Freia, (Frejya, Freyia, Frøya, Frøjya, and Freja) in Old Norse the "Lady", one of the Vanir gods, rules over the heavenly afterlife field Fólkvangr and there receives half of those that die in battle.

Freyja is also the goddess of love, beauty, fertility, gold, witchcraft, war, death…and cats

FREIA – a reflectometer for kinetics and liquid surfaces

H.Wacklin (ESS) & Anette Vickery (NBI)

IKON5

Lund, 2013-09-25 & 26

EUROPEAN SPALLATION SOURCE

FREIA:
Fast Reflectometer for Extended Interfacial Analysis

- Science Drivers
  - A broad spectrum of science:
  - thin film deposition/growth, structure and phase behavior
  - self-assembly and interactions of soft matter at interfaces
  - structure and interactions in biological materials and membranes
  - (thin film magnetism, interfacial effects)
  - gas-liquid, liquid-liquid, solid-liquid, solid-gas interfaces
  - (towards 2D-3D thin film structure)

- Technical Challenges and Opportunities
  - optimisation of flux and dynamic q-range for time-resolved studies
  - a range of sample sizes (l =5-40mm)
  - flexible resolution options from $d\lambda/\lambda$ = 2-20%
  - horizontal sample geometry for free liquid surfaces (~25% users, up to 55% on optimised instruments e.g. FIGARO).

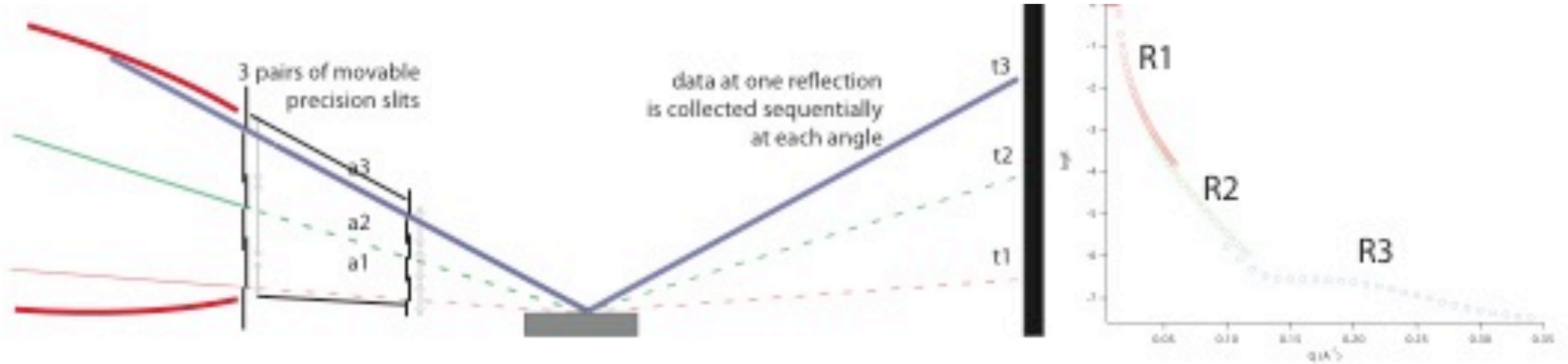
Science case

- Time-resolved reflectometry for interfacial kinetics primarily in
  - self-assembly of surfactants, polymers and proteins
  - rearrangement processes in thin films: e.g. lipid flip-flop, polymer interdiffusion, annealing effects in e.g. photovoltaic films
  - solvent penetration and drying/exchange effects
  - switchable materials (chemically, electrically, light driven)
  - surface reactions on the colloidal time scale; e.g. enzyme catalysis, receptor-ligand binding, drug-target interactions, surface functionalisation etc. etc. etc.
- Deuteration give unique contrast not available using other techniques (e.g. AFM, fluorescence methods, QCM-D, SPR, DPI…)
- Requirement: time-resolution in the range of ms-s-min with a dynamic q-range that allows structural analysis - unique to tof-reflectometry (cf XRR and ellipsometry, which measure a single q-point at a time).
- Challenge: typically need to reconfigure instrument 2-3 times for different angles of incidence to measure whole Q-range
- thin films on liquid surfaces can easily be disturbed by mechanical motion

The FREIA Solution

- Ven 2009: two guides?
- Lars van Moos, K.Lefmann 2011 - elliptical guide

- Deliver a broad angular divergence focused onto the sample surface
  - no need to move sample
  - angle selection by slits

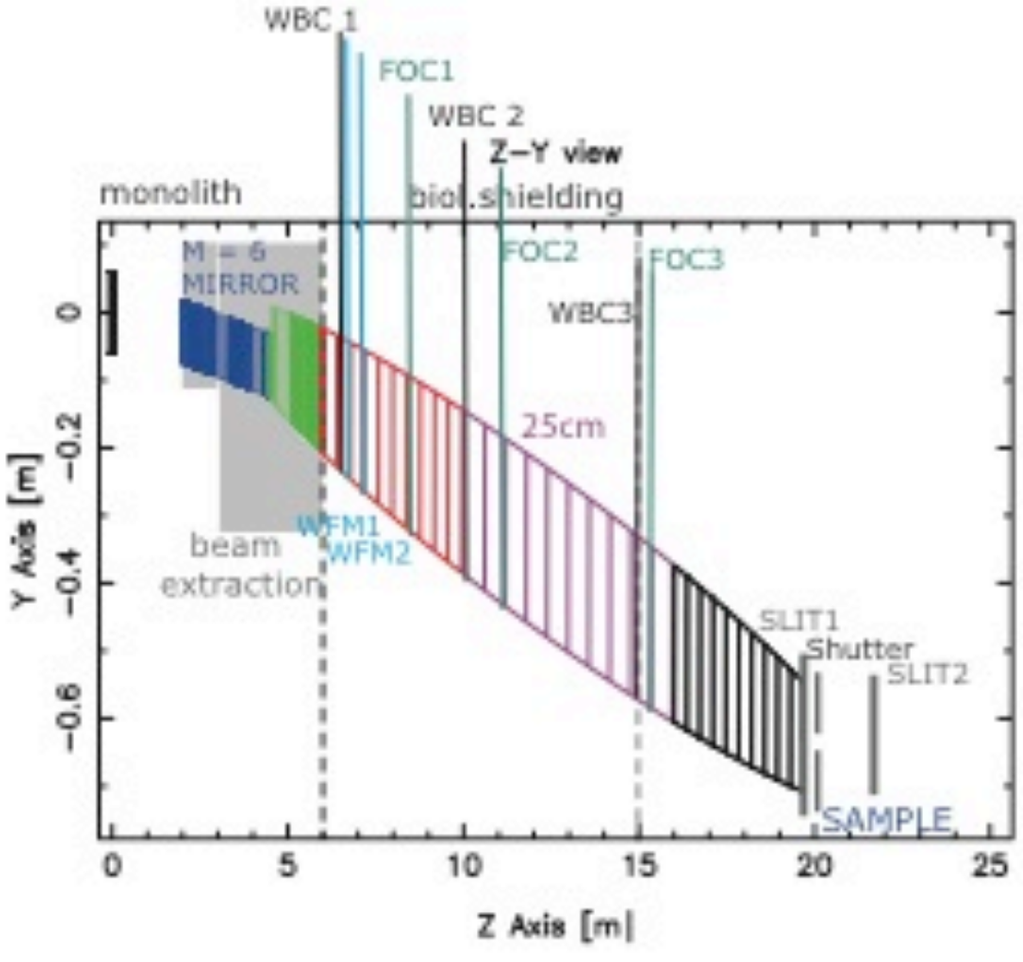


- Inclined guide to access horizontal surfaces
- Angular range for Q-range for liquids ($0.005 < Q < 0.4 Å^{-1}$) (bkg ~10^{-6})
- Low resolution mode for high flux measurements of fast kinetics
- Higher resolution mode for thicker structures/stationary measurements
- Higher q-values can be accessed by rotation of sample

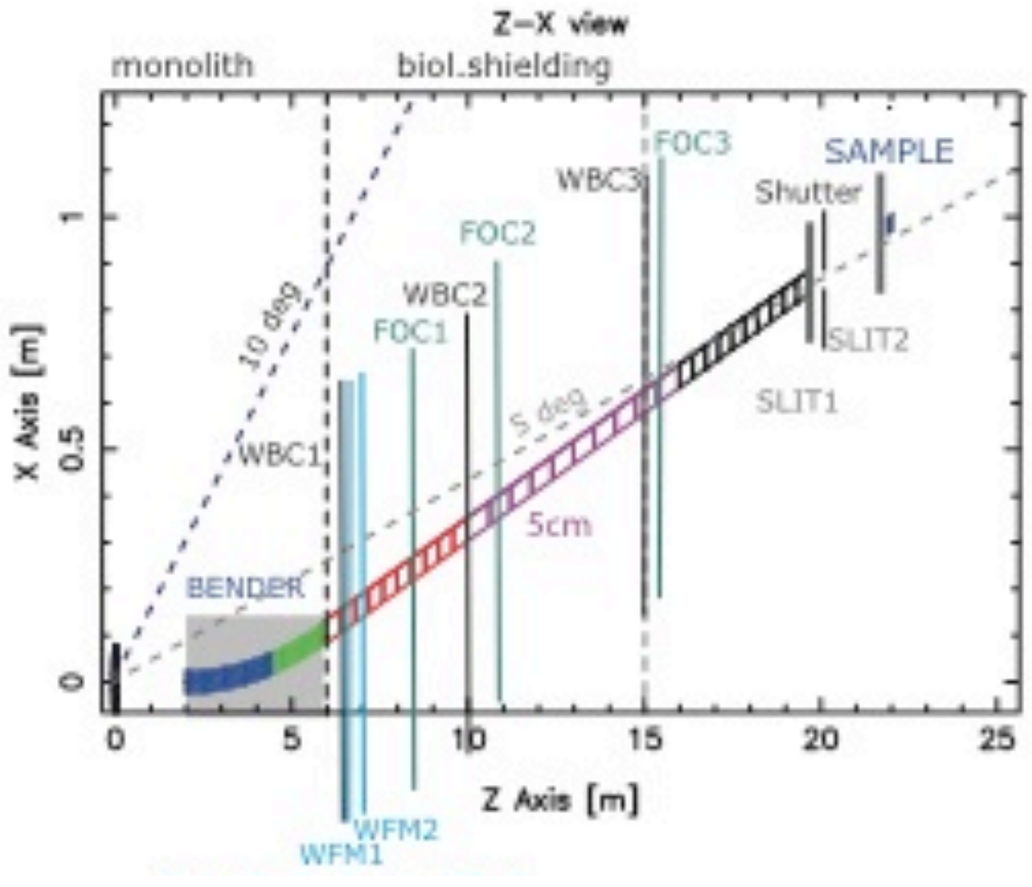
Instrument layout

- 25m instrument, elliptical guide inclined at -2°, transported divergence 4°
- m=6 mirror (-1°) deflects beam into guide, 4m bender ROC=56m (tbc)
- sample at 22m from moderator, 2m collimation, detector distance 1-3m

