Sample environment devices

Eddy Lelièvre-Berna

lelievre@ill.eu - http://www.ill.eu/sane

Content

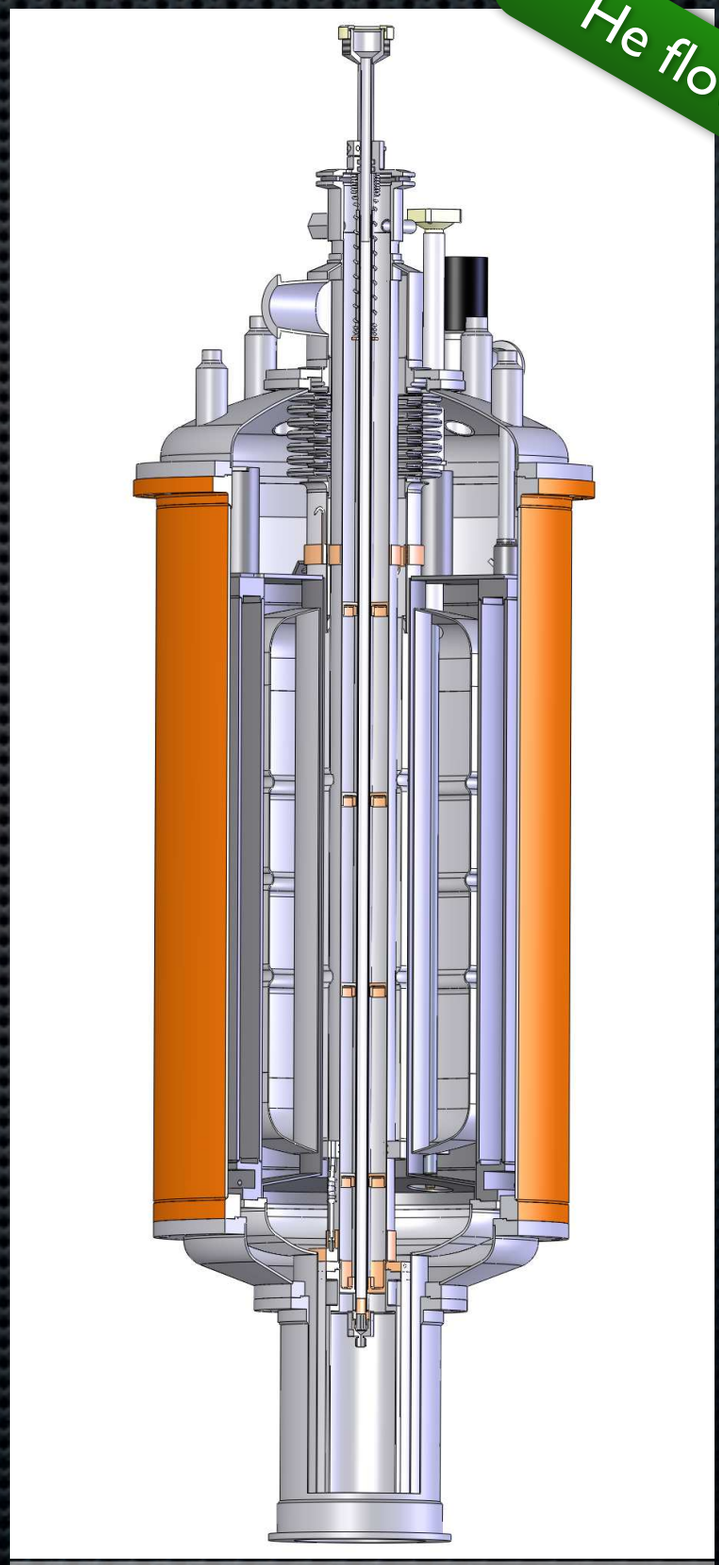
- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

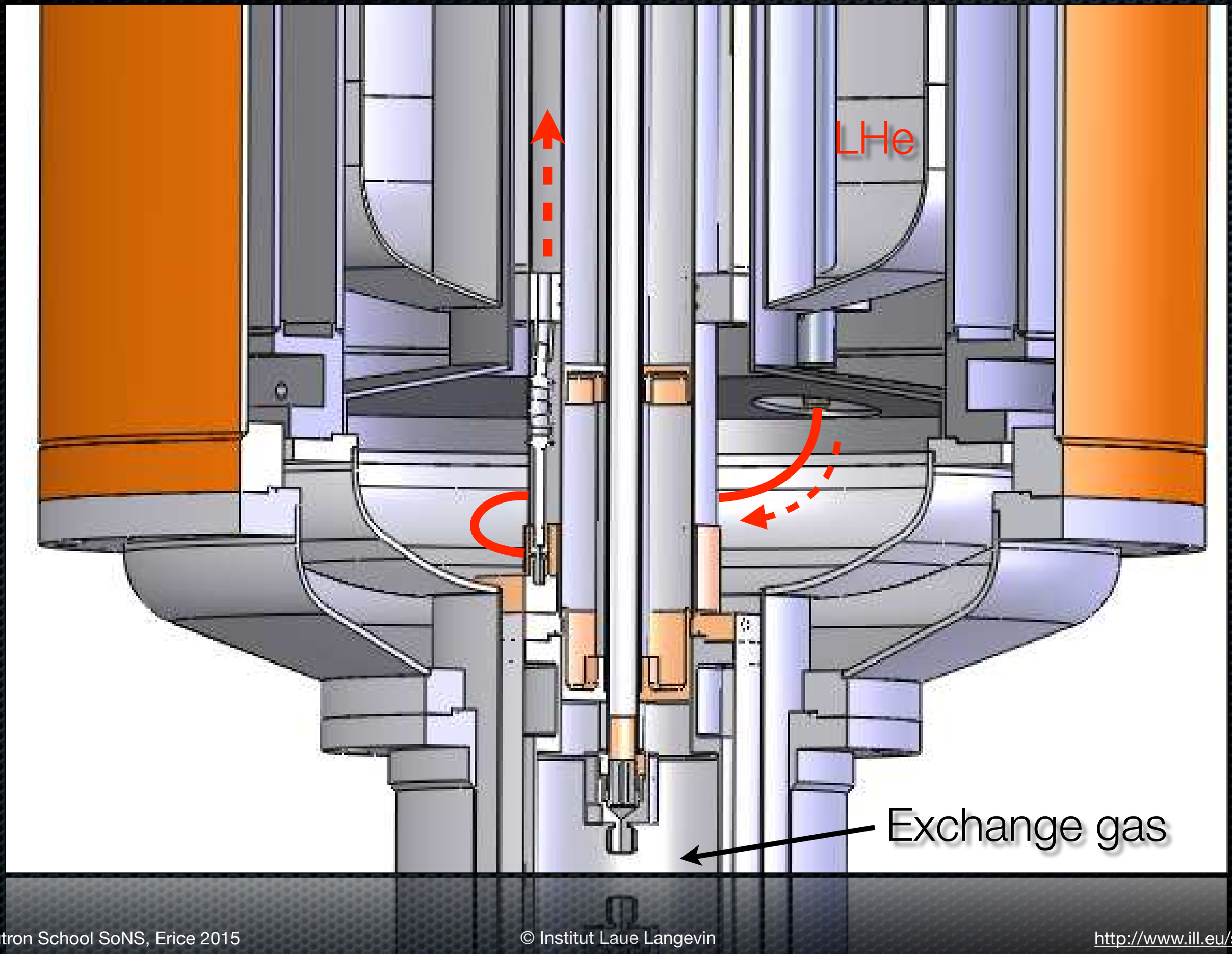
Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Cryogenics

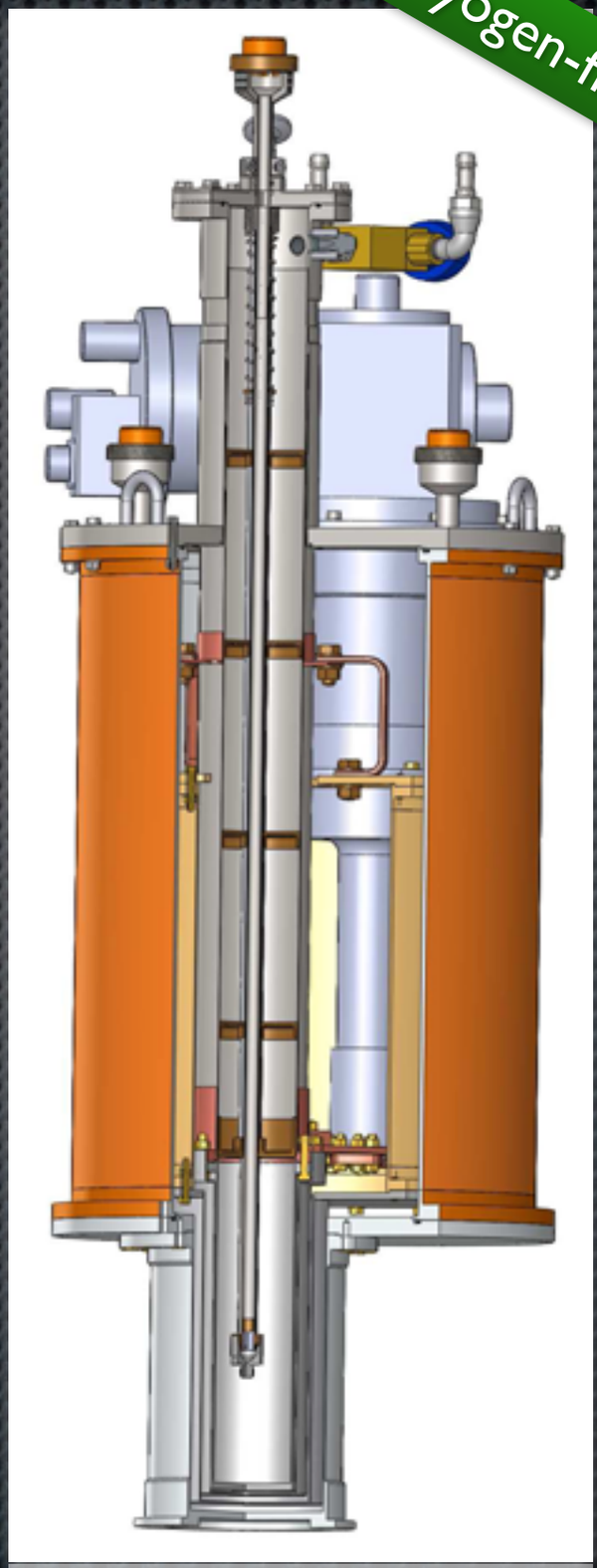
- **Cryostats (Ø330-450 mm)**
  1.5 / 2.8 to 320 K
- **Cryofurnaces (Ø330-450 mm)**
  1.5 to 550 / 650 K
- **Dry cryostats (cryogen-free)**
  1.8 to 320 K with JT
  2.7 to 620 K without JT
- **Sample changers, gonios ?**





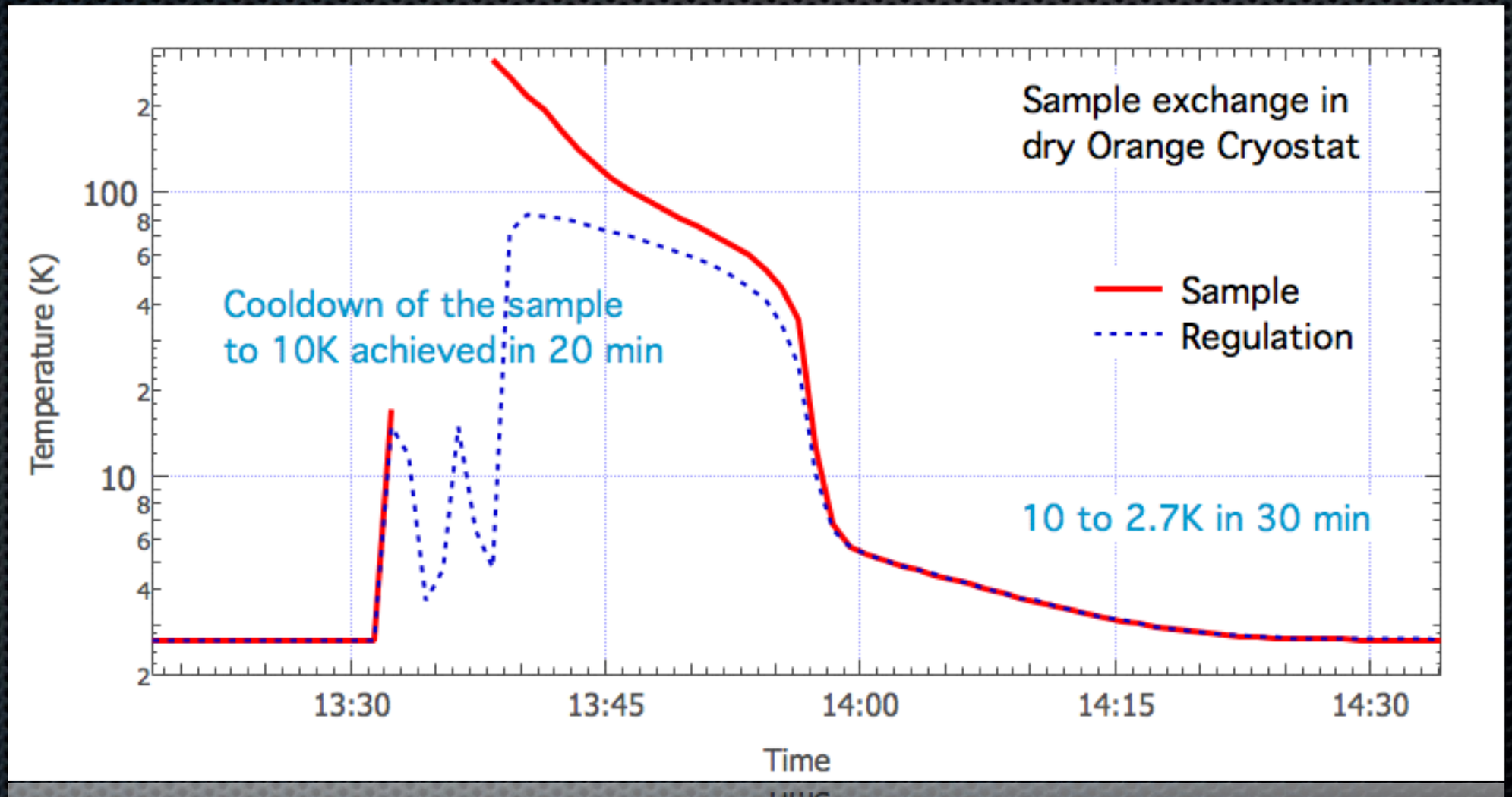
Cryogenics

- **Cryostats (Ø330-450 mm)**
  - 1.5 / 2.8 to 320 K
- **Cryofurnaces (Ø330-450 mm)**
  - 1.5 to 550 / 650 K
- **Dry cryostats (cryogen-free)**
  - 1.8 to 320 K with JT
  - 2.7 to 620 K without JT
- **Sample changers, gonios ?**



