ESRF	**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**: From: 12-05-2004 to: 17-05-2004	**Date of report**: 18-08-2004
Shifts: 15	**Local contact(s)**: Irene Margiolaki	*Received at ESRF:*

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Report:

The technical settings at the start of the experiments were λ = 1.25007116 Å, zero shift =0.00037719 º2θ. 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 overal acceptable temperature control (1-3 ºC) was feasible.

For data collection a general strategy was adopted, consisting of scanning with 4-5°/min a set of overlapping 2θ intervals (0-50, 10-50, 20-50, 30-50 and 40-50 º2θ). In most cases after each scan the capillary was translated 2.5 mm. Meta-stable samples were usually measured up till 40 ° 2θ (the same strategy). All individual and combined scans were binned at 0.004 º2θ

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-2,3pentadecanoylglycerol (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. Meanwhile, this problem has been discussed with the local contact and some additional data collection time in the near future has been offered.

Although several data sets have been indexed and structure determinations have been started, final refinement Rietveld refinements will be carried out with the new data.