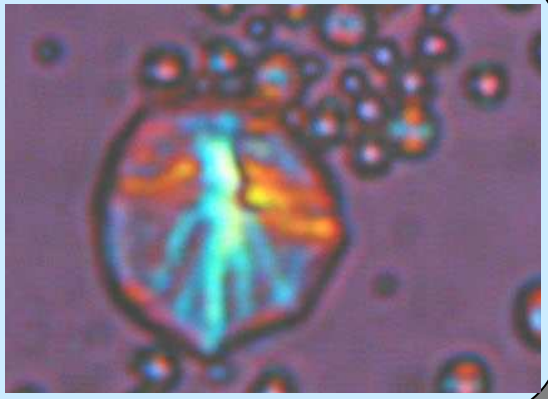
Microcalorimètre permettant des mesures couplées SAXS/WAXS/DSC



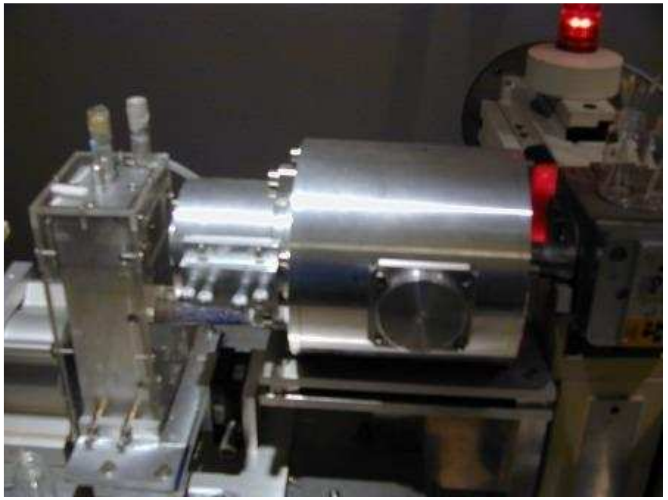
Michel OLLIVON, C. BOURGAUX, G. KELLER, P. LESIEUR, JB BRUBACH, D. KALNIN

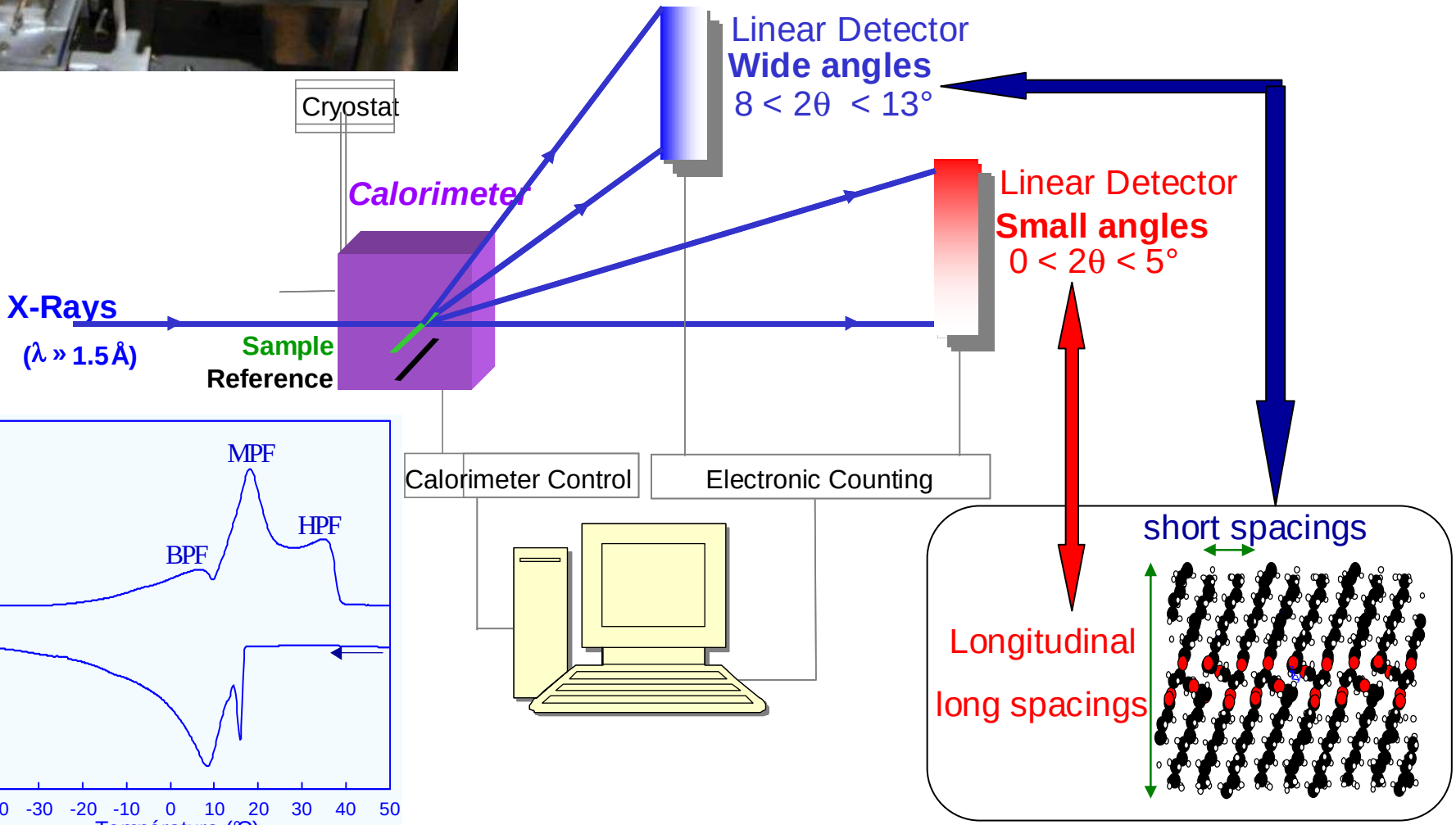
– Equipe Physico-Chimie des Systèmes Polyphasés, UMR CNRS 8612 Université Paris-Sud, Faculté de Pharmacie, 92296 Châtenay-Malabry, France.

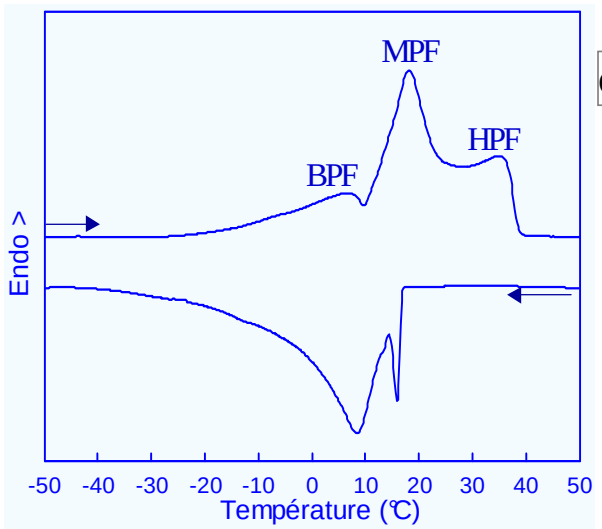
michel.ollivon@u-psud.fr

SAXS+WAXS+DSC *Setup*

Sample : 20 µl
Lin. glass Capillary (f= 1,4 ± 0,1 mm)