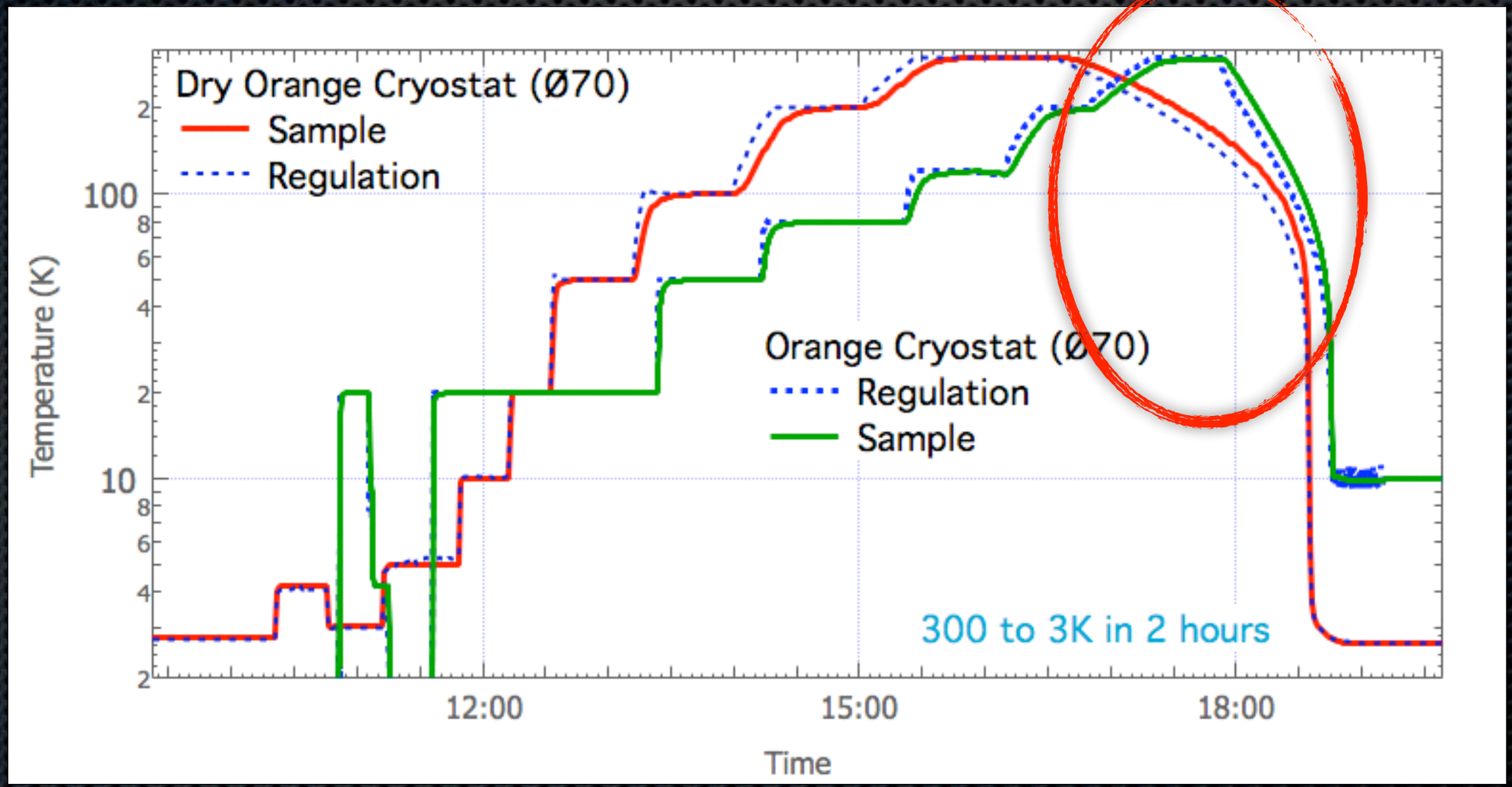
Cryogenics

dry cryostats are easy to use and look fast…



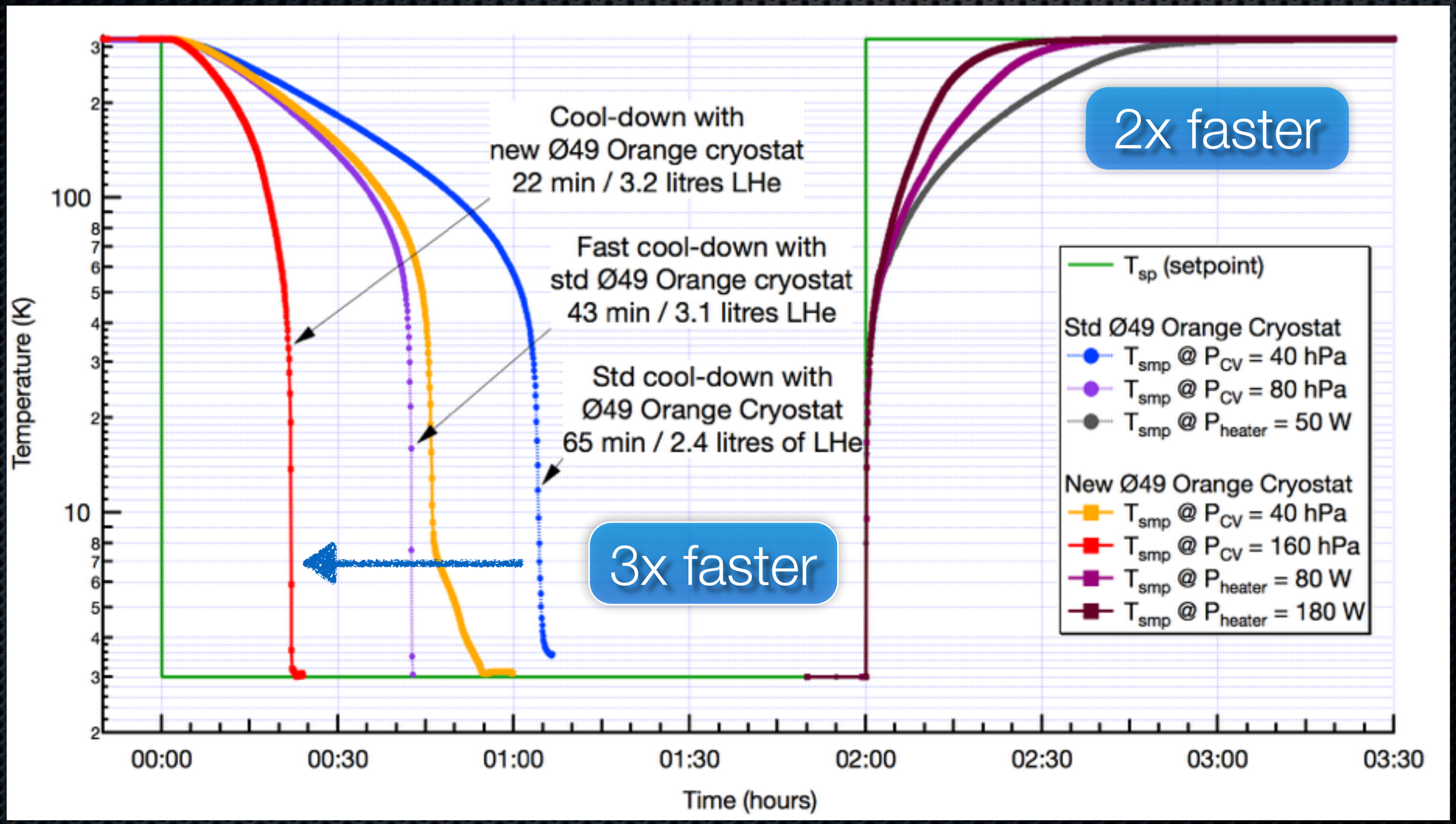
Cryogenics

…but cryogen-free cryostats are 2x slower !



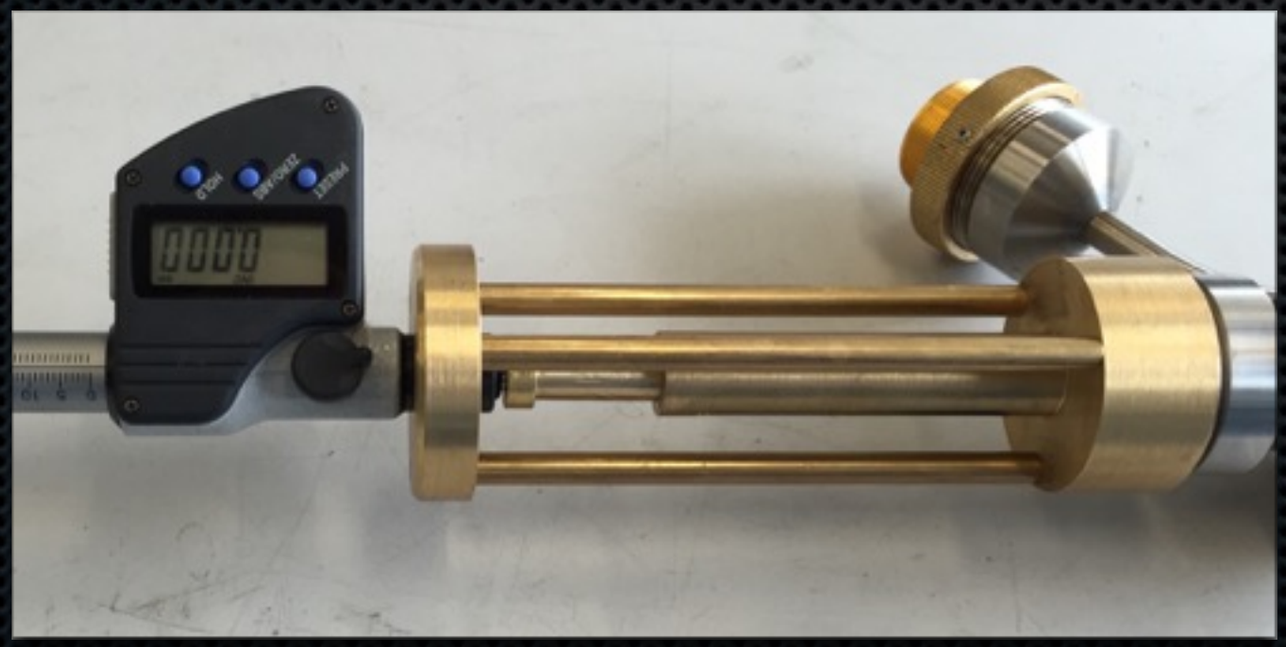
Cryogenics

Faster cryostats for new-generation instruments ?



Cryogenics

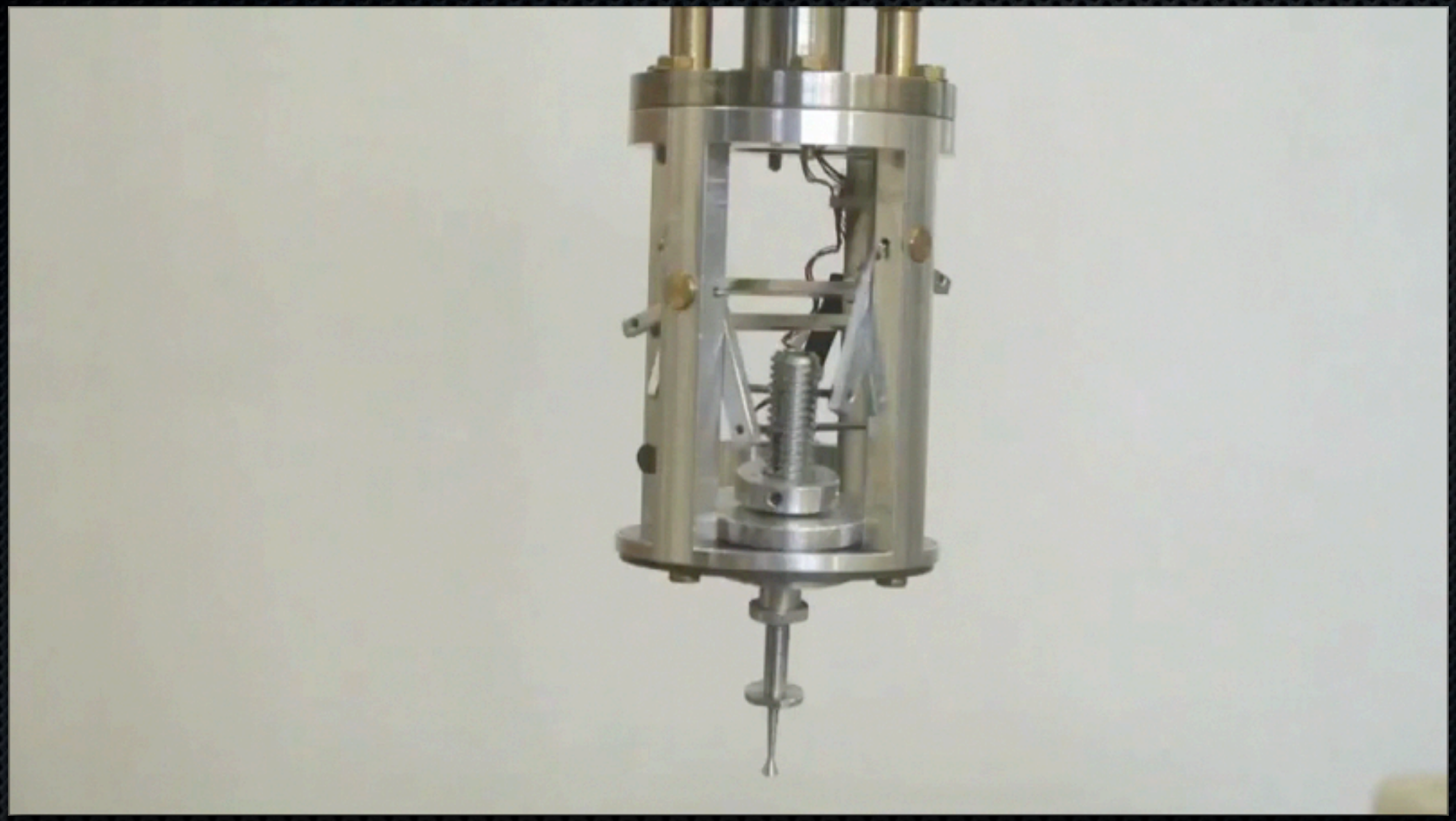
De-twinning crystals remotely inside cryostats…



Cryogenics

Orienting samples inside cryostats…

- **Cryostat position fixed by optics (detector, guide…)**
  - ➥ **Goniostick**
  - ±7° sample tilting (±0.02°)
  - ±10 mm along Oz
  - ±180° around Oz
  - non-magnetic
  - remotely controlled



Goniostick

Manual operation recorded at the lab.

Cryogenics

Orienting samples inside zero-field polarimeters…

- **Cryostat inside zero-field chamber (polarimetry)**
  - ➥ **Cryocradle**

$3 < T < 320K$

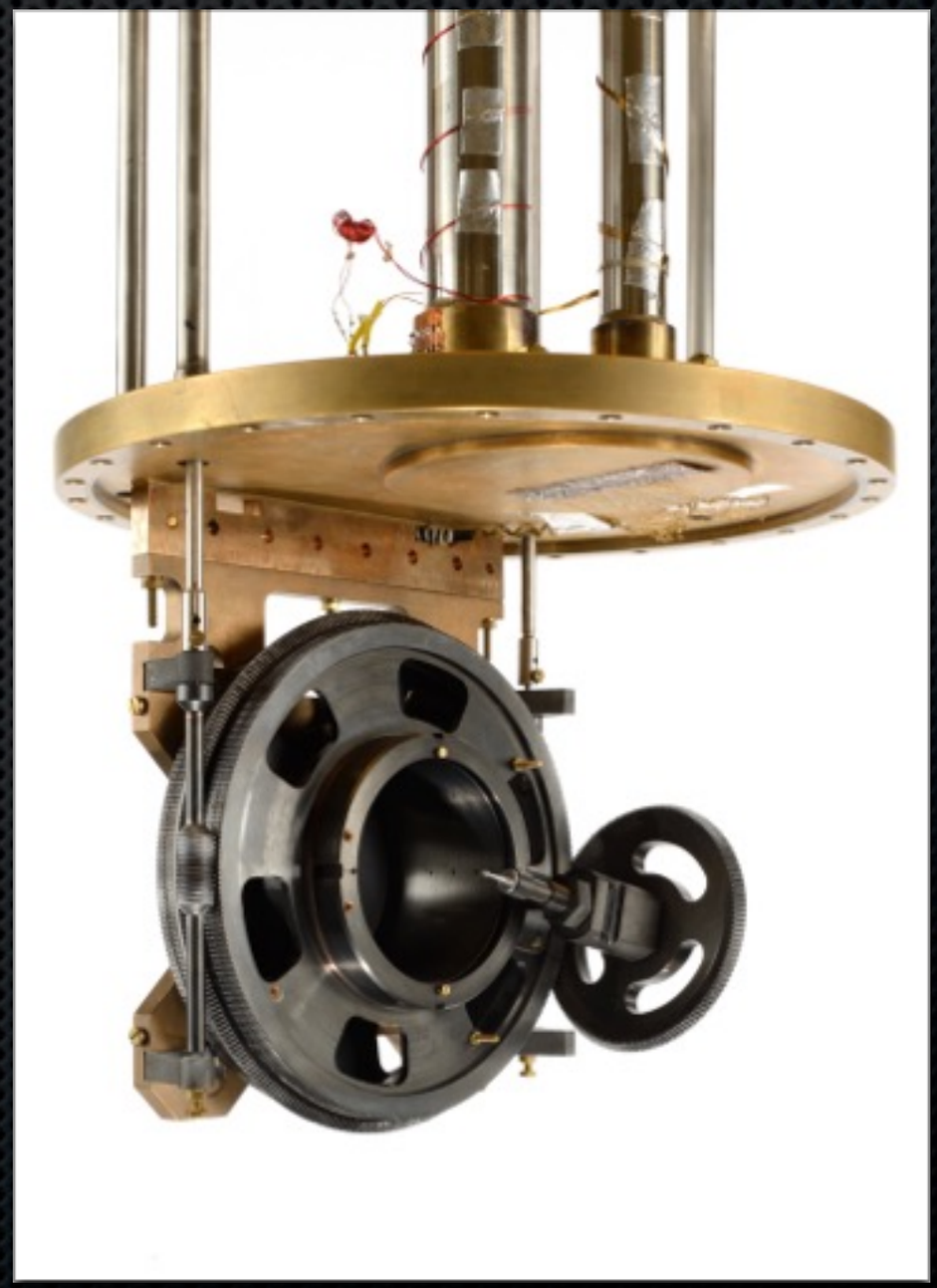
$-30 < \chi < +210°$

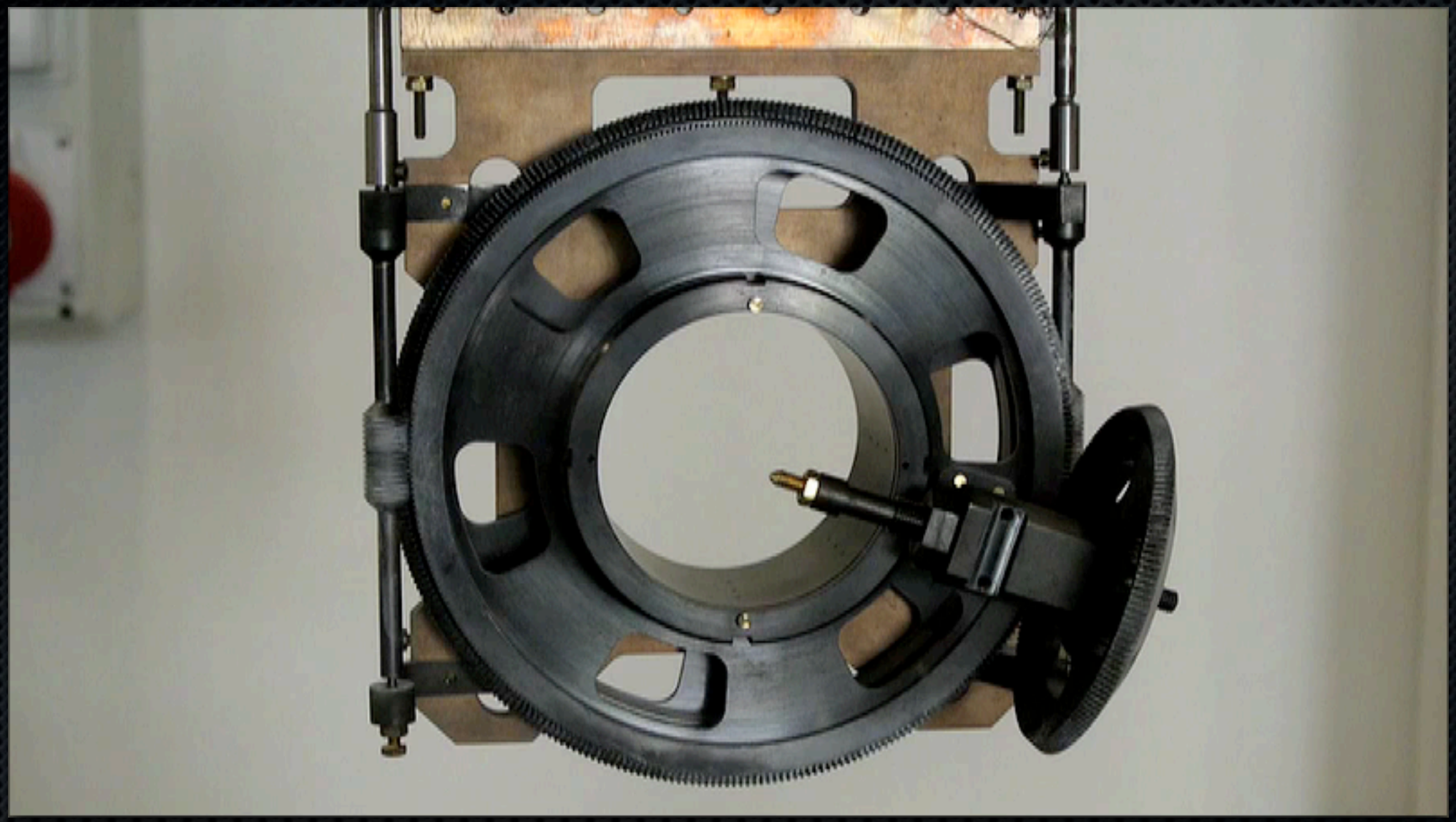
$-180 < \phi < +180°$

$-40 < 2\theta < +120°$

non-magnetic

remotely controlled





Cryocradle

Remote operation recorded at the lab.

