



	Experiment title: Crystal structure determination of the metastable beta-prime phases of the 1,3 disaturated-2-unsaturated	Experiment number: CH-1731
Beamline: ID31	Date of experiment: A From: 12-05-2004 to: 17-05-2004 B From: 16-12-2004 to: 20-12-2004	Date of report: 22-02-2005
Shifts: 15 (A) 9 (B)	Local contact(s): Irene Margiolaki	<i>Received at ESRF:</i>

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Report:

Session A

The technical settings at the start of the experiments were $\lambda = 1.25007116 \text{ \AA}$, zero shift = 0.00037719 °2 θ . The asymmetry of the peak-profile caused by the instrument can be taken (for GSAS) as: S/L = 0.0012 and H/L = 0.0027. Beam width = 2.5 mm and beam height 1.0-1.5 mm. Because a multiple-sample environment was scheduled, no goniometerheads were available, so making alignment of the capillaries a somewhat difficult task. According to the local contact and Dr Fitch, the velocity of spinning was so high that the statistics were OK even when the sample was wobbling a bit.

During the first trial experiments at room temperature, serious sample degradation was observed, and in particular mono-unsaturated triglycerides were suspected to be affected by the high radiation flux. To avoid radiation damage, it was decided to cool the samples to 250 K, after preparing the wanted (meta-stable) polymorph, and to translate the capillary after each scan. Though temperature overshoot could not be avoided, an overall acceptable temperature control (1-3 °C) was feasible.

For data collection a general strategy was adopted, consisting of scanning with 4-5°/min a set of overlapping 2 θ intervals (0-50, 10-50, 20-50, 30-50 and 40-50 °2 θ). In most cases after each scan the capillary was translated 2.5 mm. Meta-stable samples were usually measured up till 40 ° 2 θ (the same strategy). All individual and combined scans were binned at 0.004 °2 θ

Data collection was carried out for several types of triglycerides, with β , β' , γ and δ denoting the basic type of polymorph and the subscripts '1' and '2' denoting a lower-melting and higher melting phase, respectively.

- 1) *cis*-9-monounsaturated triglycerides: 1,3-distearoyl-2-oleoylglycerol (Pseudo- β' -SOS), 1,2-distearoyl-3-oleoylglycerol (SSO, precise phase not clear), 1,3-dipalmitoyl-2-oleoylglycerol (γ -POP, δ -POP, β_2 -POP)
1-stearoyl-2-oleoyl-3-arachidoylglycerol (β' -SOA) and 1,3-dimyristoyl-2-oleoylglycerol (β_2 -MOM)
- 2) *trans*-9-monounsaturated triglycerides: 1,2-distearoyl-3-elaidoylglycerol (β_1' -SSE, β_2 -SSE), 1,3-distearoyl-2-elaidoylglycerol (β -SES), 1,2-dipalmitoyl-3-elaidoylglycerol (β_1' -PPE, β_2' -PPE), and 1,3-dipalmitoyl-2-elaidoylglycerol (β_1' -PPE, β_2 -PEP)
- 3) the *cis*-9-*cis*-12-unsaturated triglyceride 1,3-distearoyl-2-lineoylglycerol (γ -SlinS)
- 4) asymmetric mixed-chain saturated triglycerides: 1-stearoyl-2,3-dipalmitoylglycerol (β' -SPP, β_2 -SPP), 1-lauroyl-2,3-dimyristoylglycerol (β_1' -LMM, β_2 -LMM), 1-heptadecanoyl-2,3-pentadecanoylglycerol (β' -171515, β -171515)
- 5) symmetric mix-chain saturated triglycerides: 1,3-dilauroyl-2-myristoylglycerol (LML), 1,3-distearoyl-2-myristoylglycerol (β' -SMS, β_1' -SMS, β_2' -SMS), and 1,3-distearoyl-2-lauroylglycerol (β' -SLS, β -SLS)
- 6) the monoacid trisaturated triglyceride tricapryloylglycerol (C888)
- 7) the fatty-acid soap triethanolamine stearate (Y283)
- 8) the material Y45

Analysis of the data afterwards pointed out that in most of the data sets an oversaturation of the detector (max. 100000 counts) had occurred in the low 2θ region. After discussion with the local contact, some additional data collection time was offered and effectuated as session B.

Session B

The technical settings at the start of the experiments were $\lambda = 1.2499263 \text{ \AA}$, zero shift = -0.00052865 $^\circ 2\theta$.

To avoid overflow of the detector, always the first peak was monitored and the beam size adjusted accordingly. Also to avoid sample degradation, after preparation of the (metastable) phase, data collection was carried out at 250 K. For data collection the general strategy of session A was adopted, consisting of scanning the set of overlapping 2θ intervals (0-50, 10-50, 20-50, 30-50 and (in most cases also) 40-50 $^\circ 2\theta$, though at a higher scanning speed (10 $^\circ$ /min) than in session A. After each scan the capillary was translated 2. mm. All individual and combined scans were binned at 0.004 $^\circ 2\theta$

Data have been collected for the following materials:

- 1) *cis*-9-monounsaturated triglycerides: 1,3-distearoyl-2-oleoylglycerol (Pseudo- β' -SOS, β_2 -SOS), 1,2-distearoyl-3-oleoylglycerol (β_1' -SSO) 1,3-dipalmitoyl-2-oleoylglycerol (δ -POP, β_2 -POP)
1-stearoyl-2-oleoyl-3-arachidoylglycerol (β' -SOA) and 1,3-dimyristoyl-2-oleoylglycerol (β_2 -MOM, β_2' -MOM, β_1' -MOM, α -MOM), 1-palmitoyl-2-oleoyl-3-stearoylglycerol (β' -POS), 1,3-dilauroyl-2-oleoylglycerol (β' -LOL)
- 2) *trans*-9-monounsaturated triglycerides: 1,2-distearoyl-3-elaidoylglycerol (β -SSE), 1,2-dipalmitoyl-3-elaidoylglycerol (β' -PPE),
- 4) asymmetric mixed-chain saturated triglycerides: 1-stearoyl-2,3-dipalmitoylglycerol (β' -SPP, β -SPP), 1-lauroyl-2,3-dimyristoylglycerol (β' -LMM), 1-heptadecanoyl-2,3-pentadecanoylglycerol ($\beta?$ -171515)
- 5) symmetric mix-chain saturated triglycerides: 1,3-dipalmitoyl-2-stearoylglycerol (β' -PSP), 1,3-distearoyl-2-myristoylglycerol (β' -SMS), and 1,3-distearoyl-2-lauroylglycerol (β' -SLS)
- 6) the monoacid trisaturated triglyceride tricapryloylglycerol (C888)
- 7) Ivory coast cocoa butter (β -6) phase
- 8) The organic material Z1000.
- 9) The liquid crystalline materials LC-1, LC-4, L32, L33 and L68