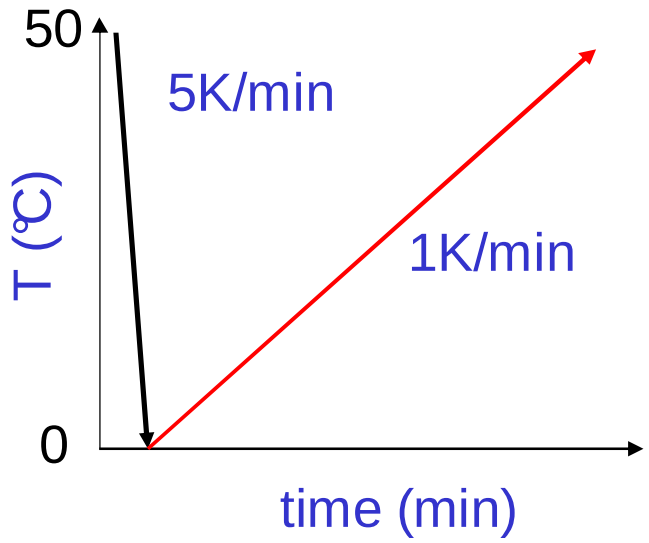
Techniques de caractérisation 2. DSC

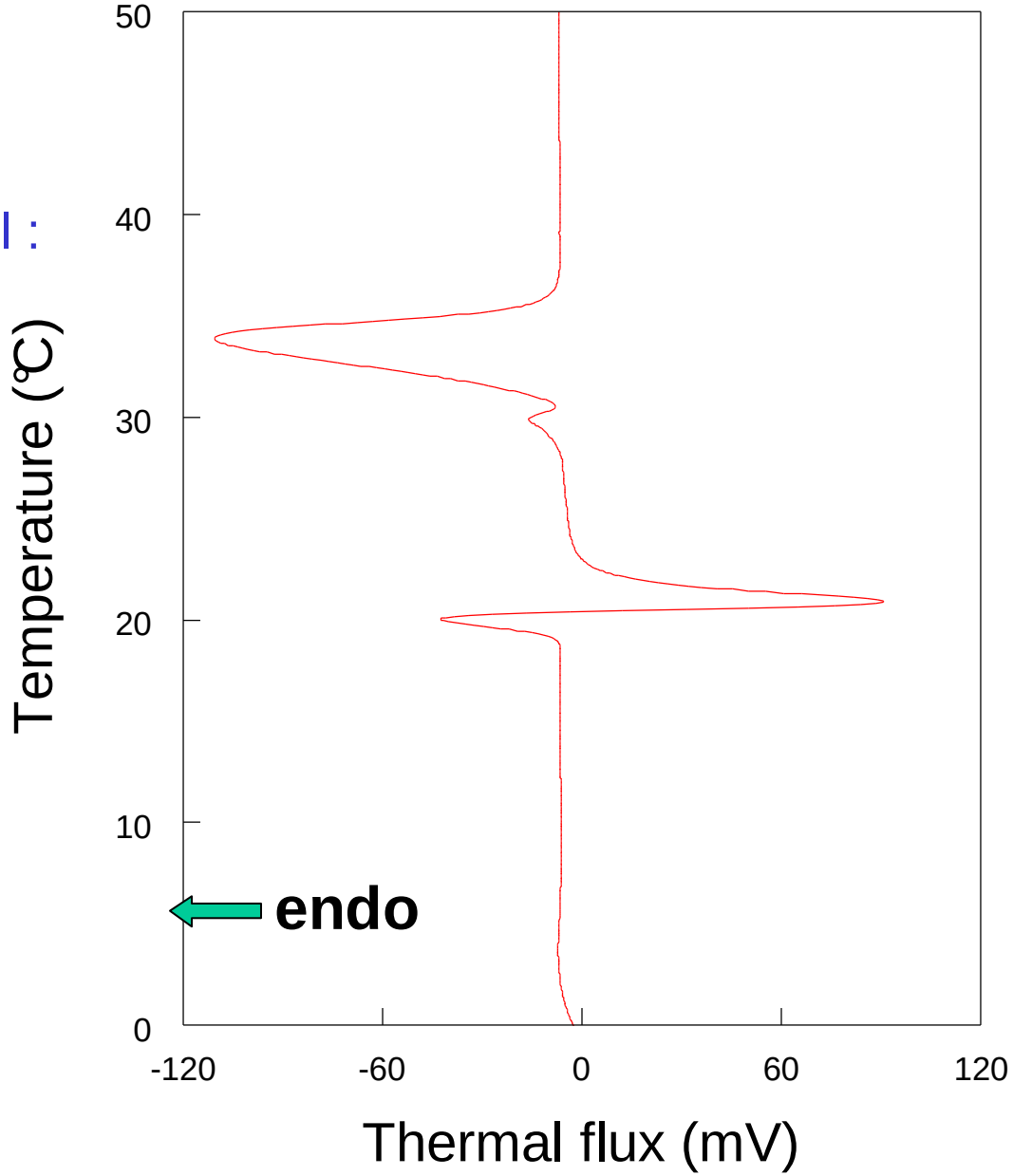
Triglyceride Polymorphism

PPO

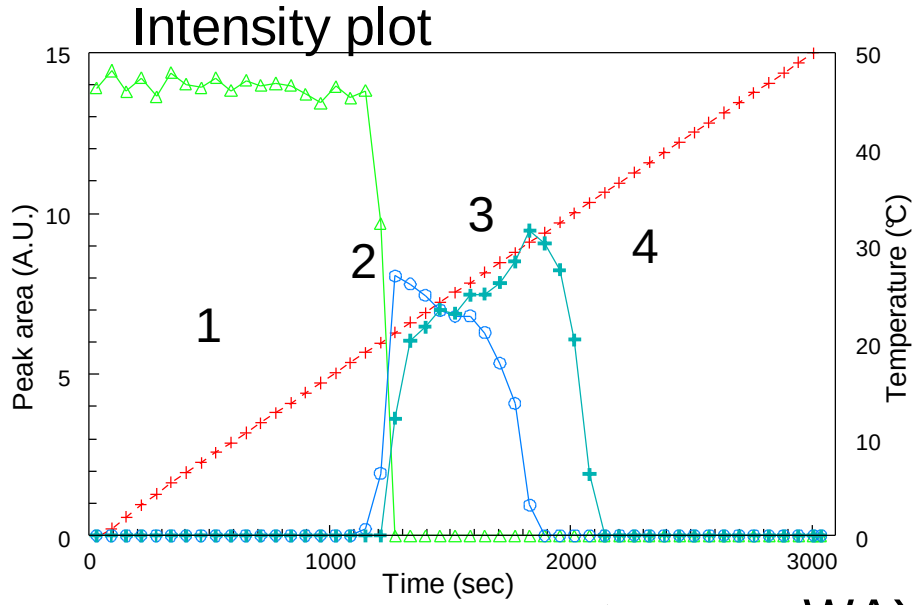
1,2-diPalmitoyl, 3-Oleyl glycerol :