side view

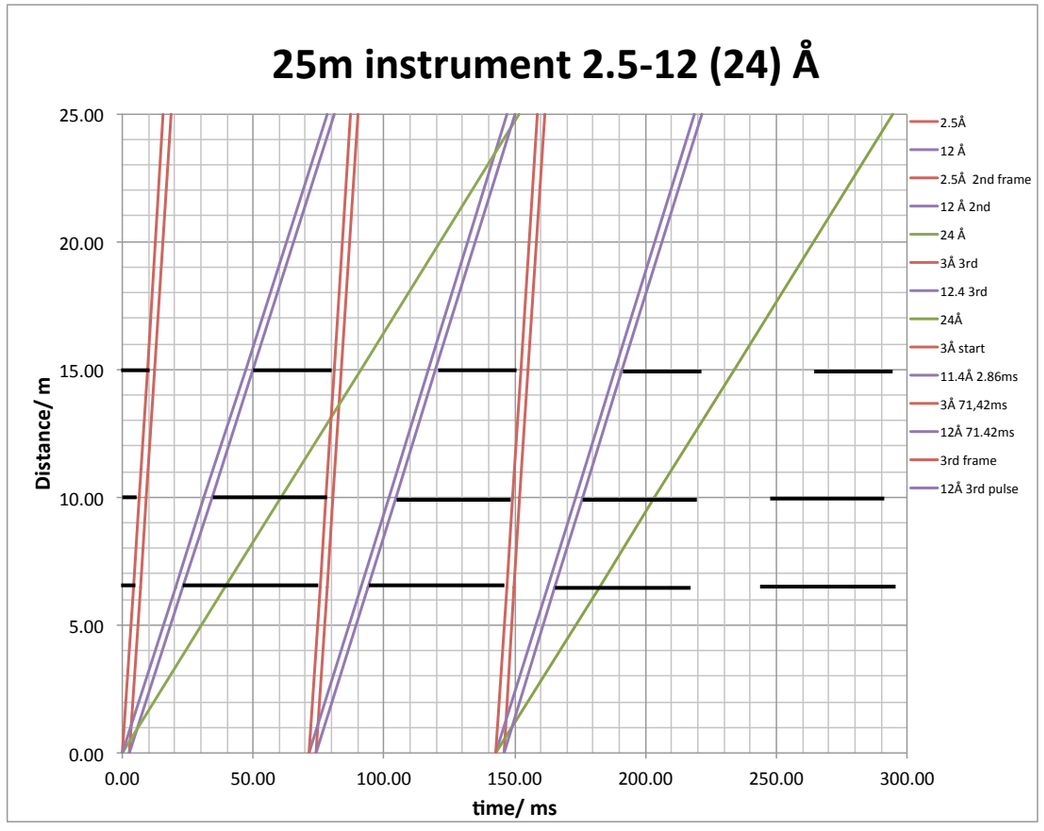


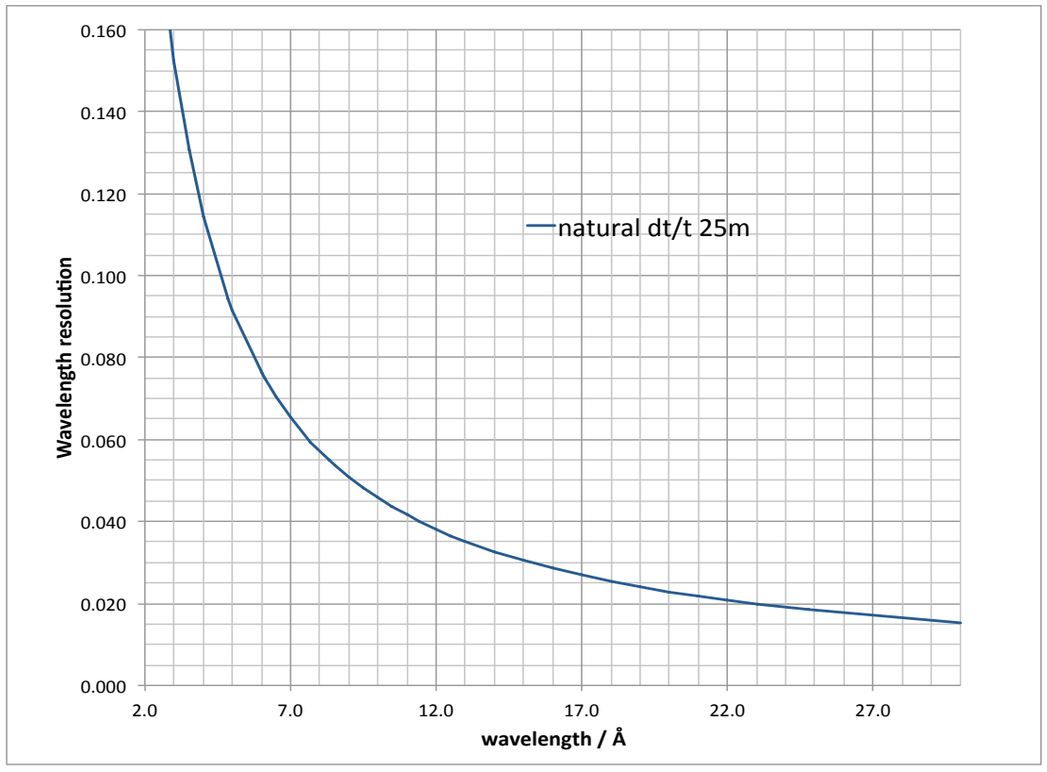
top view



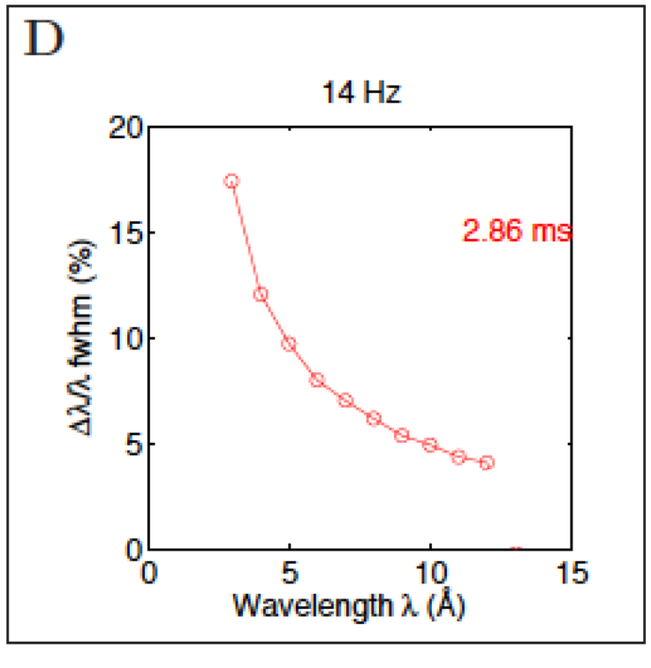
Bandwidth, resolution, etc.

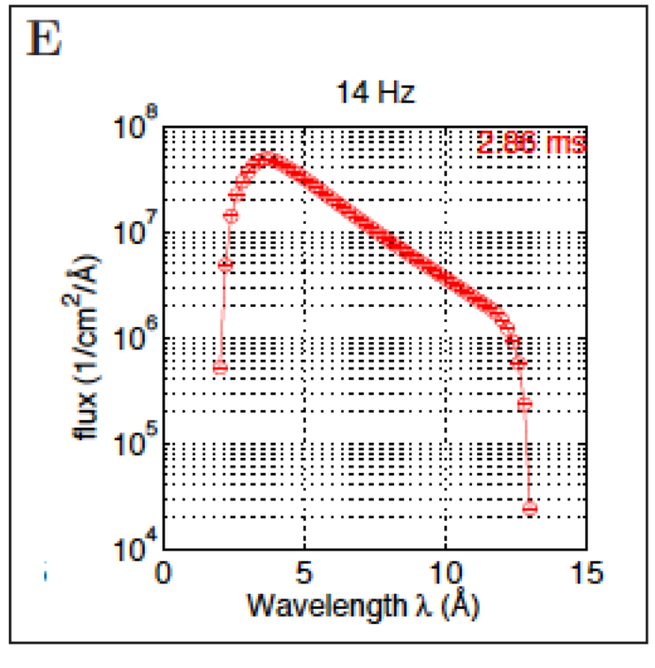
- Aim to use natural wavelength resolution and maximum bandwidth for high flux
- e.g 2.5Å < λ < 12Å, $d\lambda/\lambda$ = 3.8-18.3%





