MEASUREMENT SYSTEMS

BL2A Fluorescence Apparatus for Studies of Vapor Phase Photochemistry

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An apparatus for studies of photochemical processes in the vapor phase using fluorescence spectroscopy has been constructed on the beam line BL2A. Figure 1 shows a schematic of the apparatus. The optical system was designed such that (i) synchrotron radiation with a horizontal divergence of 40 m radians can be accepted by a 1 m Seya-Namioka monochromator (Hitachi SN2), and (ii) final dispersed synchrotron radiation

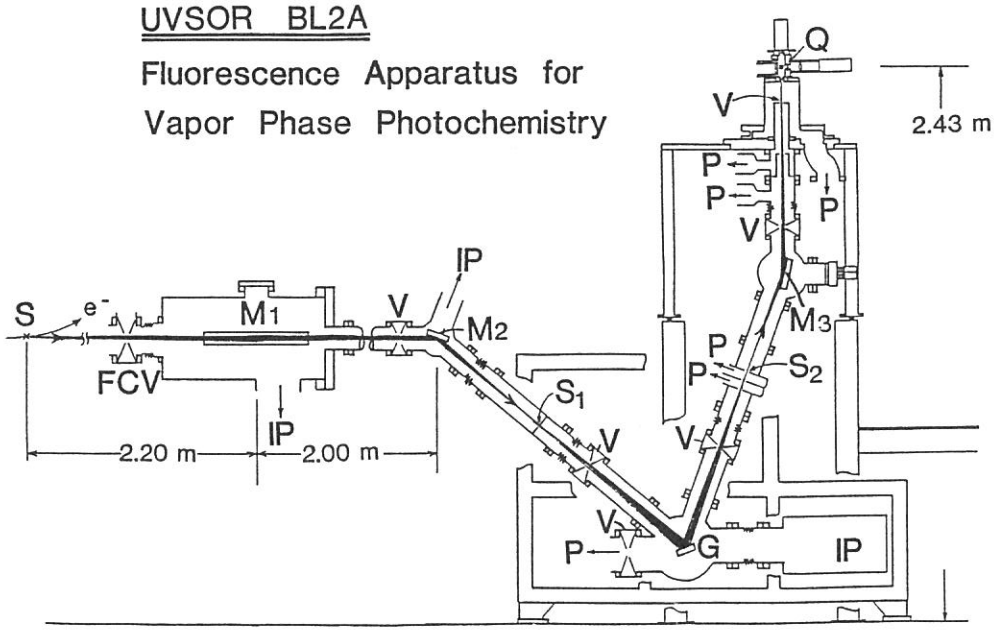

Fig. 1. Schematic side view of the fluorescence apparatus for vapor phase photochemistry. From left to right, S: source point of synchrotron radiation; FCV: fast closing valve; M1: first focusing mirror; M2: second focusing mirror; S1: entrance slit into 1 m Seya-Namioka monochromator; G: grating; S2: exit slit; M3: post focusing mirror; Q: synchrotron radiation- molecule interaction region (spot position). IP: ion pump; V: shutoff valve; P: turbomolecular pump.

goes upward. This apparatus was designed to use both samples in a gas cell and supercooled molecules or molecular complexes in a free jet. So far gas cell experiments have been done. Dispersed SR, after being focused by a toroidal mirror(M3), enters through a LiF window into a gas cell with a 10.9 cm pass length. Photon flux was monitored by a combination of sodium salicilate converter and photomultiplier (Hamamatsu R585). Fluorescence was detected perpendicularly to the incident light by a photomulti-plier tube (Hamamatsu R585). Intensities of the transparent light and fragment fluorescence, the sample pressure, and the electron beam current in the storage ring were concurrently monitored for every stepwise scan of the monochromator wave length, and were inter-faced into a microcomputer (NEC PC9801) via GP-IB interface (see Fig. 2). The photoabsorption and the relative fluorescence cross section were determined vs wave length.

Dispersed fluorescence spectra have been measured by using a Nikon P250 monochromator, installed between the gass cell and HP R585 photomultipier tube for emission detection. With purging N_2 gas flow emission above 180 nm was recorded. Fluorescence polarization measurement and studies of photochemical processes by means of molecuar beam vacuum UV spectroscopy of molecular complexes are under way.

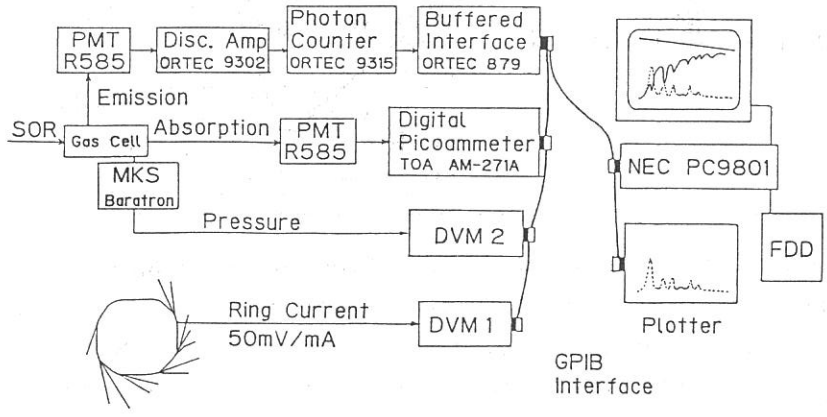

Fig. 2 Block diagram for absorption and fluorescence measurements. Data was transfered via IEEE 488 (GP-IB) interface from and to NEC PC-9801 microcomputer.

CONSTRUCTION OF A SUPERSONIC MOLECULAR BEAM APPARATUS FOR BEAM LINE BL2B2

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A supersonic molecular beam apparatus for the beam line BL2B2 has almost been completed. A technique of supersonic beam is apparently suitable and desirable for spectroscopic studies of molecular clusters as well as free molecules. Particularly a combination of this technique with a synchrotron radiation light source would undoubtedly open new aspects in the fields of vacuum UV photophysics and photochemistry.

As shown in Figure 1, in the BL2B2 the synchrotron radiation light beam is focused on the entrance slit of a 1 m seya monochromator by three pre-focusing mirrors, and then after passing through the monochromator, the light beam is further focused by a toroidal mirror.

