Which metallic alloys can be used as pressure cell windows. Case of SANS

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Task 2: "Kinetics and Dynamics"

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Aim of the study

✓ Pressure cell for SANS (and NSE) measurements, up to 6,000 bar, with small volumes (≈ 100 µL) to study structure (and dynamics) of molecules in soft matter and biophysics (*e.g.* conformation of proteins, ...)

✓ Pressure cell windows in **sapphire**: no scattering but thick windows (several cm)

→ very fragile!!

✓ Alternative solution:

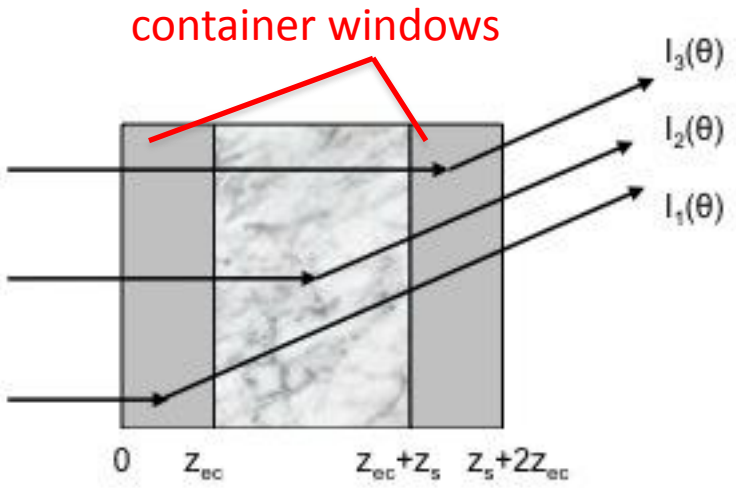
* thick **windows** in stainless materials (**alloys**), which display good mechanical properties, reasonable transmissions, and (if possible...) a « low » Q-scattering

* use of a method developped at LLB for **cell subtraction**:

• A. Brûlet *et al. J Applied Crystallography* 40: 165-177, 2007

• « Pasinet » reduction software: http://didier.lairez.fr/pasinet2/doku.php

SANS data corrections with scattering containers



Hypothesis:
neutrons are scattered once

Sample Empty container Empty beam

with

Ref.: A. Brulet, D. Lairez, A. Lapp, and JP. Cotton. Improvement of data treatment in small-angle neutron scattering. *J Applied Crystallography* 40: 165-177, 2007

Pressure cell with removable windows

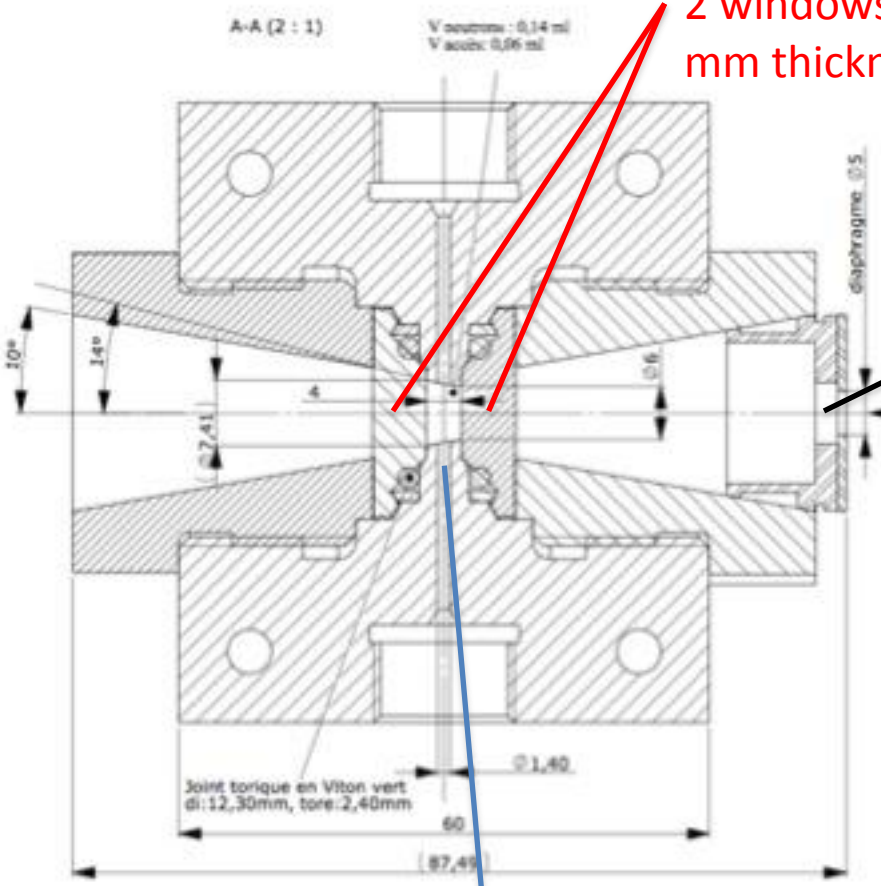
container removable windows

SANS: small beam, small angle → flat cell with windows:

- difficulties: machining, sealing
- advantages: thick body in resistant material (stainless steel)
- removable windows in suitable material

