Sample Environment Road Map: viewpoint of Structure Research

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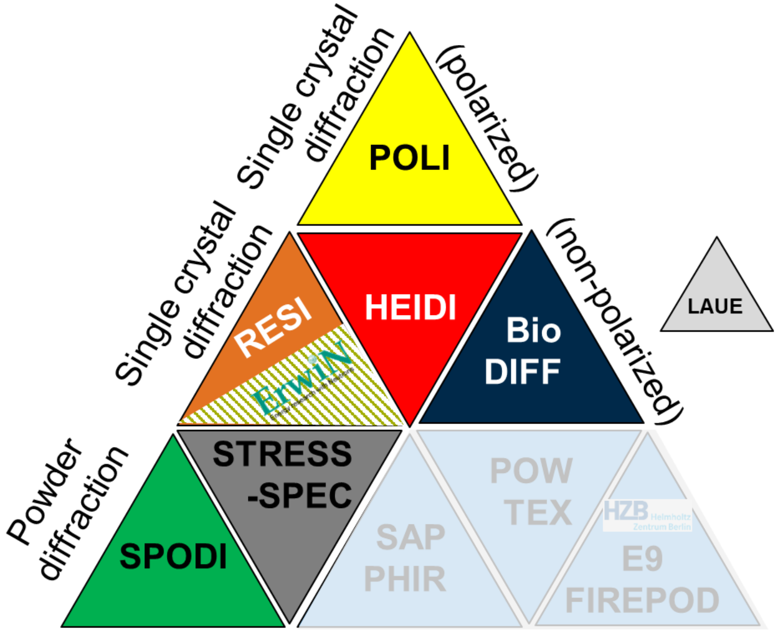
MLZ Group Structure Research



Sample Environment

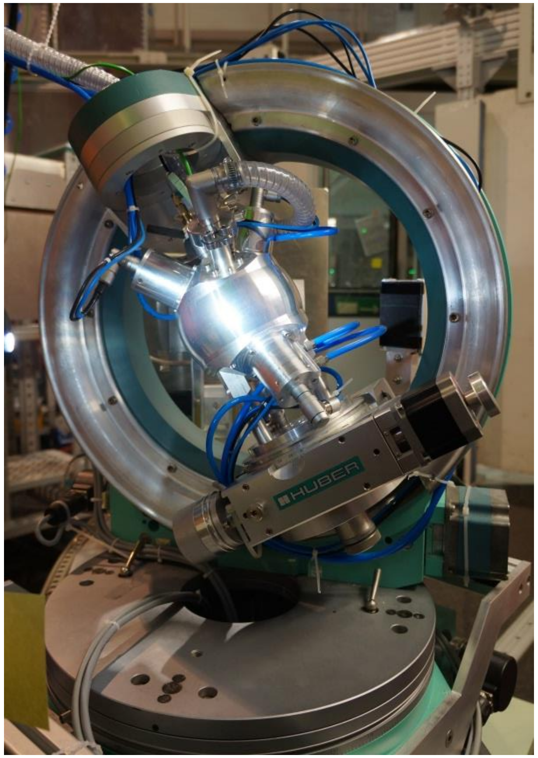
Courtesy: Dr. M. Meven

T< RT, Closed Cycle cryostat

- **T_{min} = 2.5 K** (1.9 K at heater)
- Accessible reciprocal space > 90%
  Angular limits: -45° < χ < +81°
- E-Field option up to ≈ 2kV

T > RT

- Micro furnace (RWTH), T < 500 K
  air cooled
- **Mirror furnace (TUM), T> 1000°C**
  2017: Gas handling in sample chamber