Schematic side view of our molecular beam apparatus is shown in Figure 2. The photon-molecular beam collision chamber is separated from a beam-source chamber with a skimmer, and each chamber is evacuated by two turbo molecular pumps (1500 l/s, 1000 l/s) or an oil diffusion pump (5000 l/s). There is a two-stage differential pumping system between the molecular beam apparatus and a monochromator exit slit to keep the vacuum in the monochromator below 2×10^{-9} Torr under operation. This apparatus would enable us to study; 1) vacuum UV absorption spectra of ultracold free molecules, 2) half collisional reaction processes of superexcited or ionic molecular clusters using a mass selected photoionization technique, and 3) photoelectron energy and angular dependence spectra of molecules and molecular clusters.

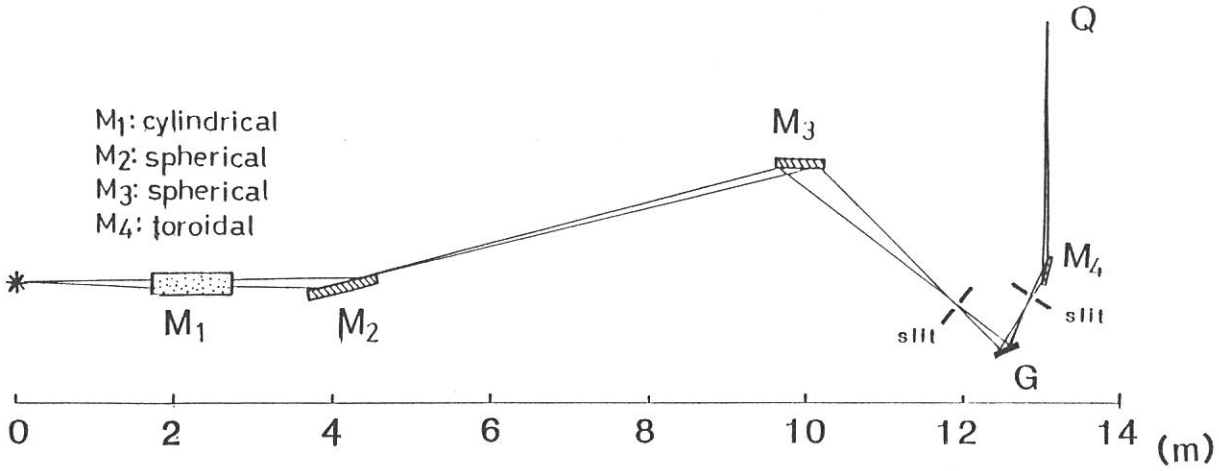

Figure 1 An optical system for beam line BL2B2.

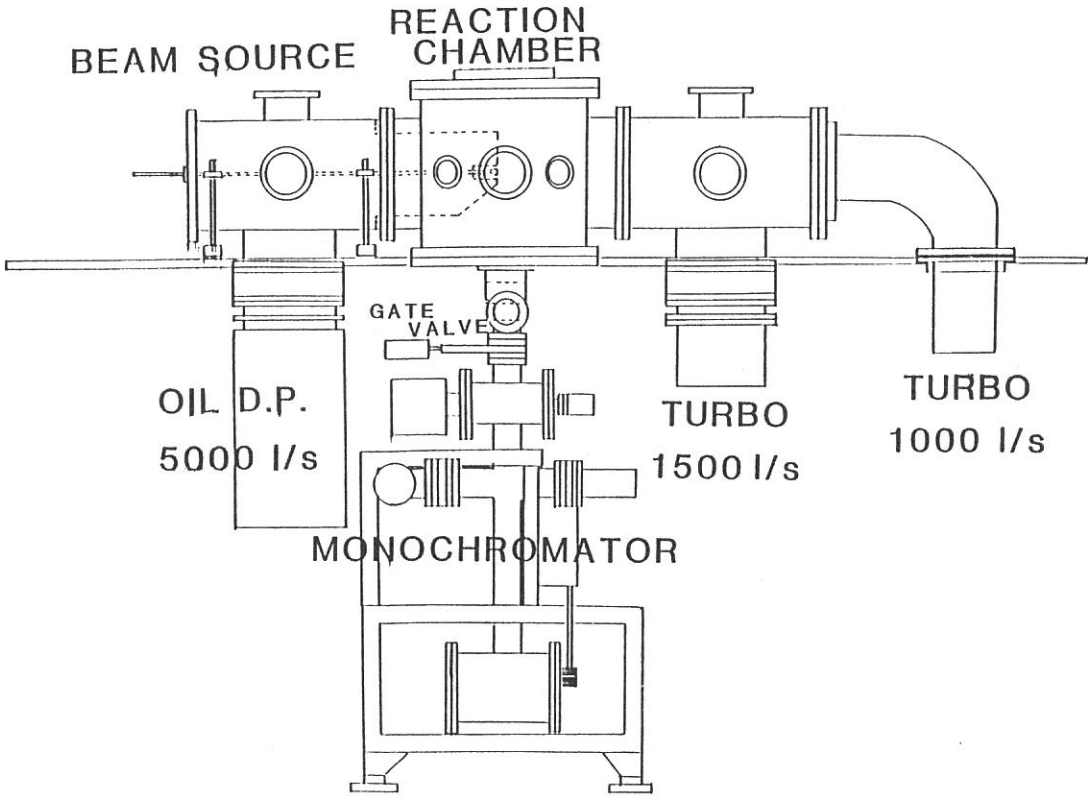

Figure 2 A schematic side view of a supersonic nozzle beam apparatus.

STATUS OF BL3A1

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BL3A is a beam line for the undulator radiation. At a pre-mirror chamber of BL3A the beam line is split into two, which are BL3A1 without a monochromator and BL3A2 with a monochromator (2 m constant deviation grazing incidence) under construction. Fig. 1 shows the cross sectional view of the pre-mirror chamber. At the entrance of the pre-mirror chamber, movable diaphragms are located to limit acceptance angle of the undulator radiation for both horizontal and vertical directions. Mirrors M_{-1}, M_0 and M_1 introduce the synchrotron radiation from B_3 bending section to the monochromator. At BL3A1, one can use the undulater radiation as the quasi-monochromatic light between 500-230 Å (at 600 MeV) by the use of filters. Before the sample chamber an ion-chamber and a photo-electron spectrometer of gases will be attached with a differential pumping section to measure the intensity and the line shape of the undulator radiation, respectively. An Al photo-cathode beam monitor is also mounted before the sample chamber. The plan view of the S_3 straight section and BL3A1 is given in Fig. 2.

