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FIRST X-RAY DIFFRACTION EXPERIMENTAL RESULTS AT THE ADONE WIGGLER LINE BX1

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One of the research fields which expects really advantages in the use of synchrotron radiation is the X-ray diffractometry, whose applicability to some particular research problems like the experimental phase determination, the anomalous scattering, etc. has been conditioned, till now, by the low intensity and the high divergence of the traditional X-ray sources.

By using synchrotron radiation and specially wiggler magnets, like the one which became operative at the Adone storage ring in Frascati since 1980, a further enhance of the photon flux in the X-ray region has been obtained, associated to a decrease in the vertical beam divergence.

For the Adone-wiggler BX1 line, after the Be window which separates the experimental apparata from the vacuum line connected to the storage ring, the typical photon flux value is about 10^{11} ph/s.mA.mrad ($E=E_c=2.77$ Kev, 0.1% bw, E=1.5 GeV) with an angular acceptance of 1.5 mrad horizontally and 0.8 mrad vertically. A channel-cut monochromator with a Si(111) or Ge(220) crystal, working in the range 3-30 KeV with an intrinsic resolution of about $\Delta E/E=1.3\times10^{-4}$, completes the line. The mean life time of the beam during each run, in order to pass from 100 mA to 10 mA, is about five hours[(1)].

The availability of such a good, tuneable, light source stimulates a joint project between the PWA Lab. (INFN) and the Istituto di Strutturistica Chimica (CNR) to built up and set up a diffraction station both for internal and external users. This station, based on a completely fully automatized four circle Huber diffractometer, became operative on the BX1 line at the beginning of the 1986[(2)].

In Table I the mechanical characteristics of the ϑ and 2ϑ goniometer and of the χ and φ Eulerian Cradle are given.

TABLE I - Mechanical features of the four circle diffractometer.

Circle	ϑ & 2ϑ	χ	φ
Axial load force (N) max	3600		
Vernier scale unit in degrees	0.005	0.01	0.02
Absolute angle deviation in arcsec	≤30	≤30	≤30
Positioning reproducible in arcsec	≤2	≤2	≤2
Reduction ratio	1/360	1/360	1/180
Radial and axial deviation max. degrees	≤0.1		
Sphere error in mm		≤0.02	
Parallelism χ-plane/φ-axis in arcsec		≤5	
Weight in Kg	36	17	

To avoid intensity losses due to the horizontal polarization of the synchrotron radiation, the diffractometer has been mounted vertically within an aluminum shielding box. The whole system is supported for alignment purposes by a tiltable and turnable table actuated by five step motors, remotely controlled by a microcomputer. The four step motors of the diffractometer are also controlled by microcomputer. A program written in Assembler gives the maximum flexibility during routinary operations and data collections; while an intelligent interface allows the use of a host computer for the subsequent data elaborations (Fig. 1).

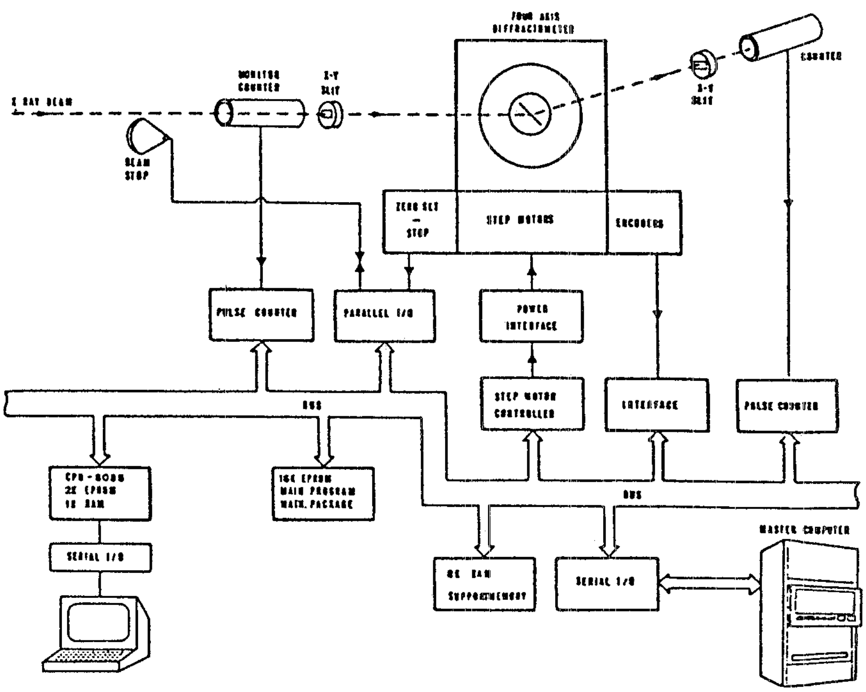

Fig. 1 - Electronic control and data acquisition system for the four circle diffractometer.

