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भाभा परमाणु अनुसंधान केंद्र

Bhabha Atomic Research Centre

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ATOMIC ENERGY COMMISSION

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| 21 | *Affiliation of author(s) :* | 1) Spectroscopy Division, Bhabha Atomic Research Centre, Mumbai<br>2) Centre for Design and Manufacture, Bhabha Atomic Research Centre, Mumbai<br>3) Physics Group, Bhabha Atomic Research Centre, Mumbai |
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# इण्डस-I सिन्क्रोट्रॉन विकिरण स्रोत पर उच्च विभेदन निर्वात-पराबैंगनी बीम लाइन का विकास


## सारांश

परमाणुओं और अणुओं के अवशोषण वर्णक्रम का अध्ययन करने हेतु एक उच्च विभेदन निर्वात-पराबैंगनी बीम लाइन का 450 MeV इण्डस-I सिन्क्रोट्रॉन विकिरण स्रोत पर विकास किया गया है। इस बीम लाइन के मुख्यतः तीन भाग हैं : (i) संकेन्द्रण प्रकाशिक निकाय (तंत्र), (ii) अवशोषण कोष्ठिका तथा (iii) ईगल मॉउन्ट में एक 6.65 m उच्च विभेदन निर्वात-पराबैंगनी वर्णक्रममापी। इस वर्णक्रममापी की तरंगदैर्घ्य सीमा 700 Å से 2000 Å के बीच है तथा इसकी विभेदन क्षमता 0.01 Å है। इण्डस-I सिन्क्रोट्रॉन विकिरण स्रोत का उपयोग करके ऑक्सीजन, अमोनिया और कार्बन डाईसल्फाइड के अवशोषण वर्णक्रम क्रमशः 1750 Å, 1881 Å तथा 3100 Å के तरंगदैर्घ्य पट्टी पर अभिलेखित (रिकार्ड) किया गया। इस रिपोर्ट में उच्च विभेदन निर्वात-पराबैंगनी बीम लाइन की बनावट और विकास के विभिन्न पहलुओं के बारे में विस्तार से चर्चा की गयी है।

# Development of High Resolution Vacuum Ultraviolet Beamline at Indus-1 Synchrotron Source

R.P. Shukla, N.C. Das[+], D.V. Udupa, Saraswathy P., Sunanda K., S.N. Jha, Aparna Shastri, Paramjeet, Singh, Manika Mallick, A. P. Mishra, N K Sahoo

*Spectroscopy Division.*

A.K. Sinha, S. Bhatt

*Centre for Design and Manufacture*

V.C. Sahni

*Physics Group*

*Bhabha Atomic Research Centre, Trombay, Mumbai 400 085*

## Abstract

High Resolution vacuum ultraviolet beamline at Indus-1 450 MeV synchrotron source has been developed for carrying out absorption spectral studies of atoms and molecules. The beamline consists of three major parts i.e., a focusing optical system, an absorption cell, and a high resolution 6.65 m vacuum ultraviolet spectrometer in Eagle mount. The wavelength range of the spectrometer is from 700 Å to 2000 Å and the resolution of the spectrometer is 0.01 Å. Using the synchrotron source Indus-1, the absorption spectra of oxygen, ammonia and carbon disulphide have been recorded at the wavelength band of 1750 Å, 1881 Å and 3100 Å respectively. Details of different aspects of design and development of the high resolution VUV beamline are described in this report.

[+] Ex- Head, Spectroscopy Division, BARC, Mumbai 400 095 and currently BRNS Senior Scientist.

## 1. Introduction

Synchrotron radiation is an intense source of electromagnetic radiation emitting in the wavelength range from X-rays to infrared rays. It is a very useful continuum source of electromagnetic radiation for absorption spectral studies in the soft X-ray and vacuum ultraviolet region as there are no intense continuum sources or tunable lasers available in this wavelength region. A 450 MeV Indus-1 synchrotron with critical wavelength of 61 Å is in operation at Centre for Advanced Technology, Indore. The wavelengths of radiation are in the range of 40 Å – 6000 Å with photon flux of 7.2 x $10^{11}$ photons/ sec/mrad/0.1% bandwidth at critical wavelength of 61 Å. A high resolution VUV beamline has been developed for carrying out photo absorption studies of atomic and molecular species in the wavelength range of 700 Å – 2000 Å. The development of the beamline consists of three parts viz. a) Focusing optics b) Absorption cell c) High Resolution VUV spectrometer.

The development of high resolution VUV spectrometer and its performance evaluation have been described in earlier papers [1, 2]. The high resolution VUV spectrometer consists of a concave spherical grating of radius of curvature 6.65 m and the R number (radius of curvature/effective aperture) of 40. This R number is equivalent to a semi-numerical aperture of about 10 mrad. Therefore the acceptance angle of the spectrometer for entrance beam is 20 mrad in vertical direction. We have chosen a vertical divergence of 6 mrad and horizontal divergence of 60 mrad of synchrotron radiation for use in the high resolution VUV spectrometer. It is, therefore, necessary to expand the synchrotron radiation beam in the vertical direction by a factor of three and reduce the horizontal divergence also by a factor of three such that the synchrotron beam should illuminate the whole aperture of the grating. Hence we have designed and developed a focusing system consisting of three cylindrical mirrors to condense the beam to a horizontal line which fills the entrance slit of the spectrometer. The details of the focusing system are presented in this paper. The focusing system is coupled to the absorption cell, which in turn is coupled to the VUV spectrometer. Synchrotron radiation passes through the absorption cell filled with the gases to be studied and is dispersed by the spectrometer. The output signal is detected by a photomultiplier and recorded using data acquisition system (DAS) coupled to personal computer (PC). The first experiment has been carried out for the performance evaluation of the VUV beamline by recording the absorption spectrum of carbon disulphide ($CS_2$) and ammonia ($NH_3$). The experimental results are presented in this report along with the description of focusing optical system, absorption cell, spectrometer and evacuation procedure.

