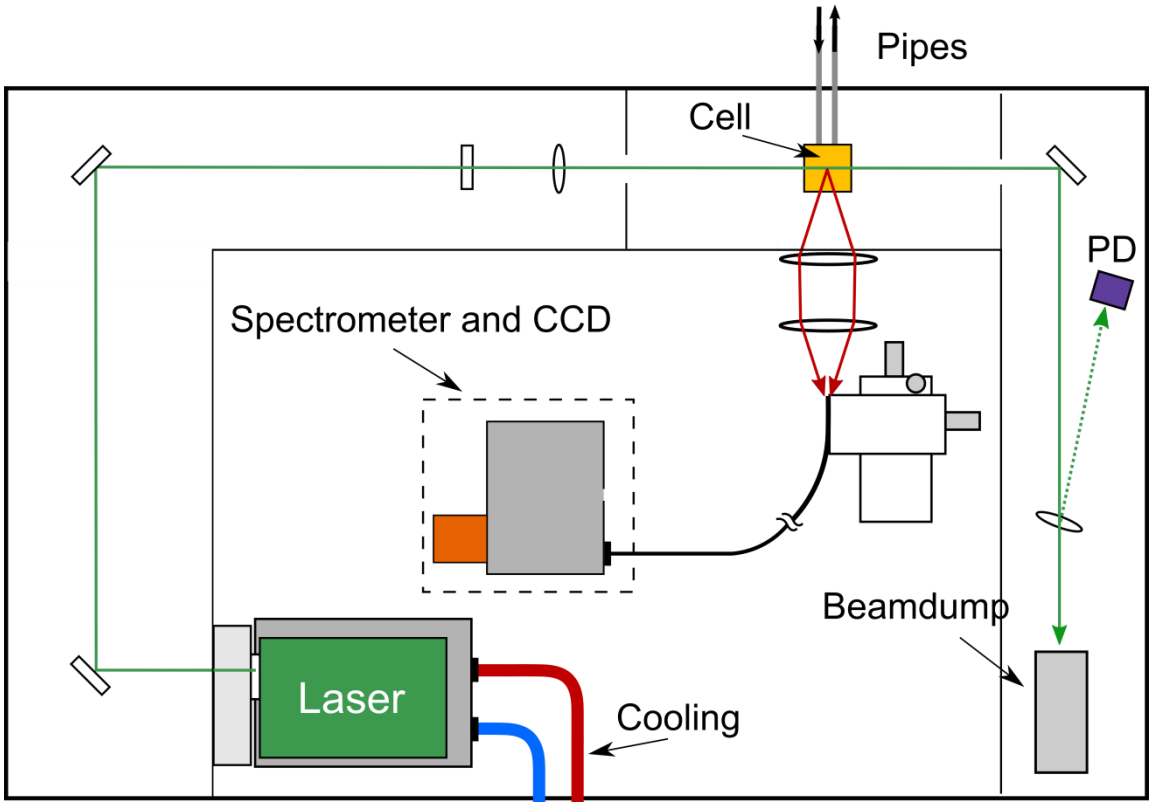

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- Raman scattering of the laser photons
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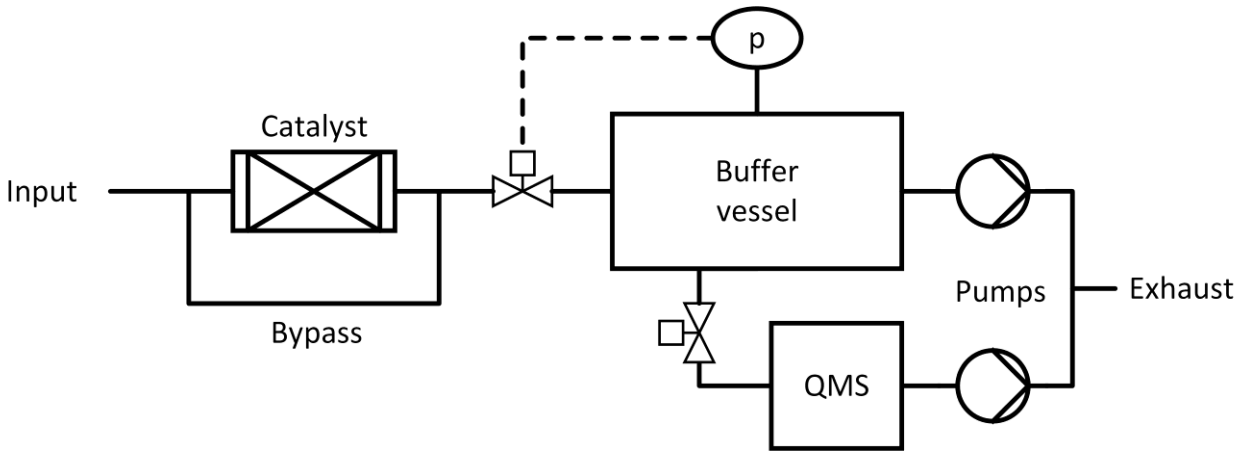
Linear calibration functions for all isotopologues

mQMS (mobile Quadrupole Mass Spectrometer)



