

SINE2020 General Assembly

Bilbao, 28-29 May 2019

WP 7

Sample Environment

Eddy Lelièvre-Berna - ILL

— CSEC, ESS, HZB, LLB, ICMA, ILL, IMPMC, ISIS, MLZ, NPI, PSI —

Sample Env. WP objectives

neutrons & muons

- Establish standards to reduce development costs, strengthen relations with industry (7.1)
- Increase efficiency by reducing beam time losses and improving equipment (7.2),
- Open new fields of science by developing new equipment or extending physical parameter ranges (7.3 & 7.4)

Work carried out — Task 7.1

- **Sample Env. Communication Protocol: SECoP**
 - 15 meetings + discussions with ISSE working group
 - Libraries coded at HZB to facilitate SECoP adoption
 - Version 1.0β available on GitHub
 - Deployment started at ESS, MLZ, PSI, ILL, ISIS
 - First experiment with MLZ furnace at ILL successful
 - Discussed with industry: attocube, Hall Scientific, HTS-110, Kurt J. Lesker, OINS, etc.



Search or jump to...

Pull requests Issues Marketplace Explore



SampleEnvironment / SECoP

Unwatch 1 Star 2 Fork 2

Code Issues 0 Pull requests 0 Projects 0 Wiki Security Insights Settings

SECoP — Sample Environment Communication Protocol <https://sampleenvironment.org>

Manage topics

268 commits 1 branch 0 releases 4 contributors

Branch: master New pull request Create new file

EnricoFaulhaber add minutes of vidconf 2019-05-08

protocol add minutes of vidconf 2019-05-08

.gitignore consistent scaling of railroad diagrams

LICENSE Initial commit

README.rst release v2018-11-07 (v1.0\beta)

README.rst

SECoP

Sample Environment Communication Protocol

- [SECoP Motivation](#)
- [SECoP Specification](#)
- [SECoP Issues](#)

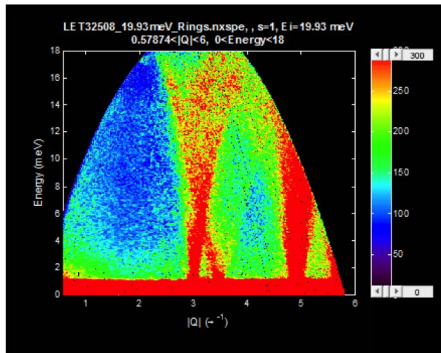


Work carried out — Task 7.2

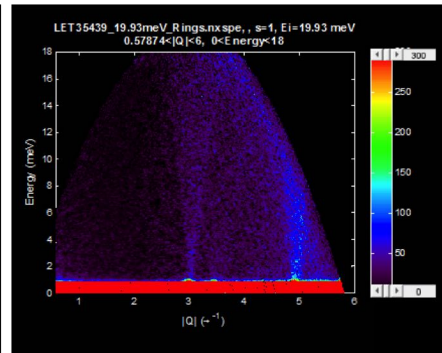
- Towards efficient Sample Env.: Simulations
 - ICMA has coded a Union component of McStas taking into account diffuse, elastic/inelastic and coherent/incoh. scattering, microstructures, etc.
 - Al7049A, Al7075, CuBe2, MP35N, NiCrAl, TAV6, TiZr data collected at ISIS on several instruments
 - Code benchmarked successfully with ISIS data
 - Now allows the simulation of any environment

Work carried out — Task 7.2

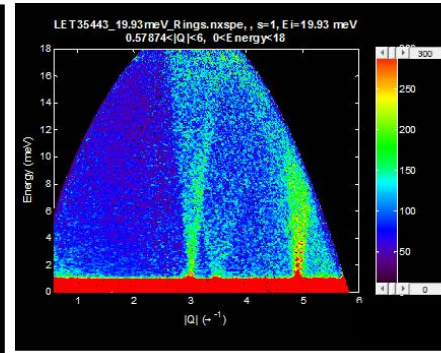
CuBe2



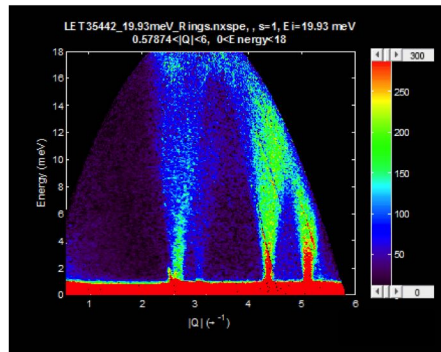
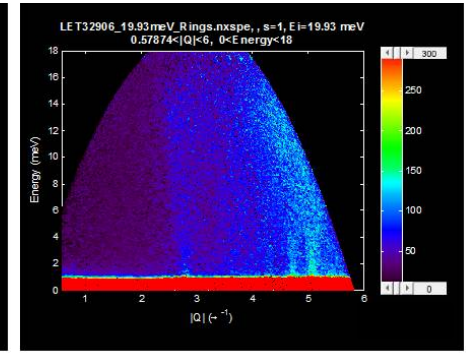
MP35N



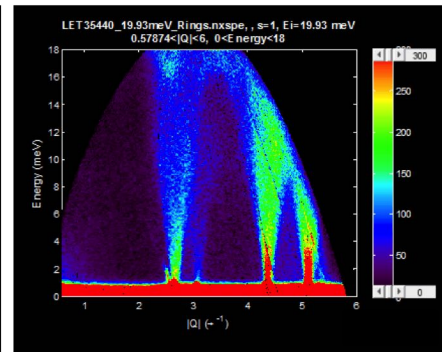
NiCrAl



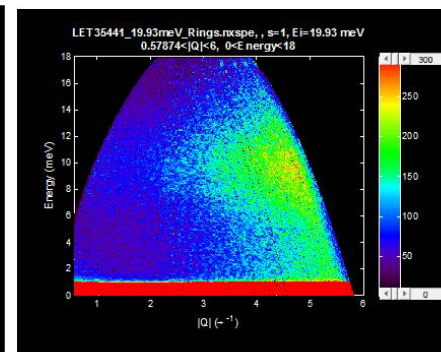
TAV6



Al 7049A



AL 7075

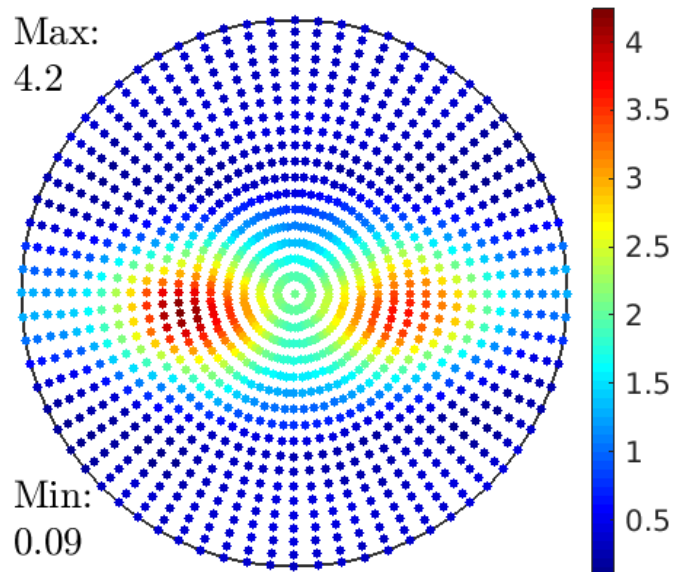


TiZr

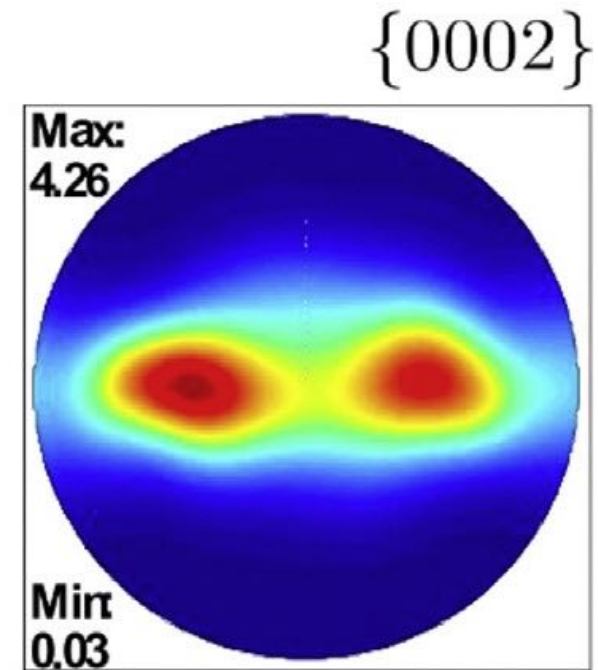


Work carried out — Task 7.2

Pole figure of the (0002) crystal planes of a Zircaloy-4 plate benchmarked against Texture.comp developed by ICMA