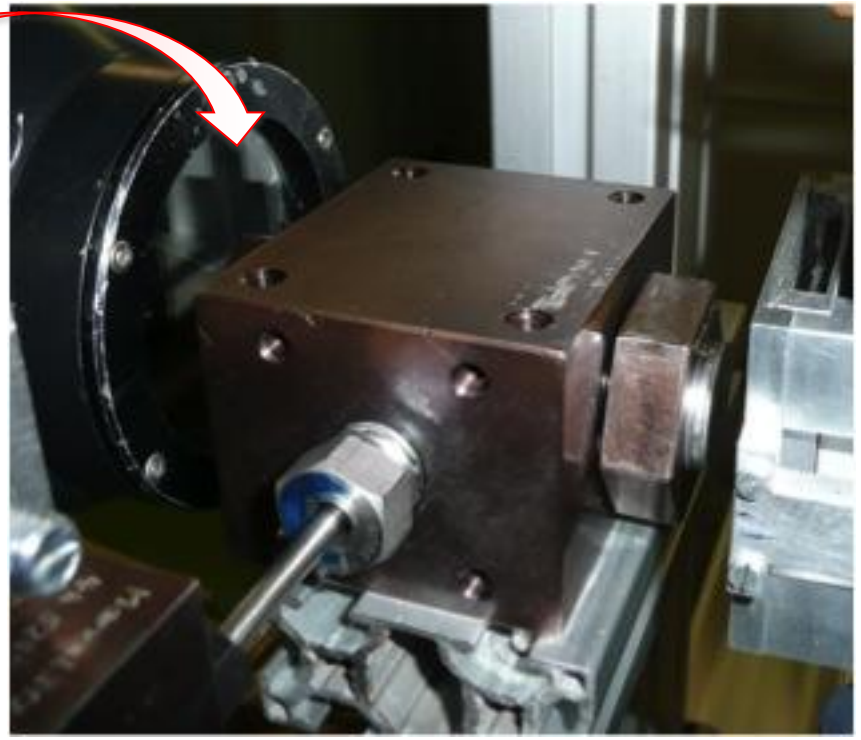
Pressure cell with removable windows

- ✓ cell body: Marval x12 stainless and extremely resistant → reusable
- ✓ windows: cheap (≈ 100 €) and easy to machine
  →can be changed
  → adaptable to the experiment
- ✓ easy to clean and purge
- ✓ temperature control (15-140°C)



Device of pressure cell for SANS measurements

pressure device on (the late) PAXE SANS spectrometer



Suitable alloys for the pressure cell windows

✓ **Titane/Zirconium alloy** (TiZr)

TiZr (Ti52Zr48, in mass) null matrix alloy (« zero scattering alloy »):

$b_{coh}(Ti) = -3.44$ fm
$b_{coh}(Zr) = 7.16$ fm
} b_{coh}(67.7% Ti + 32.3% Zr) = 0 (in mole)

but: high absorption (σ_{abs} ($Ti_{0.677}Zr_{0.323}$) = 4.33 barn) and incoherent scattering (σ_{inc} ($Ti_{0.677}Zr_{0.323}$) = 1.96 barn) → corrections

but: high coherent scattering at small Q due to microcrystallites

→ Q > 0.07 $Å^{-1}$ (*e.g.* small proteins)

P_{max} ≈ 6,000 bar

Suitable alloys for the pressure cell windows

✓ **Aluminium**

Al 2017A T4 (AlCu4MgSi)

good transmission (≈ 0.96 for 10 mm at 6 Å, better than pure Nb), reasonable low Q-scattering (essentially coherent: decreasing with Q) → Q > 0.01 $Å^{-1}$

P_{max} ≈ 4,500 bar; easier to machine than pure Nb (very soft)

Al 7049A T6 (AlZn8MgCu)

good transmission (≈0.95 for 10 mm at 6 Å) but high low Q-scattering (decreasing with Q) → Q > 0.1 $Å^{-1}$

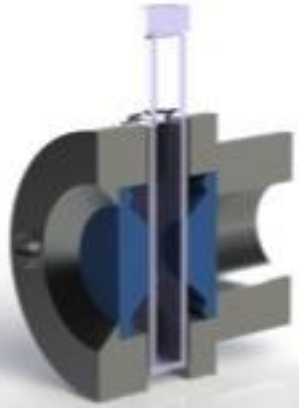
P_{max} ≈ 6,000 bar

 Zirconium Niobium (ZrNb1 = Zr99Nb1): M5™ alloy (from AREVA), P_{max} ≈ 3,000 bar

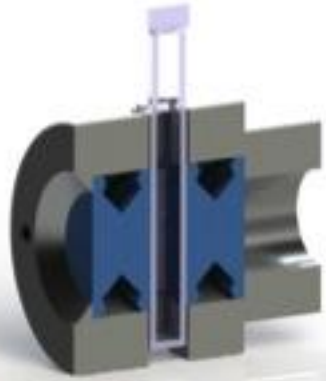
✓ **Stainless steel**: M30NW alloy (from Aubert & Duval) = X4CrNiMnMoN 21.9.4, P_{max} ≈ 6,000 bar

SANS measurements on the removable windows

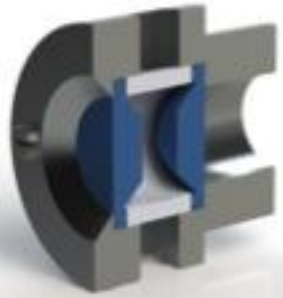
Use of a device dedicated for the measurements:



