Sample Environments for Controlled Gas Deposition up to 10 kbar

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Outline

- Neutron scattering combined with in situ vapour adsorption: applications & goals
- Automated vapour dosing devices for low and medium pressures and temperatures
- High pressure hydrogen compression system

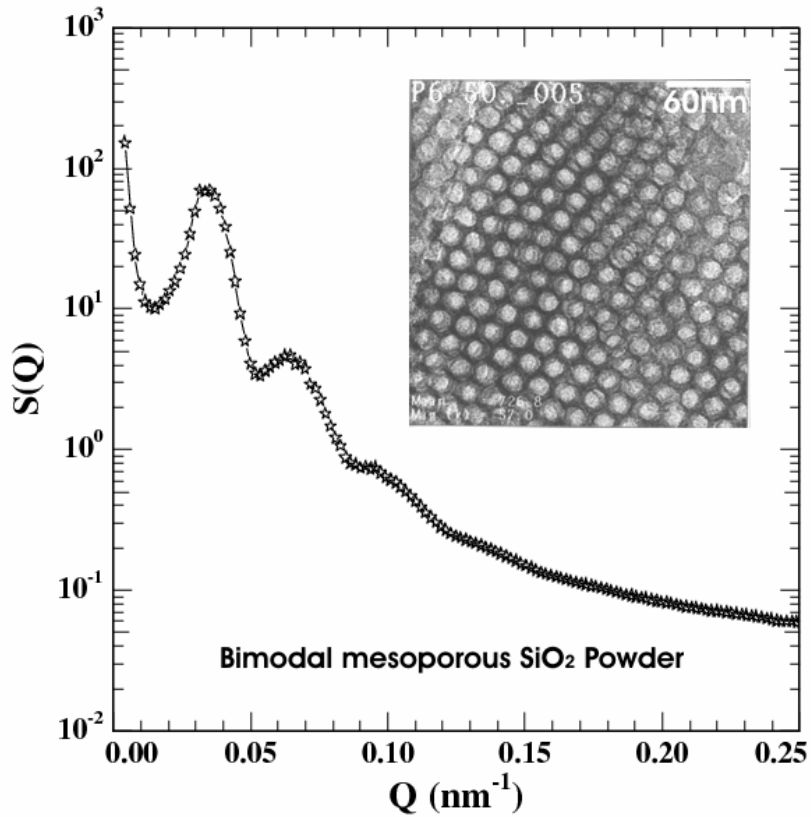
Neutron Scattering with in situ Vapour Adsorption: Applications

- Characterization of (nano-)porous materials (SANS-ADSO)
- Structure and dynamics in confined geometries (pore condensed matter)
- Controlled sample exposure to water, ammonia and hydrocarbon atmospheres in biological, material, and geological sciences
- Solar energie, fuel cells: Hydrogen storage and intercalation in host systems

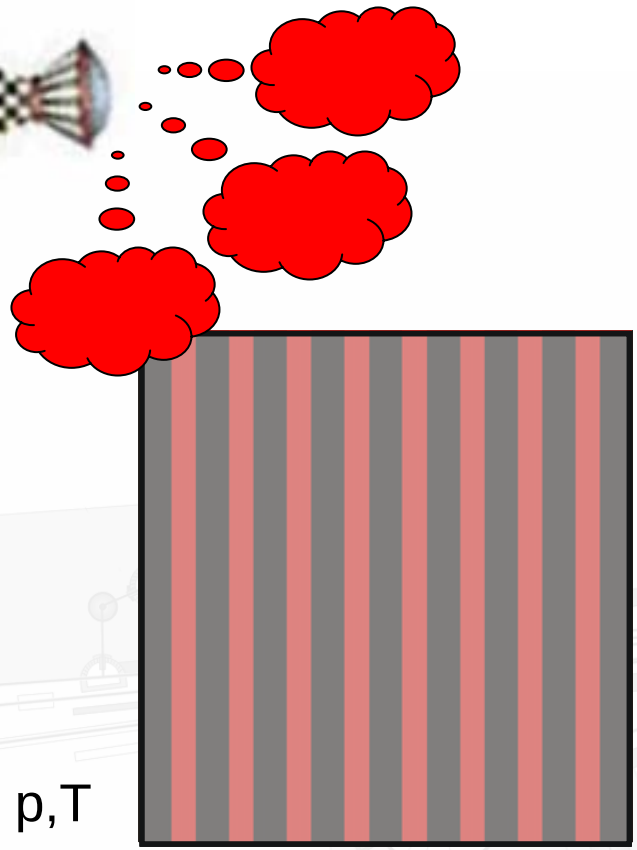
Neutron Scattering with in situ Vapour Adsorption: Example

Porous materials:

SANS



Adsorption in porous materials: dosing



p,T

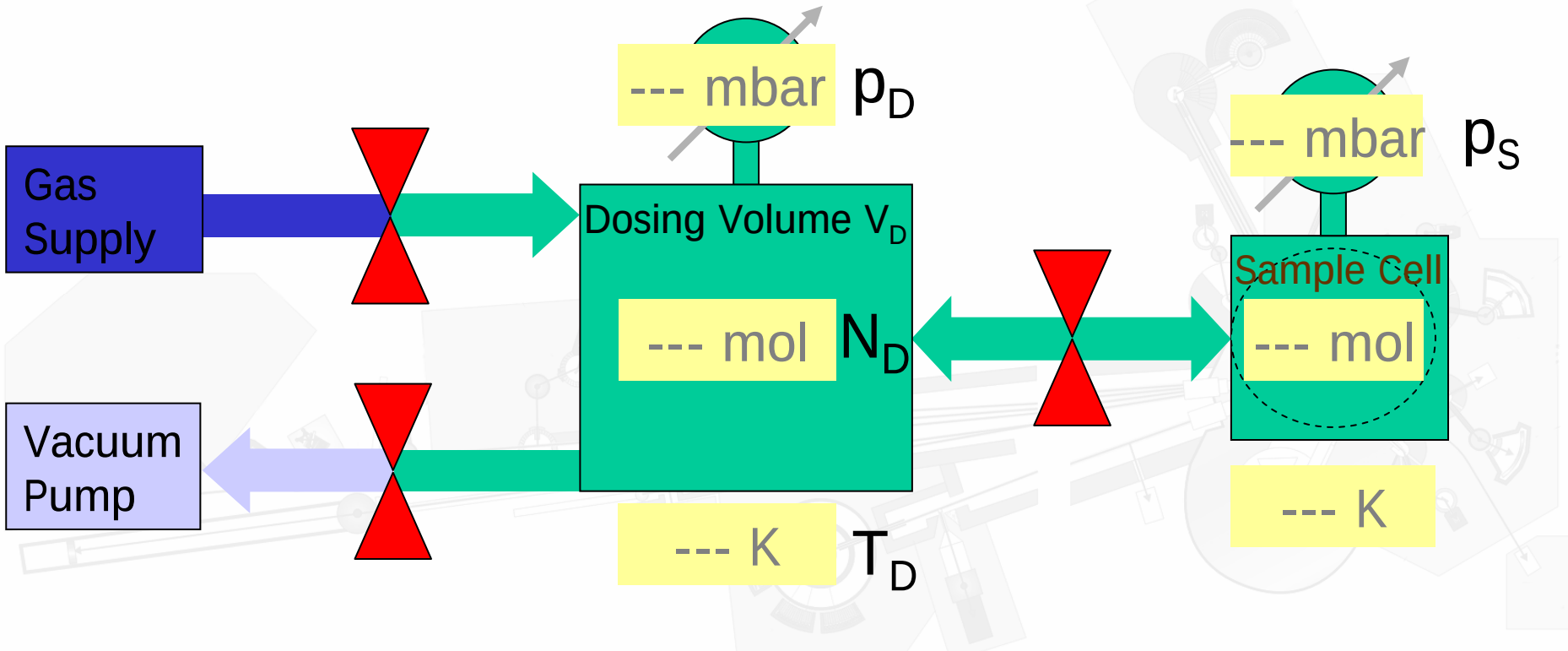
Problem:

determination of the amount of adsorbed material

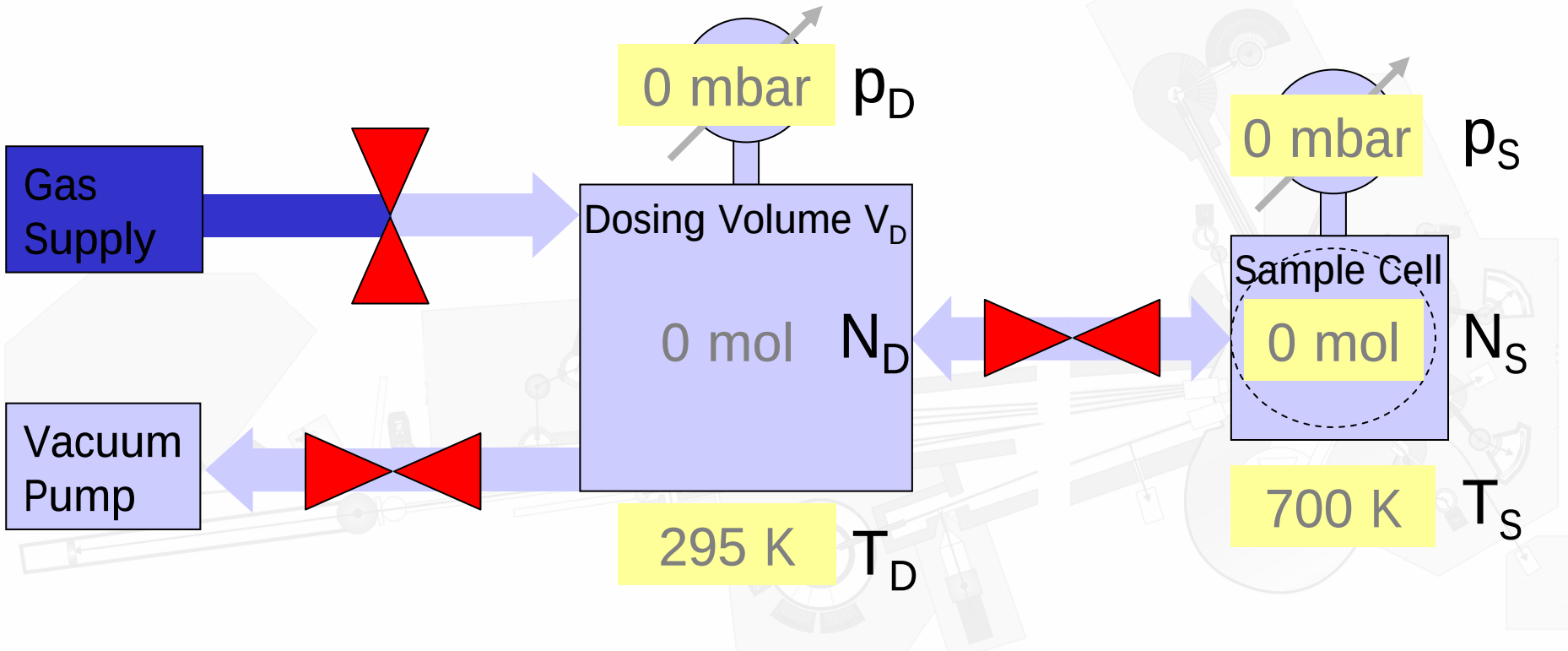
dosing device

Automated Vapour Dosing Devices: Measurement of Gas Uptake

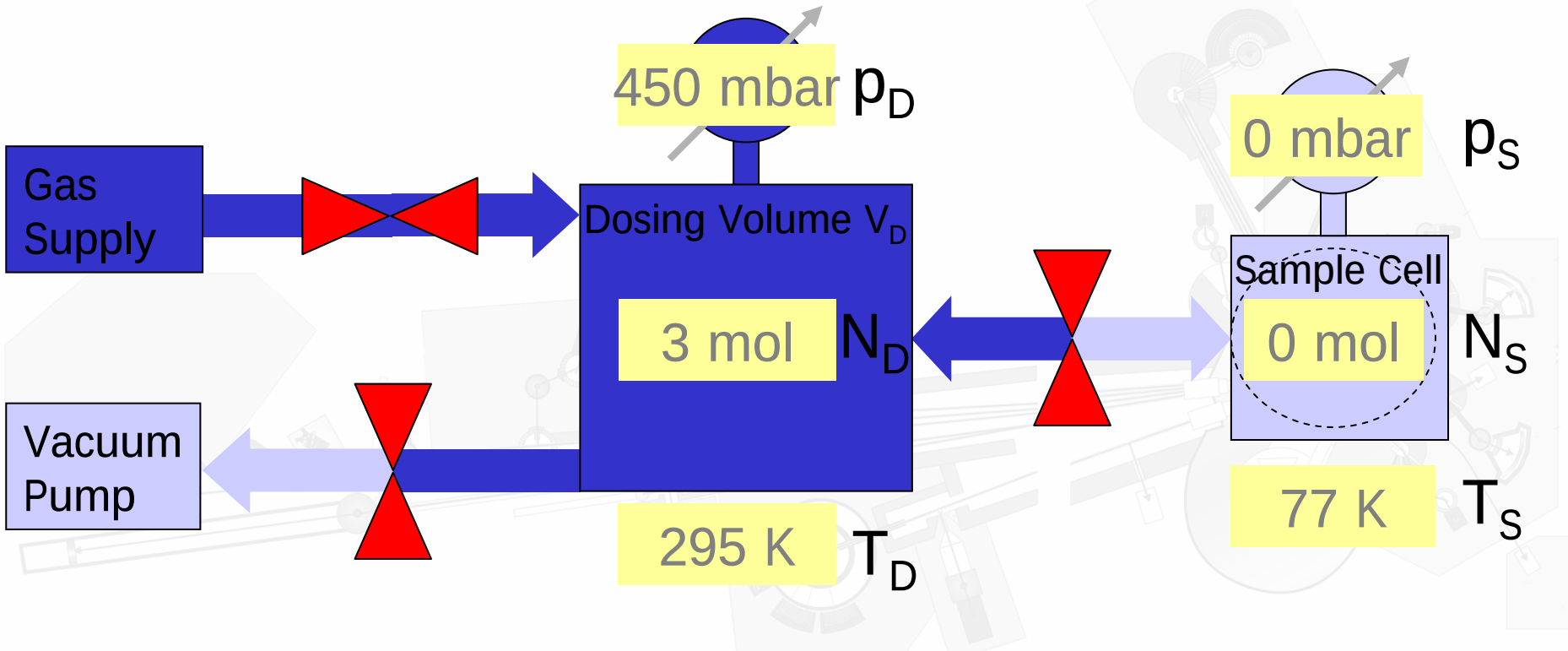
- Gravimetric (mass uptake)
- Dynamic flow (differential flow measurement)
- Volumetric (pV=nRT)



