ESRF	**Experiment title:** **Disorder in Perhydrotriphenylene Inclusion Compounds**	**Experiment number:** 01-02-241
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Shifts: 3	**Local contact(s):** Philip Pattison	*Received at ESRF:*

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Report:

Perhydrotriphenylene (PHTP) inclusion compounds consist of (almost) homochiral stacks of PHTP molecules, forming a honeycomb-like tunnel framework in which different types of molecules can be included as a guest (here: 1-(4-nitrophenyl)piperazine, NPP). Due to racemic and positional disorder of the host, orientational (up - down) and positional disorder of the guest and further, because of interactions between host and guest, a variety of complex diffuse and satellite scattering phenomena can be observed with X-ray diffraction experiments.

The aim of this experiment was to get high quality information about diffuse and satellite scattering. The experiment was done at room temperature using the MAR-image plate system. Since most of very narrow diffuse scattering features exhibit a small width parallel to **c***, the crystals **c*** axis was mounted parallel to the oscillation axis, which lead to an optimum resolution along the desired direction.

The needle-like crystal was irradiated at its tip. With 900 frames of oscillation range of 0.2° per frame a total range of 180° was covered. The reconstruction of reciprocal layers was successfully done using the program XCAVATE [1].

The analysis of diffuse scattering coming from the host showed that a preferred R-S correlation of laterally neighbouring PHTP is present. The highest probability for finding a R-S contact is parallel to the b-axis, where an almost perfect face-to-face contact of PHTP molecules is present. Further, a shearing of rows of PHTP stacks could be found parallel to the a-axis. The later effect is independent from the distribution of R and S molecules. Fig. 1 shows a section of the hk1 layer comparing experimental data with calculated intensities from the model given above.

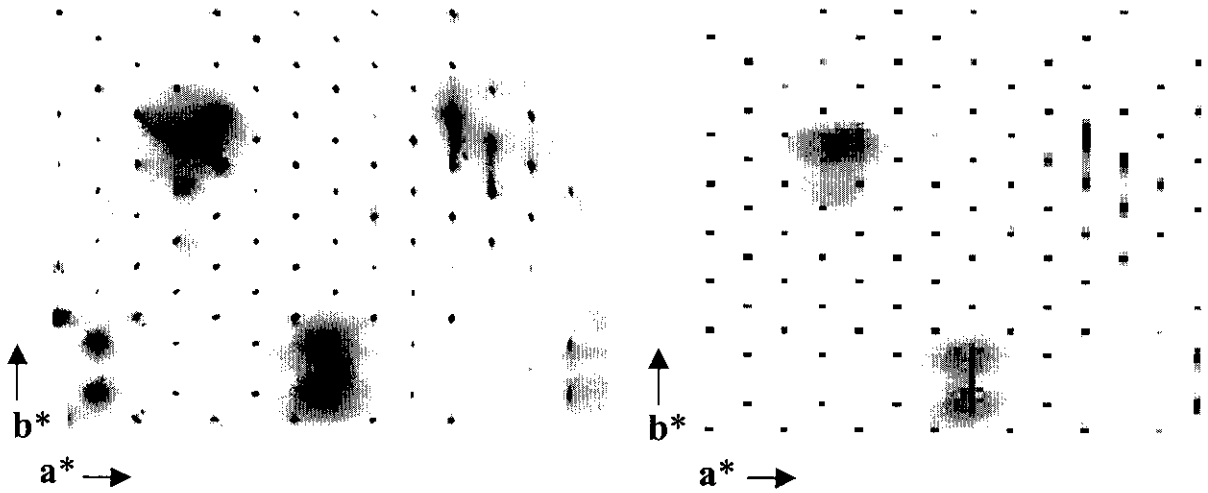

Fig. 1: Observed and calculated diffraction pattern (hk1)

Beside a number of additional phenomena the analysis of the diffuse intensities coming from the guest showed that diffuse layers, which are due to positional disorder of the guest parallel to the tunnel axis, are not completely sharp along **c*** as assumed before, but slightly broadened and of asymmetric shape. The asymmetry of the profiles indicates that orientational disorder of the guest molecules may be associated with translational disorder. Another interesting result was that guest diffuse scattering and satellite are of lower symmetry than the Bragg scattering (monoclinic *vs.* orthorhombic). A more detailed investigation is still in progress.

Reference:

[1] M. A. Estermann & W. Steurer (1998). Phase Trans., **67**, 165 -195.