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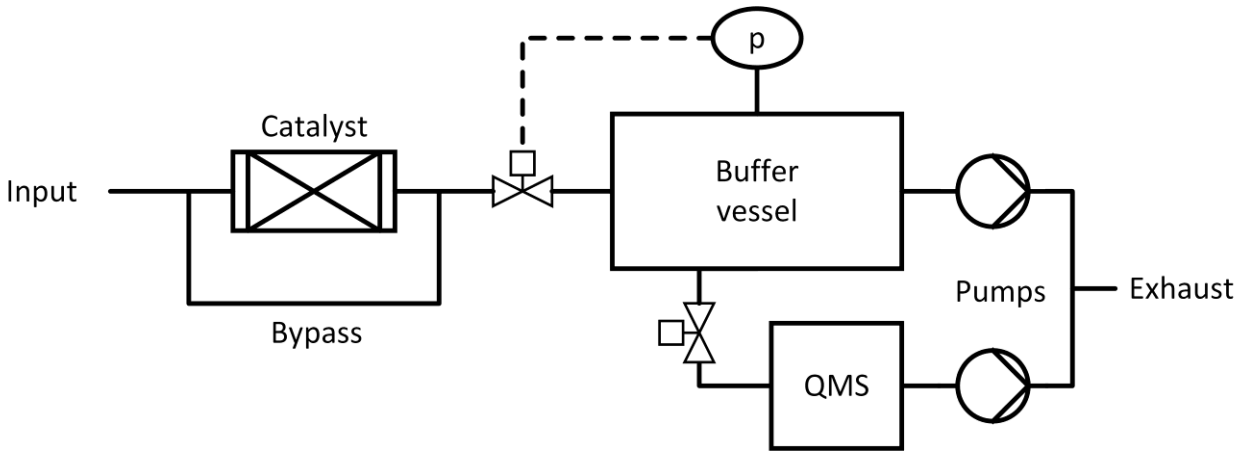