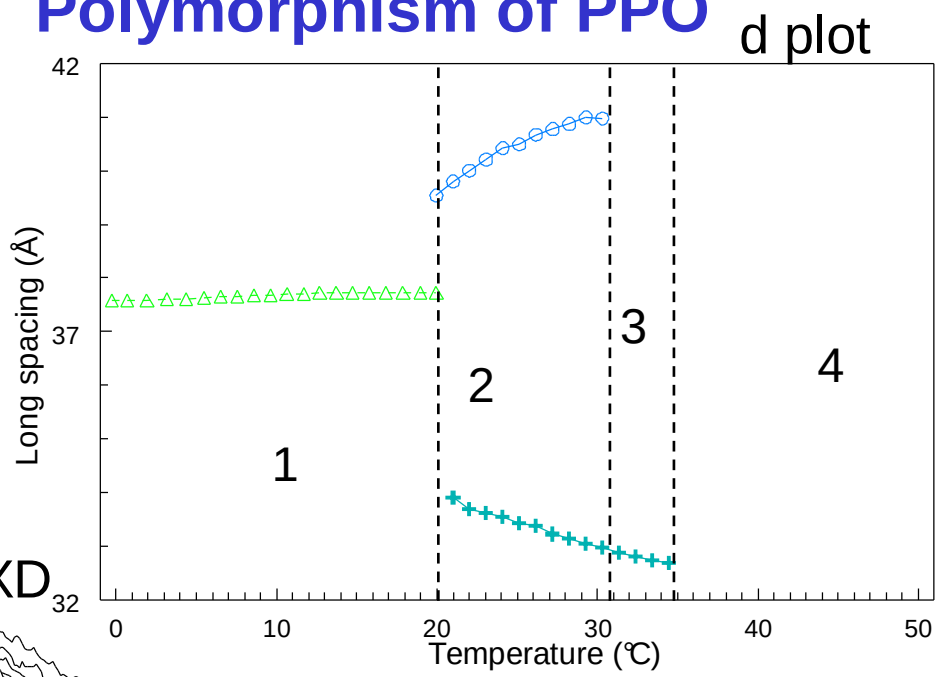
Monotropic transitions

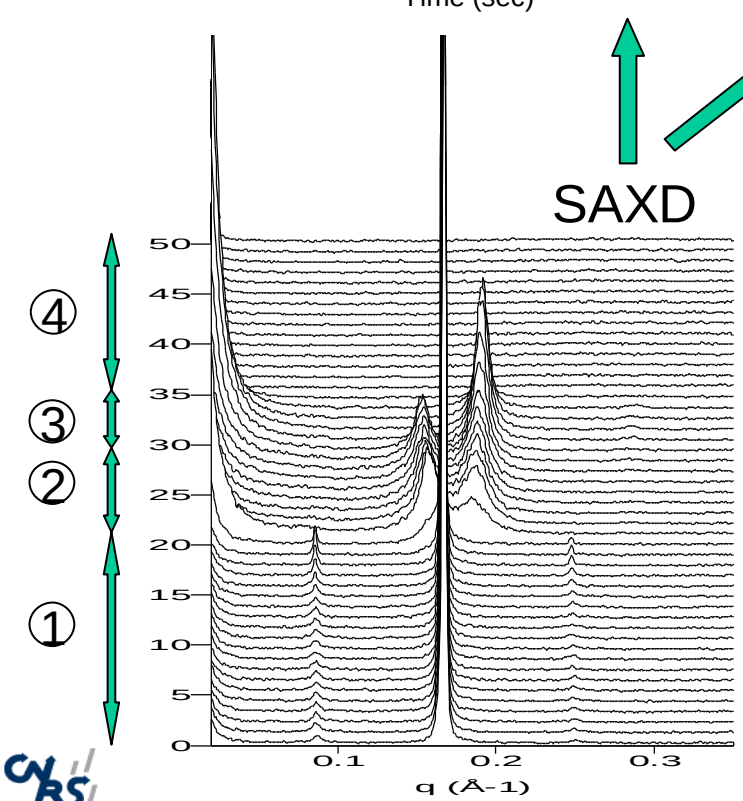


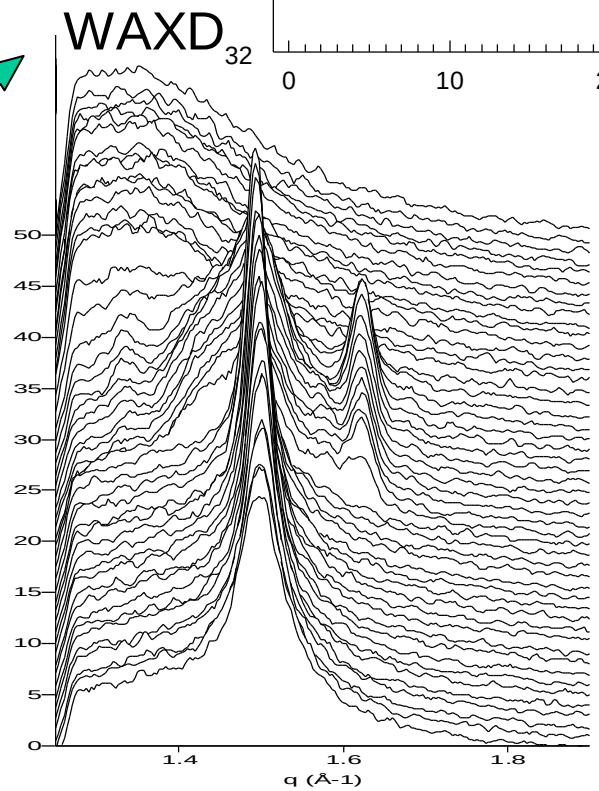


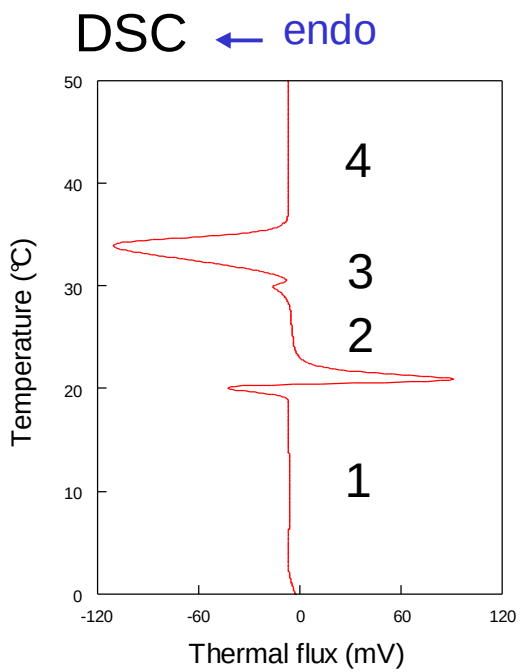
Polymorphism of PPO











An exemple of lipid polymorphism : PPO Triacylglycerol

unstable form
α (3L) 75 Å

20°C →

intermediate form
β'$_2$(2L)39-41 Å

→

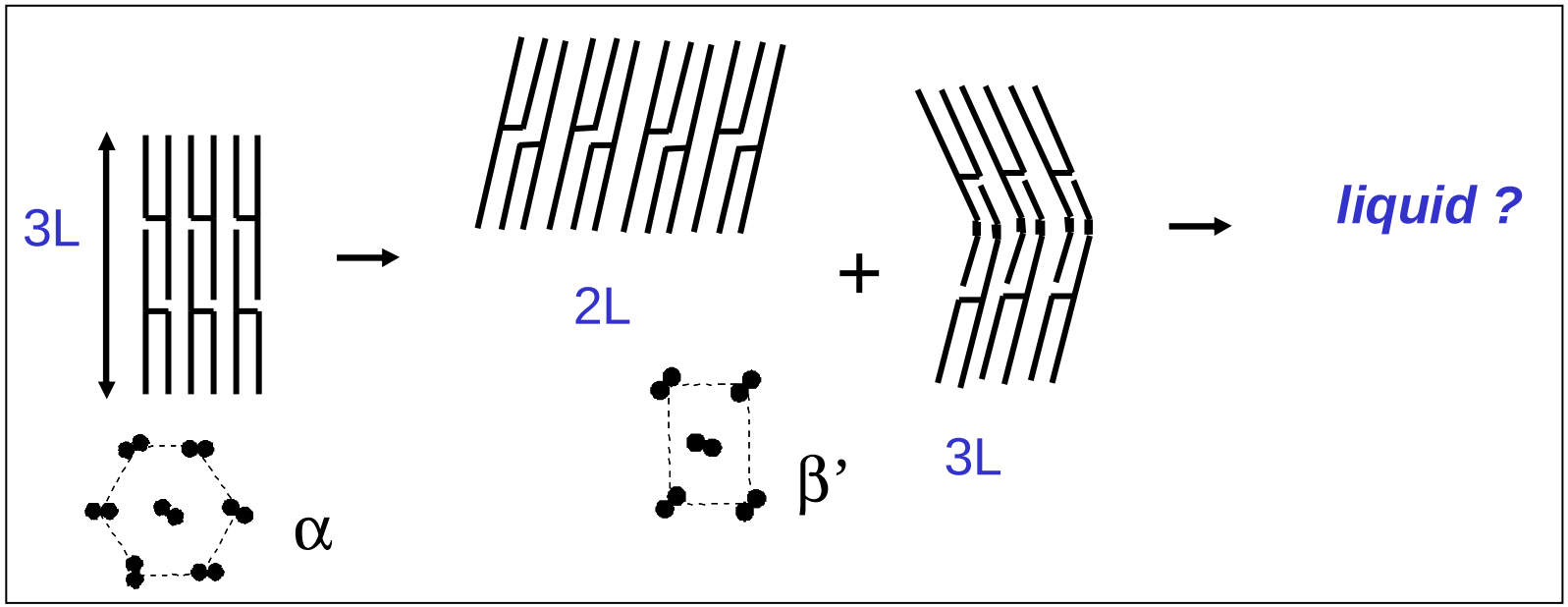
30°C →

21°C →

stable form
β'$_1$ (3L ?) 64-66 Å

35°C →

liquid

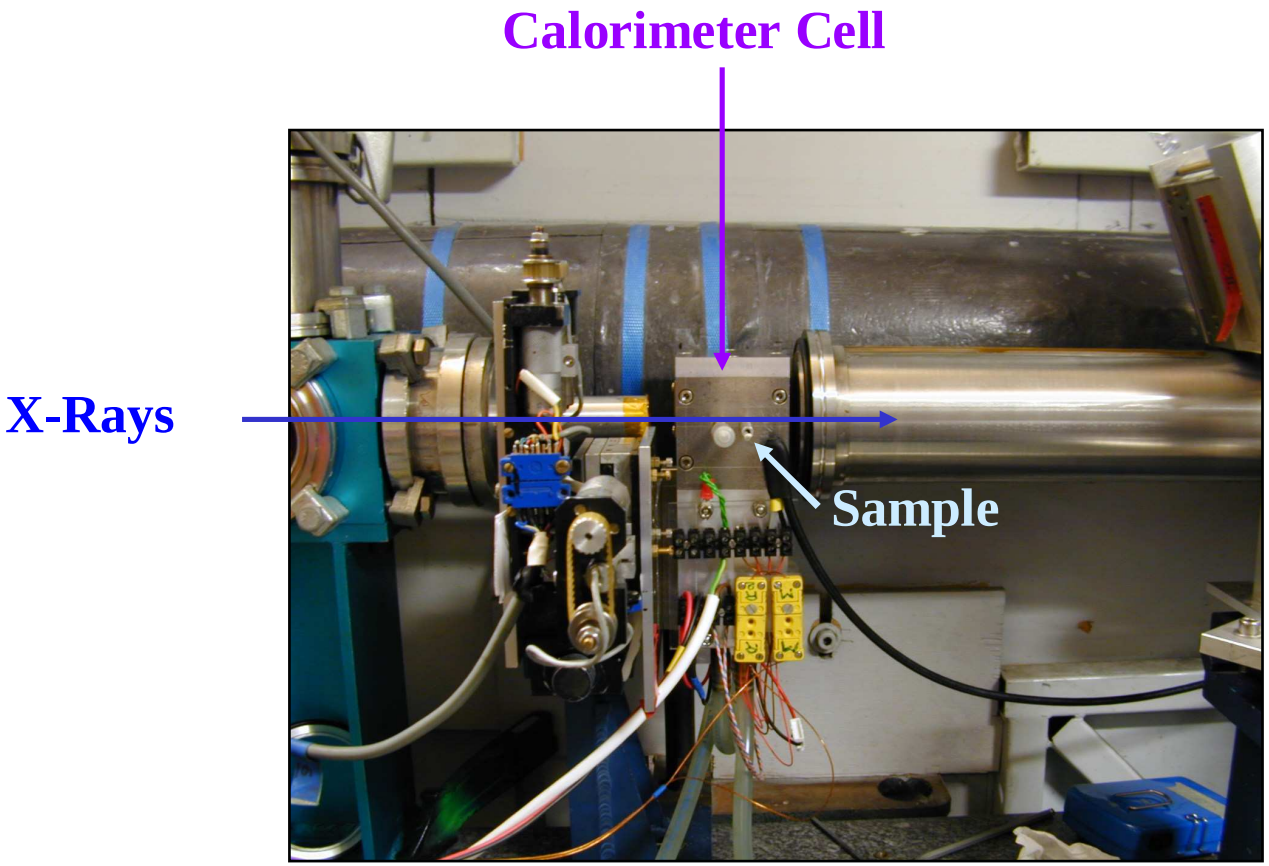


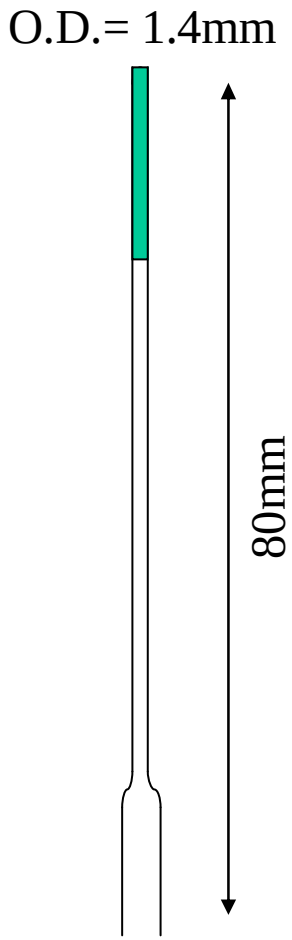
Ollivon et al., 2005, NATAS proceedings + J. Therm. Anal. and Cal. in press

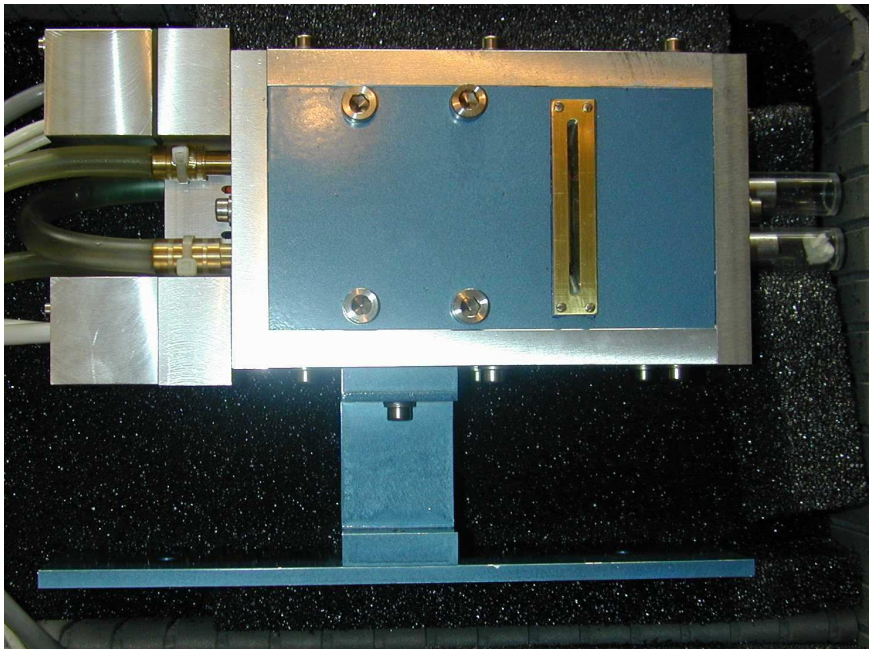
Coupled synchrotron time-resolved XRDT/DSC (SAXS and WAXS)

Insertion of a calorimeter cell on D22 beamline (LURE, Orsay, France)

Keller *et al*., 1998, J. Thermal Anal., 51:783-791.