McStas Simulation
V. Laliena *et al.* (ICMA)



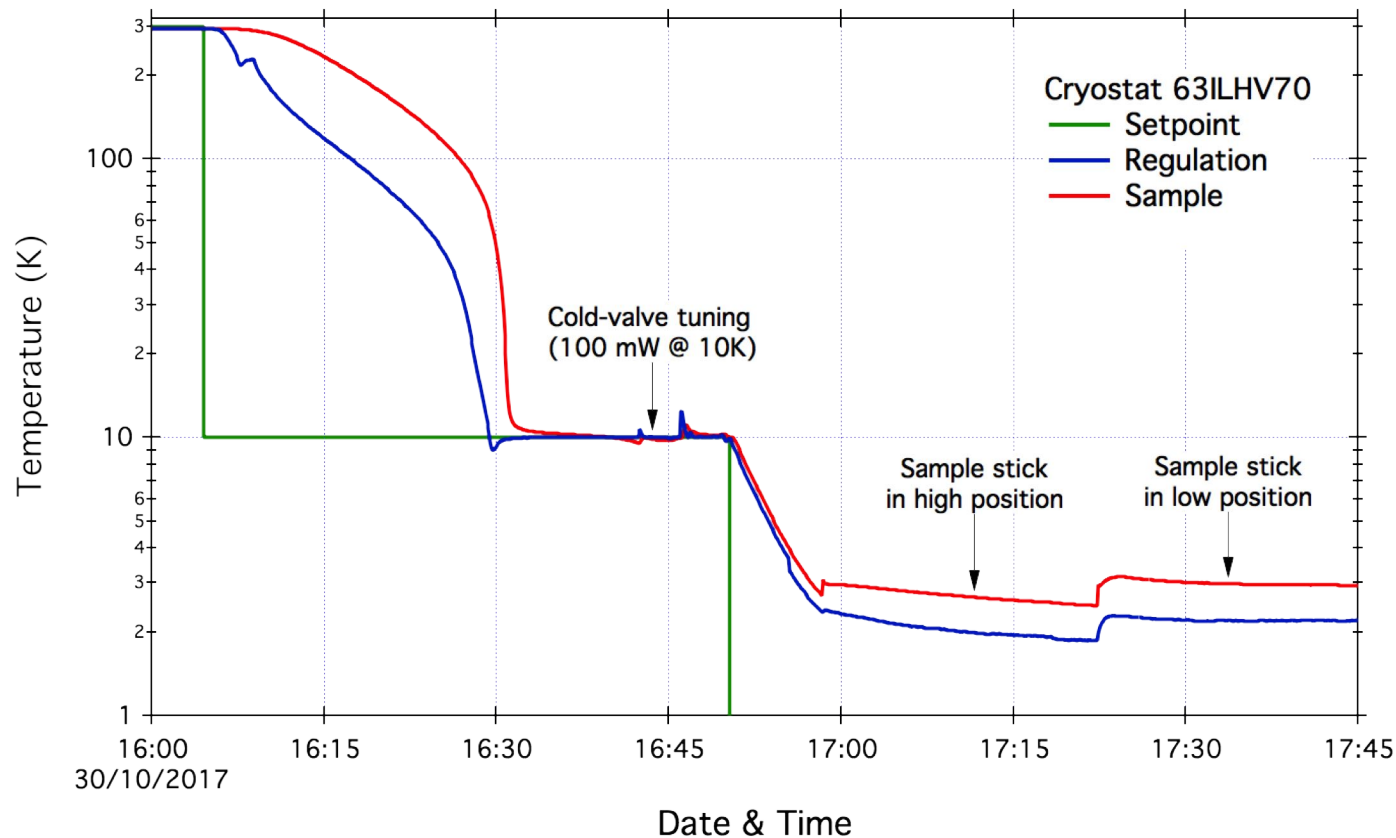
F. Malamud *et al.*
J. Nuclear Materials **510** (2018) 524

Work carried out — Task 7.2

- Towards efficient Sample Env.: Cryostats
 - Cooling/heating
 - 3x faster wet cryostats at ILL
 - Technical transfer to **AS Scientific Ltd** (UK)
 - Cryostats are getting faster at ISIS
 - Signal/background ratio
 - 30% less background produced by vanadium cryofurnaces
 - 5x less background at low-Q with TOF cryostat

Work carried out — Task 7.2

Measurement of the cool-down time, the base temperature and the gradient of temperature in different/thinner calorimeters at ILL

