The crystal structure of ferrierite is built up of rings of fivecorner-shared SiO_4 tetrahedra (known as five-membered ringsor 5MRs) building units, which form layers in the *ab* plane.The layers are connected to form a matrix of 10MR channels running parallel to the *c* axis, which are intersected by 8MR channels running parallel to the *b* axis. Six-membered rings connect the 10MRs alongthe *c* axis direction.

The HP diffraction experiments were performed at BM01a beamline (ESRF), at the fixed wavelength of 0.71 Å, using a modified Merril-Basset DAC and a mixture of methanol- ethanol and water (16:3:1) as penetrating P-transmitting medium. The powder patterns were collected from P_{amb} to 6.2 GPa. Some patterns were also measured upon pressure release up to P_{amb}, to check the reversibility of the compression effects. The unit cell parameters were refined by means of Rietveld method.

The main results of this study are:

1) No complete X-ray amorphization is observed up to about 6.6 GPa;

2) No abrupt change of the elastic behavior is observed in the whole pressure range. Between P_{amb} and 6.2 GPa the reduction of the cell parameter are 4%, 5% and 6% for a, b and c respectively, accounting for a volume reduction of about 14 %.

3) The bulk modulus obtained using a second order Birch-Murnaghan equation of state and data weighted by the uncertainties in *P* and *V* was K_0 = 30.1(3) GPa. This compressibility determined in m.e.w. is one of the highest when compared with other natural and synthetic zeolites studied with "penetrating" aqueous media [2], [3].

4) The P-induced effects on as-syntehsized ferrierite structure are completely reversible.

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Keywords: high pressure, zeolite ferrierite, synchrotron XRPD

MS05.P17

Variable-temperature studies of three polymorphs of 1,3-nitrobenzoic acid

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The reported results concern the multi-disciplinary investigations including: variable-temperature single crystal and powder X-ray diffraction, differential scanning calorimetry (DSC) and IR and Raman spectroscopy.

The thermodynamic relationship between three polymorphs has been elucidated. Three polymorphs can be grown at ambient temperature, two of them as metastable phases. Enantiotropic transformations between polymorphs occur at temperatures above ambient (353 K and 378 K) and are due to thermally enhanced vibrations of the nitro groups. The double hydrogen bonding linking carboxyl groups remains preserved in two polymorphs. One transformation has been also observed as single crystal – single crystal transition.

Structurally two polymorphs differ by the position of nitro groups in the molecular dimers, that is *trans* in the low-temperature form and *cis* in the high-temperature form. As a consequence the interactions involving the nitro groups are different. In the low-temperature form the nitro…nitro intermolecular interactions are observed. In the high-temperature form the nitro…HC intermolecular weak hydrogen bonds occur. The crystal structure of the third polymorph still remains the subject of our investigation.

The relevant role of the out-of-plane torsional vibrations of the nitro groups for the crystal structure stability is consistent with the thermodynamic properties of the crystals of other nitrobenzene derivatives, able to form strong hydrogen bonds. The crystals exhibit phase transitions above ambient temperature. The transitions are driven by large-amplitude torsional vibrations of the nitro groups while intermolecular hydrogen bonds remain persistent [1], [2].

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Keywords: polymorphism, transformation, vibration

MS05.P18

Parametric symmetry mode refinement of the structural phase transition of $CuInSe_2$

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As it is the case for many structural phase transitions, the low symmetry structure of $CuInSe_2$ can be described as a distorted form of its high symmetry structure by adding a set of symmetry modes to the atomic structure of the high symmetry phase. The decomposition of the structures in terms of symmetry modes can be easily done by web based software packages [1], [2]. Symmetry modes of one given type (e.g. lattice strain, displacement or occupancy) comprise an order parameter according to Landau theory.

This alternative approach to describe the structural evolution during ferroelastic phase transitions can also be applied in Rietveld refinement, simply refining the amplitudes of the symmetry modes instead of refining free atomic positions, occupancies and lattice parameters. To investigate the behaviour of the relevant modes over the phase transition parametric Rietveld refinement [3] offers the possibility to model the characteristics of those modes using power law trends as proposed in Landau theory.

For the structural phase transition of $CuInSe_2$ from the tetragonal chalcopyrite to the cubic sphalerite structure type the temperature dependence of all symmetry modes influencing lattice parameters, atomic coordinates and occupancies as well as the evolution of the isotropic temperature factors have been modeled with a phenomenological power law behaviour. The results of this study reveal superior statistics and much higher stability of parametric as compared to sequential Rietveld refinement leading to a more detailed understanding of the nature of the phase transition (Figure 1).
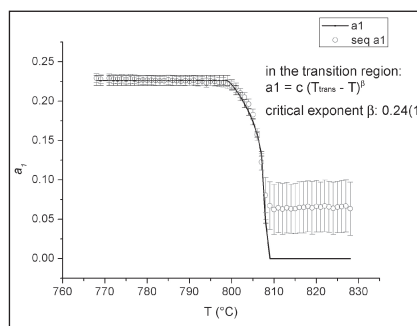

Figure 1: Comparison of the results of traditional sequential refinement (dots) and parametric refinement (line) for the a1 symmetry mode describing the evolution of the x-position of the Se atom.