Consideration of High Pressure Conditions

Courtesy: Dr. M. Meven

A. Collection of Bragg data set

Significant shading of accessible reciprocal space from DAC

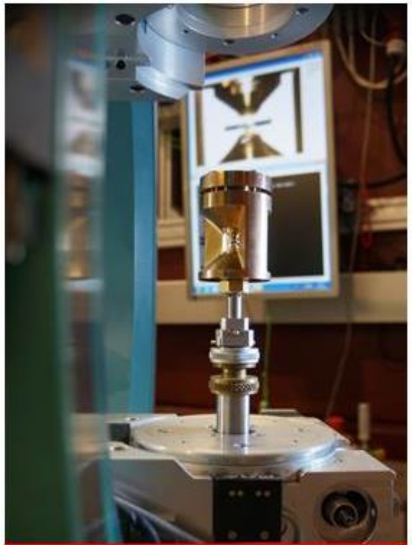
transmission DAC

tube/cylinder
with conical
entrance & exit

transmission DAC - HEIDI

panoramic DAC

tip connected cones
and posts



panoramic DAC - HEIDI

B. Correction and reduction of raw data

- Remove additional absorption effects of beam path through DAC to/from sample, e.g. absorption in gasket and/or other DAC components

- Consider „diamond dips“

occasional Bragg scattering in diamond anvils → intensity weakening

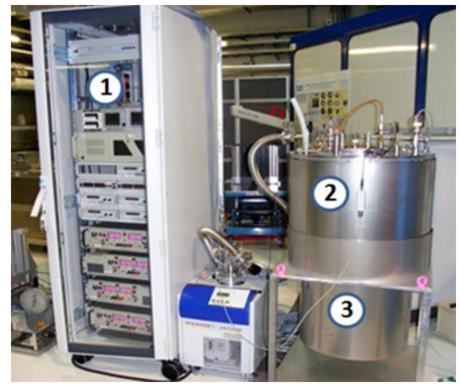
Courtesy: Dr. J. Xu

Single crystal diffraction - POLI

General

- **Cryostat: Janis**
- **8 T vertical magnet**
- **Dilution insert**
- **CryoPAD (like a Cryostat)**
    - Cooling down
    - Connect auto-N2 filling
    - Fill liquid He every a few days