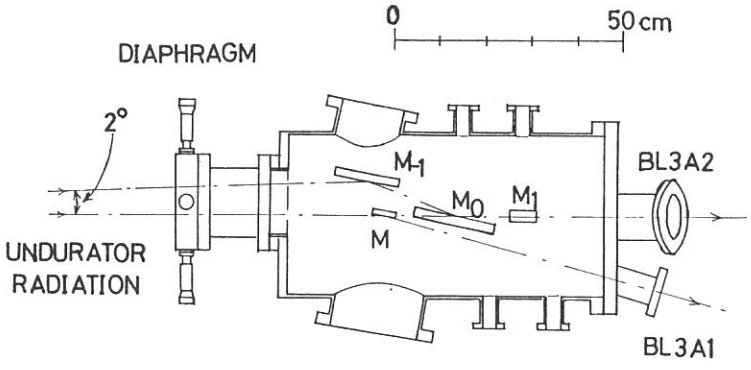

Fig.1 Cross sectional view of BL3A pre-mirror chamber. M, M_{-1}, M_0 and M_1 are pre-mirrors.

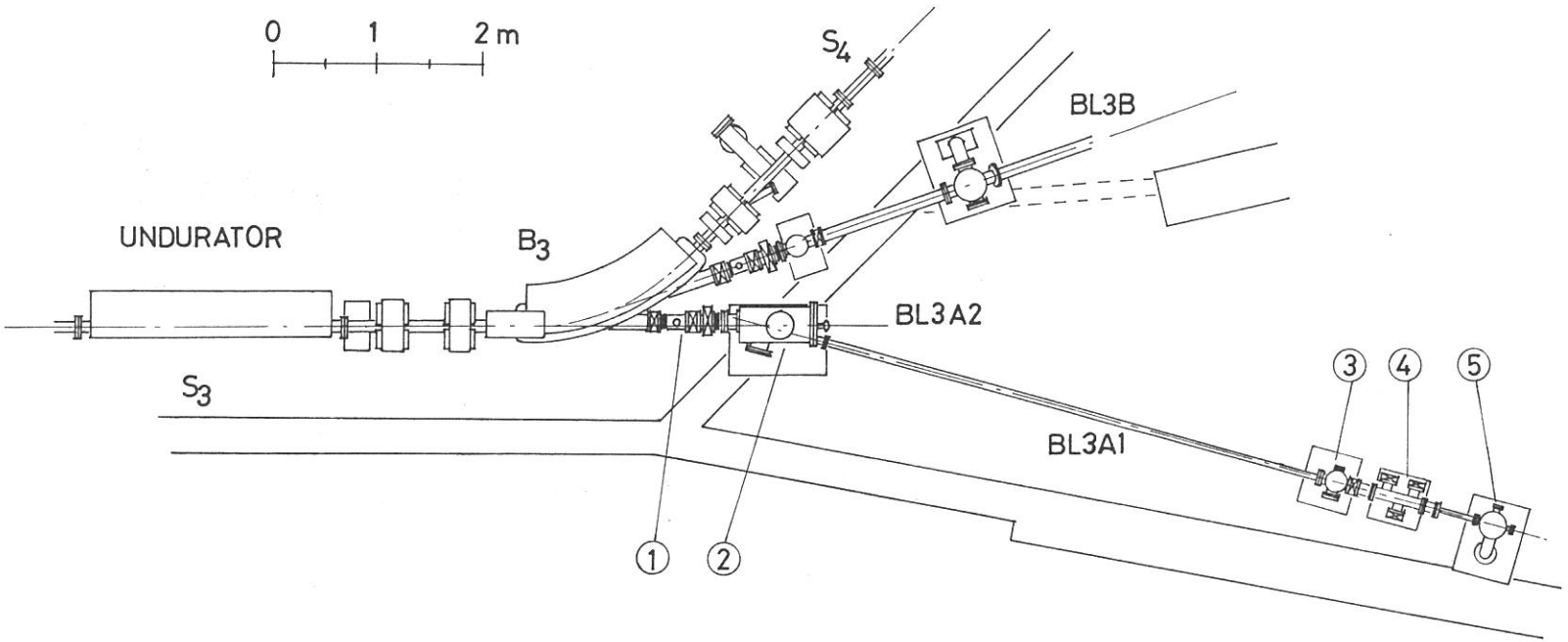

Fig.2 Plan view of the S_3 straight section and BL3A1.
1: front end, 2: pre-mirror chamber, 3: pumping station,
4: differential pumping section and 5: sample chamber.

THE TEPSICO-II APPARATUS AT BL3B FOR GAS PHASE PHOTOIONIZATION STUDIES

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At the UVSOR facility, several experimental stations for the study of elementary atomic and molecular processes in the gas phase have been constructed intensively over the last few years. Here, we report a system which has been designed for the study of secondary reactions of molecular ions produced by synchrotron radiation. The main objective is the state selection of the reactant ions, and for this purpose a novel coincidence technique TESICO[1] (threshold electron - secondary ion coincidence) is incorporated.

A schematic drawing of the apparatus, nicknamed TEPSICO-II, is shown in Fig. 1. The monochromator is a vertically dispersed normal incidence type (McPherson Type) with 3 m focal length and 10° angle between incidence and diffracted beam. It has a mechanically ruled aberration-corrected concave grating[2] with 1200 lines/mm groves. A cylindrical mirror M_1 and two spherical mirrors M_2 and M_3 focus SR of 20 mrad horizontally and 6 mrad vertically onto the entrance slit S_1, as shown in Fig. 2. The monochromatized light is ultimately directed upward and focused at a point Q in the interaction region by a toroidal mirror M_4.