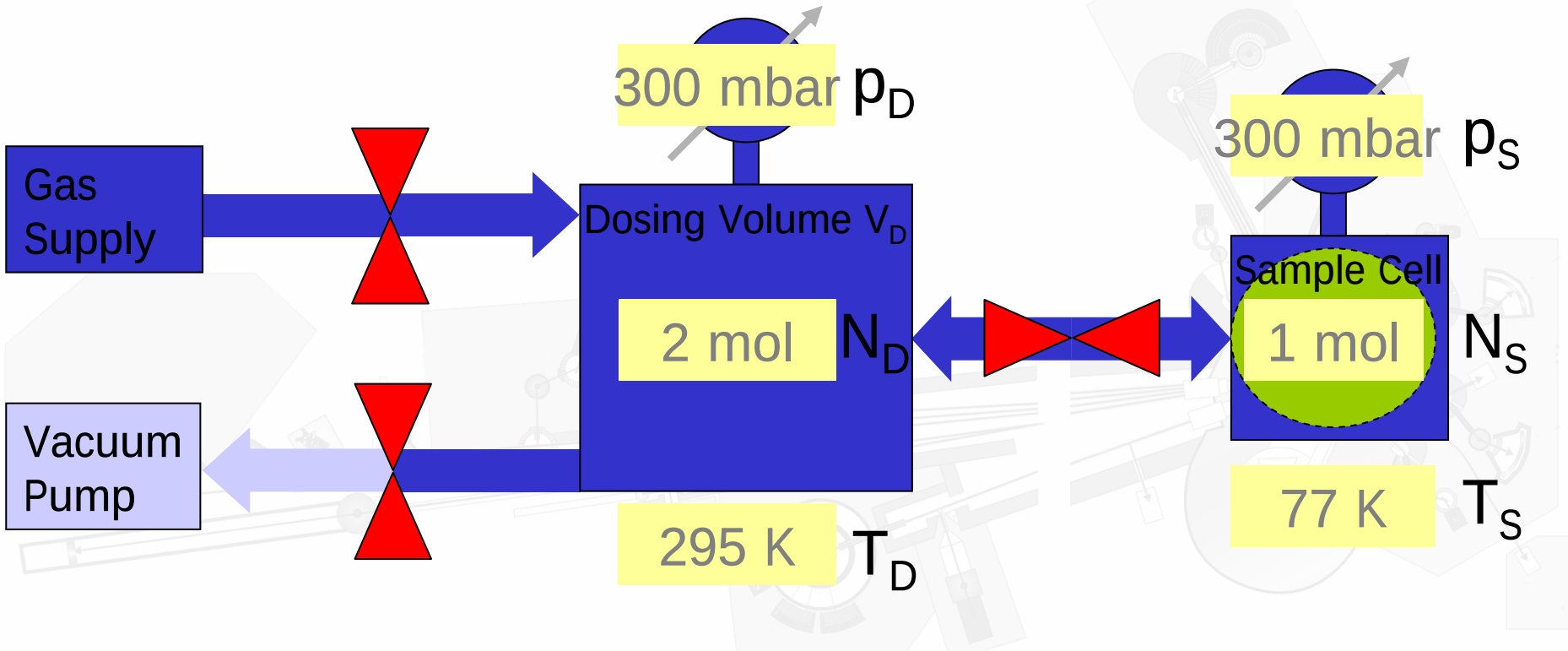
Measurement of Gas Uptake: Outgasing



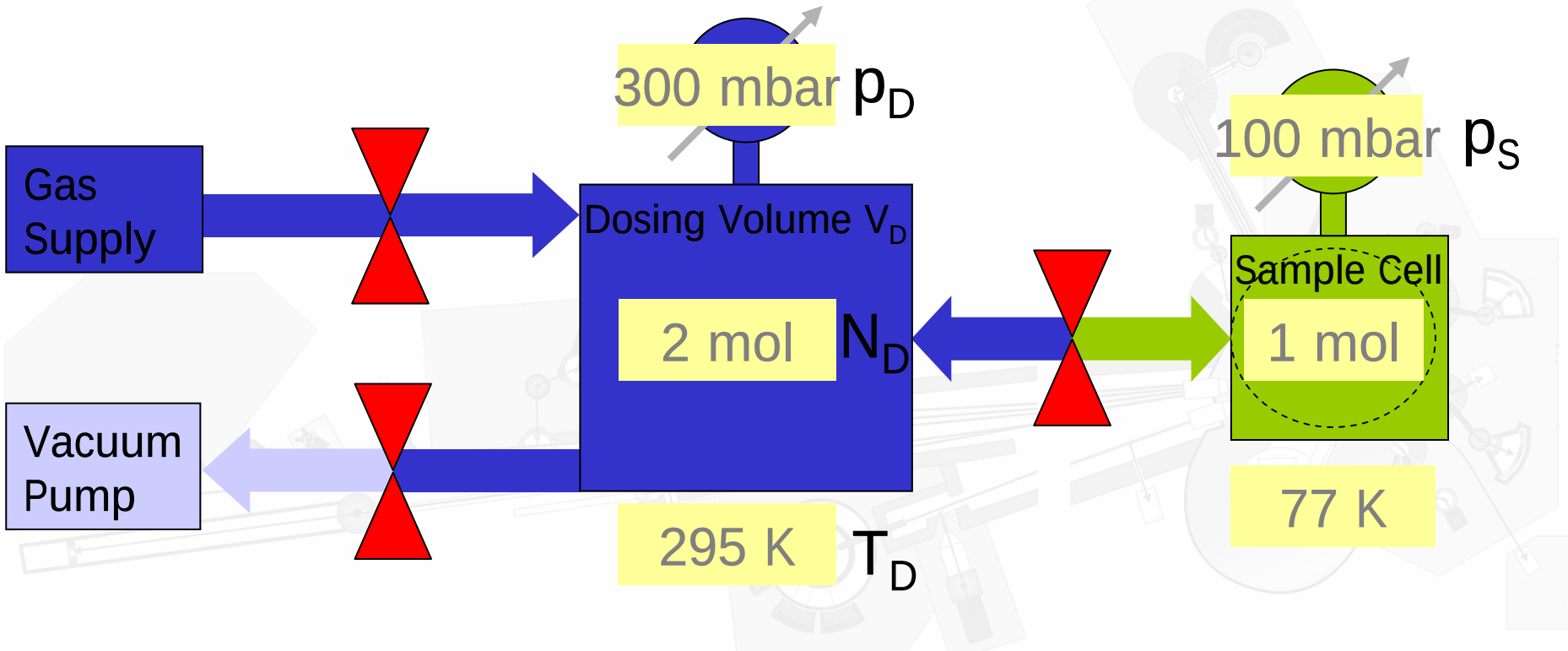
Measurement of Gas Uptake: Dosing



Measurement of Gas Uptake: Exposure



Measurement of Gas Uptake: Equilibrating



HMI-Dosing Devices (projected)

- 3 Devices for different pressure and temperature ranges

  low: 0.001 mbar < p < 1 bar, 1.5 K < T< 290 K
  med: 0.1 mbar < p < 200 bar, 1.5 K < T< 470 K
  high: 1 bar < p < 10 kbar, 1.5 K < T< 600 K