**2. Focusing optical system**

Fig.1 shows the optical system for focusing the synchrotron radiation beam on the entrance slit $S_2$ of the spectrometer. A cylindrical mirror $M_1$ of radius of curvature r = 0.881 m is used to collimate the synchrotron beam in the horizontal direction. The synchrotron beam falling on the first mirror is reflected through an angle of 79.85°. The focal length of the concave cylindrical mirror in the sagittal plane is calculated by the following relation:

$$f = \frac{r}{2\cos i} \qquad (1)$$

where r is the radius of curvature of the mirror, and i is the angle of incidence.

By taking r = 0.881 m, i = 79.85°, the distance between point $S_1$ and the cylindrical mirror $M_1$ is calculated to be 2.5 m for collimating the synchrotron beam in the horizontal direction. The cylindrical mirror $M_2$, of r = 4.513 m is used at an angle of incidence i = 72.81° for focusing the synchrotron beam. The focal length of the cylindrical mirror $M_2$ in the sagittal plane is calculated to be 7.6 m by the relation (1) and this mirror focuses the horizontal component of the synchrotron beam at the entrance slit of the spectrometer. Cylindrical axes of the mirrors $M_1$ and $M_2$ lie in the vertical plane. The axis of the third mirror $M_3$ is kept parallel to the horizontal direction and therefore it acts like a plane mirror in the horizontal plane. Consequently the emerging synchrotron beam of collimated radiation from

$M_2$ in the horizontal plane is not affected by this mirror. Hence the mirror $M_2$ focuses the beam at a distance of 7.6 m.

Let us now consider the focusing of synchrotron beam of 6 mrad vertical divergence in the vertical plane. We observe from the side view shown in Fig. 1 that the cylindrical mirror $M_1$ is acting as a plane mirror in the vertical plane. Similarly mirror $M_2$ kept at angle of incidence of 72.81° also acts as a plane mirror in the vertical direction. Thus it is the mirror $M_3$ which focuses the vertical component of the synchrotron to the entrance slit of the spectrometer $S_2$. The optical path length of the beam from source point $S_1$ to the mid point of mirror $M_3$ is 10.28 m. Radius of curvature of $M_3$ is chosen to be 38.6 m such that the focusing distance of the beam from the mirror is 3.07 m. These distances are given by the equation,

$$\frac{1}{l}+\frac{1}{l_t'}=\frac{2}{r\cos\phi} \quad (2)$$

where $l$ = object distance i.e. distance from $S_1$ to the central point of the mirror $M_3$, $l'_t$ = image distance in the tangential plane i.e. distance of the slit $S_2$ from the mirror $M_3$, r is the radius of curvature of the mirror $M_3$, $\phi$ is the angle of incidence of the synchrotron beam (central ray) on the mirror $M_3$. The above equation is satisfied for the parameters $l$ = 10.28 m, r = 38.6 m, $\phi$ = 82.97° .The specifications of the cylindrical mirrors $M_1$, $M_2$ and $M_3$ which contracts the 60 mrad synchrotron beam to 20 mrad beam in the horizontal direction and expands the 6 mrad synchrotron beam to 20 mrad beam in the vertical direction, are summarized below:

| Cylindrical mirror | Size | Radius of curvature | Angle of incidence (i) | Reflective coating |
|---|---|---|---|---|
| $M_1$ | 150 mm X 150 mm X 25 mm thickness | 0.881 m | 79.85° | Gold |
| $M_2$ | 150 mm X 150 mm X 25 mm thickness | 4.513 m | 72.81° | Gold |
| $M_3$ | 100 mm X 500 mm X 50 mm thickness | 38.6 m generated along the length 500 mm of the mirror blank | 82.97° | Gold |

The synchrotron radiation beam is focused to a horizontal line of length 10 mm and width 0.5 mm by the focusing system shown in Fig. 1. The final image of synchrotron beam was evaluated by ray tracing technique [3-5]. The measured value of the image size of the synchrotron beam on the exit slit $S_2$ is comparable to the image size predicted by theoretical calculations [3-5].

### 3. Design and fabrication of a gas absorption cell

Fig. 2 shows the diagram of a gas absorption cell which has been designed to carry out the experiments using VUV beamline. The 0.45 m absorption cell is basically a cylindrical stainless steel chamber with several ports for entrance and exit of the beam, evacuation, measurement of pressure and introduction of the sample. The design of the absorption cell must be such that we should be able to load the cell with minimum down time in between the entrance slit and $M_3$ mirror chamber exit port without disturbing the precision optical alignment of the synchrotron beam. The cell must be high vacuum compatible. Taking these important points into consideration, we have designed the absorption cell by making use of edge welded flexible bellow with KF coupling which facilitates the quick assembly to the beamline. The cell consists of edge welded bellow with adjustable tie-rods and a beam pipe of diameter 60 mm. Ports have been provided to monitor the gas flow rates, evacuation and gas introduction. Gas molecules under study are introduced from port A and evacuation can be done from port B. A gauge head is mounted on port C to measure the gas pressure in the absorption cell. Based on the above design, the absorption cell has been fabricated and leak tested with a He mass spectrometry leak detector. Leak rate was found to be less than $10^{-10}$ torr-litre/sec. The entrance and exit ports have the facility to mount sapphire windows, which transmits electromagnetic radiation down to 1500 Å. After optical alignment with synchrotron beam, the absorption cell has been coupled to the high resolution beamline for carrying out the absorption studies. It is found that the beamline alignment is not disturbed after integrating the gas absorption cell. Loading/unloading time of the cell in the beamline is also minimized considerably so that beam time can be used more effectively. Fig. 3 shows the photograph of the gas absorption cell coupled to the beamline.

