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Routine ion beam analysis of impurities
collected on solid probes during plasma experiments

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probes during plasma experiments**

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Abstract

A common problem with the use of collector probes in plasma experiments is, that a sensible measurement of distributions of deposits may require the analysis of a very large number of points. For routine application this requires a fast, semiautomatic measurement and data analysis of many data points. The use of RBS and PIXE for this particular purpose is investigated.

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1. INTRODUCTION

A widely used method, both for the study of plasma-surface interactions and for the measurement of impurity fluxes in the plasma boundary layer, consists of the exposure of collector probes to plasma discharges, and the subsequent analysis of the probes by one or more surface analysis techniques /1/. The aim of such experiments varies from the determination of surface conditions on long term wall samples to the measurement of particle fluxes onto collector probes exposed to one or a few discharges only. The reader is referred to a recent review /2/ for further details.

In some cases the behaviour of a given probe material due to exposure may be part of the investigation. The analysis technique may then be uniquely determined by the material and properties in question. In the present work, however, we shall limit our interest to those experiments in which the collected material is of primary interest. Analysis technique and probe material may then be chosen accordingly.

The interpretation and value of a collector probe experiment is often more controversial than it appears from the literature: Simultaneous erosion and deposition /3-5/, unknown sticking coefficients and mass- and energy-dependent transmission of apertures /6-8/ are all effects that are frequently acknowledged in general, but due to the difficulties in evaluating their influence on the results, they are often neglected during data evaluation. The present work is not concerned with the validity hereof, although we shall show an example where it is likely that erosion has strongly distorted the results.

Instead, recognizing the fact that collector probes are indeed in routine use at present /2,5/ as well as being planned for future plasma experiments /9/, we choose to address an even more basic question: May one at least identify and quantify deposited impurity fluxes as safely as commonly assumed? It would appear from the following, that this is far from always the case. For instance, heating of the collector probe may cause deposited material to diffuse into the substrate /10/, or to collect in substantial 'islands' on the surface /11/. This is an obvious problem for very surface sensitive techniques, but may also lead to significant errors for techniques sampling larger depths (see below). The situation is further complicated by the possibility that original impurities in the substrate may segregate to the surface and appear as deposited, or influence the collection of other materials during or after exposure. Also, a multitude of different elements is usually deposited simultaneously, often leading to overlapping signals, etc. Such problems have to be taken into account when choosing analysis methods and probe materials. We shall see that for practical probe materials and typical experimental conditions confidence limits may deviate strongly from ideal values.

A particular question is the determination of oxygen fluxes. In today's plasma machines carbon and oxygen are among the most important impurities, and several groups /4, 12-29/ have been tempted to measure the oxygen deposited on collector probes. However, the effect of the probe material may have been seriously underestimated.

There is little doubt, that some of the problems encountered below may be avoided, if sufficient time, money, and effort is invested, but this is rarely done. We shall attempt to keep the discussion as generally valid as

possible, and may thus not be able to consider very specialised requirements. We shall be particularly, although not only, concerned with such experiments, where a large number of points have to be analysed within a limited time. This may occur in space resolved or time resolved measurements, where a single discharge is resolved in up to 40 datapoints /30, 27/, as well as in the analysis of stationary or long term samples, where a large surface area may have to be studied in quite small steps /31/. The use of nuclear microprobes for the fast analysis of several thousand points on a single target /32/ is a particular case, which is not specifically treated here, although much of the discussion will be relevant to this also. We are thus primarily interested in analysis techniques with which we may, within a single work-day, determine deposited impurity amounts on perhaps a hundred different points on our probes, without excessive use of man-power. The choice is here naturally limited.

As argued by Zuhr et al. /2/, the best quantitative analysis of heavy impurities is usually obtained using Rutherford Backscattering Spectrometry (RBS) and Particle Induced X-ray Emission (PIXE). Auger Electron Spectroscopy (AES) and Secondary Ion Mass Spectroscopy (SIMS) are the two other most commonly used techniques. They both have their advantages, but lack the quantitative precision of the RBS and PIXE. Furthermore, because of their extreme surface sensitivity one must often combine them with sputter depth-profiling in order to determine total impurity amounts. The two methods thus pose problems with regard to fast, automatic measurements. Similar arguments apply to the many other surface analysis techniques /2/, so that particularly for automated analysis and a semiautomatic data evaluation RBS and PIXE are usually preferred. Although the following discussion

and investigation is based on these two methods, several results should, however, have consequences for other analysis techniques too.

When investigating a great number of samples one should of course prefer to use just one analysis technique, or, if both RBS and PIXE are needed, to perform these simultaneously. The practicability of this will be discussed on the basis of practical examples. The two most popular probe materials are carbon and silicon. Of these, carbon has potential advantages for routine ion beam analysis, and a major aim of the present work is to investigate, whether carbon is also in reality the best probe material.

A flexible carbon foil (Papyex) is particularly easy to handle, but contains a number of impurities which are found to limit sensitivities severely. Carbon, however, is also available in very pure form, so the eventual advantage of such substrates, after handling and normal exposure conditions, was studied too. Results are compared to the advantages and disadvantages of silicon and other potential probe materials (e.g. Be).