- Safe and user-friendly control management

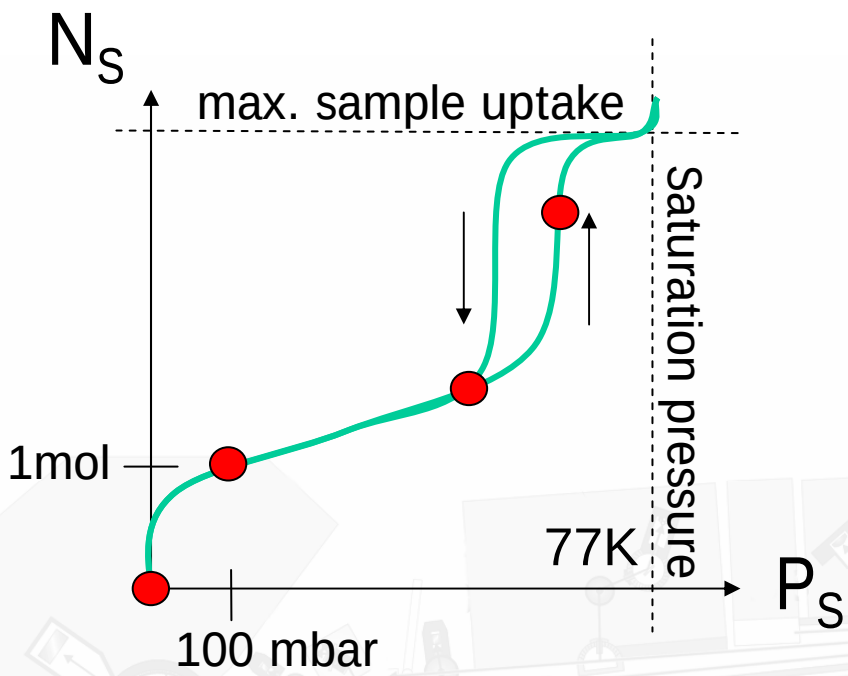
- Automation of the gas dosing and deposition process

- Implementation in the standard instrument software: bidirectional interface to e.g. CARESS, MAD, …

- Short preparation times of the requested MpT *) equillibrium adsorption states

*) Molar number/Mass, Pressure, Temperture

Preliminary Laboratory Work: Sorption Isotherm

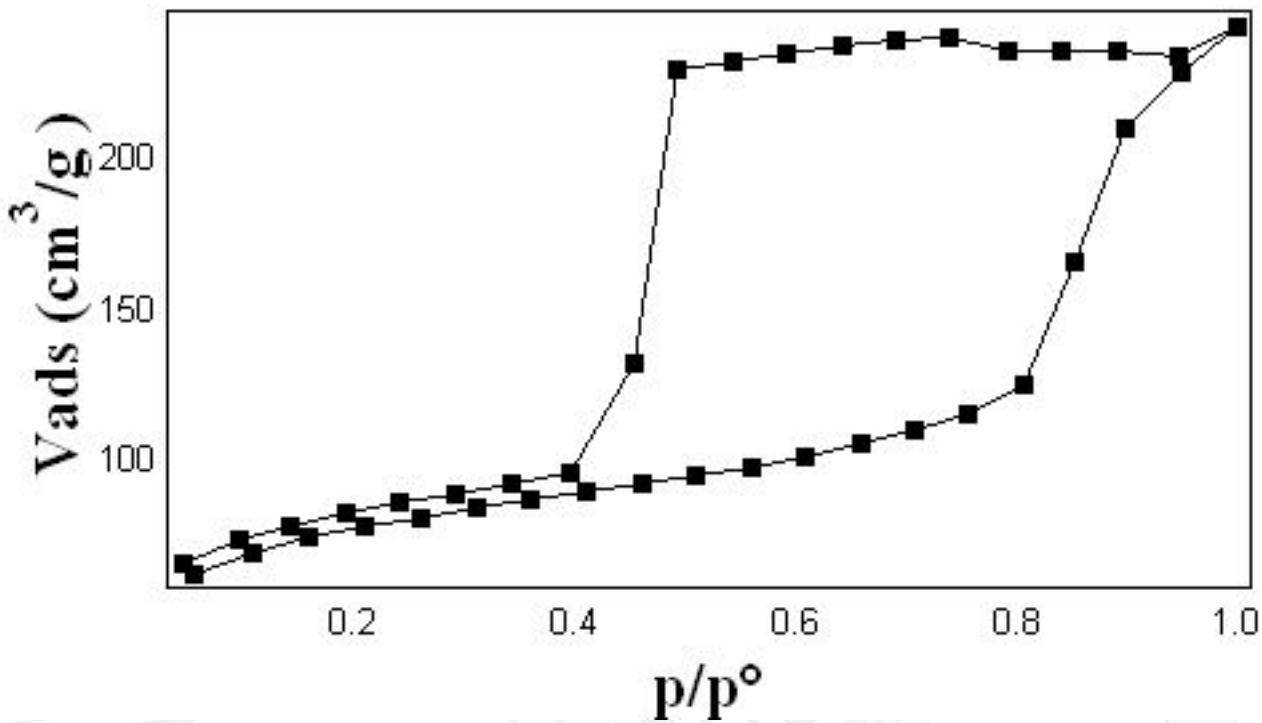


Successive filling (or emptying) steps

Sorption Isotherm

(Defined thermodynamic MpT-states of adsorbate-substrate-system)