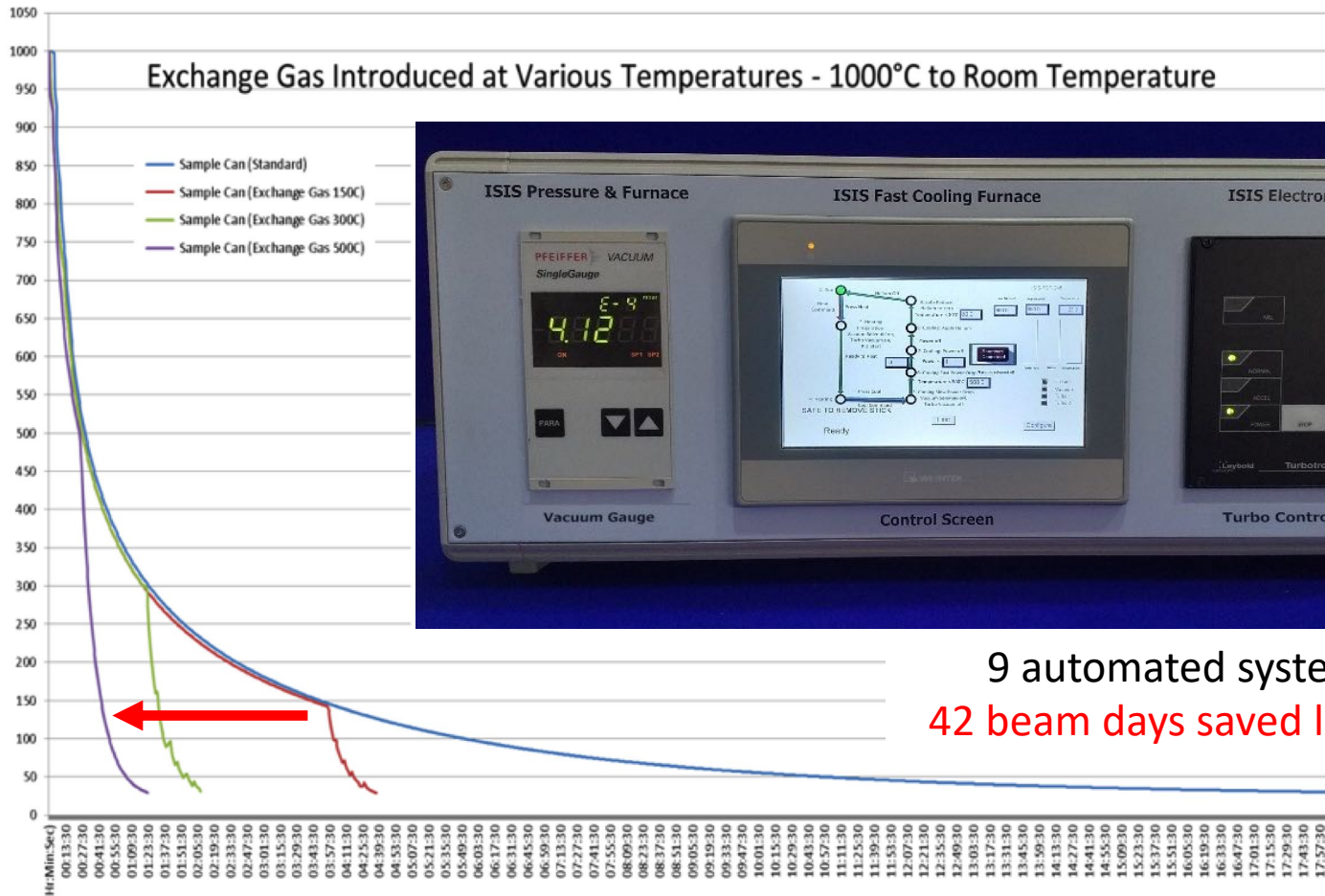


Work carried out — Task 7.2

- Towards efficient Sample Env.: Furnaces
 - Furnace type selected from survey
 - Technique developed by ISIS team
 - Results: 1000 to 150°C in less than 45'
 - 6x faster cool-downs at ISIS with 2 l/min He flow
 - 4x faster cool-downs at ILL with 7 cc He injection
 - Full automation being finalised at ISIS and ILL
 - Technical transfer to **AS Scientific** envisaged (UK)

Work carried out — Task 7.2

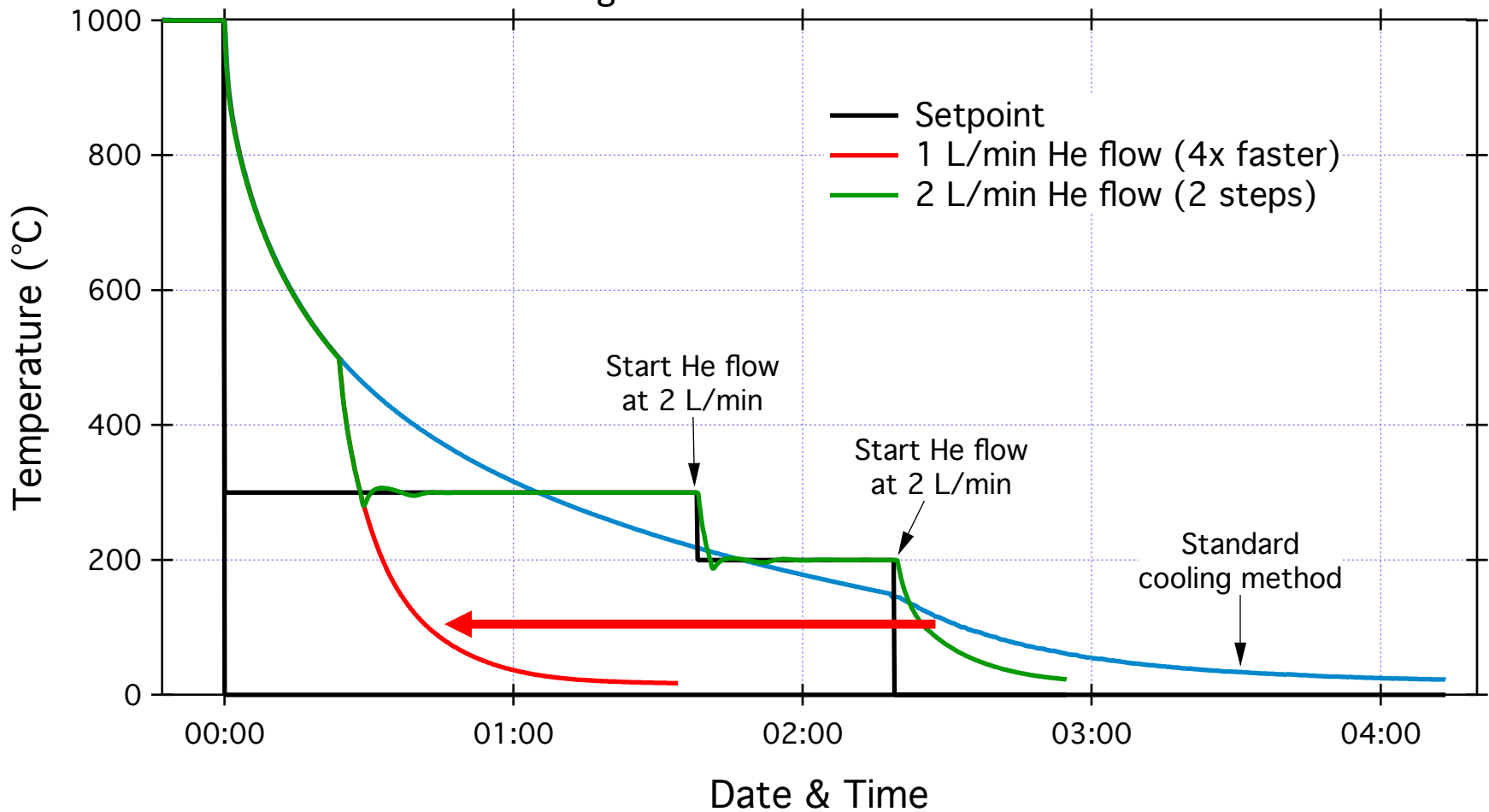
Fast cooling tests in resistive furnaces at ISIS



9 automated systems soon
42 beam days saved last year!

