FREIA science case, requirements and layout

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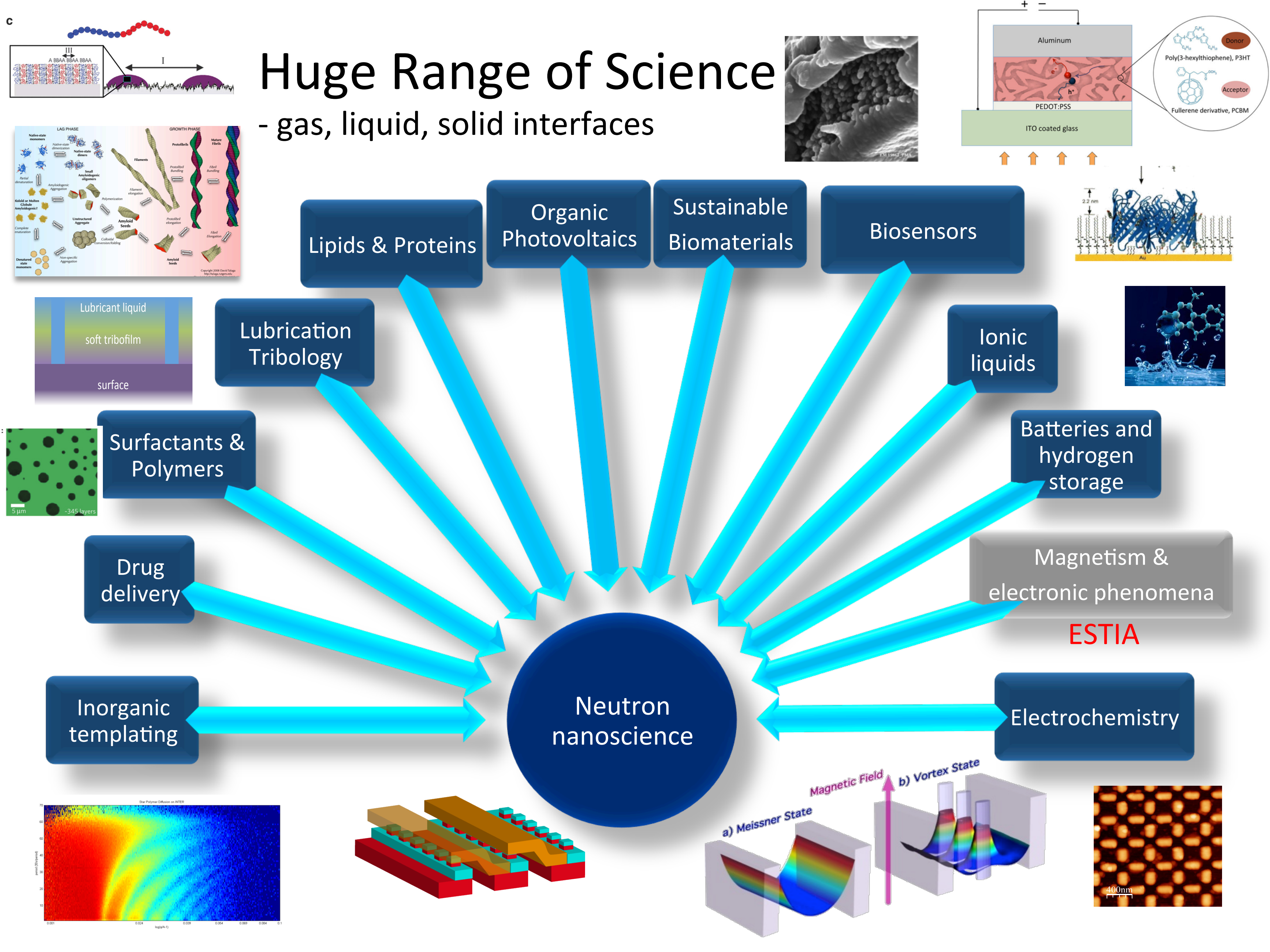
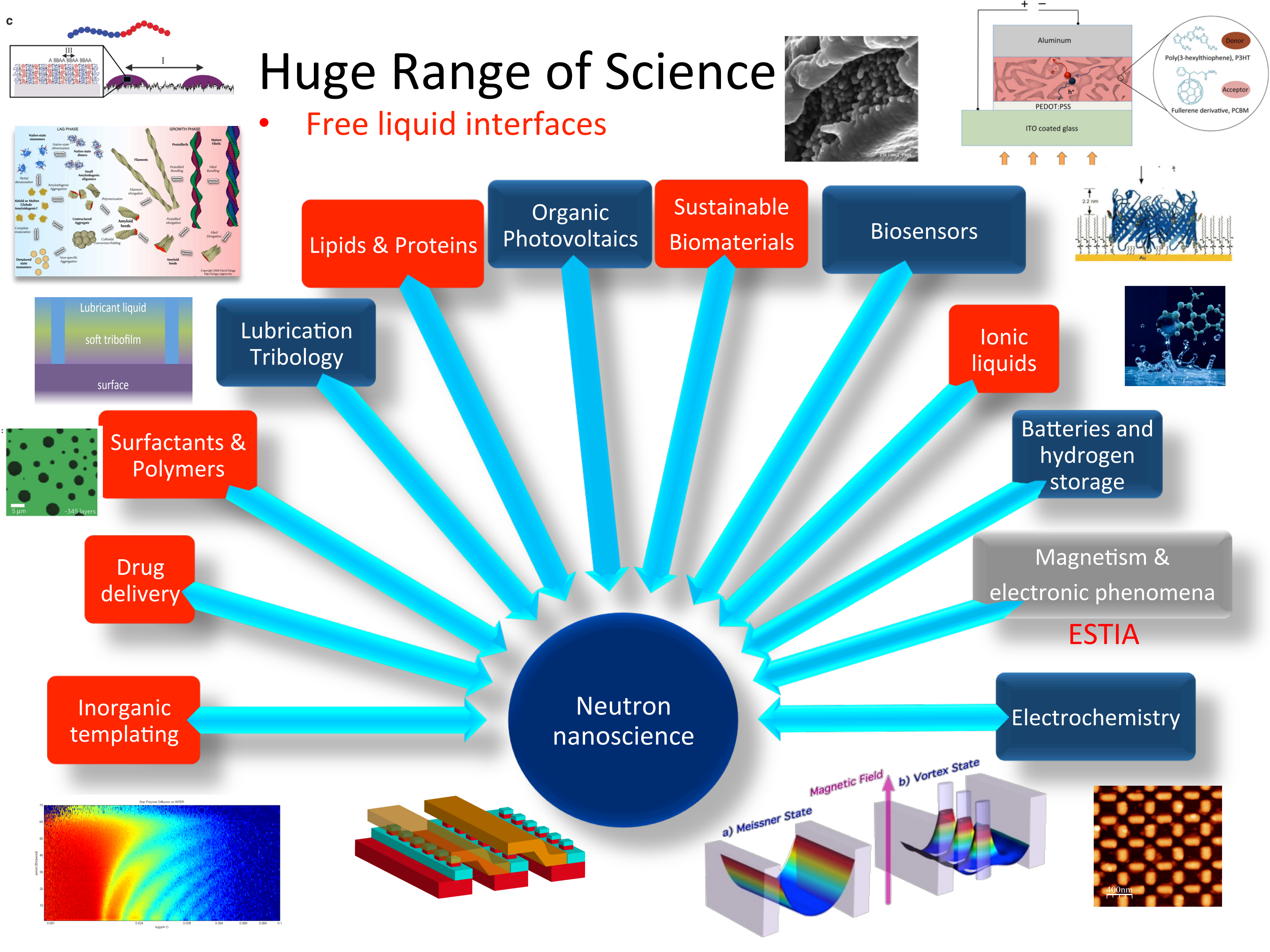
17th of October 2016

Summary

- Science case and Complementarity with ESTIA
- Operation modes and High-level requirements
- Instrument layout and components
- Sample environments
- Scientific Impact of Options 1-3


Huge Range of Science
- gas, liquid, solid interfaces
Lipids & Proteins
Organic Photovoltaics
Sustainable Biomaterials
Biosensors
Lubrication Tribology
Ionic liquids
Surfactants & Polymers
Batteries and hydrogen storage
Drug delivery
Magnetism & electronic phenomena
ESTIA
Inorganic templating
Neutron nanoscience
Electrochemistry
Aluminum
PEDOT:PSS
ITO coated glass
Donor
Acceptor
Poly(3-hexylthiophene), P3HT
Fullerene derivative, PCBM
LAG PHASE
GROWTH PHASE
Lubricant liquid
soft tribofilm
surface
5 µm
~345 layers
a) Meissner State
Magnetic Field
b) Vortex State
400nm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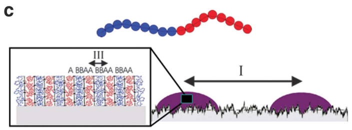
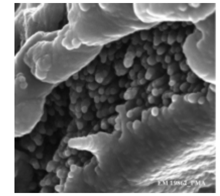
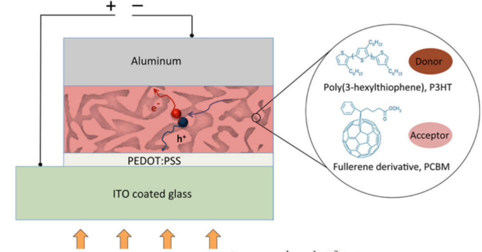
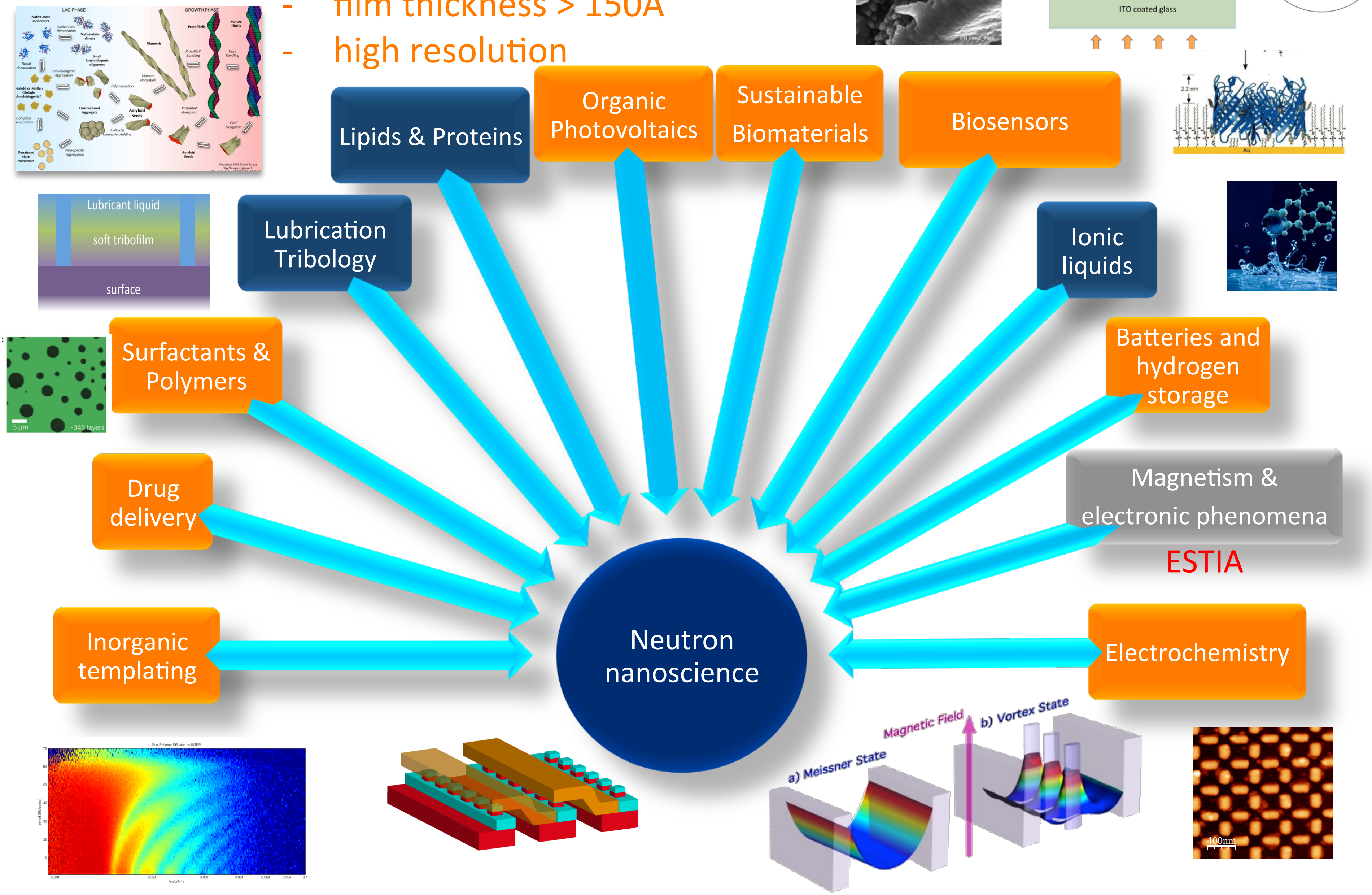