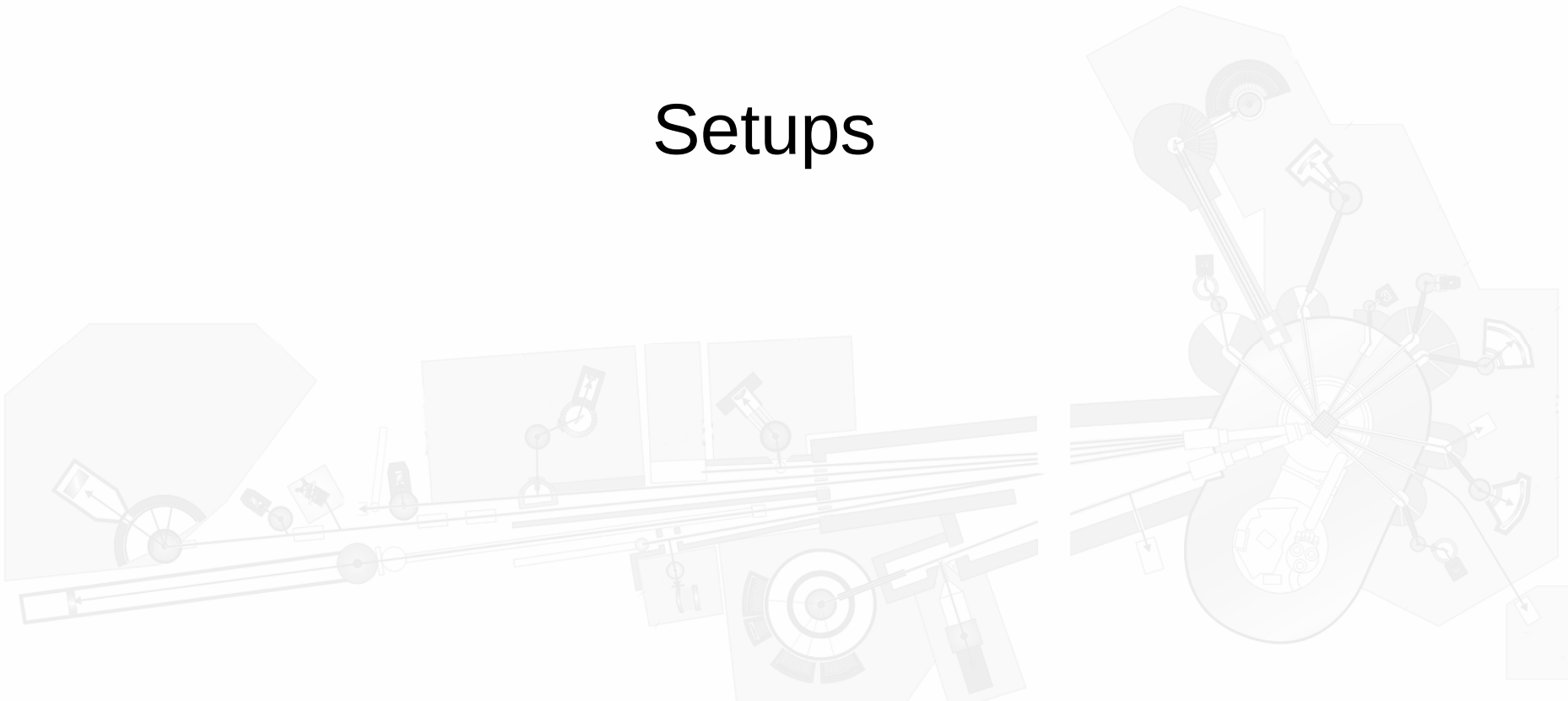
Example: Isothermal Adsorption/Desorption



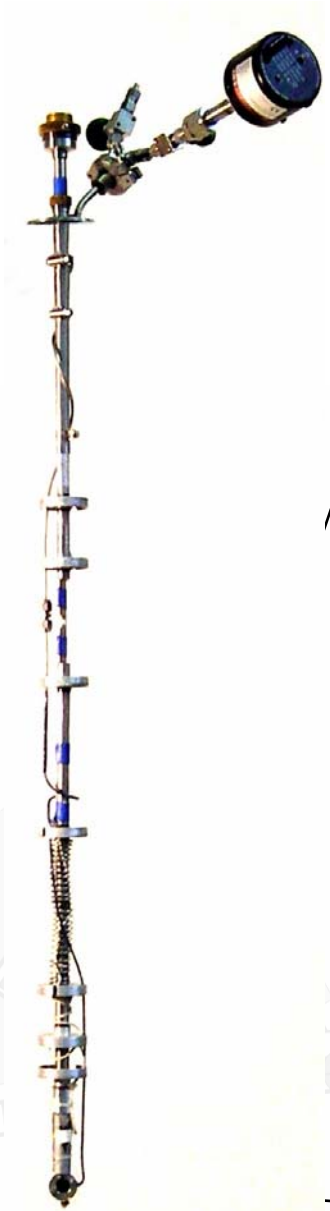
Measurement of Gas Uptake: Problems

Problem	Solution
Long equilibration times	Pre-charaterization of sample Optimized dosing algorithm Sample caroussel
Void volume, dead volume	Short connections
Cold spots	Heated manifold and capillary
Outgasing	External outgasing station