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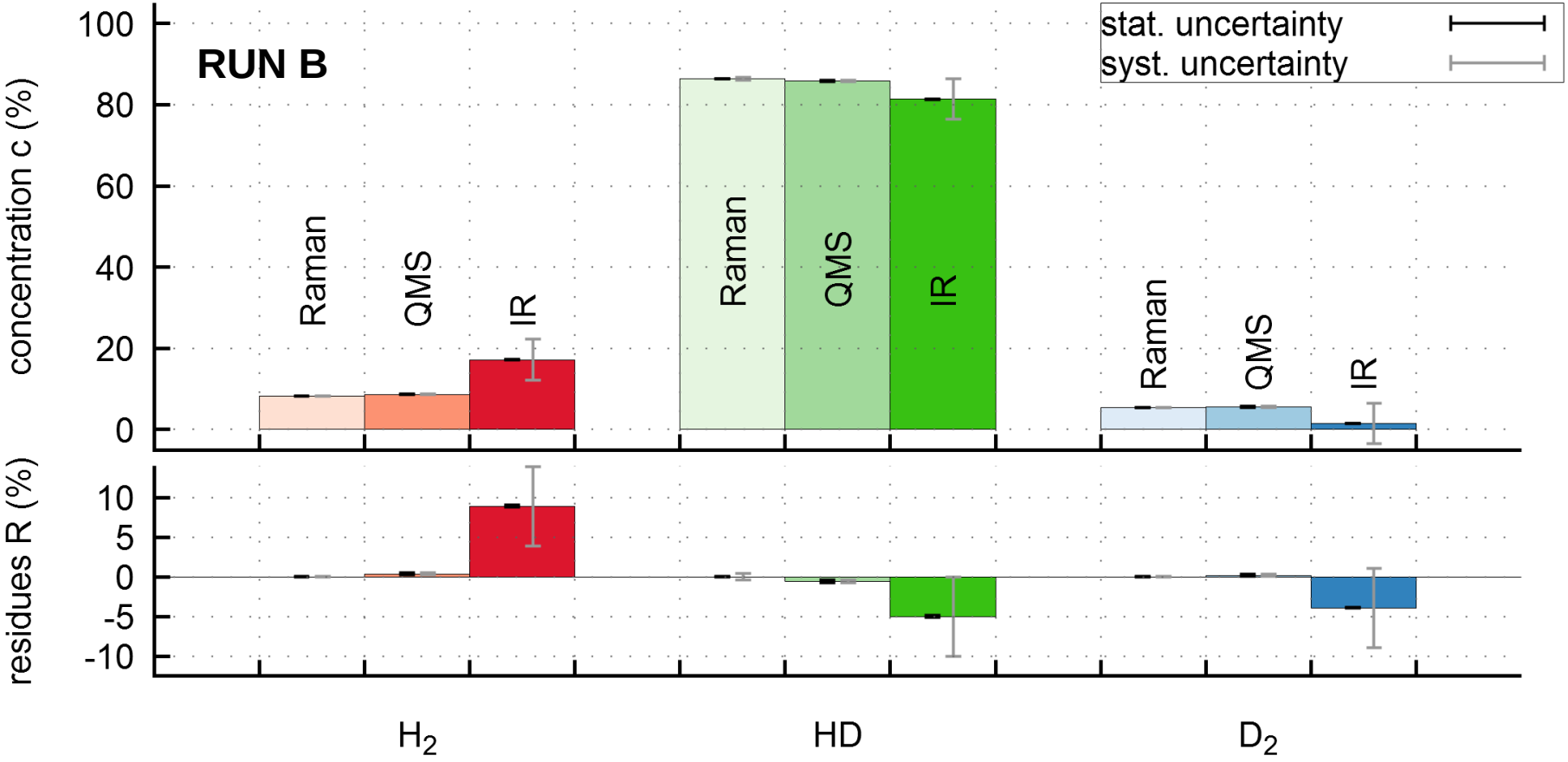
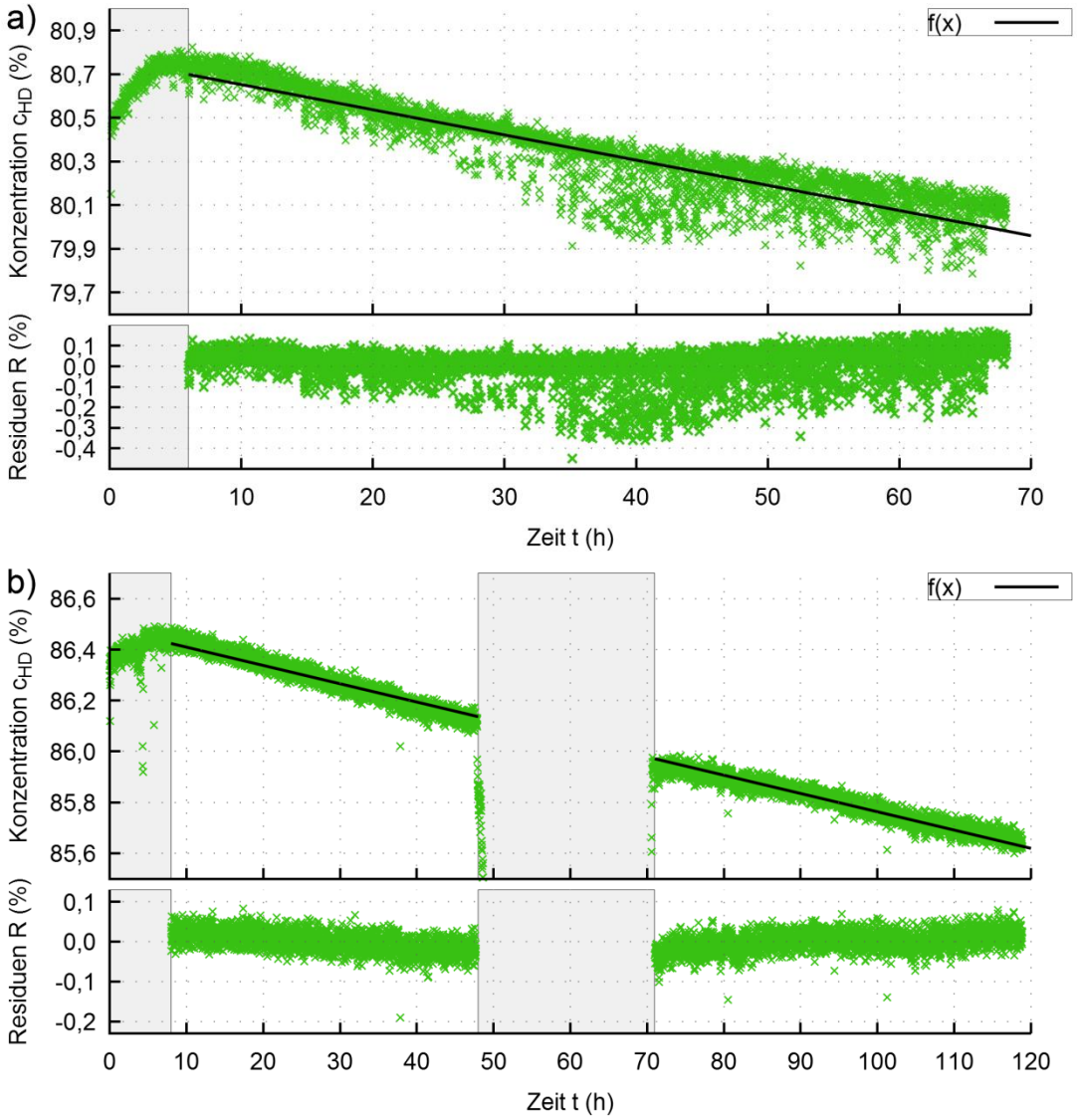
calibration functions
H2, HD: linear
D2: second order