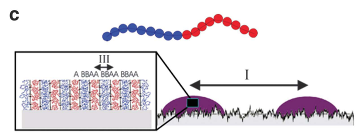
Huge Range of Science

- film thickness > 150Å
- high resolution



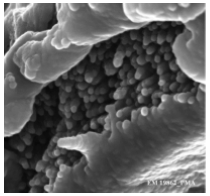


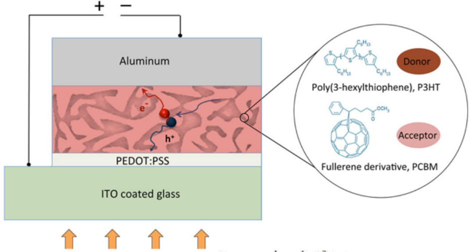


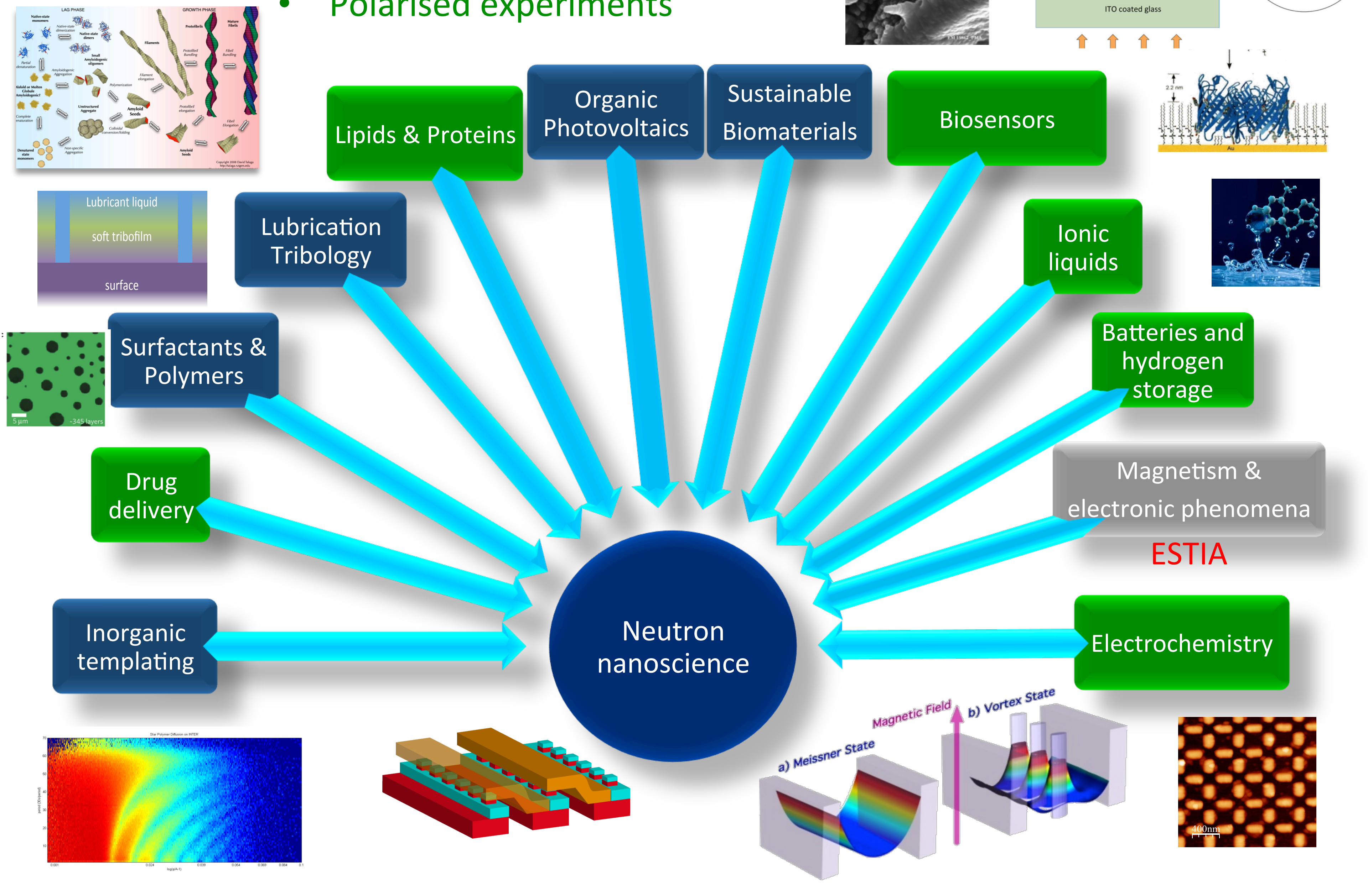


Huge Range of Science

- Polarised experiments

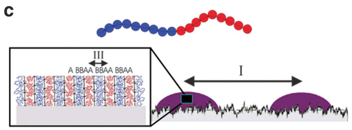


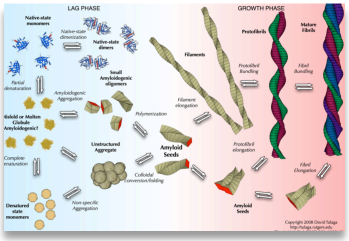


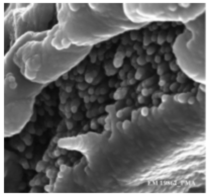


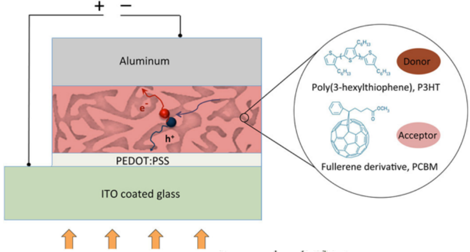
Huge Range of Science

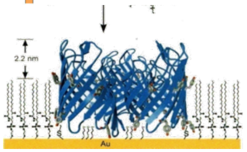
- Time-resolved experiments
- GISANS

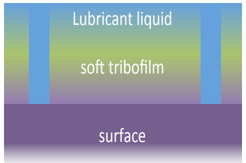


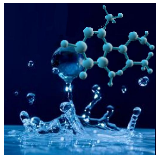


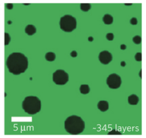












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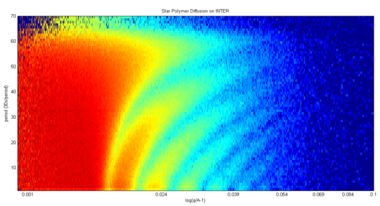
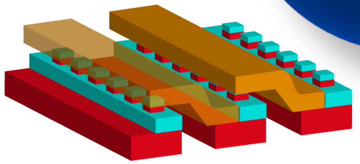
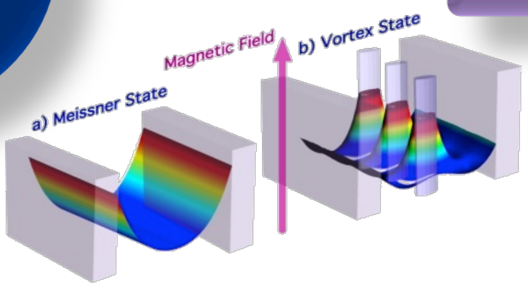
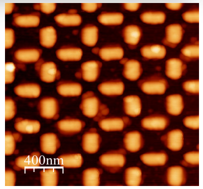




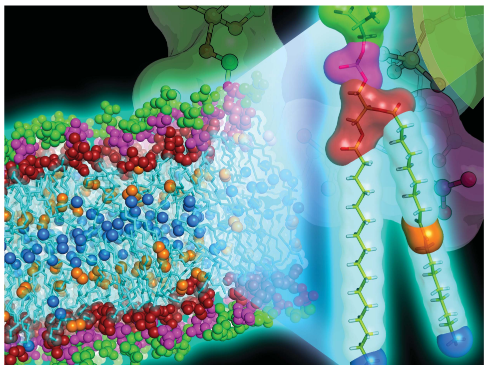




FREIA Science Case

In-situ time-resolved reflectometry for soft condensed matter, life science and functional materials

Structure

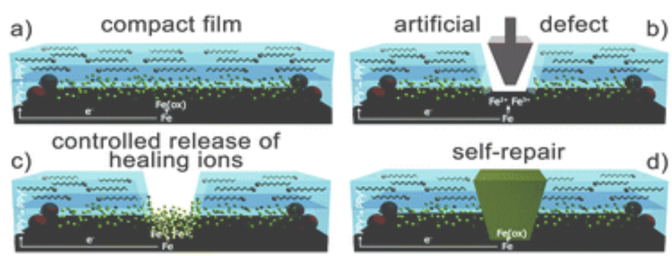


Design

Instrument needed:

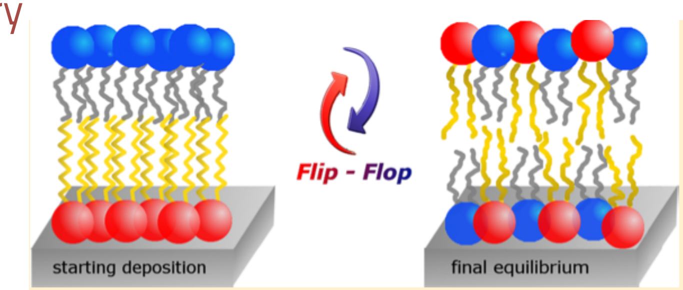
- Horizontal sample geometry
- Flexible collimation
- Variable resolution
- Broad simultaneous Q
- No sample movement

Applications



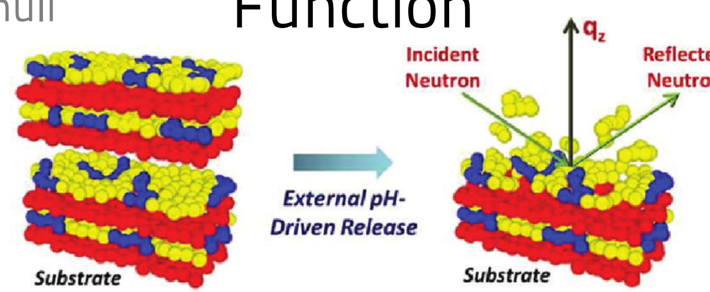
- response to external stimuli
- in situ and in operando
- complex sample environments

Dynamics



- deposition, structure and phase behavior
- adsorption, self-assembly and reactions
- gas/liquid/solid interfaces

Function



FREIA Business Case

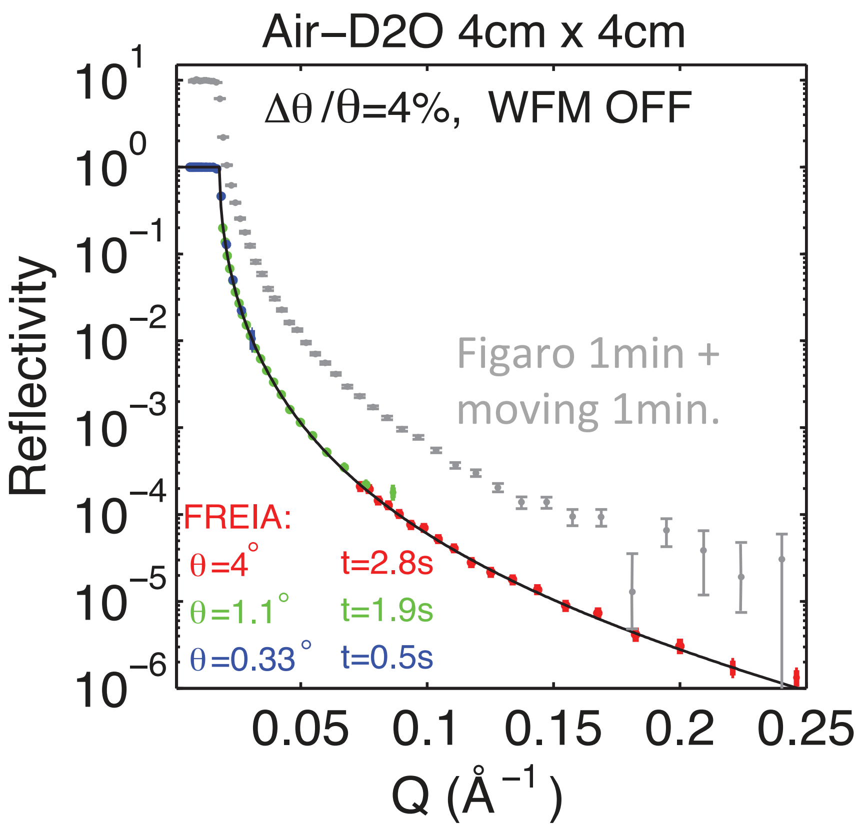
Time-resolved and high-throughput reflectometry to match ESS long pulse performance

Configuration/angle changes to record full Q-range often take several minutes for liquids samples.