**4. Vacuum ultraviolet spectrometer**

Design and development of high resolution VUV spectrometer has been published in reference [1-2]. We will review here briefly the design details, signal detection of the spectrometer. A schematic of the optical arrangement of the spectrometer is shown in Fig. 4. The spectrometer is based on off-plane eagle mounting utilizing a concave spherical grating of radius of curvature : 6.65 m, diameter: 180 mm, ruled area : 125 mm x 110 mm (groove length), Ruling : 4800 lines/mm, Blaze angle : 13° 52', blaze wavelength: 1000 Å. The spectrometer covers a wavelength range of 700 Å to 2000 Å. The reciprocal linear dispersion of the grating is about 0.3 Å/mm. The distance between the entrance slit and the concave grating for zero wavelength setting is 6.65 m. The distance between the entrance and exit slits is 16.8 cm in the horizontal plane. The grating is rotated about the horizontal direction for recording various wavelengths on the vertical plane. The maximum rotation of the grating is 45 °. The grating is translated linearly by a maximum distance of 84 cm for focusing the spectral lines of various wavelength regions 700 Å to 2000 Å along the Rowland cylinder in vertical plane. The spectral lines are recorded by moving the exit slit and photomultiplier (PMT) coupled together along the Rowland cylinder. The maximum displacement of the exit slit and photomultiplier tube assembly along the vertical focal plane is 160 mm giving a scanning bandwidth of 48 Å. The output of the photomultiplier tube is fed to a pre-amplifier coupled to a PC based data acquisition system (DAS). The DAS consists of a precision multifunction analogue to digital converter system based on National Instruments NI-PCI-6013 card mounted in the PC which records the output through a software program based on visual basic-6 graphic interface (GUI) platform. The intensity of radiation at the exit slit is converted to PMT current, which is amplified and converted to voltage and subsequently to a 16 bit digital signal. The intensity profile of a spectral line is recorded in the form of variation of digital signal with PMT displacement.

**5. Beamline evacuation procedure**

The complete mechanical layout of the high resolution beamline consisting of front end, focusing system having three mirror chambers, absorption cell and VUV spectrometer is shown in Fig. 5. The synchrotron radiation enters from the left hand side in the figure. The front end consists of two all metal pneumatic gate valves and a fast closing valve connected by pipes and sputter ion pumps (SIP). Front end, mirror camber $M_1$, mirror camber $M_2$, mirror camber $M_3$, absorption cell and spectrometer are connected through beam pipes. The beam pipes are assembled to the mirror chamber through bellows to facilitate proper alignment of the optical components to allow the synchrotron beam along the optical axis of the system. Four numbers of gate valves are connected to isolate the mirror chambers from the beam pipe. All the mechanical components used for assembly of the beamlines are UHV compatible and use CF flanges. Several ports are provided for connecting the beam pipes to the pumps and vacuum measuring equipments. The vacuum requirement in the beamline is $10^{-9}$ mbar. To achieve this vacuum, the vacuum components are thoroughly cleaned chemically as well as ultrasonically before assembling into the beamline. To create fore vacuum of the order of $10^{-6}$ mbar in the beamline, turbomolecular pumps are used. The SIP are used to create ultra high vacuum of $10^{-9}$ mbar. Since the volume of the beamline to be evacuated is large, three turbomolecular pumping stations of capacity 500 litres/sec are connected to the three chambers through 35 CF all metal right angled values. A helium mass leak detector is used (180TD+, Alcatel make) for checking the leak tightness in the beamline. A leak rate of $5 \times 10^{-11}$ cc atm/sec is obtained with a fore vacuum of $10^{-4}$ mbar in the beamline. The beamline components are then degassed at a temperature of $100^{\circ}$ C- $150^{\circ}$ C. The baking of the beamline is done by wrapping heating tapes around the beam pipes and mirror chambers. The heating tapes are connected through temperature controllers set to the desired temperature. Utmost care is taken to safe guard the delicate components like cylindrical mirrors, feed-throughs, bellows and knife edges of the flanges. The beamline system is degassed for 8-12 hours, then allowed to cool and then the SIP are triggered for obtaining the ultimate vacuum. There are in all five SIP connected in the current beamline. A 140 litres/sec capacity SIP on mirror chamber $M_2$, and 270 litres/sec capacity on mirror chambers $M_1$ and $M_3$, on beam pipes between $M_1$ - $M_2$, and $M_2$ - $M_3$. Once the SIP start evacuating the beamline, the turbomolecular pumps are isolated through the angle value and switched off. The whole procedure to create vacuum in the beamline to an order of $3 \times 10^{-9}$ mbar takes 3 to 4 days. For initial monitoring of the vacuum, the current in the SIP power supply is used. A current of $<20$ $\mu$A corresponds to a vacuum of $< 10^{-9}$ mbar in the vacuum chamber. This provides only an approximate value as quite often the power supplies are not accurately calibrated for pressure measurement. Nude ion gauge heads and gauges (of Granville Phillipe and Varian make) meant for pressure monitoring in the ultrahigh vacuum range have been procured for accurate pressure measurements. This will be incorporated in the system soon. A Residual Gas Analyzer (of Pfeiffer make) will be assembled in the vacuum system for evaluating the quality of the vacuum by measuring the partial pressures inside the beamline.

### 6. Recording the Absorption Spectra in Ultraviolet Wavelength Region

For recording the spectrum in ultraviolet wavelength region, the grating of frequency 1200 grooves/mm was used and the experiment was carried out in air. The carbon disulphide sample was introduced into the evacuated absorption cell through a fine control needle valve. The pressure inside the cell was maintained at 5 mbar. The $CS_2$ spectrum was recorded in the wavelength range of 3120 Å - 3270 Å with and without sample inside the cell by focusing the synchrotron radiation to the entrance slit of the spectrometer. The exit slit-photomultiplier assembly was moved along the focal plane to record the spectrum. By applying the Beer-Lambert law, the absorption spectrum of $CS_2$ is generated from the intensity profiles recorded with and without the sample. Fig. 6 shows the 3100 Å band of $CS_2$ molecule.

### 7. Recording the Absorption Spectra in Vacuum Ultraviolet Wavelength Region

For recording the absorption spectrum of ammonia in the vacuum ultraviolet wavelength region, we have used a grating of frequency 4800 grooves/mm. The spectrometer was evacuated to a pressure of $5\times10^{-4}$ mbar. Two sapphire windowed gate valves (flange size 65 KF and window size 43 mm dia) were assembled to the absorption cell to extend the wavelength region up to 1500 Å. The procedure for recording the spectrum is similar to the one explained earlier. Since the absorption coefficient for these molecules is large in the VUV region, the pressure of sample admitted to the cell was of the order of $1\times10^{-3}$ mbar or less. Capacitance gauges and gauge heads capable of measuring the pressures in this range ($1\times10^{-1}$-$1\times10^{-3}$ mbar) are connected to the absorption cell. Fig. 7 shows the 1881 Å absorption band of ammonia recorded in the spectrometer. Fig. 8 shows the absorption spectrum of oxygen in the wavelength range of 1730 Å to 1770 Å recorded in the spectrometer. Fig. 9 shows the photograph of the high resolution VUV spectrometer.