Comparison of spectroscopic methods

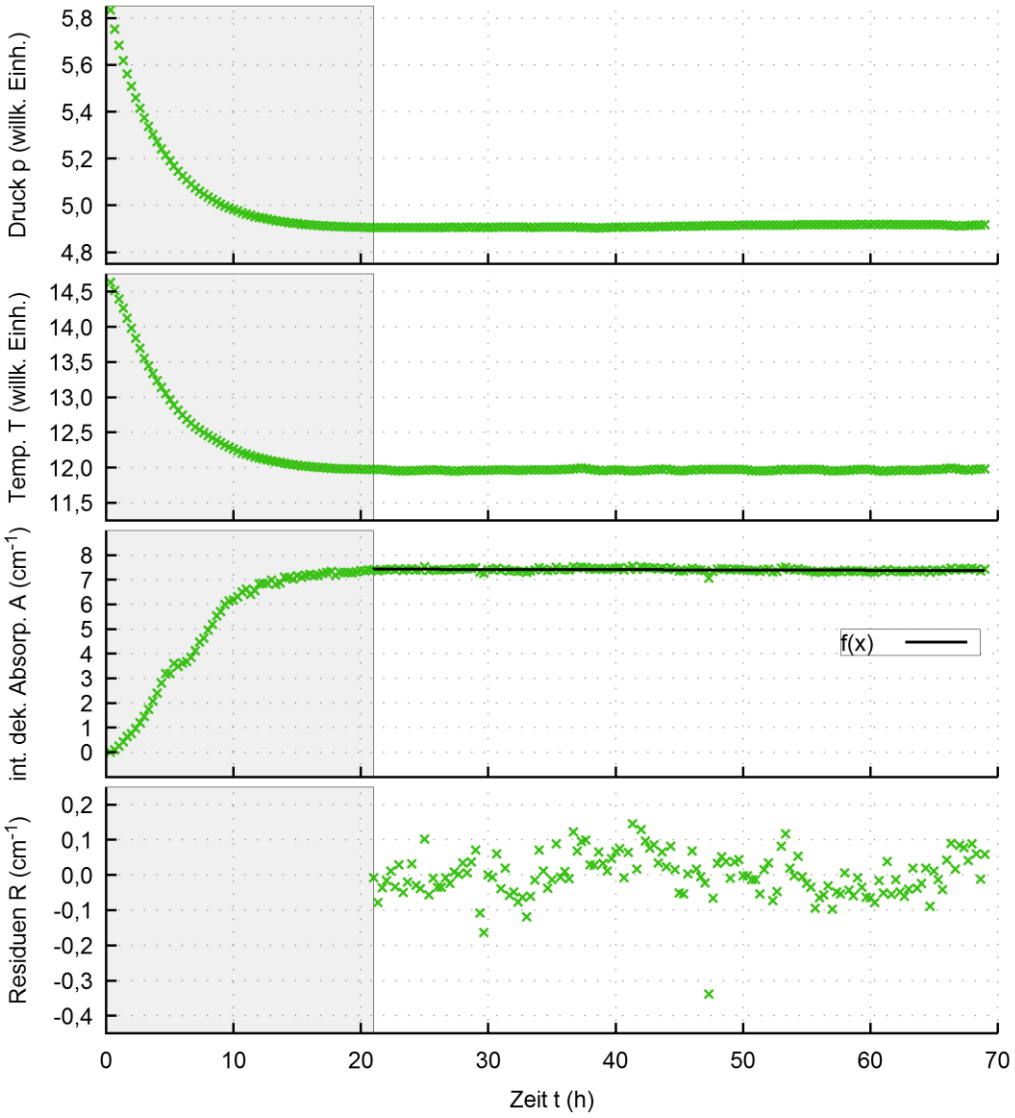


Stability analysis



Stability analysis

RUN A



Stability analysis

RUN B