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Transformable Single-Crystal Adsorbent Based on 1-D Coordination Polymer Skeletons
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Recently, we found a single crystal host, $[M(II)_2(bza)_4(pyz)]_n$ (bza and pyz = benzoate and pyrazine, M = Rh and Cu), which is suitable for the study of gas-containing structure through gas adsorption. They generated gas inclusion crystals by transition from a closed α to an open β phase through the process of smooth physisorption; guest gases are adsorbed into the generated narrow channels of the β lattice. I will present a new convenient procedure for crystallizing gas into a co-single crystal state by putting the crystal adsorbent in a gaseous guest atmosphere, which is efficient to ascertain the exact molecular/atom structures with high resolution for included light aggregates.[1]

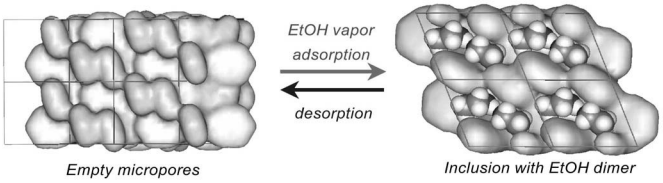

Fig. An exampple: ethanol dimer inclusion formation accompanying bulk phase transition.[2]

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Keywords: metal-organic complexes, porous materials, solid-state phase-transition

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Is your Crystal Representative of the Bulk?
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Structure analysis by X-ray diffraction is commonly carried out on just **ONE** single crystal. The physical, chemical, pharmaceutical, biological and structural properties of this one crystal can only be characteristic of the bulk under certain circumstances. In particular for bulk samples which are mixtures, or whose purity is questionable, an excellent ploy is to characterize the one single crystal used for the diffraction experiment. However until recently a difficulty has been its small mass *ca*. 1 μg. Thermochemical characterization by way of differential scanning calorimetry (DSC) can provide clear evidence on purity, phase transitions and solid-solution formation. For enantiomeric mixtures both circular dichroism (CD) and enantioselective chromatography, but not optical activity, may be applied to such a single crystal taken into solution.

As a first case study, a determination of absolute configuration was achieved from X-ray diffraction and CD measurements on crystals obtained from a racemate in the bulk by spontaneous resolution to give a tricky racemic conglomerate. Crystals were either enantiopure but twinned by a pure rotation or twinned by inversion in various proportions. In the second case study of an absolute-configuration determination from an enantiopure sample, the optical activity and CD spectrum were far too weak to be useful to characterize the enantiomer. However enantioselective chromat-ography on the single crystal taken into solution provided the necessary characterization.
Keywords: single-crystal, chiral, characterization

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Model Wave Function for Glycyl-L-Alanine from Experimental Diffraction Data
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The XCW method is a relatively new technique for analysing X-ray diffraction experiments [1-5]. Rather than using the more common pseudo-atom expansion of the electron density [6], the X-ray diffraction data are introduced directly into the calculation of the quantum mechanical molecular wave function in such a way that the wave function is constrained to reproduce the X-ray data, at the expense of the smallest possible change in the quantum mechanical energy. The XCW method has been applied to a number of small-molecule organic crystals [2-4] showing, for example, that the constrained wave functions could model the effects of higher level calculations, simply by including experimental data or that the effects of the crystal lattice could be incorporated [2]and that a number of derived properties, such as electrostatic potentials and multipole moments could be obtained [3-4]. In order to address the limitations of the XCW method as compared to other forms of analysis of the electron distribution in crystals, like the pseudo-atom model, and to extend the application of the XCW method to more complex systems for which ab-initio calculations cannot be expect to give very accurate results (e.g. for proteins), synchrotron and neutron diffraction data have been measured on the model dipeptide Glycyl-L-Alanine at multiple-temperatures in the range 10 to 295K. The multi-temperature data were used in a molecular Einstein model [5] to get a more complete description of the thermal motion in the crystal and to recover information on the atomic correlation of motion. These latter were then included in the XCW fitting procedure to see their influence on the predicted structure factors and on the derived electronic properties. The neutron data where essential to evaluate the contribution of the motion of the hydrogen atoms to the final XCW-derived electron density.

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Keywords: quantum crystallography, experimental wavefunctions, peptides

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Dynamics of Nucleoside and Nucleotide Hydrates by MD Simulation
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Humidity-governed phase transitions of nucleoside and nucleotide hydrates are triggered by adsorption or desorption of crystal water molecules.[1] Lowering temperature, the phase transitions coupled with ordering of crystal water molecules in the hydrates were also observed. To know the dynamics of the phase transitions, we have started molecular dynamics simulation of hydrates of guanosine[2] and disodium inosine 5'-phosphate.

The simulation was performed with the AMBER 6 program in the periodic boundary conditions. The calculation cell was constructed to be about 60 × 60 × 60 Å by replicating the crystallographic unit in the directions of the *a*-, *b*-, and *c*- axes, respectively. The AMBER ff99 energy parameters and the TIP3P water parameters were used. Electrostatic interactions were calculated by the particle-mesh Ewald method.

Averaged structures, atomic displacement parameters, time profile of hydrogen bonding probability, formation of large translocation clusters etc. were analyzed based on the simulation trajectory. The origins of the phonons observed in Raman spectra[3] were also suggested.

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