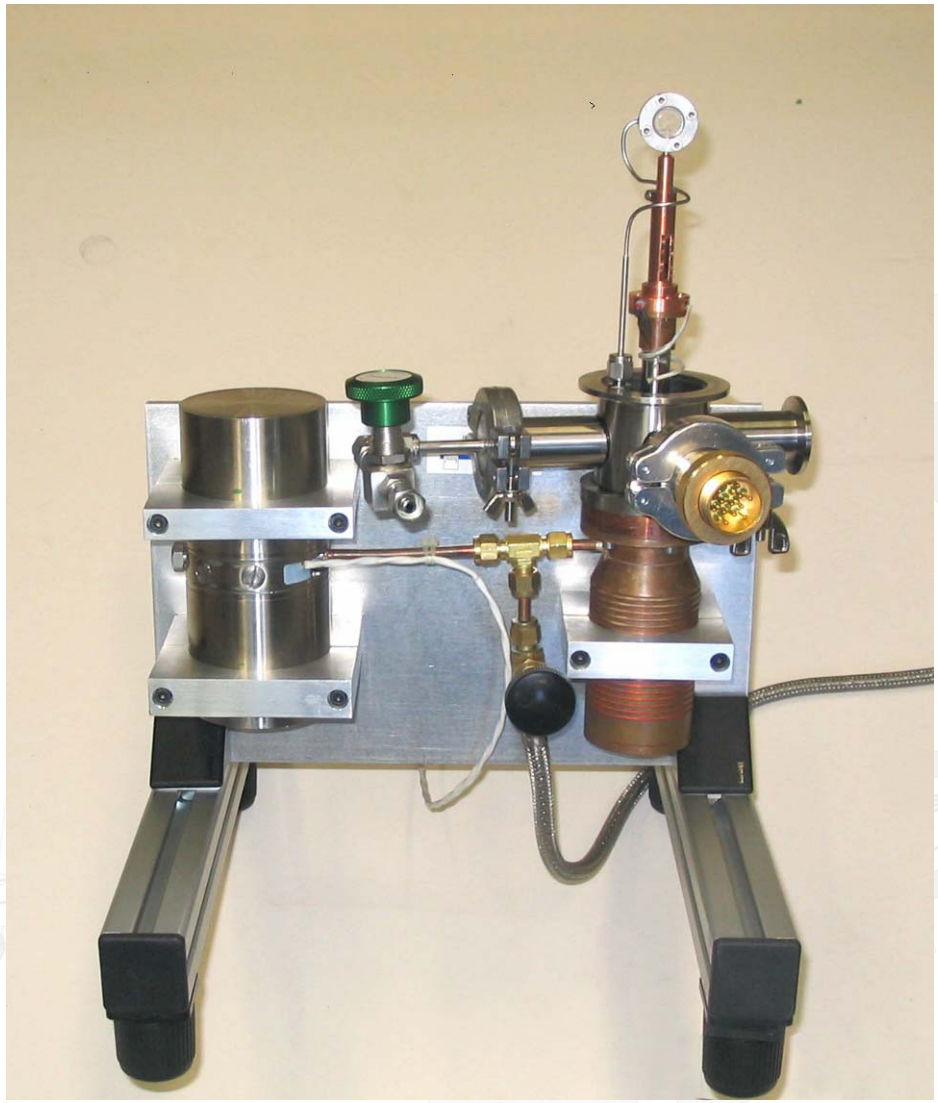
Setups



Sample stick



Pulstube



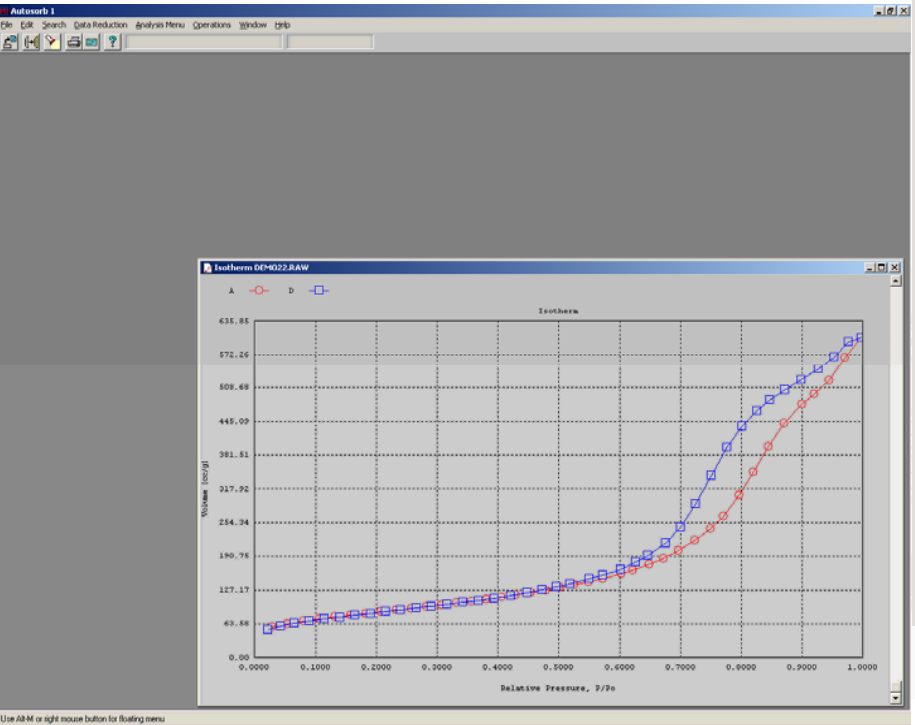
Automated Vapour Dosing Devices
1: Low pT-Range

Modified Commercial Gasadsorption System

"Quantachrome Autosorb"

0.001 < p < 1300 mbar

1.5 K < T < 290K



Quantachrome Autosorb®
Surface Area and Pore Size Analyzer

- A fully automated analyzer for surface area and pore size measurements.
- Offers simultaneous analysis and sample preparation.
- Low maintenance, vacuum volumetric system with stainless steel manifold construction.
- Speed and precision ensured a proprietary dosing algorithm.
- High sensitivity ensured by multiple pressure transducers, minimum dead volume and accurate coolant level control.
- Operates with any non-corrosive adsorbate and a wide variety of coolants.
- DoseWizard™ learn mode and InitialFill™ for analysis time savings of up to 60 percent.
- System supplied complete and ready for operation.

Automated Vapour Dosing Devices 2: Low and Medium pT-Range

Automized Gas Manifold
(200bar and 200°C)