Some of the present results are clearly specific to the probe materials or analysis techniques investigated, but in many cases it remains to be convincingly documented that similar problems do not occur with other materials or techniques.

2. GENERAL CONSIDERATIONS

As discussed below, an optimization of the experimental parameters involves compromises between resolution, sensitivity and speed of measurement. Of particular importance is the choice of collector material. Finally, we find it necessary to specify what is meant by 'detection limits' or "confidence limits".

2.1 Analysis techniques

i) RBS. The simplest and most common ion beam analysis technique is the Rutherford Backscattering Spectrometry (RBS), the basic principles of which are described in Ref. 33. The application to the analysis of collector probes is particularly simple:

The probe surface is irradiated with ions of mass M_1 and energy E_0 , and projectiles are detected after being backscattered through an angle θ (Fig.1). Impurity atoms of mass M_2 collected on the surface of the probe backscatter projectiles into the detector with the energy

$$E_1(0) = K_{M_2} \cdot E_0 \quad (1)$$

where /33/

$$K_{M_2} = \frac{1}{(1+M_2/M_1)^2} [\cos \theta + (M_2^2/M_1^2 - \sin^2 \theta)^{1/2}]^2 \quad (2)$$

and one may then in principle identify the mass M_2 . In reality the limited resolution of the detector makes it impossible to distinguish between two heavy impurities of almost equal mass, e.g. W and Ta. Clearly, mass resolution is improved by raising E_0 or M_1 , whenever the available accelerator allows this. However, the detector resolution deteriorates rapidly and energy straggling increases with increasing M_1 . Thus, during RBS-analysis of carbon probes for instance Cr- and Fe-signals were apparently not separated any better using 3.5 MeV nitrogen ions /34, 35/ than could have been done with 2.5 MeV ^4He -ions. Also, at energies above 1 - 2 MeV several elastic scattering cross sections (notably from carbon) deviate from those of Rutherford scattering, and an increasing amount of nuclear reactions are observed. Such effects may be advantageous /33/, but are often a complication in routine measurements.

Factors:

During plasma exposure impurity ions may be implanted or diffuse to significant depths below the probe surface, depending on ion energies, probe composition and heating effects /10/. Projectiles scattered from these impurities will lose energy on the way in and out through the target, and thus be detected at a lower energy than that of the surface signal. A significant distribution in depth will thus result in broader peaks in the energy spectrum, and perhaps a poorer mass resolution. A similar effect of course results from large amounts of some impurity (thick layer) on the surface. A particular case is the substrate signal: If the probe material has atomic mass M_s , the corresponding RBS-signal is distributed over all energies below $E_s(0) \sim \frac{K}{M_s} \cdot E_0$.

Energy:

Line:

back:

Figure 2 shows an idealized RBS-spectrum, in which small amounts of two heavy impurities are found only at the surface, while a medium mass element is distributed up to depths of perhaps 1000 Å. The figure also illustrates what is a good probe material for RBS-analysis: The substrate signal should not interfere with the impurity signals, i.e. the probe material should be lighter than the impurities investigated. It may often be possible to separate an impurity signal from a superposed substrate-signal, but this is usually too complicated for semi-automatic evaluation of many spectra.

Heavier bulk impurities in the probe material will result in corresponding RBS-signals distributed towards higher energies. This is often observed as a background signal under the peaks investigated (Fig. 3), and may thus influence the detection limits as far as collected impurities are concerned.

The experimental scattering yield from a surface deposit with atomic number Z_2 is described by the Rutherford scattering cross-section

$$\left(\frac{d\sigma}{d\Omega}\right)_R = \left(\frac{Z_1 Z_2 e^2}{4 E_0}\right)^2 f(\theta) \quad (3a)$$

where

$$f(\theta) = \frac{4}{\sin^4 \theta} \frac{\{\cos \theta + \sqrt{1 - M_1^2/M_2^2} \sin^2 \theta\}^2}{\sqrt{1 - M_1^2/M_2^2} \sin^2 \theta} \quad (3b)$$

Z_1 is the atomic number of the projectile, and e is the electronic charge. This cross-section is, however, only valid at medium energies. At low E_0 the cross-section must be corrected for screening /36/, whereas at high E_0 nuclear reactions and enhanced cross sections are observed /33/.

It follows immediately from Eq. 3a, that RBS is most sensitive to heavy elements. The use of a low-Z substrate thus also minimizes the background due to pile-up (see also Sect. 2.3).

The sensitivity is improved by lowering E_0 (see Eq. 3a), but this on the other hand reduces the mass resolution (see Eq. 1). Instead, the use of heavier projectiles seems more promising /34, 35/. However, for many accelerators the available light-ion intensities are sufficiently much larger to compensate for the smaller scattering cross section.

ii) PIXE. Another ion beam analysis technique, which is extensively used for routine analysis of impurities on environmental 'collector probes' (aerosol samples, etc. /37-39/), is the measurement of Particle Induced X-ray Emission (PIXE). For a description and discussion of the basic principles the reader is referred to Ref. 40.

