european photon & neutron science campus

NEUTRONS FOR SCIENCE®

Update on ILL activities

Eddy Lelièvre-Berna

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

New site entrance

with tramway line — early until after midnight

New IBS building on site

connected to ILL He recovery line — 430 m

New Science Building

with a blast-room in the basement for H.P. dev.

Master glass blower/maker

for building toys we could not anticipate…

Sample Env. Staff

- S. Baudoin
- N. Belkhier
- E. Bourgeat-Lami
- J. Gonthier
- J.-P. Gonzales
- J. Maurice
- O. Losserand
- A. Ratel
- F. Marchal
- P. Martin
- Y. Memphis
- P. Mendes
- C. Payre
- X. Tonon
- S. Turc
- V. Duchasténier

+ new position soon…

Projects…

- Modernised He recovery
- Vertical low-pressure stick
- Goniostick & cryocradle commissioned
- Liq./Liq. interface cell, stopped-flow head, pressure cells

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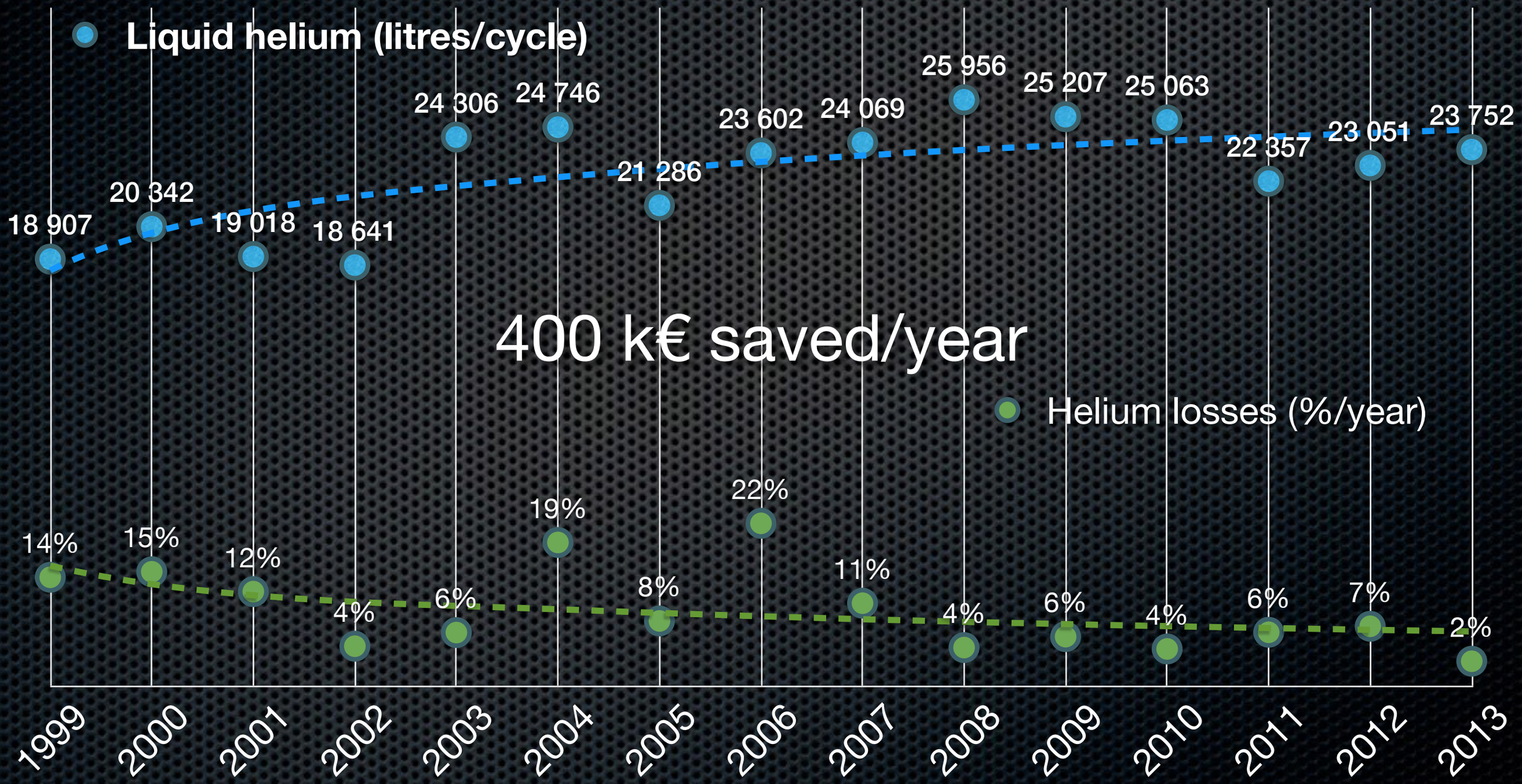
Helium recovery system