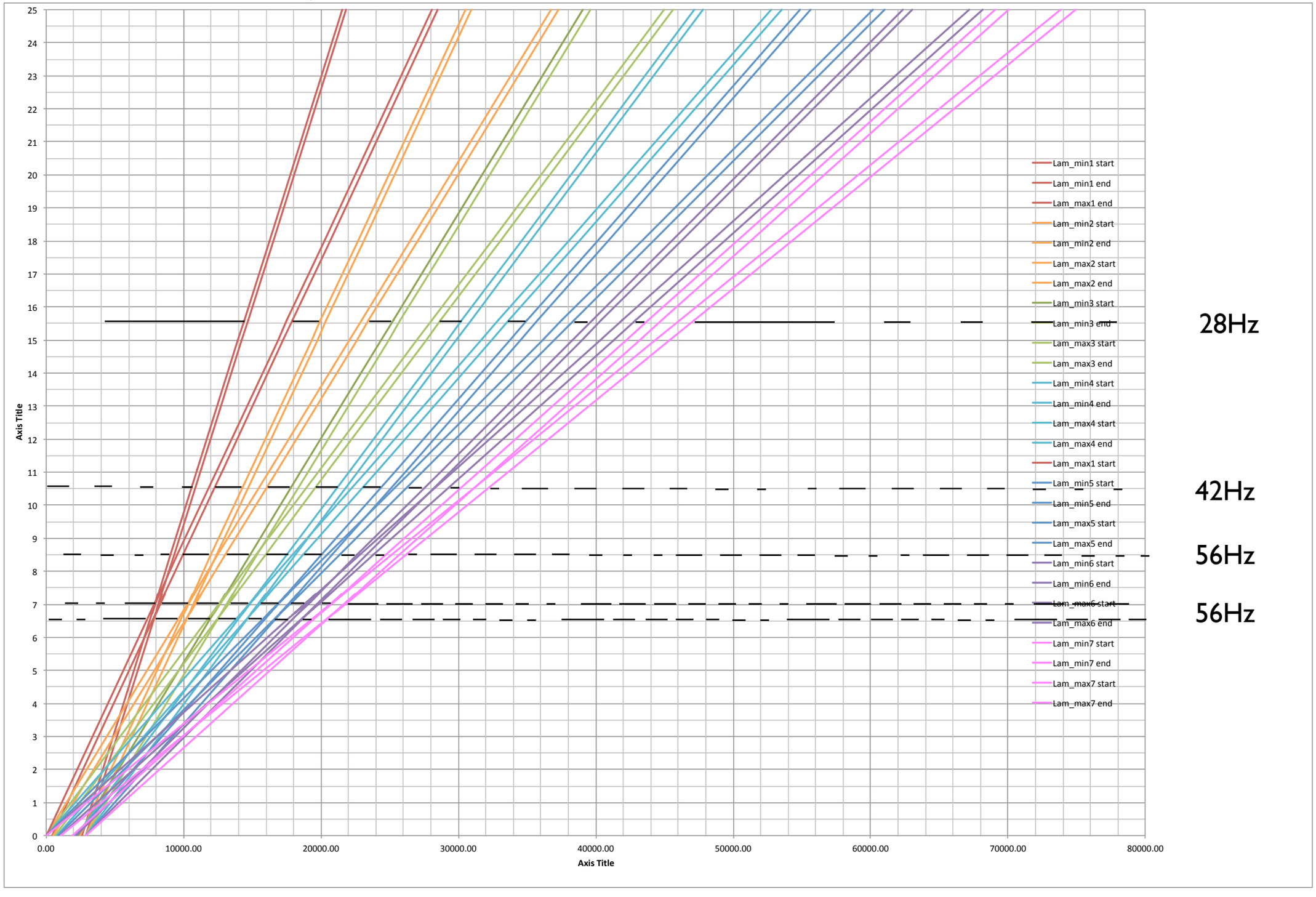
- 3 wavelength choppers at 6.5, 10m and 15m, 14Hz/7Hz
- 2.86ms
- Counter-rotating pairs with variable opening.





Bandwidth, resolution, etc.

- WFM challenging for 9.5Å bandwidth and 5cm guide, but possible (just):
- 7-fold WFM with $d\lambda/\lambda = 2\%$



FOC3 28Hz

FOC2 42Hz

FOC1 56Hz

WFM2 56Hz

WFM1

Bandwidth, resolution, etc.

- WFM challenging for 9.5Å bandwidth and 5cm guide, but possible (just):
- 7-fold WFM with $d\lambda/\lambda = 2\%$