For the analysis of plasma probes the technique offers potential advantages over RBS, but it has not yet been used so often for this purpose /11,30,31,41,42/:

As in RBS the probe is irradiated with MeV light ions, but instead of scattered projectiles we detect the characteristic X-rays from target atoms ionized by the incident beam. A typical spectrum is shown in Fig. 4. It consists primarily of a number of peaks corresponding to the K_α and K_β X-ray lines of the target elements. For the heavy elements also the L and M lines may be observed. The peaks are superposed upon a continuous background, mainly due to bremsstrahlung in the substrate.

Whereas the power of RBS lies mainly in its absolutely quantitative nature, a distinct advantage of PIXE is the capability of distinguishing between elements, also heavy neighbouring elements. As the individual peak-energy is independent of the depth of origin, this resolution is also not influenced by a significant distribution in depth. Unfortunately, this also constitutes a weakness of the method: The ionization cross section is generally strongly energy dependent, and thus via the projectile energy loss in the target depth-dependent. In order to extract quantitative information we therefore need to know the approximate depth from which a given signal originated. In particular, varying heat loads, and corresponding diffusion, during exposure may even distort the apparent relative distributions measured.

Experimental yields vary with the X-ray production cross-section, the detector efficiency, and the energy dependent absorption in optional absorber foils (see Section 3). The detector should therefore be calibrated to known standards.

With a normal solid-state detector (with window) we are not sensitive to elements much lighter than aluminum; hence, by using a lighter probe material the substrate signal is effectively eliminated. This is particularly important for fast measurements, because total count rates of more than a few thousands per second will result in significant dead time in the detector (see also Section 3). For the same reason, we may also want to reduce some of the strongest impurity signals, relatively to the weaker (or more interesting) ones. This may often be accomplished by a suitable choice of absorber /40/.

For the data evaluation heavier bulk impurities in the probe material have a different effect from that in RBS-measurements: Only elements having X-ray lines near those of the surface deposits are of any importance. These, on the other hand, may be detrimental to the measurement (see Section 4.2).

Optimization of the primary beam energy E_0 generally constitutes a compromise between signal-to-background ratio and speed of measurement:

The bremsstrahlung is strong at low photon energies, but decreases rapidly above the maximum energy

$$T_m = \frac{4mM_1}{(M_1+m)^2} E_0 \cong \frac{4m}{M_1} E_0 \quad (4)$$

that the projectile (mass M_1) may transfer to a free electron (mass m).

Hence, T_m should be kept well below the X-ray lines of interest, if possible. Furthermore, at high energies (a few MeV) γ -radiation will be produced in the target, giving rise to a high energy tail in the spectrum due to Compton scattering in the detector. On the other hand, for the X-rays of interest the production cross sections increase significantly with energy.

iii) A potential alternative to PIXE is the Electron Induced X-ray (EIX) analysis /21/, which offers obvious advantages for the investigation of plasma probes:

The system is considerably cheaper, and better suited for in-situ analysis, i.e. transport of the probe through air may be avoided. Production, handling and focussing of keV electrons is comparatively simple, and an electron microprobe may facilitate the analysis of also strongly varying distributions.

The X-ray production cross section for electrons in the 10-100 keV range is comparable to the cross sections of interest for PIXE, but the bremsstrahlung due to the beam is 3-4 orders of magnitude larger. It is therefore generally necessary to reduce background count rate and part of the elemental signals by an appropriate combination of beam energy and absorber foils. The method thus seems less suitable for the simultaneous measurement of several elements.

2.2 Probe materials

It was argued above, that only the lightest materials are of interest for general purposes. Among these, however, only a few are sufficiently pure and easy to handle. Furthermore, unlike for environmental probes /37-39/ plasma exposure requires materials with sufficient heat resistance, usually ruling out otherwise practical materials such as plastics or aluminium. It should be noted, though, that a volatile collector material may sometimes be preferred in order to ensure that a probe was only exposed to negligible heat loads /25/. If one wishes to collect hydrogen isotopes as well, several candidate materials, notably Al, are excluded because of

insufficient hydrogen retention. We are then essentially left with the choice between silicon, beryllium and carbon. Clearly for alternative surface analysis methods, such as AES /25,27,28/, the material choice may be less restricted.

Silicon is available to a very high degree of purity. Although machining it is rather difficult, measurements have indeed been made using Si single crystals and ion channeling to determine the plasma induced damage profiles /43/, but the channeling technique is not practical for fast, automatic measurements. Si yields a quite large PIXE-signal, which however can be largely reduced by means of appropriate absorbers (see Sect. 3). More seriously, without the use of channeling the material gives more than a factor of 5 higher substrate signal and considerably more pulse pile-up in RBS-measurements (sect. 2.3) than does pure carbon. Nevertheless, a consequence of the present work may be that Si remains as the least unattractive probe material.

Beryllium has the potential advantage of being the lightest candidate material and furthermore traps implanted hydrogen very well /44/. Unfortunately, the material is brittle, and even high-purity commercial grades /45/ contain up to an order of magnitude more iron than Papyex (compare Sect. 4.1). For RBS-analysis Hori et al. /29/ therefore also estimated detection limits just as large as we do for Papyex (Sect. 4.2).

