MS25-1-4 Structure determination study of beam-sensitive organics with 3D electron diffraction #MS25-1-4

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Abstract
Three dimensional electron diffraction has been proven suitable for a large range of materials including highly beam sensitive crystals [1][2]. In order to speed up data acquisition thus decresing the electron beam damage the majority of these crystal structures have been performed using fast hybrid pixel detectors. Here we demonstrate different settings for successful data acquisition using a slow CCD camera. One of the examples used to demonstrate this is the crystal structure solution of Vitamin B2 (riboflavin) [3] also known as lactoflavin. Riboflavin is a naturally occurring yellow pigment that was first isolated from whey and egg white in 1933 [4]. Riboflavin has the function of a vitamin and is commonly found in milk, liver, kidney, muscle, yeast and plant matter. The human body is supplied with vitamin B2 through nutrition [5]. Depending on which of the eight riboflavin polymorph is present [6], the solubility and thus the bioavailability of the vitamin changes. 3D Electron Diffraction (3DED) combined with electron precession technique was used to determine the structure of the anhydrate riboflavin polymorph.
The three-dimensional electron diffraction data was collected by using Fast-Automated Diffraction Tomography (Fast-ADT) in a FEI Tecnai F30/S-TWIN Transmission Electron Microscope (TEM) equipped with a ULTRASCAN4000 CCD camera. Fast-ADT is a straightforward method and allows a quick data acquisition and crystal tracking with no need for a manual crystal pre-orientation. This can reduce the electron dose during the acquisition and therefore the technique has been proven suitable for beam-sensitive organic materials like the riboflavin [7].
The polymorph A of riboflavin occurs in the orthorhombic space group P212121. Each unit cell contains four riboflavin molecules showing an elaborated hydrogen bonding network where the sugar alcohol D-Ribit side chains of the molecules face each other. Moreover the molecules in the unit cell occur twisted against each other so they are not stacked but appear similar as a herringbone pattern.

References
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Structure solution model of Riboflavin Polymorph A