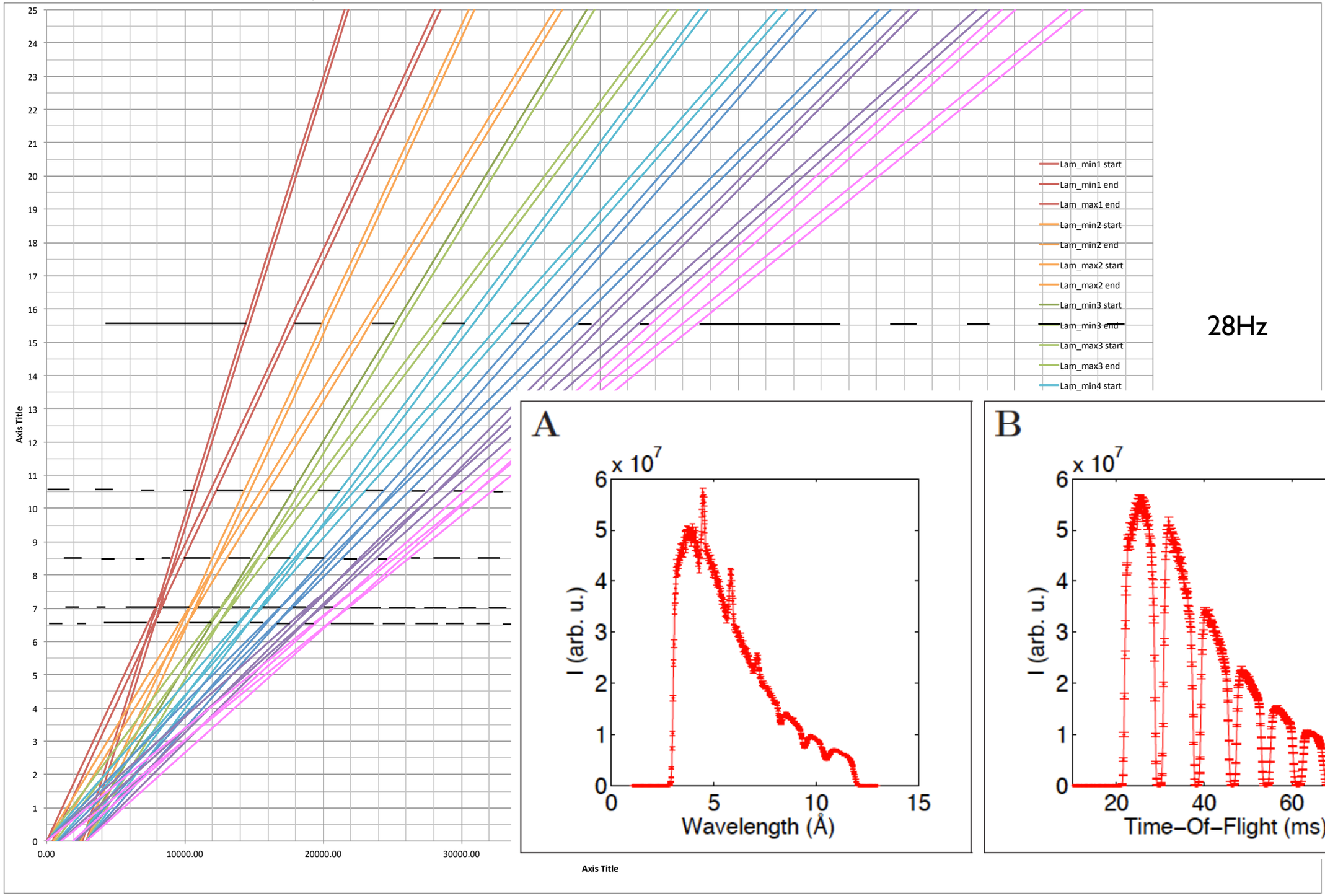
FOC3

FOC2

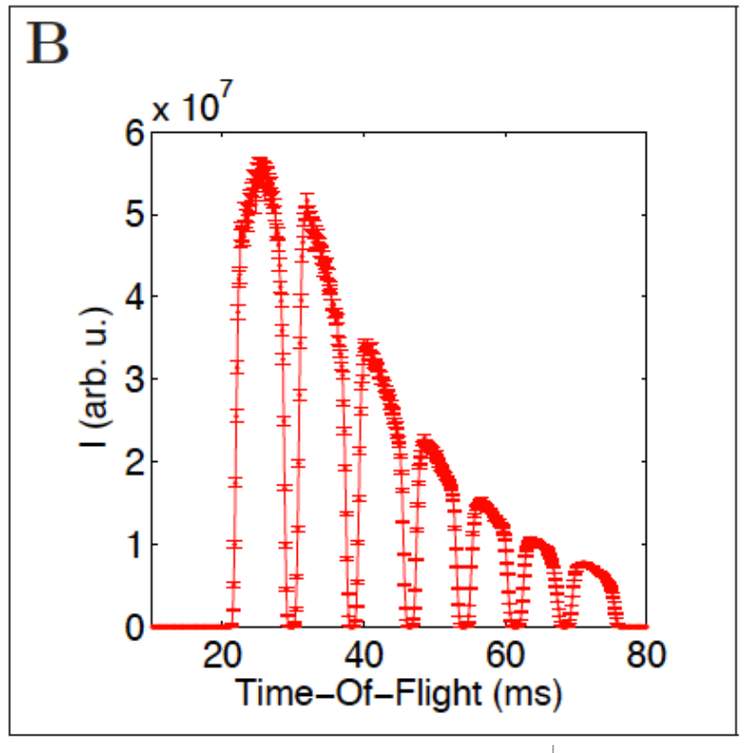
FOC1

WFM2

WFM1

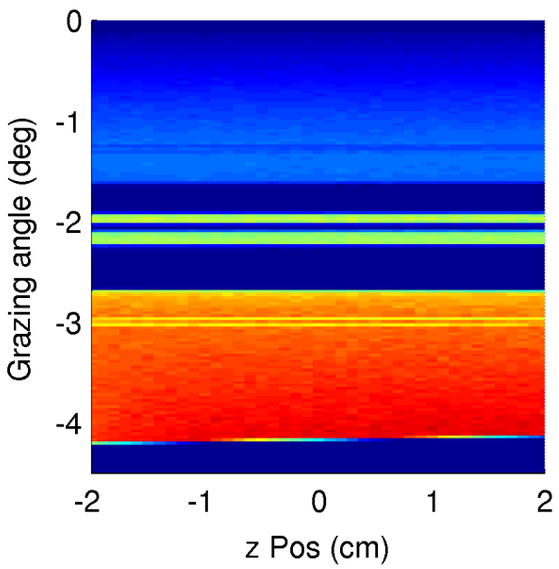


28Hz

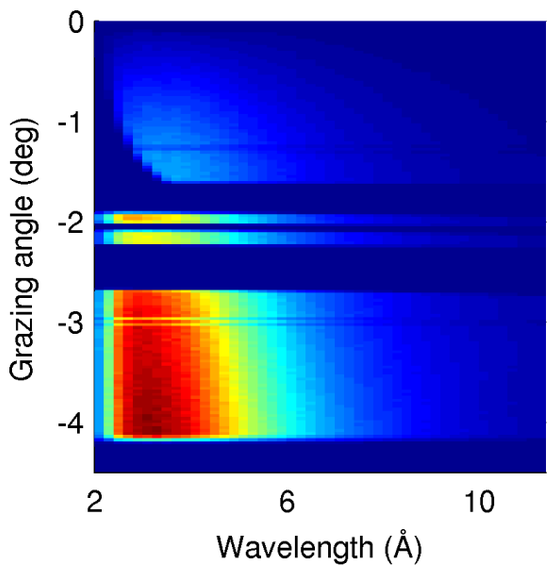


Simulations (without WFM)

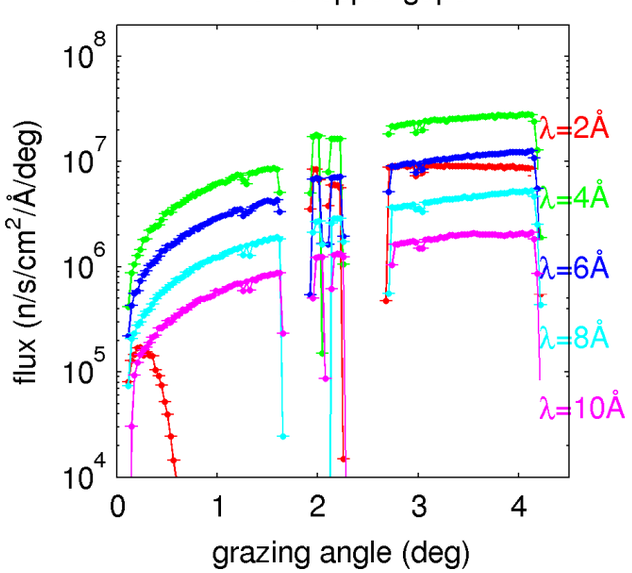
With chopper gaps
horizontal monitor



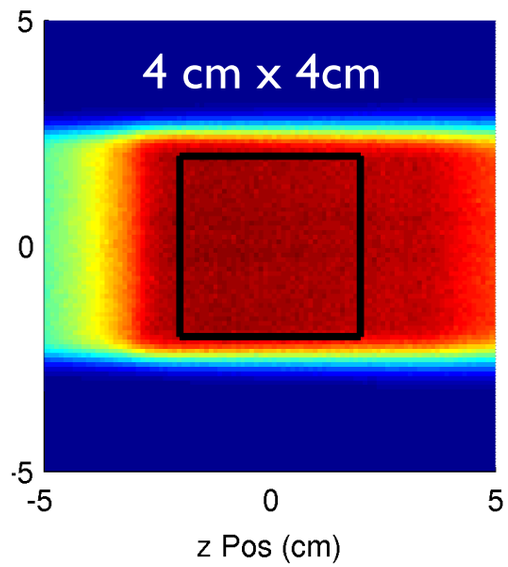
With chopper gaps
horizontal monitor



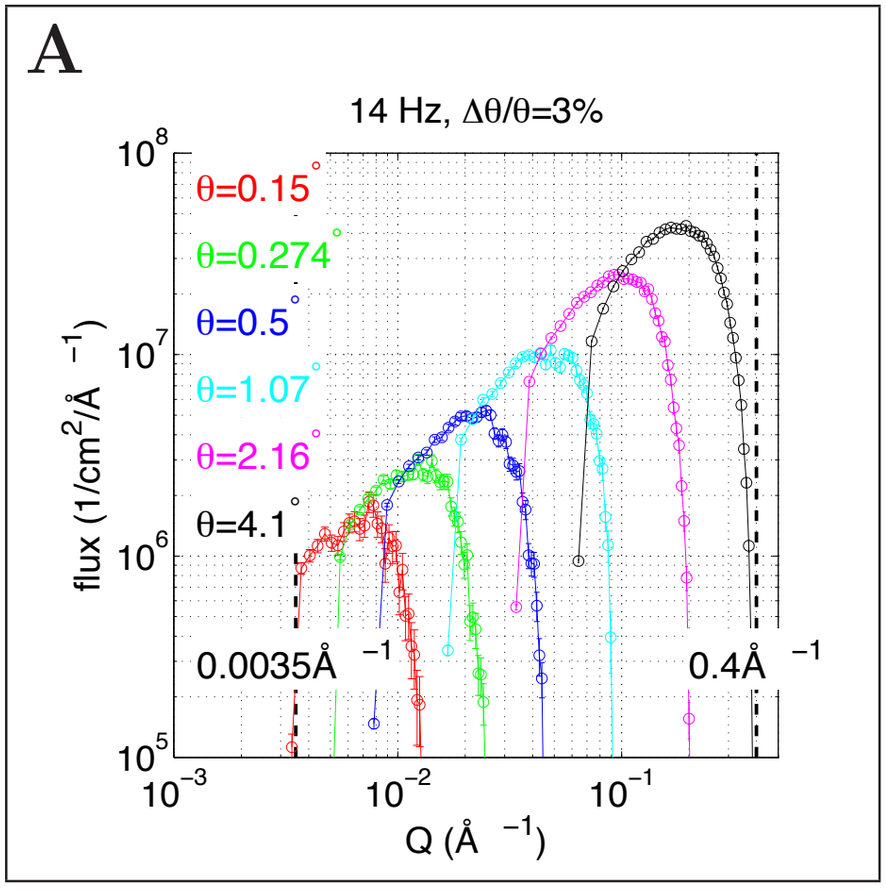
With chopper gaps



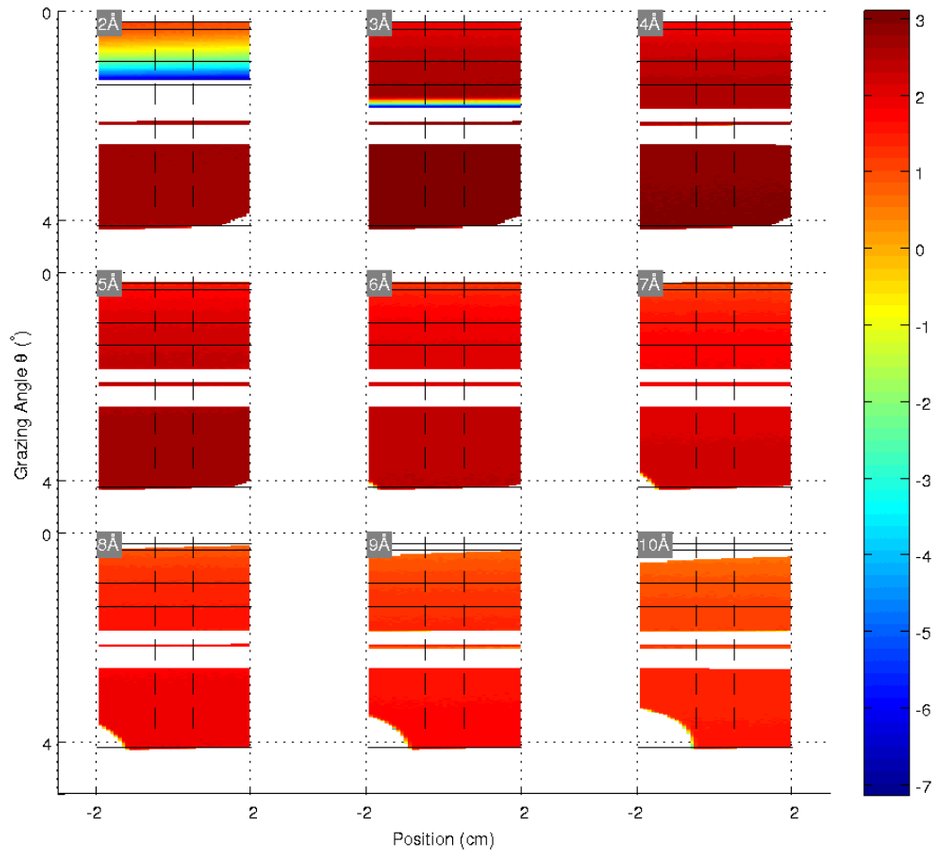
With chopper gaps
horizontal monitor



flux on 4cm x 4cm horizontal sample (collimated)