Today: record time-resolved data at one angle with limited Q-range.

At ESS, it will be possible to measure a reflectivity curve to $R=10^{-6}$ from D_2O in 1-2s

-> need to change angles/samples fast

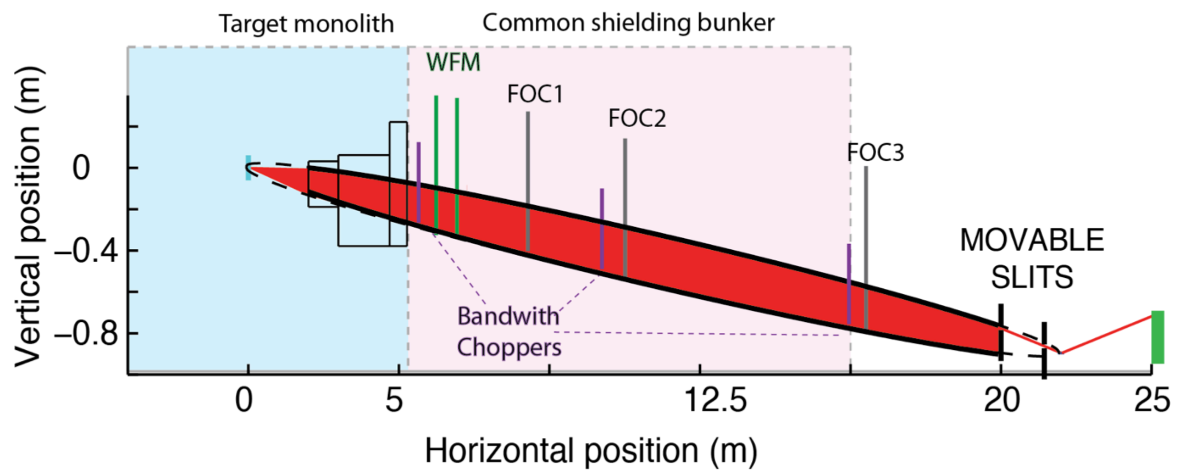


From instrument proposal (TDR moderator)

FREIA Business Case

Time-resolved and high-throughput reflectometry to match ESS long pulse performance

At ESS, it will be possible to measure a reflectivity curve to R=10^{-6} from D_2O in 1-2s.



ESS will allow ms-s measurements over a wide Q-range:

- Avoid unnecessary movements/configuration changes
- Long-pulse/14Hz ideal for fast kinetics using a broad λ band

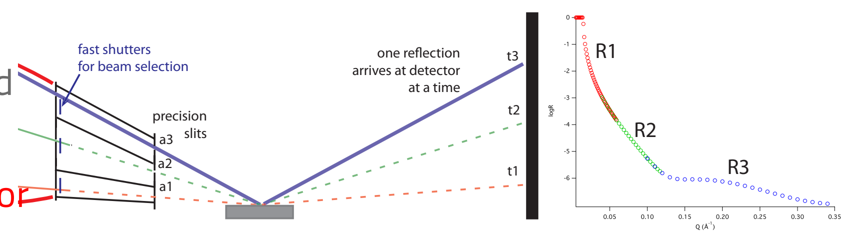
FREIA is optimised for time-resolved/high throughput studies:

- Wide vertical divergence - no movement of sample/detector
- Extended simultaneous Q range with fast shutters
- High flux mode: kinetics, thin films (<150Å)

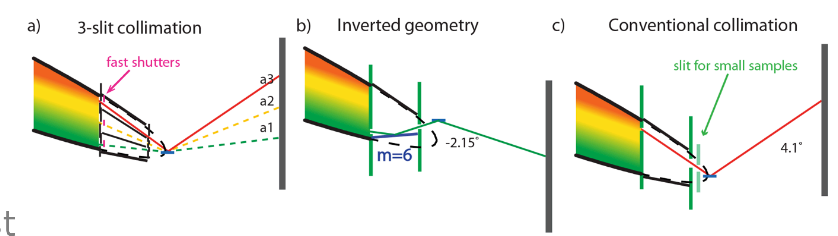
FREIA is a flexible instrument for broad science coverage

- High resolution mode: thicker films, GISANS
- Flexible collimation for liquids, in situ SE, small samples
- Polarised beam /PA option for magnetic materials/contrast

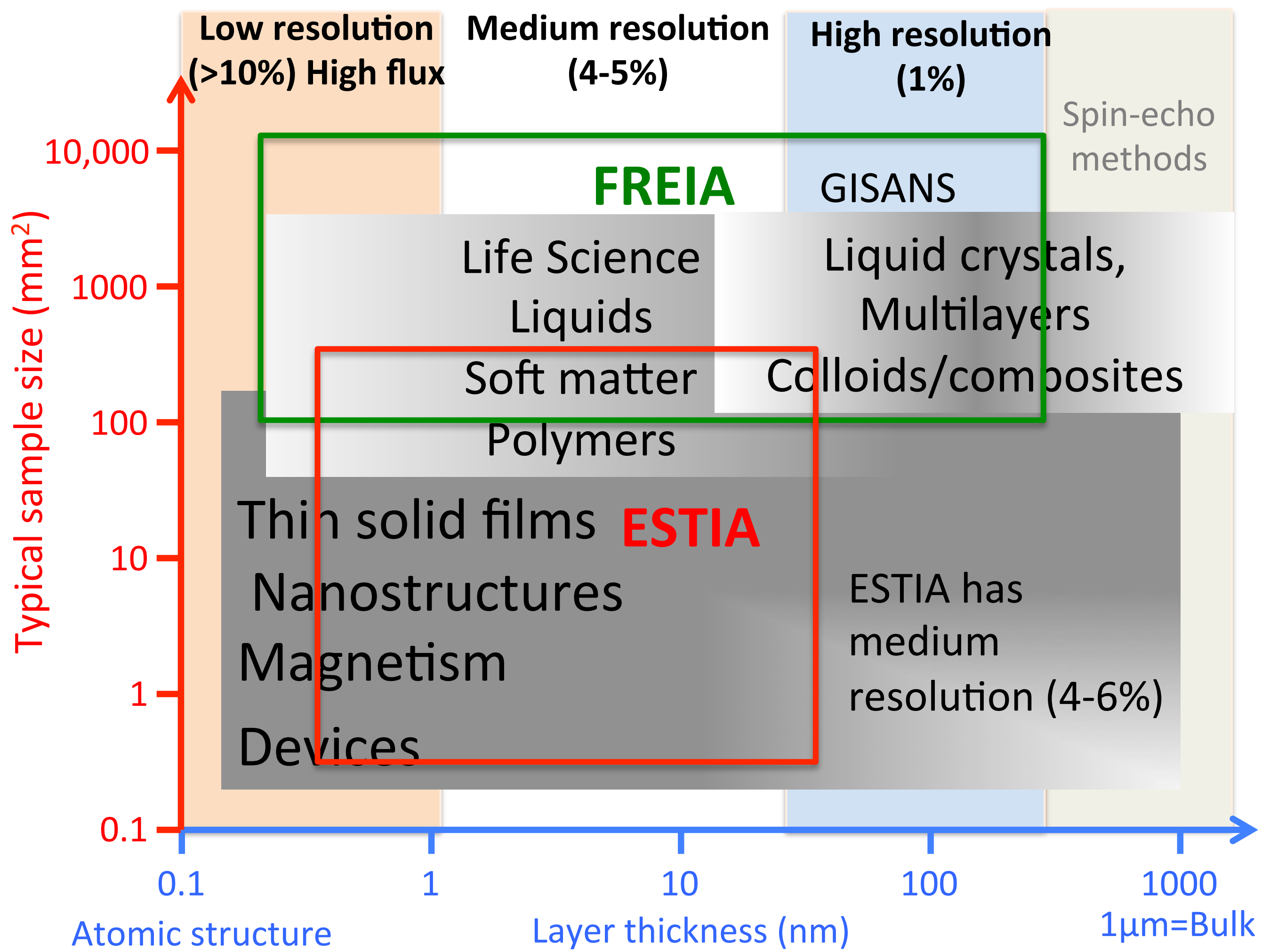
Fast shutters: 0.0035 - 0.4$Å^{-1}$ in one shot



Flexible collimation options:



Resolution and sample sizes



Complementarity

- ESTIA can cover many solid-liquid soft matter experiments, but the the high-flux mode of ESTIA is not for liquid samples.
- GISANS and liquid-liquid experiments (inverted geometry) are limited in Q-range and flux on FREIA due to the optimisation for time-resolved experiments.
- ESTIA has small sample sizes and fixed detector distance – not ideal for GISANS from soft matter (also not day 1 option).
- ESS should ideally build a third instrument optimised for GISANS and the inverted geometry.

Operation modes on FREIA

EUROPEAN SPALLATION SOURCE

I. High-Intensity Specular and Off-specular Reflection 50% of experiments
II. High-resolution Specular and Off-Specular Reflection 50%
III. Fast kinetic measurements (ms-s) with broad Q-range 50%?
IV. Polarized time-of-flight (TOF) neutron reflection 15% but could grow
V. Inverted Beam Geometry at least 25%
VI. Grazing Incidence SANS 30-40%?