2 windows + sample in quartz cell



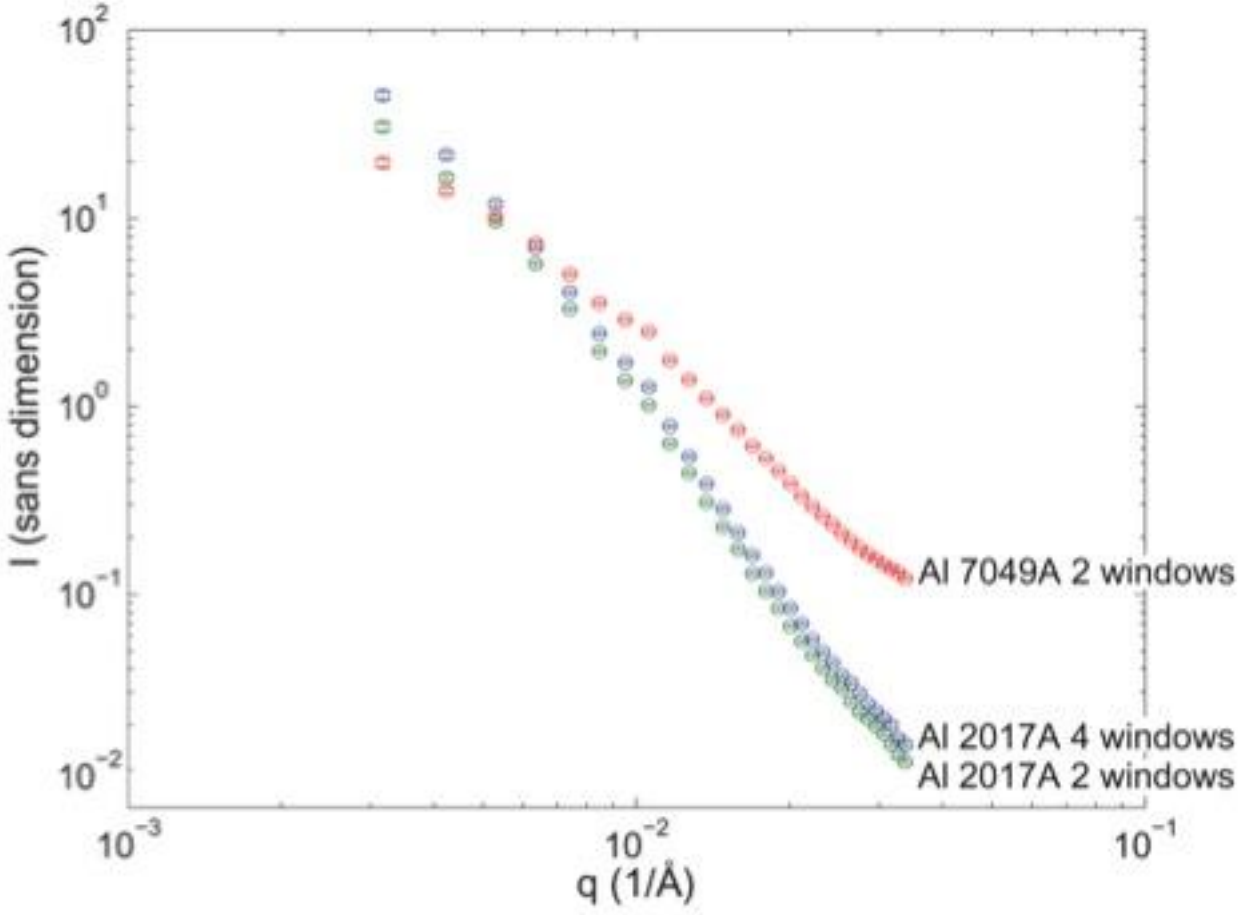
4 windows + sample in quartz cell



« empty cell » (2 windows)