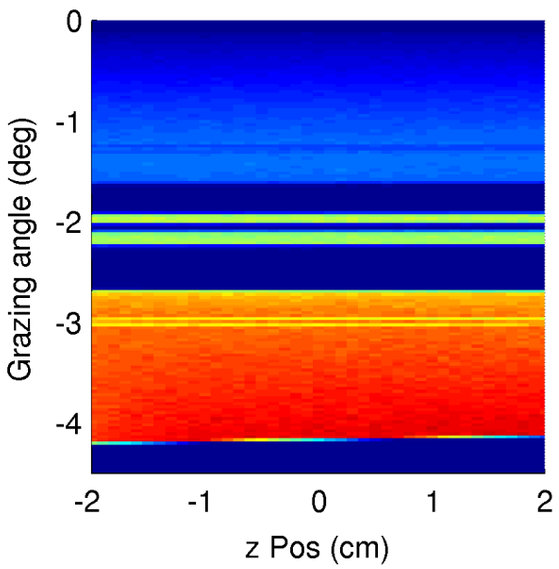


footprint as function of wavelength and angle (uncollimated)

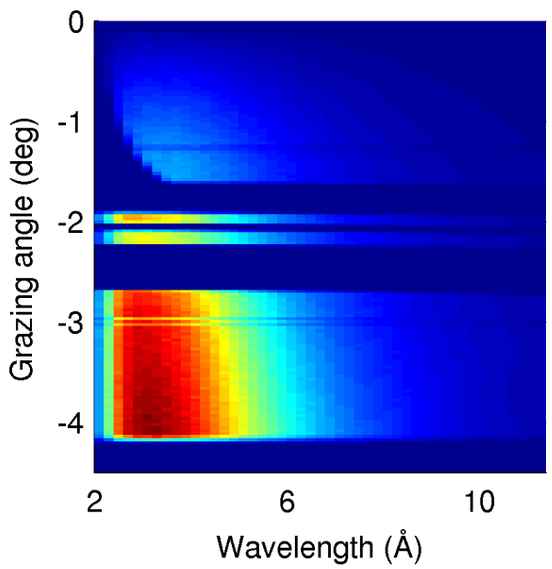


Simulations (without WFM)

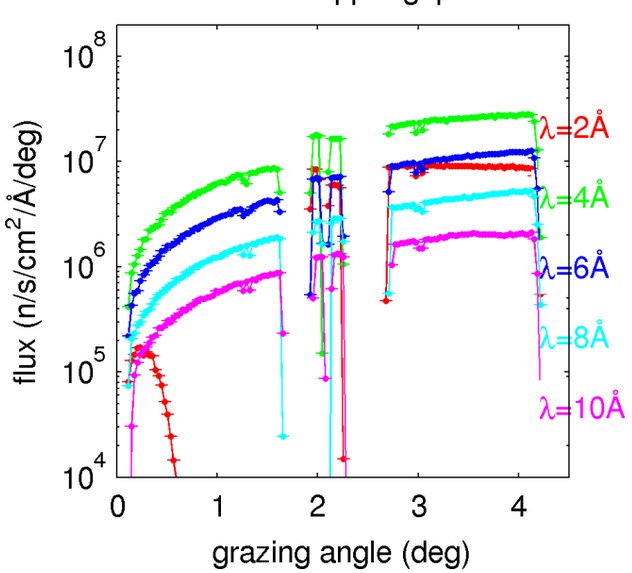
With chopper gaps
horizontal monitor



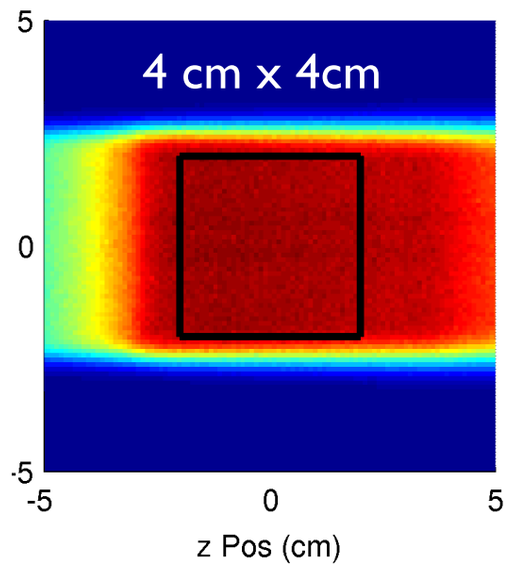
With chopper gaps
horizontal monitor



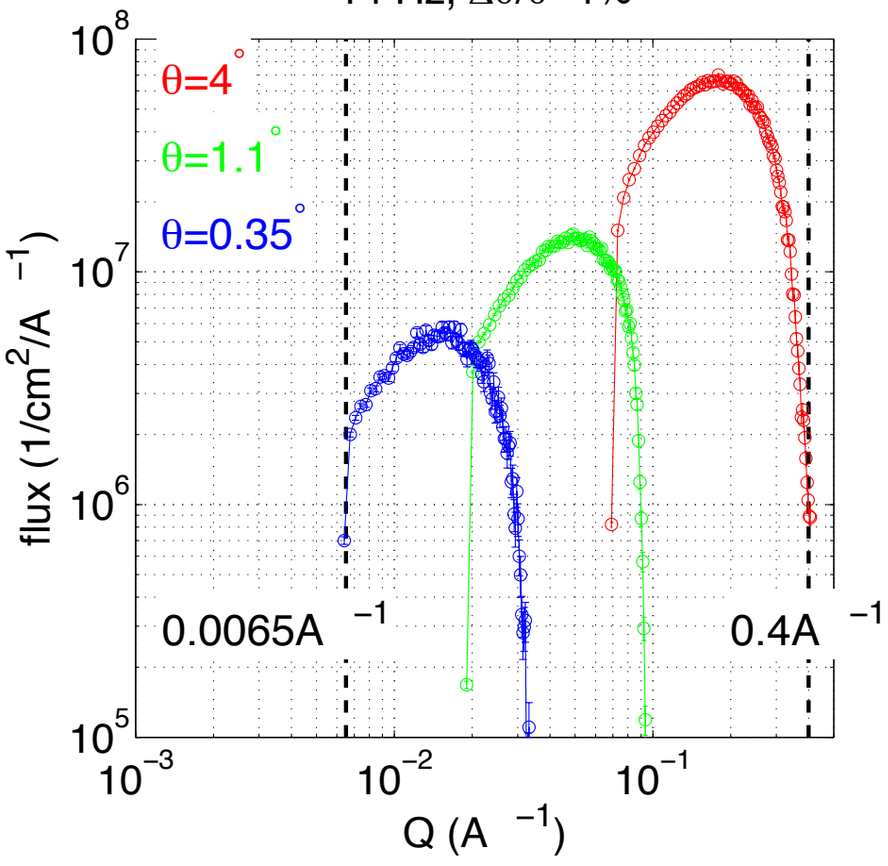
With chopper gaps



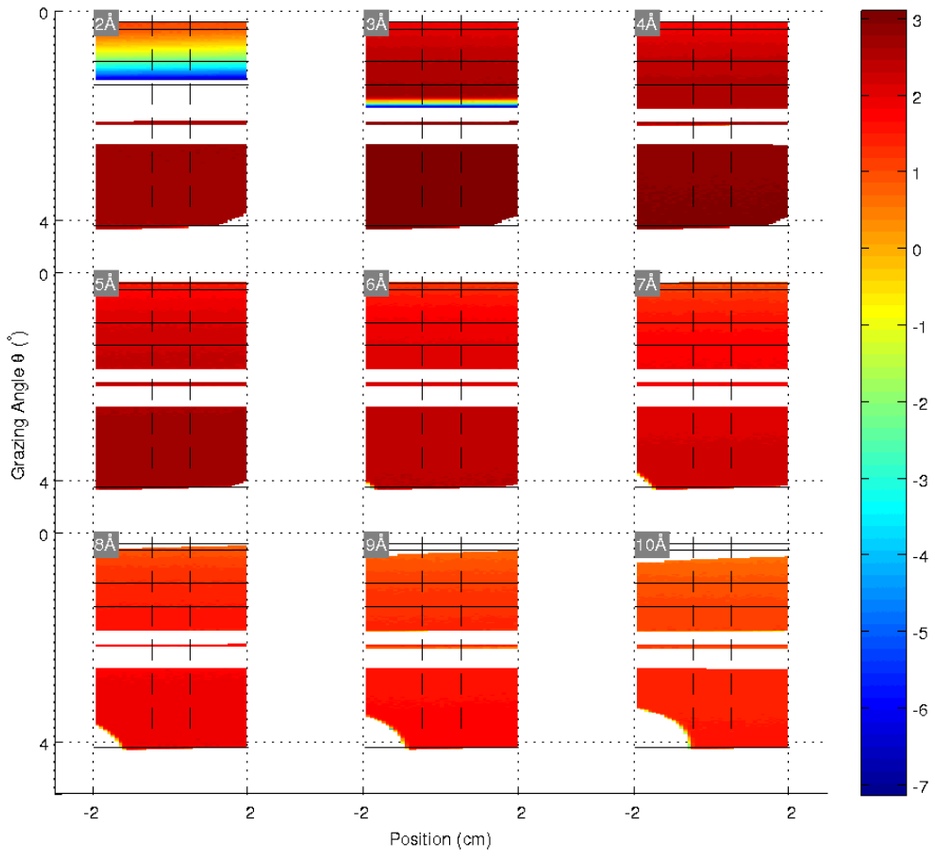
With chopper gaps
horizontal monitor



flux on 4cm x 4cm horizontal sample (collimated)