During the first experimental works X-ray diffraction pattern has been obtained from different single crystal and powder samples. In Fig. 2 the rotating crystal pattern of 3.5-dicyano-nitro-benzene compound is shown. The symmetrical disposition of the spots declares the good instrument alignment, while their net shape proves the intrinsic source collimation. The first experimental results also guarantee the possibility to determine in situ the crystal orientation matrix which is necessary for the successive data collection.

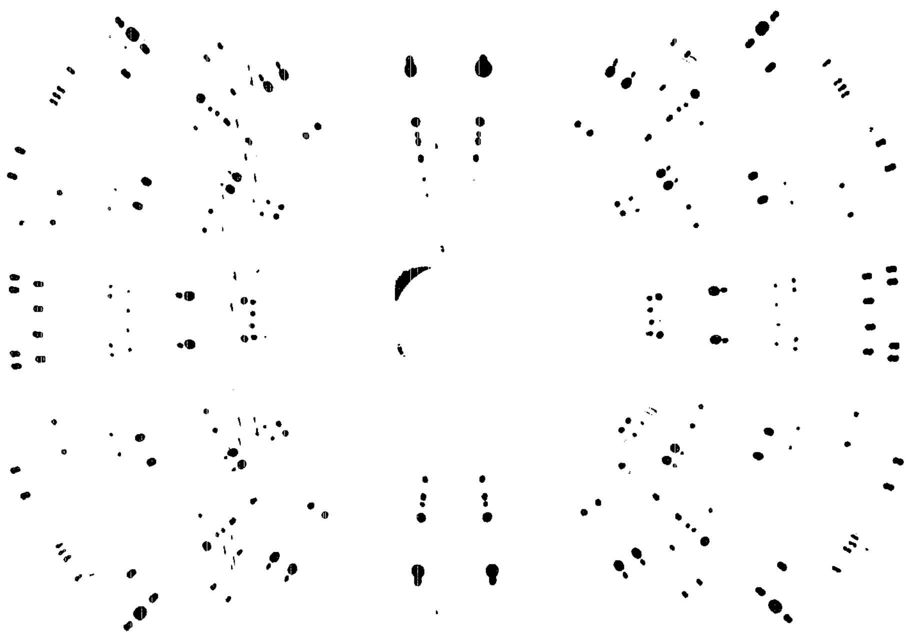

Fig. 2 - Rotation pattern of 3.5-dicyano-nitro-benzene single crystal.

A removable Debye-Scherrer Camera, has been also placed inside the supporting box. This device allows to obtain rotating-crystal photographs both from micro-powder and from liquid-crystal samples. The effectivenes of using the intense synchrotron radiation to study liquid samples is proved by the diffraction pattern given in Fig. 3. The net and sharp image of a lauril-sulfate sample has been easily obtained with an exposition of only three hours in comparison with one day usually required, by using traditional X-ray sources, in order to have the same image quality.

In the near future measurements will be made on a quartz microcrystal in order to solve the phase problem according to the three beam theory(3).

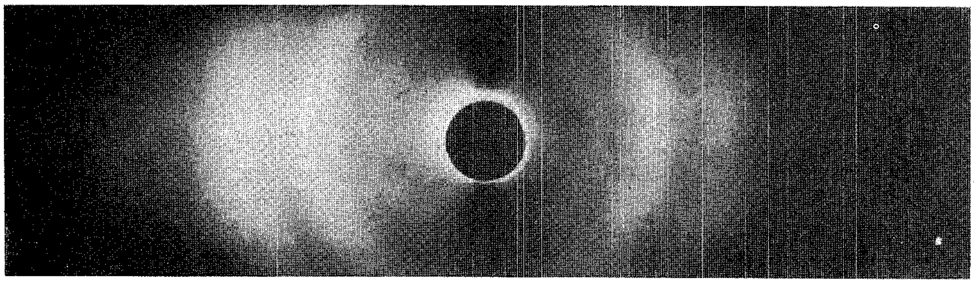

Fig. 3 - Diffraction pattern from an aqueous solution of lauril-sulfate.

References:

(1) E.Burattini et al., Nuclear Instr. and Meth. 208, 91 (1983).
(2) Proceedings of the IX European Crystallographic Meeting, Torino 1985, Abstracts (1-014).
(3) R.Colella, Acta Cryst. A30, 413 (1974).