ESRF	**Experiment title:** High-resolution powder diffraction of asymmetric triacyl-glycerols of the types CnCnCn+2 (n=14,16)	**Experiment number**: 01-01-246
Beamline: BM1B	**Date of experiment**: From: 05-10-2000 to: 09-10-2000 (A) and 12-04-2001 to: 17-04-2001 (B)	**Date of report**: 31-08-2001
Shifts: 6 (A) 9 (B)	**Local contact(s)**: Hermann Emerich (A) Wouter van Beek (B)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Eva Dova, Kees Goubitz*, Rob Helmholdt*, Rene Peschar, Henk Schenk (A)
Mihaela Pop*, Kees Goubitz*, Dirk De Ridder*, Rene Peschar, Henk Schenk (B)
Laboratory for Crystallography
Institute for Molecular Chemistry (IMC)
Universiteit van Amsterdam
Nieuwe Achtergracht 166
1018 WV Amsterdam
The Netherlands

Report:

Session A

Although 6 shifts had been allocated, eventually more beam time turned out to be available and therefore, in addition to the compounds mentioned in title of the proposal, also data collection has been carried out for some other compounds on behalf of various other on-going projects in our laboratory. In this session complete high-resolution powder diffraction patterns were collected of sixteen compounds, here for shortness referred to by our acronym code: SMM, SPP, PMM, MSM, PSO, SOA, Z1066, Arbian, Y160, Z246OMT, Hist_50, Bock12, Bock15, Hartl, Y179, CuBZAC

The last six compounds mentioned above were measured first at the start of the session using the λ = 0.5010 Å of the previous user. After that, the wavelength was changed to λ = 0.85019 Å and the first ten (organic) compounds were measured. In this way the wavelength needed to be changed only once.

Except for the three pigments, data collection was carried out in the interval interval 0 - ~ 50.0º using special scan protocols in which the exposure time increased as function of 2θ. Especially for structure determination and refinement of organic samples this approach has been shown to be very useful.

The samples Hist_50, Bock12 and Bock15 are yellow-colored pigments (so-called Naples Yellow), presumably used on oil paintings and preserved in the Darmstadt Pigment Collection (Hist_50) and the collection of the Swiss painter Arnold Böcklin (Bock12 and Bock15) respectively. On the basis of the synchrotron data Hist_50 could be shown to consist of a mixture of $Pb_2\ Sb_2O_7$ and K_2PbSO_4 instead of pure $Pb_2\ Sb_2O_7$ while the two Böcklin samples did not contain any $Pb_2\ Sb_2O_7$ at all. A paper containing these results and others on the subject of Naples Yellow will be submitted soon to the Zeitschrift für Kunsttechnologie und Conserviering.

The compound Hartl, a Re-dioxolene complex that had measured at an earlier occasion at BM16 but not successfully because of sample-related problems, has been measured upto 30º 2θ. The Rietveld refinement is in progress.

The organo-metallic complexes Y179 and CuBZAC were also measured upto 30 º 2θ respectively. Both patterns have been indexed but structure determination has not yet been undertaken.

The samples SMM, SPP, PMM, MSM, PSO, SOA are all different triacylglycerols, main constituents of fats and oils. In all cases the patterns have been indexed. Because of a delay in the appointment of new personnel, the (time-consuming) structure determination has only started recently.

The compound Z1066 is a substituted derivate of trans,trans-1,4-bis[2-phenylethenyl]benzene [one of the compounds from Yang et al (1990)]. The pattern has been indexed and the structure determination has been completed.

The patterns of both the compounds Arbian and Z246OMT could be indexed but structure determination has been postposed because of relatively large amount of impurities.

References

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.

Session B

On behalf of various on-going projects in the lab, in this session complete high-resolution powder diffraction patterns were collected of the following compounds: POS, SOS, 13.15.13, 15.17.17, alpha-PPP, Camerun, Congo, Ivory, Togo, Ag-behenate, Boke, PW1246, Katal, Melphos, Mel4phos3, $Fe(teec)_6BF_4$, $Fe(teeF)_6BF_4$, $Fe(teeBr)_6BF_4$, $Fe(teei)_6BF_4$ and $Fe(teec)_6ClO_4$.

The wavelength in all experiments was 0.75003 Å and data collection was done at T = 294 K unless indicated otherwise. Samples were either prepared in capillaries or cut into a capillary shape (Camerun, Congo, Ivory and Togo)and rotated during exposure. In most instances data collection was carried out with the purpose to index the pattern and to undertake crystal structure determination. Therefore, scan protocols were used in which the exposure time increased as function of 2θ.

The first four compounds (POS, SOS, 13.15.13 and 15.17.17), different types of triacylglycerols with the former two being main constituents of fats and oils, have been measured in the interval 0.- 48.º 2θ. The patterns of POS (= β-1-palmityl,2-oleoyl,3-stearylglycerol and SOS (= β-2-oleoyl,1,3-distearyglycerol) as well as those of 13.15.13 and the asymmetric 15.17.17 have been indexed using a local program while structure determination of the first two is in progress.

Data collection for the compounds Camerun, Congo, Ivory and Togo, cocoa butters originating from different countries, was carried out in the interval 0.- 48.º 2θ. All these samples were carved into a capillary shape (~ 2-4 mm) suitable for data collection. Although being co-crystallized mixtures of various triacylglycerols, the diffraction patterns are of good quality and they are now being analyzed.

Alpha-PPP is the so-called 'rotator' alpha phase of the triacylglycerol tripalmitin. This instable phase was obtained by melting PPP and fast cooling.

The compounds Melphos and Mel4phos3 are melaminephospate compounds. The crystal structure of the former has been solved and the Rietveld refinement has been finished. A preliminary paper has been written but for strategic reasons it can not be submitted yet.

The compound Katal, an inorganic catalyst, has been solved and the refinement is in progress.

The patterns of Ag-behenate (well-known calibration standard in SAXS data collection), Boke (a supramolecular compound) and PW1246 (organic compound) have been indexed. Stucture determination of the former two is in progress.

The compounds $Fe(teec)_6BF_4$, $Fe(teeF)_6BF_4$, $Fe(teeBr)_6BF_4$, $Fe(teei)_6BF_4$ and $Fe(teec)_6ClO_4$ are tetrazole complexes showing spin-transition behaviour as function of temperature. The crystal structure of $Fe(teec)_6BF_4$ at room temperature has been solved recently by us on the basis of BM1B powder diffraction data (Dova et al., 2001) . In this session it was attempted to use a cryostream in data collection of this compound at 150 K. Unfortunately, the temperature reading of the cryostream differed more than 40º from that of the thermocouple so the precise temperature at the capillary remains unknown. The other tetrazole compounds listed have been measured at room temperature. Most of the patterns have been indexed and two compounds have been solved. Refinement is in progress.

References

Dova E., Stassen A.F., Driessen R.A.J., Sonneveld E., Goubitz K.,Peschar R., Haasnoot J.G., Reedijk J. and Schenk H.(2001). Structure determination of the [Fe(teec)6](BF4)2 metal complex from laboratory and synchrotron x-ray powder diffraction data with grid search techniques.Acta Cryst B57, 531-538.