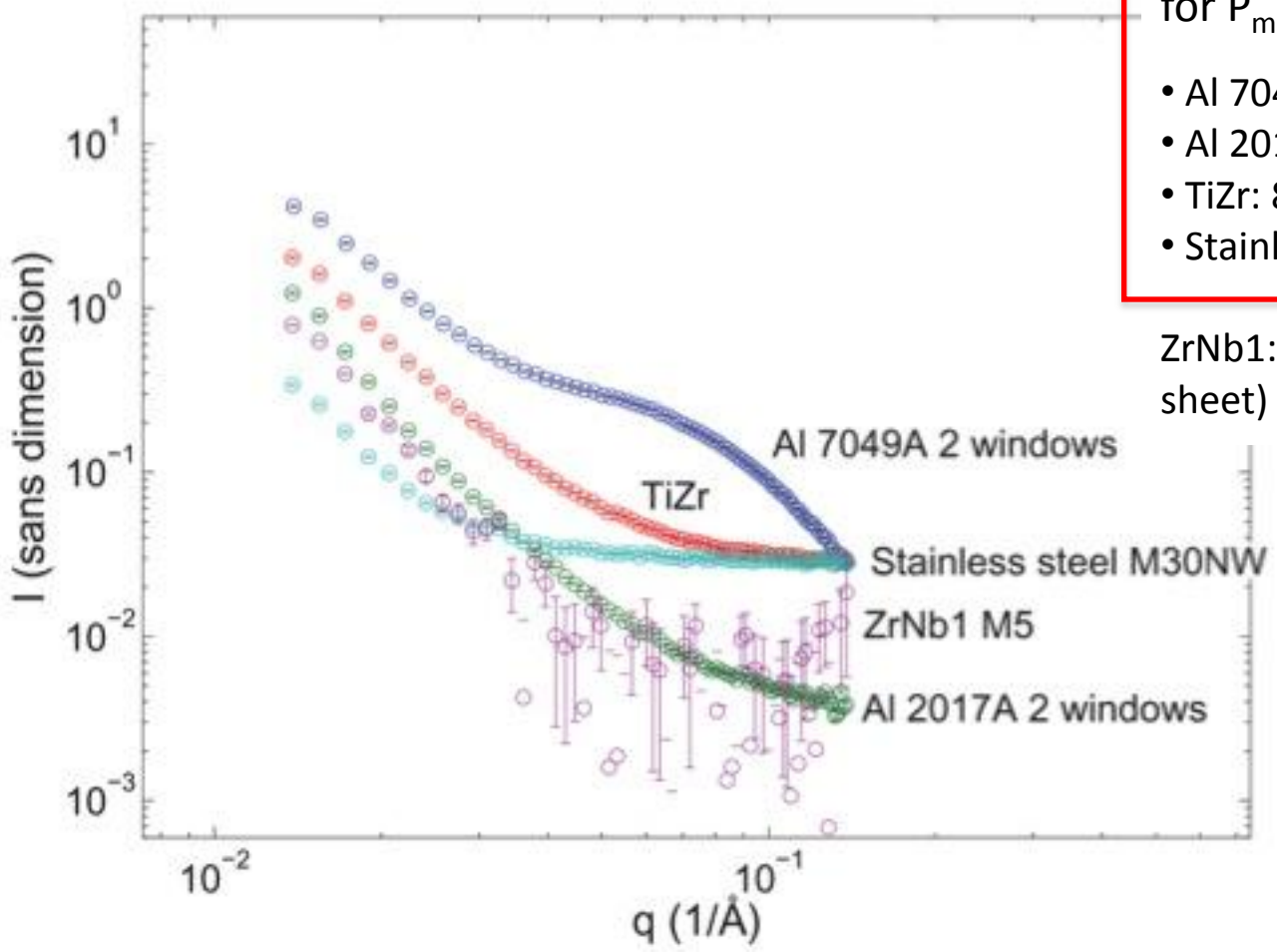
- Aluminium
-low Q
-windows alone

PACE, 13 Å-4.5 m : 0.003 < Q < 0.035 $Å^{-1}$



- other alloys
- higher Q
- windows alone

PAXY, 6 Å-3 m : 0.01 < Q < 0.15 $Å^{-1}$, **normalization by the thickness**



Window <u>thickness</u> for P_{max} ≈ 6,000 bar:

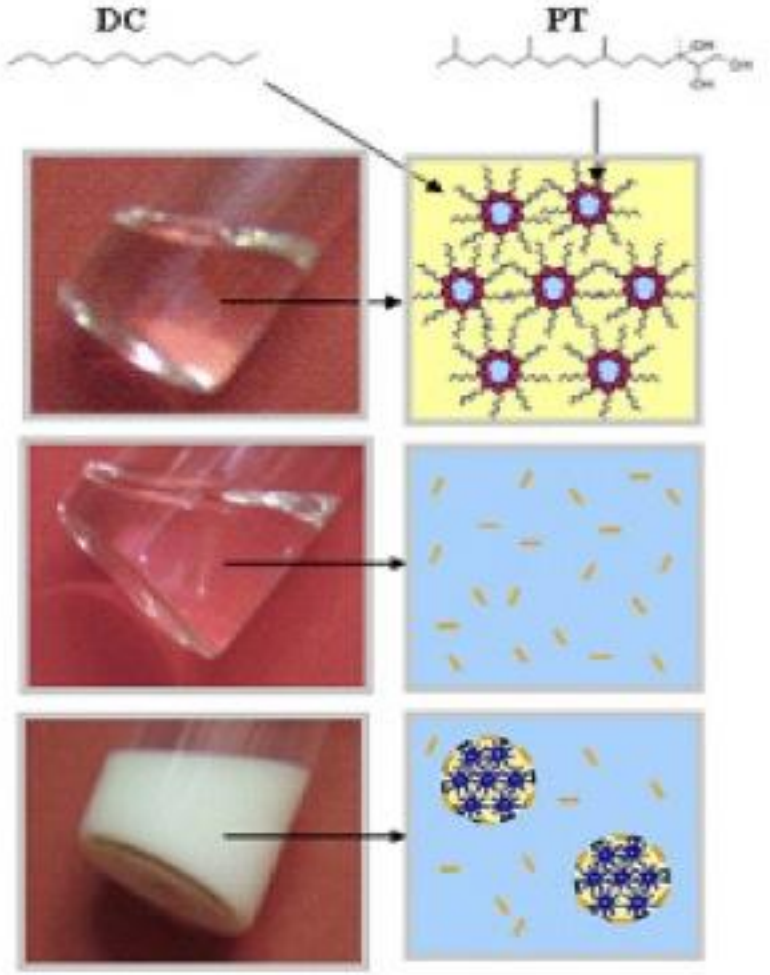
- Al 7049A: 12.2 mm
- Al 2017A: 12.2 mm
- TiZr: 8.6 mm
- Stainless steel: 10 mm

ZrNb1: 0.4 mm (thin sheet)

Transmission of the windows

	Nb of windows	Tickness of a window (mm)	Total thickness (mm)	Transmission at 13 Å	**Tr at 13 Å for 10 mm thickness**	Transmission at 6 Å	**Tr at 6 Å for 10 mm thickness**	Maximum pressure (bar) **for 10 mm**	Q range ($Å^{-1}$)
Al 2017A	2	6.1	12.2	0.785	**0.817**	0.948	**0.961**	~4500	Q > 0.01
Al 2017A	4	6.1	24.4	0.524	0.768	0.896			
Alu 2017A + Mb	2	6.1	12.2 + Mb	0.506		0.556			
Alu 2017A + laponite/emulsion	2	6.1	12.2 + lapo.			0.724			
Alu 2017A + laponite/emulsion	4	6.1	24.4 + lapo.			0.708			
Al 7049A	2	6.1	12.2	0.760	**0.801**			~6000	Q > 0.1
Al 7049A	4	6.1	24.4			0.884	**0.951**		
Alu 7049A + Mb	2	6.1	12.2 + Mb	0.497		0.544			
Alu 7049A + laponite/emulsion	4	6.1	24.4 + lapo.			0.675			
TiZr (52.5%-47.5%)	2	4.3	8.6			0.416	**0.361**	~6000	Q > 0.07
TiZr + Mb	2	4.3	8.6 + Mb			0.261			
Stainless steel (M30NW)	2	5	10			0.295	**0.295**	~6000	Q > 0.01
ZrNb1 (M5)	--	--	0.4			0.993	**0.842**	~3000	Q > 0.01
Nb			10			0.8	**0.801**	~2500	Q > 0.01