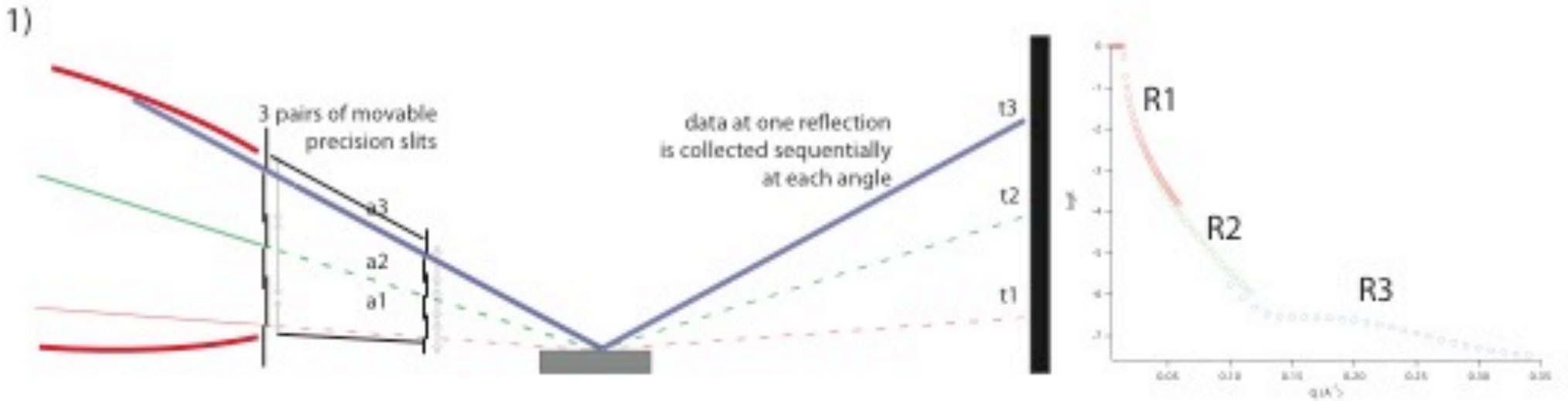


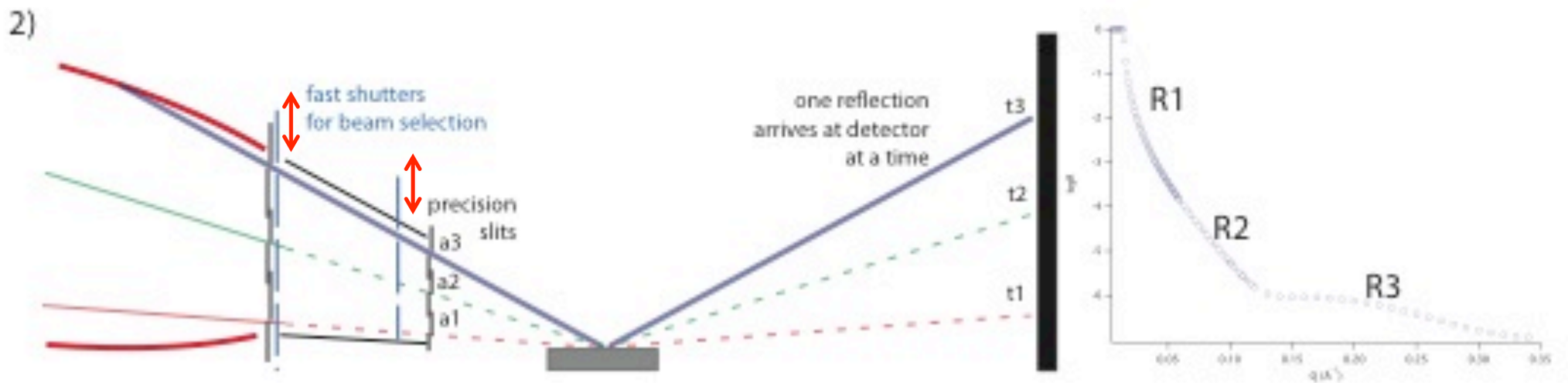
footprint as function of wavelength and angle (uncollimated)



Collimation

- a collimating guide (2m) to clean up off-specular scattering from the guide
  - with three pairs of slits that are are pre-positioned, possibility to open/close record full q-range with high time-resolution

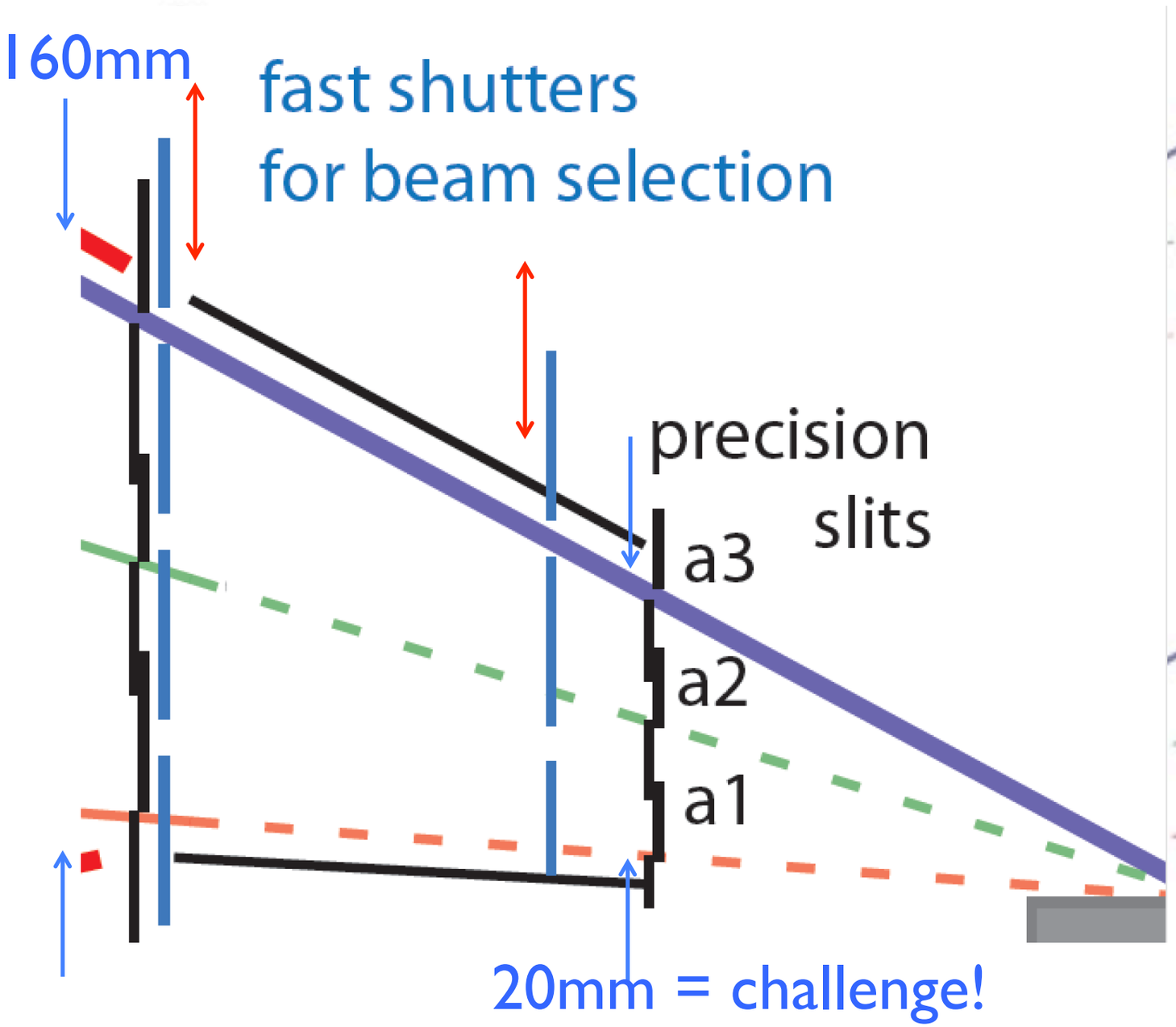


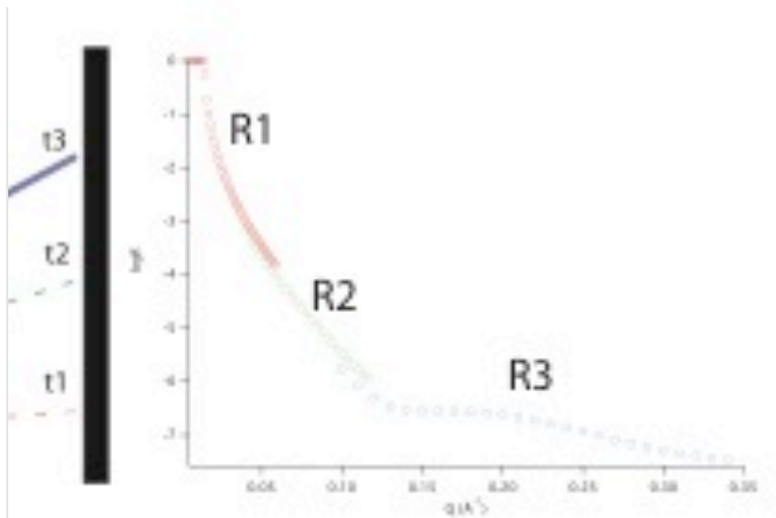


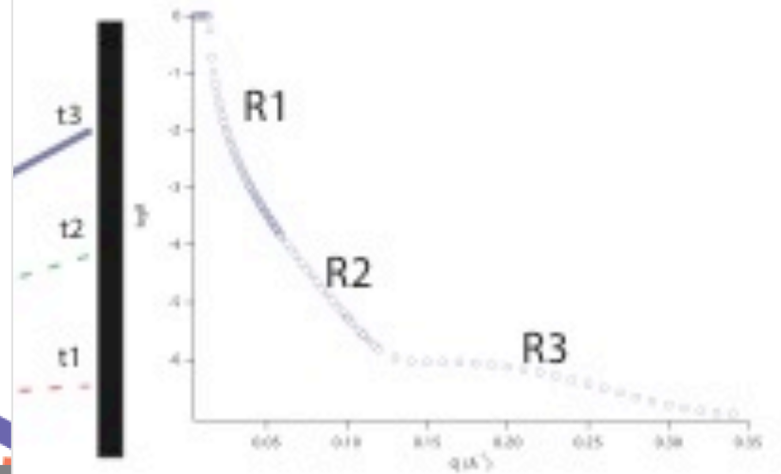
  - beam selection on pulse time scale to record full q-range 'simultaneously".

