ESRF	**Experiment title:** High-resolution powder diffraction of cyclodextrins complexed with hydrophobic organic guest molecules	**Experiment number**: CH-1209
Beamline: BM01B	**Date of experiment**: From: 10-05-2002 to: 13-05-2002	**Date of report**: 25-02-2003
Shifts: 9	**Local contact(s)**: Hermann Emerich	*Received at ESRF:*

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Report:

In total eight complete data sets have been collected at room temperature (set temp = 293 K, hutch temp ≈ 301 K) in theta mode, using continuous scans. The wavelength was 0.79942 Å, beam width 5.0 mm., beam heigth 1.0-1.5 mm. All data sets were finally binned at 0.005 º2θ.

Initially, we intended to use a data collection protocol that consists of an amount of overlapping 2θ sections with the lower 2θ boundary being increased after each iteration. In this way each reciprocal lattice point is being exposed to approximately the same amount of radiation, like in a single-crystal diffraction experiment. According to the local contact, this scheme has two other additional advantages: (a) the slow intensity drop of the beam during the experiment, that can not (yet) be taken into consideration, has little effect on the data collection and (b) little influence of the shift of the detectors. This shift can vary in an unpredictable and non-constant way while, as yet, in the software only a constant shift is corrected for. A disadvantage of this scheme, however, is that the amount of dead time increases with the amount of iterations and therefore it was to advised to use another scheme: divide the total 2θ interval in a set of intervals that become smaller as 2θ increases and let the integration time increase proportionally. This scheme was adopted but, in retrospect, we do not support this scheme for an additional reason: data collection using overlapping 2θ sections has the advantage that from the beginning data of the complete 2θ interval is

available. Thus, data collection might be stopped voluntarily after each scan. Even more important, if the data collection is stopped unvoluntarily, still data in the highest 2θ section are available.

Six β cyclodextrin inclusion compounds (organic guest molecules: tolfenamic acid, metoprolol, ciprofloxacin, flufenamic acid, nimodipin and atenolol respectively) were measured in the interval 1.0 – 30.0 $^{o}2\theta$. Each complete interval was divided in four (tolfenamic acid) or six (the other five cyclodextrins) subintervals, with the lowest being measured with an increasing integration time of 1s, the highest with 4 s and 6 s, respectively.

Data collection of the sixth cyclodextrin was unvoluntarily aborted (at 15.45 on 11-05-2002) because of a major front-end computer crash at another beamline. As a result, no data collection was possible for almost two days. Eventually, data collection of this sixth β-cyclodextrin has been carried out but no time was left to perform data collection for any of the α-cyclodextrins. Indexing and structure determination of the six β cyclodextrin inclusion compounds has been started