Test #1 - high scattering sample: laponite/emulsion

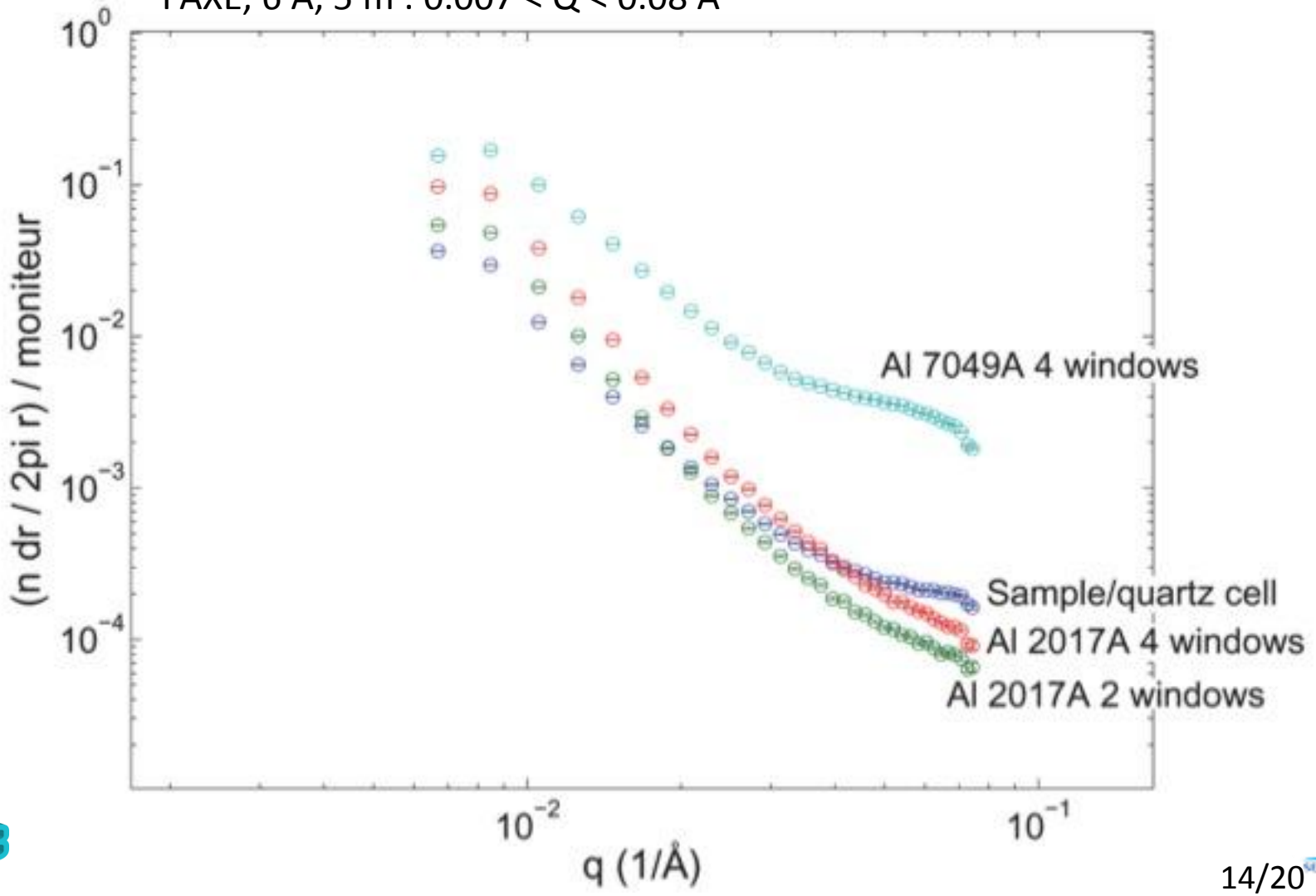


Laponite/emulsion:
phytantriol (PT) and dodecane (DC) mixture (50:50) in water stabilized by Laponite clays (disc-like particles with a mean radius of 12.5 nm and a thickness of 1 nm) to form Pickering emulsions