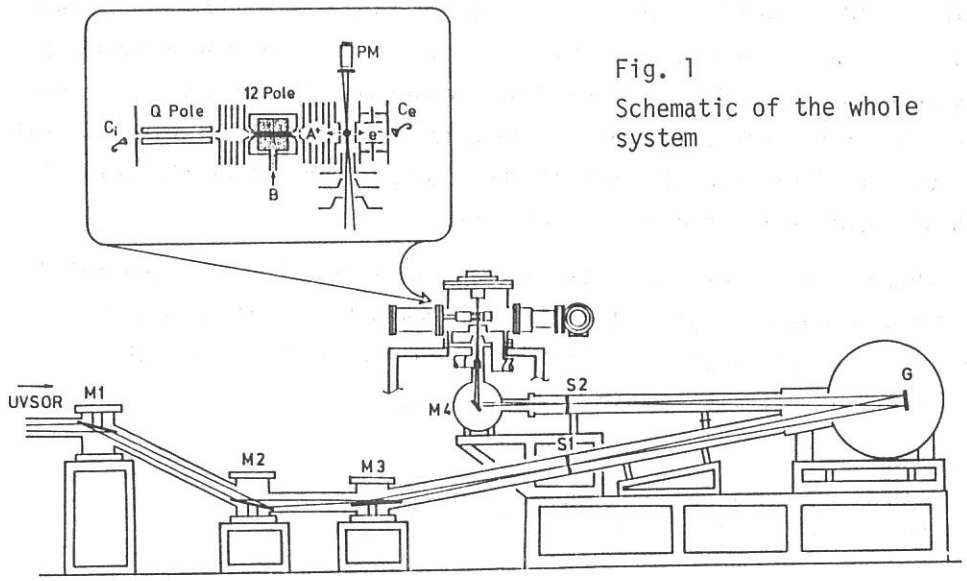

Fig. 1
Schematic of the whole system

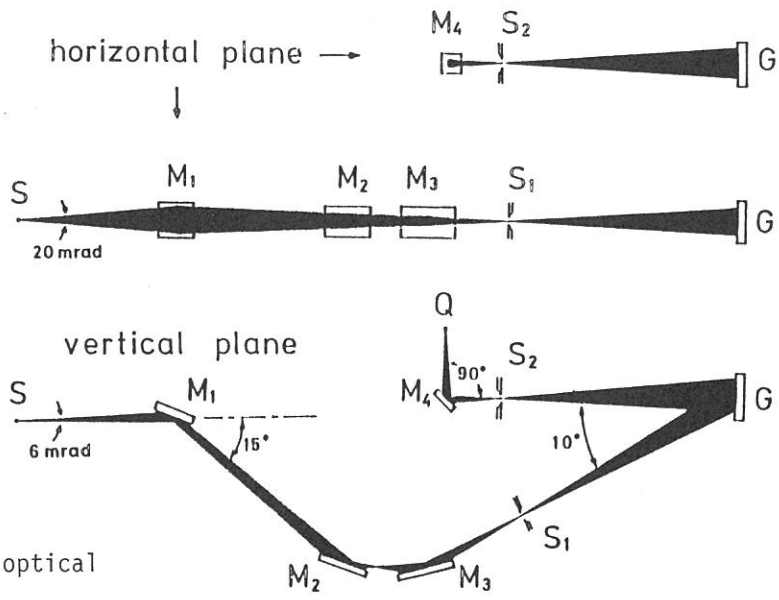

Fig. 2
Scheme of the optical layout

Sample gas A is introduced into the interaction region as a free jet and photoionized therein. Ions A^+ and photoelectrons e^- produced are expelled from the ionization region by a weak field in the directions opposite to each other and perpendicular to the photon beam. Threshold electrons are selected from the photoelectrons with finite kinetic energies by means of a non-line-of-sight steradiancy analyzer of our own design. Ions, on the other hand, are led into the reaction zone filled with neutral reactant B and surrounded by 12 poles on which RF voltages of opposite signs are applied alternately. Reaction takes place in this confining field produced by these 12 poles, so that all product ions, as well as all reactant ions entering this region, pass through the quadrupole mass spectrometer and are detected by channel multiplier C_i. This ion signal is then counted in coincidence with the threshold electron signal. The 100 % collection efficiency of the product ions allows the determination of absolute reaction cross sections.

These sections of the system are assembled together via several differential pumping systems. Experiments showed that, with a stagnation pressure of sample A of 2 atm, no deterioration of the ultrahigh vacuum (1×10^{-8} torr) in the monochromator is caused at all.

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FAR-INFRARED SPECTROSCOPY OF SOLID AT BL6A1

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A system for spectroscopy in the far-infrared region at the beam line BL6A1 covers a wavenumber region from 5 to 250 cm^{-1}.

I. Collection of Long Wavelength Light

Figure 1 shows the optical system. The radiation from the orbit is reflected by the plane mirror M_1^p and collected by a spherical mirror M_2^s. The long wavelength radiation diverges considerablly and a large acceptance angle is necessary for the collecting system. The light is reflected by two plane mirrirs M_3^p and M_4^p which are beneath M_1^p and M_2^s. After passing through a Si window, it is guided to the collimating system. A wedge shaped Si window of 2mm thickness separates the high and low vacuum parts. The mirror M_1^p is made of copper and has a small cut through which the short wavelength light within 20×10 $mrad^2$ passes to another system. The surface of the all mirrors are coated with gold.

II. Spectrometer and Sample Chamber

The collected light beam is made parallel by the toroidal mirror M_5^T in Fig.1. The cross sectional size of the parallel beam is about 36×45 mm^2. The horizontally polarized component is selected by the wire grid polarizer WG_1 and lead to a Martin-Puplett interferometer made by Specac Co.

The interferometer is separated from the sample chamber by a polyethylene window of 1 mm thickness. The light is polarized horizontally or vertically according to the direction of a wire grid polarizer WG_2. The light beam is collected by a toroidal mirror M_7^T on a sample S. In the case of the observation of the reflectivity, reflected light from the sample is collected by a spherical mirror M_8^S to a detector D. The angle of incidence is 7.5°. In the case of the transmission measurement, the detector is displaced to a position D' behind the sample as shown by the dashed line in Fig.1.

III. Spectral response

In Fig.2, spectral response is shown from 10 to 250 cm^{-1} detected by a Golay cell at the position D' in Fig.1 without any sample. The current of the UVSOR ring is 45.5 mA. The spectra show the intensities of light passed through different sizes of a diaphram placed at the sample position S. When the diameter of the aperture is larger than 3 mm, the intensity on the high energy region does not decrease. This result shows that owing to the low emittance of the source, an effective size of the beam at the sample position is as small as about 3×3 mm^2. This is a remarkable property because a small spot of slightly convergent light is obtained with a high intensity. The dashed curve in Fig.2 shows the intensity of light from a high pressure mercury lamp through 3 mm aperture. The intensity is much weaker than that of the synchrotron radiation at the current of 45.5 mA. Using this property, we have observed anisotropic reflectivity spectra of a K_2AgI_3 single crystal of 2 mm diameter at 80 K.