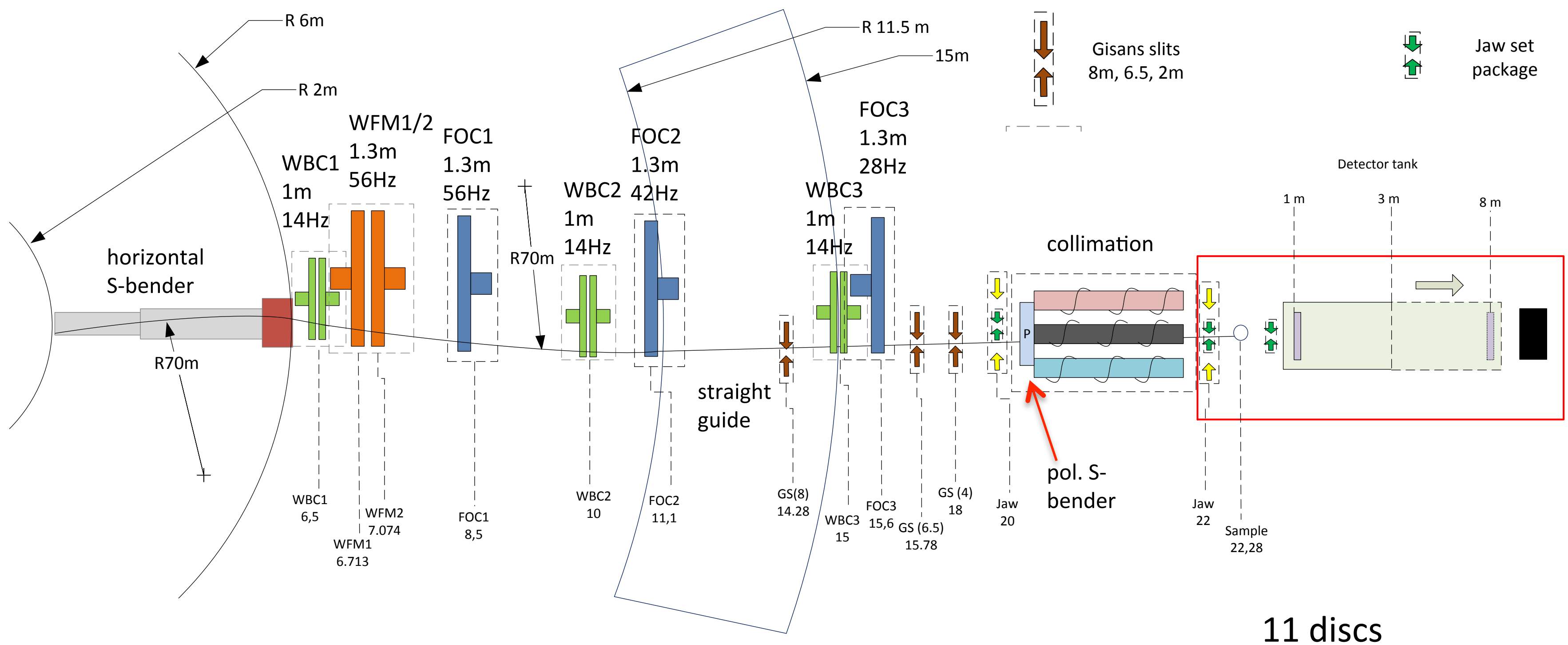
High-level requirements

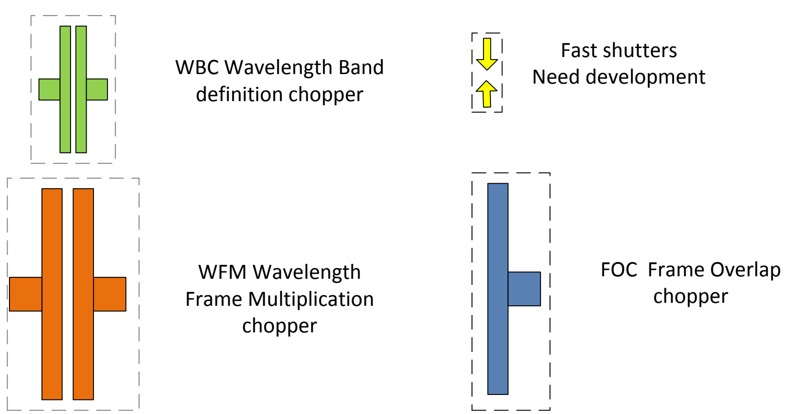
EUROPEAN SPALLATION SOURCE

1. The instrument shall be capable of measuring specular reflection on free liquid surfaces without moving the sample or detector between Qmin = 0.0035 Å-1 and Qmax = 0.44 Å-1. **Elliptical guide, Detector area, WBC1-2 choppers**
2. The instrument shall be capable of measuring specular reflection from solid samples in the range of Qmin = 0.005 and Qmax = 1Å-1 **Detector motion range, WBC1-2 choppers**
3. The instrument shall allow the illumination of horizontal sample areas between 1cm(x) x 1cm(l) and 4cm (w) x 8cm (l).**Width of guide, width of detector**
4. The instrument shall be capable of providing a minimum angular resolution of dθ = 0.01°. **Detector resolution**
5. The instrument shall have a minimum wavelength band Δλ of 7Å without frame-overlap. **Length, WBC1-2 choppers**
6. The instrument shall have a maximum wavelength resolution δλ/λ = 10.5% fwhm **Length, WBC1-2 choppers**
7. The instrument shall have a high resolution option with δλ/λ = 1.5% fwhm **WFM Choppers**
8. The instrument shall provide essential temperature-controlled sample environments for liquid and solid samples and mechanisms for changing samples automatically. **Sample environments**
9. The instrument shall be able to skip every second source pulse (pulse-skipping) **WBC3 chopper**
10. The instrument shall allow fast collimation changes for kinetic experiments **Three-slit system and Fast shutters**
11. The instrument shall be able to measure specular reflection from below the sample interface up to Q = 0.2Å-1 **Inverted beam configuration**
12. The instrument shall be able to polarise the full wavelength band of 7Å. **S-bender polariser, guide fields, flipper, electro magnet**
13. The instrument shall provide a GISANS option for horizontal samples that is capable of detecting length scales of up to 190nm with resolution δQy/Qy = 5%. **Collimation and detector distance, area and resolution**

FREIA LAYOUT

TOP VIEW





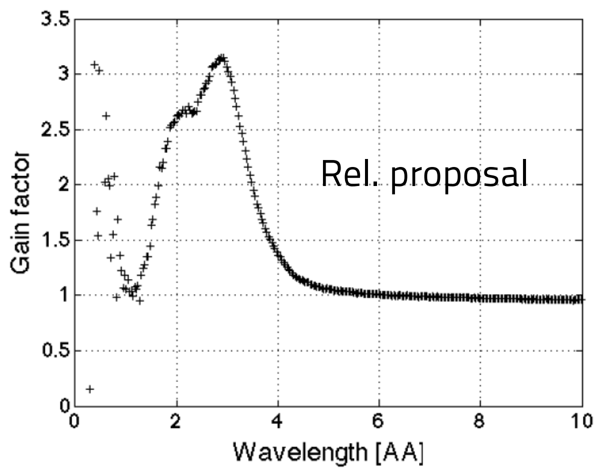
11 discs

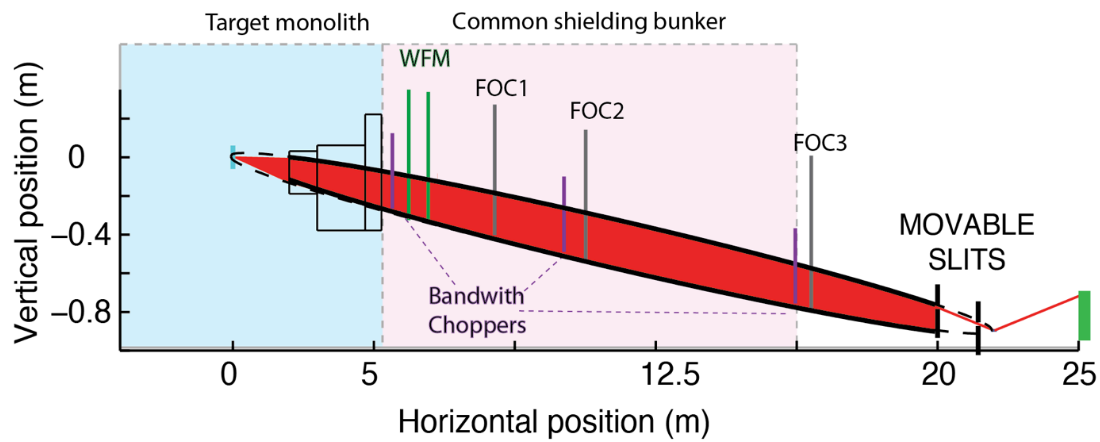
Chopper	Position/ m	Hz	Windows	Radius/ mm	Approximate Beam h/w mm	Description
WBC1	6.5±0.025	14/7	1	500	190/40	Double disc
WFM1	7.527	56	7	650	200/40	Co-rotating
WFM2	7.873	56	7	650	210/40	blind pair
FOC1	8.5	56	7	650	230/40	Single disk
WBC2	9±0.025	14/7	1	500	240/40	Double disc
FOC2	11.1	42	7	650	250/40	Single disk
WBC3	15.6±0.025	14/7	1	500	240/50	Double disc
FOC3	16	28	7	650	240/50	Single disk

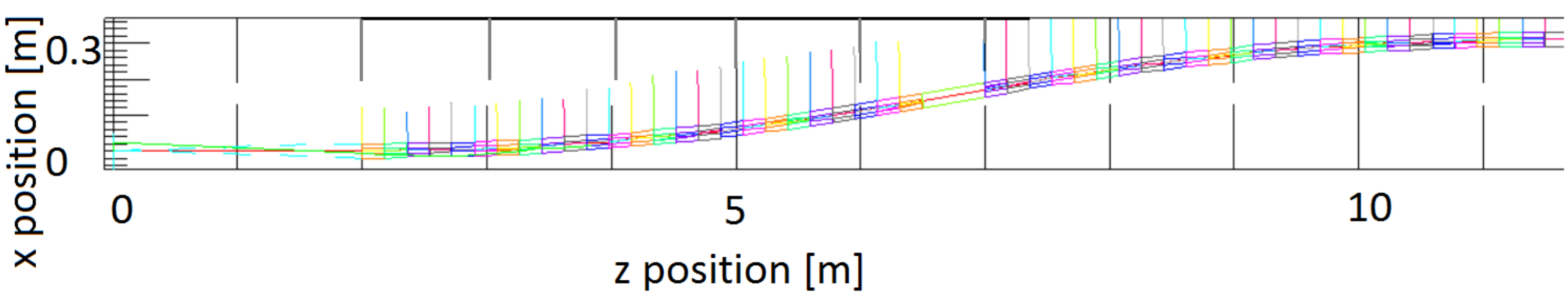
Guide

EUROPEAN SPALLATION SOURCE

- 4cm guide width
- 3-channel S-bender (4cm) with ROC = 70 m, 2 x 4.5m, m=5.4/3 – *height 19-26cm*
- 2 x out of LOS by 11.5m – Reflectometry is a background-limited technique! Aim to measure $R = 10^{-8}$
- Ellipse (b=25cm) with m = 3-6 needed for focusing/vertical divergence.

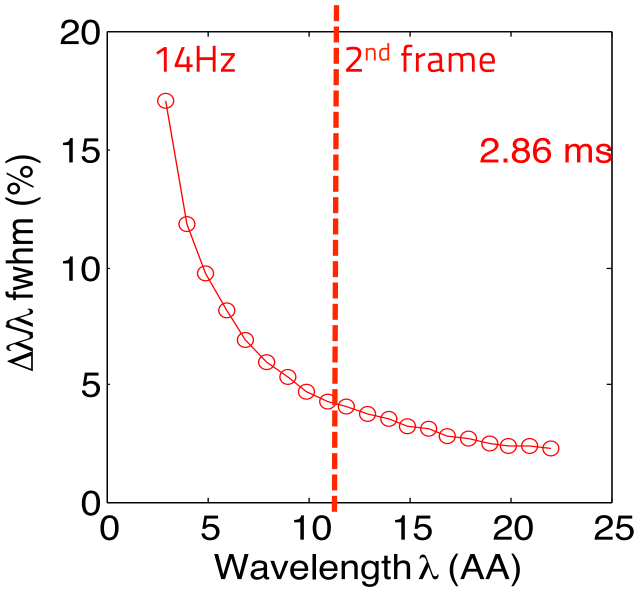




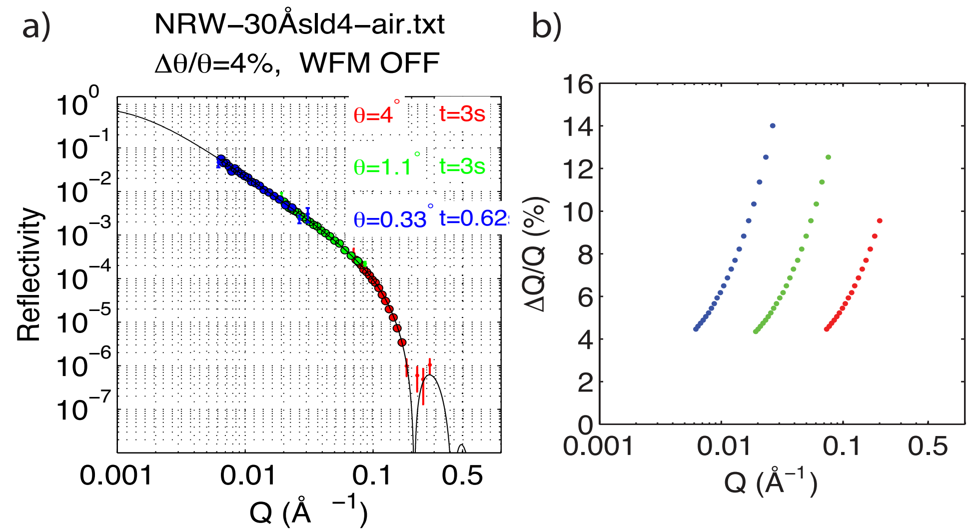


Bandwidth Choppers WBC 1-3

- Maximum bandwidth and flux: e.g 2.5Å < λ < 11.3 Å, dλ/λ = 2.6-12.5% (fwhm)

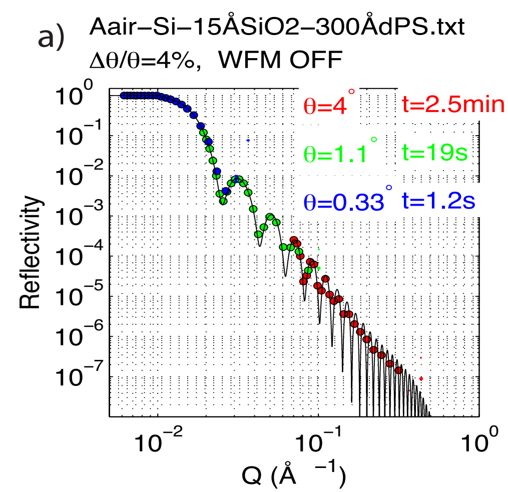


Resolution suitable for thin films only (<150Å):



- 3 WBC choppers at 6.5, 10m,15m
- counter-rotating, variable opening
- 14Hz (7Hz), full 2.86ms pulse
- min. d = 1m (motor clash with guide)
- WBC1-2 for selection of band
- WBC3 for pulse-skipping and higher resolution