CryoPAD



Piezo-goniometer

Special

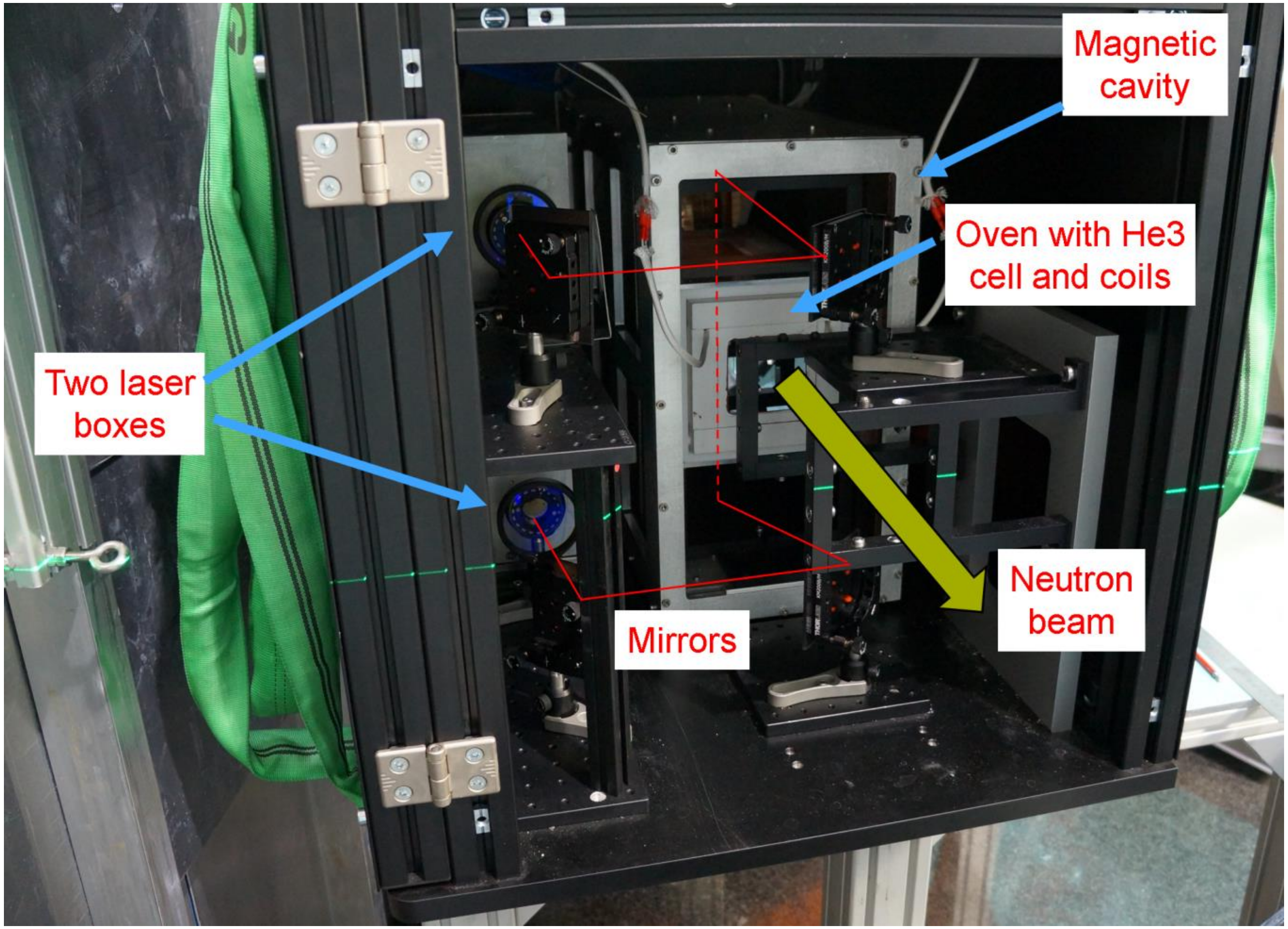
- **Pressure cells**
- **Electric fields**
- **Piezomotor goniometer and translation stages on the sample stick**
- **SEOP neutron polarizer**
  contains oven, laser, cooling, coils