## 8. Conclusion

The design details of high resolution VUV beamline at Indus-1 synchrotron source, Centre for Advanced Technology, Indore have been discussed in this report. The performance of the beamline with respect to the photo-absorption spectral studies of atoms and molecules in the VUV region has been demonstrated successfully by recording the spectra of carbon disulphide, oxygen and ammonia at the central wavelength of 3100 Å, 1750 Å and 1881 Å respectively

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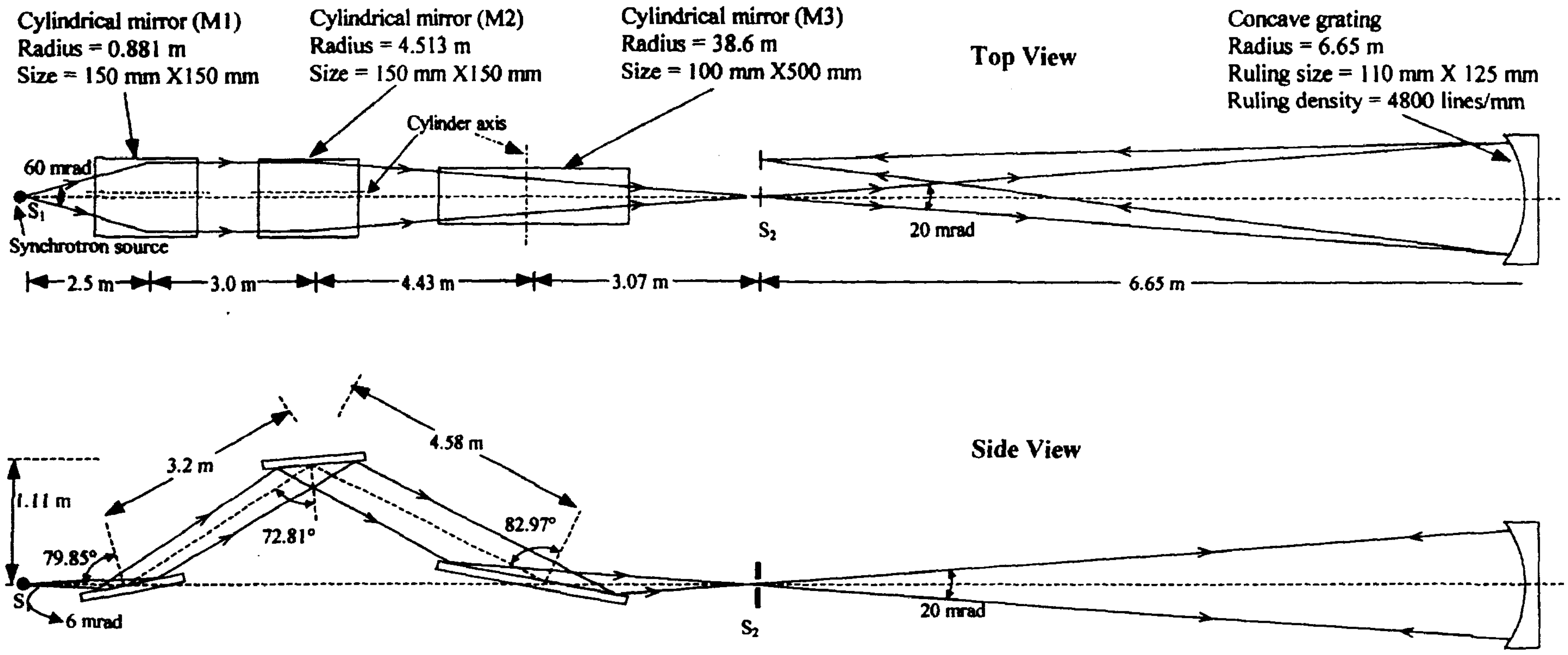


Fig. 1. Schematic optical diagram of the high resolution VUV beamline at INDUS-1 synchrotron source showing the various geometrical parameters.

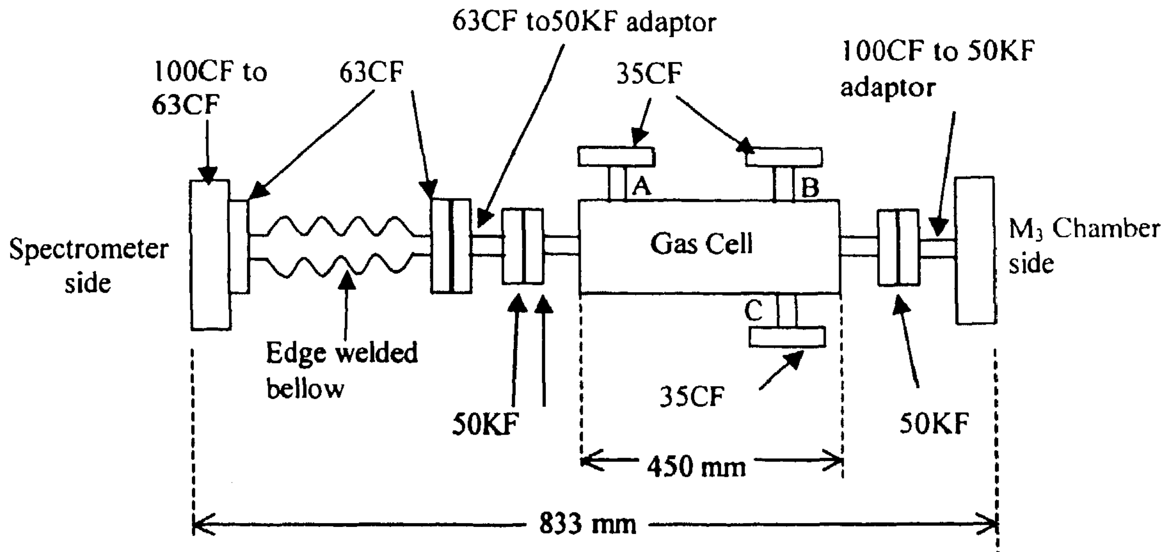


Fig. 2. Schematic diagram showing the mechanical arrangement of the gas absorption cell.

