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|  | <b>Experiment title:</b><br>Crystal structure determination of meta-stable beta-prime and stable beta phases of triacylglycerol constituents of | <b>Experiment number:</b><br>CH-1616 |
| <b>Beamline:</b><br>BM01B  | <b>Date of experiment:</b><br>From: 21-09-2003 to: 23-09-2003   | <b>Date of report:</b><br>18-08-2004 |
| <b>Shifts:</b><br>6  | <b>Local contact(s):</b><br>Hermann Emerich   | <i>Received at ESRF:</i>             |
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## Report:

With respect to the experiments, the same settings as in session CH-1536 were used, except for the use of the cryostream as discussed below.

One of the goals of this session was to obtain some meta-stable phases of triacylglycerols. The sample (in a 1.0 or 1.5 mm cap.) should be completely molten. As it turned out, the cryostream available at BM01B heated the capillary only locally so upon cooling re-crystallization into the stable phase took place. Eventually, this problem was solved by heating the capillary for a few minutes at  $T = 60\text{ }^{\circ}\text{C}$  on a hot plate, quenching it in a water/ice mixture and drying it. The capillary was fixed with red clay because of the low melting point of the wax ( $T_m = 52\text{ }^{\circ}\text{C}$ ). One of the meta-stable phases wanted was the  $\gamma$  phase of 1,3-distearoyl-2-oleoylglycerol (SOS). A short low angle test scan revealed the presence of the expected  $\gamma$  phase long spacing ( $d \sim 70\text{ \AA}$ ) and complete data collection was carried out at  $T = 293\text{ K}$  in the interval  $0.5 - 35.0\text{ }^{\circ} 2\theta$ .

In spite of several attempts, one other meta-stable phase of 1,3-dilauroyl-2-oleoylglycerol (LOL) that had been observed in laboratory experiments did not crystallize, for unknown reasons.

Also, four complete data sets have been collected of the beta-stable triacylglycerols SMS, SLS, LLM and MML. Meanwhile, the pattern of LLM has been indexed and the crystal structure has been solved. Surprisingly, the data of MML showed several sets of low-angle peaks with a small difference in  $2\theta$  position, suggesting the presence of two polymorphs, while in laboratory data just single peaks had been observed. The patterns of SMS and SLS are still being processed.