Work carried out — Task 7.2

Fast cooling tests in resistive furnaces at ILL

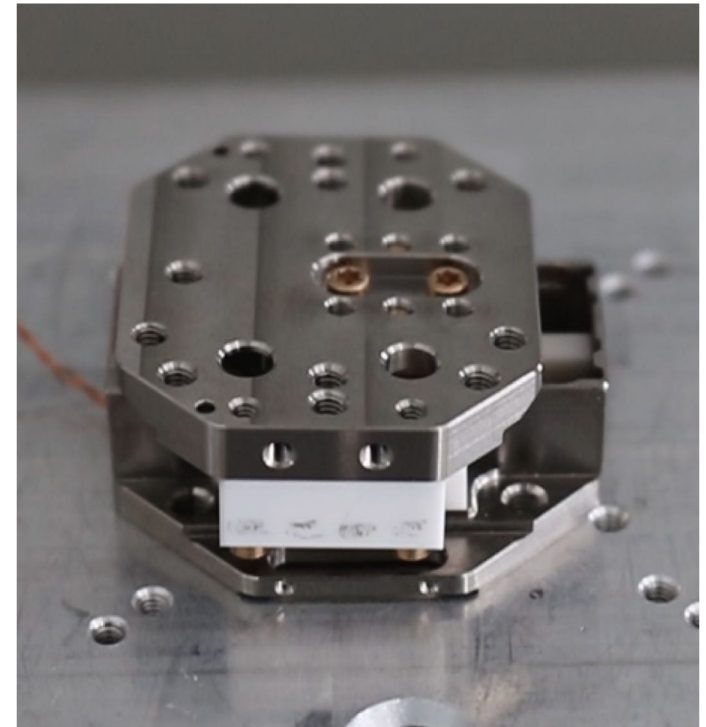
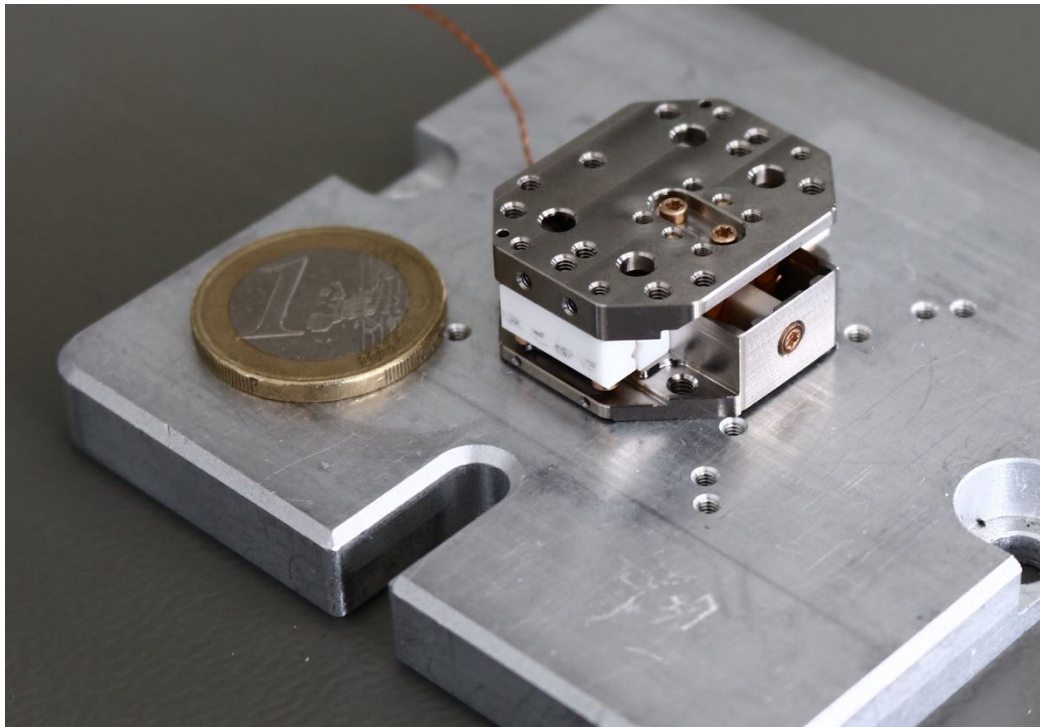


Work carried out — Task 7.2

- Towards efficient Sample Env.: Goniometers
 - Ultra-low temperature goniometer specifications defined from survey => Ø36 outer diameter!
 - Concept design agreed (amongst 3 proposed by PSI)
 - Resistive and/or capacitive encoders
 - Prototype will soon be tested with dilution inserts at PSI and ILL
 - **attocube** will distribute the final version

Work carried out — Task 7.2

Ultra-low temperature goniometer designed by attocube, PSI & ILL



Work carried out — Task 7.3

- Next-generation high-P cells for NS & μ SR
 - Kick-off meeting gathering μ SR, NS and HP experts
 - CSEC high-pressure expertise transferred to PSI
 - Multi-layer design adopted for μ SR clamp cell
 - 1.5 gain factor established with 2.6 GPa μ SR cell
 - Same concept adopted for neutron clamp cell
 - Material survey performed at ISIS (task 7.1)
 - In-situ pressure measurement tested at ILL
 - Multi-layer clamp cell to be tested at ISIS soon

C: Static Structural

Equivalent Stress 2

Type: Equivalent (von-Mises) Stress

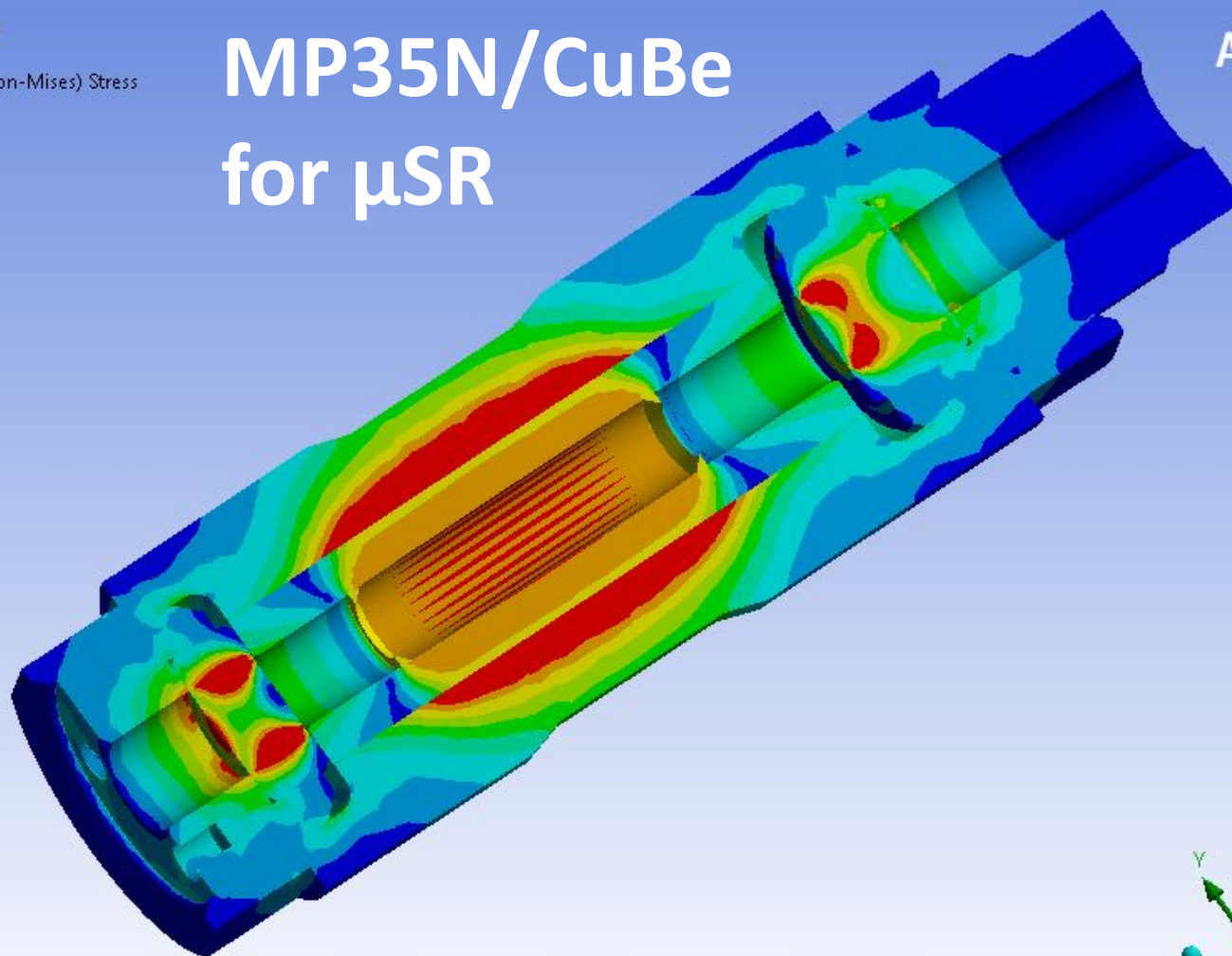
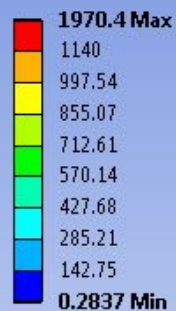
Unit: MPa