Gases:
Water, Hydrocarbons, Ammonia

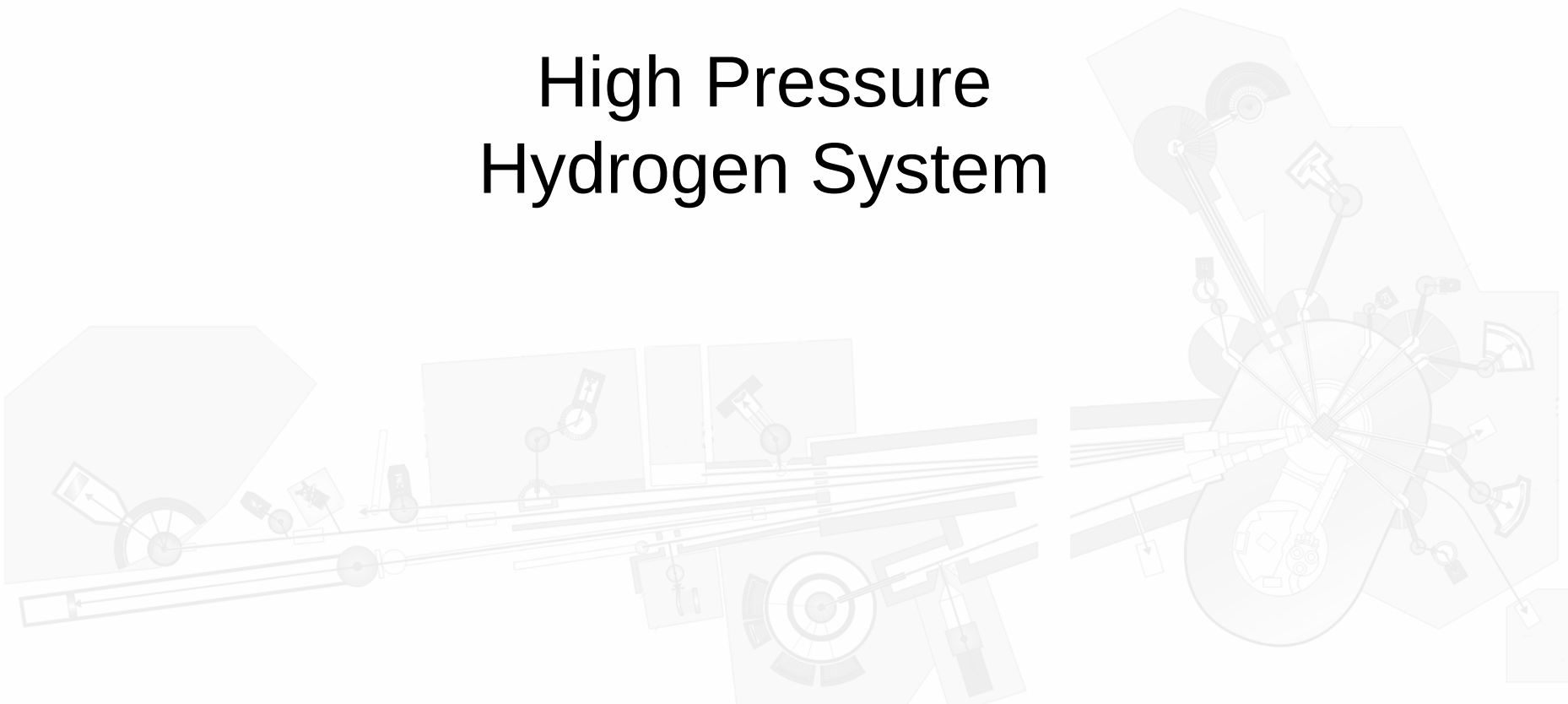
Applications:
Biological systems

Condensates in confined geometries

Cryo-environments:
Orange furnace (sample stick)

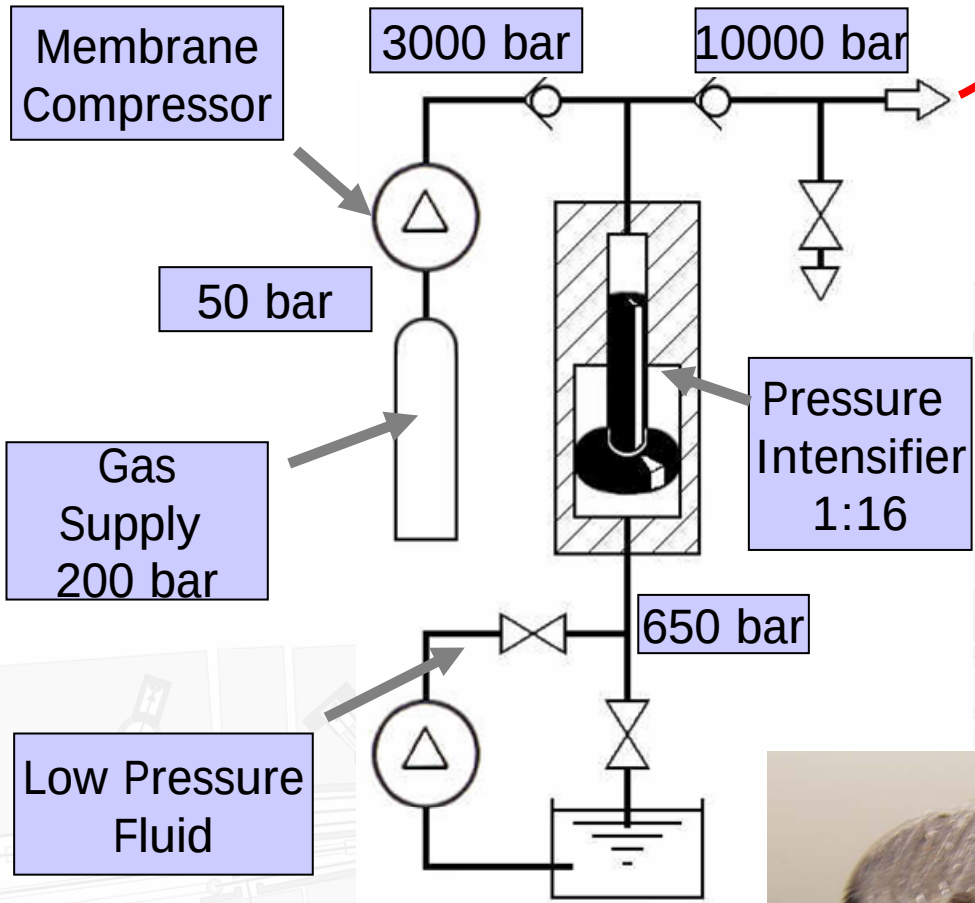
Closed cycle refrigerators (equipped with Sapphire)

High Pressure Hydrogen System



High Pressure Hydrogen System

3000 bar Compressor
(Nova Swiss)



Sample Stick with High Pressure Capillary (AS Scientific)

Conclusion

- Increasing demand for neutron scattering measurements with in situ gas/vapour adsorption
- Increasing demand for neutron scattering measurements with Hydrogen under pressure
- HMI is on the way