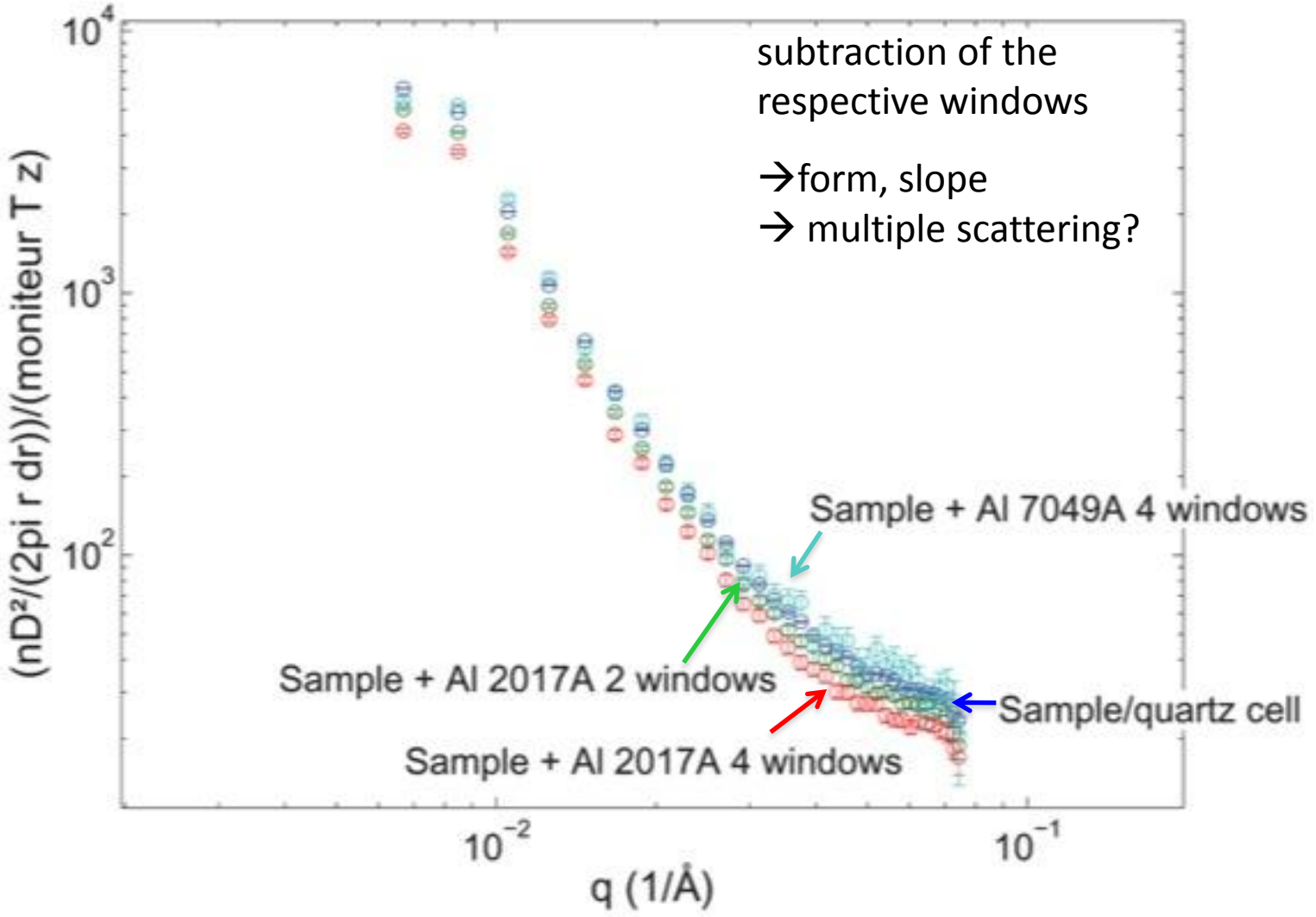
Sample: in D_2O, in a 5 mm Hellma Suprasil quartz cell

sample/quartz cell *vs.* windows alone

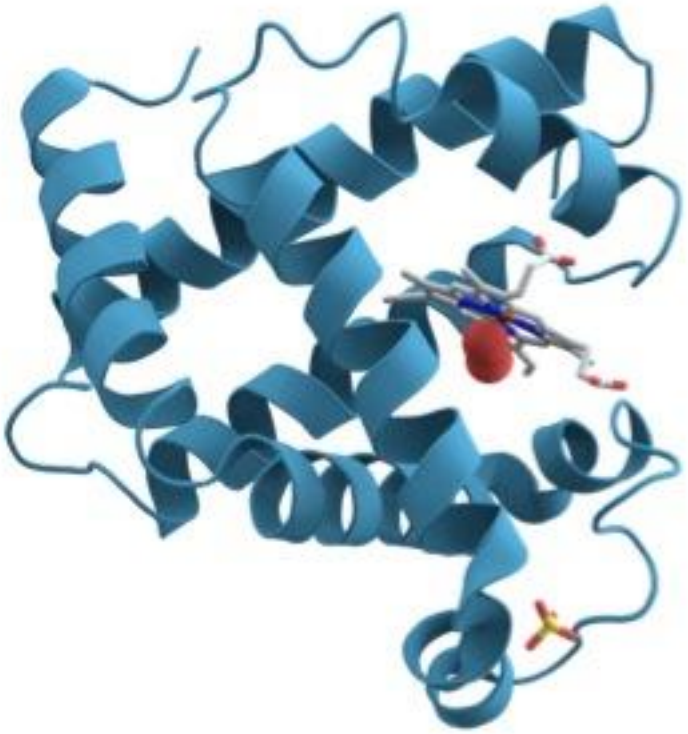
PAXE, 6 Å, 5 m : 0.007 < Q < 0.08 Å^{-1}