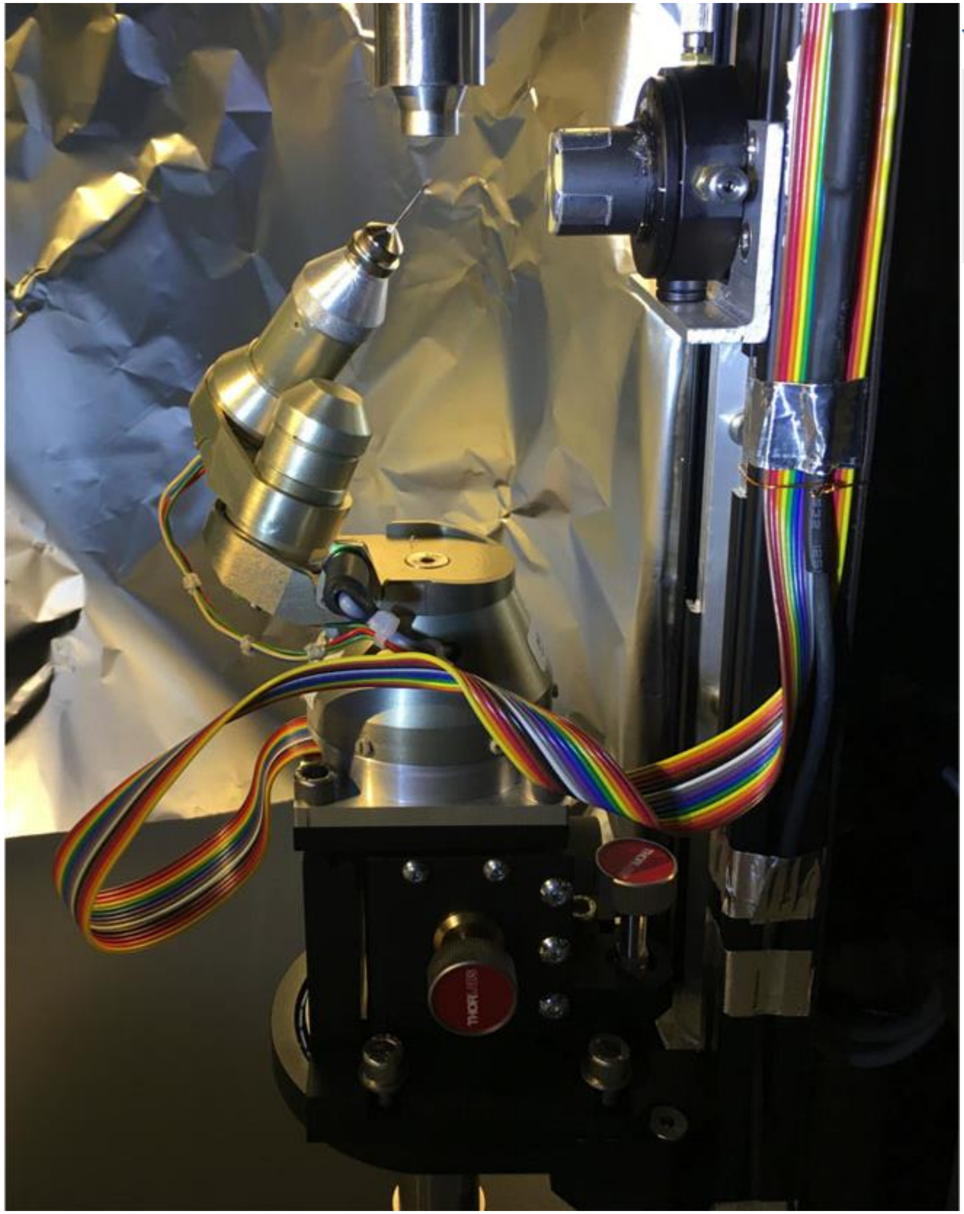
Pressure cells

Electric field

In-situ SEOP He3 neutron polarizer

Courtesy: Dr. J. Xu





▾ **Sample Environment**

Besides standard sample environment BIODIFF provides:

- Oxford Cryosystems Cryostream 700 plus with a temperature range of 90 K to 500 K
- Closed cycle cryostat 3.5 – 325 K

