

# Construction and Preliminary Test of a 7-axes-diffractometer for Structure Analysis Beamline

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## 1. Out line of the diffractometer

The Crystal Structure Analysis beam-line was constructed by the collaboration of four subgroups, that is, Structural Phase Transition, Highly Precise Molecular Crystallography, Diffuse Scattering and High-Resolution Powder groups. Since the requirements to the diffractometer and apparatus are widely spread among these four groups, the basic concept to construct the diffractometer was settled so as it is able to be used for the general purpose. First, we decided to use the four-circle type diffractometer with an off-center x-cradle. And then, various type of apparatus, such as cryostat, furnace, camera, diamond anvil cell, etc., were designed for the widely spread interest of each group. By considering the conventional set up of the diffractometer, we ordered a commercially available machine, and some part was modified to fit the special purpose of the experiments. The central part of the system of this beamline is the seven-axes diffractometer, whose system is very similar with the conventional six-axes diffractometer commonly used at many beamlines of synchrotron radiation facilities. One extra two-theta axis is added. The purpose of the extra axis is to be specialized for

the conventional structure analysis to give the high speed motor function. The basement for the diffractometer was carefully designed in order to align the optical beamline easily. We will use a standard type monochromator of SPring-8. The beam height, 1400mm before the monochromator, will be fixed at 1430mm after the monochromator. The energy range we can use at this beamline is from 5keV to 50keV. For lower energy range ( $5 < E < 22\text{keV}$ ), two mirrors are settled-in to eliminate the higher order harmonics and to focus the beam. When the mirrors are used, the beam height changes from 1525mm(22keV) to 1640mm(5keV).

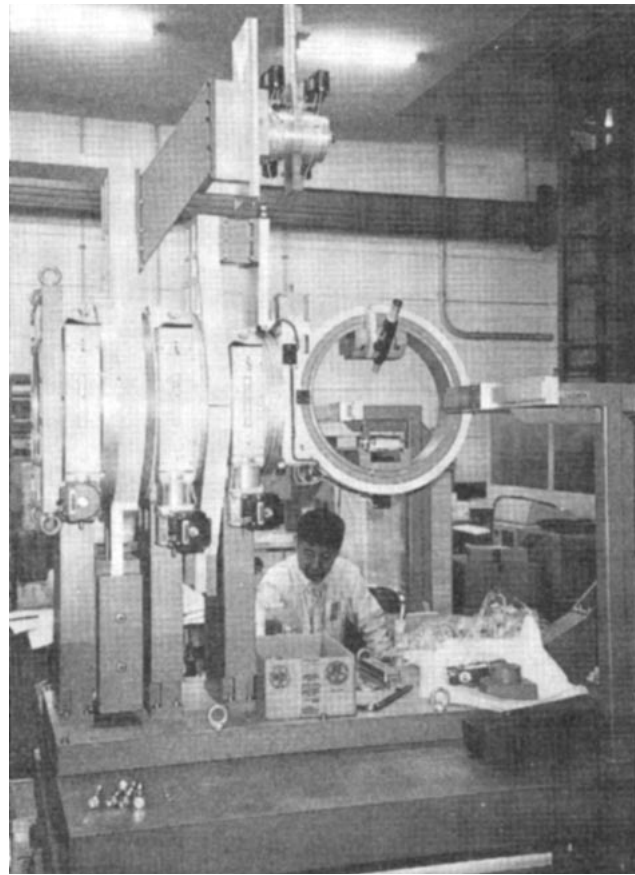
## 2. Preliminary test of the diffractometer

The diffractometer itself was delivered to MAC Science Co. Ltd. at Kaga on the beginning of February by Niki Glass. Shown in the Photograph 1 is the Huber four-circle diffractometer just arrived at the machine-shop of MAC Science. Schematic drawings are also shown in Fig. 1.

We tested the motor speed, range of the angles and the translational movements without any special loading. The angles to be controlled are  $2\theta_{S1}$ ,  $2\theta_{S2}$ ,  $\omega_S$ ,  $\chi_S$ ,  $\phi_S$ ,  $\omega_{A1}$ ,  $2\theta_{A1}$  for the diffractometer. Here, s denotes a sample and A denotes an analyzer. We have another set of  $\omega_{A2}$  and  $2\theta_{A2}$  to measure the polarization of the scattered X-ray beam. All of motor system have the ability to tell the own absolute zero position and the angle limit positions to the computer system. The diffractometer is put on the basement table to tune the crystal and incident collimator positions just along the incident X-ray beam. Four motor systems are available for the

basement table:  $R_B$ ,  $\omega_B$ ,  $X_B$  and  $Z_B$ . Here,  $R_B$  is for the tilting,  $\omega_B$  is for the rotation, and  $X_B$  and  $Z_B$  are for the translations of the basement table. In Table 1, the interval of angles or strokes for one pulse of the motor, observed speed of motors and the range of the movement of each axis are tabulated.

The observed motor speeds are satisfactory, and the ranges of the movements are good enough. However, we found that the precision of the rotating center are slightly disaligned during the transportation from Germany to Japan. We will readjust the alignment at the experimental hutch of SPring-8 on the end of March after the set-up of the all instruments .



Photograph 1. 7-axes Huber diffractometer

	step	mm or deg/min	range
$2\theta_{s1}$	$0.0001^\circ$	120	- 48 to +204
$2\theta_{s2}$	$0.0004^\circ$	300	- 58 to +201
$\omega_s$	$0.0001^\circ$	120	-180 to +180
$\chi_s$	$0.0002^\circ$	240	-180 to +180
$\phi_s$	$0.0001^\circ$	180	-180 to +180
$\omega_{A1}$	$0.0001^\circ$	180	-180 to +180
$2\theta_{A1}$	$0.0001^\circ$	180	-180 to +180
$R_B$	$0.000022^\circ$	5	-1.5 to +1.5
$\omega_B$	$0.0000572^\circ$	3.3	-2.5 to +3.0
$X_B$	0.0004mm	93	-37 to +63
$Z_B$	0.0001mm	25	0 to +210

Table 1. The observed interval of angles or strokes for one pulse of the motor, speed of motors and the range of the movement of each axis of the seven-axes diffractometer and the basement table.

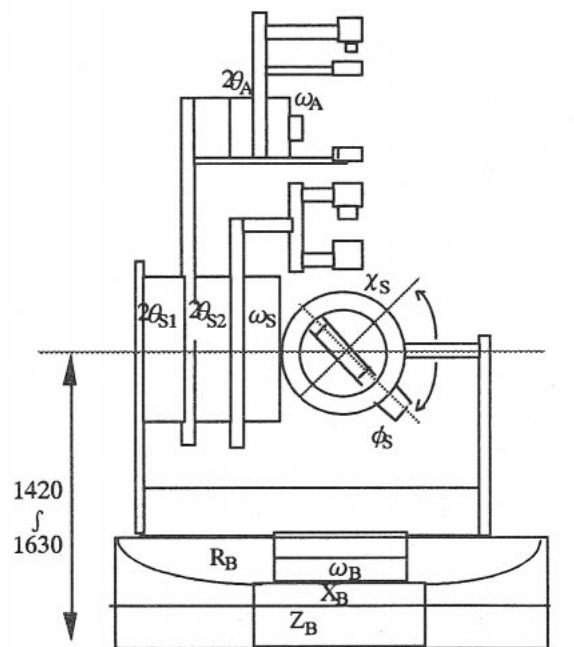


Fig.1 Schematic drawing of the seven-axes diffractometer with the basement table.