Microcalix

T range : –30 to +230℃
Scan rates : 0.01– 20℃/min

Sample: 0.5 –30 mg (µl)

Sample change: 2 sec.

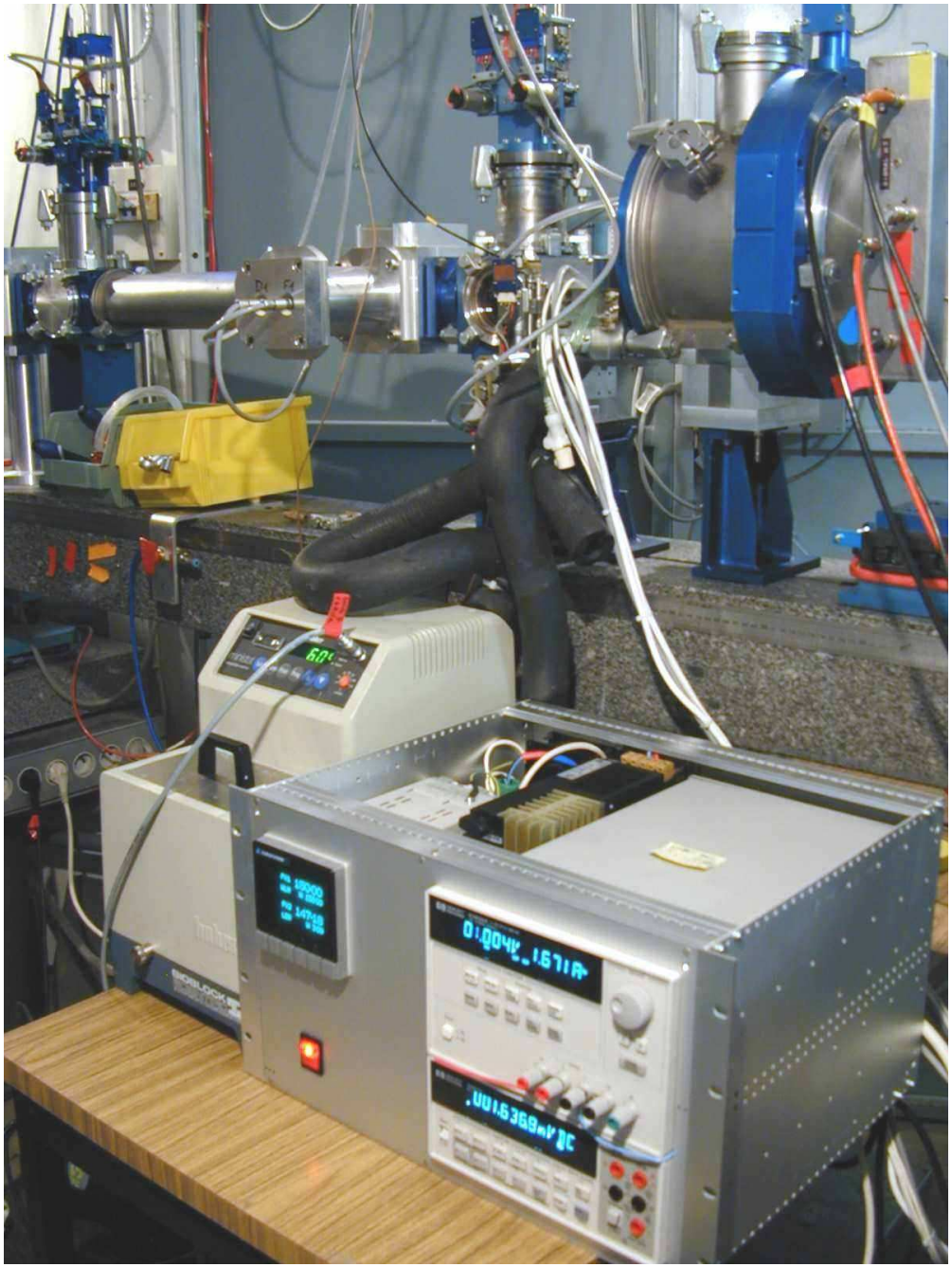
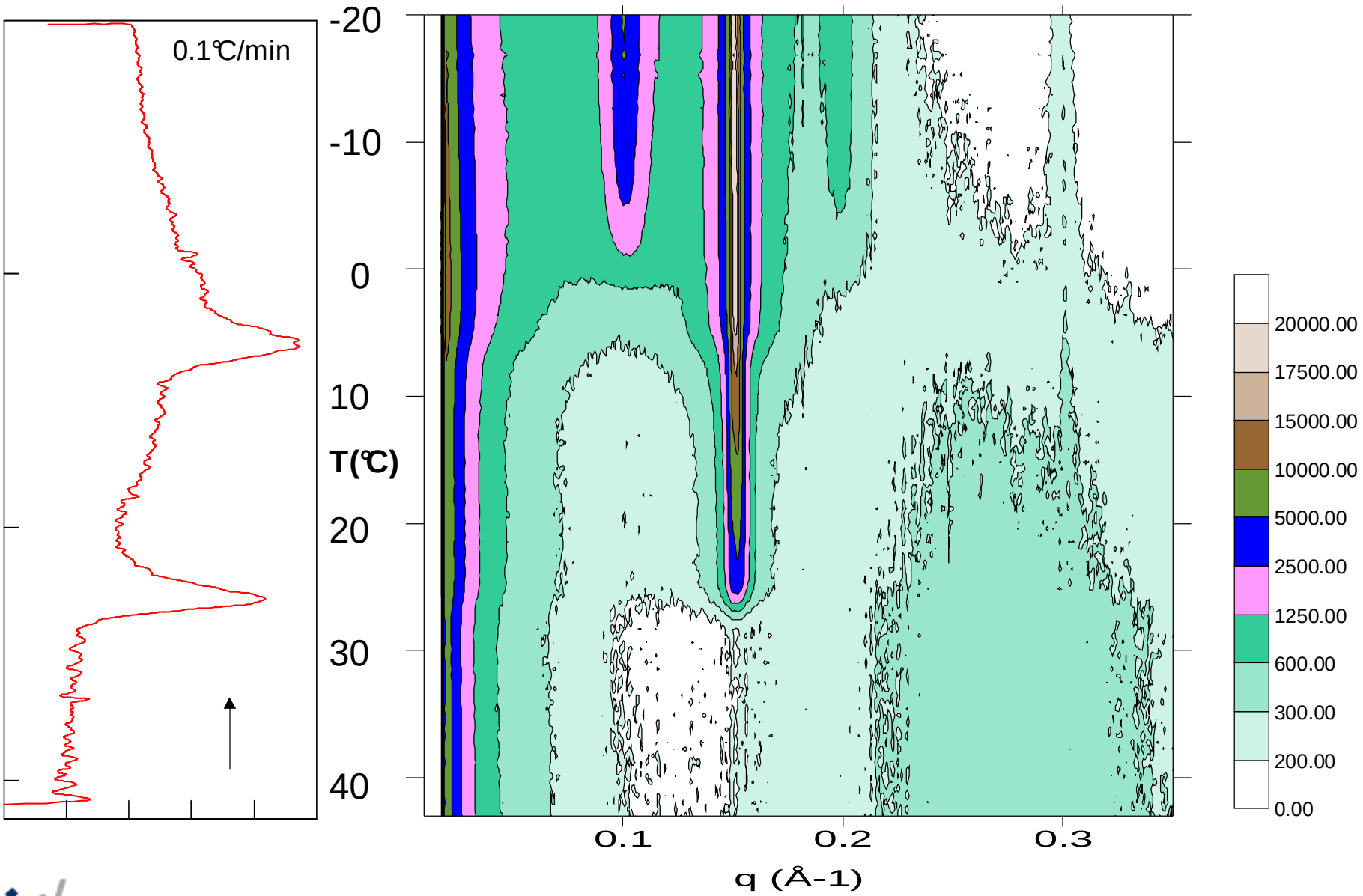