At least for fast, automatic measurements carbon probes thus a priori seem the best choice - the material itself yields no PIXE-signal in a normal detector (with window), and only a very small RBS-signal, and it retains collected hydrogen very well /46/.

Carbon is available in many shapes and grades, from particularly practical foils (e.g. Papyex) to extremely pure rods, but of course there is a trade-off.

Papyex is a brand name for a commercial graphite foil, which is very handy as a probe material: It is easily bent and cut to any shape, and quite strong. From the producer it is specified to contain less than 0.5 % 'ashes', particularly ~ 100 ppm Cl and ~ 300 ppm sulphur. The density is relatively low ($\sim 1.1 \text{ g/cm}^3$), but the porosity is not large anyway. We have investigated the material further (Chapter 3 and Ref. 11), and indeed found the impurity level to be the limiting factor in many measurements.

Other graphite foils are commercially available (e.g. from Union Carbide), but they are in general not purer than Papyex. Carbon may be purified by heating (in a reactive atmosphere or vacuum), but this would make the foils brittle and thus reduce their practical advantages.

In contrast to such foils, most other carbons are quite hard and somewhat brittle, but some of them can be obtained considerably purer. For some purposes (e.g. limiter-probes) one may actually prefer a massive material, but one could also construct a time resolving probe of massive carbon (e.g. Ref. 47). The applicability of different types of graphite has been investigated elsewhere /11/.

Carbon is available with ash contents below 1 ppm from the producer, but this does not necessarily lead to correspondingly superior results as a certain minimum of handling, even after purification, cannot be avoided. In

order to gain an impression of practically achievable results, a high-purity carbon (<1 ppm 'ashes') was analysed after being handled with the maximum, practical, care, but being exposed to air without subsequent cleaning.

2.3 Detection limits - confidence limits

In the discussion of experimental techniques for surface analysis the concept of 'detection limits' is commonly used, but not always well defined. For RBS-measurements with 2 MeV ^4He -ions of surface impurities (Z_2) on a substrate (Z_s) reference /33/ states an empirical estimate of the sensitivity:

$$\min \cong \left[\frac{Z_s}{Z_2} \right]^2 \cdot 10^{14} \text{ atoms/cm}^2 \quad (5)$$

This is the minimum amount of surface impurities on a lighter substrate - $Z_2 > Z_s$ - that can be detected by 2 MeV ^4He backscattering. The equation takes pulse pile-up into account, but assumes an absolutely pure substrate, and is thus generally a lower limit only. The use of ions with $Z > Z_s$ would obviously eliminate pile-up effects due to the substrate /34, 35/, but not background signals from bulk impurities. For PIXE-measurements a general estimate is more difficult.

As mentioned above, we shall be particularly concerned with practical detection limits for routine analysis of plasma probes. These may differ significantly from optimum values for several reasons:

- 1) When analyzing many probes, we should not use more than a few minutes of measuring time pr. point, i.e. a typical dose does not exceed 100 μC . For substrates heavier than carbon we may actually have to measure slower (i.e. smaller dose) in order to avoid pulse pile-up or dead time.

- 2) A variety of impurities are already present in the 'virgin' substrates /11/. These may contribute to the general background in the spectrum (RBS), or add specifically to a signal of interest (primarily PIXE).

- 3) During exposure to a plasma the original impurities may diffuse, segregate to the surface or even evaporate from the substrate. This again will reduce or enhance signals in a subsequent measurement. As demonstrated in Section 4.3, segregation of sulphur to the surface of a carbon substrate can result in an increased RBS-signal just as well as if the sulphur had been deposited from the plasma, and the same type of effect might of course affect other impurities too.

If one could assume, that the original impurities in the substrate were unaffected by plasma and handling, one might attempt to account for these in the data analysis. However, as it is not practical, for routine purposes, to analyse the individual probes before exposure, one would then have to rely on 'typical values' for the substrate. One aim of the present work is the determination of such values. Unfortunately, for some probe materials, notably Papyex, the impurity levels are not always reproducible. The concept of 'detection limits' is thus less relevant than that of 'confidence limits':

For a given substrate we define our confidence limit for an element as the minimum amount of material which, in a routine measurement, may safely be identified as deposited on the substrate. Due to lack of reproducibility this is not always easy to quantify (see sect. 4.2). As discussed in Section 3 a routine analysis would typically be RBS with 1 MeV ^4He -ions and a backscattering angle of 165° , or PIXE with 1.5 MeV protons and an angle of 135° . A series of measurements were performed with these techniques in order to gather experience as to possible problems and error-sources. On the basis hereof we shall attempt to estimate typical confidence limits for the detection of various elements, as well as point out potential problems.

Clearly, for particular purposes sensitivities may be enhanced by another choice of experimental parameters. Thus, one may often enhance the sensitivity a factor of 2-3 by tilting the target. The present empirical estimates may, however, help to predict the obtainable confidence limits in a routine analysis. On the basis of our experiences we shall later discuss (Section 5), whether a better set of experimental parameters could be chosen for our own routine measurements. In particular, it would of course be of interest if one could perform PIXE- and RBS-measurements simultaneously, i.e. with the same beam.

Confidence limits are given, unless otherwise specified, in units of atoms/cm², as we are interested in quantities originally deposited on, or near, the substrate surface.