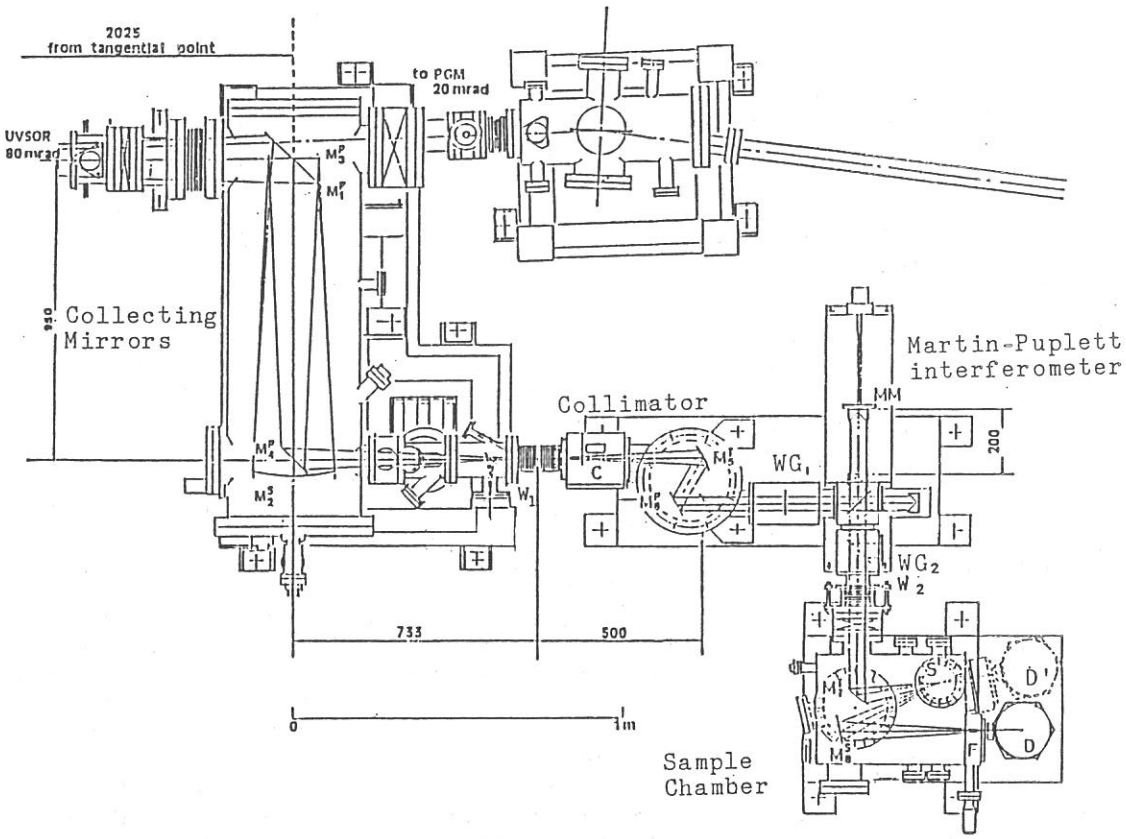

Fig.1 Optical system for far-infrared spectroscopy at BL6A1.

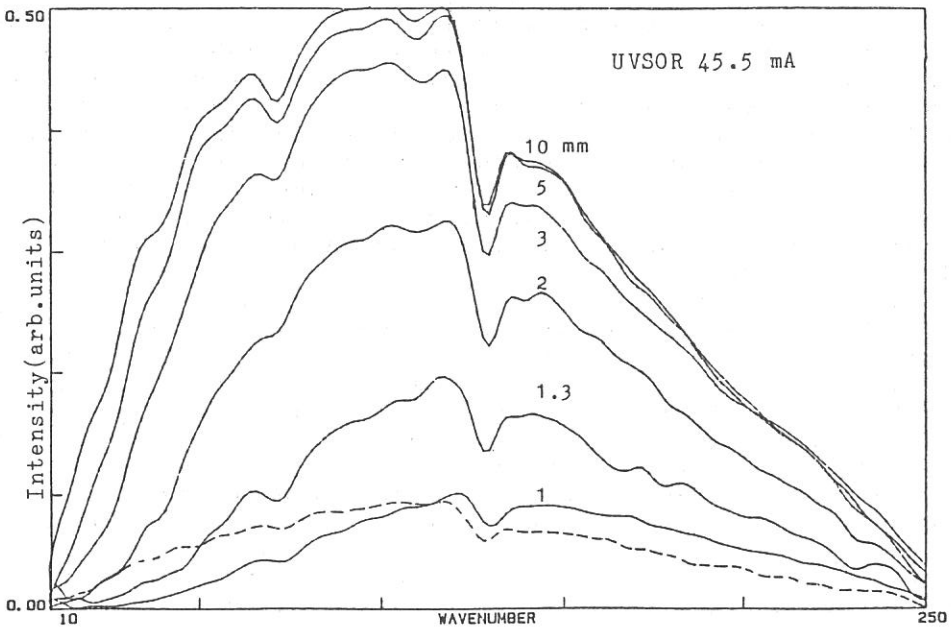

Fig.2 Intensity of spectra detected by Golay cell through apertures of different diameters placed at the sample position. Spectral resolution $\Delta\nu$ is 6 cm^{-1}. The dashed curve is the intensity of a Hg lamp through 3 mm aperture.

PLANE-GRATING MONOCHROMATORS FOR 2 EV < hν < 100 EV AT BL6A2 AND BL8B2

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At BL6A2 and BL8B2, we adopt two plane-grating monochromators (PGM) of the same design for studying solids by photoelectron spectroscopy and optical measurements, with moderate energy resolution (0.1 - 0.2 eV) and intensity. PGM can cover a farily wide range of photon energy with a good spectral purity.[1-6] The hν range of the present monochromator covers the whole valence excitations of various solids, and is also sufficient for mapping the energy-band dispersion relation E = E(k) by angle-resolved photoemission technique.