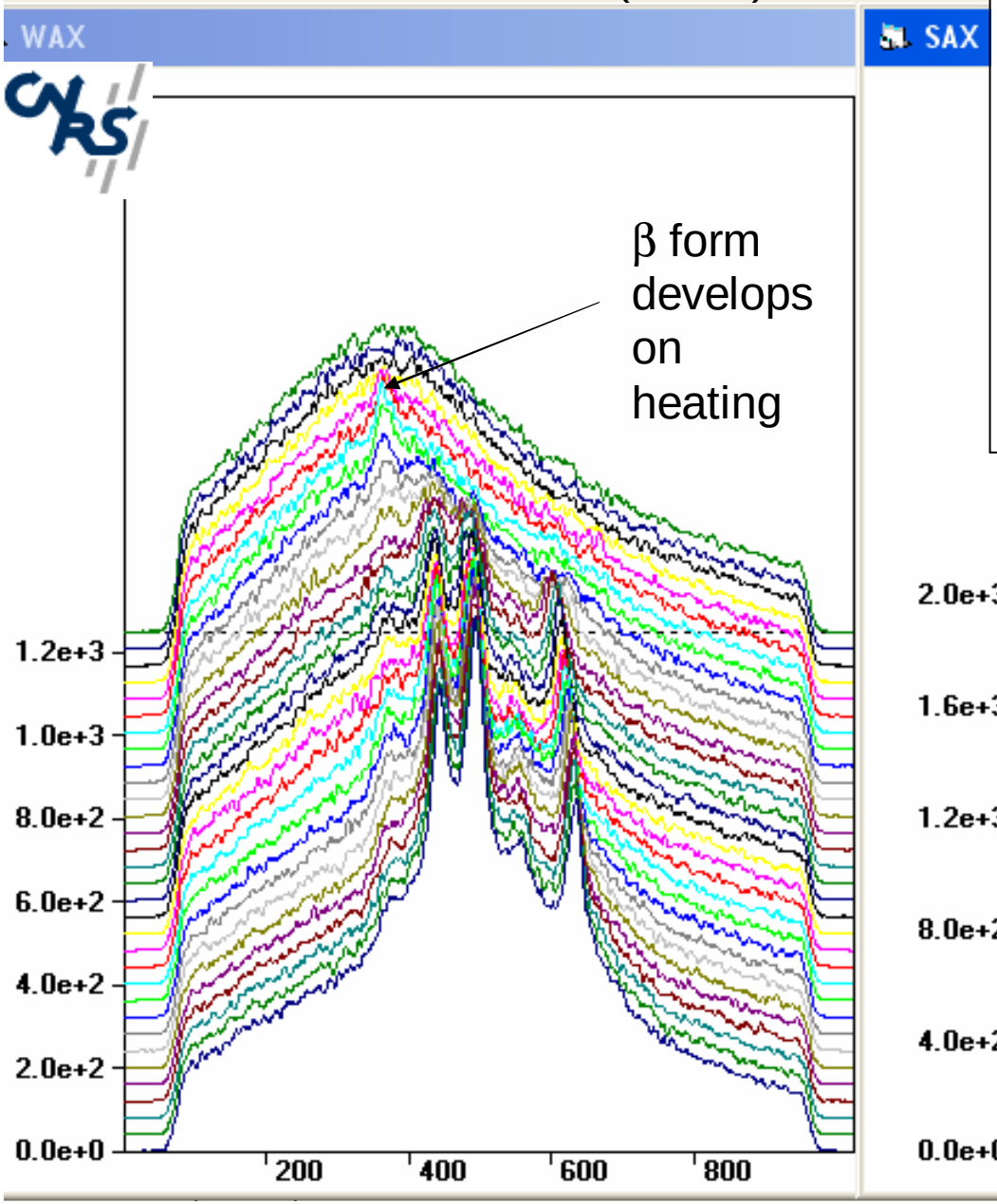
Figure 3

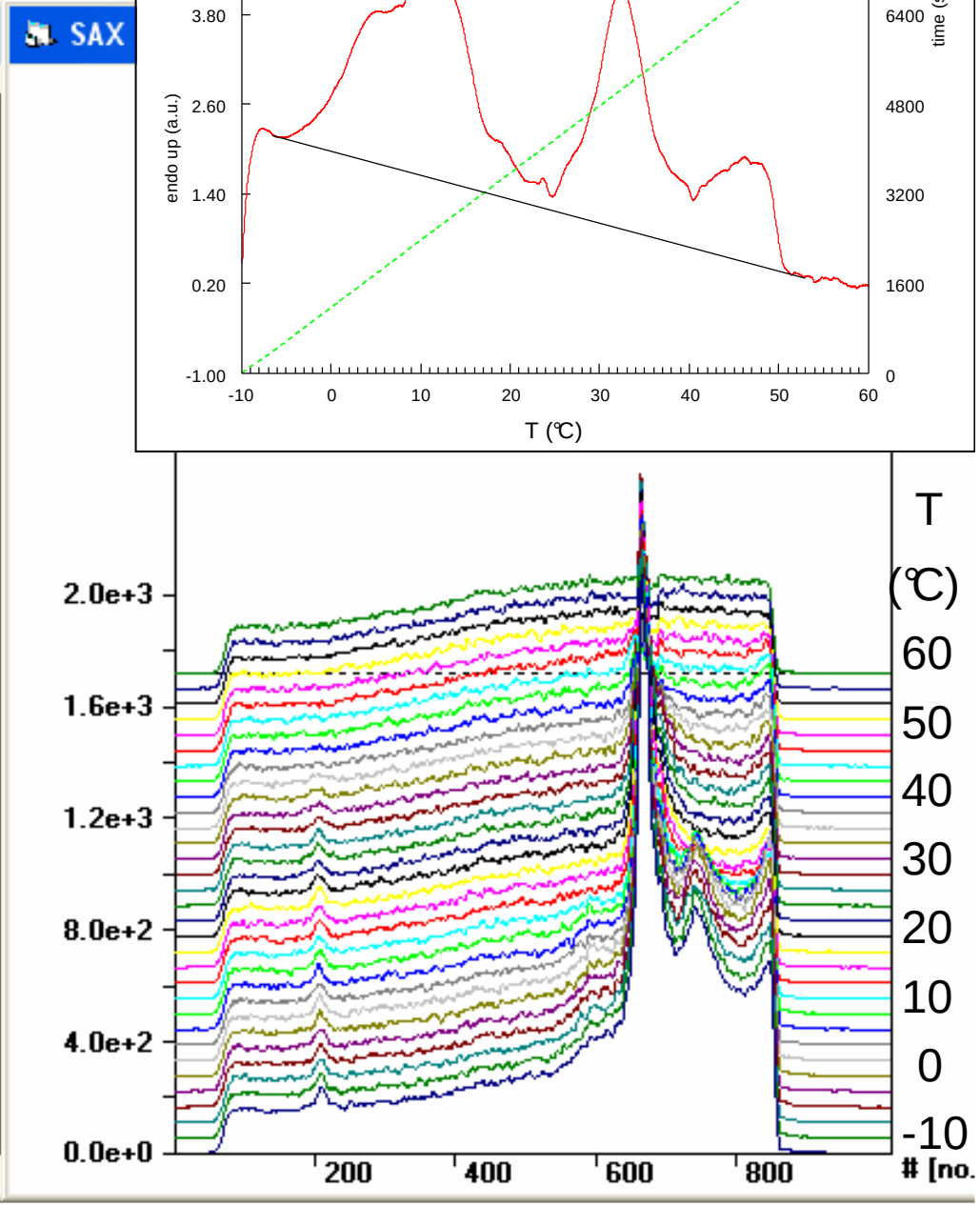


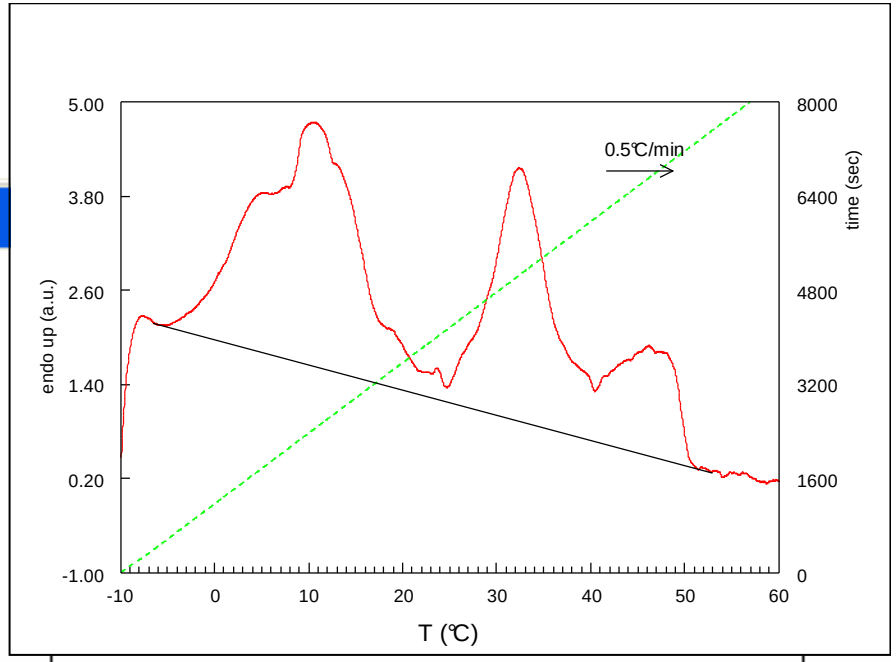
C.L. Chong et al., Eur. J. Lipid Sci. Tech. 2007, 410-421