Cryogenics

- **^{3}He fridges (Ø400 mm)**

  350 mK to 320 K

- **Dilution fridges/inserts**

  15 / 35 mK to 320 K

- **Compact dilution fridge**

  110 mK to 320 K

- **Large dilution fridges**

  for high-pressure cells, complex environment…

Cryogenics

- **^{3}He fridges (Ø400 mm)**

  350 mK to 320 K

- **Dilution fridges/inserts**

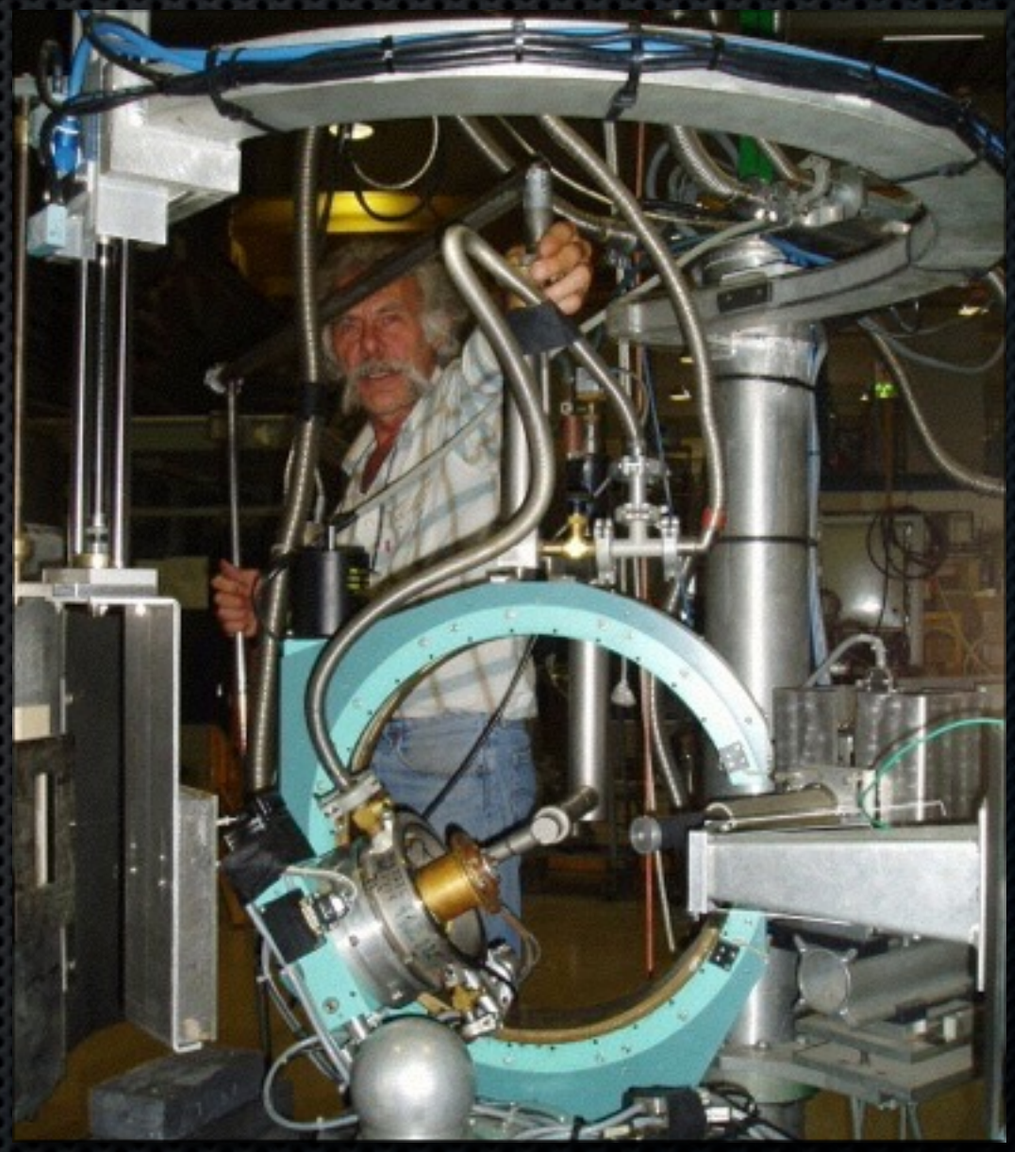
  15 / 35 mK to 320 K

- **Compact dilution fridge**

  110 mK to 320 K

- **Large dilution fridges**

  for high-pressure cells, complex environment…



Cryogenics

- **^{3}He fridges (Ø400 mm)**

  350 mK to 320 K

- **Dilution fridges/inserts**

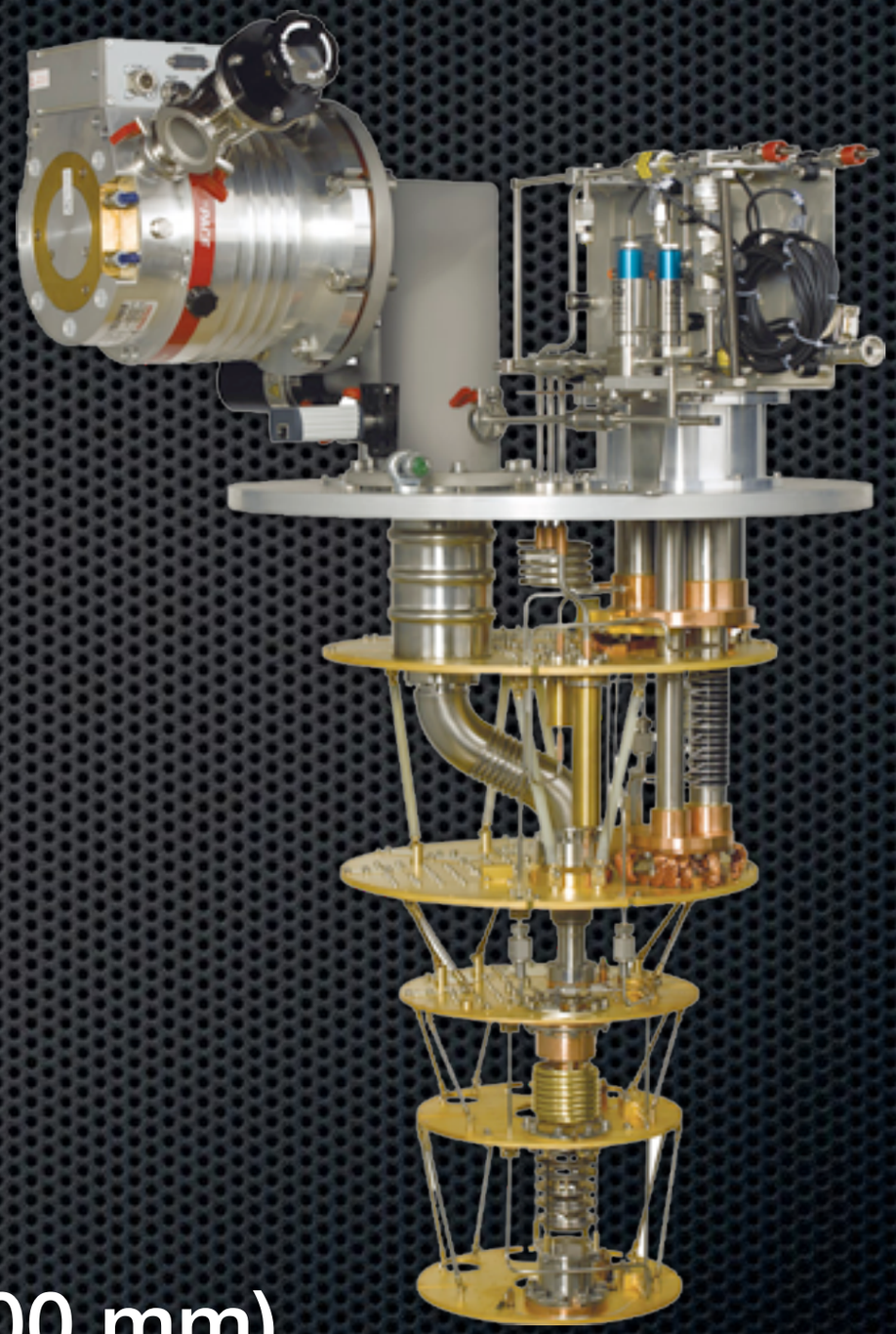
  15 / 35 mK to 320 K

- **Compact dilution fridge**

  110 mK to 320 K

- **Large dilution fridges (>Ø800 mm)**

  for high-pressure cells, complex environment…



Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Content

- Low temperature cryostats
- High temperatures furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

High temperature

- **Resistive furnaces (Ø340)**
  - 320 to 2000K
  - V, Nb resistors in beam
  - Sapphire windows
  - Compact version
- **Auto-power racks (350 kg)**
  - 2 kVA and 3.5 kVA
  - Ethernet I/O

High temperature

- **Resistive furnaces (Ø340)**
  - 320 to 2000K
  - V, Nb resistors in beam
  - Sapphire windows
  - Compact version
- **Auto-power racks (350 kg)**
  - 2 kVA and 3.5 kVA
  - Ethernet I/O

High temperature

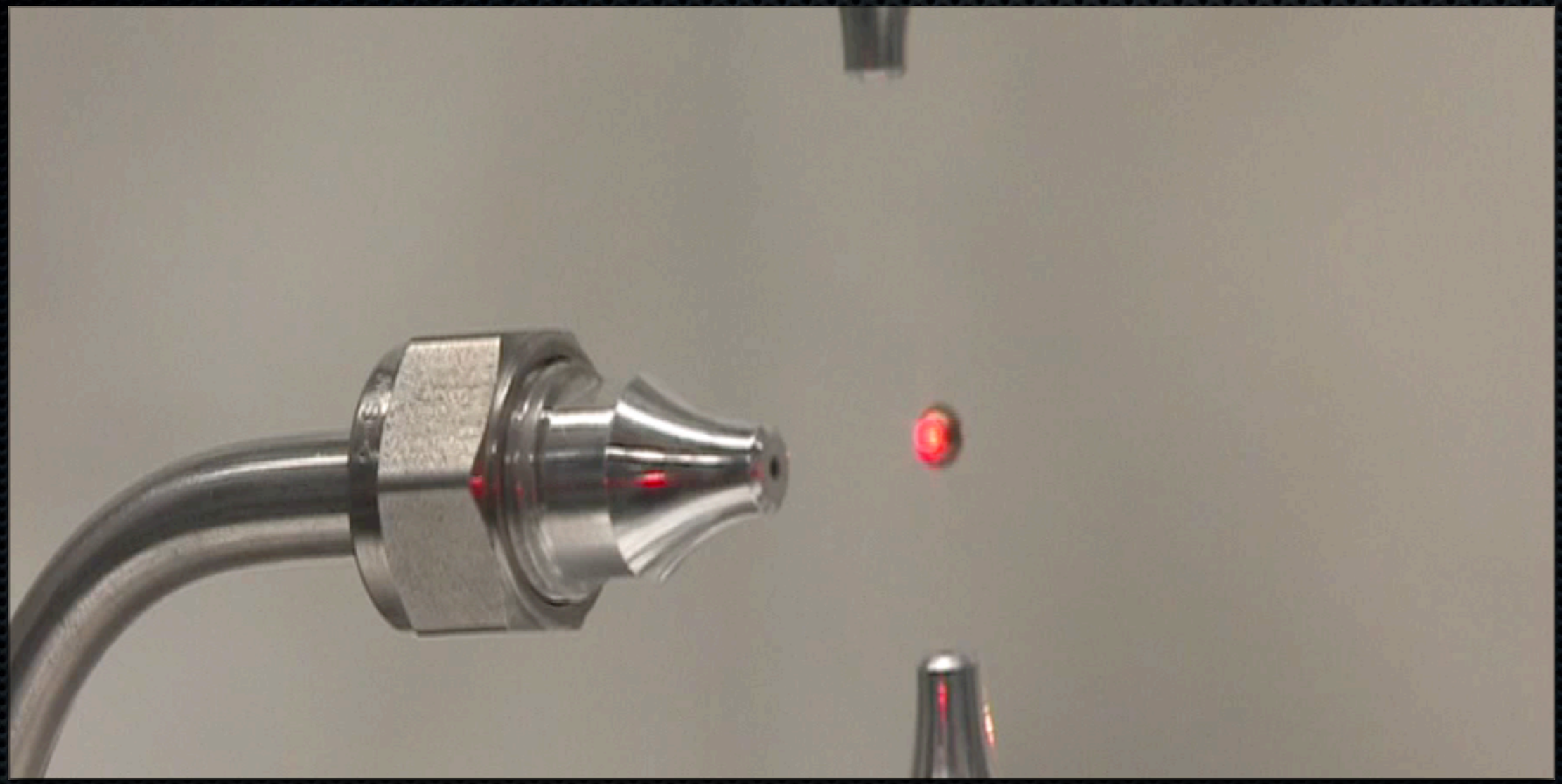
- **Resistive furnaces (Ø340)**
  - 320 to 2000K
  - V, Nb resistors in beam
  - Sapphire windows
  - Compact version
- **Auto-power racks (350 kg)**
  - 2 kVA and 3.5 kVA
  - Ethernet I/O

High temperature

- **Resistive furnaces (Ø340)**
  - 320 to 2000K
  - V, Nb resistors in beam
  - Sapphire windows
  - Compact version
- **Auto-power racks (350 kg)**
  - 2 kVA and 3.5 kVA
  - Ethernet I/O

Levitation furnace (electro.)

Requires more than 3 m^2 on the instrument



Levitation furnace (aerodyn.)

Requires more than 3 m^2 on the instrument

Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

High E-field

- **AC or DC field**

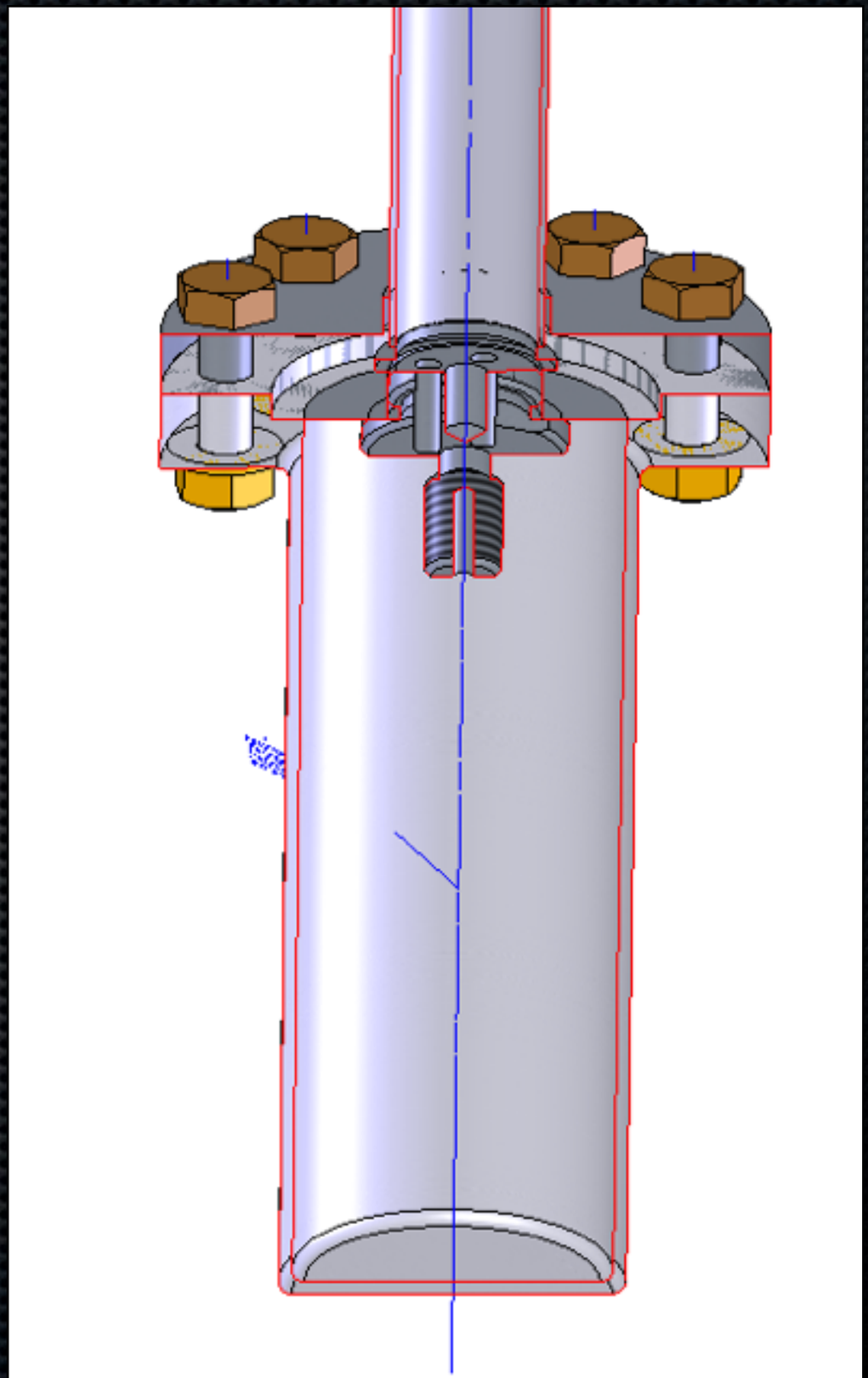
  10 kV power supply

  Custom electrodes

  Thermometers fixed to sample holder

  Sample in He during temperature change

  Sample in vacuum when applying E-field



High B-field

- **Vertical field (Ø800 mm)**

  up to 15T, top-loading

  40 mK dilution insert,

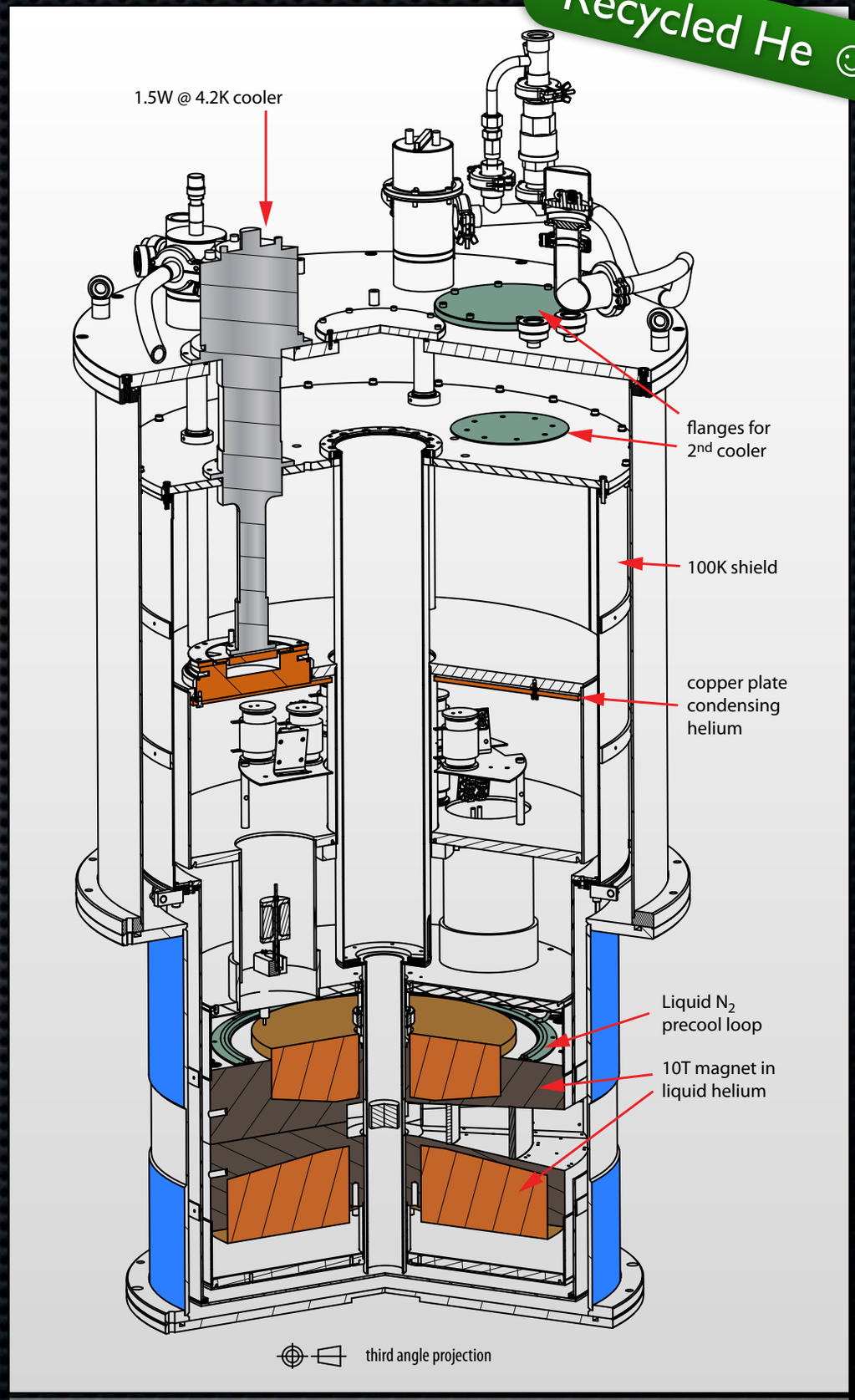
  symmetric or asymmetric ?

  self-shielded or not ?