The optical design is illustrated in Fig. 1. Divergent synchrotron radiation is made parallel by two premirrors (M_0 and M_1) and vertically

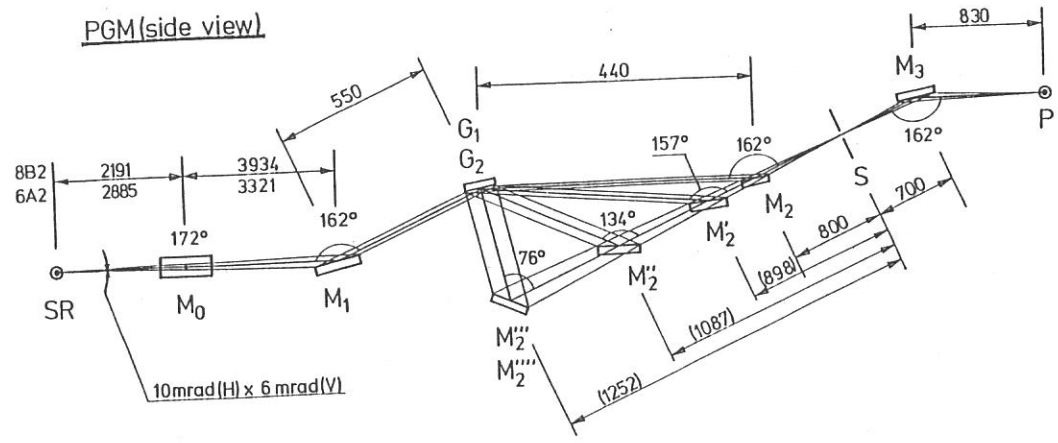

Fig. 1 Schematic of the optical design of the PGM

diffracted by one of the two innterchangeable, mechanically-ruled gratings (G_1; 1200 l/mm, G_2; 450 l/mm). The diffracted light is focused by one of the five cylindrical mirrors (M_2 - M_2'''') onto the exit slit S. Finally, the toroidal mirror M_3 focuses the divergent radiation onto the sample P in the measurement chamber. The deterioration of the resolution by the finite source size is minimized by virture of the small source-size of UVSOR (design values σ_x = 0.3 mm and σ_y = 0.2mm).

Rather similar performances have been observed for the two monochromators. The observed relative intensity distribution, at a slit width of 500 μm, is shown in Fig. 2. It was measured with a photomultiplier with a sodium salicylate energy converter. The unit of the ordinate could be calibrated for an absolute value of ∿2 x 10^{11} photons/s at a ring current of 27 mA, by measuring the photoemission from an Al-foil and using a reported value of photoemission yield.[7] Later, the exchange of a miss-blazed G_1 led to an improvement of this distribution, including the increase of the intensity at 10 eV $h\nu$ < 20 eV. The energy calibration was performed by measuring sharp features in the absorption spectra of rare gases, O_2 gas, CH_3I gas and alkali halide films. The resolution at a slit-width of 300 μm was found to be 0.015 - 0.3 eV in the wavelength range from 230 to 13.5 nm, from the sharpness of the features in these spectra. The spot size of the zeroth-order light at the sample surface (P) is < 1 x 1 mm^2, as designed.

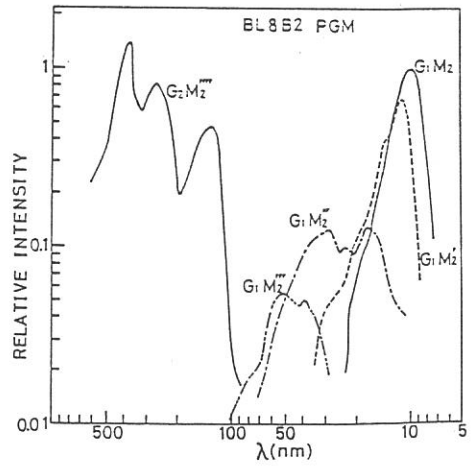

Fig. 2 Spectral intensity distribution of the PGM

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PERFORMANCE OF A DOUBLE CRYSTAL MONOCHROMATOR FOR BL-7A OF UVSOR

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A high vacuum compatible constant offset double crystal monochromator (DXM) was constructed and tested at BL-7A using soft x-ray emitted from a normal bending magnet section of UVSOR. A schematic layout of the beam line of BL-7A is given in Fig. 1. The constant offset during the rotation of crystal plane was realized by a simple linkage of linear motions of the two crystals with two linear guides placed on both imput and output beam levels. The mechanism is similar to that of Cowan et al.[1] and will be described in detail elsewhere. The mechanical movement was found to be very smooth through a full rotation of 60° (between 15° and 75° of Bragg angle), and the constant offset was successfully achieved.

The intensity of the monochromatized light was monitored with Hamamatsu R-595 electron multiplier with a first dinode of Cu-Be or evaporated CuI. The output current was lead into TOA AM-271A digital picoammeter. The control of goniometer and data acquisition were made with NEC PC-9801E microcomputor through digital I/O and/or IEEE-488 standard interface bus.

The monochromator crystals such as KAP(100), mica(001) and beryl(10$\bar{1}$0) whose 2d value is 26.64Å, 19.80Å, and 15.98Å, respectively were tested. In Fig. 2 is shown the throughput transmission spectrum of the DXM for beryl crystal at the operating condition of the ring of 750MeV and about 30 mA. No radiation damage was observed for both beryl and mica crystals. The KAP crystal, however, suffered a serious damage on the surface even in the 600MeV operation.

Fig. 3 shows the Na K-edge near edge absorption spectrum of a thin film of NaCl which was evaporated in situ on a collodion film in the sample chamber. Despite the presence of many structures due to impurities and constituent atoms in the monochromator crystal in the throughput transmission spectrum, no structure appears in the absorption spectra of NaCl. All the structures were eliminated by the division of signals without and with a sample. From the doublet separation of the spectrum in Fig. 3 we can estimate the resolution of about 1eV which is practically sufficient for this energy range.

Further studies of other crystals such as InSb (111) and Quartz (10$\bar{1}$0) along with the spectrum of x-rays emitted from a superconducting horizontal wiggler are also planned.