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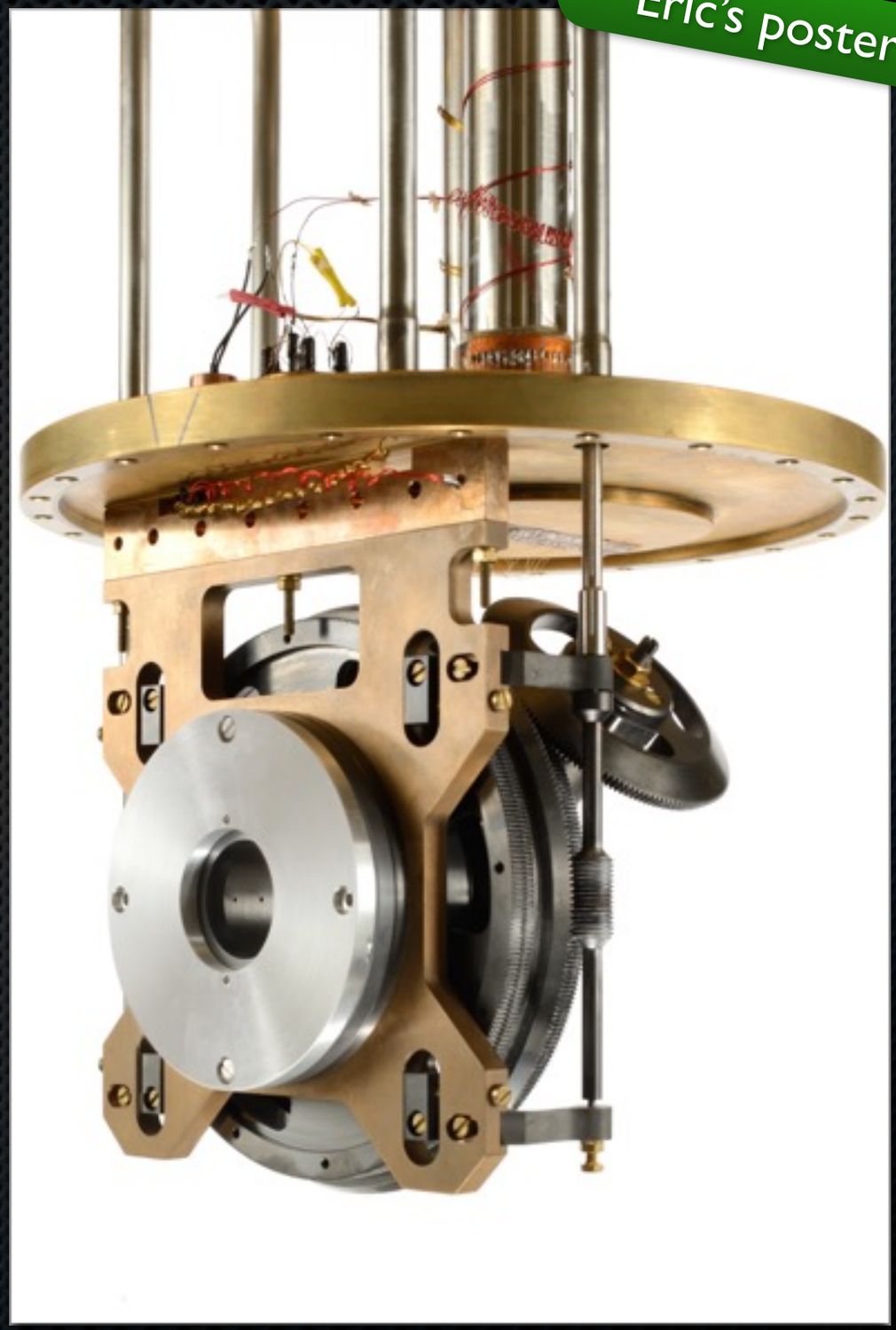

