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A PHOTODIODE CALIBRATION FACILITY FOR THE VUV AND SOFT X-RAY REGION

by

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1. INTRODUCTION

Many experiments in atomic and molecular spectroscopy require knowledge of the photon flux incident on the gas cell or gas beam before a cross section, for example, can be measured absolutely. Standard diodes can be purchased, from the National Bureau of Standards, USA, which have been calibrated against a reference standard, but the calibration of these should be checked every two years or so, for which they must be returned to the USA. The equipment described here was developed to check diode calibration over the range of wavelengths 100-1000 Å. It proved esential for the measurements on photoionisation cross sections of atomic ions (Lyon et al, 1987), and over a part of the wavelength range above its performance was checked against an NBS calibrated diode. More general applications have been for measurements of photon flux from a monochromator, which, provided higher order contamination is below the 10% level, can be quoted with an accuracy of this order. The equipment was also designed to permit intercomparison of diodes, and found applications in spectrometer calibration for an experiment on x-ray lasers at the Rutherford Appleton Laboratory.

The wavelength range is limited at the long wavelength end by the ionisation potential of the gas being used in the ion chamber, and at the short wavelength end by the onset of multiple ionisation. Generally the rare gases are used, and the chamber is a double-plate type, described some time ago by Samson (1967), where details of the principles involved are to be found. It relies on the assumption that all ions generated in the region between its plates are collected, and also on secondary ionisation processes being unimportant. Quite sophisticated plate shapes have been developed to ensure this, but for the accuracy required here this proved unnecessary. By the inclusion of an MKS Baratron capacitance type manometer to measure the gas pressure absolutely, the photoabsorption cross section of the gas being used can also be measured, although this relies on the Baratron calibration being accurate. As will be seen later, a knowledge of the gas pressure or cross section is not required for the diode calibration measurement.

2. ION CHAMBER CONSTRUCTION

Figure 1 is a drawing showing the layout of the ion chamber, which is UHV compatible, and it can be seen that a substantial amount of it is constructed from standard vacuum hardware. The chamber (12) contains the ion collector (11) and two equal length repeller plates (23), which are of flat, polished etainless steel and are supported on three BNC feedthroughs (13) as shown, the distance between them being maintained by the ceramic insulators (10). The repeller plate (23) is electrically connected to the front baffle supported in the PTFE spacer (6); at the centre of this there is an interchangeable pinhole collimator, ~ 1 mm bore, and this assembly serves two purposes: (i) to collimate the light beam and (ii) to provide differential pumping so that a gas pressure between 10^{-3} and 2×10^{-2} millibar can be maintained in the chamber. Collimation of the light beam is essential; it must not overfill the diode photocathodes, nor must it strike the ion collector or repeller plates. It will be noticed that the light beam passes closer to the repeller plate than to the collector plates; this is done to provide the minimum practical distance for the electrons to travel, thus reducing the chance of secondary ionisation by these electrons. The collimator is held at the same potential as the repeller plate to ensure that all ions generated near the collimator are directed towards the collector plate.

The diodes for calibration, or intercomparison, are shown on the left of the diagram. The calibration diode (20) is mounted on a standard electrical feedthrough and consists of a cylindrical anode (the label (20) actually points to this), with a central circular plate as the photocathode, generally made of aluminium. The anode is held at a potential of ~ 10 V, though ia not critical over the range 10-40 V. The intercomparison diode (17), is of similar construction (the number (17) indicates the cathode in this case), but also allows an investigation to be made of the response of different areas of cathode, using the linear feedthrough (16). In addition, it is a simple matter to examine different cathode materials.

The MKS Baratron capacitance manometer (1) ia used to measure the absolute pressure in the chamber. This is not essential for the calibration measurement, but can be used to measure the photoionisation cross

section of the gas in the cell. Its reference side is held at $\sim 10^{-7}$ millibar by the main vacuum system of the monochromator or by a separate pumping system, connected by the flexible tube (5). This is also used to pump out the ion chamber itself, via the all metal valve (4), which effectively bypasses the differential aperture and collimator [7]. In use, this valve would be closed, and gas admitted through the leak valve (3), via the Nupro valve shown connected to it. Nupro valve (2) is provided so that before the ion chamber is vented to atmospheric pressure, valves (2) and (4) are closed and thus both sides of the Baratron are held at good vacuum. Although the Baratron can withstand an atmospheric differential across its inlets, it is probably wise, in the interest of keeping its calibration, not to allow this to happen.

