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|  | <b>Experiment title:</b><br>High-resolution powder diffraction of various compounds         | <b>Experiment number:</b><br>CH855   |
| <b>Beamline:</b><br>BM16   | <b>Date of experiment:</b><br>from: 24-06-2000                               to: 27-06-2000 | <b>Date of report:</b><br>28-02-2001 |
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## Report:

In this session high-resolution powder diffraction patterns were measured at room temperature of thirteen different compounds, referred to below as MMM,  $\beta$ -PSP, COGHA, COMAL, Z1063, Z1070, Z1071, Z1083, Y154c, Y177, Y178, D37 and B13, respectively, on behalf of various on-going projects in the lab. The wavelength in most experiments was 0.99542 Å, except for D37, B13, Z1070 and Z1063 that were measured at 0.4997452 Å. Except for COGHA and COMAL, the samples were prepared in capillaries. All samples were rotated during exposure.

The pattern of  $\beta$ -1,2,3 tri-tetradecanoyl-glycerol ( $\beta$ -trimyristin or  $\beta$ -MMM) was collected in the interval 0-48.0° 2 $\theta$  with 0.6° 2 $\theta$  min<sup>-1</sup> and a sampling time of 100 ms during 5 hr. After data collection the scans were binned at 0.005° 2 $\theta$ . The pattern has been indexed using ITO and manual interference. Grid search and Rietveld refinement have been used to determine and refine the structure respectively. The final  $R_p$  value is 7.1%. A publication is in press (Van Langevelde et al., 2001).

Short data collections were carried out of the cocoa butters COGHA (from Ghana) and COMAL (from Malaysia), intended to support the analysis of SAXS measurements at the DUBBLE-CRG beam line. In either case thin sticks (~ 3 mm thick and 3 cm long) were carved from a block of the respective cocoa butters. Data collection was carried out in the interval 0-35.0° 2 $\theta$  with 0.6° 2 $\theta$  min<sup>-1</sup> and a sampling time of 100 ms during 1 and 2 hrs respectively. Although the cocoa butters are co-crystallized mixtures and the total exposure time was short, the diffraction patterns were of surprising good quality. Very recently, we even managed to index them.

The compound  $\beta$ -PSP is highly relevant in our research of cocoa butter and its constituent triacylglycerols because it is the only triacylglycerol of which both a stable  $\beta$ -phase and a stable  $\beta'$ -phase is available. Data was collected from 0- 48° 2 $\theta$ . The pattern has been indexed, an initial model has been found and is currently being refined.

The compounds Z1063, Z1070, Z1071 and Z1083, four differently substituted derivatives of trans,trans-1,4-bis[2-phenylethenyl]benzene [compounds 1, 8, 13 and 4 respectively from Yang et al (1990)] were measured with slightly different scan protocols and  $2\theta$  maxima (35., 35., 45. and 50. respectively), taking into account the quality of the patterns and the available beam time. The pattern of the compound Z1083 has been indexed and the structure has been solved. Structure determination of the other compounds is currently in progress.

In the framework of a project on the examination of the yellow coloured pigment Naples Yellow, commonly assumed to be lead antimony oxide ( $\text{Pb}_2\text{Sb}_2\text{O}_7$ ), the samples B13 and D37, originating from the historical Böcklin and Darmstadt pigment collections, have been measured upto 42 and 50 °  $2\theta$  respectively. The sample B13 turned to consist of a few known phases but in the D37 pattern the presence of six different crystalline phases has been established, including one not known before. A paper is in preparation.

Data collection for the compound Y154c, a Re-dioxolene complex, upto 45 °  $2\theta$  was problematic because the pattern was unexpectedly noisy, even at low  $2\theta$  and taking into account the good crystallinity and data collection time, and showed also a 'shoulder-like' behaviour at positions of completely resolved reflections. After additional experiments and analysis together with Dr. Masson, it was concluded that these phenomena could be attributed to bad particle statistics. At the time, this problem could not be solved because of the limited amount of sample and impossibility to grind it (complex is not stable when ground).

Two organo-metallic complexes (Y178 and Y177) were measured upto 45 and 88 °  $2\theta$  respectively. An analysis of the Y177 pattern on the spot showed a possible metallic contamination

## References

- Van Langevelde, A., Peschar, R. and Schenk, H. (2001). Structure of  $\beta$ -trimyristin and  $\beta$ -tristearin from high-resolution powder diffraction data. *Acta Cryst.* B57, In press.
- Yang, Z., Geise, H.J., Mehbod, M., Debrue, G., Visser, J.W., Sonneveld, E.J., Van't dack, L. and Gijbels, R. (1990). *Synthetic Metals* 39, 137-151.