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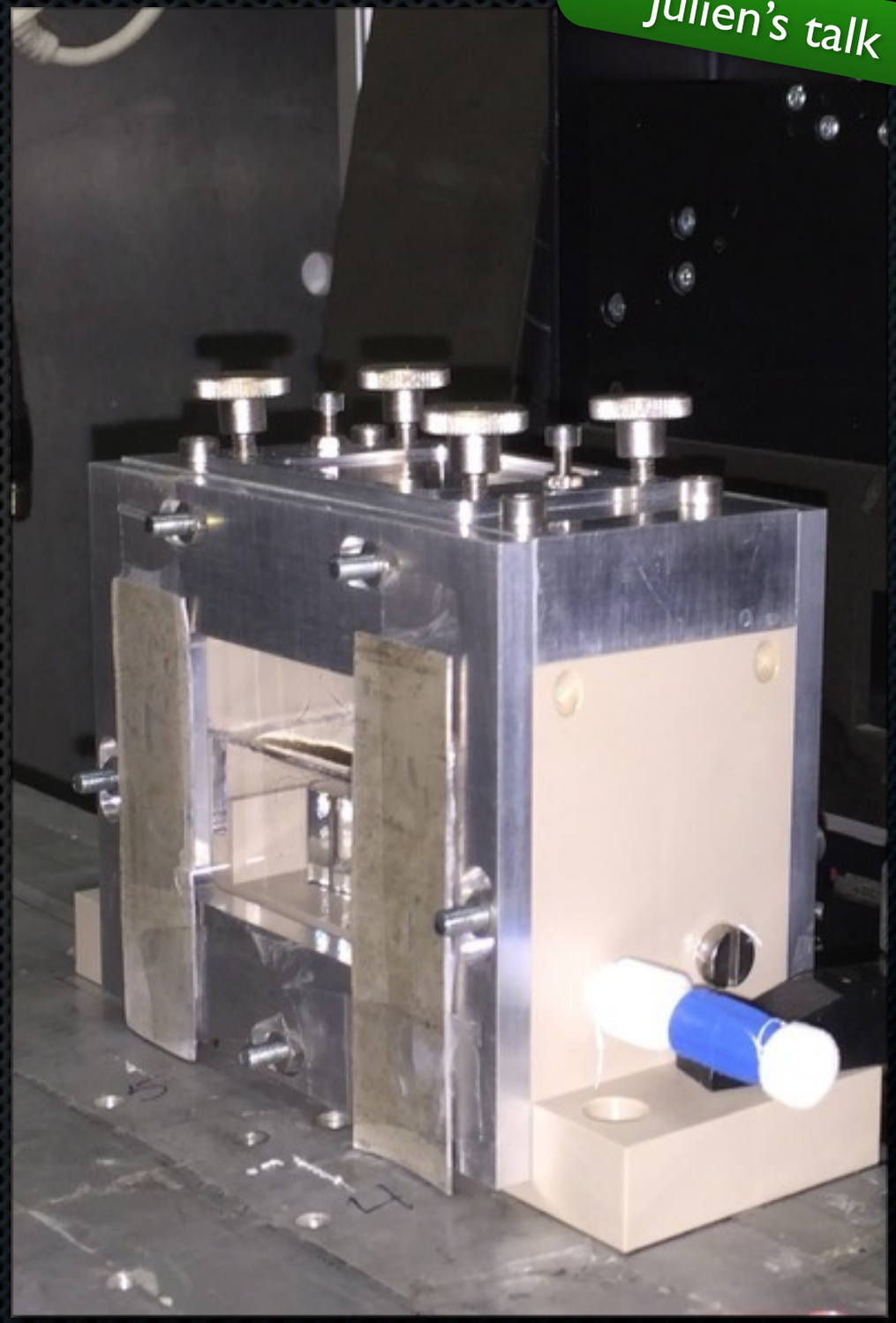

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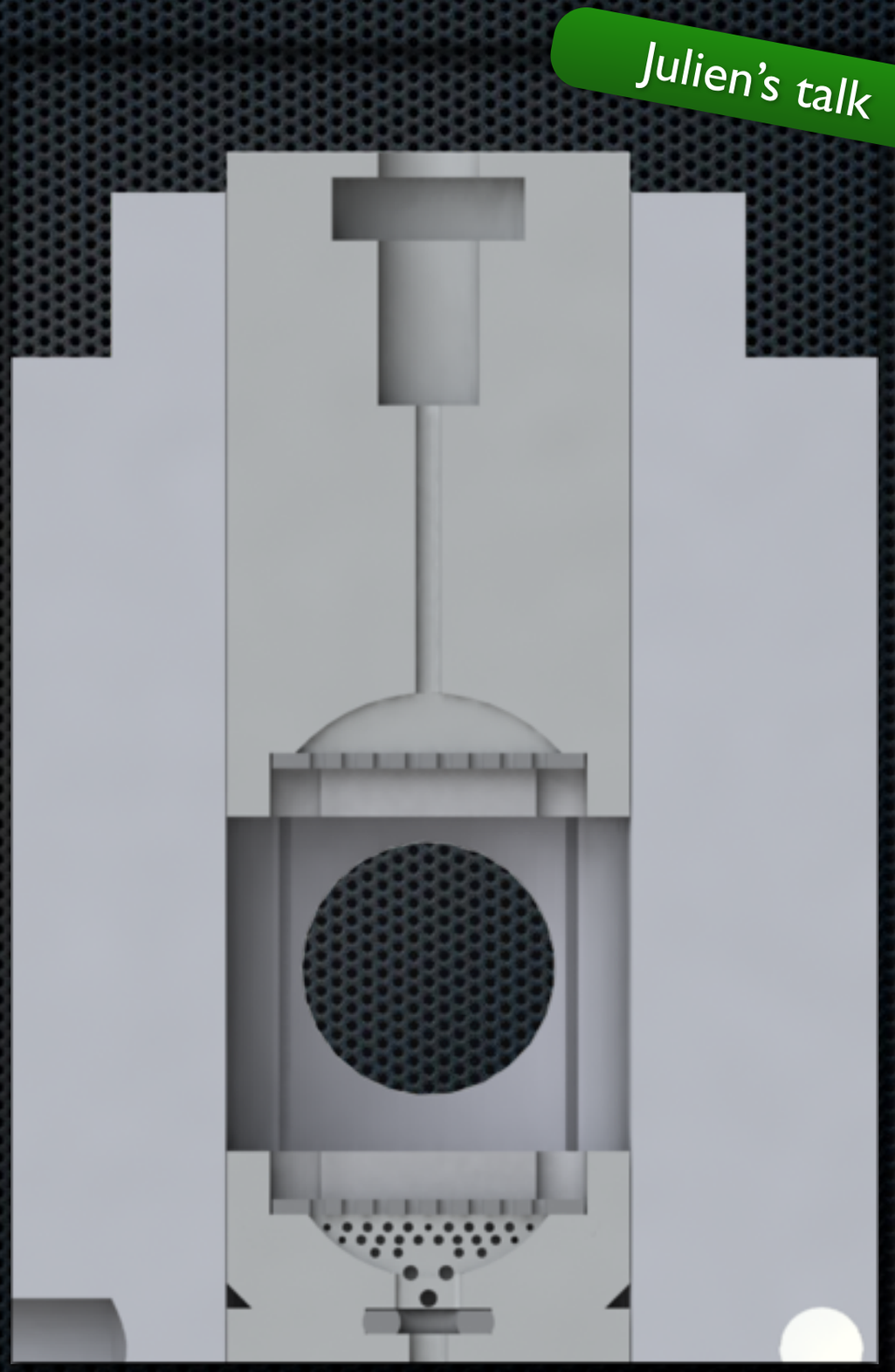