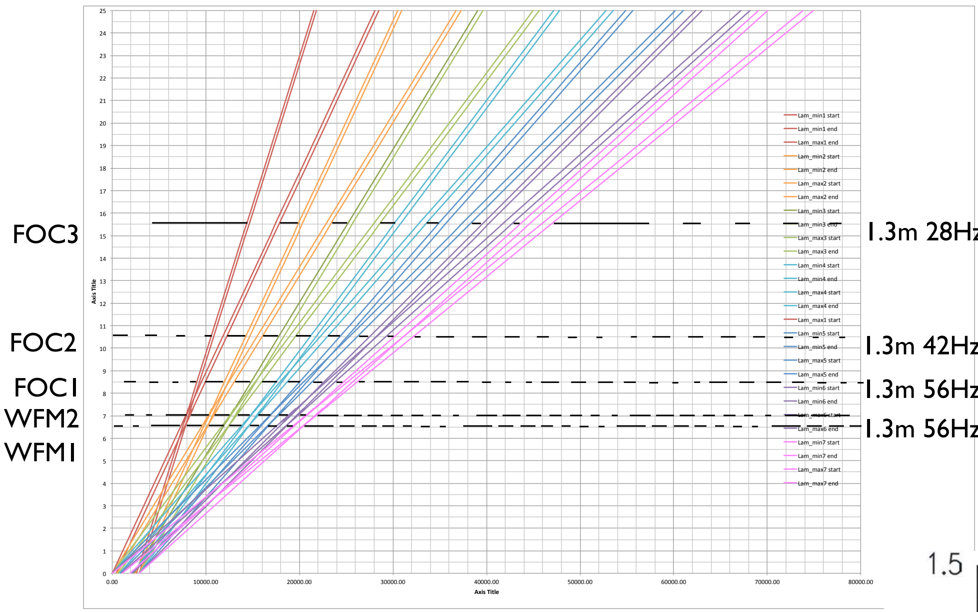
Resolution not sufficient for thicker films, e.g. polymers, multilayers, biosensors and GISANS.

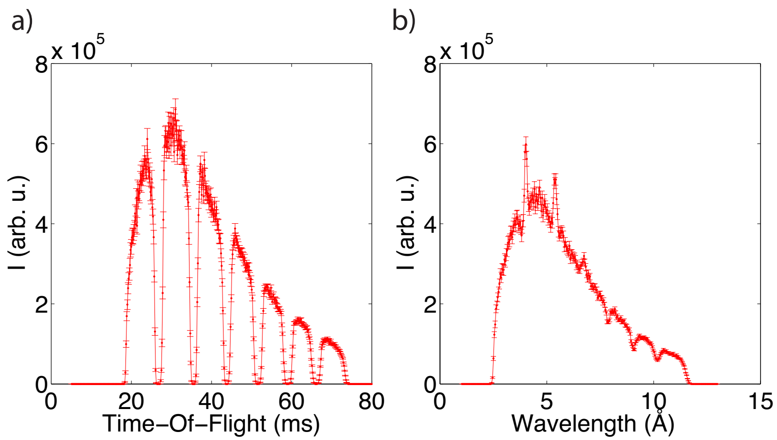


Need higher resolution for 50% of reflectometry science case + GISANS

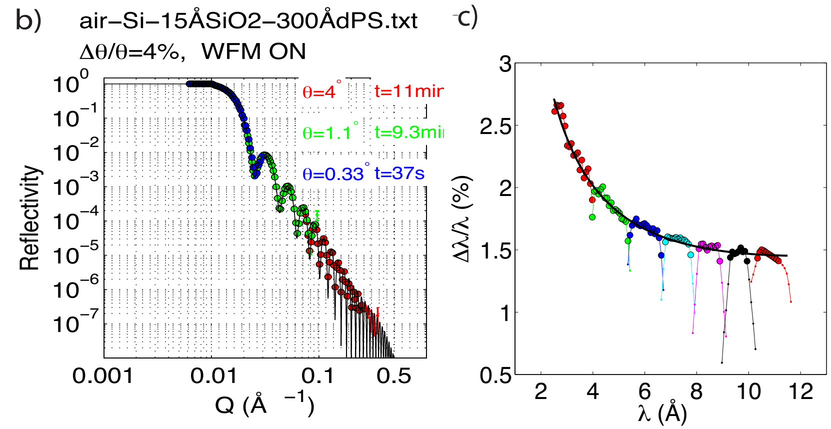
Wavelength Frame Multiplication WFM

7-fold WFM with $d\lambda / \lambda$ = 1.5% FWHM



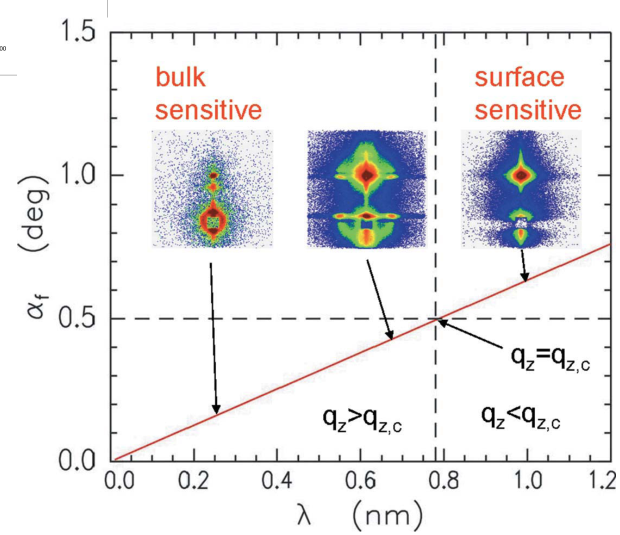


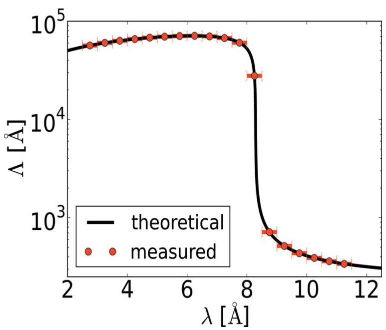
Resolution for thicker films (e.g. 300Å) – 50%



And controlling the penetration depth of GISANS

~30%





H. Frielinghaus, et al. Phys. Rev. E, 85, (2012), 041408.

P. Mueller-Buschbaum et al., J. Appl. Cryst. (2014). 47, 1228–1237

Collimation – all should work with polarised beam/guide field

EUROPEAN SPALLATION SOURCE

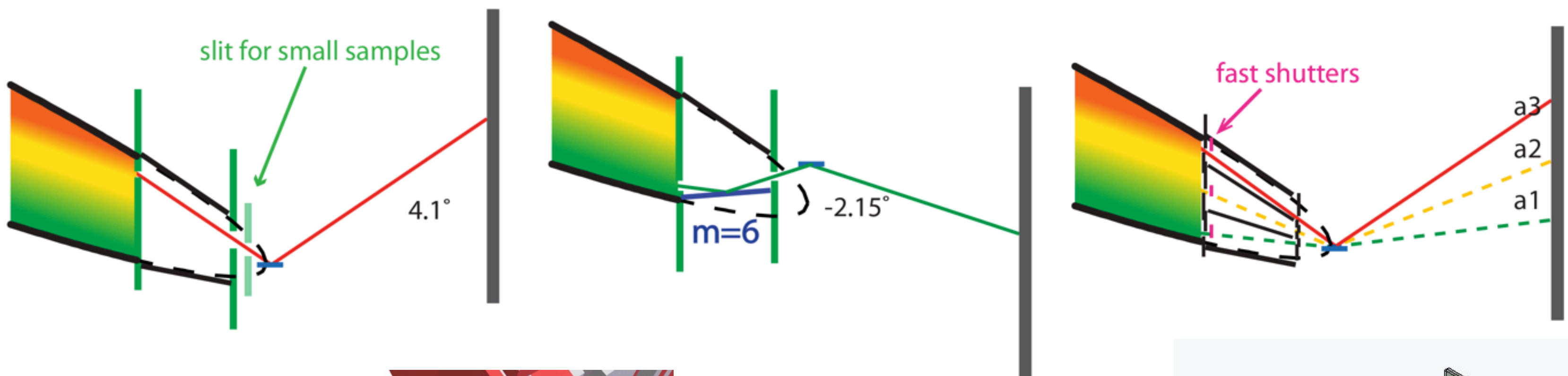
- Interchangeable 2m guide sections (20-22m from source) for :

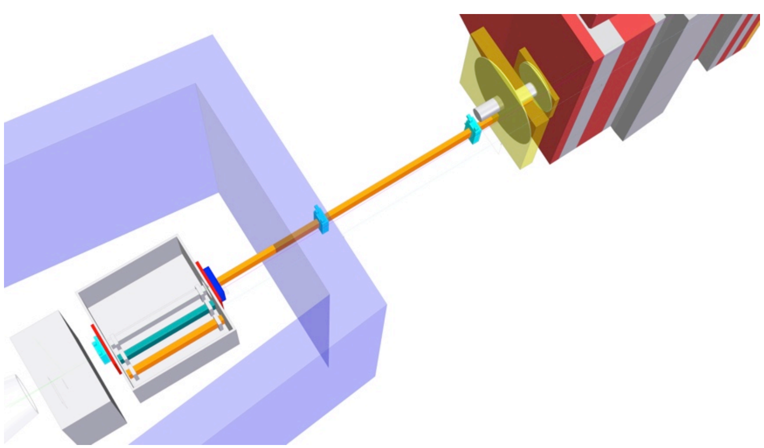
a) conventional slit collimation with extra slit for small samples (more efficient illumination)

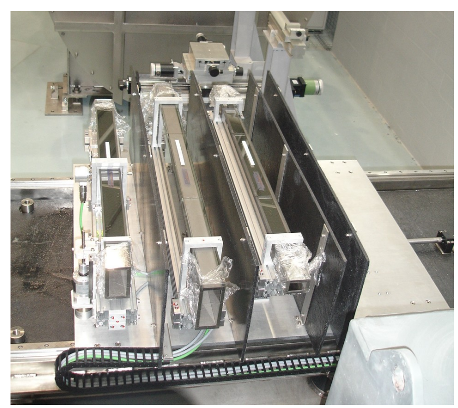
b) deflection mirror for inverted geometry (liquid-liquid interfaces, sample environments) 25%

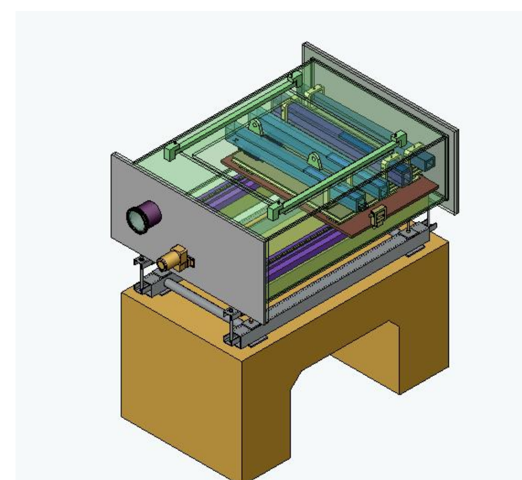
c) three-slit collimation + fast shutters for kinetics at the ms-s timescales 50%?

a) Conventional collimation b) Inverted geometry c) 3-slit collimation









e.g. collimation chamber on Platypus

Polarisation option for magnetic contrast:

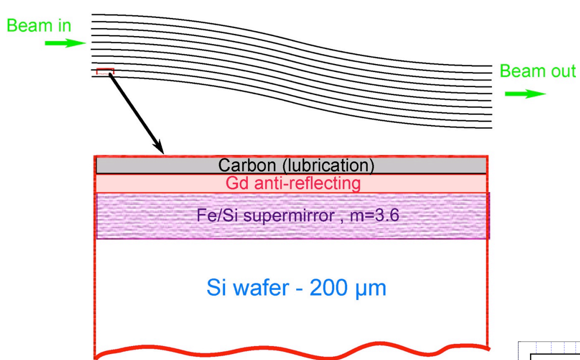
- 15% of experiments

Silicon-bender before first collimation slit:

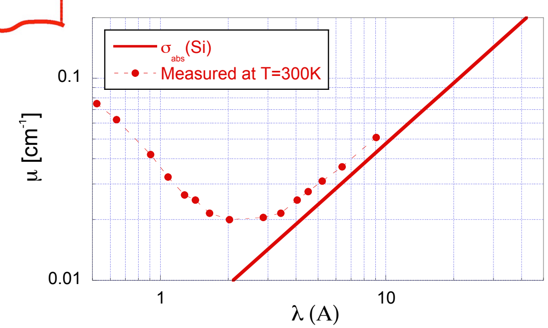
m=3.6 Fe/Si + Gd/Si. For 2.5-11.4 Å, a 6cm length,150µm Si