Laponite/emulsion sample: cell subtraction



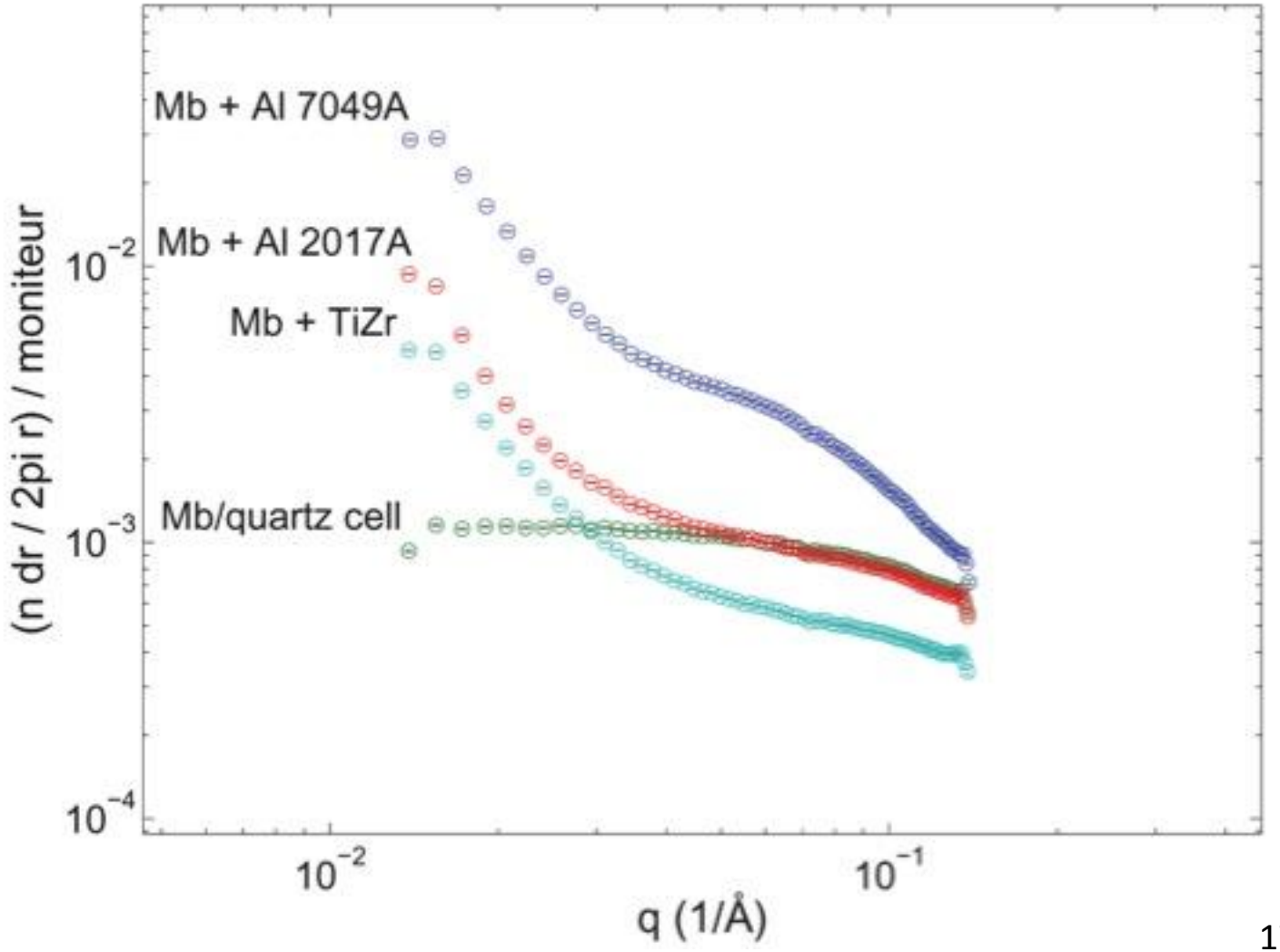
Test #2 on a lower scattering sample: the myoglobin protein



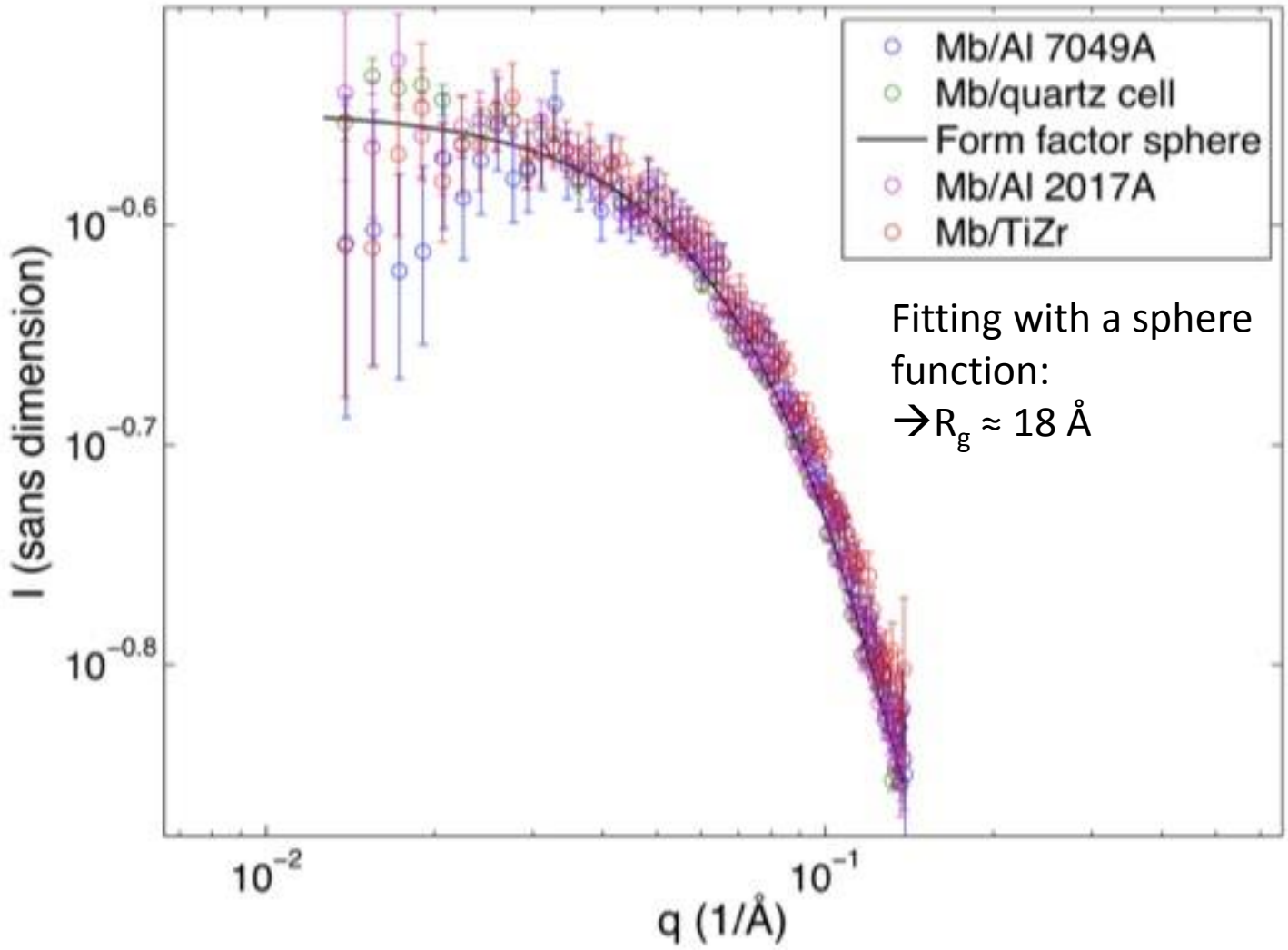
Myoglobin: small globular protein (17.7 kDa)
Sample: 20 g/L of Mb in a D_2O buffer, in a 5 mm Hellma Suprasil quartz cell

