ESRF	**Experiment title:** High-resolution powder diffraction of the C_n C_{n+2} C_n (n = even) triacylglycerol series members β'-MPM and β'-PSP at low temperature.	**Experiment number**: 01-01-245
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Report:

Powder diffraction experiments at various low temperatures have been carried out for two homologous triacylglycerols with acronyms PSP (1,3-dehexadecanoyl-2-octadecanoyl-glycerol) and CLC (1,3-dedecanoyl-2-dodecanoyl-glycerol), both being in the β'phase, in order to study the temperature dependence in relation to a orthorhombic *vs.* monoclinic chain packing.

Sample preparation

Samples had been prepared at home in 1.5 mm capillaries because of the relatively poor scattering of this material. In order to amount this size capillary, the sample holder had to be drilled out. In addition to PSP and CLC also capillaries with the homologous series members LML and MPM had been prepared but the amount of shift time was insufficient to measure them as well.

Experiments

In this session twelve powderdiffraction patterns were recorded, on 12-07-2000 six patterns of the β'phase of PSP and on 13-07-2000 six patterns of the β'phase of CLC, both with the same temperature settings (T = 300 K , 100 K, 150 K, 200 K, 250 K and 295 K) and using the Janis cryostat. Experimental characteristics of this session were a 1/3 fill mode (70 - 90 mA), λ = 0.60044 Å, zeroshift = -.011 and beam spot 10 mm.

The experiments with both β'-PSP (exps. 1 - 6) and β'-CLC (exps. 7 - 12) were carried out according to the same protocol.

At room temperature (T = 300 K) a reference pattern of β'-PSP was collected (exp. 1) Visually, the β' phase was recognized and the max. 2θ estimated to be ~ 35°. After quenching the sample to T = 100K (within

a few minutes), and letting it stabilize for about 15 minutes, a second data set (exp. 2) was collected at this temperature. Visual inspection of this pattern suggested a phase different than the expected β' but analysis afterwards showed this to be incorrect. The misinterpretation can be attributed partly to the fact that the largest β' peak was not visible because of a 2θ = 0.5˚ underlimit imposed to protect the detector. A second reason was the problems encountered with binning the data files (see below). However, the general strategy was not affected by these problems. On basis of other synchrotron data (T = 250 K) available of homologous compounds and in view of the amount of time, it was decided to spend most time on data collection at T = 250 (exp. 3), to do shorther experiments with a reduced 2θ interval at T = 150 and T = 200 (exps. 4 and 5 respectively) and to carry out again a longer data collection (during the night) at T = 295. At this moment still no binning was possible.

As noted, data collection of CLC (exps. 7 -12) was carried out in a similar way. A reference pattern was made (exp 7) at T = 300 K in which the expected β' was recognized. Cooling to 100 K and data collection (exp 8) As expected, peaks broadened at T = 300 K were now clearly split. On basis of the binned _rt and _100 data and the experience with β'-PSP, it was again decided to make shorter scans at T = 150 and 200 K (exp. 9 and 10, max. 2θ = 10.5) and longer scans at T = 250 and T = 295 K (exps. 11 and 12, max. 2θ = 18.5 and 20.5 respectively). Splitting of peaks was still present at 250 K but at the final T = 295 pattern the splitting had vanished.

(Re-)binning

The detectorsystem, consisting of 4 detectors at a 1.1 °2θ intervals, is known to have small temperature-dependent offsets because of temperature fluctuations in the hutch (no airconditioning system at the time of data collection). According to Dr. Emerich, the offset corrections are in principle listed 'on file (temp.res) and applied 'automatically' in the binning procedure but, for some unknown reason, the existing temp.res file turned out to be incorrect so the actual correction had to be determined manually. This was carried out using the four detector files psp_rt.00, psp_rt.01, psp_rt.02 and psp_rt.03. After some trial and error, a resolved peak at ~ 2θ = 7.95 was selected, its position on the four files established, the relative difference calculated and the new corrections determined

detector file	position (2θ)	rel. diff (00-0x)
.00	7.968607	0.
.01	7.954937	0.01367
.02	7.972531	-.003924
.03	8.013769	-.045

	temp.res (old)	-	rel. diff	=	temp.res (new)
.01	-1.07812	-	.01367	=	-1.09179
.02	-2.1923	-	-.003924	=	-2.1884
.03	-3.3342	-	-.045	=	-3.289

Using the new settings satisfactory patterns were obtained and, eventually, all twelve data sets were binning using a step of 0.005º 2θ.

Preliminary Results

The patterns have been analyzed using a special indexing program (Peschar, unpublished results). All six CLC patterns have been indexed while also for most PSP patterns an acceptable indexing has been found. The cells found confirm the initial assumptions that the splitting of peaks, most clearly observable at CLC, increases with at lower T and can be interpreted in terms of monoclinic cells. A more detailed analysis involving atomic models is expected to be carried out soon.