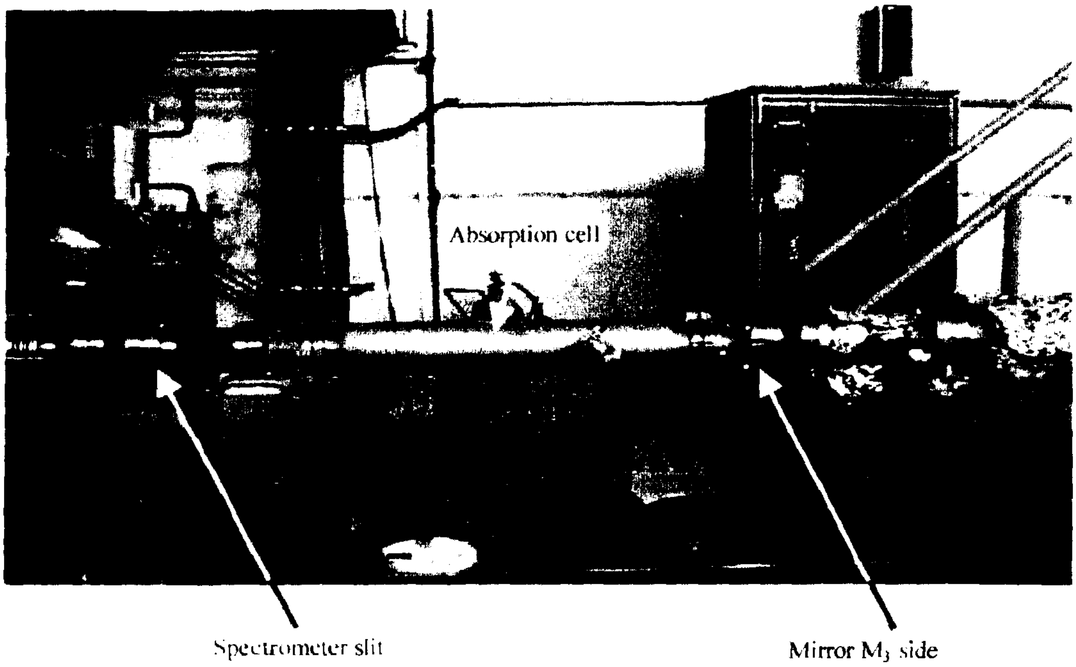


Fig. 3. Photograph of the absorption cell for filling the gas under study. Absorption spectrum is recorded at various pressures.

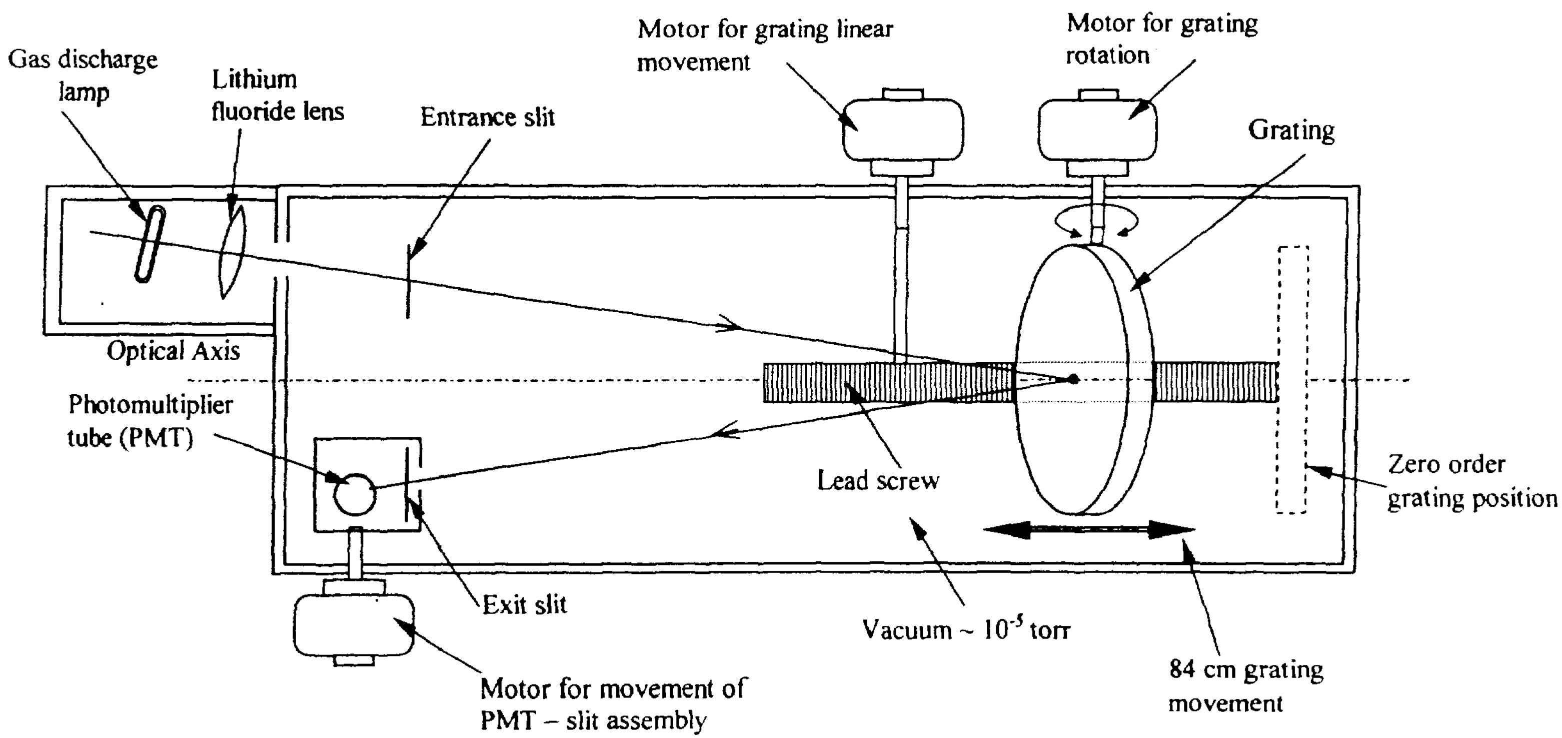


Fig. 4. Schematic diagram showing the complete assembly of the 6.65 m off-plane Eagle spectrometer