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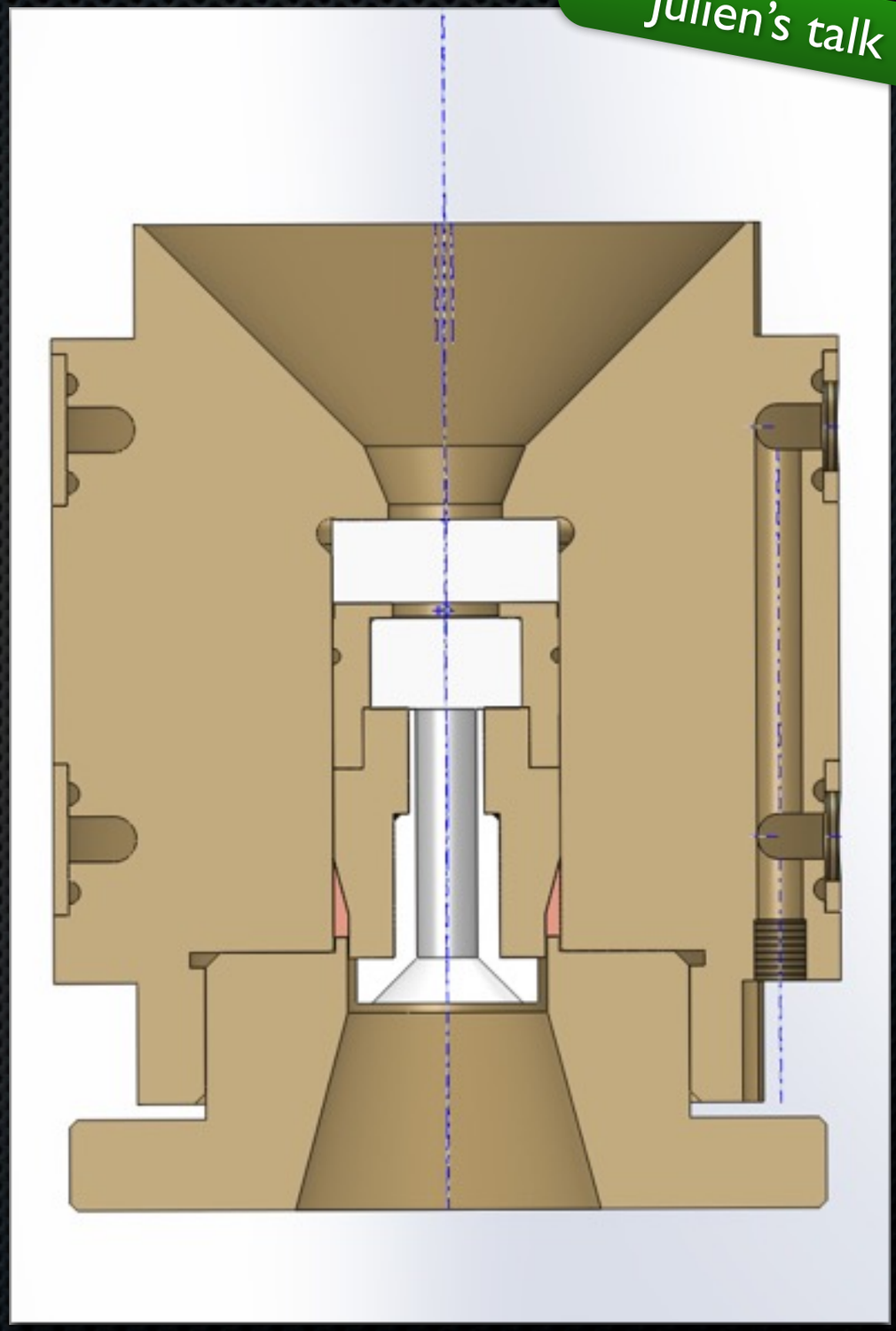