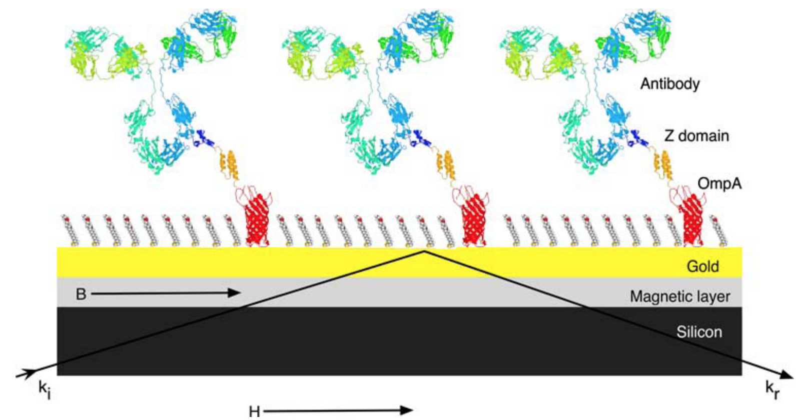
- 10cm gap reserved before collimation section
- guide field/spin-flipper 2m before sample
- 16cm,(h) x 4cm (w)

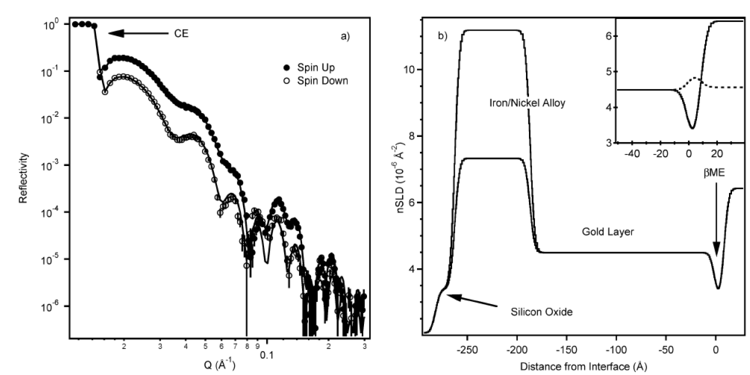
Needed for sample in which solvent contrast changes are not possible, e.g. weakly-bound antibodies in biosensor arrays.



Broad polarised wavelength band needed for kinetic PNR experiments possible at ESS.







Krist, Th., Peters, J., Shimizu, H.M., Suzuki, J., Oku, T., Physica B 356 (2005), 197-200.

Three-slit collimation and fast shutters (~50%)

- Three pairs of adjustable precision slits at 20m and 22m to minimize movement time – this needs development as conventional solutions are not applicable.

Shutter options possible for kinetics at different timescales:

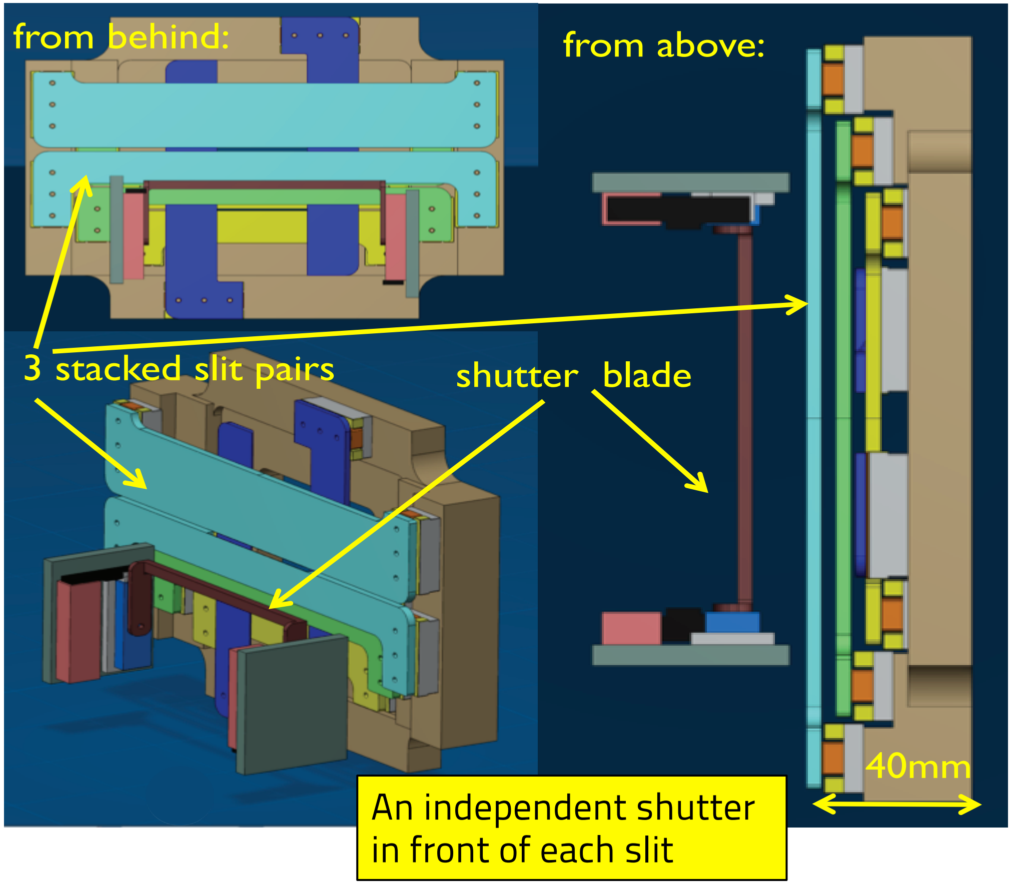
A) for fast (<10s) kinetics:
3 linear shutters + 3-channel collimating guide (Option 3)

B) 30s-2min kinetics:

three slits - open/close pre-positioned slits (Option 2)

D) > 2 min kinetics:

– scan with conventional single slit pair (Option 1)



Sample area and sample environments

EUROPEAN SPALLATION SOURCE

Large sample area to house:

- sample stack up to at least 750kg
- coarse height-stage ± 100mm – smaller stage ± 10mm
- translation table 500mm
- gas manifold
- kinematic mounting
- laser for aligning solid samples
- laser interferometer for aligning liquids
- shielding to minimize sample background
- liquid handling manifolds (HPLC, baths)

INTEGRATION and COMMISSIONING of sample environments is essential before user program can start.

ALL SE items require some degree of customisation or need to be designed in-house.

SAMPLE ENVIRONMENTS:

a) air-liquid adsorption troughs
b) Langmuir trough
c) set of solid-liquid flow cells
d) set of liquid-liquid cells
e) HPLC pump + syringe pump
f) water bath
g) overflowing cylinder
h) electrochemical cells

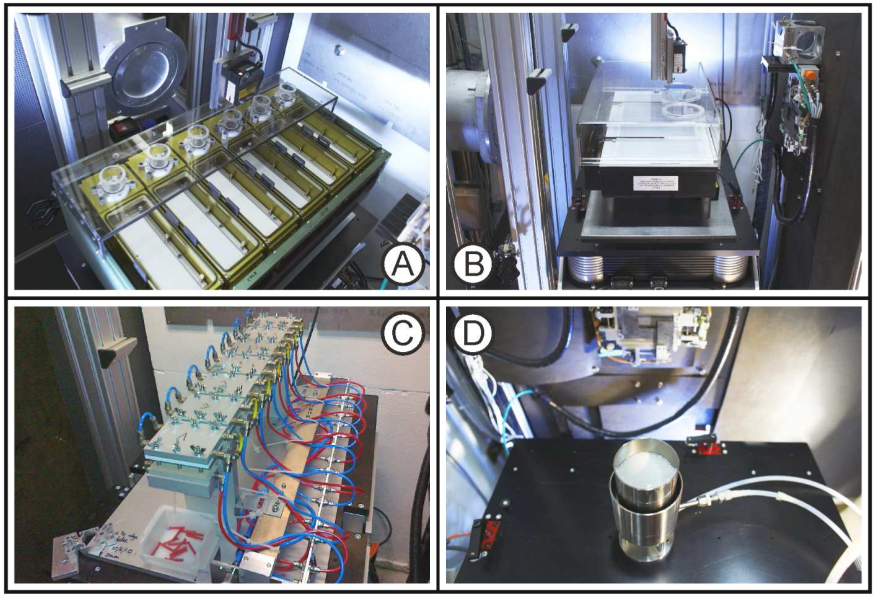
From ESS SE pool:

a) humidity chamber
b) electromagnet
c) basic vacuum chamber
d) furnace
e) rheometer
f) potentiostat (to be shared with ESTIA)

Sample changes

2-10s per sample = 1 minute per 6 air-liquid chambers!!!

- **Cleaning and reassembling sample cells takes 15-20 min. each time**
- *Need multiple sets of for user program*
- Large translation table for many samples
- Sample preparation area at instrument
- Lab for cleaning close by
- remote contrast changes (HPLC pump)
- remote sample injections for kinetics (syringe pump)



AUTOMATION WILL BE ESSENTIAL TO MACTH ESS BEAM POWER

NEED to develop robotic changers for both liquid and solid samples in the long term.

Detector and housing

FREIA will not measure high-Q data up to 2$Å^{-1}$ as proposed originally, due to the fixed detector distances.

EUROPEAN SPALLATION SOURCE

REF: 0.3m x 0.3m area detector 3m from sample position in evacuated tank (snout)

- > measure Q = 1$Å^{-1}$ (11.5°) on solid samples (#2)

The detector height is defined by need to detect reflections 0.25°- 4.1° without moving (#1)

The detector width is defined by horizontal beam divergence (±1.5°) for 4cm wide samples (#3)

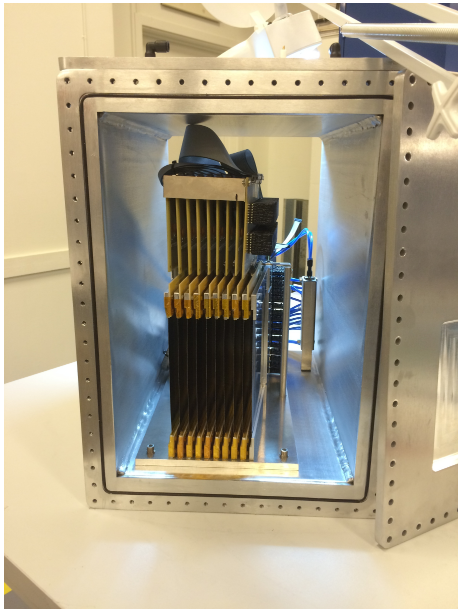
GISANS: 1m^2 area detector at 8 m from sample position in evacuated tank

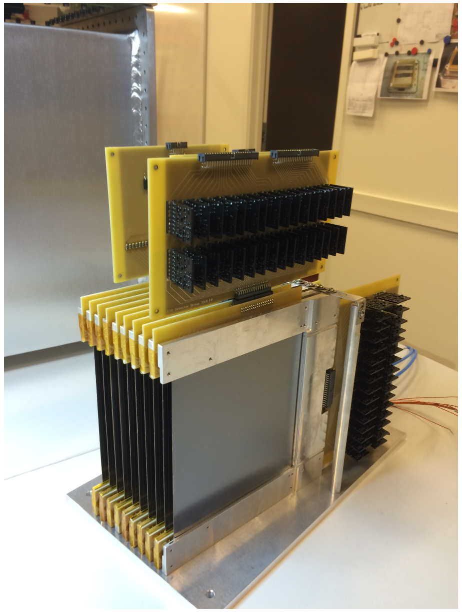
GISANS detector position and size are determined by the required Q_y range (3 x 10^{-3}- 0.25 $Å^{-1}$)

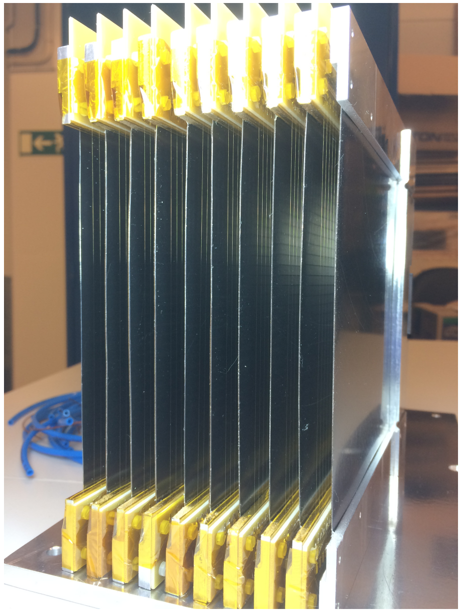
Both based on ^{10}B Multiblade detector developed at ESS (Francesco Piscitelli)

Cylindrical arrangement of blades -> fixed detector distance, radial vertical translation

Resolution requirements: 0.5mm vertically and 2.5mm horizontally.