3. METHOD OF USE

The ion chamber can be used on any soft x-ray/VUV monochromator, but it is definitely an advantage, though not essential, if the zero order beam from the monochromator can be seen, containing the visible part of the spectrum. This implies a window valve on the exit arm of the monochromator, the beam being used to align the ion chamber by eye, at atmospheric pressure. The incident beam is centred on the collimator (7), and viewed at the exit end by removing the photodiode (20), and withdrawing the cathode of photodiode (17). The ion chamber is then moved around until the beam emerges through the centre of the exit flange, and it can also be ascertained that it is not reflecting off the repeller plate (23). The ion chamber must therefore be mounted on a platform whose height is adjustable, and will need to be joined to the monochromator by a short bellows section. This was done on both the Seya and 5-metre McPherson monochromators on line 3, where in each case the exit beam emerged from the end of a 2 mm bore capillary light guide. The distance between the end of the light guide and the collimator (7), ~ 15 cms, was adjusted further to ensure that the photodiodes were not overfilled. The assembly has to be fixed fairly rigidly to ensure that the vacuum forces acting on the bellows, particularly the flexible tube (5), do not pull it out of alignment. The system is then pumped out with valves (2) and (4) open, the leak valve (3) also being open and the preceding Nupro valve closed. Should it be desired, the line from this Nupro valve to the gas cylinder regulator can also be pumped out (roughly) through the leak valve, the Nupro valve being shut off after a few minutes and the gas line leading up to it immediately pressurised with the gas intended to be used. This minimises atmospheric contamination of the working gas. When the pressure in the ion chamber has reached $\sim 10^{-6}$ millibar, it can be used. Valve 4 should be closed, valve (2) should already be open, and the leak valve should be closed, opening the Nupro valve to it as soon as it is closed. The sample gas can then be admitted using the leak valve (3), using the Baratron to monitor the pressure in the ion chamber. This should stabilise after ~ 15 minutes at most, and the monochromator base pressure should not rise onto the 10^{-8} range. Further pumping can be provided to the interspace between the collimator (9) and the end of the capillary light guide, or monochromator exit slit etc.; it should be noted that this may cause excessive "streaming" through the collimator resulting in non-constant pressure over the length of the ion chamber. The accuracy of the device depends on constant pressure of the working gas, so this excessive pumping is to be avoided if possible.

After the pressure has stabilised, a voltage between + 20 and + 40 volts can be applied to the ion repeller plate and the ion currents from plates (11) measured, using electrometers. The monochromator must first be scanned to a photon energy beyond the ionisation potential of the gas being used. The voltage on the repeller plate is then varied to ensure that a "plateau region" is reached for the two ion currents, which are generally in the range 10^{-10}–10^{-12} A, depending on gas pressure and light intensity. Excessive voltage will cause the photoelectrons to have sufficient excess energy to cause significant further ionisation, thus invalidating the measurement; insufficient voltage will cause fewer than 100% of the ions generated to reach the collector plates.

Having measured the two ion currents, the photodiode currents have to be measured. This can not be done with gas in the chamber, because ionisation of gas in the region of the diode will give a false reading. Thus it must be pumped out before taking the photodiode readings. In practice, it was found satisfactory to take a series of ion current readings for various photon energies, noting the SRS beam current for each reading. The chamber was then evacuated and a series of diode readings taken for the same photon

energies, again noting the SRS beam current for each reading. In this way a correction could be made for different light intensities; however it did assume a linear response for the SRS beam current monitor, and also a genuine zero at zero beam current, within a few per cent. Thus SRS beams of ~ 10 hours lifetime were required to reduce any errors, caused by the above assumptions, well below the level of other measurement errors.

4. INTERPRETATION OF RESULTS

A full analysis of ion chambers is given in Samson (1967), and for the present purposes, can be summarised as follows:

If the ion currents are i_1(A) and i_2(A), and the lengths of the repeller plates are d(cms), then

$$I_1 = I_0 \, e^{-\mu d}, \quad I_2 = I_1 \, e^{-\mu d} \tag{1}$$

and $i_1 = (I_0 - I_1)e_c, \quad i_2 = (I_1 - I_2)e_c$ $\tag{2}$

where I_0 is the incident photon intensity, I_1 the photon intensity leaving the end of the first collector plate and I_2 is the intensity leaving the end of the second collector plate, all in photons/sec. μ is the attenuation factor at the prevailing pressure and temperature, but need not be known when calculating I_0; e_c is the electronic charge. These equations assume the following:

(i) The photoionisation yield is unity, i.e. each photon absorbed produces an ion; in this spectral region, where other scattering processes are negligible, this is reasonable.

(ii) The Lambert-Beer law for absorption can be applied. This law is true for optically thin samples, as is the case for the gas pressures being used here.

