

**FA3-MS22-T03**

**Disorder and conformational flexibility in vitamin B12 explored at high pressure.** Francesca P. A. Fabbiani<sup>a</sup>, Gernot Buth<sup>b</sup>, Birger Dittrich<sup>c</sup>, Heidrun Sowa<sup>d</sup>. <sup>a</sup>*GZG Dept. of Crystallography, Georg-August-Universität Göttingen, Germany.* <sup>b</sup>*Karlsruhe Institute for Technology, ISS, Germany.* <sup>c</sup>*Inst. of Inorganic Chemistry, Georg-August-Universität Göttingen, Germany.*  
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The seminal work by Dorothy C. Hodgkin and her group on vitamin B12 (cyanocobalamin) [1-3] sparked a wealth of subsequent structural investigations on the vitamin and its derivatives.[4] The crystals are prone to solvent loss and disorder, in particular that of the solvent region,[5] making data collection and structural refinement not straightforward. We have determined the hydrated, “wet” structure of the title compound by a) compressing a single crystal to 1.0 GPa and by b) growing a single crystal *in situ* in a capillary at ambient conditions. Diffraction data were collected at room-temperature using X-ray synchrotron radiation. A comparison of the two structures reveals: a) dramatic increase in ordering of the water solvent region, b) smaller displacement parameters of the B12 moiety and c) substantial conformational change in the side chains of the vitamin induced by high pressure. Possible mechanisms that account for these observations are discussed and related to observations made on protein crystals.[6] The use of high pressure and low temperature as tools to probe conformational flexibility and increase order in crystals containing a large amount of water are also discussed.

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**FA3-MS22-T04**

**Symmetry analysis of extinction rules in diffuse scattering experiments** Mois I. Aroyo<sup>a</sup>, Ray L. Withers<sup>b</sup>, J. M. Perez-Mato<sup>a</sup>, Danel Orobengoa<sup>a</sup>  
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Structured diffuse scattering intensities, whether of compositional or of pure displacive origin, static or dynamic, contain important information about the symmetry of the individual compositional and/or displacive modes responsible for the observed intensities. However, the interpretation of the experimental data is very often impeded by the lack of a symmetry-based approach to the analysis of the structured diffuse scattering distributions. We have demonstrated the existence of systematic phonon selection rules for diffuse

scattering that depend on the symmetries of the mode and the scattering vector, and not on the specific structure [1]. The aim of this contribution is to show that such symmetry analysis can be successfully extended and applied also to structure-dependent diffuse scattering associated with 'disordered' materials, combining general diffuse scattering extinction conditions with the concept of non-characteristic orbits [2]. The utility of this approach is illustrated by the analysis of diffuse scattering data from three different 'disordered' systems ThAsSe [3], FeOF [4] and FeF<sub>2</sub> [5]. We show that initially unexpected extinction conditions can arise in structured diffuse distributions if the responsible atoms lie on sites with higher space-group symmetry than the whole average structure. Knowledge of the relevant supergroup symmetry enables to rationalize the observed 'extinction conditions' and allows a direct insight into the characteristics of the responsible real space structural disorder. In addition to these unexpected 'extinction conditions', it is also demonstrated that additional extinction conditions can arise at special zone axis orientations as a result of the symmetry of the modes responsible for the observed structured diffuse scattering.

An essential part of the calculations has been done using the computer programs NEUTRON (systematic phonon extinction rules in diffuse scattering) and NONCHAR (non-characteristic orbits of space groups) that are available on the Bilbao Crystallographic Server ([www.cryst.ehu.es](http://www.cryst.ehu.es))[6].

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**FA3-MS22-T05**

**Determination of disorder using the three dimensional pair distribution function.** Arkadiy Simonov, Thomas Weber, Walter Steurer. *Laboratory of crystallography, ETH Zürich, Switzerland.*  
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As detectors become better high quality diffuse scattering data becomes readily available. Consequently, there is a demand for novel tools that provide easy access to the information from diffuse scattering.

We suggest a straightforward way for finding disorder models. The method is based on a direct refinement of the pair distribution function which is calculated as the Fourier transform of x-ray intensities. The method is analogous to powder PDF refinement, however, powder PDF refinement is restricted to one-dimensional experimental data. Fourier transform of such data yields a rotational projection of the PDF. In our experiments we use x-ray scattering from single crystal and therefore we are able to calculate the PDF in three dimensions (3D-PDF). To investigate disorder, the PDF function is calculated from diffuse scattering only. The result

is the so-called  $\Delta$ PDF which contains information about deviations of the real structure from the average structure.

The method was applied to diffuse scattering from a N,N',N"-tris-*t*-butyl-1,3,5-benzene tricarboxamide single crystal [1-2]. The crystal consists of chiral stacks of molecules. The stacks are arranged in a hexagonal lattice. On average both enantiomeric stacks occupy the same position. Refinement of the disorder model showed that there is a very strong preference for nearest stacks to be of different chirality. It is also shown that pairs of stacks having a same chirality stay slightly further from each other than those with different chirality.

It was shown that this method provides an easy access to chemical ordering parameters and size effect parameters.

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**Keywords: 3D-PDF, diffuse scattering, disordered crystals**