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Local Order Determination of a Novel β-keto Ether Analogue by X ray absorption

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Among all methods of structural elucidation and analysis, stand out Single-crystal X-ray Diffraction (XRD) and X-ray Powder Diffraction (XRPD). Both methodologies are based on the diffraction phenomenon, and the difference between them is given by the preparation and the sample quantity and equipment used in the diffraction experiment. Collection of diffraction data from single crystal results in a pattern consisting of spots arranged symmetrically in a beamstop. The location of the point indicates the direction of the scattered ray, while the spot size indicates the intensity of this ray. Once the sample is in the form of powder, it can be accommodated so that its surface is flat and the sample holder may be: metal, plastic or even glass. In addition, the preferred orientation can be avoided if the sample powder has the form of a cylinder of diameter 0.3mm to 0.5mm, usually accommodated in a glass capillary. The objective of this work was the structural long range analysis of a β-keto ether analogue via Single-crystal X-ray Diffraction and X-ray Powder Diffraction. The monocrystal X-ray diffraction data of the analyzed compound were collected at room temperature using the Bruker APEX II CCD diffratometer with graphite-monochromated MoKα radiation (λ=0.71073 Å) at Chemistry Institute of the Federal University of Goiás. In addition, X ray powder patterns of β-keto ether analogue samples were collected at three different wavelengths and two different temperatures at LDRX-UFSC and XRD1 beamline of Brazilian Synchrotron using 7 and 12 keV photons at room temperature and 100K. All the measurements were carried out in transmission mode using 0.7 mm glass capillary. The Rietveld refinement of the X-ray Powder diffractogram was achieved in order to verify the crystalline structure determined by single crystal method. Polymorphic susceptibility of the compound was proposed by the root-mean-square deviation of the atomic positions held between structures obtained via single crystal and powder. In this poster we will show our X ray diffaction results in view to discuss the possibilities to understand this duality by local order determination of single crystal and powder samples using XAS (EXAFS and XANES) experiments.