ESRF	**Experiment title:** Surface crystallography with a two dimensional detector.	**Experiment number:** SI-264
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Report:

The determination of a surface crystal structure from diffraction measurements implies the accurate measurements of the intensities I(HKL). This is traditionally done by integrating the diffracted intensity measured in a scan, which starts away from the reflection, going through the peak intensity and ending again on the other side, away from the peak with the detector fixed. The six-circle diffractometer installed in ID3, using a punctual detector, works in a Z-axis mode measuring the integrated intensities as described above [1]. This particular geometry affects the measured intensities basically by two corrections when this kind of detectors are used:

(i) A geometrical correction given by the Lorentz factor coming from the particular geometry used while the peak intensity is being detected during the Theta-scan.

(ii) The acceptance of the detector delimited by the slit setting in front of the detector.

Usually the measurement of a particular reflection (depending of how intense is the peak), performing a Theta-scan, takes between 2 and 4 minutes of counting time. Each scan can contain more than 50 points, to reproduce perfectly the shape of the peak.

When the punctual detector eliminated and substituted by a “photograph” of the diffracted intensity taken by a CCD camera. In this way the 2-Dimensional angular distribution of the diffracted intensity is collected in a single shot. This may save a factor 3 in time, which could permit to measure more reflections and to obtain more complete data sets. In this last case both the Lorentz correction and the detector slit setting correction are substituted by a single geometrical factor. This factor would have into account the cross section area between the rod and the Ewald sphere.

To determine that correction we have measured two times the same reflections of a crystal, one with the punctual detector and the other with the CCD camera in identical experimental conditions. We chose the structure of the Ge(001) p(2x1) and we measured in-plane as well as out-of-plane reflections and crystal truncation rods. The analysis of the images presents as main problem the two-dimensional background subtraction. We recorded a complete data set which is being analyzed. A practical problem that it has not still been solved is the following. The punctual detector allows to determine the integrated intensity I(HKL) of a reflection typically within less than 1% error if the crystal is good enough. Therefore one needs to achieve this level of accuracy, if possible, with the 2D detector if one wants to get similar structural accuracy. This presents the practical problem of accurately adjusting the background of the CCD images which is much more intense than in the data from the punctual detector since no energy filtering (crystal analyzer) can be used. The background problem is being solved by the fit of the 2D background by a polinomy defined along both directions, x and y. The image data are not completely corrected by this term due to the used software does not treat all the images sequentially, and they have to be corrected one by one. The number of measured images is more than 500. At the same time, some software development is being do it to try to solve this problem.

The data obtained with the punctual detector were already corrected using the standard surface data analysis programs supported by ID3, which is not the case to correct the image data.

We do not have a definitive conclusion yet since this implies software development that it is presently underway. A tentative conclusion is that once the adequate and accurate background subtraction routines will be implemented, the data from the CCD camera will be of almost the same accuracy than those from the punctual detector. An important additional aspect in favour of the CCD is that as the experimentalists can actually “see” the diffracted beam, misalignment problems, double reflections coming from different crystallites in the sample and other experimental difficulties are detected at once.