Time: 1

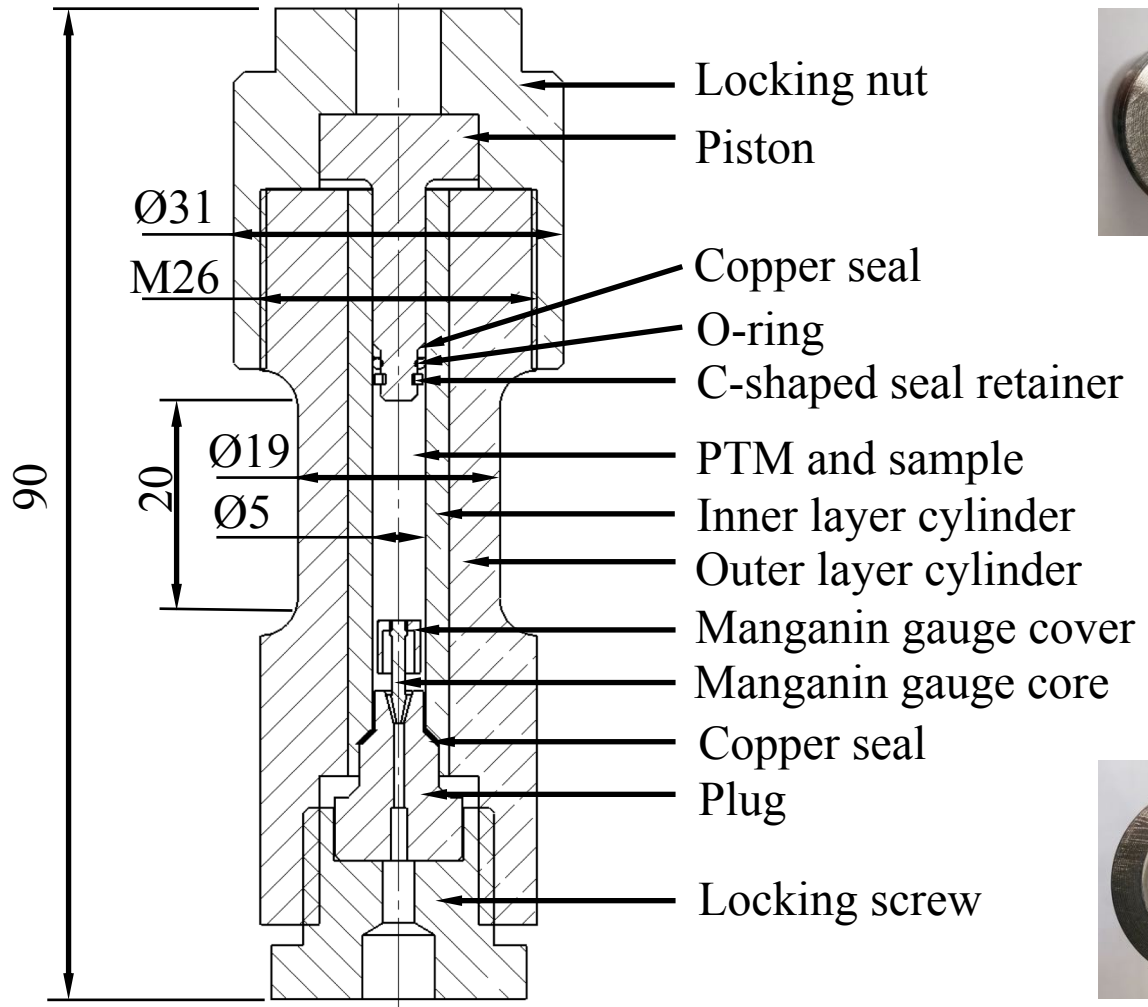
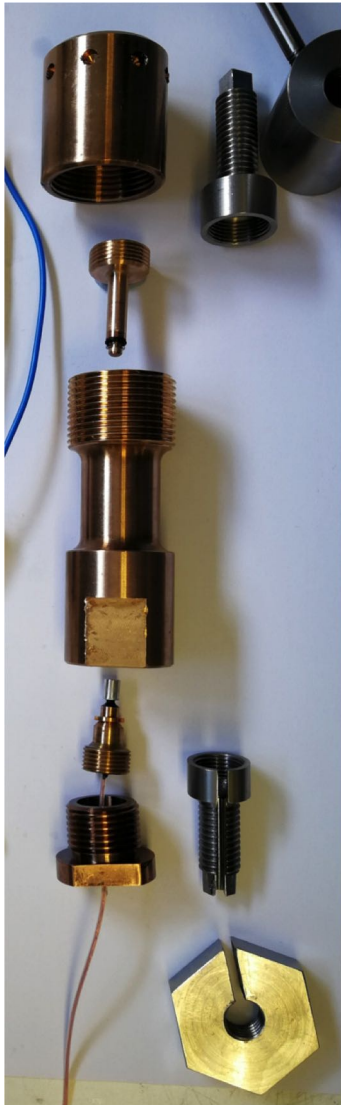
11.01.2017 17:24