PALM OIL heating from -10 to 55℃ at 0.5℃/min

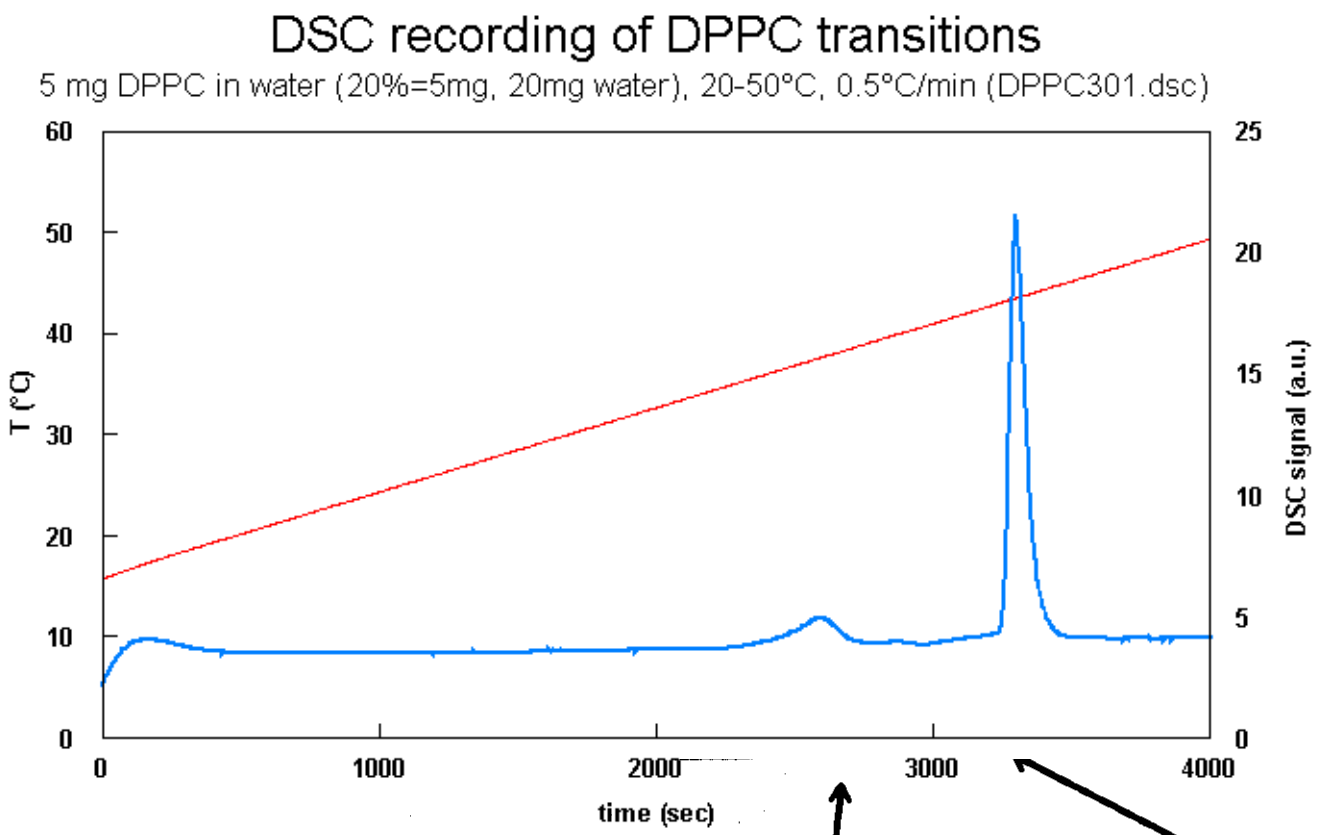
Chong et al. in preparation (in lab.)



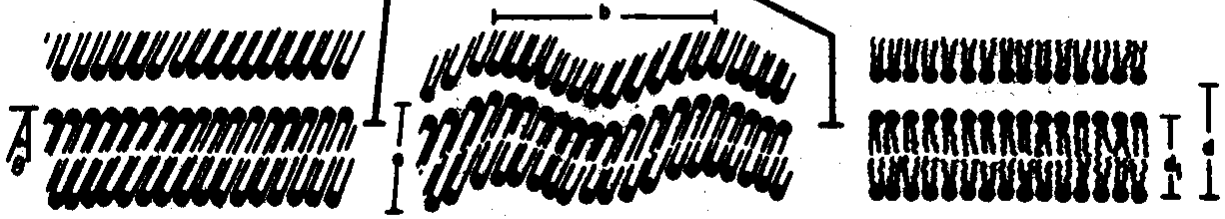




Emulsifiers (Lecithin), Dipalmitoylphosphatidylcholine = DPPC
25mg sample, 80% water, heating, (1 hour expt). 5mg DPPC