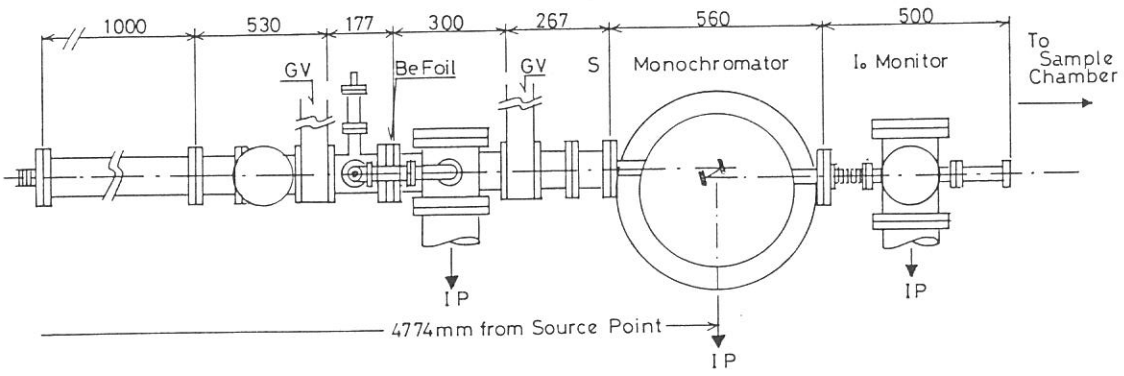

Fig. 1 Schematic layout of BL-7A. GV and IP mean gate valve and ion pump, respectively.

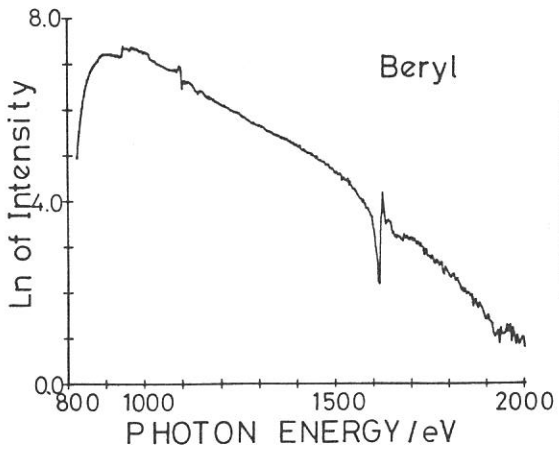

Fig. 2 Throughput transmission spectrum of DXM with beryl monochromator crystal.

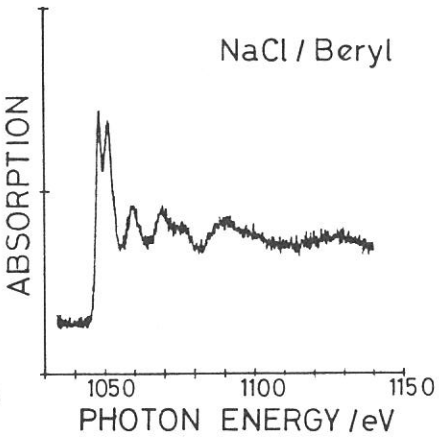

Fig. 3 Na K-edge near edge spectrum of NaCl taken with beryl crystal.

Reference

1) P. L. Cowan, J. B. Hastings, T. Jach, and J. P. Kirkland, Nucl. Instrum. Methods 208, 349 (1983)

The Vacuum-UV Spectrophotometer Installed in the BL-7B Beam Line

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A vacuum-UV spectrophotometer using a improved 1 m Seya-Namioka-type monochromator has been constructed by the members of the Equipment Development Center and installed in the BL-7B beam line. An outline of the spectrophotometer is shown in Fig. 1. In order to achieve a maximum through-put of the monochromatic light, the spectrophotometer was designed on the basis of the results of the ray-trace calculation from the light source to a sample point through two troidal focussing-mirrors. A further improvement was made by employing a new microcomputer-driven mechanism for exchange of two concave gratings (600 or 1200 and 2400 ruled lines per mm with different blaze angles) mounted in the monochromator under an ultra-high vacuum. The expected efficiency of the spectrophotometer has been almost satisfied as follows; the intensity of the monochromatic light is about 10^{10} photon/sec with $\Delta\lambda/\lambda = 1\%$ at 50 nm for the storage ring currents of 40 mA, a maximum resolution of 0.15 nm for 1200 line/mm and a beam size on a sample position of about 1x2 mm^2 with a beam aperture of 26 mrad. In Fig. 2, the relative intensities of the monochromatic light are plotted for two kinds of gratings as a function of photon energy. It is found that that, by exchanging the gratings at 18 eV, this spectrophotometer is available in a wide energy region from 0.2 to 40 eV, being free from mixing with a stray light or the second-order diffracted light. The reproducibilities of exchanging and

scanning motions of the gratings are in a same order accuracy as the maximum resolution.

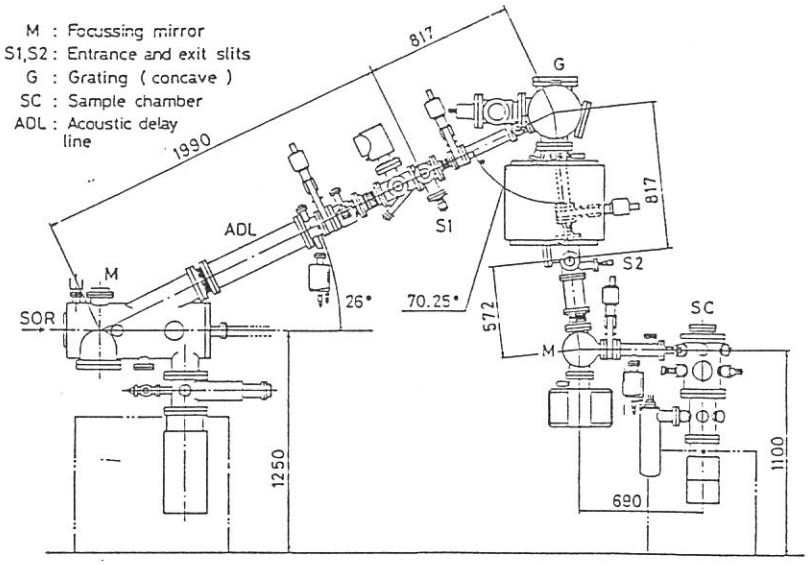

Fig. 1 Schematic diagram of the vacuum-UV spectrophotometer.