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- 3x faster cryostats with 33% more liquid He
- 100 mK closed-cycle dilution fridge in 4-circle
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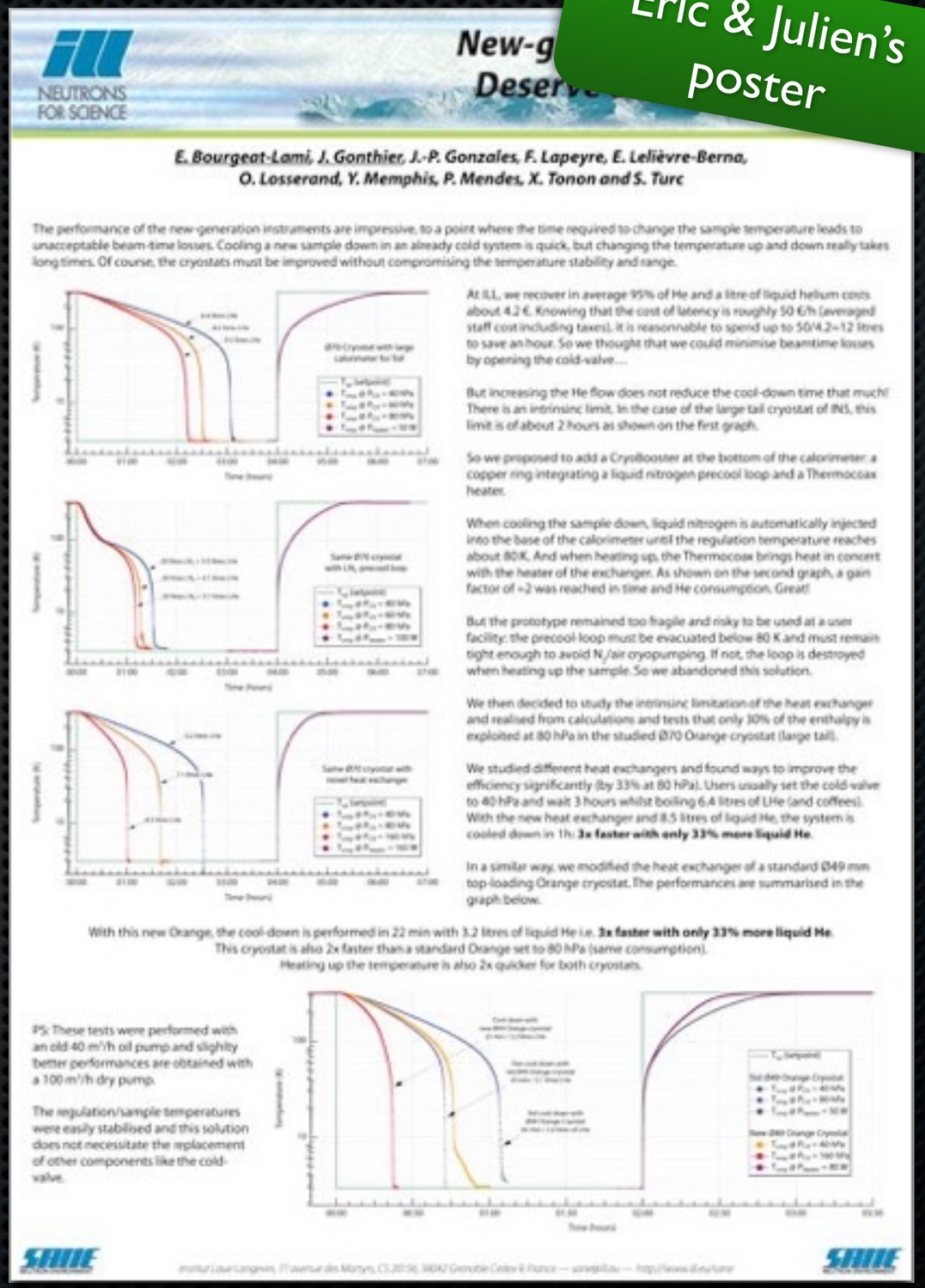
- 40T hor. pulsed field magnet commissioned

Eric & Julien's poster



NEUTRONS FOR SCIENCE

New-g… Deserv…

E. Bourgeat-Lami, J. Gonthier, J.-P. Gonzales, F. Lapeyre, E. Lelièvre-Berna, O. Losserand, Y. Memphis, P. Mendes, X. Tonon and S. Turc

The performance of the new-generation instruments are impressive, to a point where the time required to change the sample temperature leads to unacceptable beam-time losses. Cooling a new sample down in an already cold system is quick, but changing the temperature up and down really takes long times. Of course, the cryostats must be improved without compromising the temperature stability and range.