3. APPARATUS AND DATA TREATMENT

The experimental set-up was designed for simple, rapid analysis of collector probes:

Ion beams from a 2.5 MeV van de Graaff accelerator are mass analyzed in a magnet before entering a relatively compact target chamber, which is always under vacuum. The targets are mounted on a simple manipulator which is moved in the vertical direction by means of an electric step motor. This facilitates the automatic change of target position. The targets may furthermore be tilted perpendicular to the plane containing incident beam and detectors by manual rotation of the manipulator. Targets are changed by moving the manipulator through a vacuum lock to a very small chamber below the target chamber. Only this small chamber is vented when changing samples limiting this whole operation to a few minutes. Backscattered projectiles are detected in a surface barrier detector of solid angle $\sim 10^{-3}$ sr placed below the incident beam at an angle of 165° . Experiments have been performed using instead an annular detector of solid angle ~ 0.1 sr around the incident beam, but for most elements this did not improve confidence limits (Section 4.2).

X-rays are detected in a Si(Li)-detector placed above the incident beam at an angle of 135° . As the PIXE-measurement is usually count rate limited, we may generally optimize sensitivity by selectively filtering out unwanted X-rays, e.g. from the substrate, and then raising the remaining count rate by increasing the beam current. A provision for placing optional filters in front of the detector is therefore useful. In particular, when Si-probes

are analyzed (see later) the bulk signal will be absolutely dominant in the spectrum, but a 10 μm Al-foil reduces the Si-signal by a factor of $\sim 10^5$ without reducing e.g. the Fe-line by more than 25 %.

Automatic measurements of a larger number of points on a probe (see e.g. Section 4.3) are controlled by a program residing on a small personal computer. This program adjusts the vertical target position after each measurement and transfers the experimental spectra from the multi-channel analyzer to a local PDP-11 computer. On this computer reside simple programs for data analysis, as well as for printing and plotting of spectra:

RBS-spectra are evaluated by simply integrating elemental signals over predefined channels, subtracting a linear background, and calculating the corresponding areal densities in the so-called 'thin film approximation' /33/. The variation of shape and position of signals with depth and amount of material (Figs. 2-3) make a more advanced, automatic data evaluation almost impossible and a visual inspection of the individual spectra very necessary. In those instances where the latter has been neglected, very large errors have often resulted!

In contrast, the advantage of PIXE-spectra is that shape and position of a given elemental peak is always the same. Accordingly, better peak search and evaluation codes are available for the automatic analysis of many spectra, also on small computers /48, 49/. It should be noted, that unlike for RBS, a good data analysis program may not only save time/work, but may also enhance the sensitivity considerably, in many cases. Although usually

less necessary, visual inspection of the individual spectra is also recommended for PIXE.

In general, the optimum beam energy for PIXE is 1-2 MeV/amu /40/, whereby protons and alphaparticles seem equally useful /50/. For the present investigations we shall use 1.5 MeV $^1\text{H}^+$ -beams as a representative case. For the RBS-measurements we use 1 MeV $^4\text{He}^+$, although it should be mentioned that 2 MeV $^4\text{He}^+$ -ions may often be preferable (see also Section 2.1).

4. RESULTS AND DISCUSSION

It was argued in Section 2.3, that practical sensitivities are often limited by impurities in the original probe material. Examples of impurity concentrations in various carbons are given in Ref. /11/. For the particularly popular graphite foils (Papyex) the typical impurity levels were further investigated. On the basis hereof confidence limits for RBS and PIXE analysis of Papyex are estimated and compared to those obtainable with a high-purity carbon substrate. The consequence of these for actual plasma measurements is investigated. Finally, it is illustrated that the original purity of the substrate is not always the limiting factor for determining impurity fluxes.

4.1 Original impurities

The 'virgin' probe materials were analyzed with RBS and PIXE in order to determine the typical impurity levels in the probe before exposure to a plasma. Whether or not these are a problem of course depends on the impurities one wishes to collect during plasma exposure (Sects. 4.2 and 4.3).

a) Papyex. A series of Papyex stripes, cut to the shape used for plasma probes /51/, were investigated. It was assured, that the stripes came from different rolls, probably produced at quite different times. A variety of elements were identified, partly near the surface, partly apparently distributed in the bulk. For most of these elements the measured quantities varied tremendously within a single stripe, sometimes over distances of only 5-10 mm. Typical results were:

The sulphur concentration in the bulk varied between 2 and 5×10^{-4} S/C-atom, in good agreement with specifications (Section 2.2), and usually remained relatively constant within a single strip. The presence of sulphur in the bulk, as well as its occasional diffusion/segregation during exposure, poses a few problems for the evaluation of both RBS- and PIXE- results (see below).

In contrast, the considerably smaller chlorine concentration ($0-1.5 \times 10^{-5}$) is usually not so much of a problem, nor are concentrations of $1.5 - 4 \times 10^{-5}$ Ca/C or $0 - 1 \times 10^{-5}$ K/C. The measurement of the stainless steel components with PIXE is limited by the occurrence of $0.5 - 2 \times 10^{-5}$ Fe/C,

$0 - 3 \times 10^{-5}$ Cr/C and $0 - 4 \times 10^{-6}$ Ni/C, which also produce a background in the RBS-spectra. In a few cases strongly varying concentrations of zinc were also found ($0.5 - 12 \times 10^{-5}$ Zn/C).