(iii) All ions generated are collected; this depends on the repeller voltage used and on finding a plateau for the ion currents.

(iv) Single ionisation is the major process; this means that the right gas must be chosen for the wavelength range of interest. Thus helium is appropriate down to ~ 100 Å, since its double ionisation cross section is still low compared to the single ionisation cross section. However, since its single ionisation threshold is at 504 Å, it cannot be used for wavelengths longer than this.

In general, regions of autoionising structure should be avoided, where the cross section will go through rapid variations. Argon and xenon can however be used, at carefully chosen wavelengths, to cover the region up to ~ 1000 Å.

Rearranging equations (1) and (2):

$$I_0 - I_1 = I_0(1 - e^{-\mu d}) = i_1/e_c$$

$$\therefore I_0 = \frac{i_1/e_c}{1 - e^{-\mu d}} \tag{3}$$

Similarly $$I_1 = \frac{i_2/e_c}{1 - e^{-\mu d}} \tag{4}$$

So $$I_1/I_0 = i_2/i_1 = e^{-\mu d} \text{ (from (1))}$$

Thus, using (3) $$I_0 = \frac{i_1/e_c}{1 - i_2/i_1} = \frac{i_1^2/e_c}{i_1 - i_2}$$

Thus a knowledge of the charge on the electron, and the two ion currents, is all that is needed to determine the absolute value of the photon flux. Clearly leakage currents must be accounted for, and are reduced to a minimum, by choosing high quality feed-throughs and keeping the ion chamber scrupulously clean. Knowing the photon flux, and the photodiode current for that flux, corrected for any change in SRS beam current between the two measurements, it is a simple matter to calculate the diode efficiency, defined as the number of electrons emitted/incident photon, and usually expressed as a percentage.

In addition, since $i_2/i_1 = e^{-\mu d}$, the cross section of the gas in the ion chamber can also be measured; μ, the attenuation factor is given by

$$\mu = \frac{\sigma \; T_0 \; P \; N}{P_0 \; T}$$

where T_0 and P_0 are standard temperature and pressure, N is Loschmidt's number (number of atoms/cc), P and T are the prevailing pressure and temperature in the ion chamber, and σ is the cross section in cm^2, assuming d is measured in centimetres. Measurement of P is provided by the Baratron. The values so obtained can be compared to standard data, if the rare gases have been used (Marr and West, 1976; West and Morton, 1978), and this is a useful check on correct operation of the ion chamber. The most likely source of error is end effects, particularly near the end of the rear collector plate. To overcome this grounded guard plates could have been fitted, but, as the next section will show, the results indicated that this was not a serious problem.

5.   CASE STUDY

A series of measurements were taken recently to calibrate the diode used in the Newcastle University experiment to measure the absolute photo-ionisation cross section of atomic ions. On fig. 2 the results are shown comparing the calibration of the diode using the ion chamber, with some data taken by calibrating the diode against an NBS standard diode. The latter measurements were taken in a separate chamber which allowed first one diode, then the other, to see the photon beam. All the data were taken on the Seya monochromator, on beam line 3, using Argon gas in the ion chamber. The agreement between the NBS diode "intercalibration" and the ion chamber calibration is seen to be very good down to ~ 340 Å, for measurements taken at 10 μ (10^{-2} mm Hg) pressure in the ion chamber. The 5 μ measurements agree less well below 500 Å, and this is due to the lower ion currents obtained at this pressure, which in turn meant that correction for leakage currents became very important. In fact, measurements at 5 μ (5×10^3 mm Hg) pressure below 400 Å proved useless because the leakage current was of the same order as the ion current. The falling photon flux in this wavelength region from the Seya, combined with the falling cross

section of Argon, exacerbated this situation. At 10 μ pressure, measurements at 320 Å were calculated to have the error shown, and if the NBS diode is to be believed even this is optimistic. By 380 Å, the light levels were such that the error bars could be reduced to the size shown for the 10 μ point at this wavelength. The fluctuations in the 400-500 Å region may be caused by the presence of window resonances in this spectral region in Argon, except for the point at 480 Å, which cannot be explained in this way. However, the general agreement between the 10 μ data and the NBS diode is encouraging.

The efficiency curve for an aluminium photodiode is not structureless. Saloman (1980) has reviewed the behaviour of such diodes, and a continuous wavelength scan with the Newcastle diode did reproduce some of this structure. At high energies particularly, however, the Newcastle diode had a much poorer response than the NBS diode, and this was borne out by the ion chamber measurements. The difference in surface finish between the two diodes was probably responsible for this; the NBS diode had an evaporated photocathode, whereas the Newcastle diode had a rough machined one which presumably hindered the escape of photoelectrons produced by more penetrating radiation.