At ILL, we recover in average 95% of He and a litre of liquid helium costs about 4.2 €. Knowing that the cost of latency is roughly 50 €/h (averaged staff cost including taxes), it is reasonnable to spend up to 50/4.2=12 litres to save an hour. So we thought that we could minimise beamtime losses by opening the cold-valve…

But increasing the He flow does not reduce the cool-down time that much! There is an intrinsinc limit. In the case of the large tail cryostat of INS, this limit is of about 2 hours as shown on the first graph.

So we proposed to add a CryoBooster at the bottom of the calorimeter: a copper ring integrating a liquid nitrogen precool loop and a Thermocoax heater.

When cooling the sample down, liquid nitrogen is automatically injected into the base of the calorimeter until the regulation temperature reaches about 80K. And when heating up, the Thermocoax brings heat in concert with the heater of the exchanger. As shown on the second graph, a gain factor of ≈2 was reached in time and He consumption. Great!

But the prototype remained too fragile and risky to be used at a user facility: the precool loop must be evacuated below 80 K and must remain tight enough to avoid N$_2$/air cryopumping. If not, the loop is destroyed when heating up the sample. So we abandoned this solution.

We then decided to study the intrinsinc limitation of the heat exchanger and realised from calculations and tests that only 30% of the enthalpy is exploited at 80 hPa in the studied Ø70 Orange cryostat (large tail).

We studied different heat exchangers and found ways to improve the efficiency significantly (by 33% at 80 hPa). Users usually set the cold-valve to 40 hPa and wait 3 hours whilst boiling 6.4 litres of LHe (and coffees). With the new heat exchanger and 8.5 litres of liquid He, the system is cooled down in 1h: **3x faster with only 33% more liquid He**.

In a similar way, we modified the heat exchanger of a standard Ø49 mm top-loading Orange cryostat. The performances are summarised in the graph below.

With this new Orange, the cool-down is performed in 22 min with 3.2 litres of liquid He i.e. **3x faster with only 33% more liquid He**.
This cryostat is also 2x faster than a standard Orange set to 80 hPa (same consumption).
Heating up the temperature is also 2x quicker for both cryostats.

PS: These tests were performed with an old 40 m³/h oil pump and slighlty better performances are obtained with a 100 m³/h dry pump.

The regulation/sample temperatures were easily stabilised and this solution does not necessitate the replacement of other components like the cold-valve.

SANE

Institut Laue Langevin, 71 avenue des Martyrs, CS 20156, 38042 Grenoble Cedex 9, France — sane@ill.eu — http://www.ill.eu/sane

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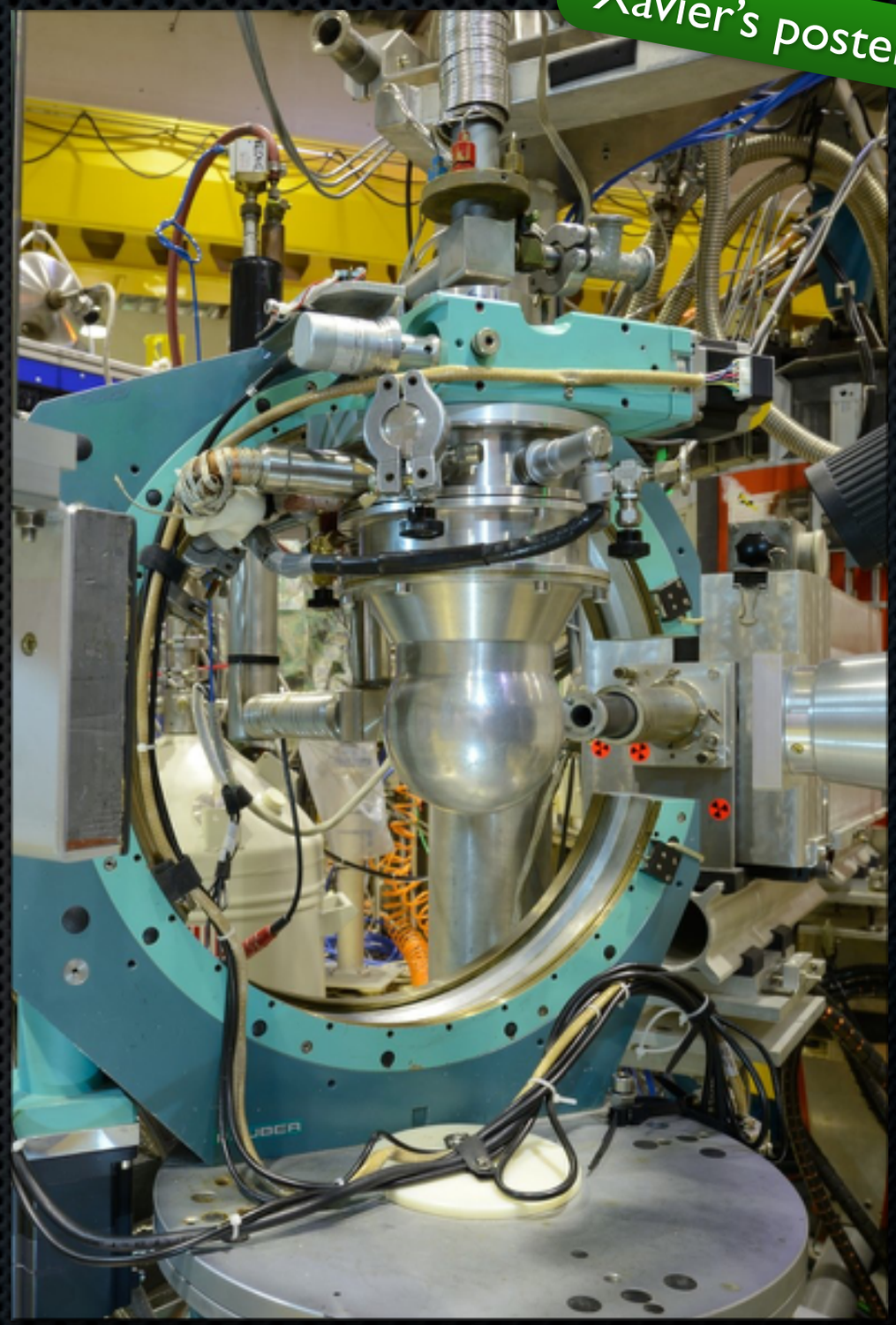