Myoglobin sample + windows

PAXY, 6 Å-3 m : 0.01 < Q < 0.15 $Å^{-1}$

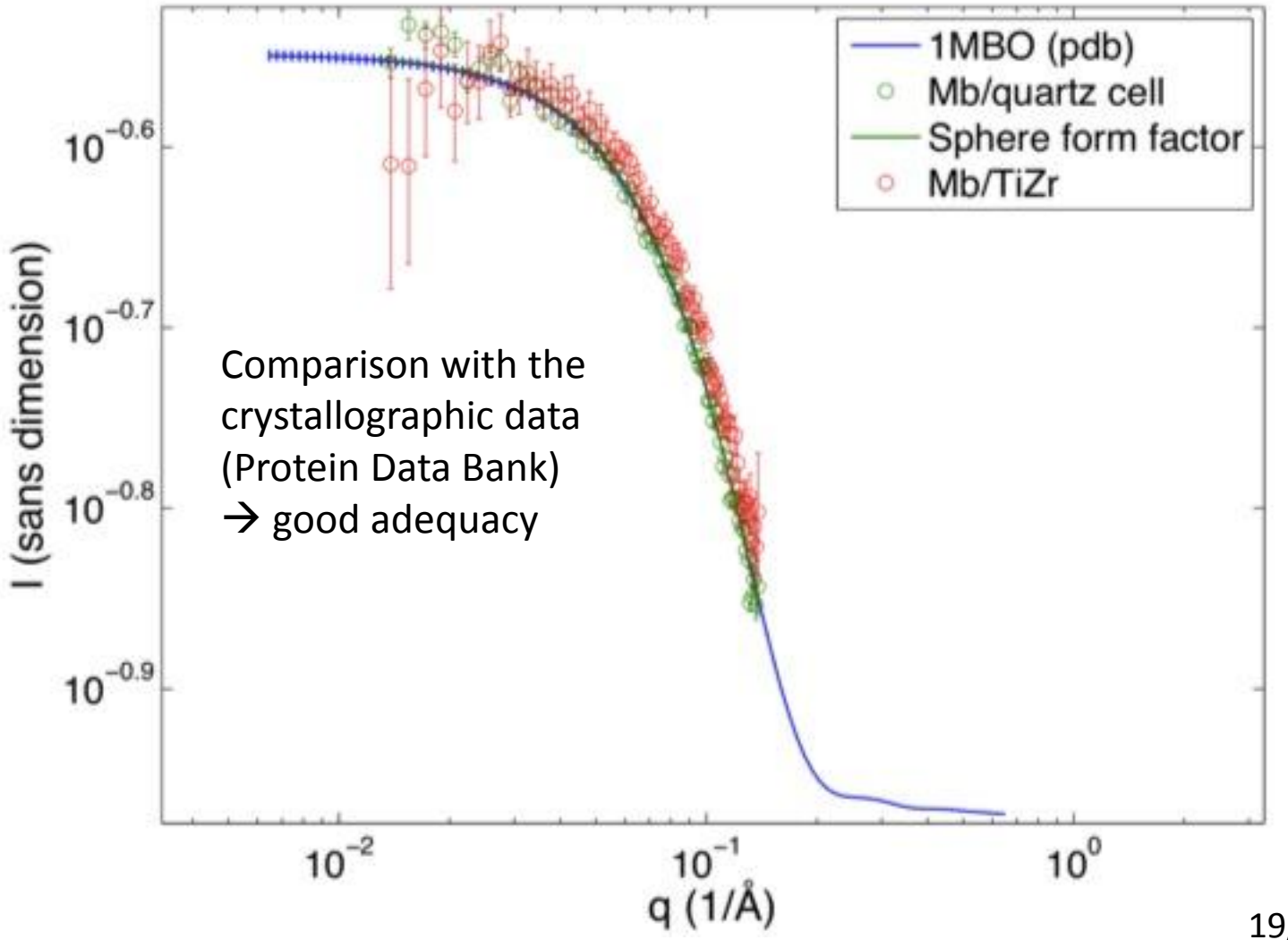


Cell subtraction and fitting



Fitting with a sphere function:
→$R_g \approx 18$ Å

Cell subtraction and fitting



Comparison with the crystallographic data (Protein Data Bank)
→ good adequacy

Conclusions and perspectives

 Good subtraction of the pressure cell windows (Al 2017A, Al 7049A, TiZr)
- both at low and high Q-values
- both for a high scattering sample (laponite) and for a much lower one (myoglobin protein) ; probably multiple scattering in the case of laponite

 Removable windows: adaptable to the experiment
- range of Q-scattering
- absorption
- P_{max}
- corrosion

 Transmission: Al 2017A, Al 7049A, and M5 (ZrNb1): better than TiZr and much better than stainless steel

 SANS test on ZrNb1 and Nb windows, with a sample

 Test of similar windows (non magnetic materials) on the neutron spin echo spectrometer (MUSES, LLB)