MP35N/CuBe for μ SR

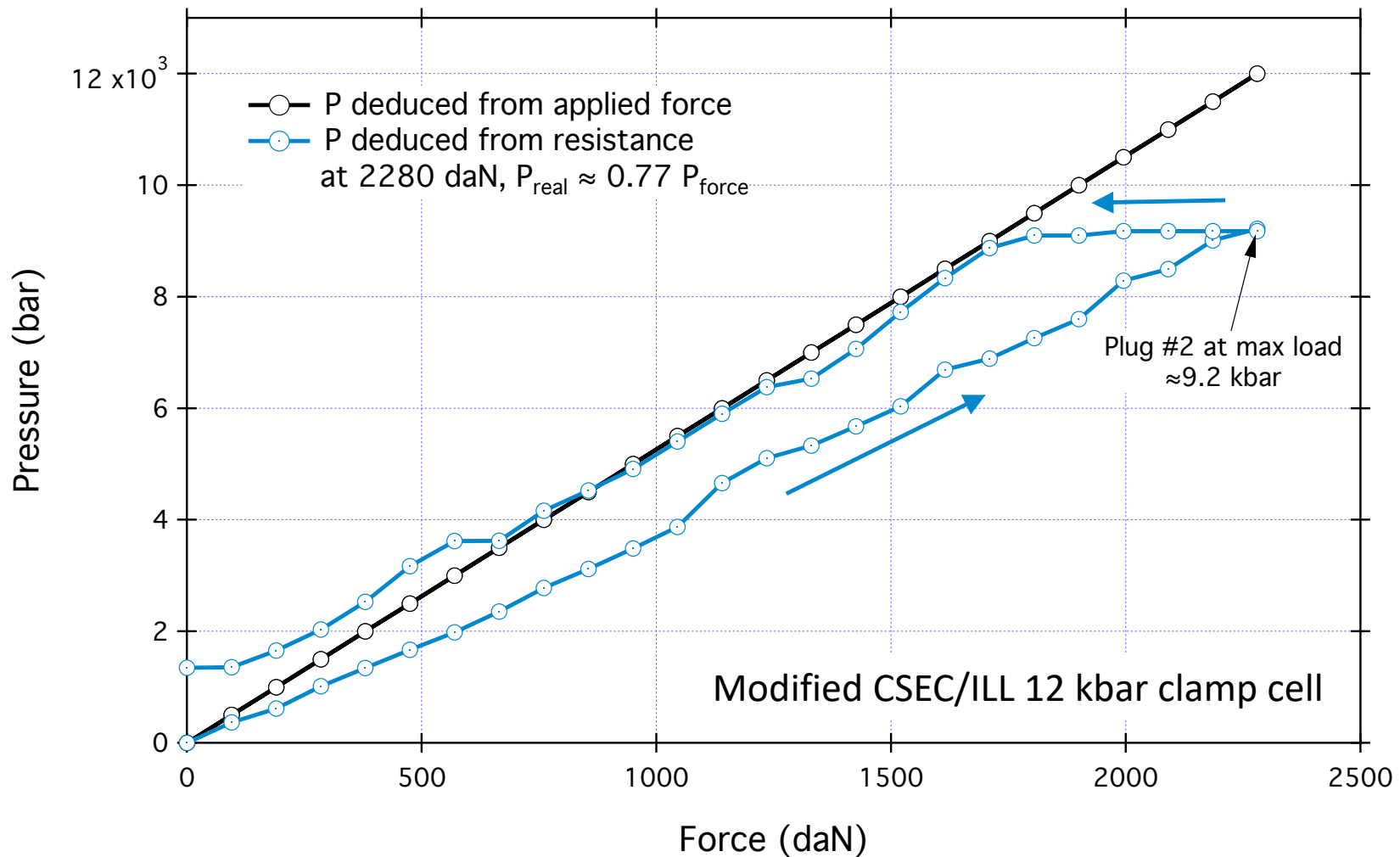
ANSYS
R17.0



Work carried out — Task 7.3



Work carried out — Task 7.3



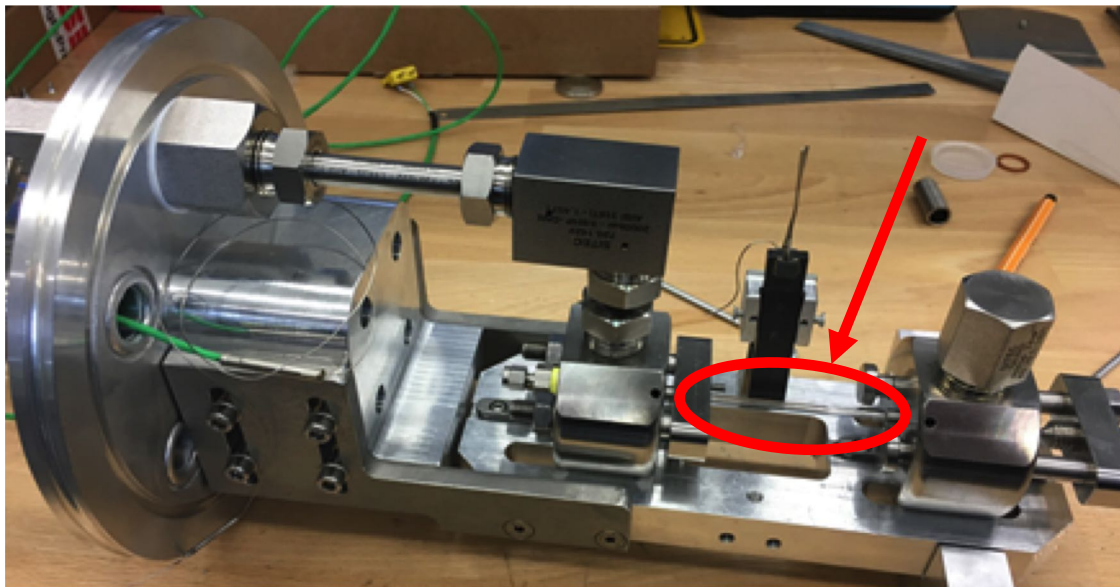
Work carried out — Task 7.3

- Next-generation high-P cells for NS & μ SR
 - Test experiments performed by IMPMC at ILL with Paris-Edinburgh cell have identified solutions for improving the signal to background ratio:
 - transparent sintered diamond anvils
 - thinner TiZr gaskets
 - First tests at ILL on D20 revealed:
 - same signal/bckg ratio with 2x less sample
 - 25% higher pressure efficiency

Work carried out — Task 7.3

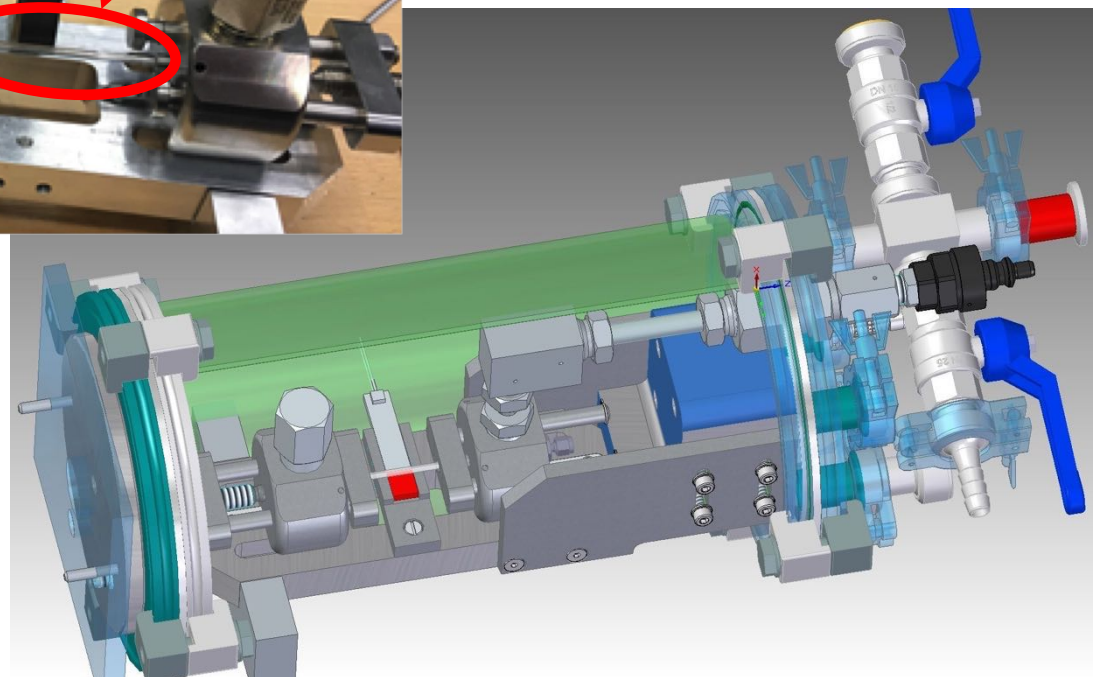
- Next-generation high-P cells for NS & μ SR
 - Review of the 700 bar H₂ container used at X-ray facilities
 - Specifications refined with the help of high-P experts and SE teams of neutron facilities
 - Concept design and engineering drawings produced by HZG and reviewed with partners
 - 700 bar H₂ container built and tested at FRM II

Work carried out — Task 7.3



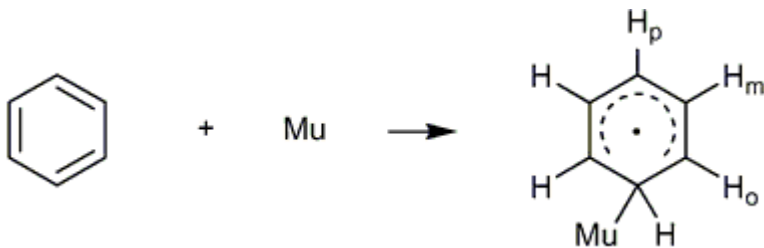
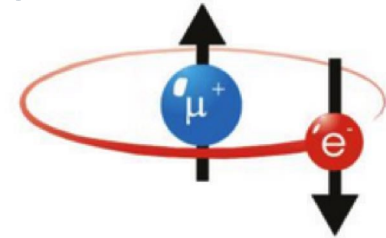
700 bar hydrogen
container for SANS
built at HZG

$\varnothing 1$ mm capillary for SANS
but much larger diameter
required for DIF and INS