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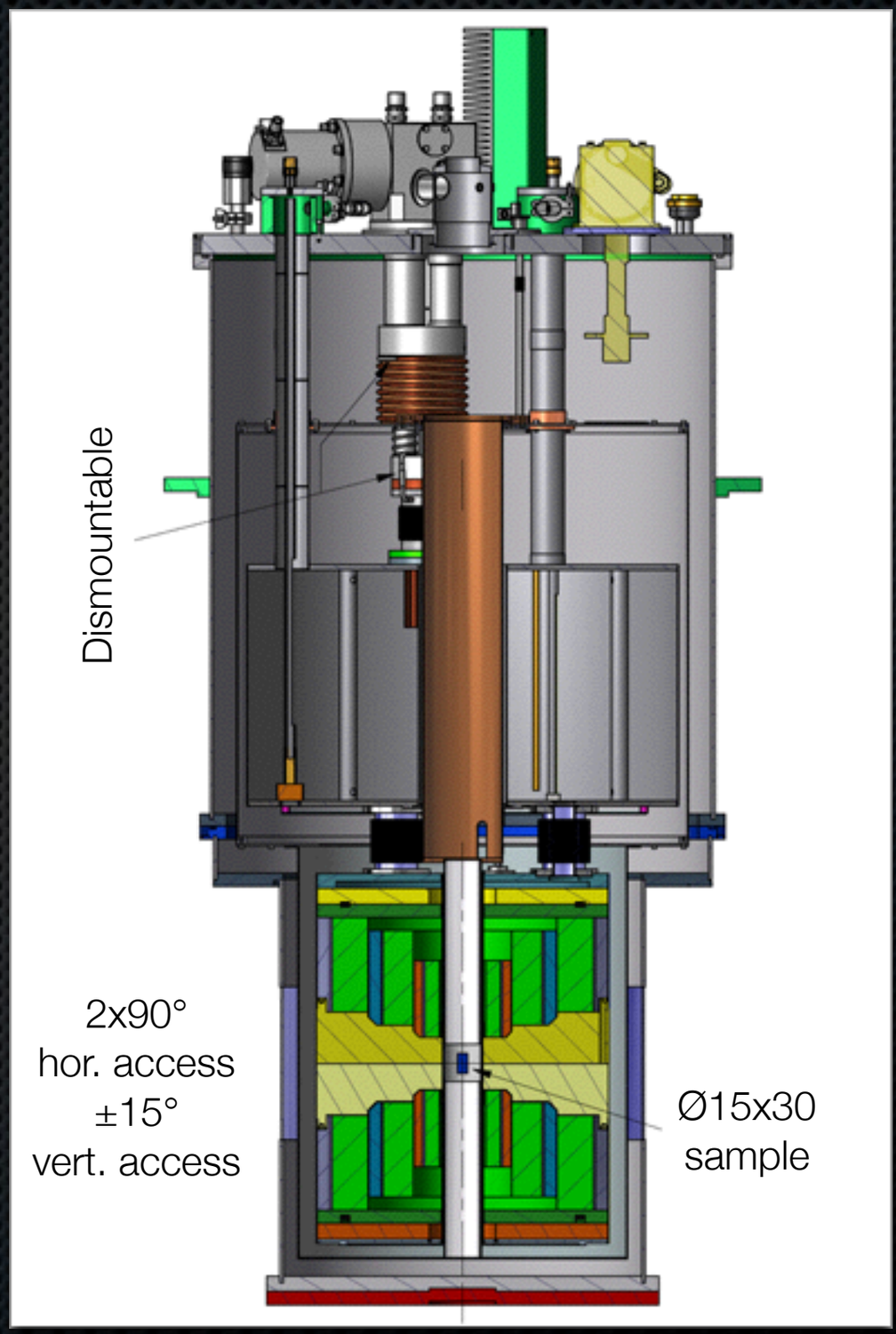