STAP Priorities:

The scientific priorities for realizing the instrument's scientific scope are as follows in order of priority:

I. High-intensity specular and off-specular reflectivity

-> thin films (>150Å) at liquid and solid interfaces with sub-min. kinetics

II. Sample environment

III. High, medium and low resolution options for specular and off-specular reflectivity due to the wide range of length scales (WFM choppers)

IV. Lateral structures over a range of length scales (GI-SANS)

V. Free liquid/liquid interfaces and rheometry (inverted beam configuration)

VI. Kinetic measurements over a broad dynamic range requiring the fast shutter system.

VII. Magnetic reference layers (polarized neutron reflectivity)

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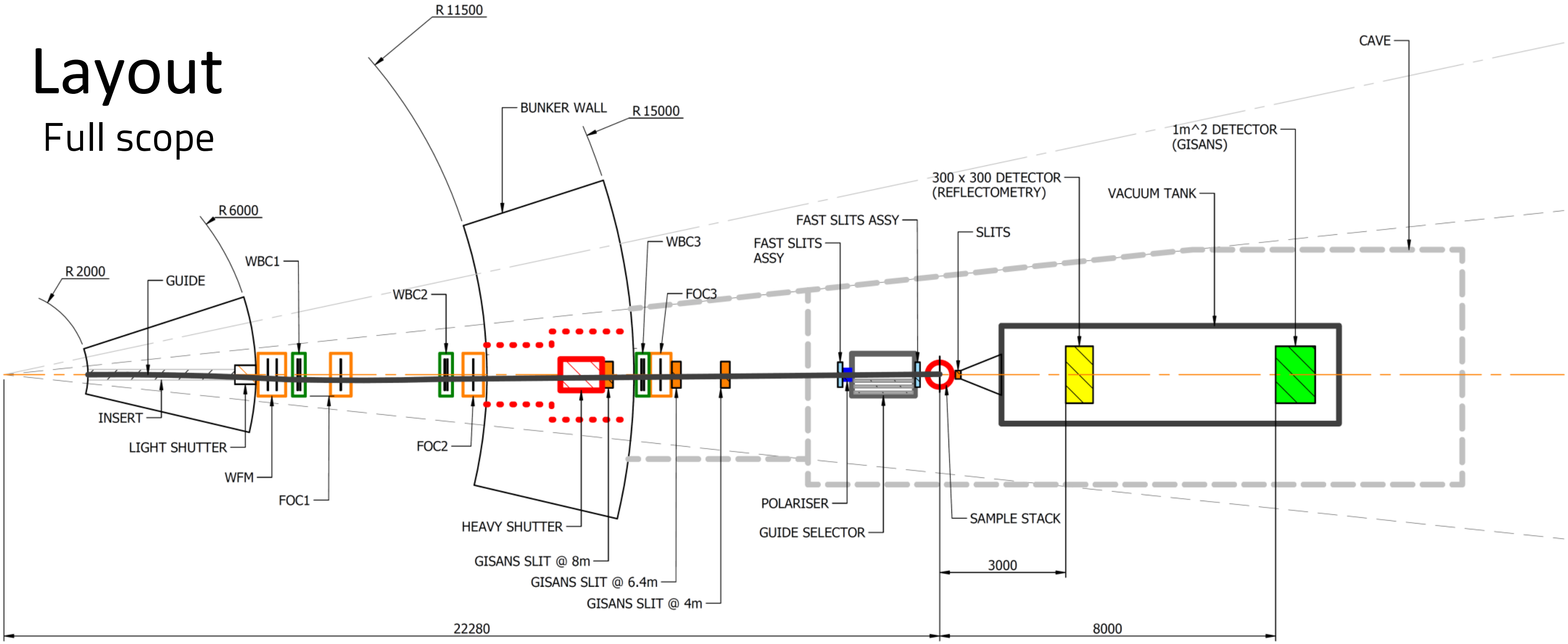
"The STAP considers the minimum acceptable scope for day 1 in order for the ESS to go ahead with the FREIA instrument is the first 3 items on the list immediately above. This will allow conventional specular reflectivity experiments to be carried out for the majority of the science case."

Layout

Full scope



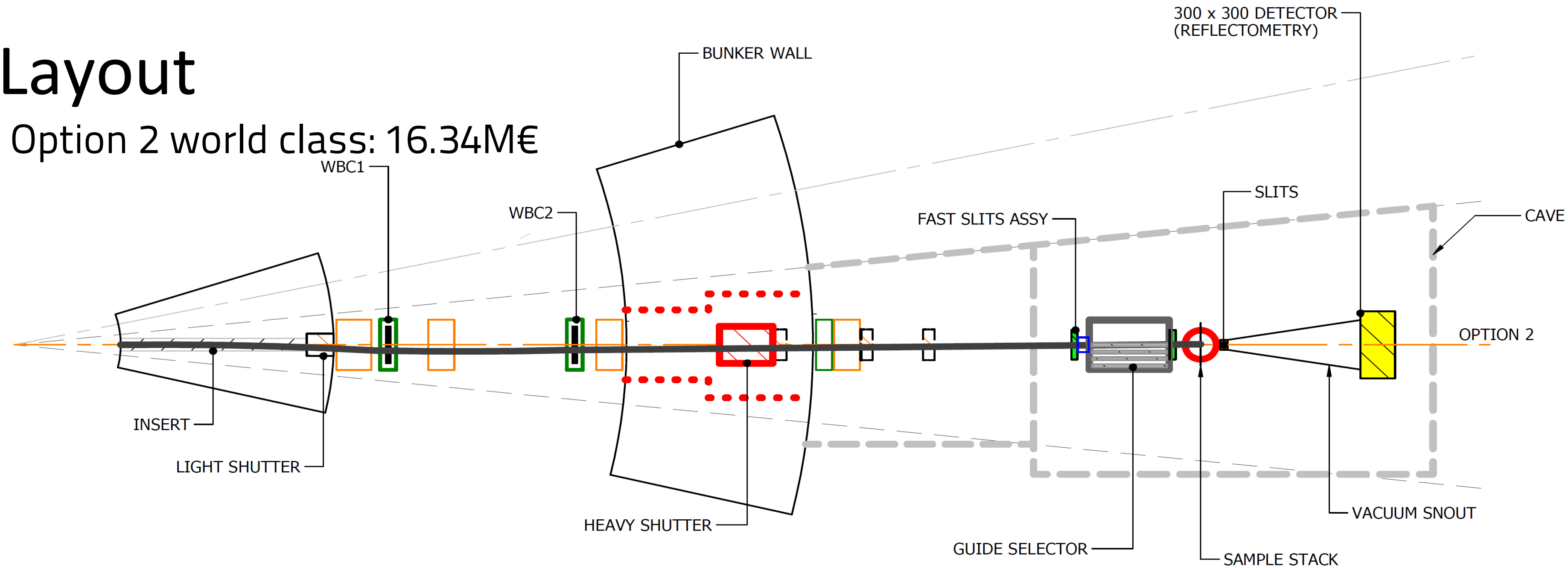
STAP report: Full configuration meets most of Reflectometry science case (~85%) at world-leading level
+ complementary GISANS (d~200nm) to what can be achieved on a dedicated instrument.

11 discs

Chopper	Position/ m	Hz	Windows	Radius/ mm	Approximate Beam h/w mm	Description
WBC1	6.5±0.025	14/7	1	500	190/40	Double disc
WFM1	7.527	56	7	650	200/40	Co-rotating
WFM2	7.873	56	7	650	210/40	blind pair
FOC1	8.5	56	7	650	230/40	Single disk
WBC2	9±0.025	14/7	1	500	240/40	Double disc
FOC2	11.1	42	7	650	250/40	Single disk
WBC3	15.6±0.025	14/7	1	500	240/50	Double disc
FOC3	16	28	7	650	240/50	Single disk

Layout

Option 2 world class: 16.34M€



This configuration meets the minimum requirements defined by the STAP as necessary for day 1.

This option will meet most of Reflectometry science case (75%)

This option is easily upgradable to polarised NR and the full sample environment. GISANS upgrade would involve cave rebuild.

11 discs

Chopper	Position/ m	Hz	Windows	Radius/ mm	Approximate Beam h/w mm	Description
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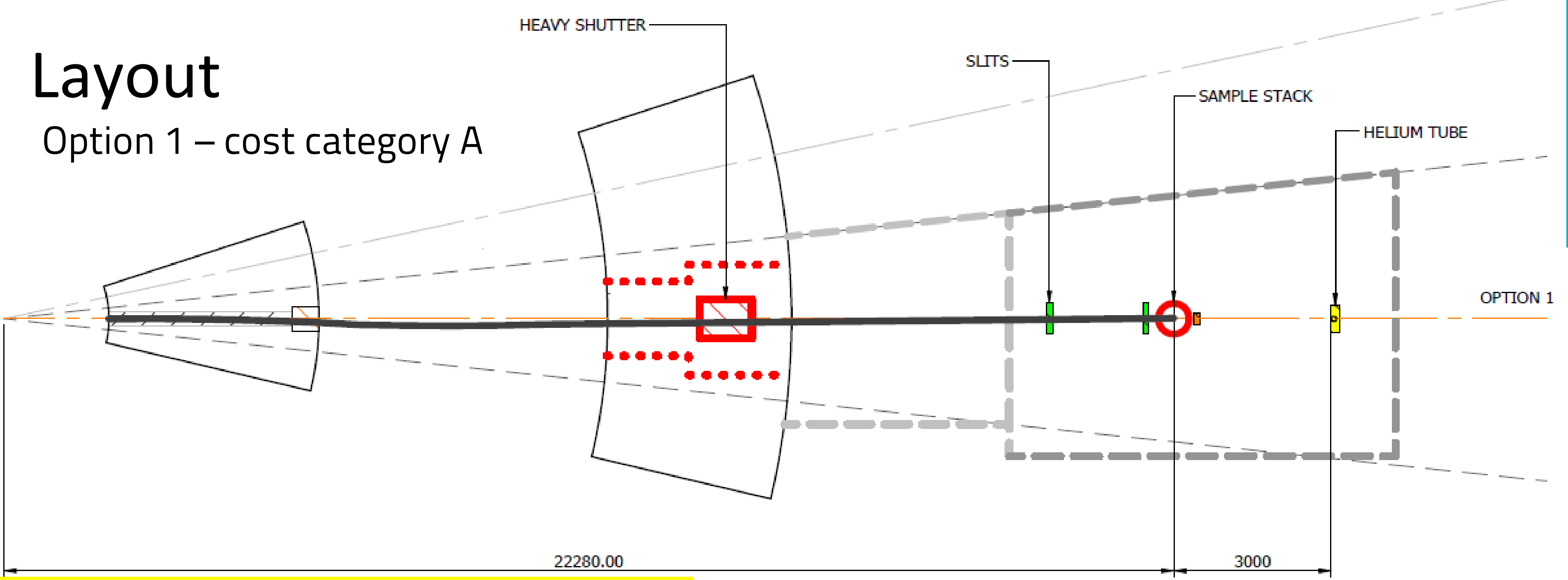
Layout

Option 1 – cost category A



It is not possible to build a functional instrument within cost category A.

A *minimal functional configuration that can measure reflectivity data would need:*

Bandwidth/frame overlap choppers (1354k€)

A sample stack (372k€)

Control hutch (178k€)

This configuration would not satisfy the STAP priorities for day 1. This option is not upgradable to Option 2 or 3.

Measurements on liquids surfaces without moving sample/detector (#1) **STAP Priorities**

The wavelength resolution (#7)

Sample environment (#8)

Pulse-skipping (#9)

Fast collimation changes for kinetic experiments (#10)

Specular reflection from below the sample interface (#11)

Beam polarisation (#12)

GISANS (#13)

Further considerations

EUROPEAN SPALLATION SOURCE

- WFM choppers need development and time for commissioning before user program – too late to start this after 2019.
- Later installation WFM choppers would lead to a massive disruption to user program on several instruments
- Sample environments are essential for early science success – only the bare minimum set is included even in the full scope option 3.
- Development of robotic sample changers using external/operational funding will be essential for a successful user program that is able to benefit from the source performance
- Cave rebuild for GISANS is feasible but the upgrade is expensive. Although dedicated instrument would perform better, need to keep this option on the table at least until this is decided.