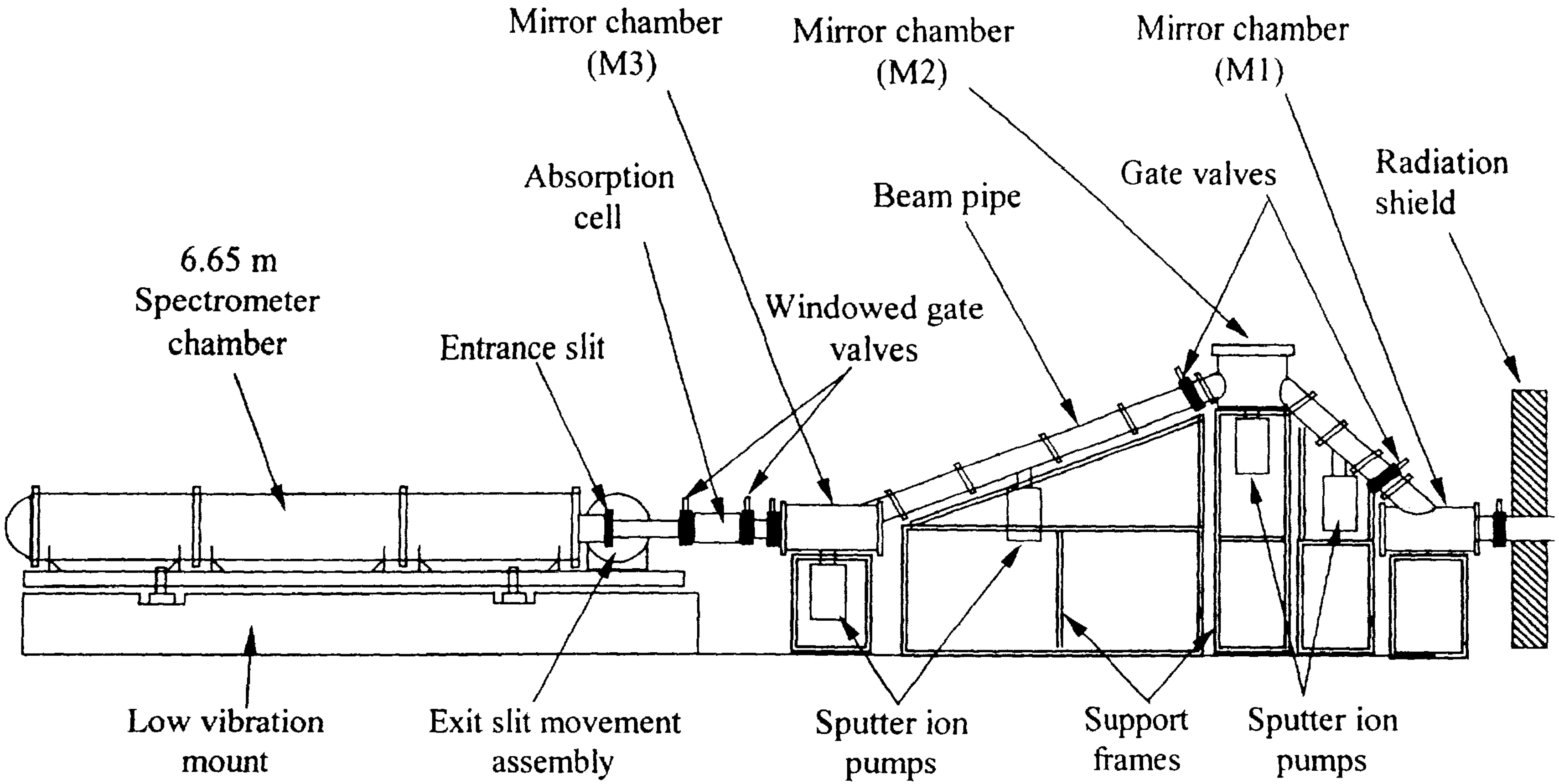


Fig. 5. Mechanical layout of the high resolution VUV beamline at INDUS-1

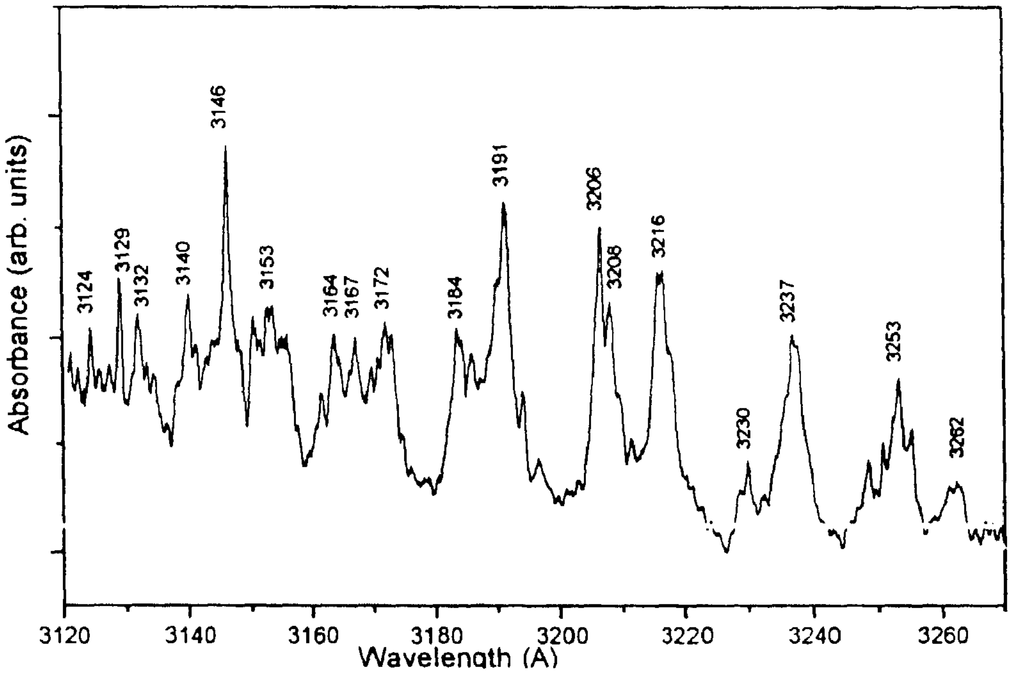


Fig. 6. Absorption spectrum of carbon disulphide recorded using Indus –1 synchrotron radiation source in the wavelength range of 3100 Å to 3300 Å.