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Collaborative Projects

- ANR High Pressure for Bio — until 2015
- ANR 40T Mag. Field for NS — until 2015
- NMI3 Soft & Bio Materials JRA — until 2016
- NMI3 Adv. Methods & Technics JRA — until 2016
- SINE2020 Sample Environment JRA — submitted
- TASUM 30T All-Superconducting Magnet — submitted
- ANR Mol. Switchable under High-P — in preparation
- ILL Endurance (NESSE: NEw Standards for Sample Env.)

NESSE Project

Bio Materials, Liquids, etc. [1/2]

- Humidity chambers
- Stopped-flow systems (T-jump option, N chambers)
- Dedicated high pressure cells and splitters
- Specific high temperature equipment
- Adsorption troughs, sorption sticks at low/high-T
- T-controlled reaction cells (acids, solid/liq., solid/gases)
- T-controlled acoustic levitation for bio samples

NESSE Project

Bio Materials, Liquids, etc. [2/2]

- Novel and spare rheometers
- T-controlled UV/Vis/IR light irradiation cell
- T-controlled electric field cells
- SAXS X-ray components for cooling, aligning, etc.
- Acquire DLS/SLS setups for SANS
- T-controlled in-situ dialysis
- etc…

NESSE Project
Low Temperature

- Upgrade He recovery system
- Upgrade ageing gas handling systems of dilution refrigerators with automated units
- Speed up existing cryostats (x3 with 33% more LHe ?)
- Reduce significantly the background produced by cryostats, dilution inserts, etc.
- Deploy goniometers, cryostats with sample changer

NESSE Project
High Pressure & Temperature

- Liquids: Acquire 2 kbar syringes, build +7 kbar regulators, develop novel hybrid clamps with in-situ pressure measurement
- Gases: Acquire 3 / 7 /10 kbar controllers, build cryostat for VX-1 press, provide nano-polycrystalline or VTA anvils for PE cells, warming components for PE cells
- Speed up cool-down in furnaces, reduce background issues encountered with furnaces

NESSE Project
High-Field Magnets, Polarimeters

- Provide 12T horizontal field split-pair magnet for SANS with dilution insert, rotating table, goniometer
- Replace ageing 4T horizontal field magnet for TAS with dilution insert, rotating table, goniometer
- Build zero-field polarimeter for Nucl. & Part. Physics

PS: The 11T magnet for IN5 is being manufactured and covered by the Millennium Project

NESSE Project (2015-2023)

Bio Materials, Liquids, Films & Glasses	3.0 M€
Low Temperature	2.8 M€
High Pressure & Temperature	1.8 M€
High-Field Magnets, Polarimeter	2.4 M€
Total investment including external staff	10.0 M€

Organisation

- Dynamic process ! Scientific priorities refined on regular basis: Scientific Councils, IGLs' meetings, Experiments needs, etc.
- Highest priority given to experiments: few sub-projects in parallel — too many will lead to demotivation and lower efficiency.
- Harmonisation with external grants whenever possible to increase staff resources and financial support
- Don't reinvent the wheel, unless you're asked on learning more about wheels !

Organisation

- We shall build short-term sub-project teams:
  - With directly concerned instrument teams
  - Through collaborations with facilities and labs (not only through EC and ANR projects).
  - Studentships (e.g. draftsmen, engineers, technicians)
  - Internal mobility

In 2015...

- Humidity chambers with D16 team & HZB
- Liquid-liquid interface cell with G. Fragneto & ESRF
- HP cell for bio samples with J. Peters (ANR)
- HP cell for SANS/NSE with D11 team, ToF and LLB.
- Stopped-flow observation heads with I. Grillo & ISIS
- Commission 11T with Cryogenic and IN5 team
- Boost performance of cryostats, cryofurnaces
- Replace ageing He level monitors of dewars

Special thanks to a great team…

Simon Baudoin
Eric Bourgeat-Lami
Julien Gonthier
Frédéric Marchal
James Maurice
Yohan Memphis
Sébastien Turc

—

Agnès Ratel
Frédéric Ivens

Nadir Belkhier
Jean-Paul Gonzales
Olivier Losserand
Paul Martin
Patrice Mendes
Claude Payre
Xavier Tonon

—

Valérie Duchasténier
Christian Lazzarotto

Many thanks for your attention