One further problem to guard against, but which did not affect the present measurements, is the presence of higher order radiation and scattered light in the monochromator output. Below 700 Å, the output of the Seya is not expected to contain much second order; also, both the gas cross section and photodiode response are falling and thus the effect of higher order radiation would be diminished with respect to the first order response. At longer wavelengths this would not be the case, and the higher order content would have to be investigated. One way in which this problem could be recognised is through the measurement of the gas cross section, and its correspondence, or otherwise, with published data.

6. CONCLUSION

A double ion chamber has been constructed, for the purpose of photo-diode calibration, and shown to produce reliable data in the 350-700 Å wavelength region when compared with an NBS calibrated photodiode. Since its performance at shorter wavelengths was limited by the light flux from the monochromator, and at longer wavelengths by the gas being used, in general it should be useable over the wavelength range 100-1000 Å, within the limitations discussed in the text.

REFERENCES

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FIGURE CAPTIONS

Fig. 1   Drawing of the ion-chamber, detailing the major components.

Fig. 2   Comparison of the photodiode efficiencies measured by the ion
         chamber with those referred to a calibrated NBS diode.

         ● Ion chamber measurements using 5μ of argon
         θ Ion chamber measurements using 10 μ of argon
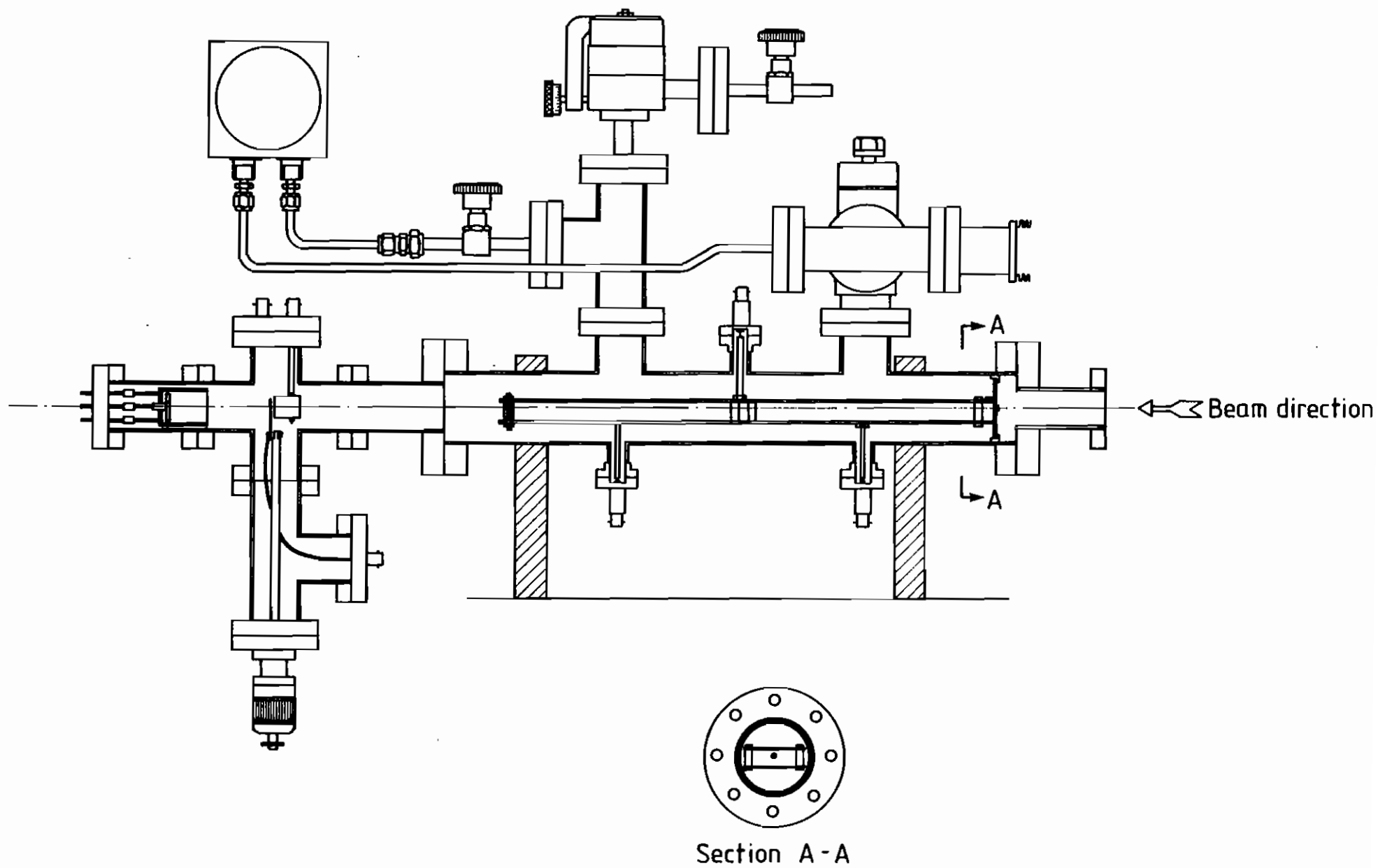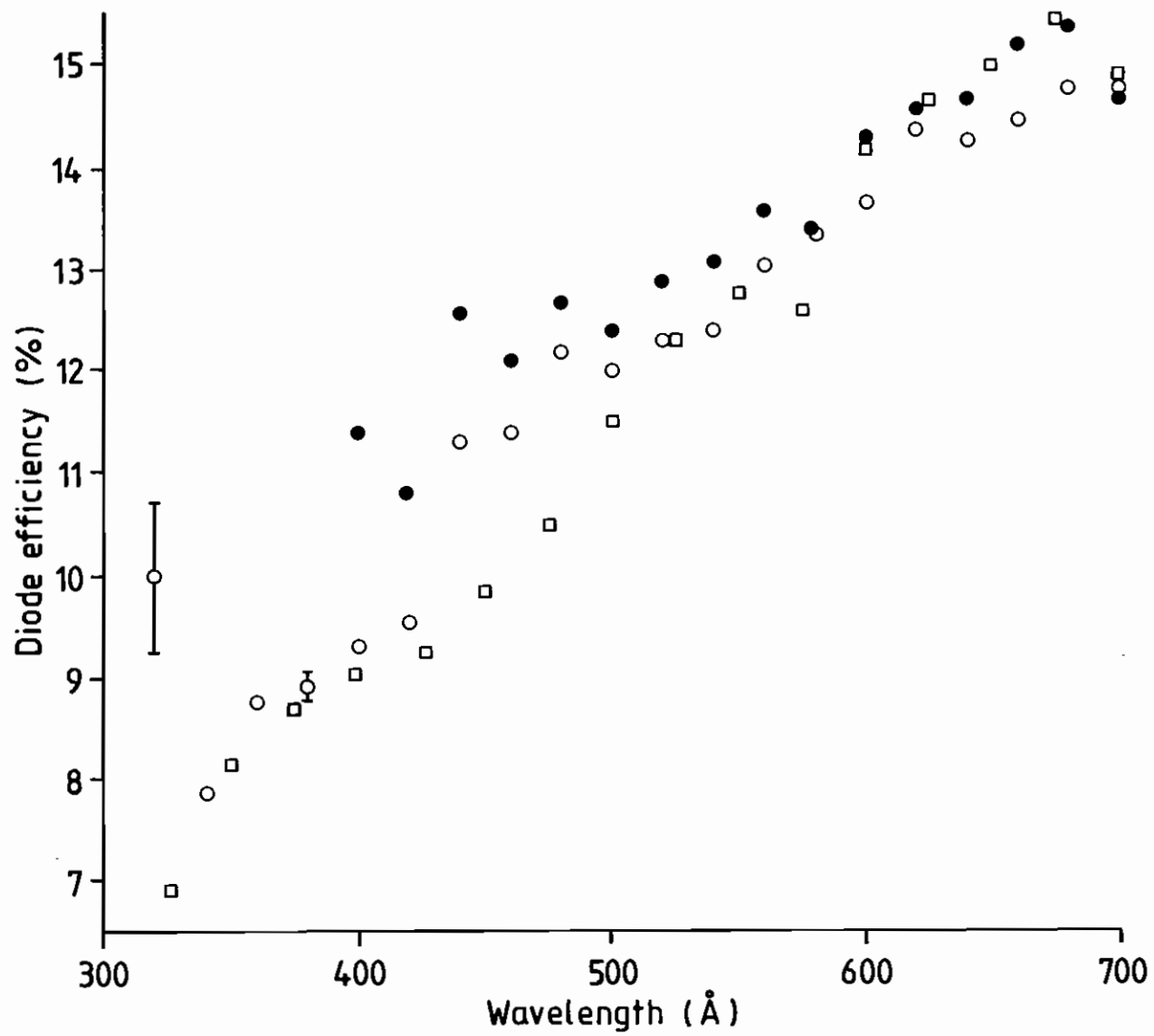   ▣   Measurements referred to NBS diode

Beam direction

Section A-A

Fig. 1

Fig. 2