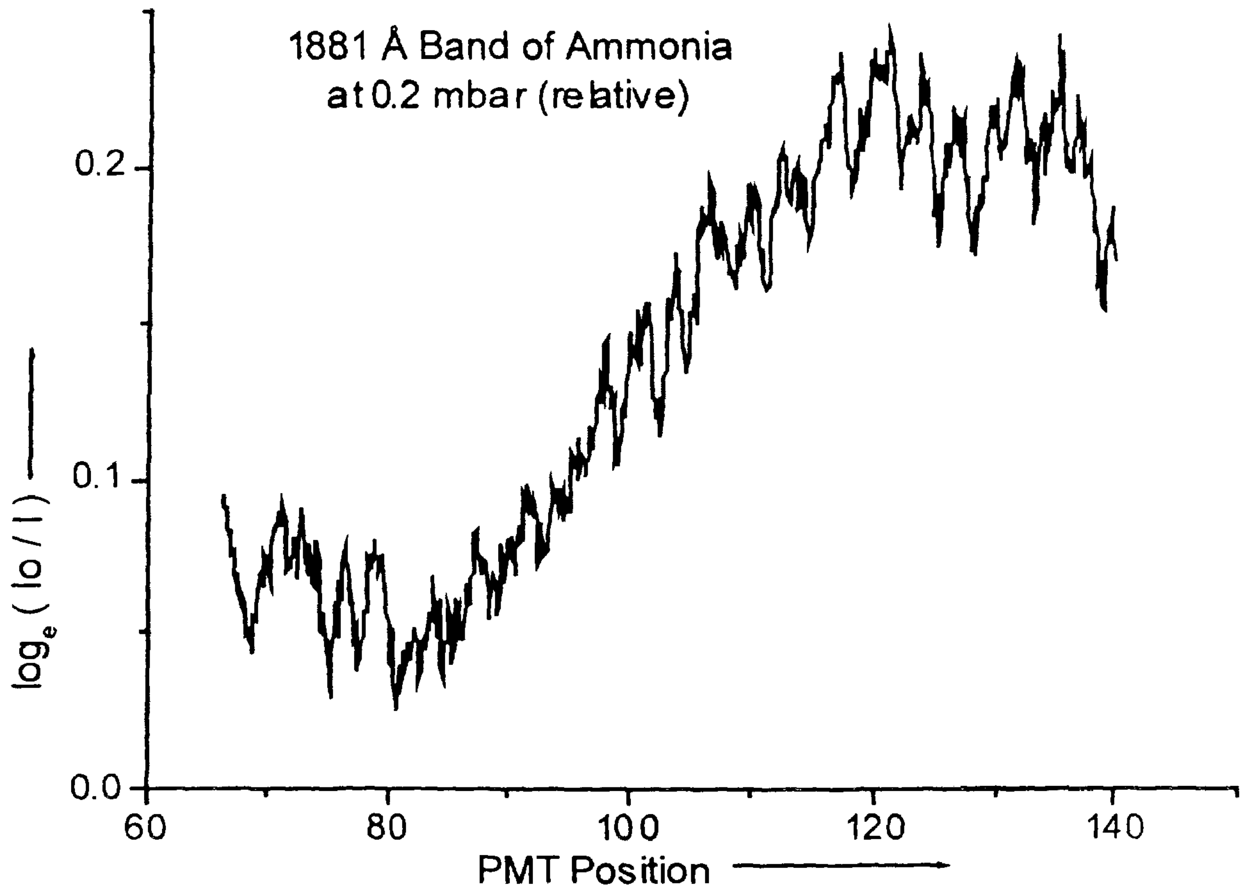


Fig. 7. Absorption spectrum of ammonia recorded using Indus – I synchrotron radiation source in the wavelength range of 1868.4 Å – 1893.6 Å.

