ESRF	**Experiment title:** Absolute atomic configuration of chiral crystals: a novel solution	**Experiment number**: HC-1779
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Report:

Scientific Background

Determining of absolute atomic configurations of chiral crystals is extremely important because most of biomolecules and medicaments are chiral and the sign of chirality is crucial for their efficiency.

X-ray diffraction can determine the spatial arrangement of the atoms, but don't distinguish the model of inversion. X-ray methods used to determine the absolute atomic configuration with a model of invertion [1, 2] exploits the anomalous dispersion of X-rays by atoms in crystal. It is widely used for determining of absolute atomic configurations inorganic compounds but does not work for crystals consisting of identical atoms (like Te) and with biologically important crystals consisting of atoms lighter than chlorine (C, H, N, O, F). In case of light atoms the interference pattern is determined only by the presence of nuclei, and not by their nature.

We suggest a new method to determine the absolute configuration of any crystals. Our method is based on the chiral asymmetry of multiple X-ray scattering. Every reflection on x-ray diffraction pattern can be obtained in several ways leading to scattering at the same angle. The intensity of each reflection is composed of the contribution of the main two-wave reflection and a variety of three-wave (Renningers) contributions. Far from absorption edges, the two-wave contribution is not sensitive to the chirality and does not change when the crystal rotates around the azimuthal axis. In case of forbidden reflection, this part vanishes whereas multiple scattering part is not zero and sensitive to chirality. Rotating the crystal about the axis perpendicular to surface, we get the azimuthal dependence of the Renninger plot which shape is determined by the atoms positions and looks different for the right and left configurations. Beauty of this approach is that it is model independent, rather flexible in choosing the wavelength of x-rays and uses only one reflection.

Experimental details

To test our method we chose untwinned quartz single crystals (space groups *P*3121, N 152 and *P*3221, N 154), because it's absolute atomic configurations is well known and can be used for checking correctness of our measurements.

The highest intensity of rennigers peaks could be reached using the scattering of circularly polarized X-ray radiation. So, we measured the azimuthal Renninger plots for the forbidden 001 reflection with the right and left circular polarizations of incident radiation in both right and left crystals. To exclude resonant contributions, we have chose the beam energy 4.5 KeV (far from the absorption edges of Si and O).

Results

We have observed a serie of the Renninger reflections for the angles between 0 and 70^{o} . Azimutal dependencies of reflection intensity looks mirror-symmetric for right and left quartz. So, we defined samples handedness by comparing with calculated curves (Fig.1).

Theoretical plots were calculated with the perturbation theory (Fig.2). We considered the interference between different three-wave scattering channels and specified on the picture the indexes of reflections which give the largest contribution to the corresponding peaks. From the position of the peaks were obtaned unit cell dimensions and coordinates of atoms. For the right quartz sample a=b=4.9141 A, c=5.4060 A; for the left quartz a=b=4.9132, c=5.4038 A.

References

[1] J. M. Bijvoet, A. F. Peerdeman, A. J. Van Bommel. "Determination of the Absolute Configuration of Optically Active Compounds by Means of X-Rays". Nature 168 (4268): 271-273 (1951).

[2] H. D. Flack. "On Enantiomorph-Polarity Estimation". Acta Crystallographica, A39: 876 881 (1983).

Report Summary

We proposed a new solution for determining the absolute configuration of chiral crystals and confirmed it in the case of crystals with light elements - left and right alpha-Quartz. Later we are going to extend our method to organic crystals where absolute atomic configuration are especially important.

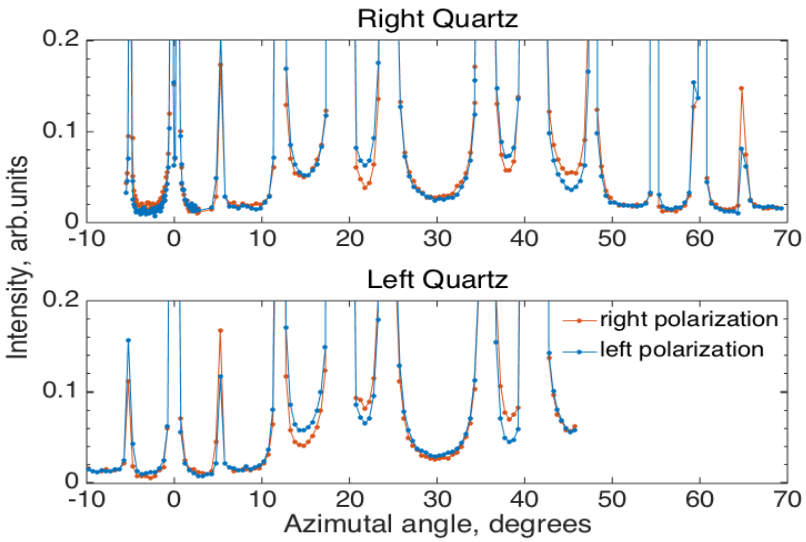

Fig.1: Intensity of 001 reflection for right and left polarisation in right and ledt quartz cristals.

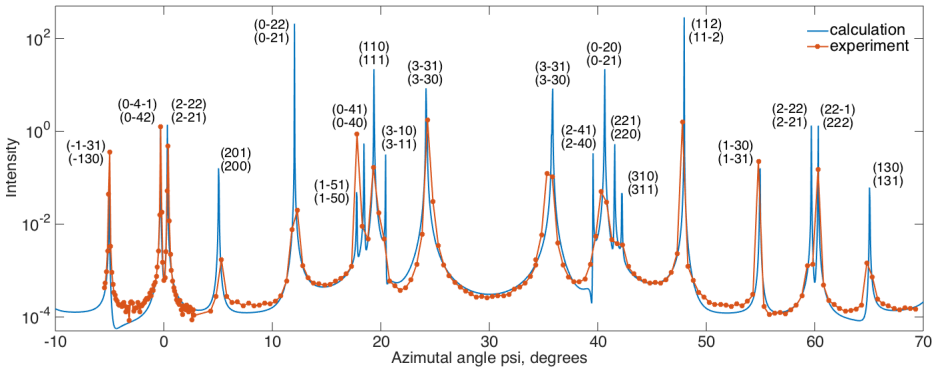

Fig.2: Experimental and calculated intensity of forbidden 001 reflection in the right alpha-Quartz.