Project

EUROPEAN SPALLATION SOURCE

- Neutron Instrument proposal SAC May 2014 ✓

  Scientific case for Neutron Instrument, scientific relevance, impact and usage. Conceptual design and a preliminary costing.

- Tollgate 1 STC October 2014 ✓

  Proposal accepted into ESS baseline

- Phase 0: January 2015 – April 2016 ✓
- Phase 1 May 2016 – 1 July 2017

FREIA is at a very early stage of preliminary design

EUROPEAN
SPALLATION
SOURCE

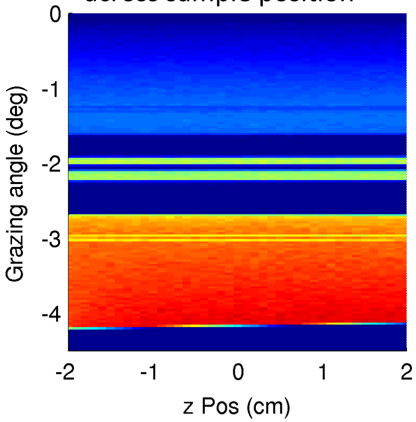
www.europeanspallationsource.se

May 2016

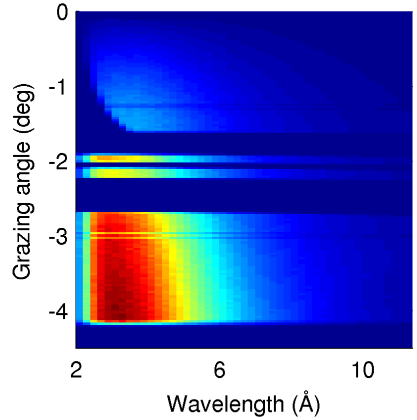
Beam properties

TDR moderator – multiply intensities with ~3.5 for updated moderator + S-bend

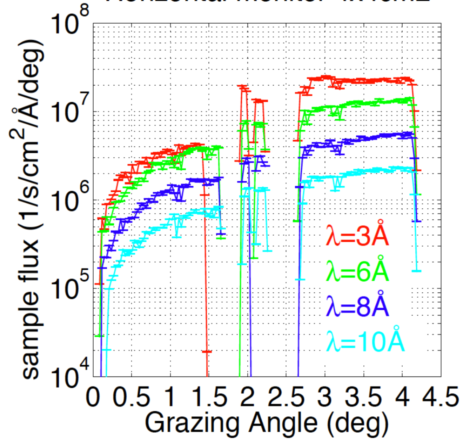
Vertical divergence across sample position



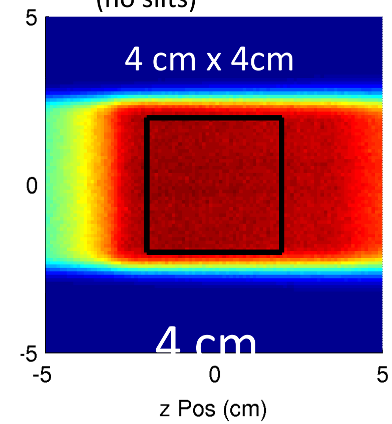
Wavelength vs divergence over 4cm x 4cm



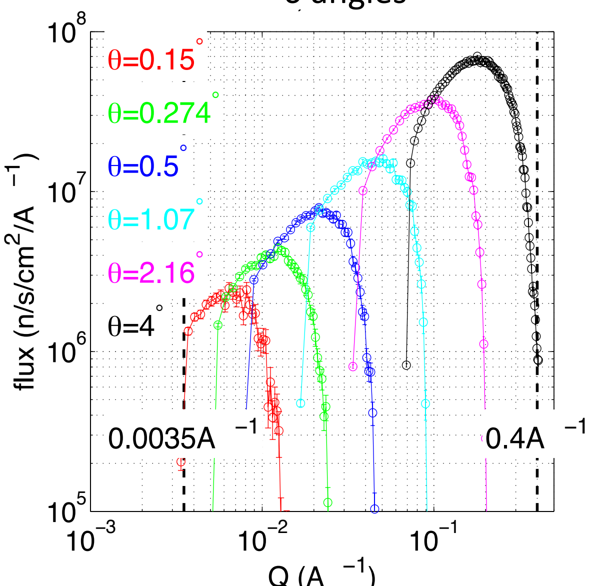
With chopper gaps
Horizontal monitor 4x4cm2



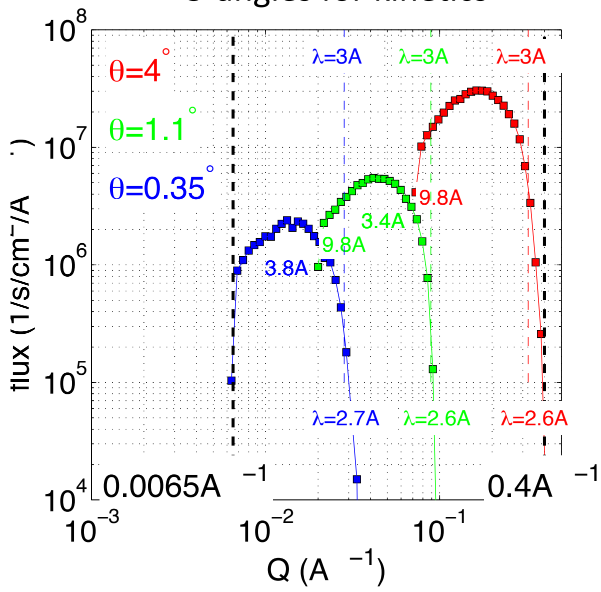
Full beam footprint (no slits)



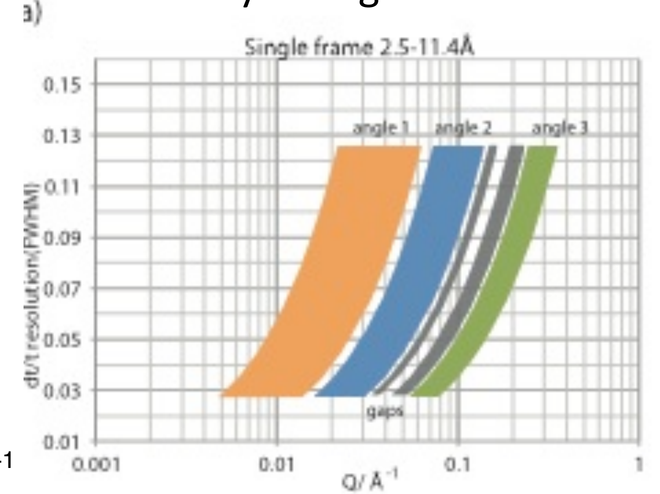
6 angles



3-angles for kinetics



flexibility in angle selection



Beam properties

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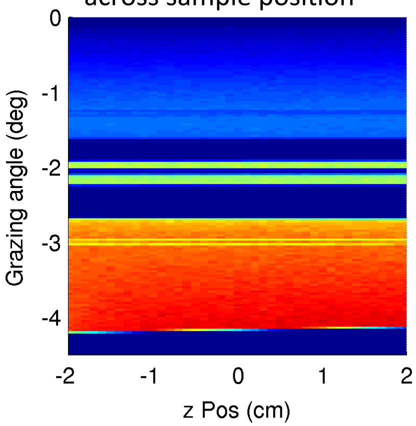

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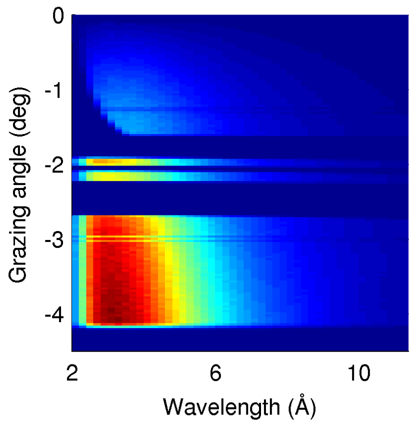
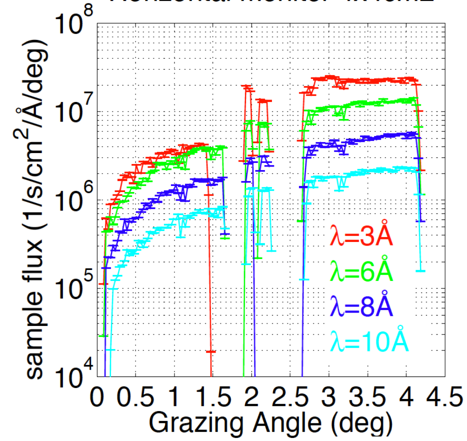


With chopper gaps
Horizontal monitor 4x4cm2



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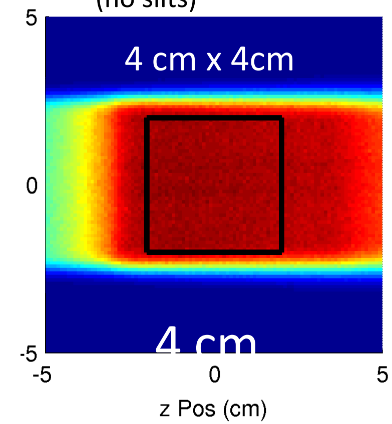

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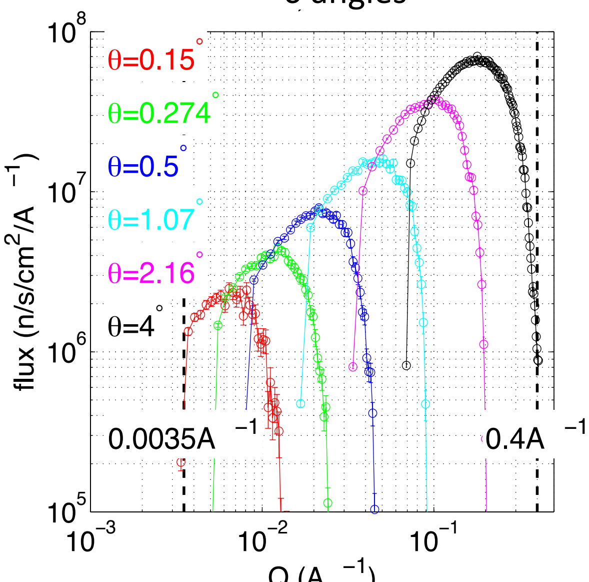

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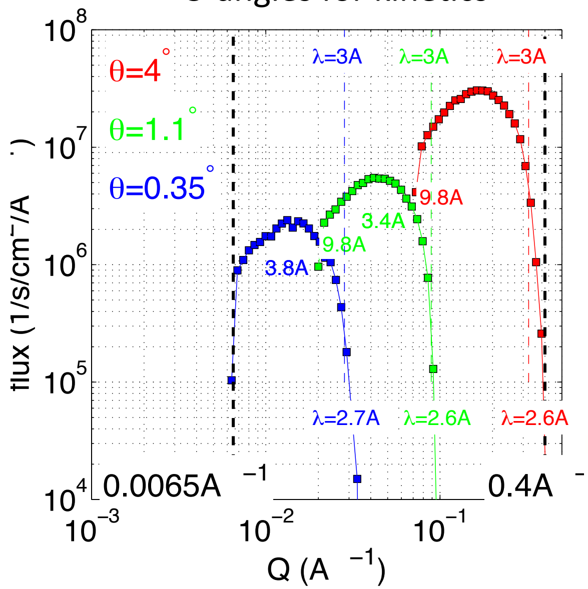

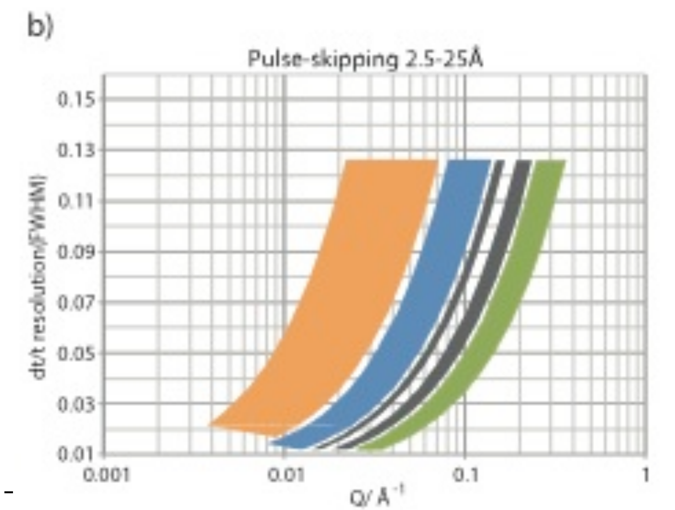

two angles – pulse skipping