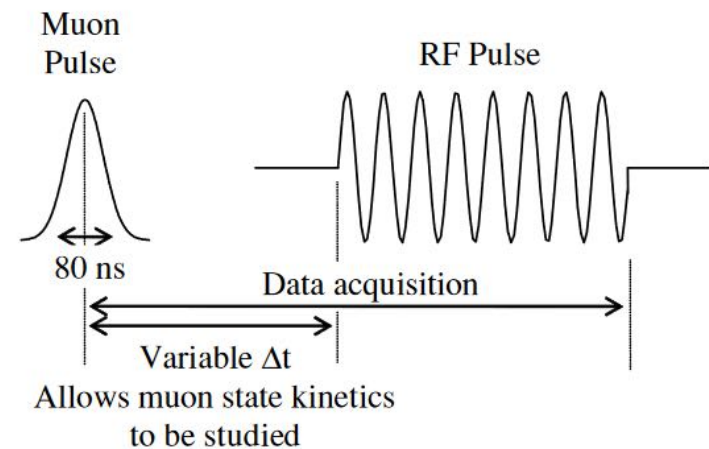


Work carried out — Task 7.4

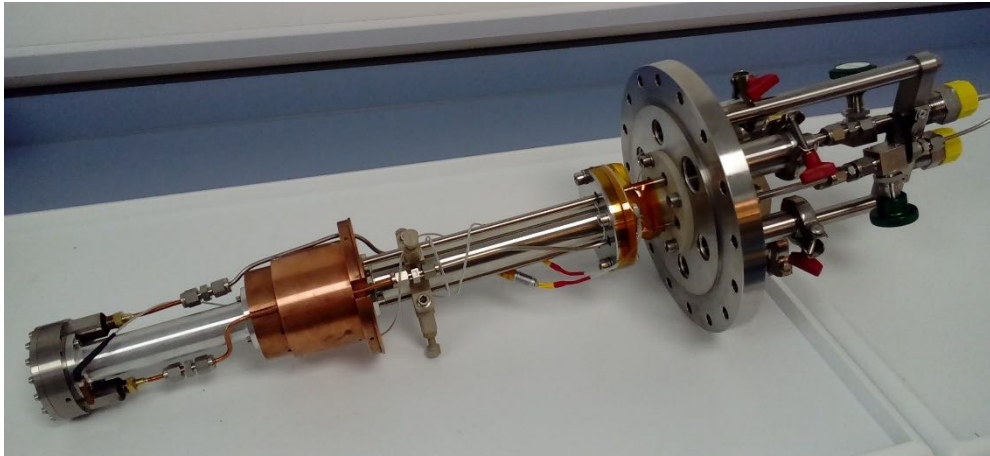
- In-situ muonium studies for μ SR
 - Stainless steel and glass rigs removing O_2 in liquids
 - Insert developed for standard 4-400 K cryostat
 - RF Mu chemistry chamber developed



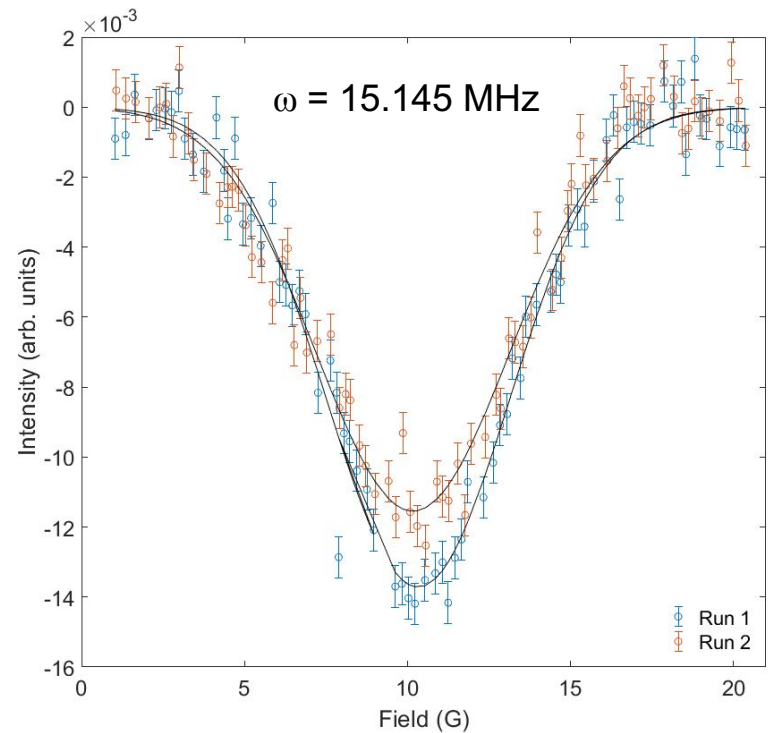
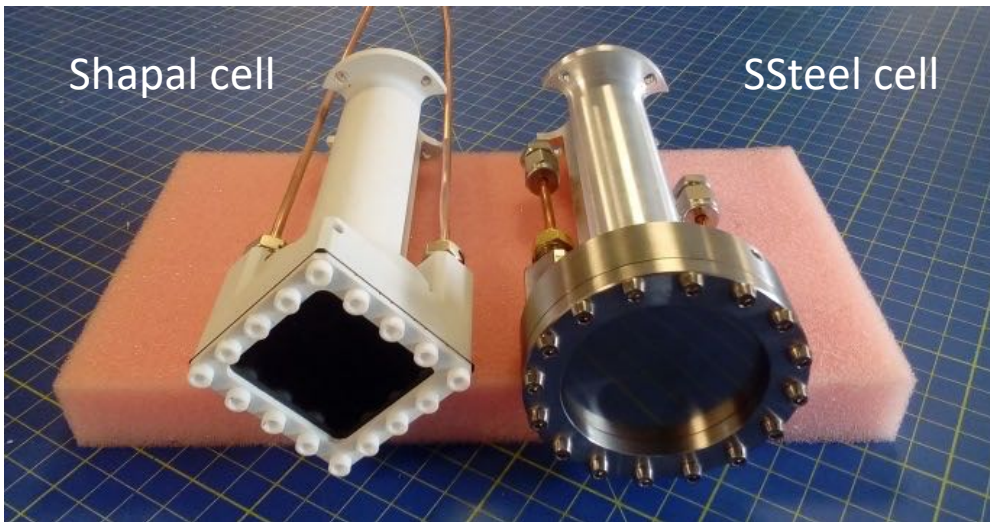
RF-MuSR: Probing reaction final states!