  2T Dy booster + focusing ?

- **Horizontal field (≈400 mm)**

  up to 17T, bottom-loading



High B-field

- **Vertical field (Ø800 mm)**

  up to 15T, top-loading

  40 mK dilution insert,

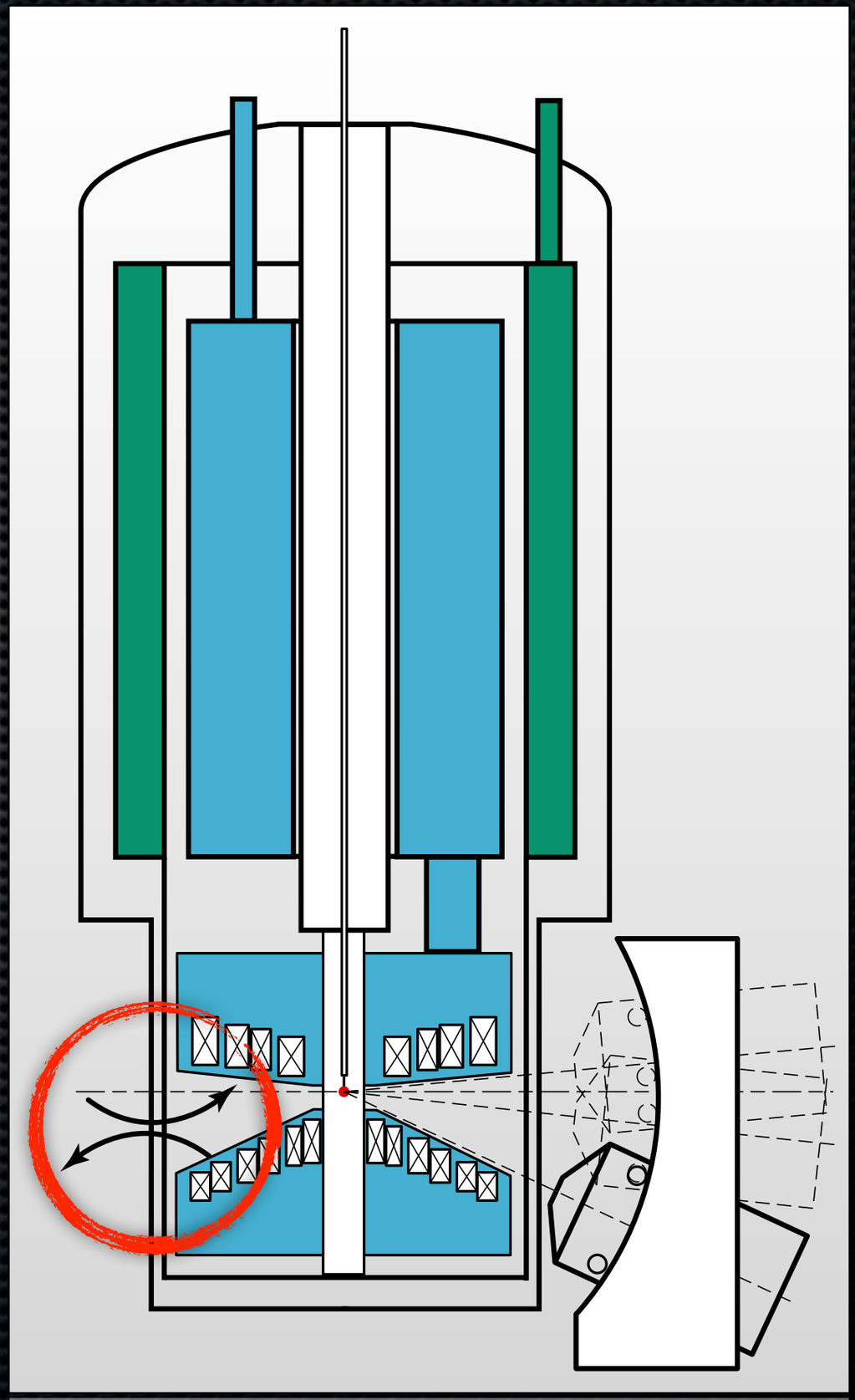
  symmetric or asymmetric ?

  self-shielded or not ?

  2T Dy booster + focusing ?

- **Horizontal field (≈400 mm)**

  up to 17T, bottom-loading



High B-field

- **Vertical field (Ø800 mm)**

  up to 15T, top-loading

  40 mK dilution insert,

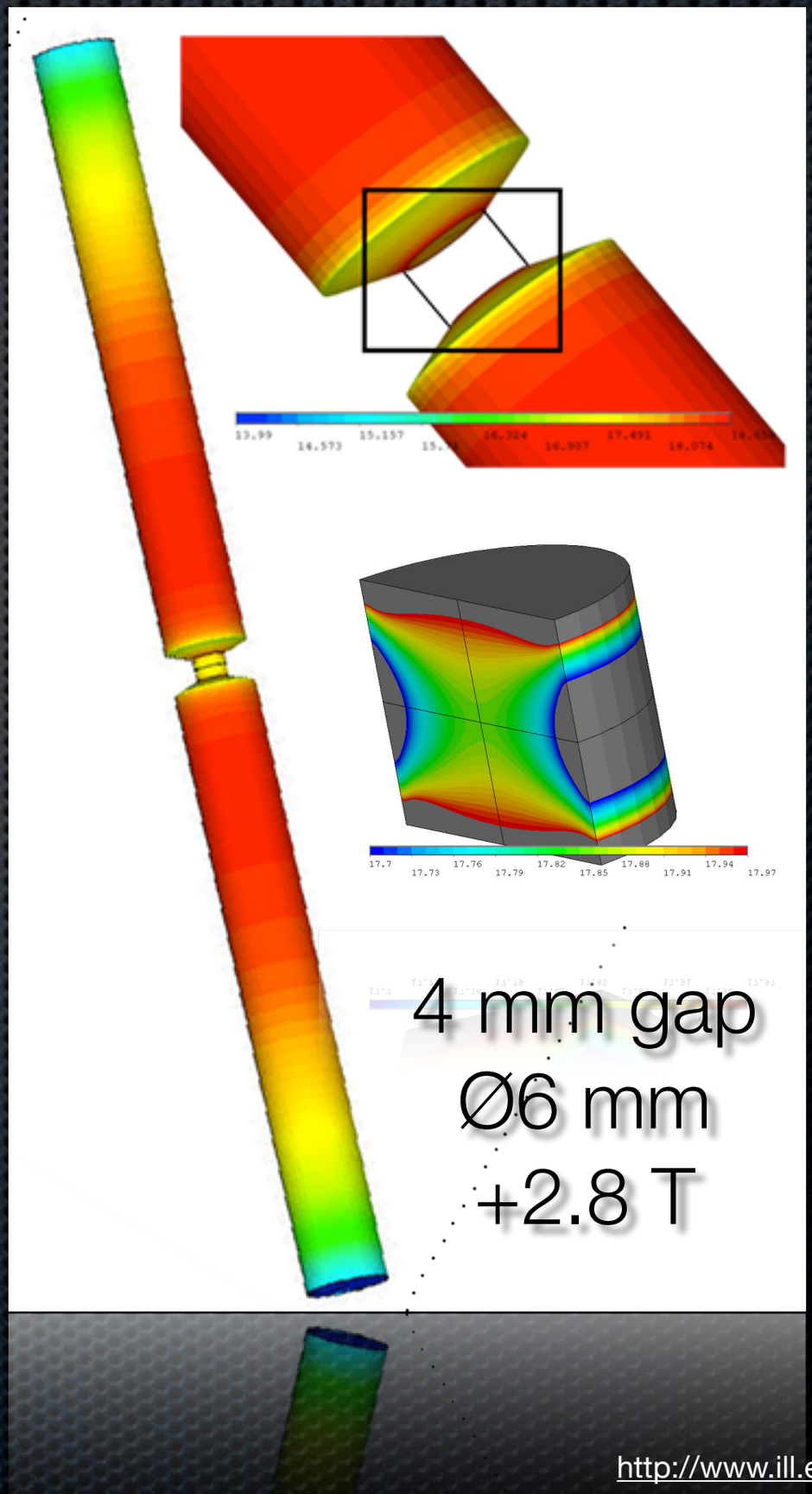
  symmetric or asymmetric ?

  self-shielded or not ?

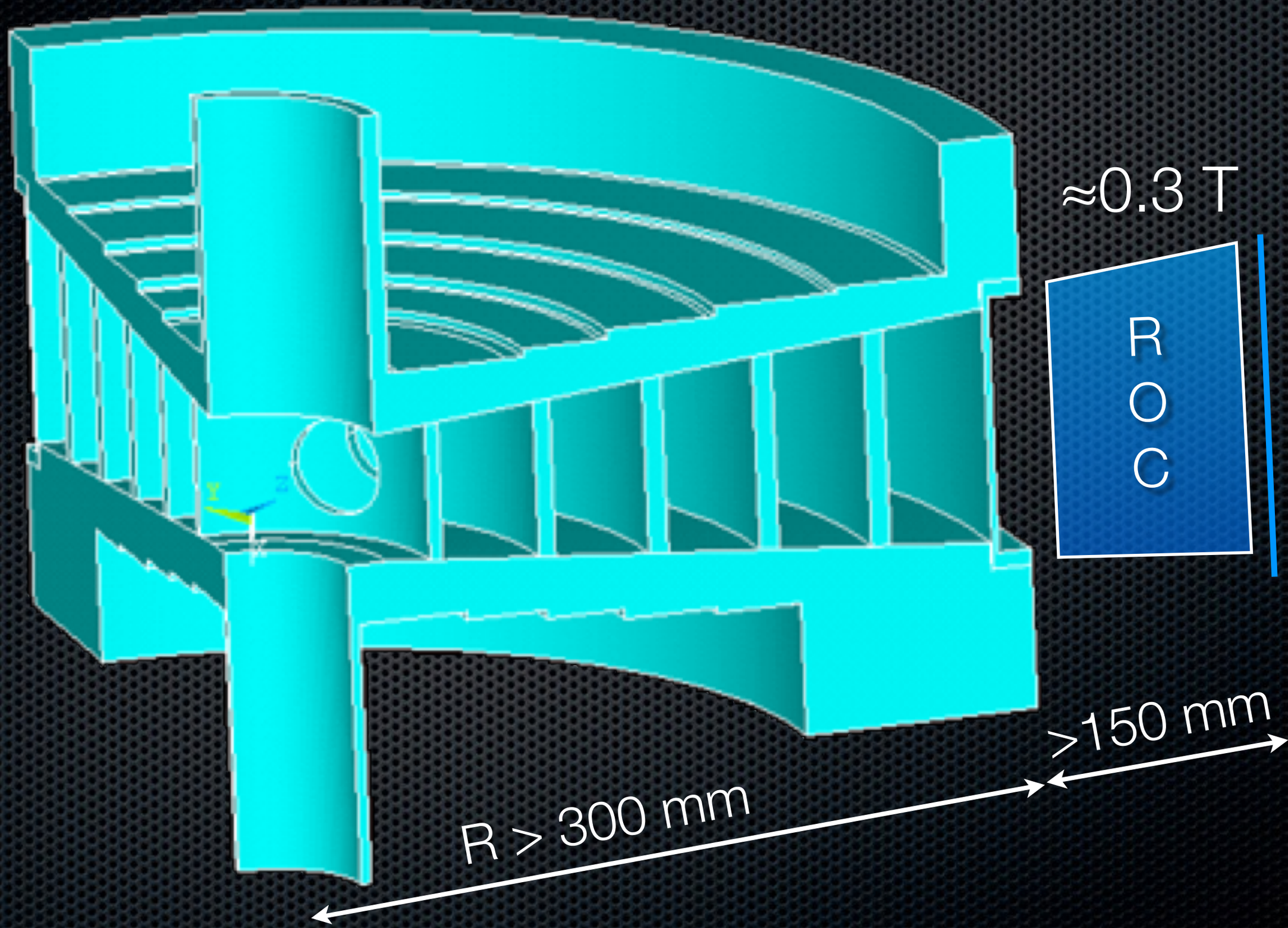
  2T Dy booster + focusing ?