▾ **Technical Data**

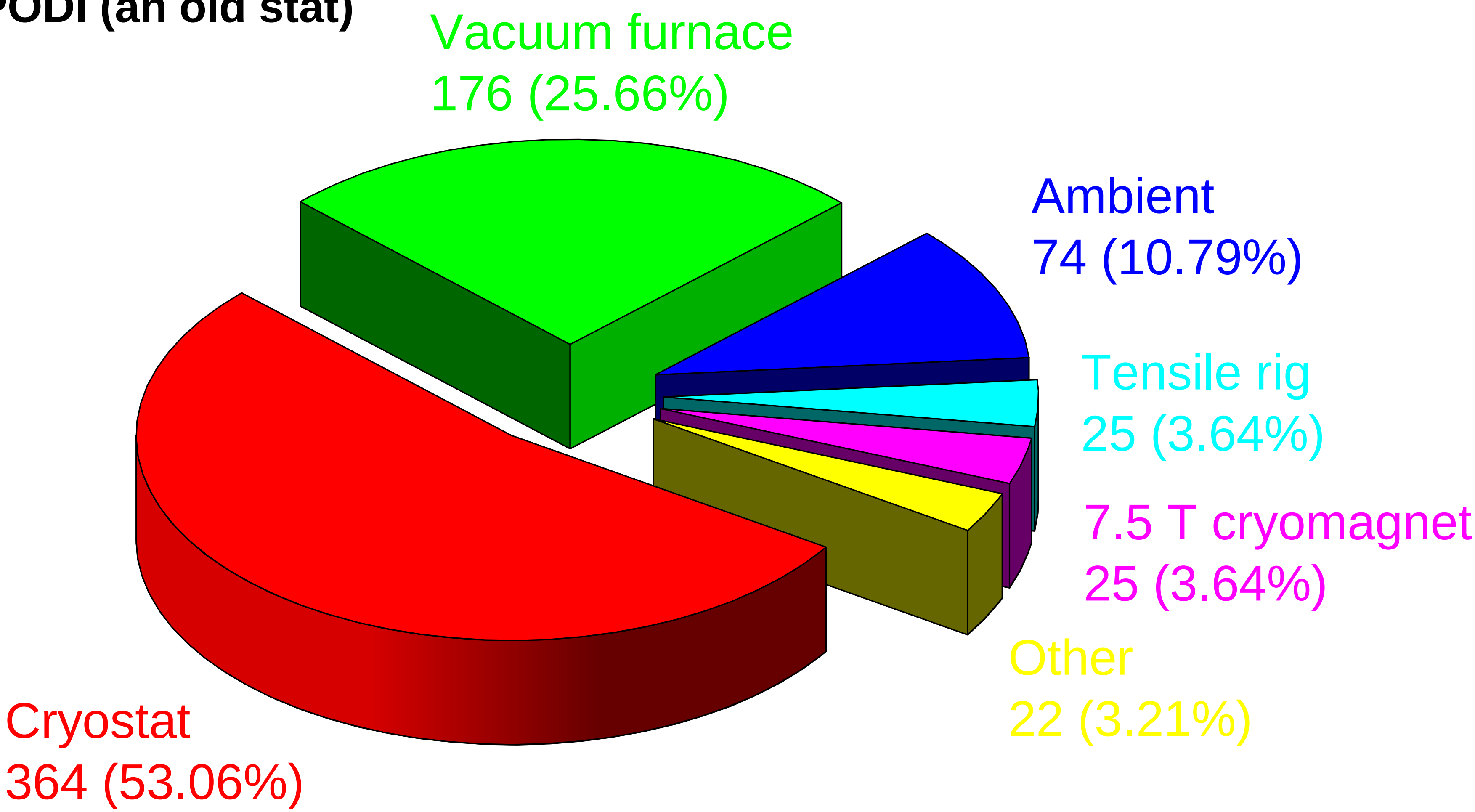
Primary beam
- Neutron guide NL-1; supermirror m = 2
- Monochromator:
  - Pyrolytic graphite (PG), mosaicity: 0.4 – 0.5°
- Higher order filter:
  - Astrium type velocity selector
  - transmission 87 % for 2.7 Å
- Wavelength range:
  - 2.0 Å with PG(004): $3.0 \cdot 10^6$ n cm^{-2} s^{-1}. Limit: lattice constants up to 80 Å.
  - 2.7 Å with PG(002): $4.0 \cdot 10^6$ n cm^{-2} s^{-1}. Limit: lattice constants up to 110 Å.
  - 3.4 Å with PG(002): $1.8 \cdot 10^6$ n cm^{-2} s^{-1}
  - 4.0 Å with PG(002): $1.0 \cdot 10^6$ n cm^{-2} s^{-1}
- Collimation by adjustable slits between 1 – 4 mm

Beam properties at the sample position
- Wavelength resolution at sample position: $\Delta\lambda/\lambda$ = 2.9 % at 2.7 Å with PG(002)
- Wavelength resolution at sample position: $\Delta\lambda/\lambda$ = 1.5 % at 2.0 Å with PG(004)
- Beam divergence (no slits):
  - 0.8° FWHM horizontal
  - 0.7° FWHM vertical