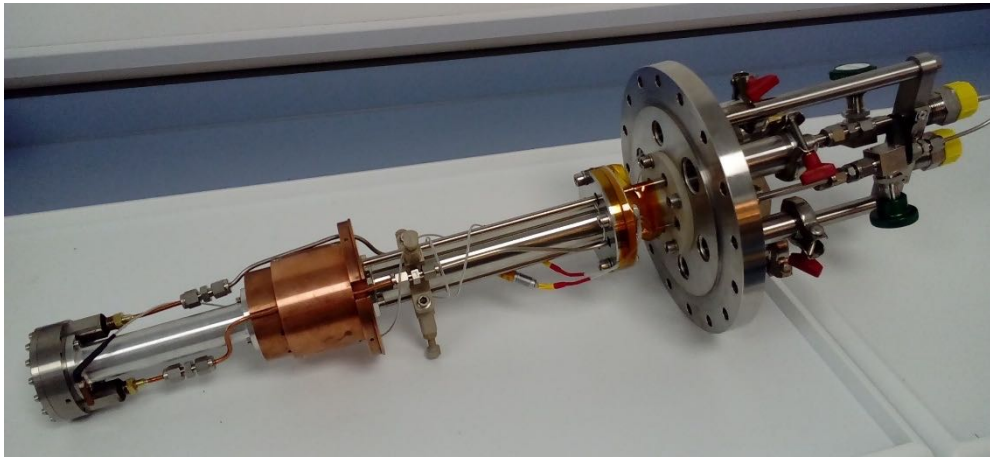
Work carried out — Task 7.4



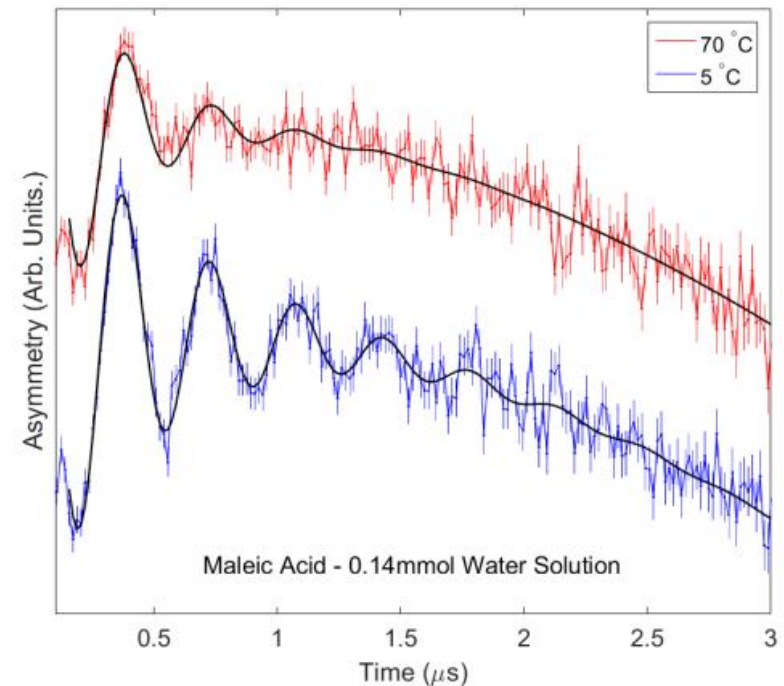
with Shapal cell:
Mu formed in water, sample
flowing, in-situ degassing



Work carried out — Task 7.4



with Stainless-steel cell:
Relaxation rate constants
determined for maleic acid
in water at 5 and 70°C

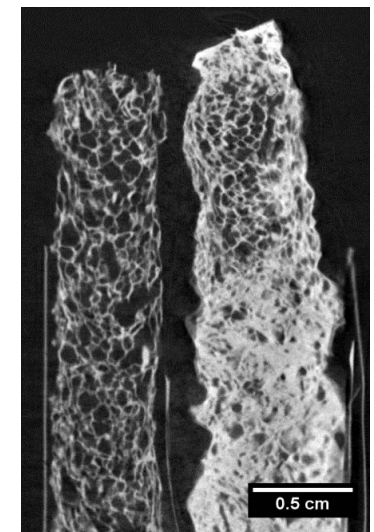
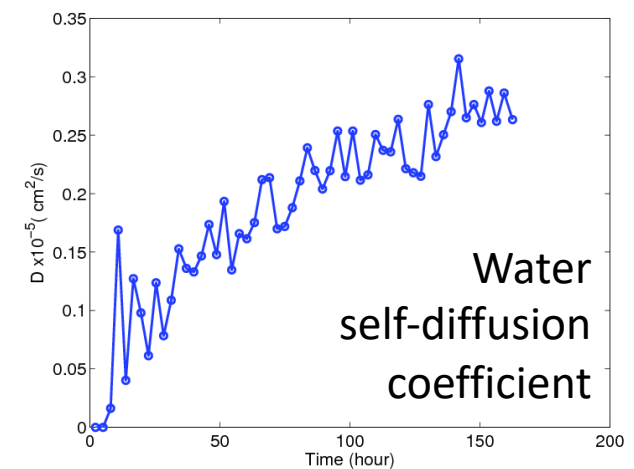
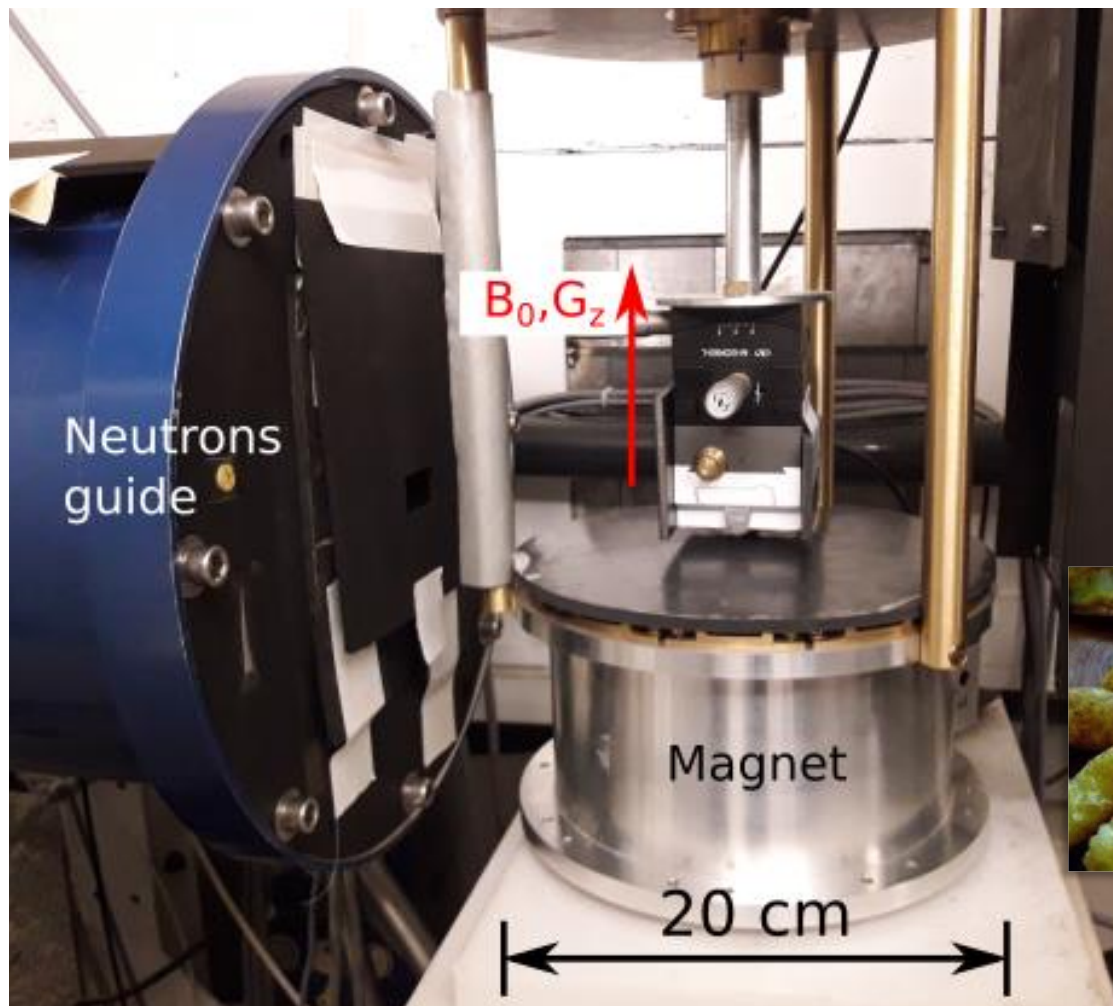


Work carried out — Task 7.4

- In-situ NMR for neutron scattering
 - NMR probe head designed and commissioned successfully on food science case
 - In-situ setup tested successfully at LLB on PRAXY (SANS) and IMAGINE (tomograph) instruments



Work carried out — Task 7.4



Impact / KPIs

- Facilities and contacted companies are adopting the SECoP international standard
- 1.5x better clamp cells, 4x faster furnaces, 3x faster cryostats, 1.5 to 5x lower background cryostats, new in-situ techniques for NS & μ SR
- AS Scientific Ltd upgrade cryostats, attocube will distribute ULT non-magnetic goniometers
- Works soon published in a special issue of JNR

Future...

- Deploy SECoP at all facilities, adopt standard high-P cells featuring in-situ pressure measurement, adopt new PE anvils, etc.
- Meet twice a year, review progress, select common projects and define milestones
 - Work with experts e.g. high-P, high-B, etc.
 - Work with x-ray community on e.g. standards, robotics, He management, SECoP, etc.

**Warm thanks
to all WP7 ~~participants~~ friends
for their great efforts!**

**...and to you
for your attention**