Of frequent interest in many plasma machines is the measurement of titanium. Fortunately, here bulk concentrations were always less than 2×10^{-6} Ti/C.

Finally, near the surface oxygen concentrations of the order of 10^{-3} O/C are detected, sometimes extending to depths of a few hundred nm, possibly in the form of water. Depth distributions may not be meaningful because of surface roughness, but it should be noted that a total amount of $0.5 - 2 \times 10^{-15}$ atoms/cm² is typical, whereas at some spots considerably less was found. Undoubtedly, the occurrence of oxygen depends on handling conditions (see also Sect. 4.4).

ii) High-purity C. A very pure carbon was further cleaned by the producer⁺), after machining, to a specified purity of less than 1 ppm 'ashes'. In our laboratory it was then exposed to air and handled with as much care as is practical for collector probes. Subsequent analysis revealed 10^{-6} Fe/C, as well as less than 10 ppm S, Cl and Ar (which would not show up as "ashes").

+) Ringsdorf Werke, FRG

4.2 Experimental confidence limits

Over the years a large number of elements, including such "strange" ones as Au /13/, Li /52/, Zn /18/, V /5/, and Pd /11/, has been studied with collector probes. Sometimes elements not already present are even introduced on purpose for exactly that reason /4,11,52/. Obviously, though, the stainless steel component, originating from inconel or steel wall structures, as well as carbon and oxygen are usually of primary interest. The present estimate of confidence limits is far from conclusive. In general a large number of elements, in very different quantities, will contribute to an experimental spectrum. Overlapping signals from major components (e.g. Figs. 3-4) may thus significantly reduce the sensitivity to other elements. This will have to be accounted for in the individual case. If the material partly diffuses into the bulk, or is co-deposited with larger amounts of other materials (including carbon), this may also change the sensitivity, particularly for RBS. Furthermore, it has been seen /11/ that previous heating of the substrate may significantly alter the background level, and thus the confidence limits. This implies that heating of the probe during plasma exposure might also influence the measurements. Indeed, segregation of bulk impurities to the surface is not easily distinguished from deposition.

For these reasons the present estimates are only indicative: If the material in question is deposited on the surface of the original substrate, without disturbing effects and without the simultaneous deposition of any other element, this is the minimum amount of material that we will confidently identify as deposited.

The scatter in the results above was often too large for a mean value to have any relevance - we want confidence in all our data. Thus only when impurity signals plus general background was reasonably reproducible, was the confidence limit taken as five times the scatter. For most cases, however, a 'worst case' estimate was made. Results are listed in Table 1 for RBS and PIXE, respectively. .

a) RBS. For the 'virgin' Papyex-strips only oxygen sometimes yields a definite peak in the RBS-spectra (Sect. 4.1). Correspondingly, we should define a 'confidence limit' of $\sim 4 \times 10^{15}$ atoms/cm², unless special measures are taken to remove the original oxygen. However, we shall see in Sect. 4.4 that this estimate is usually of doubtful value, as the measurement of oxygen faces other severe problems.

For the heavier elements the situation improves with the atomic number. As, for instance, the detection of Au on the surface cannot be obscured by lighter impurities, we may thus in the present set-up detect $\sim 10^{12}$ Au-atoms/cm². Actually, measurements with an annular detector of solid angle ~ 0.1 sr have demonstrated, that one may detect 5×10^{11} Au-atoms/cm², in good agreement with Eq. 5. For the lighter elements, however, a larger detector solid angle would not help so much: Already for Mo the limiting factor is the background signal. For the stainless steel components, and all lighter elements, a considerable background is observed.

For the high-purity carbon no definite peaks are observed in the RBS-spectra, but a decreasing background signal due to pile-up and impurities extends up to energies corresponding to backscattering from Ca.

For the sake of comparison the 'ultimate confidence limits', as estimated from Eq. 5, are also included in Table 1. Good agreement is found with the experimental values for the pure carbon, except for the light elements.

ii) PIXE. Because of the strongly varying bulk-impurity levels in Papyex the large sampling depth of PIXE can be a serious problem and for the stainless steel components, and lighter elements, we find high confidence limits.

In particular, until the sporadic occurrence of large Zn-contaminations has been satisfactorily accounted for, we may not believe in collected amounts of less than $\sim 2 \times 10^{16}$ Zn-atoms/cm².

For the heavier elements the situation is more complicated: The high-energy signals are not obscured by background, and easily identified, but the ionization cross-section is very low. The M and L lines for these elements are much easier to excite, but here the signals from the many lighter impurities also appear. For instance, the Mo-L_α line lies only ~ 15 eV from the S-K_α, which is the most dominant PIXE-signal from both Papyex and the high-purity carbon. By means of a good data analysis program one may include also the other (smaller) Mo-peaks, notably Mo-L_{β₁}, and the known ratio between them, but at least for Papyex the sensitivity is just as good when evaluating only the Mo-K_α line. In general, for the heavier elements RBS is clearly the more sensitive technique, and PIXE is here primarily of interest for element identification.

