ESRF	Experiment title: Test of diffraction properties of an unusual protein/lipid complex	Experiment number: LS.574
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Report:

Low-density lipoproteins (LDL) are spherical particles (total molecular weight, 2000 kDa) that consist of a core of cholesterol esters and have a surface composed of a monolayer of phospholipids and one molecule of apolipoprotein-B (molecular weight, 5 13 kDa). We have obtained crystals of a LDL subfraction that, when dissolved, run like native LDLs in agarose gels.

The reason for applying for beam time at the ESRF was to test whether the small-size crystals that we obtained so far permit detection of diffraction properties. We have brought the following samples to the ESRF:

- "multi-crystal" sample consisting of 30- 100 crystals of size about $(40\mu m)^3$, embedded in gel-like matrix (1 mm $\varnothing$, 200μm wide) which forms during crystallization. We have taken exposures (rotation angle 1° to 10°) of duration between 1 min and 10 min at different positions and angles with respect to the sample. We observed several diffraction rings at Bragg spacings between 10 and 3Å, which at the temperature of the experiment might correspond to the crystalline state of cholesterol esters known to be present in the core of the LDL particles. The diffraction pattern also showed tiny spots of about the detector's pixel size, most of which arose from detector noise, as was revealed in control experiments. However, long exposures with larger rotation angles resulted in broadening of some of those spots.

. yellow single crystals of size up to $(70\mu m)^3$, hexagonal habitus. Several of these were exposed (1° rotation, 3-20 min), but no clear evidence of diffraction spots was obtained.

In summary, the experiments perfomed were not conclusive, but point towards the necessity of further crystallization trials and other biochemical and biophysical characterization of the crystals. Due to the expected large unit cell size, necessary prerequisites for future experiments with LDL crystals at a synchrotron appear to be

1. substantial increase in crystal size
2. even higher flux than available at beamline BL19
3. long exposure time and low-noise detector; a CCD detector like the one at BL 19 appears to be more suitable for short-time exposures.