

s10.m1.p15 **Structure Determination of Low-Symmetry Naturally Originated Compounds Using SR Powder Diffraction.** P. Serda^a, J. Grochowski^d, R. Czarnecki^b, T. Librowski^b, ^a*Regional Laboratory, Jagiellonian University, Ingardena 3, 30-060 Kraków, Poland;* ^b*Chair of Pharmacodynamics, Collegium Medicum Jagiellonian University, Kraków, Poland.*

Keywords: powder diffraction, advanced methods, structure determination.

The investigated substances belong to the group of naturally originated organic compounds extracted from plant sap. Poor crystallisation in this group of compounds is frequent and physiologically justified (e.g., large day-night thermal amplitudes). Crystal structure solution requires in such cases either microcrystalline single crystal diffraction, or high-resolution powder diffraction.

The present work is devoted to structure investigation of three substances, two of which are of natural origin: (1) khellol glucoside (chromone derivative, C19 H20 O10), (2) marmasenin (coumarin derivative, C20, H24 O9), and (3) a carane derivative, C32 H62 O4 N4 Cl2, synthesized from a naturally originated substrate.

Available diffraction data consist of the following sets: for substance (1) high-resolution synchrotron powder diffraction data, for substance (2) single crystal diffraction data and high-resolution synchrotron powder diffraction data, and for substance (3) two single crystal diffraction data and two powder diffraction data sets – using laboratory diffractometer and collected with a synchrotron source. Crystal data of the studied compounds are as follows: (1) $a=23.055(8)$, $b=17.872(5)$, $c=4.852(2)$ Å, $V=1979$ Å³, orthorhombic; (2) $a=7.868(1)$, $b=6.007(1)$, $c=40.727(2)$ Å, $V=1924.9$ Å³, orthorhombic; (3) $a=7.350$, $b=7.530$, $c=17.492$ Å, $\alpha=82.66$, $\beta=83.82$, $\gamma=84.39^\circ$, $V=953.7$ Å³, triclinic.

The above substances are a difficult case of *ab initio* structure determination from powder data due to large unit cell volumes and, in the case of (3), low space group symmetry as well as very close values of different lattice parameters.

Comparison will be made between *ab initio* structure determination from powder data with single crystal results in terms of geometrical features and accuracy of bond length and angles determination. Also, a comparison between various methods of *ab initio* structure solution will be attempted.

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s10.m1.p16 **Texture as a tool for structure determination from powder diffraction data.** T. Wessels, Ch. Baerlocher, L.B. McCusker, *Laboratory of Crystallography, ETH, Zurich, Switzerland.*

Keywords: structure determination, powder diffraction, texture

Powder diffractionists usually take great care to ensure that the crystallites in their sample are randomly oriented because preferred orientation distort the intensities. However, in principle, a textured powder sample can be used to determine the relative intensities of overlapping reflections to provide a single-crystal-like data set. In addition, a textured powder sample is useful also in allowing peaks from impurity phases to be identified and making the indexing unambiguous. Both problems often prevent structure determination if real materials are investigated.

A sample with pronounced preferred orientation can be regarded as being something between an “ideal” powder and a single crystal and can, therefore, provide more diffraction intensity information than can an “ideal” powder. To extract the three-dimensional intensity information of a textured powder sample a two step procedure was applied. First the texture in the sample was determined from the intensities of a few non-overlapping reflections measured at a series of sample tilt and rotation angles (pole figures). Then, full diffraction patterns were collected for several different sample orientations. By applying a deconvolution procedure, a set of single-crystal-like integrated intensities was extracted from the diffraction data.

The data collection can be performed either in reflection or transmission mode. Both geometries have their advantages and their limitations. By exploiting the parallel nature of a synchrotron beam and using a pre-detector analyzer crystal, high quality diffraction data can be collected in reflection mode. However, to collect the necessary amount of data approximately three days of synchrotron beam time are required. With a two-dimensional detector and using the transmission geometry this time can be shortened and only a few hours of beam time are required for data collection. The application of a two-dimensional detector leads to a decrease in the resolution of the powder diffraction pattern. However, the loss of resolution in 2θ can be partially compensated by a better resolution in orientation space, because complete diffraction patterns are measured for all sample orientations rather than just four or five as in reflection mode.

The texture method was successfully applied to different materials with complex crystal structure. With this approach it was possible to extract a set of intensities, that was sufficiently single-crystal like for the crystal structure to be solved *ab initio* by applying standard crystallographic methods. From conventional extracted intensities using only one diffraction pattern collected on an untextured powder sample none of these structures could be solved. Especially the structure solution of the zeolite UTD-1F with 69 non-H atoms in the asymmetric unit provides a clear demonstration of the power of the texture approach.