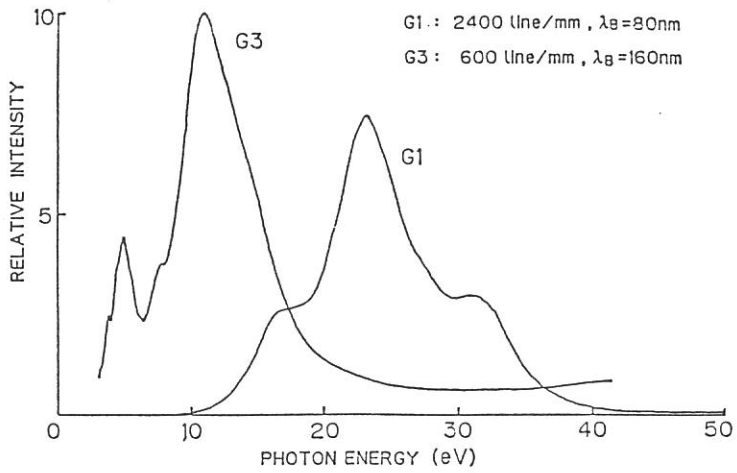

Fig. 2 The intensity of the monochromatic light for two kinds of gratings.

STRUCTURE OF BL8A

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Institute for Molecular Science

BL8A is a free port. Its plan and side views are shown in Fig.1 and Fig.2, respectively. It is composed of a front end, an acoustic delay line and a separation chamber. The front end is different a little from common ones described in p. 11. That is, a fast closing valve has a viton gasket so that it can shut out air completely. Its closing time is 20 ms. The acoustic delay line is 1 m long. It has eight diaphragms, the aperture of which is 80 mm x 27 mm, and two ion sputter pumps. It delays propagation time of leakage of 1 atm air by 40 ms. It also acts as a differential pumping system and can keep pressure difference of one order. A pneumatic valve after the acoustic delay line (indicated as 16 in Fig.2) has a glass window. One can aligne instruments by the use of SOR passing through the window. After that, the separation chamber is located. Users can connect their own instruments after this chamber. When users make irradiation experiments, such as lithography, they can use this chamber as a sample chamber.

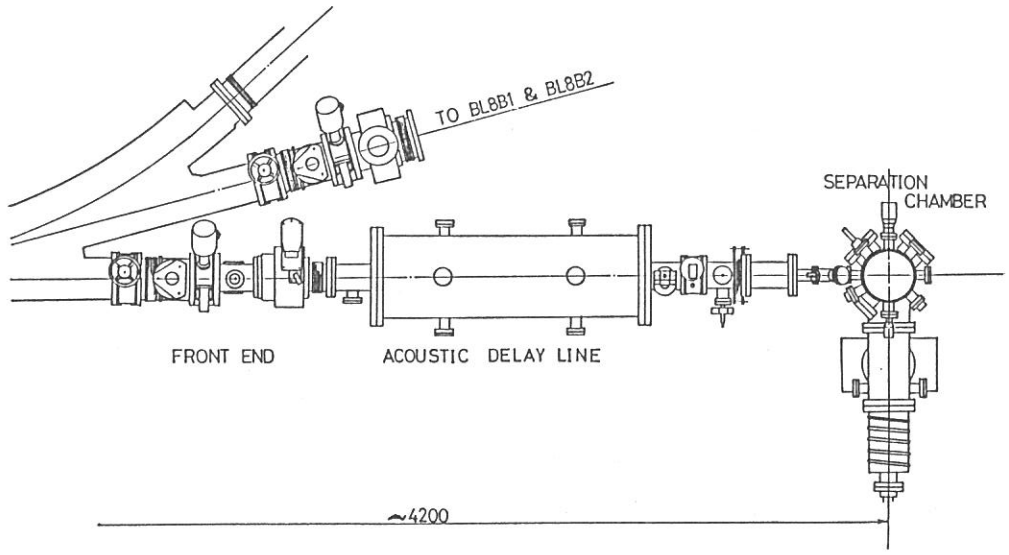

Fig. 1 Plan view of BL8A.

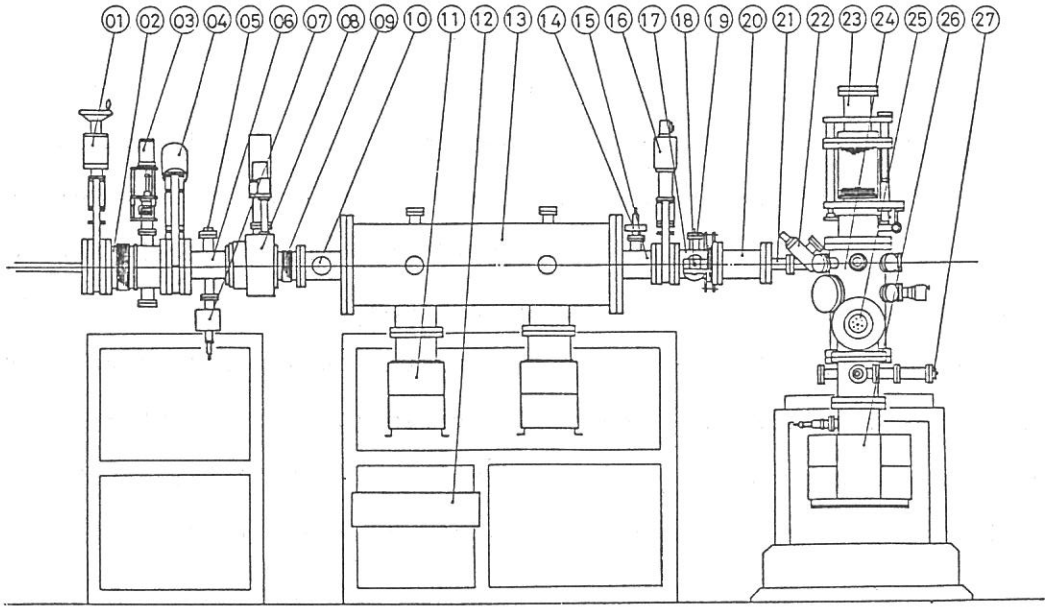

Fig. 2 Side view of BL8A. 01: manual valve, 03: beam shutter, 04 and 16: pneumatic valves, 05 and 27: ion gauges, 07,11 and 26: ion sputter pumps, 08: fast closing valve, 14: sensor for fast closing valve, 18: appendage pump, 19: pirani gauge, 22: straight through valve and 25: titanium sublimation pump.