- **Horizontal field (≈400 mm)**

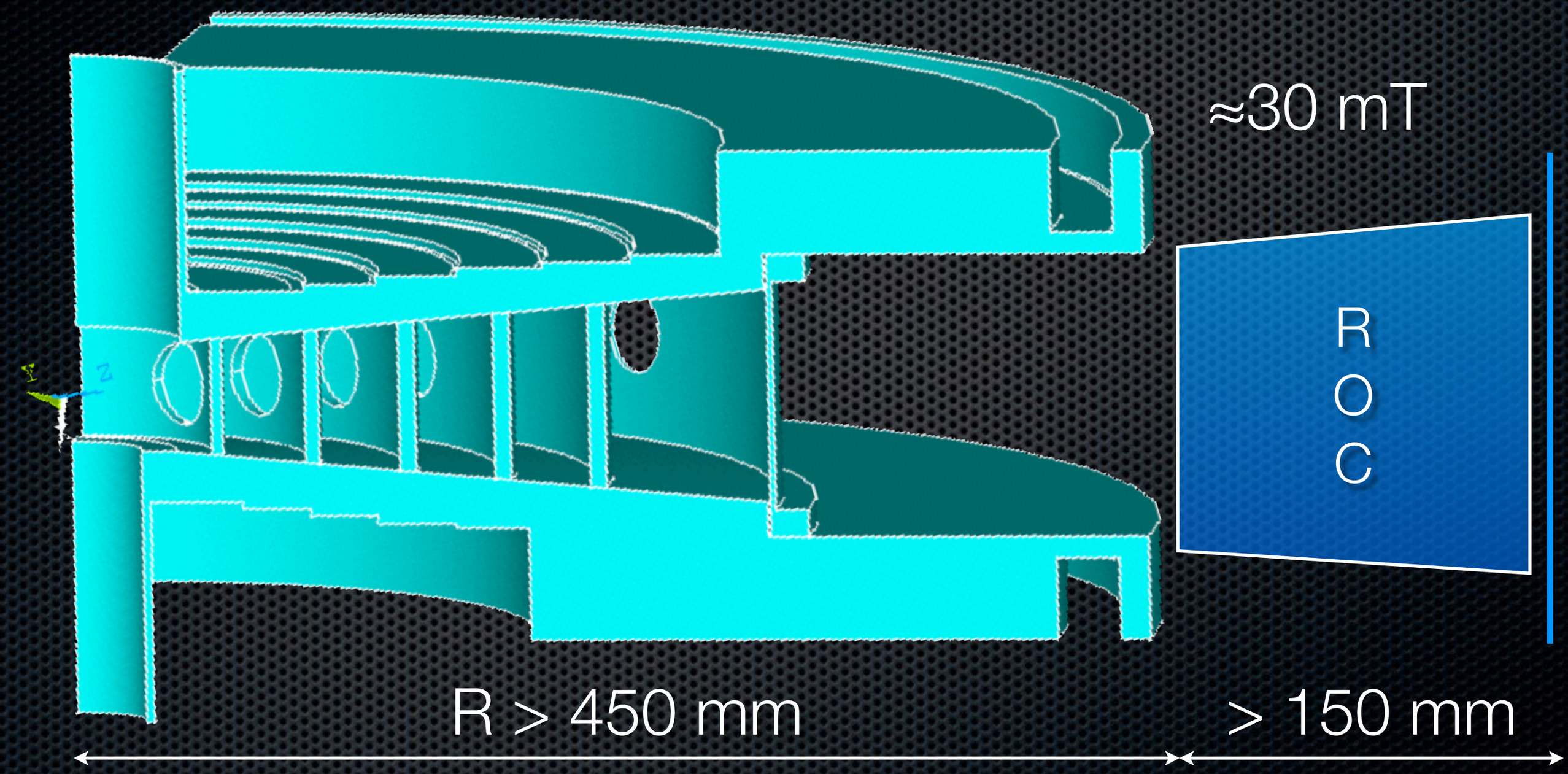
  up to 17T, bottom-loading



10T non-shielded magnet

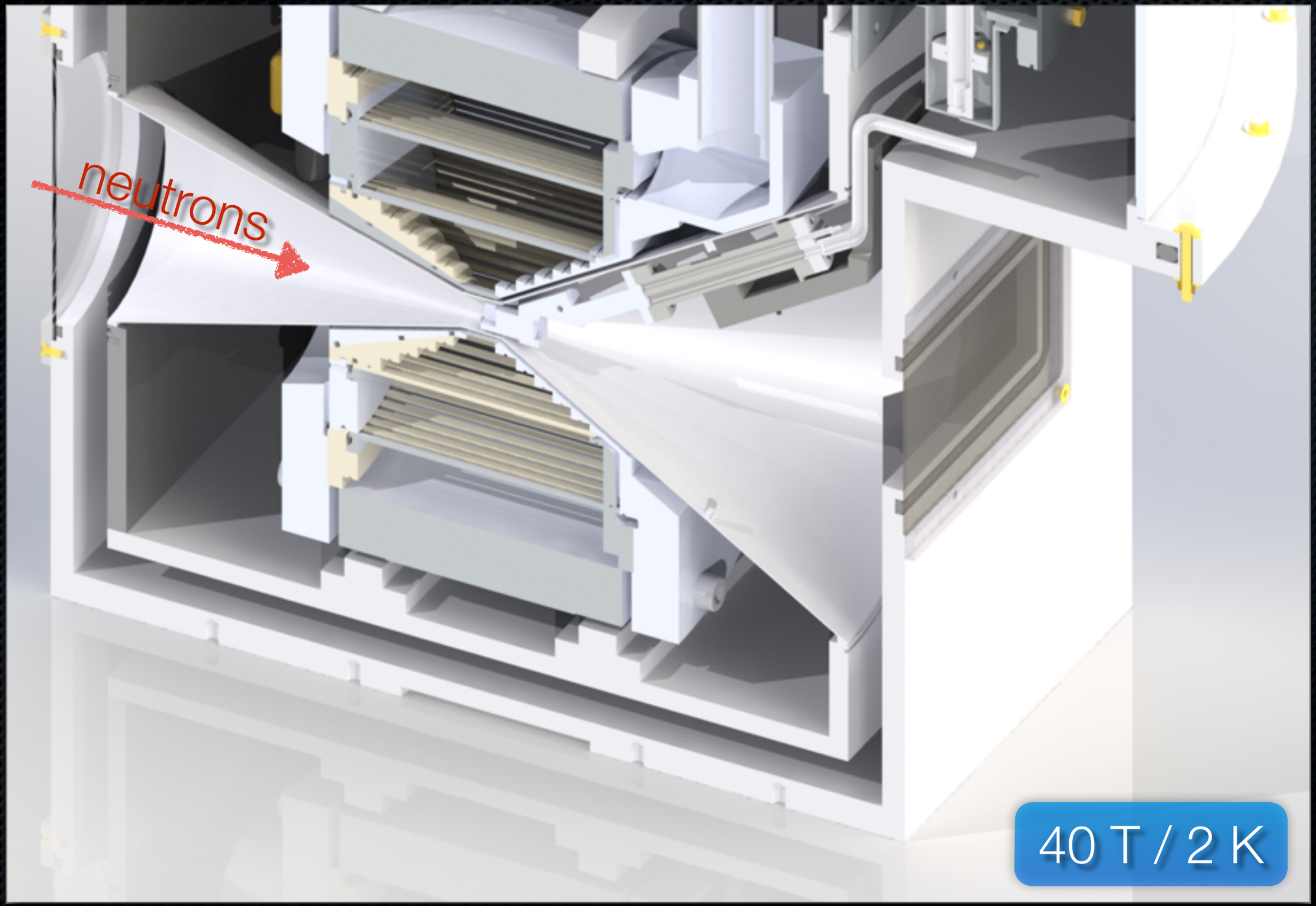


10T actively shielded magnet

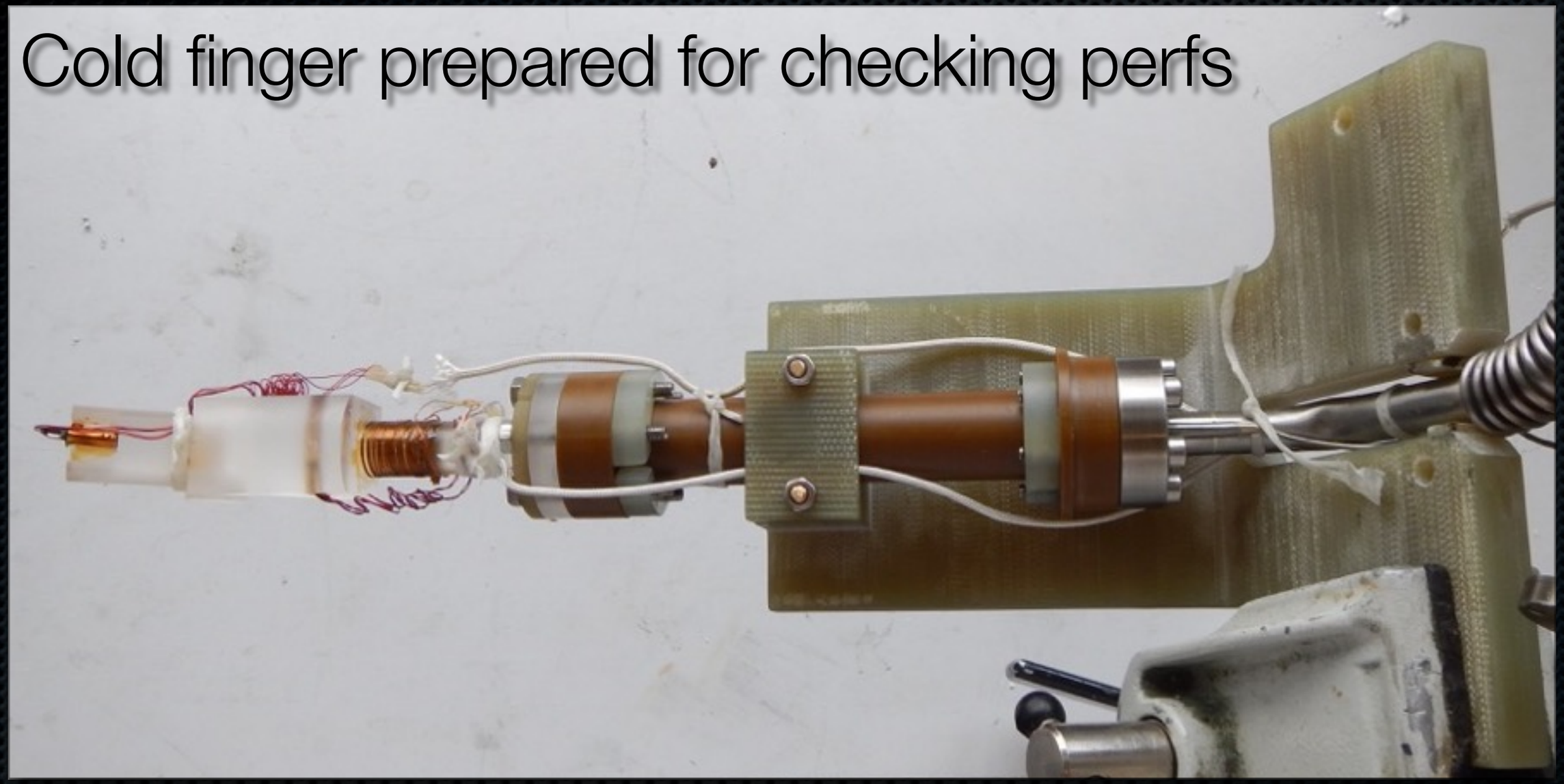


15 T / 30 mK

15 T / 30 mK

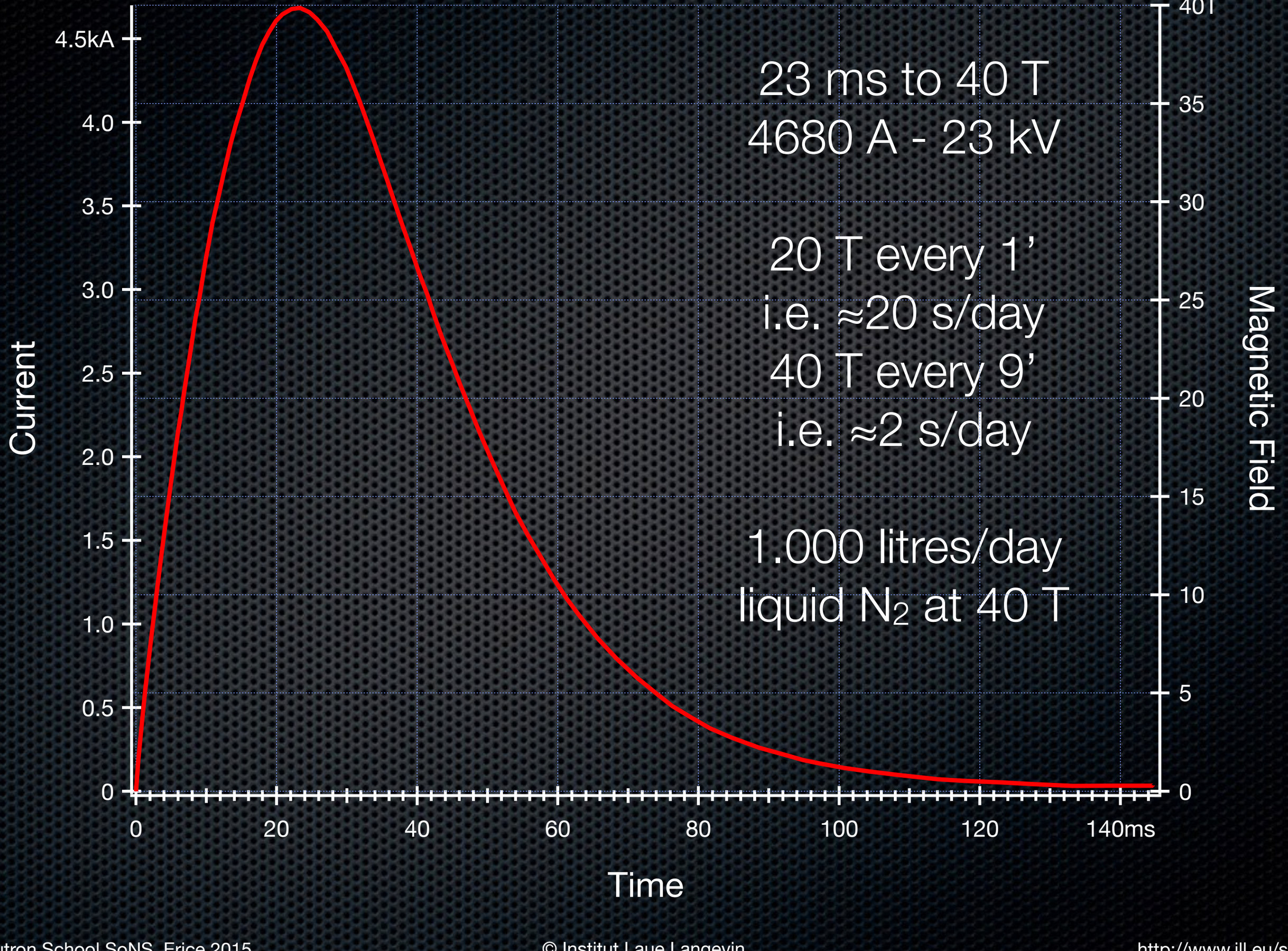


Cold finger prepared for checking perfs



2 K in 40 T pulsed field

Joule-Thomson expansion directly on the finger



Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

High pressure

- Which materials can we use in the beam ?
  - Aluminium 7049A-T6: transparent but strong signal into detector above ΔE ≈ 5 meV
  - Hardened CuBe alloy: less signal above ΔE ≈ 5 meV but activates quickly in high flux beams
  - TiZr alloy: no Bragg peak but incoherent scattering
  - Sapphire: transparent but fragile
- Pressure transmitter: He, Fluorinert FC-770…

High pressure

- **Clamped cells**
  up to 15 — 30 kbar
- **Gas pressure cells**
  up to 5 — 10 kbar
- **Liquid pressure cells**
  up to 7 — 10 kbar
- **Paris-Edinburgh presses**
  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

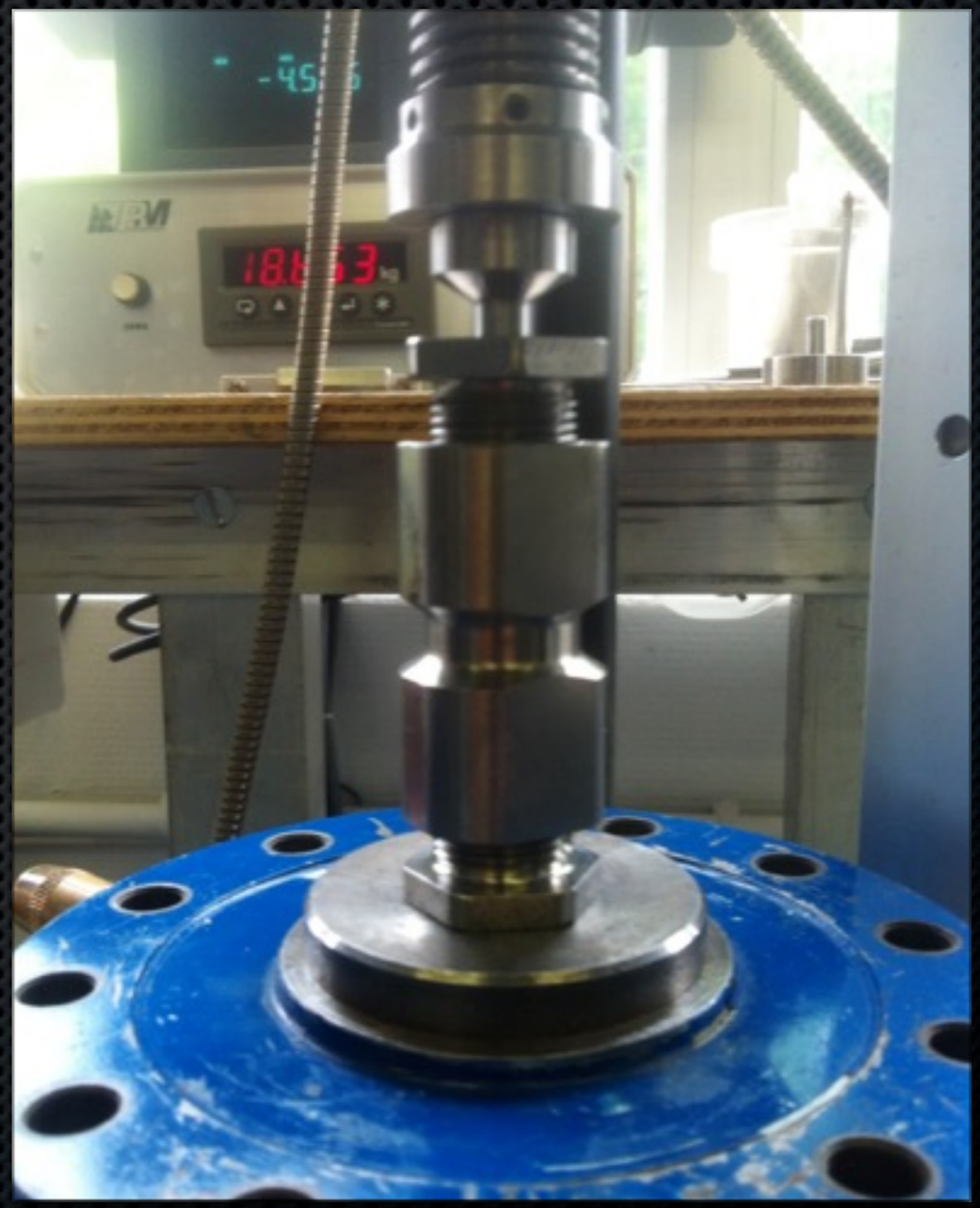
  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**
  up to 15 — 30 kbar
- **Gas pressure cells**
  up to 5 — 10 kbar
- **Liquid pressure cells**
  up to 7 — 10 kbar
- **Paris-Edinburgh presses**
  up to 200 kbar…



High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

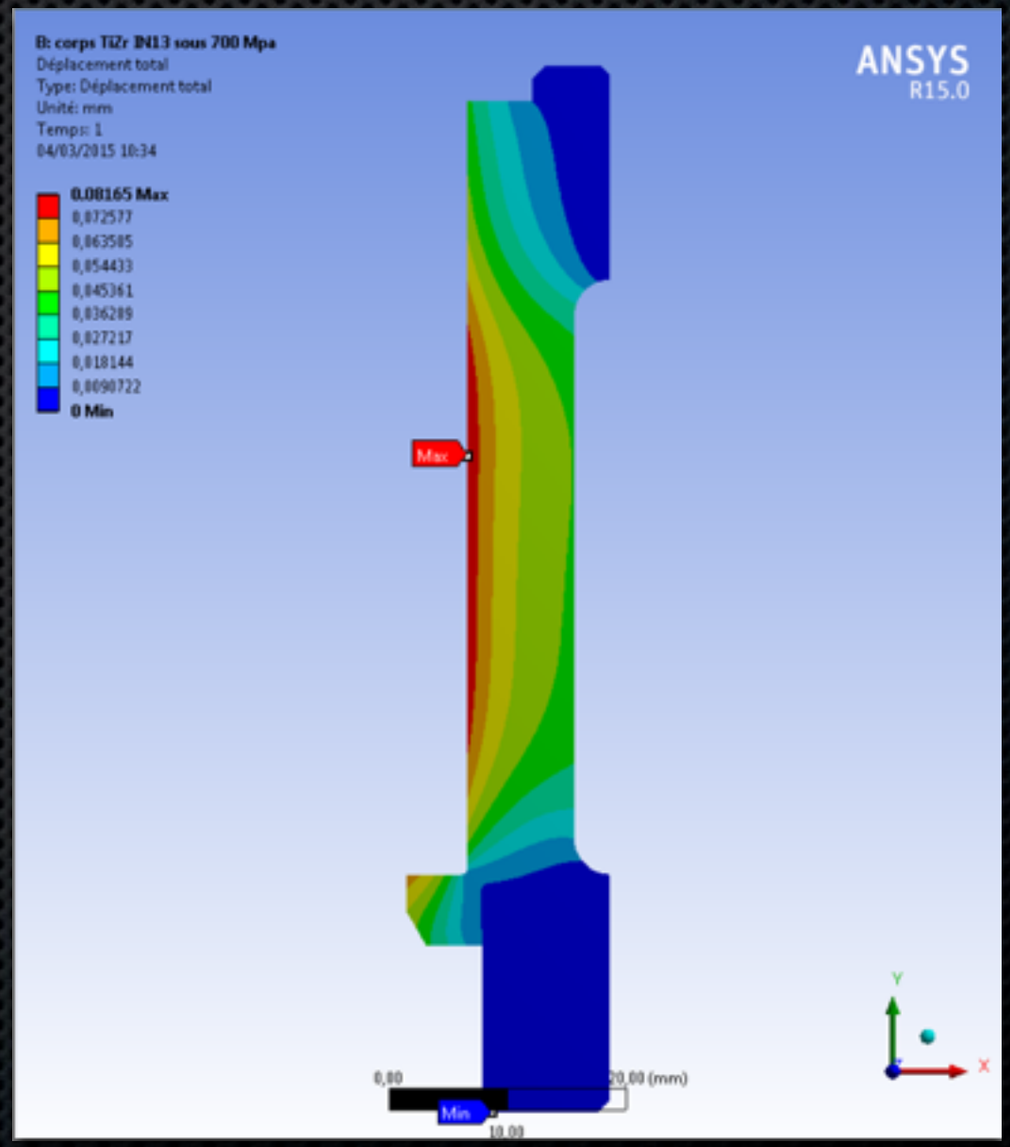
  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…