Main detector Neutron image plate (cylindrical)
- $BaFBr:Eu^{2+}$ mixed with Gd_2O_3
- Dimensions:
  - radius: 200 mm
  - angular range:
    - ±152° horizontal

Powder diffraction

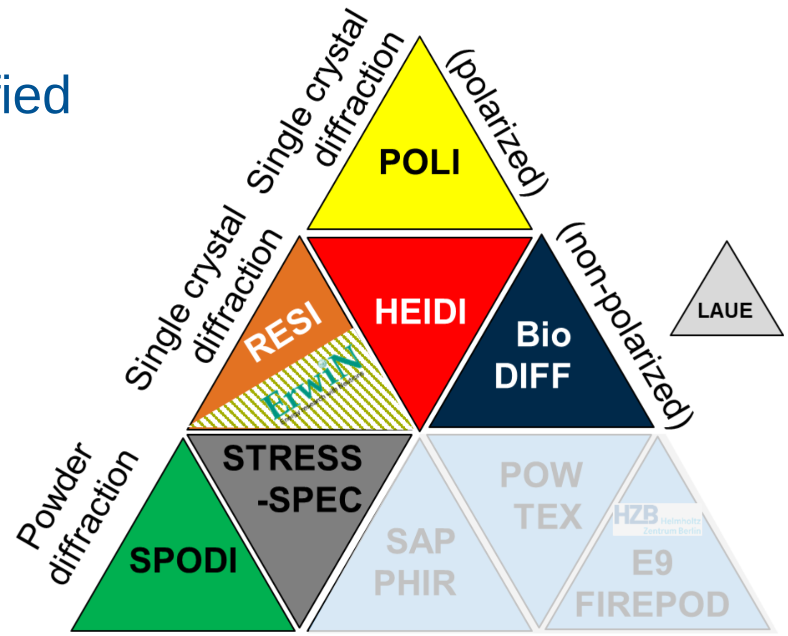
Sample environment usage at SPODI (an old stat)



Sample environment at SPODI: past, present and future

Sample environment already in use at SPODI

- CCR cryostats (SPODI equipment, modified FRM II standard)
- High-temperature furnace (SPODI equipment, FRM II standard)
- Automatic sample changer (SPODI equipment)
- Potentiostats for batteries (SPODI equipment)
- Tensile rig (SPODI equipment)
- Electric field setup (SPODI equipment)
- 5.5 T magnet (from FRM II pool)
- ^{3}He inserts



Operation of ERWIN and FIREPOD besides SPODI will enhance the demand for sample environment

- Common pool for three diffractometers
- Supply of standard sample environment equipment
- Maintenance of standartizised sample environment equipment

Courtesy: Dr. M. Hölzel

(SPODI) Assistance and support from sample environment group needed

- Maintenance of cryostats and high-temperature furnaces
- “Automated sample change at low temperatures" projects
- Set-up and commissioning of ADR cryostats and its extension towards automated measurements
- "High temperature measurements at different atmospheres“
- Adaptation and usage of magnets (5.5 T and 8.0T POLI magnet)