For a carbon substrate the effective sampling depth is determined by beam energy and direction, rather than absorption of X-rays. Unless the target surface is particularly rough, the sampling depth may be reduced somewhat by tilting the target, leading to an enhanced sensitivity to surface deposits without changing the yield from bulk impurities. The influence of the bulk may furthermore be strongly reduced by lowering the beam energy /10/, which also has the advantage of reducing T_m (Eq. 4). As one has often considerably more beam intensity available than can be exploited (see section 3), the resulting reduction in ionization cross section also at the surface is not always important.

It may be worthwhile noting, that a reduced sampling depth is more conveniently obtained with electron beams (EIX).

4.3 Actual plasma measurements

Below we shall consider various experiments performed with collector probes exposed in the Garching fusion device ASDEX. All of these experiments have involved routine analysis (mostly RBS) of a larger number of probes, and the results have been, or will be, published elsewhere. For the purpose of the present work selected samples were investigated both with RBS and PIXE, in order to determine the superior method in the individual case.

Experimental parameters were as described in Section 3. The experiments involved both time-resolved and stationary probes.

i) The first example illustrates the significance of our estimated confidence limits (Section 4.2):

A row of Papyex stripes were mounted /13/ on a cylinder behind 4 mm circular apertures and first exposed to the divertor plasma as stationary probes. The exposed part of a stripe is denoted as positions 15-19 mm in Fig. 5. During a later discharge the probes were rotated past the apertures, yielding a time resolved measurement /13/. This time positions 24-38 mm were exposed. Thus for instance positions 20-23 mm have not been directly exposed to the plasma.

RBS-analysis of one of the Papyex stripes yielded, among other things, the distribution of metals vs. position shown in Fig. 5a. With 1 MeV $^4\text{He}^+$ -ions one may not separate signals from e.g. Ti, Cr, Fe and Ni very well, so the sum is shown.

PIXE-analysis of the same stripe showed, that contributions from Cr and Ni were negligible compared to those of Ti and Fe. Evaluating the data under the assumption that all signals originate at the probe surface, we obtain the Fe-distribution shown in Fig. 5b. We notice large discrepancies: Not only do we find more peaks than with RBS (notably one near position 23 mm, which was supposedly not exposed at all), but in several points the apparent Fe-concentration is much larger than the sum of Fe- and Ti-concentrations determined with RBS. The additional PIXE-signal from Fe can only be explained by a significant depth distribution of this metal. However, a diffusion into the bulk of deposited Fe would be visible in the shape of the RBS-spectra. A small number of large (μm) metal droplets on the surface /35/ could have the observed effect, as PIXE sums up over large depths whereas the RBS-signal depends on the relative areal density near the surface (Section 2.1), but Scanning Electron Microscopy (SEM)

combined with X-ray analysis /53/ did not reveal any droplets on the present surface.

The question remains, whether the discrepancies can be explained by the original Fe-concentration in the Papyex, and indeed Table 1 shows that the observed PIXE-signals from iron are all below the confidence limits. In contrast, the RBS-results are clearly significant. Apparently, the values in Table 1 are by no means too pessimistic.

ii) Let us consider another example, where the PIXE-results are only useful in connection with RBS:

A series of time resolved measurements were made in the divertor of ASDEX, just like in the previous example, except this time the cylinder rotated faster /34/, thus improving time resolution. During the experiment 40 stripes of Papyex were exposed to a high-density plasma during neutral injection, the exposure of each stripe being repeated in four discharges, in order to improve the sensitivity. Despite the repeated exposure, however, the deposited amounts were not very large. Obviously, the analysis of collected amounts versus position (~ 15 points) on each stripe calls for a fast, semi-automatic procedure (section 3).

We selected one of the stripes for investigation. Figure 6 shows the detected amount versus position. In the RBS-measurements the mass resolution is certainly not optimized (section 2.1), but one can still distinguish a large number of peaks. These peaks were ascribed to Fe, Ta, Mo, O, Br, K, Cl, S, Si and Cs, but in most cases contributions from neighbouring elements may not be excluded - for the heaviest elements the

identification is even questionable. Thus, we have no way of knowing whether the "Ta-signal" is for instance caused by W instead!

Before we start to calculate deposited impurity fluxes, etc., we have to convince ourselves that we are indeed measuring deposited material. Although all the signals correspond to substantial amounts of material (at least in the maximum), we find that the signals from Cl, S and Si are clearly below the confidence limits (Table 1). The maximum in the Br-distribution barely exceeds the limit, and for O, K and Cs only the maxima are apparently significant (see broken lines in Fig. 6). On the basis of Table 1 alone, we may then ignore many of the signals.

A common argument in support of deposition has been the correlation between distributions of different elements. However, although in the present case the iron is undoubtedly deposited, the apparent correlation between the S- and the Fe-distributions is not an argument for sulphur also to originate from the plasma (Sect. 4.4). Similarly, the oxygen distribution might be due to oxidation of the carbon or enhanced water adsorption.

In Fig. 6 the K- and the Cl-distributions seem strongly correlated, and both largely independent of the other distributions, so in conclusion only the distributions of Ta (or W), Fe and Mo seem convincingly related to the plasma, and traces of Cs might be significant.