High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…

High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

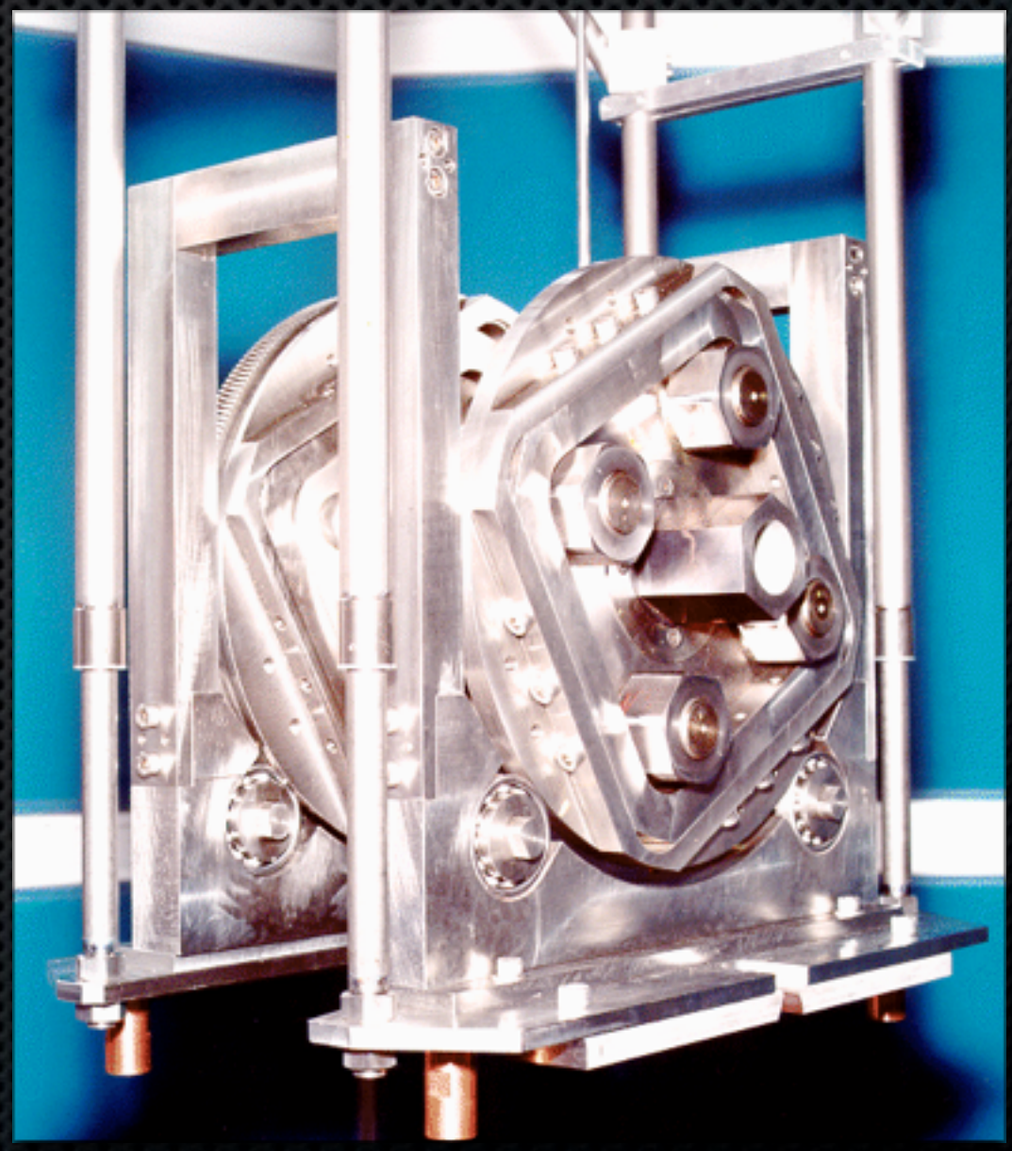
  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…



High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

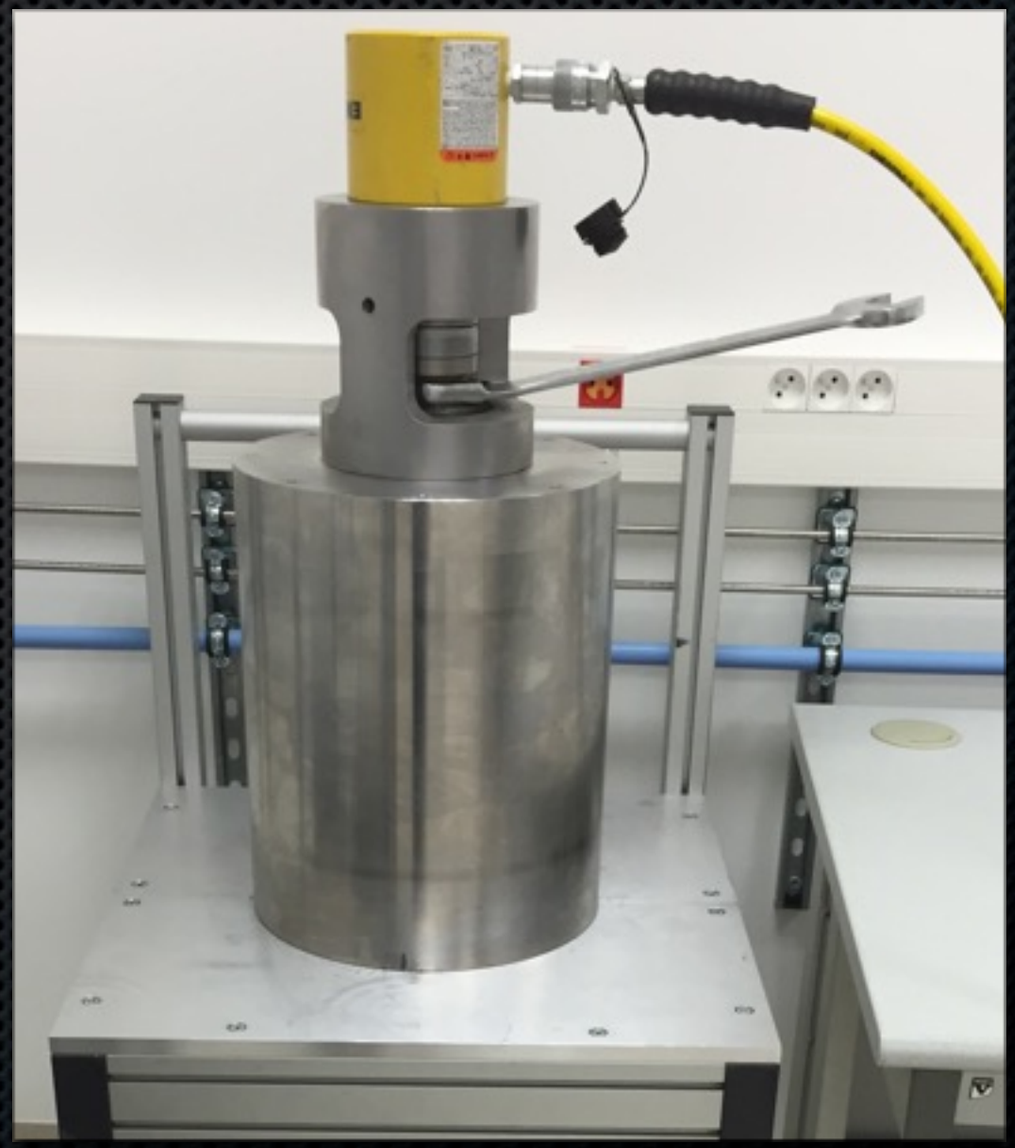
  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar…



High pressure

- **Clamped cells**

  up to 15 — 30 kbar

- **Gas pressure cells**

  up to 5 — 10 kbar

- **Liquid pressure cells**

  up to 7 — 10 kbar

- **Paris-Edinburgh presses**

  up to 200 kbar or more…

Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Content

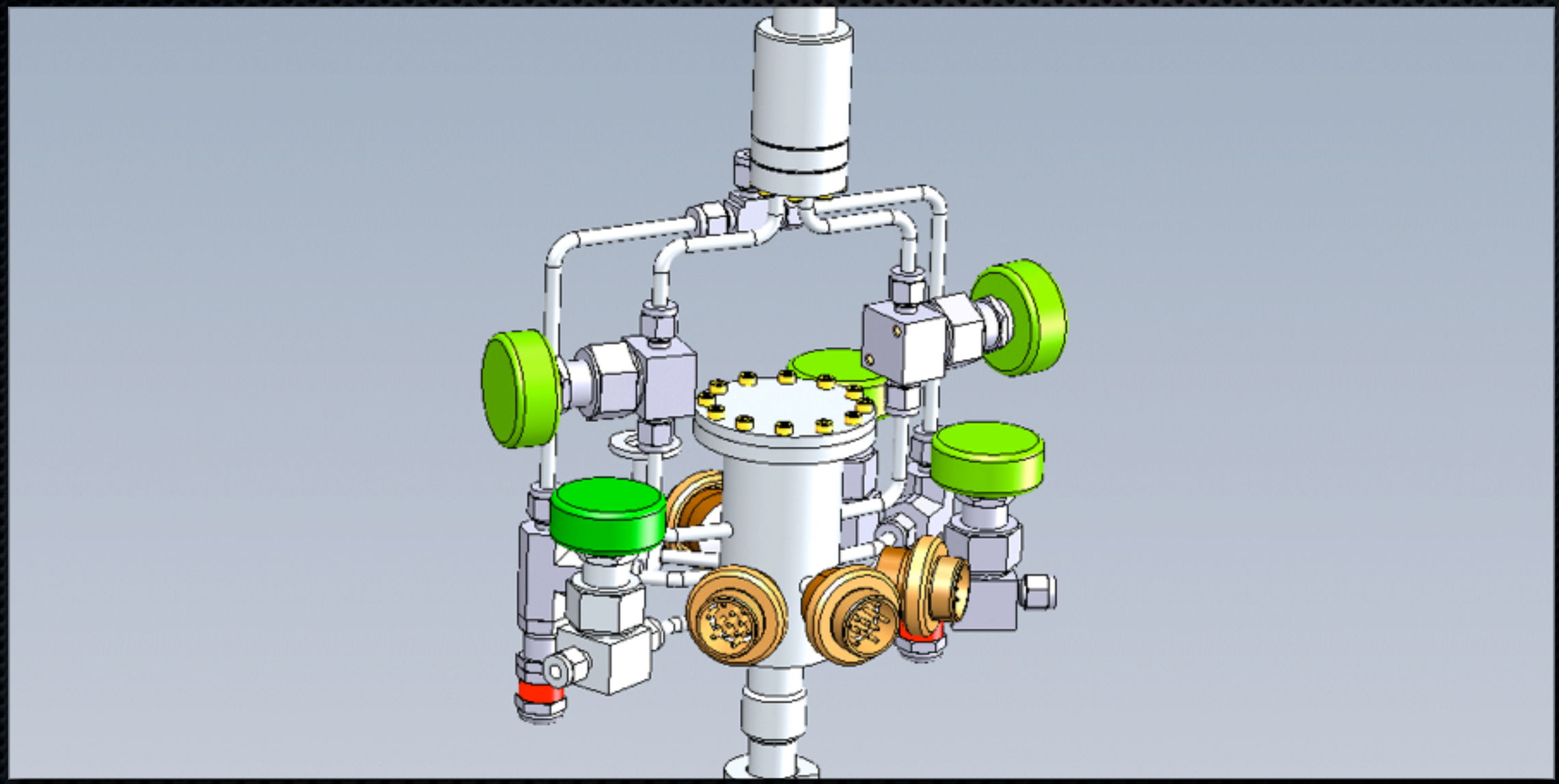
- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- **Gas sorption analysis with H_2, O_2, CO_2, etc.**
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Gas Sorption

Volumetric sorption analyser + inserts + …

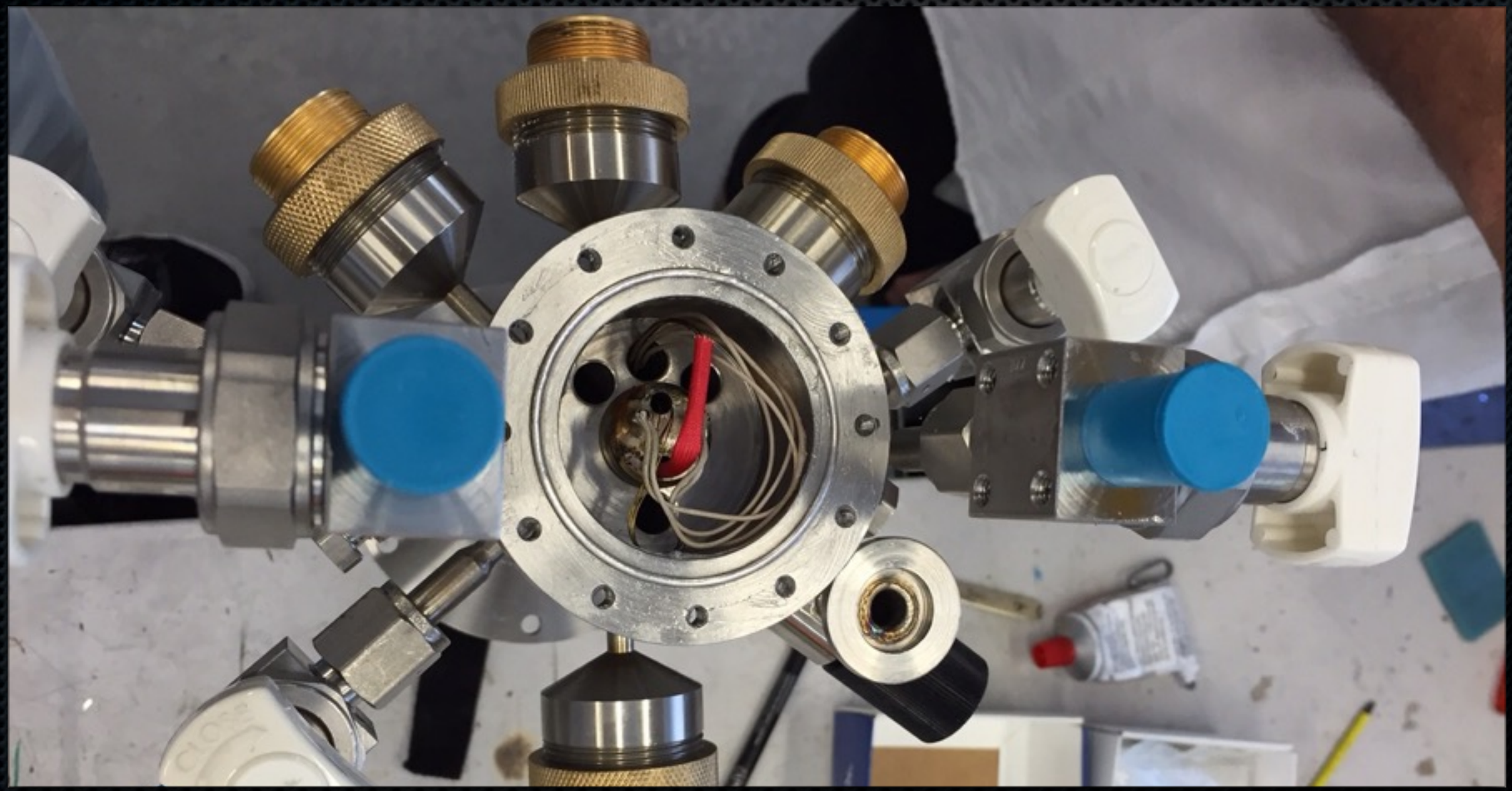
Gas Sorption

Volumetric sorption analyser + inserts + …

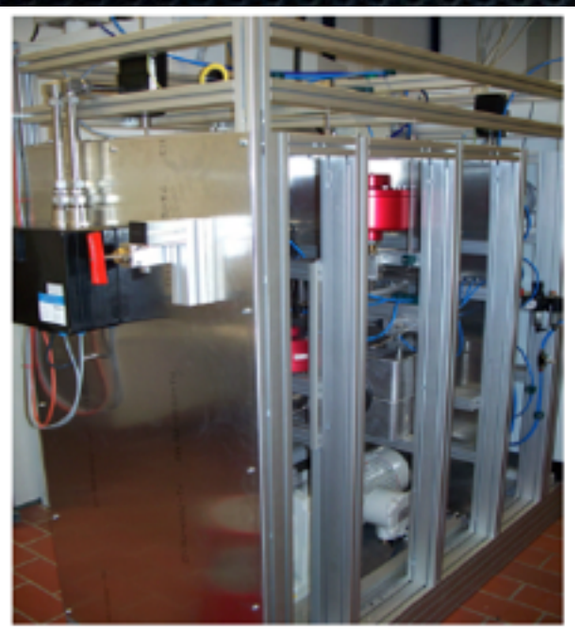


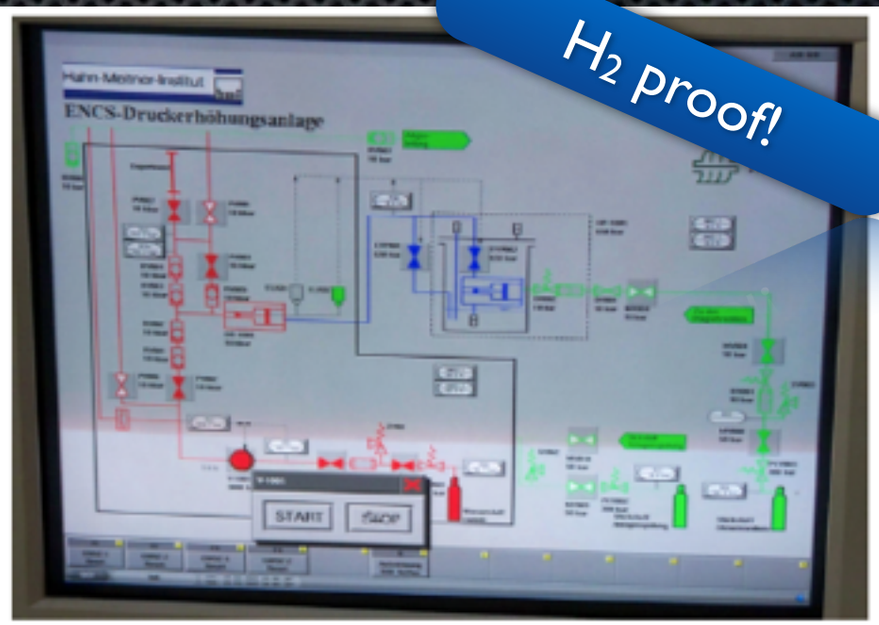
Gas Sorption

Volumetric sorption analyser + insert + …



Gas Sorption at 10 kbar





H_2 proof!