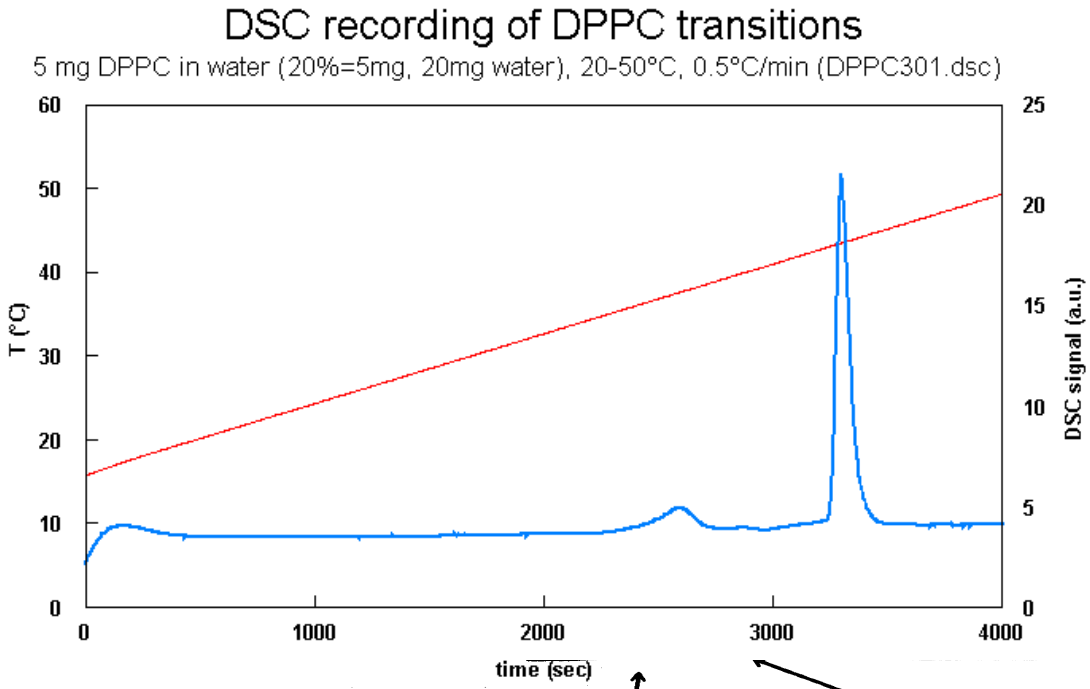


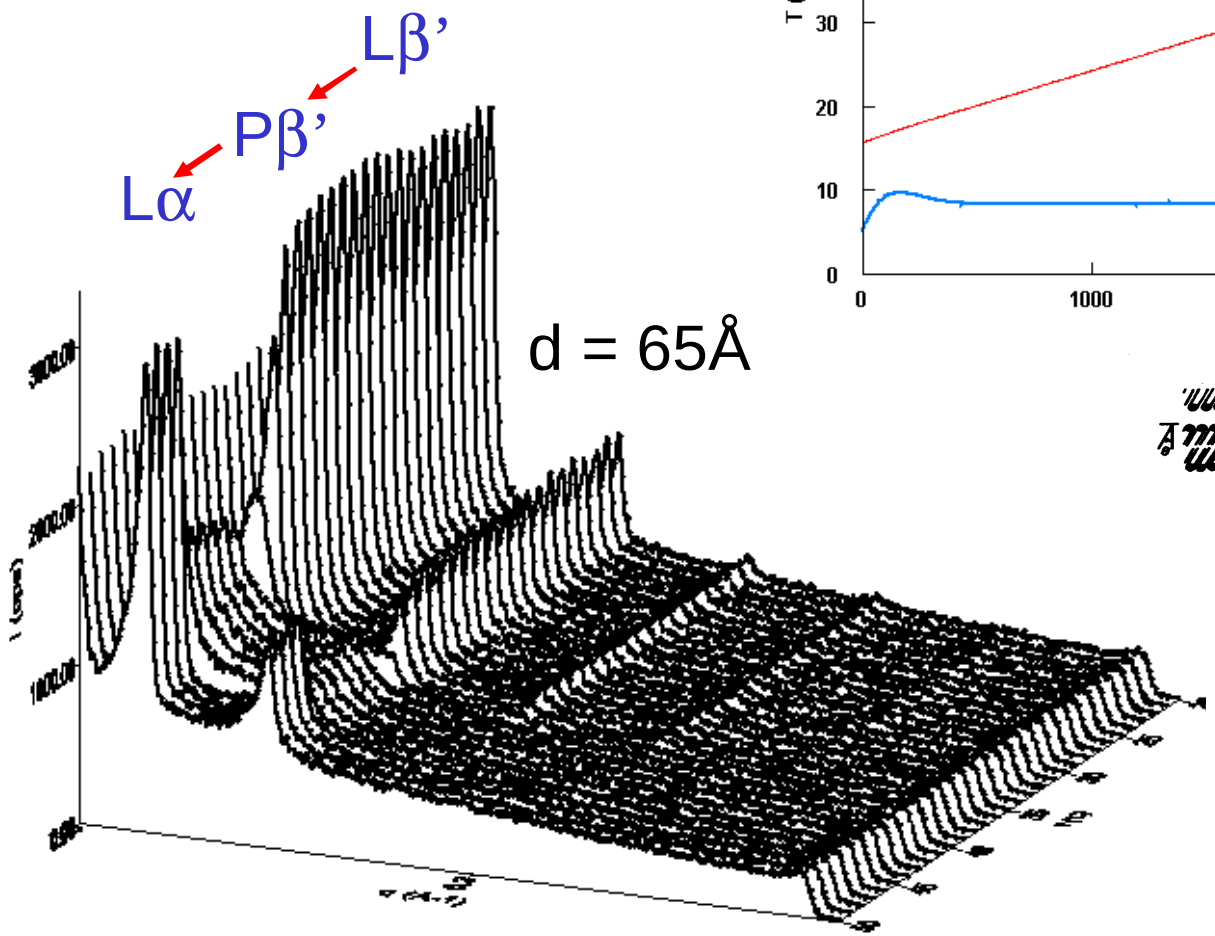
d = 65Å

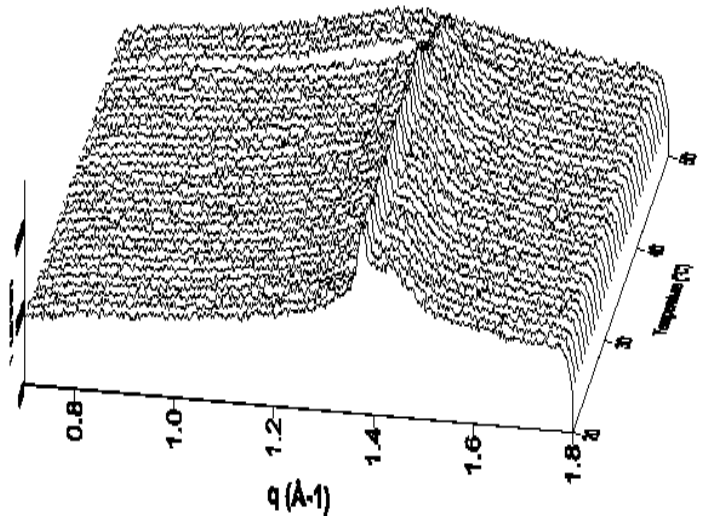


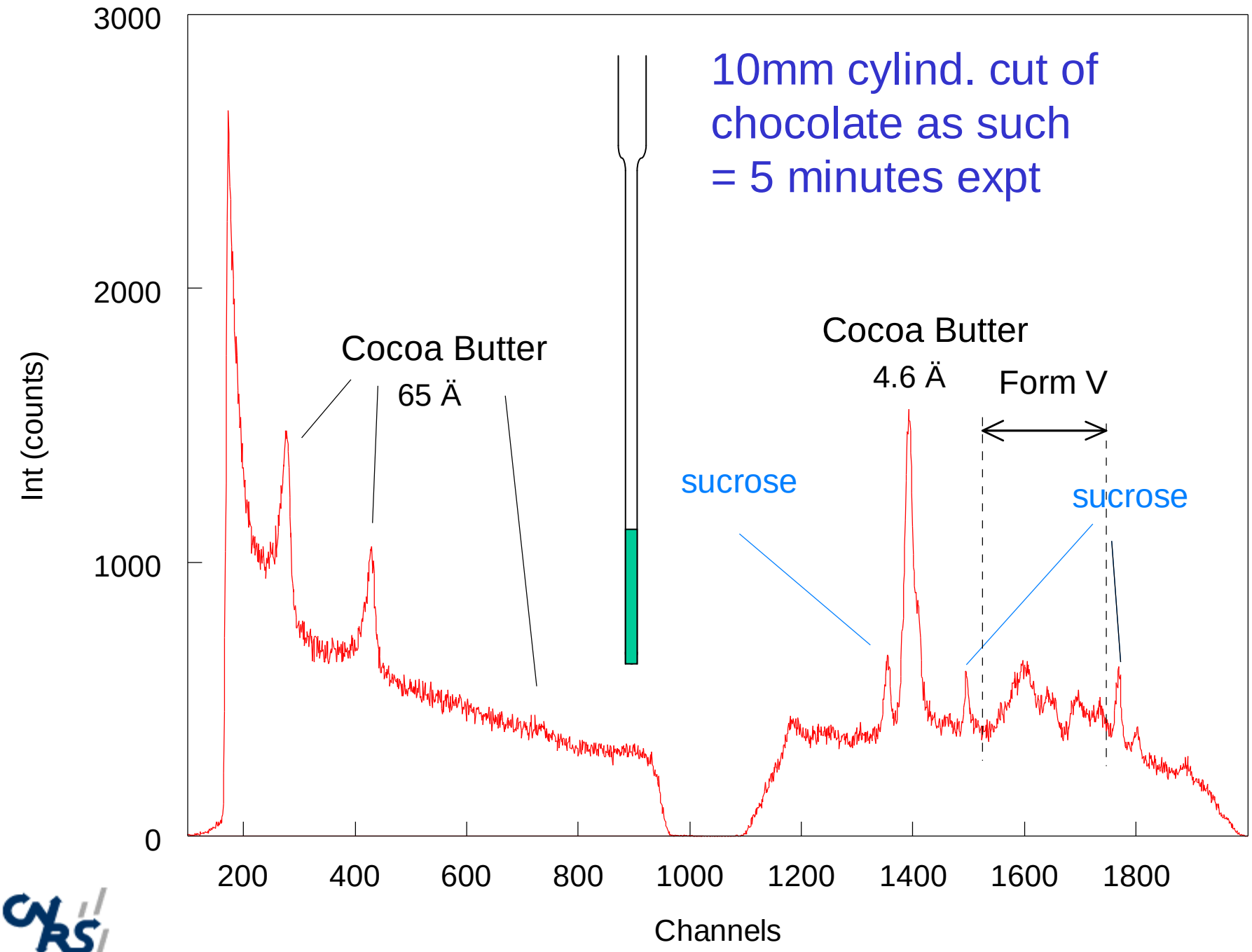
Lβ’ Pβ’ Lα

Emulsifiers (Lecithin), DPPC 5mg, 80% water, heating, simultaneous SAXS and WAXS

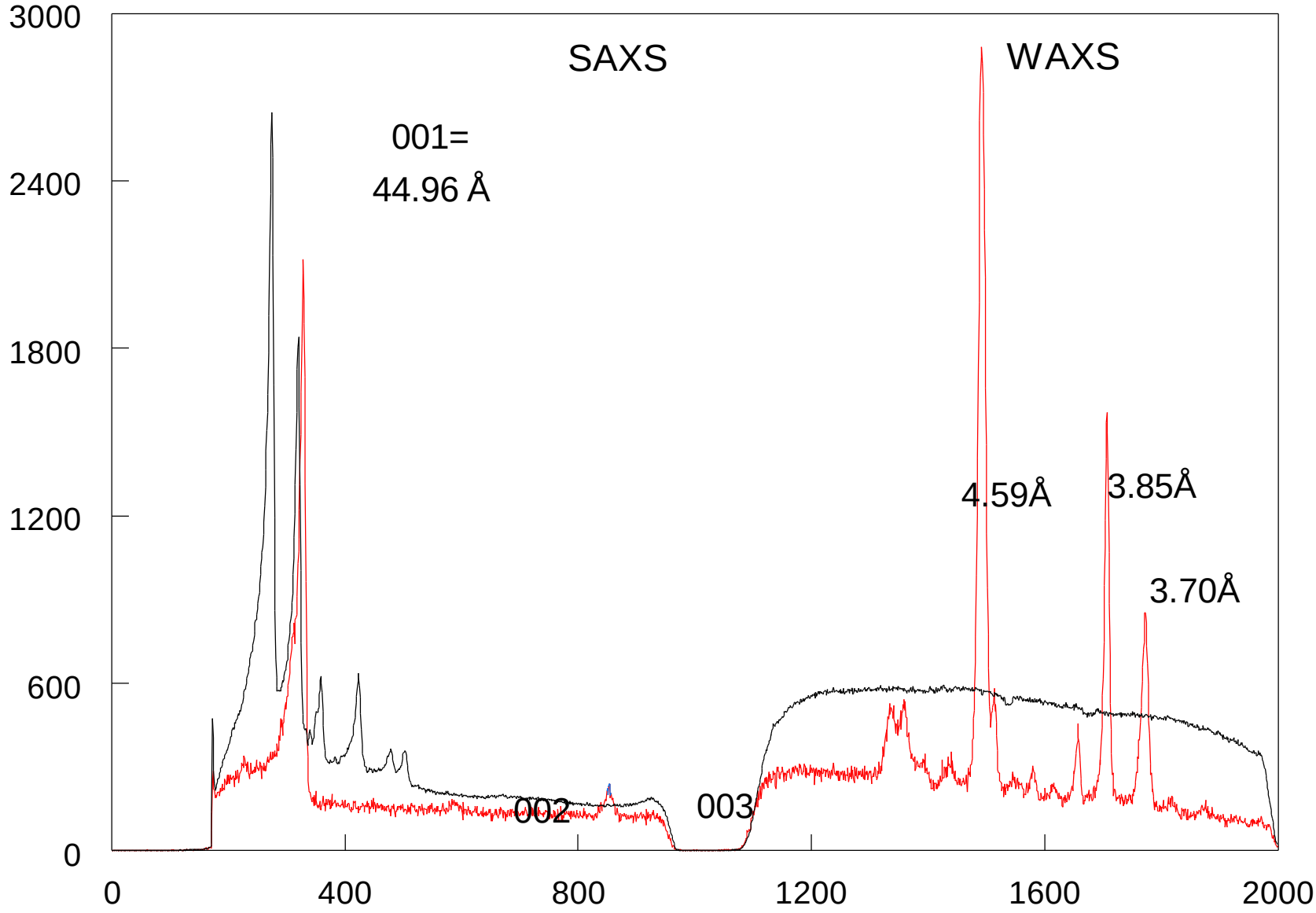







10mm cylind. cut of chocolate as such = 5 minutes expt
Cocoa Butter
65 Ä
Cocoa Butter
4.6 Ä
Form V
sucrose
sucrose
Int (counts)
Channels
3000
2000
1000
0
200
400
600
800
1000
1200
1400
1600
1800

SSS tristearin standard beta form



When do we need synchrotron X-ray to interpret lipid DSC?