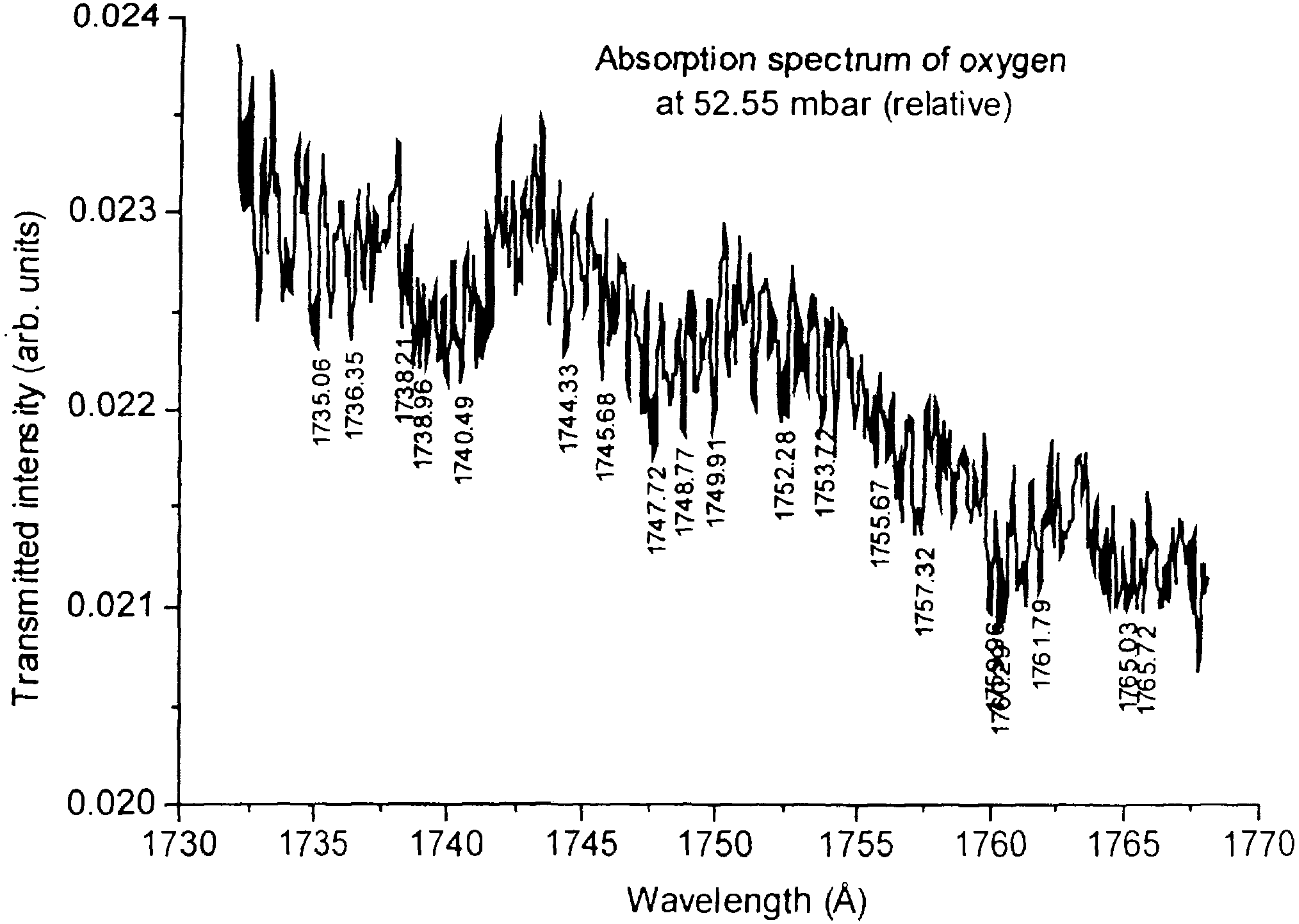


Fig. 8. Absorption spectrum of oxygen recorded using Indus –I synchrotron radiation source in the wavelength range of 1730 Å – 1770 Å.

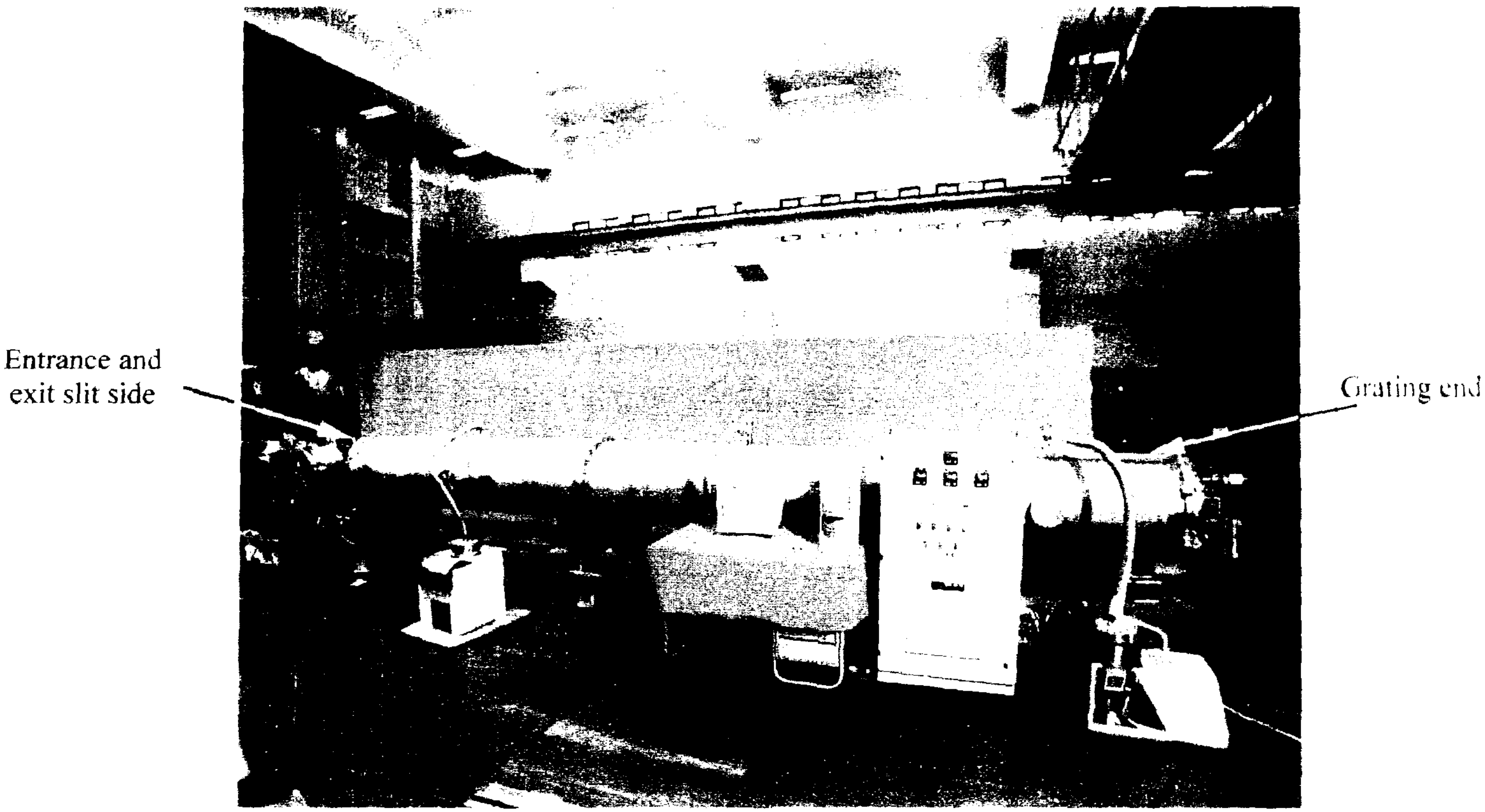


Fig.9. Photograph of the 6.65 m off – plane Eagle vacuum ultraviolet spectrometer