Content

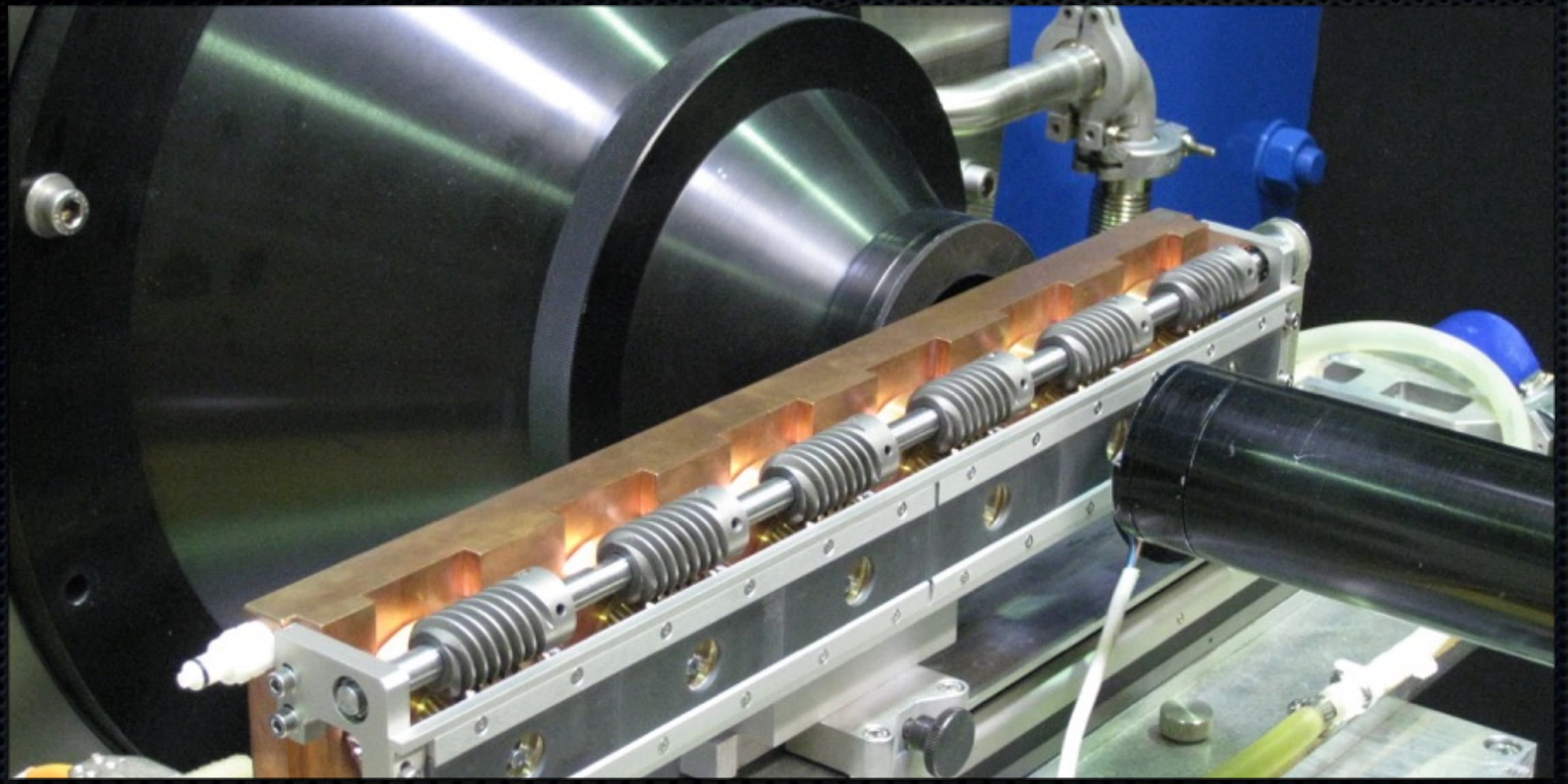
- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- **Gas sorption analysis with H_2, O_2, CO_2, etc.**
- Other devices for soft and bio materials
- Constraints to consider before building an instrument

Content

- Low temperature cryostats
- High temperature furnaces
- Electric and magnetic fields
- High pressure cells and presses
- Gas sorption analysis with H_2, O_2, CO_2, etc.
- **Other devices for soft and bio materials**
- Constraints to consider before building an instrument

for Soft & Bio Materials…

- Humidity chambers for SANS, ToF, Reflectometry,
- Stopped-flow observation heads,
- High-pressure cells for SANS, NSE,
- Electric field cells for SANS, ToF,
- Light-scattering setups for SANS (static & dynamic),
- Acoustic levitation, Rheometers,
- Adiabatic calorimetry, etc…

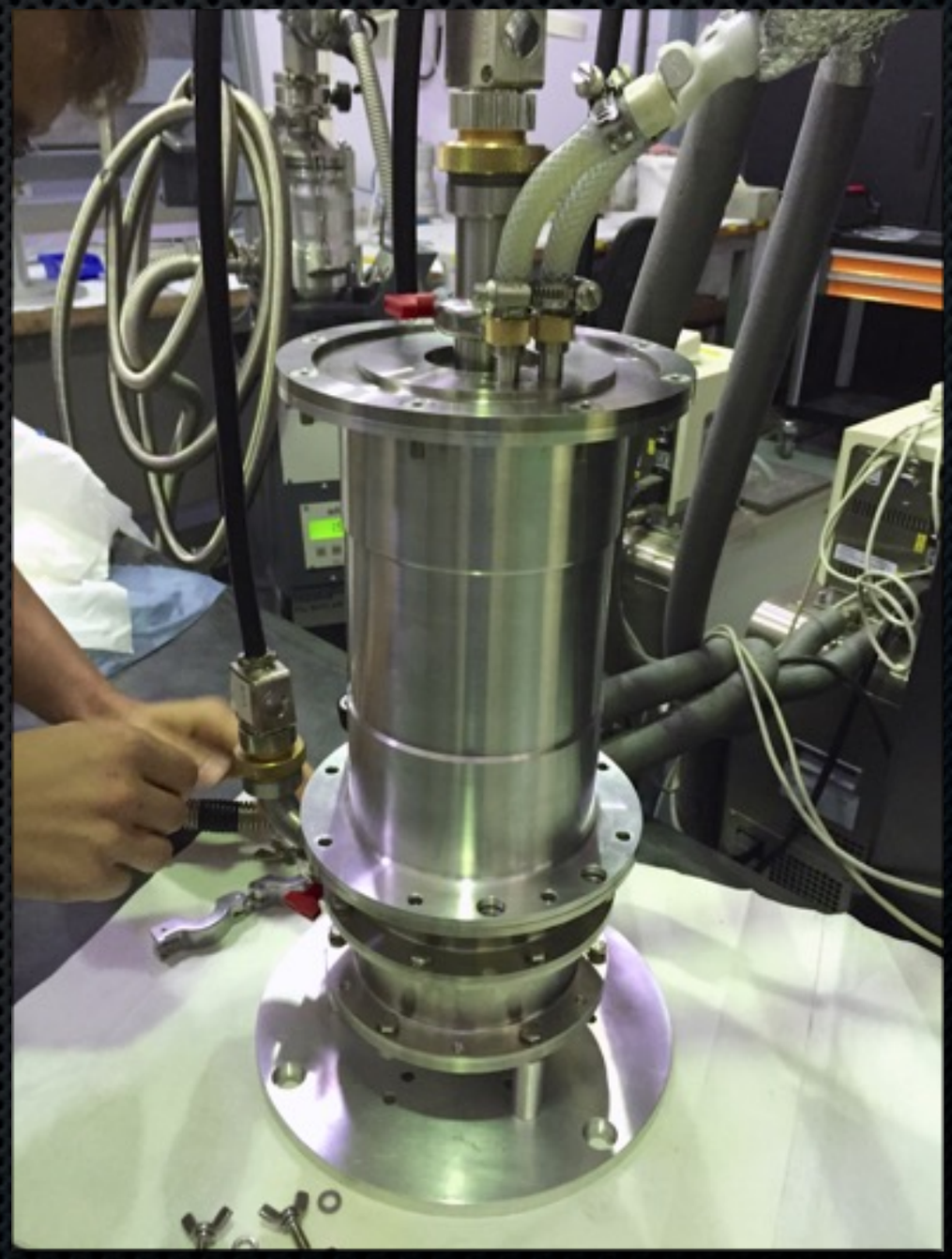


Soft & Bio Mat.

SANS sample changers

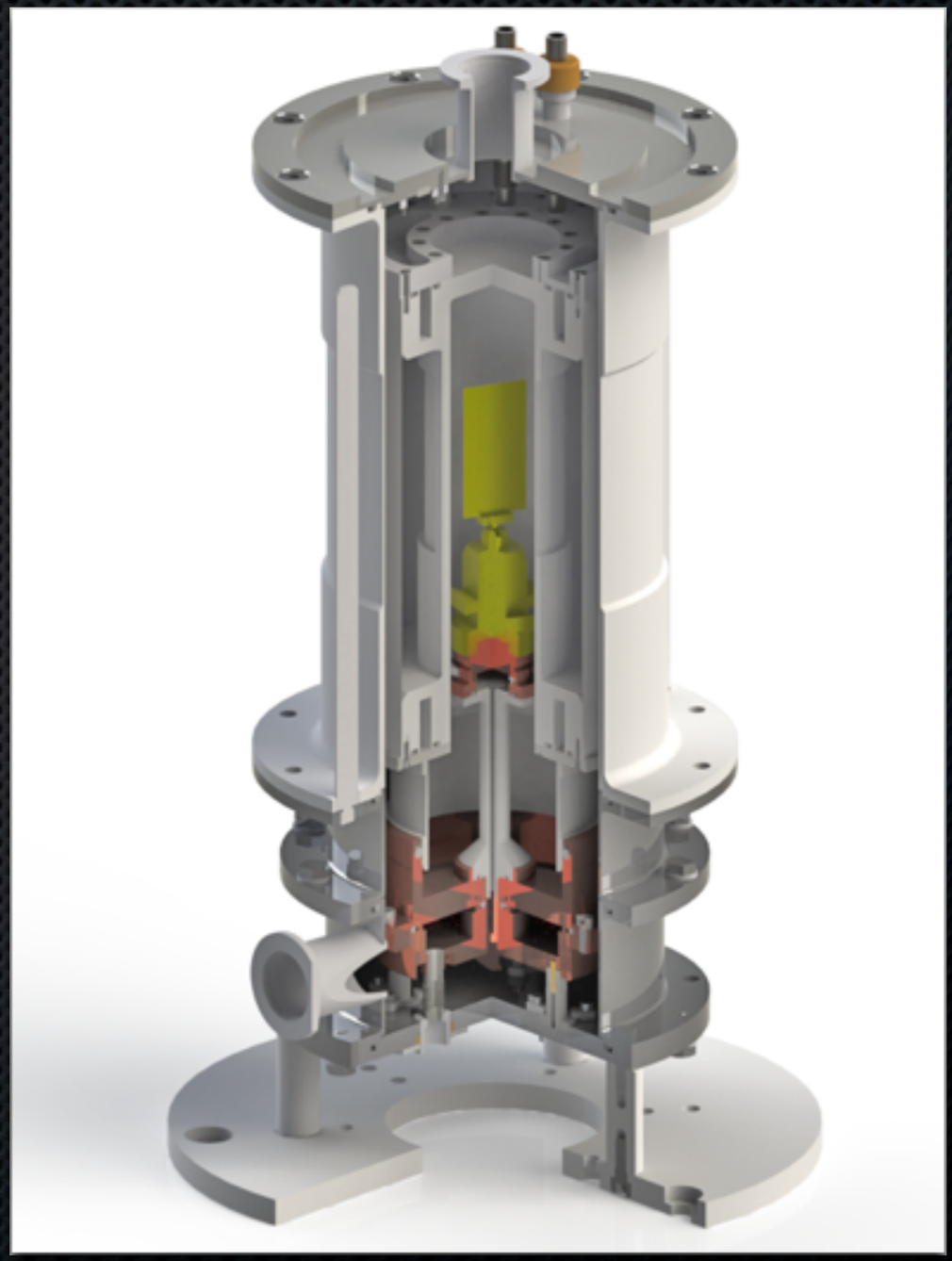
Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume



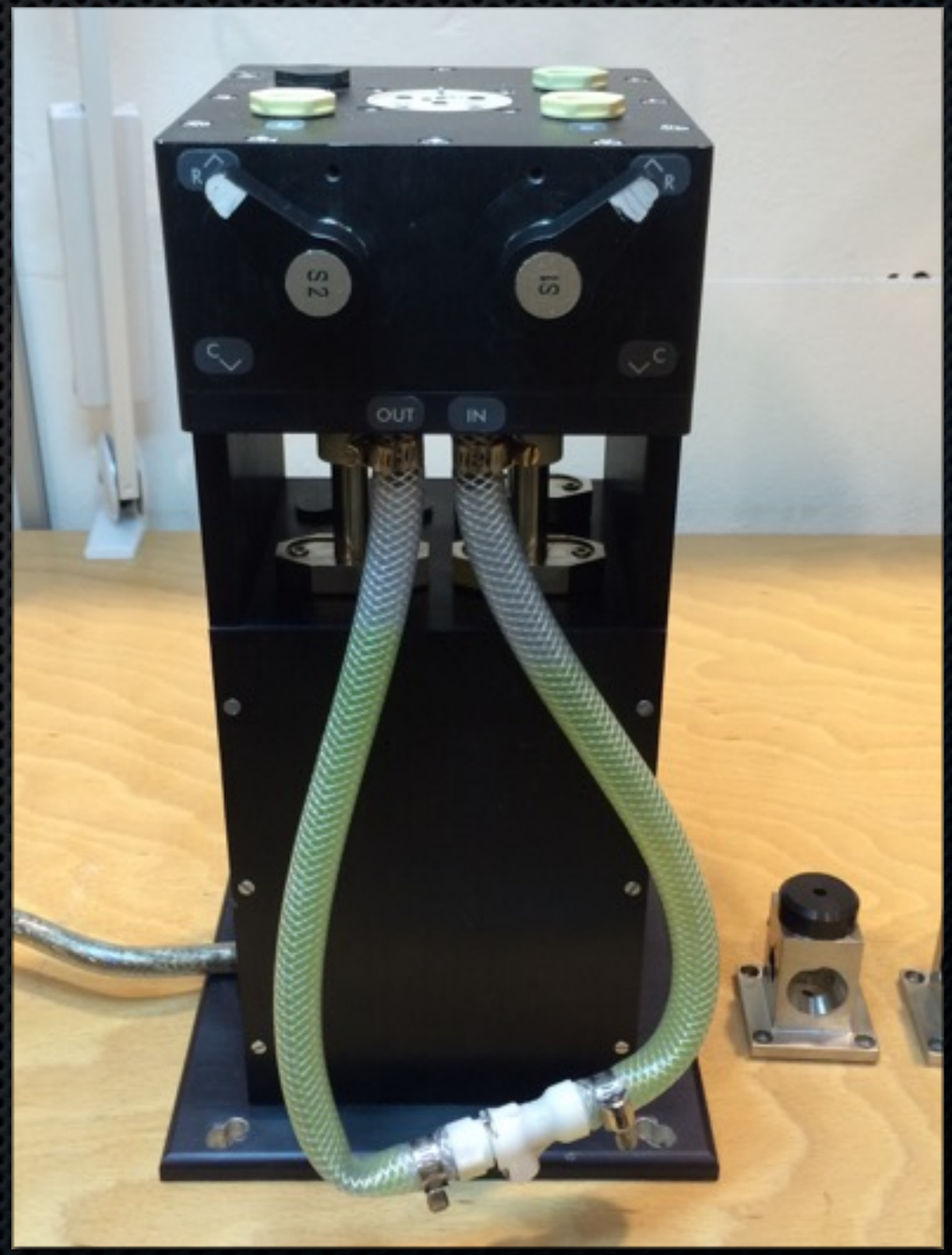
Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume



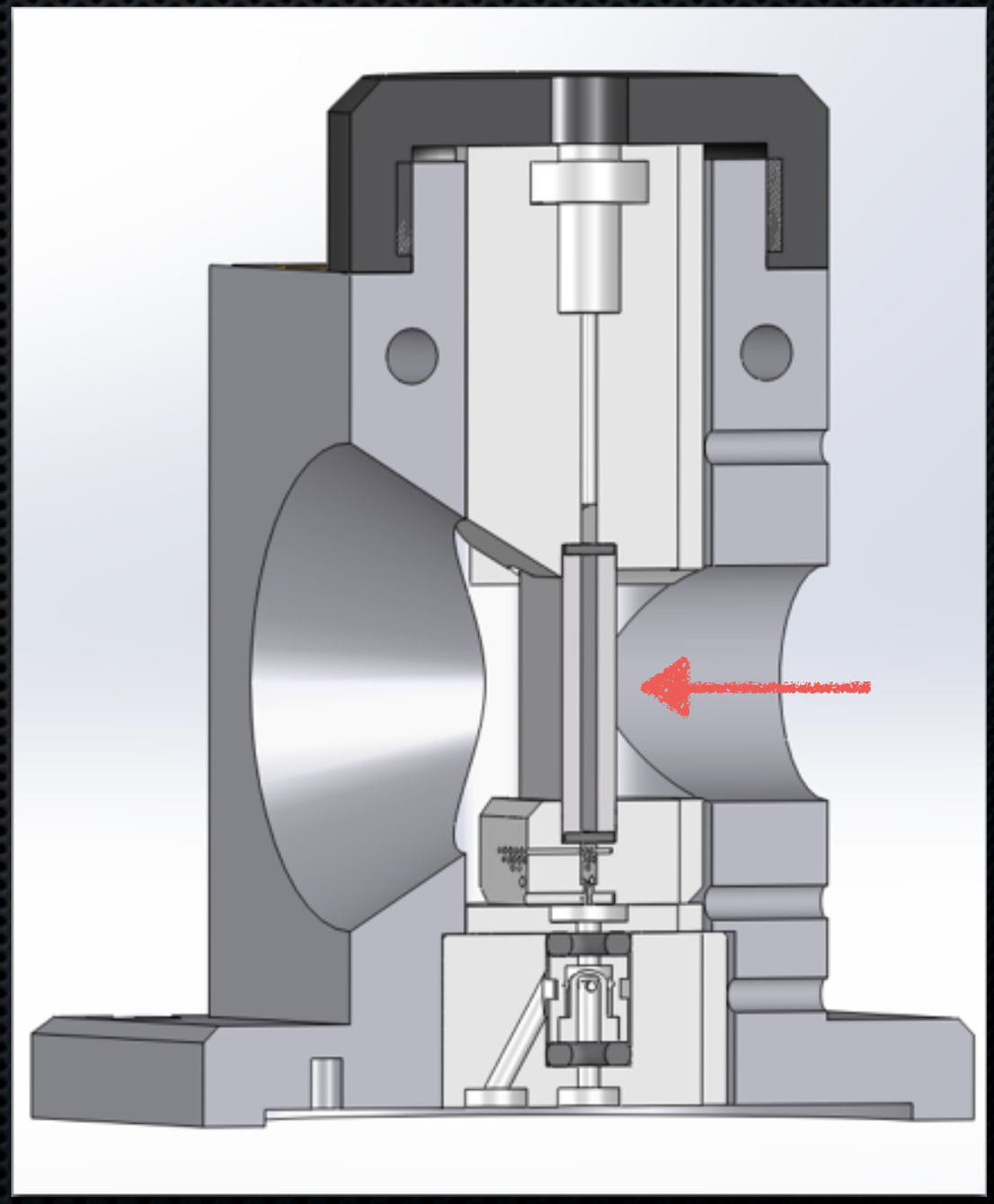
Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume



Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume



Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume

Soft & Bio Mat.