MWs — MICROCALIX is used with — < 1KW

Synchrotron source for :

Identification at q < 0.05 Å-1 (d > 120Å)

Resolution at low q

(e.g. ΔD = 1Å at 50Å)

Complex phases identification

Time resolved experiments

(µsec, msec)

Highly diluted samples or

low electron density or

weak differences of electron densities

Interfaces studies

Bidimensional detection (e.g. oriented samples)

Regular X-ray source for :

Coupled DSC-SAXS-WAXS studies

Identification at q > 0.05 Å-1 (d <120Å)

Time-consuming studies (e.g. > 1hr)

at equilibrium

slow DSC scan

Slow evolutions of sample

Special conditions :

Dry sample, T, time

Samples difficult to prepare

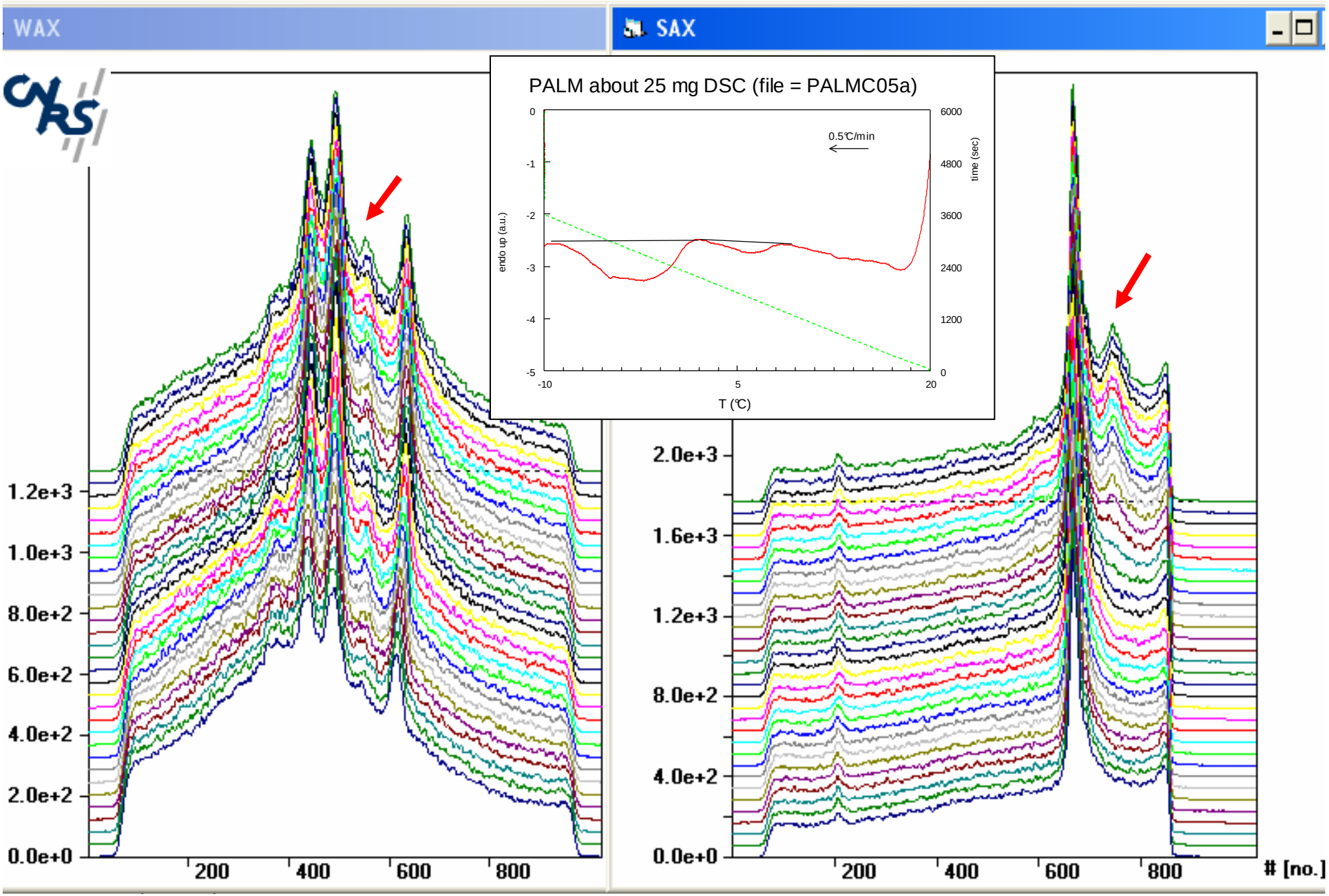
Convenience (at home studies)

Concentrated samples

Confidentiality, Cost, Fastness

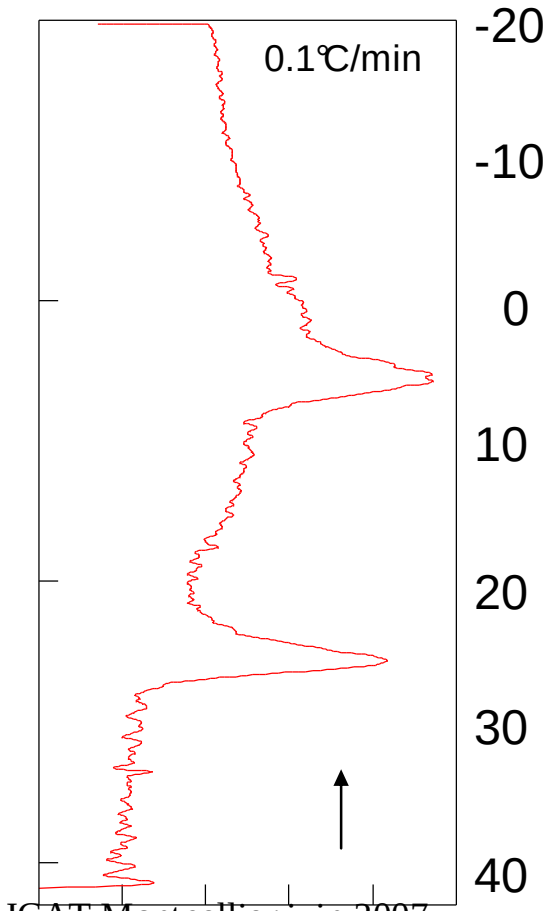
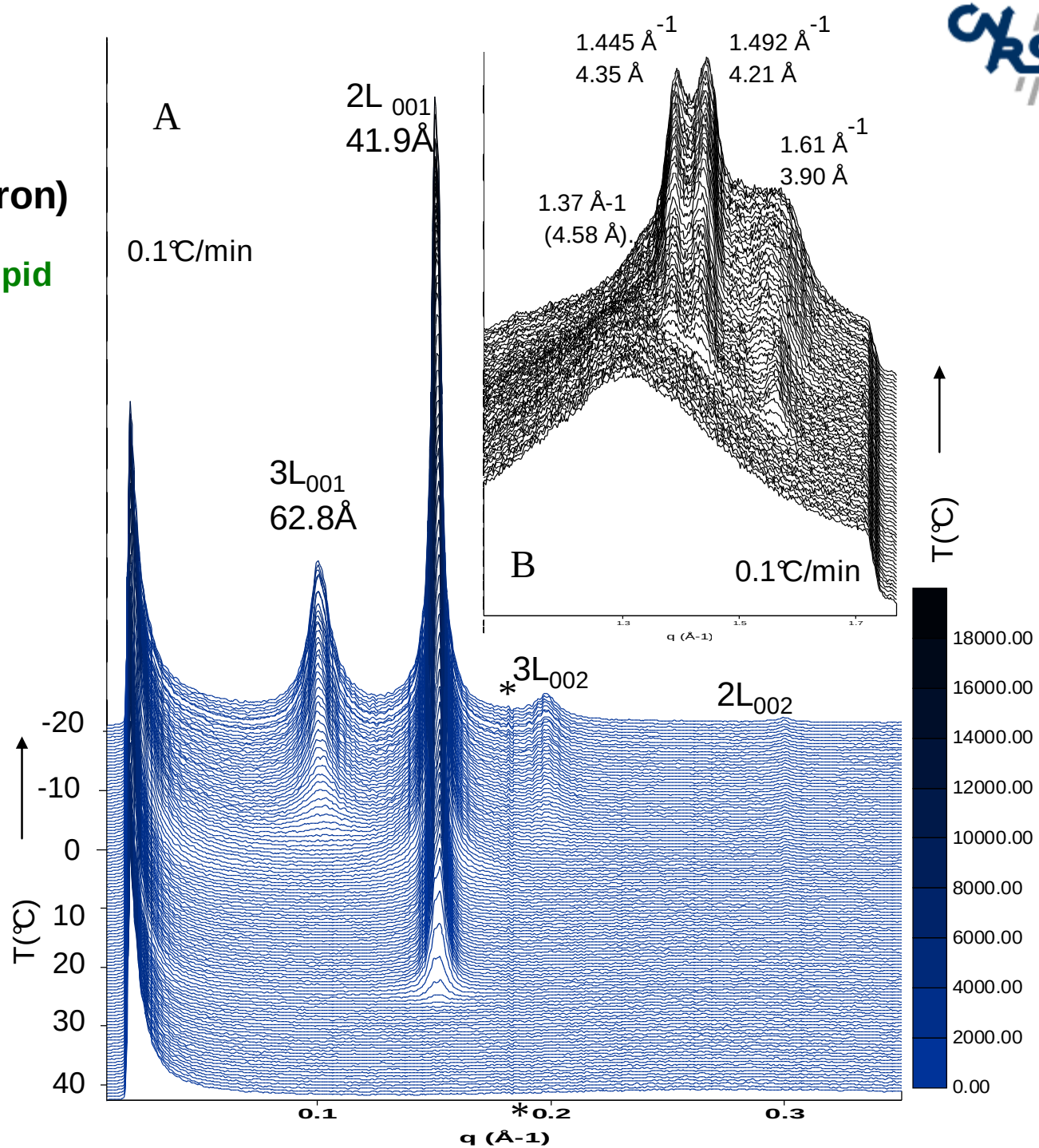
Thank you for your attention

PALM cooling from 20℃ to -10℃ at 0.5℃/min + platea u at -10℃ (file = PALMC05a)



PALM OIL cooling

from 40℃ to -20℃

at 0.1℃/min (Synchrotron)

C.L. Chong et al. Eur. J. Lipid Sci. Tech. 2007, 410-421