(SPODI) Projects with sample environment group

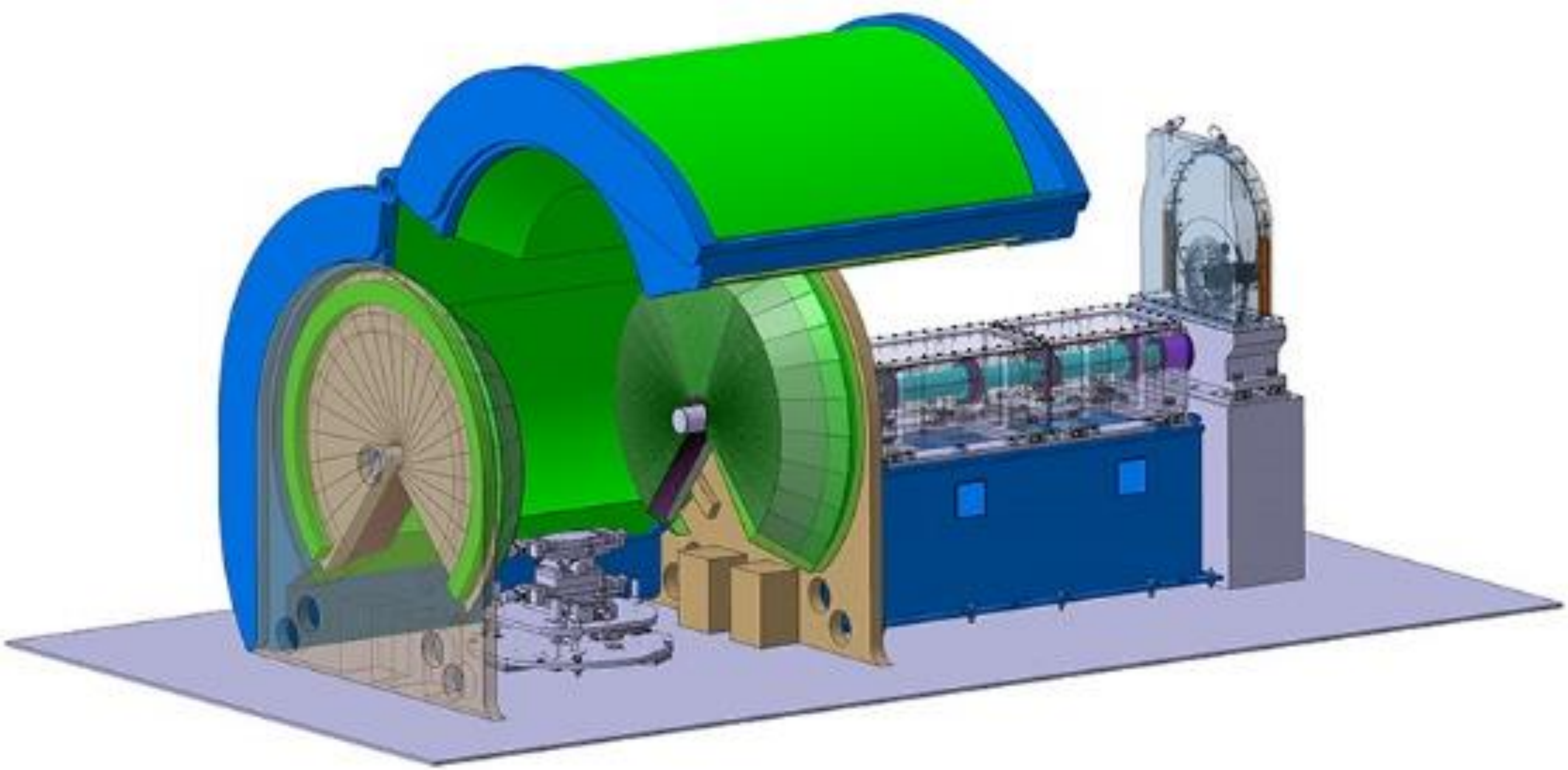
-Completion of the cryogenic sample changer for CCR

-Use of robotic arms on cryostats - automated sample stick exchange

-High temperature measurements at different atmospheres, including gas flow rates using an exchange sample stick

Courtesy: Dr. M. Hölzel

Sample environment at POWTEX



Sample Environment
Road Map: viewpoint of Structure Research

- Maintenance of existing projects (low and high temperatures, high magnetic fields)
- Extension and upgrade of existing sample environments
- New types of sample environments: development and conceptualisation (gases, high pressures)
- Automated sample change and alignment, robotics