- **Humidity chambers**
  up to 99.8 %RH
- **Stopped-flow systems**
  reduce wasted sample…
- **Liquid-liquid interface cells**
  flat meniscus, neutron path
- **Optimum chillers**
  optimum power and volume

Soft & Bio Mat.

- **Humidity chambers**

  up to 99.8 %RH?

- **Stopped-flow systems**

  reduce wasted sample?

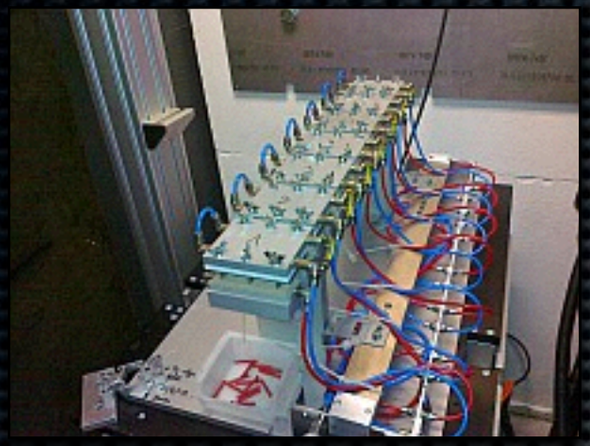
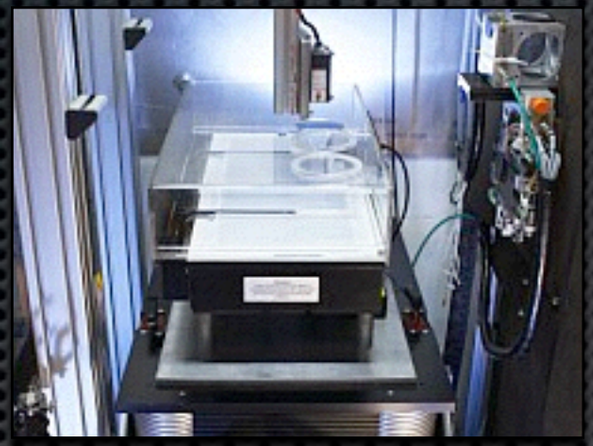
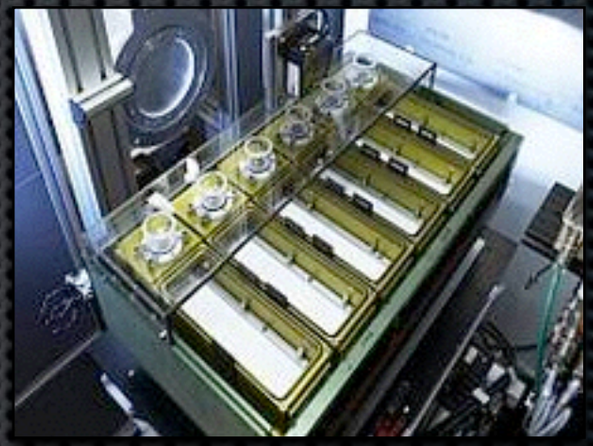
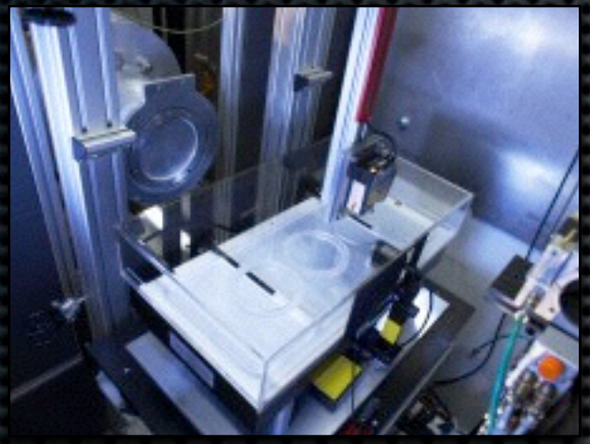
- **Liquid-liquid interface cells**

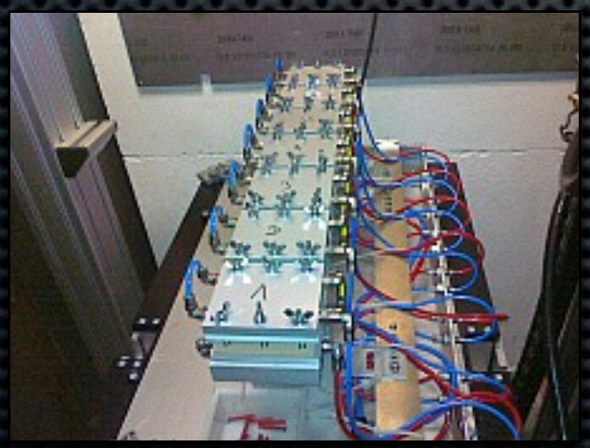
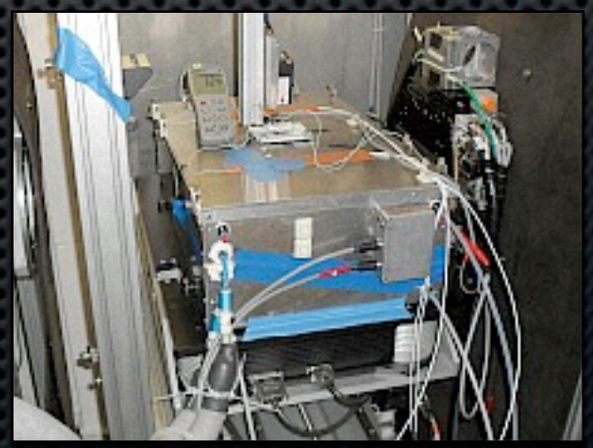
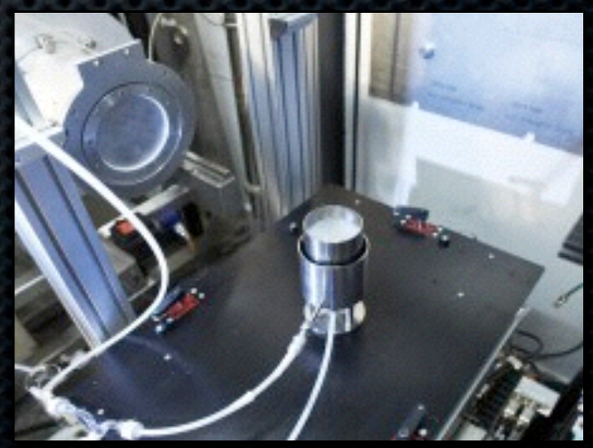
  flat meniscus, neutron path

- **Water baths**

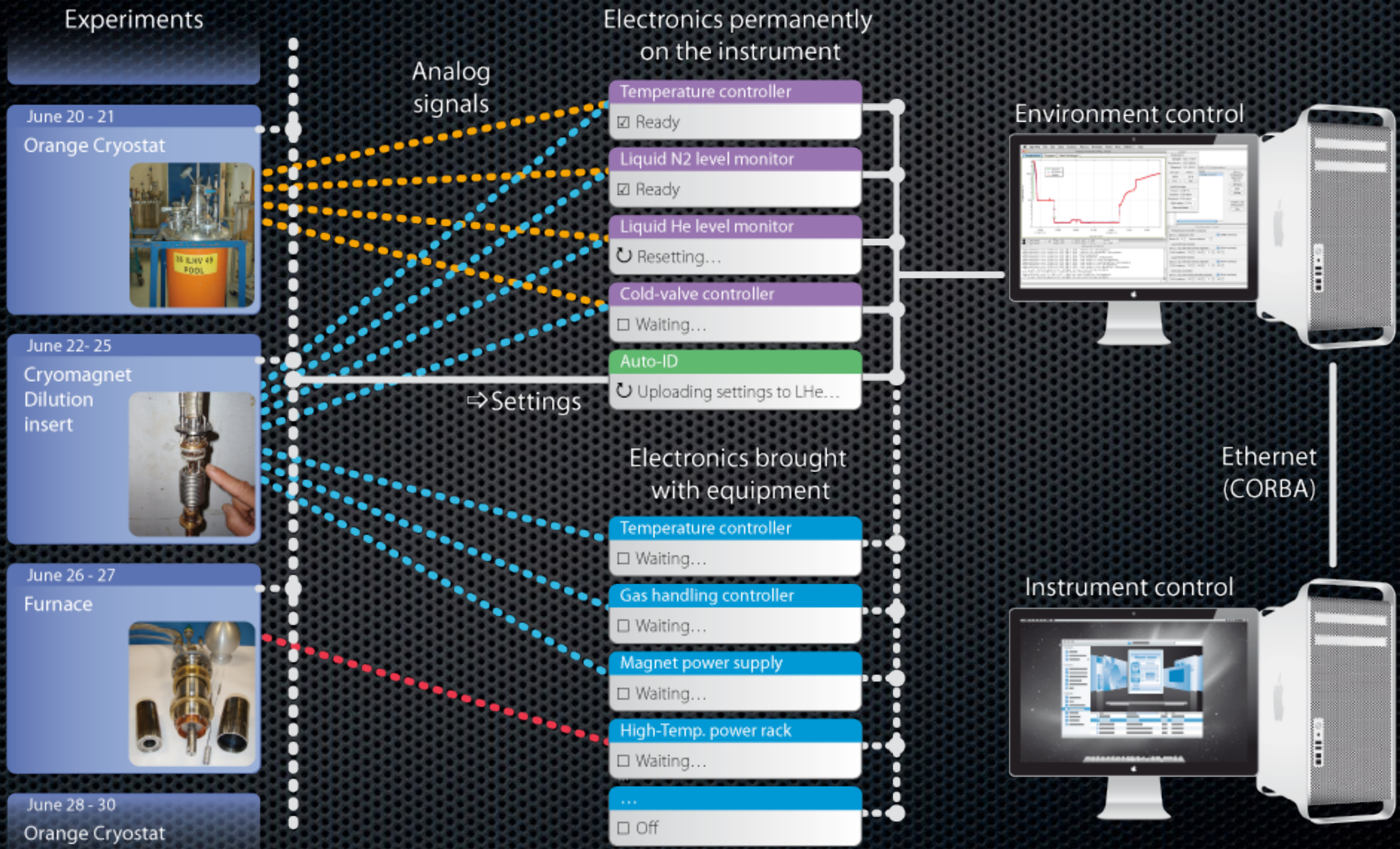
  optimum power and volume

And many more…

Organisation…



To summarise

- Non magnetic material in > Ø3 m around sample: use Al alloys, Stainless steels 304L or 316L, brass, etc.
- > Ø800 mm sample volume (inside ToF chamber!)
- > 1000 kg allowable weight on sample table
- > 400 mm height between sample table and beam
- > 3 m access above beam (magnets, dilution fridges)
- > 4 m^2 near sample area for installing shared equipment + space for equipment of the instrument

To summarise

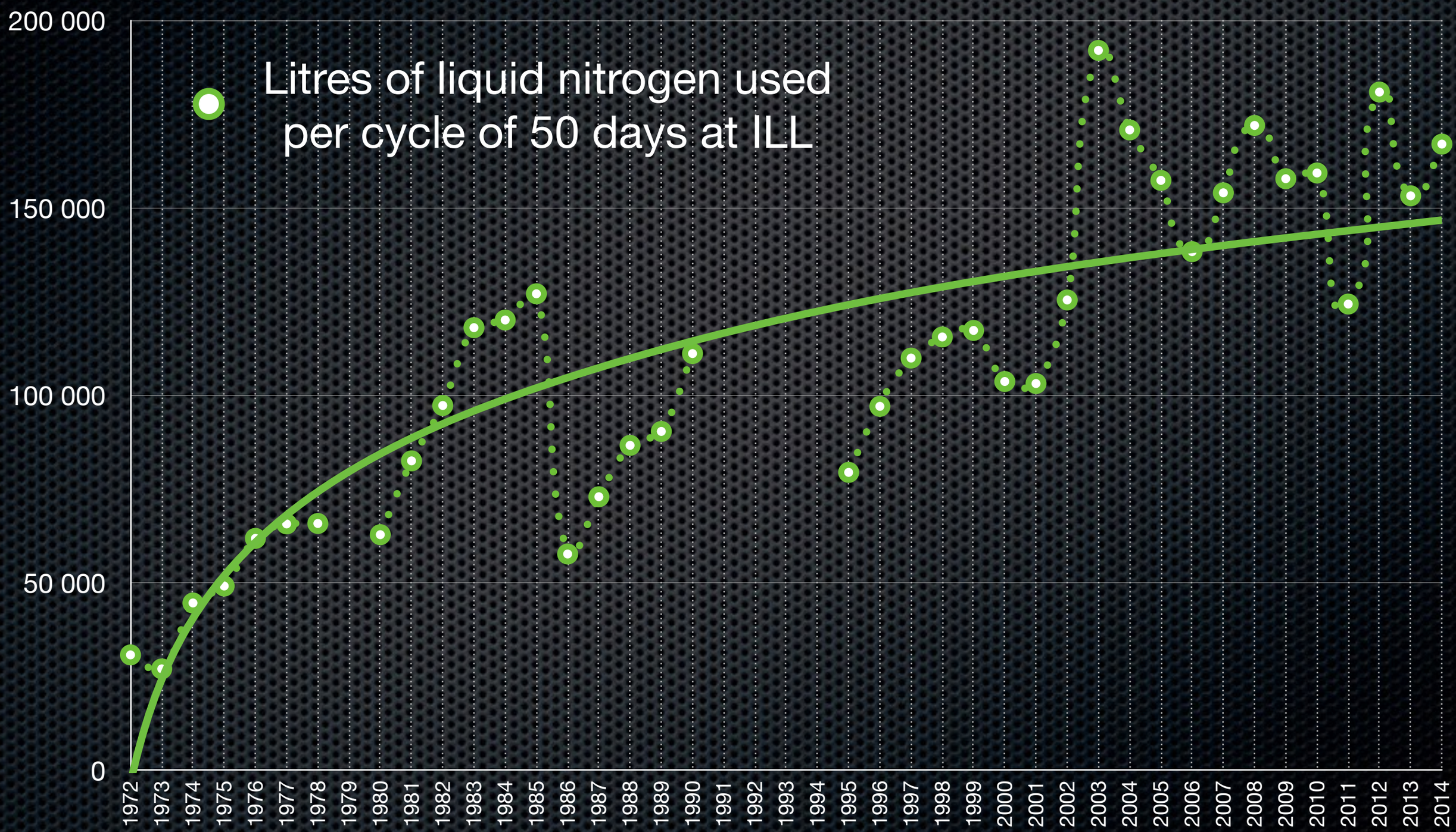
- > 10 kW.h water cooling circuit on each instrument
- > 10 kVA on instrument only for sample environment
- > 1.000 kg slow crane with access > Ø4 m

- Background issues can be avoided during the design phase when considering environment from start.
- Services above the sample area: He recovery line, compressed air, power line, liquid nitrogen, Ethernet…
- Standard plugs, electronics, communication protocol…

To summarise

- Train technicians and scientists !
- Provide enough liquid N2 and Ar…

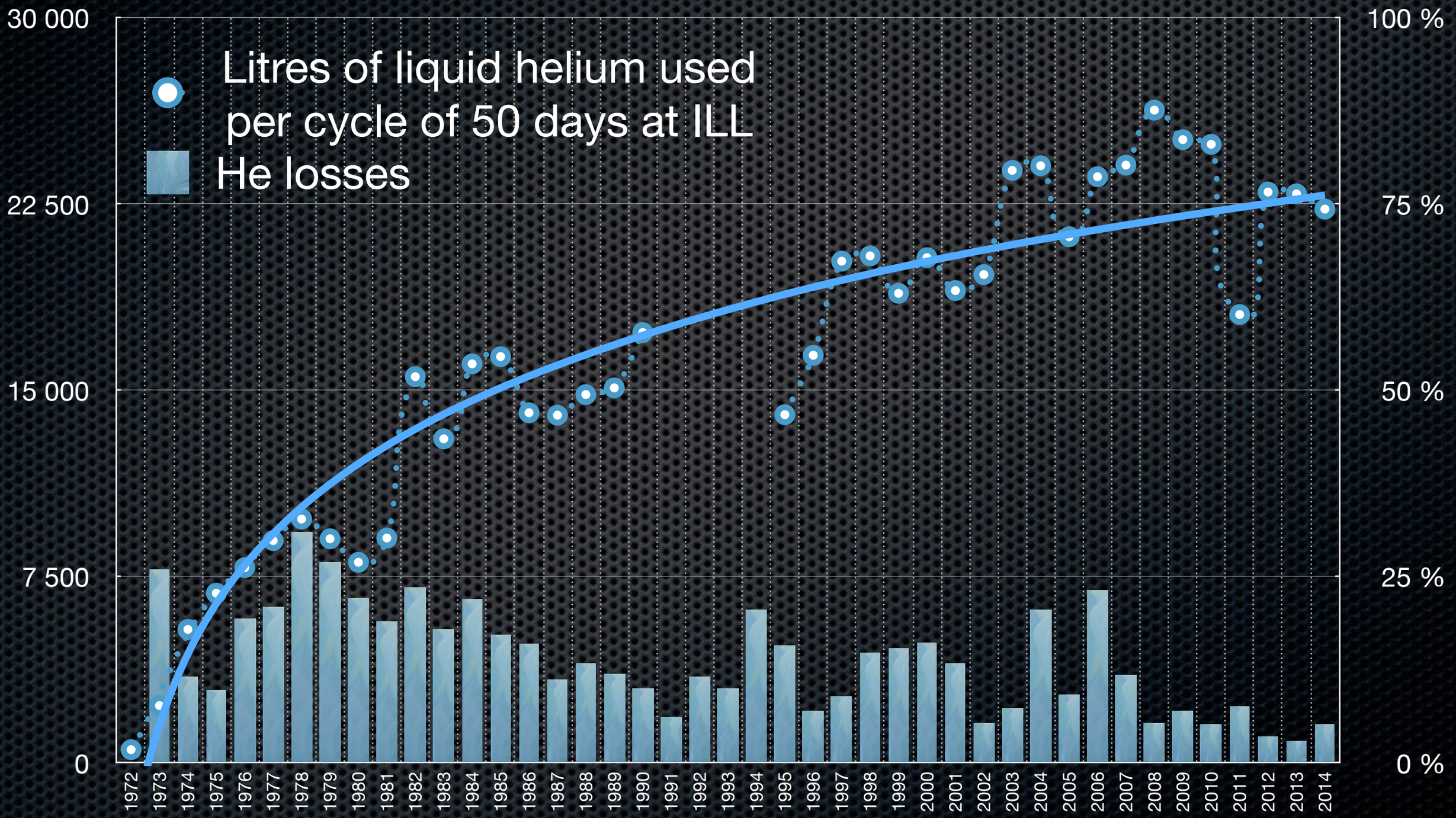
To summarise



To summarise

- Train technicians and scientists !
- Provide enough liquid N2 and Ar…
- Install a He recovery line to save He and money…

To summarise



To summarise

- Train technicians and scientists !
- Provide enough liquid N2 and Ar…
- Install a He recovery line to save He and money…
- And never assume that you know which type of environment will be used. The sample environments suite will be science driven, not the reverse !

Many thanks
for your attention