Collimation

- a collimating guide (2m) to clean up off-specular scattering from the guide
  - with three pairs of slits that are are pre-positioned, possibility to open/close with pulse-sequence and record full q-range with high time-resolution







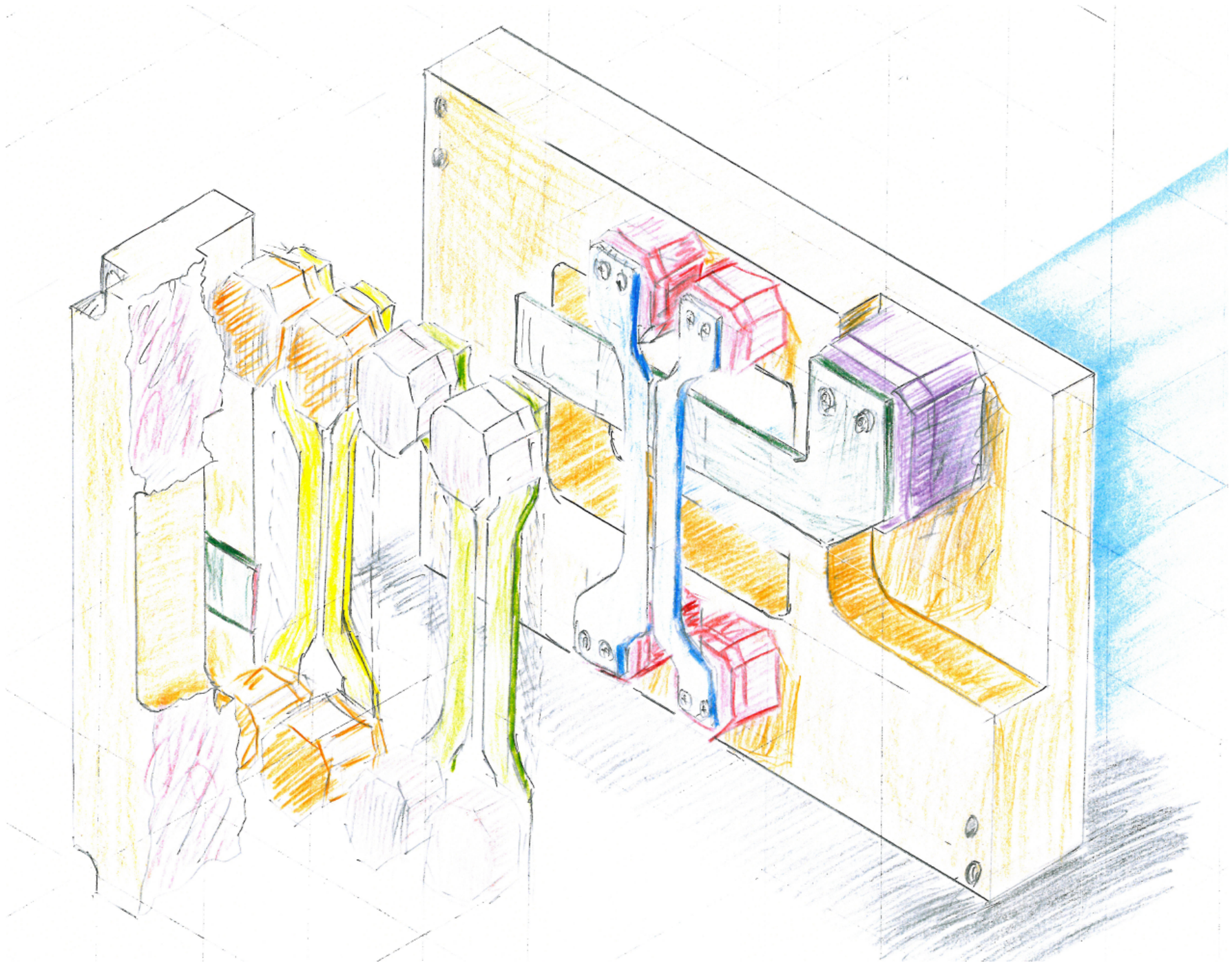
Collimation

- Not much space available - concept for triple slit package:

3 pairs of horizontal slit pairs, each movable to cover full theta range:

a common pair of vertical blades to limit beam width;

slit pair position in beam direction should not vary more than 40mm

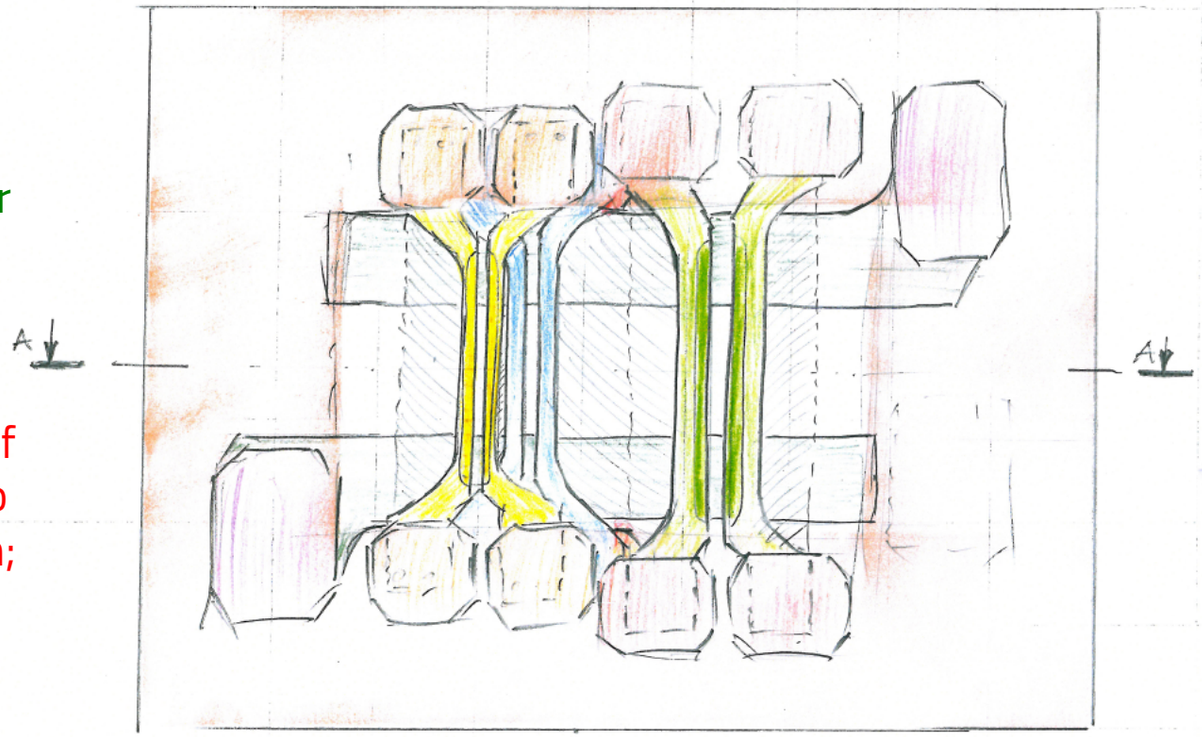


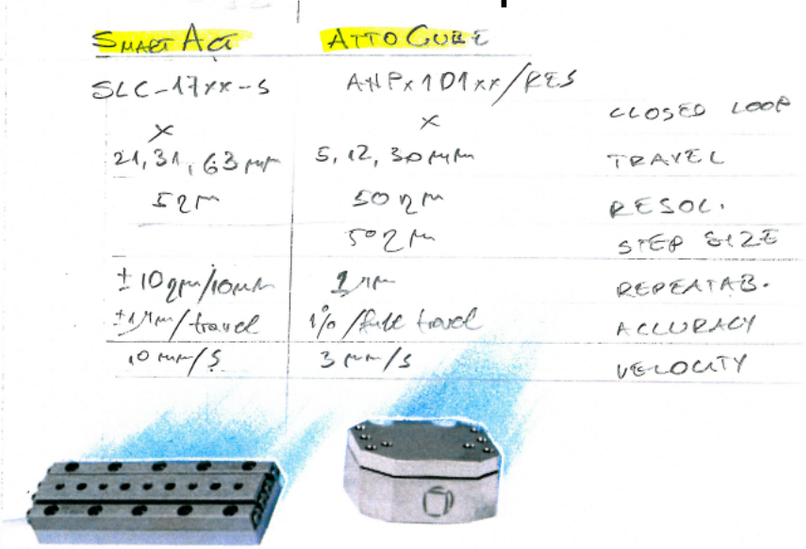
Zvonko Lazic, ESS

Collimation

- Not much space available: use piezo-driven positioners with nm-precision

3 pairs of horizontal slit pairs, each movable to cover full theta range:

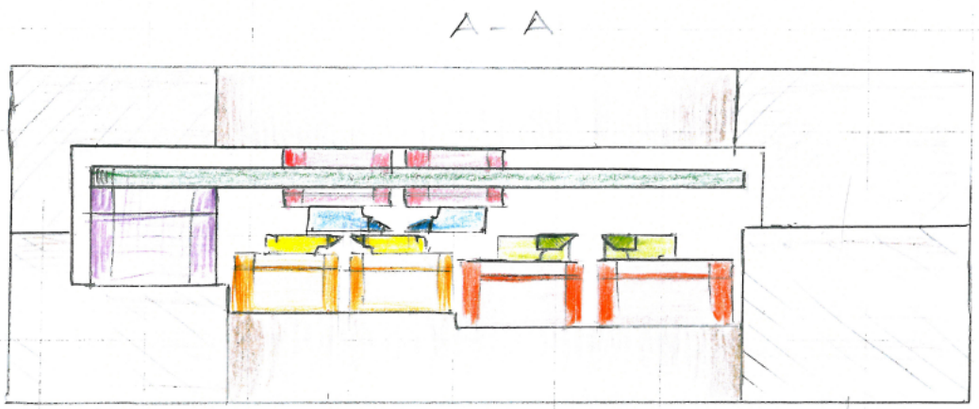


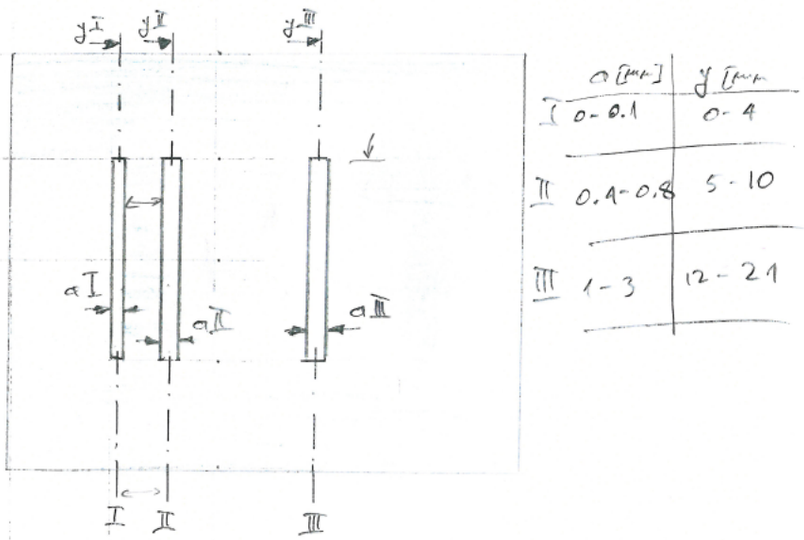


piezo devices
24mm x 24mm x 11mm

a common pair of vertical blades to limit beam width;

slit pair position in beam direction should not vary more than 40mm

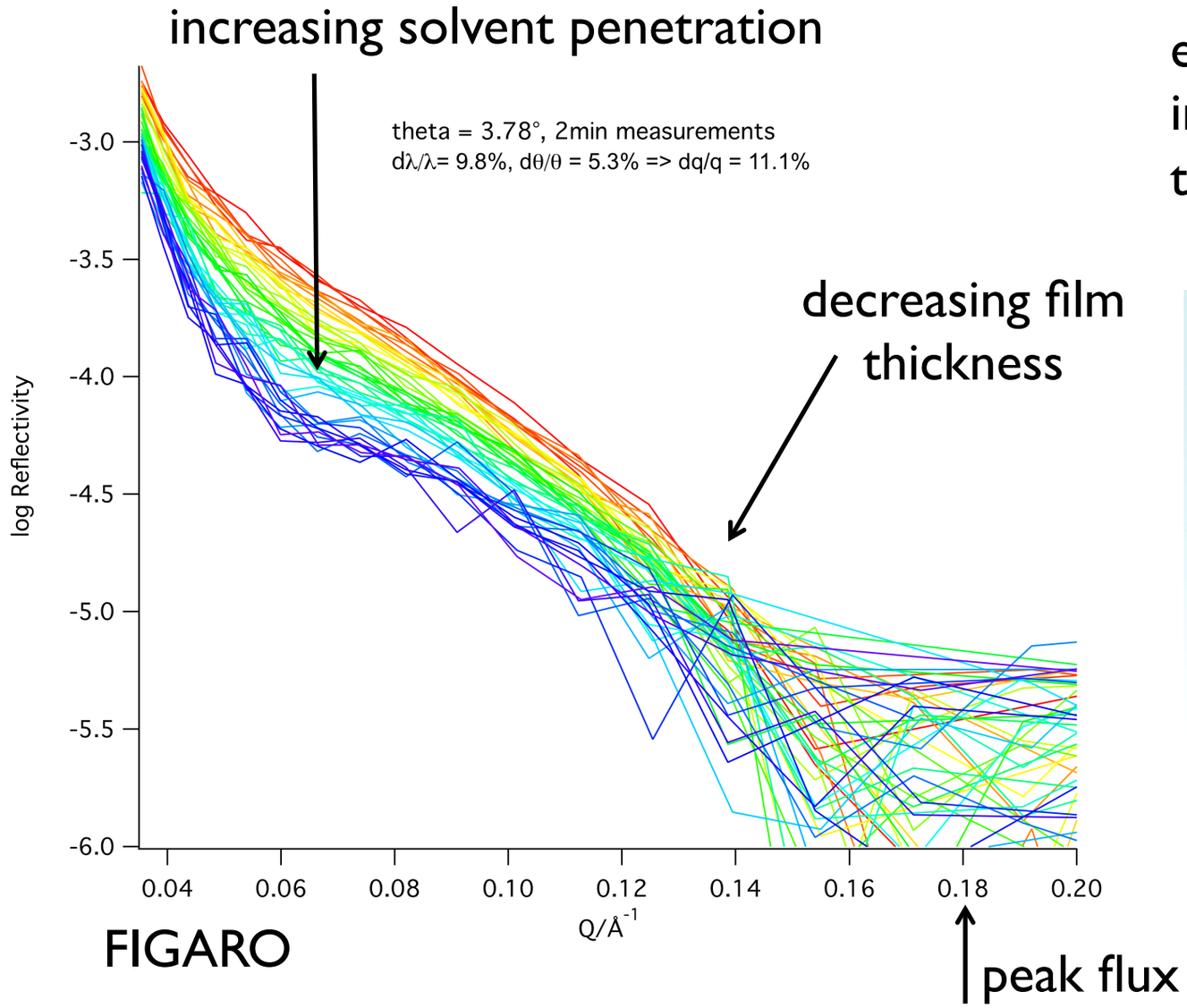




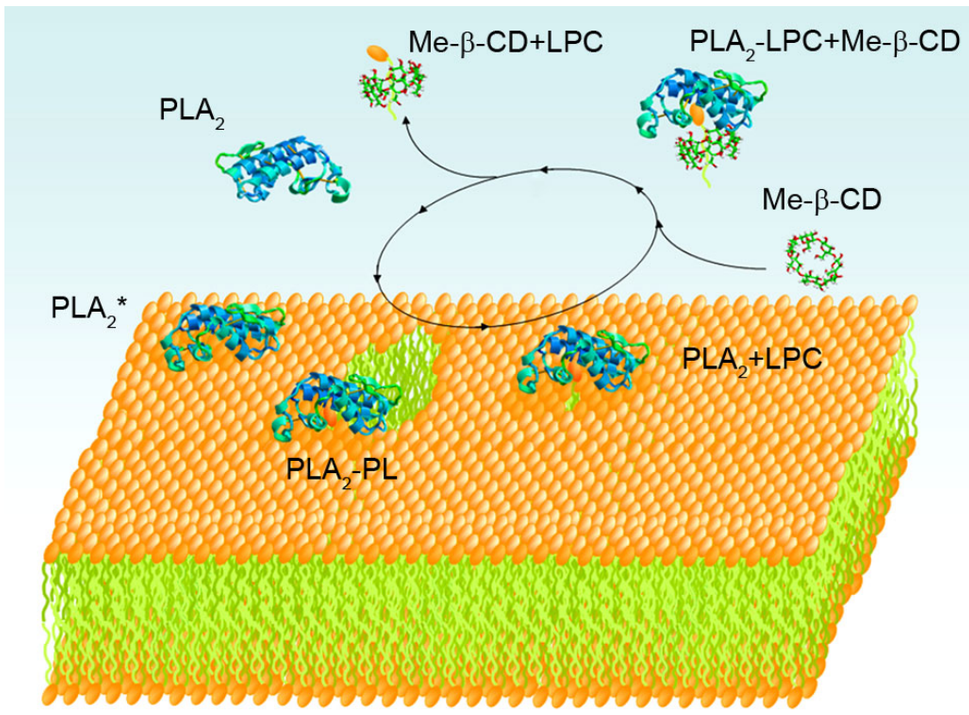
Zvonko Lazic, ESS

Summary

- FREIA will be a versatile reflectometer for soft matter, optimised for kinetic studies on thin films
- no existing reflectometer operates without moving the sample
- Varying angle with pulse sequence has potential to allow a very broad dynamic q-range (needs development and prototyping).



enzyme kinetics slowed down by 10x in D2O – today we can not measure this in H2O and avoid isotope effects…



Acknowledgements

Anette Vickery
Simulations

Zvonko Lazic
Slit concept

Thomas Gähl
Motion control for slits

Ken Andersen

Iain Sutton
Choppers

Phil Bentley
Optics

Richard Hall-Wilton
Detectors

Freia, (Frejya, Freyia, Frøya, Frøjya, and Freja) in Old Norse the "Lady", one of the Vanir gods, rules over the heavenly afterlife field Fólkvangr and there receives half of those that die in battle.

Freyja is also the goddess of love, beauty, fertility, gold, witchcraft, war, death…and cats

Thank you!

Questions?

EUROPEAN SPALLATION SOURCE

Thor, the god of thunder, lightning, storms, oak trees, and strength, is one of the once rival god tribe of Æsir