A study of Table 1 shows, that none of the observed deposits might have been safely identified as such by means of PIXE. Nevertheless, for the sake of illustration let us take a look at the results of a PIXE-measurement

(Fig. 7): In the PIXE-spectra we identify signals from Fe, Cr, Ti, Zn, K, Ca, and S, although only the Fe- and K-signals ever exceed the confidence limits (Fig.7). In agreement with these limits the RBS-measurement excluded the deposition of the other elements in these amounts. In particular, the deposition of almost 10^{15} Zn-atoms/cm² would easily have been identified with RBS.

For a detailed comparison of the remaining distributions it should be mentioned, that RBS and PIXE were made with two different, independently focussed, beams and that the target had been dismantled and remounted in between. Beamspots may thus deviate by fractions of a mm, which may be important for sharp peaks and structures. This may explain the position of the K-peak in Fig. 7, but the magnitude can only be explained by an extension to large depths. Unless this is caused by diffusion it is thus unlikely that we are observing deposited material, although the signal exceeds the confidence limits of Table 1. We may note, that a comparable Cl-distribution is not observed, in contrast to Fig. 6.

In the case of iron, the maximum is shifted slightly, probably due to the differences mentioned above. The magnitude is considerably larger, but the maximum only exceeds the 'confidence limit' by an amount comparable to the RBS-result. This further supports the values in Table 1. Actually, in view of these values the 'Fe-distribution' in Fig. 6 can essentially be explained as a combination of Fe- and Cr-signals. The fact that nickel is not observed at all, even at a level below the confidence limit, indicates that this element can not contribute more than a few percent to Fig. 6.

iii) An example of a truly stationary probe was chosen among a set of graphite-blocks which had been exposed at the divertor of ASDEX during a so-called 'L-shot' with neutral injection and disruption at the density-limit /55/. Obviously, Table 1 is not directly applicable here, although the pure-carbon values definitely must be lower limits. Anyway, as indicated above, tabulated confidence limits may sometimes be too optimistic, for instance in the case of substantial diffusion/segregation or simultaneous deposition of larger amounts of other materials. Instead of establishing alternative confidence limits with a series of 'virgin' reference samples, it was thus decided to check whether further effects could obscure resulting 'deposited impurity fluxes': The probe had been positioned normal to the magnetic field lines, so that only one side was exposed directly to the plasma. The backside was now analyzed as an 'unexposed' reference, leading to an intriguing result.

The back was found to contain of the order of a few ppm tantalum, together with the 'normal' impurities. The Ta appeared to extend to large depths, but neither RBS nor PIXE could of course distinguish whether it was distributed uniformly, or concentrated in small, thick 'islands' on the surface (see e.g. Refs. 11 and 31). There is no reason to expect Ta in the original material, and the simultaneous occurrence of considerably larger amounts on the front side of the sample suggests that it is all related to the plasma exposure. The same behaviour may well occur for some of the other impurities studied. On the back side we also found $\sim 3 \times 10^{14}$ Ti-atoms/cm², probably originating from the plasma.

If we are therefore aiming at interpreting the 'direct' deposition on the exposed front side, we should rather take our reference values from the back side than from the original substrate impurity levels. This also suggests, that for other collector probe measurements as well (e.g. above) the 'confidence limits' of Section 4.2 may be only lower limits!

Let us then turn to the front side of the sample: Figure 8 shows the detected amounts versus position, as found with RBS, and the broken lines indicate the confidence limits estimated above. We note that the detected amounts are generally significantly above the confidence limits - this is the way a collector probe measurement should look! The heaviest element (Ta) is of course not identified with RBS, but with PIXE (below), and the 'Fe'-signal contains contributions from both Cr and Ti.

However, even in the present case data interpretation is not as simple as it may seem. For one thing, the signals may interfere with each other: The Ta-signal is so large that it overlaps with any other signal and thus might obscure e.g. a smaller Fe-signal. Here the confidence limits are therefore considerably larger. This illustrates the necessity of a visual inspection of the individual spectra, even with an otherwise automatic data evaluation (see Section 3).

This is not the only problem, if we wish to determine 'deposited fluxes'. It is very well possible that the originally deposited distributions are strongly perturbed by erosion. Thus, the original distributions may all have peaked in position 56 mm, but most of them subsequently been reduced by an incident heatflux correlated to the Ta-deposition: A visual

inspection of the target shows a clear discoloration at position 56 mm. Furthermore, the bulk concentrations of Ca and S (Fig. 9) are generally lower than on the back side, indicating a significant heating of the front. The variation of these concentrations with those of deposited Fe and Cr may be due either to variations in this heating, or to the formation of more or less effective segregation barriers (Sect. 4.4). Also, it is believed that the Ta originates from melting/evaporation of nearby shields, suggesting very large heat fluxes. The Ti seems not correlated to the other deposits, but this may simply mean, that it was deposited after the strongest heating. Finally we note, that in the present case the very large oxygen amounts may be explained by oxidation of the metal deposits (Fig. 8), perhaps during the final exposure to air.

The above discussion involves a significant amount of guesswork, but demonstrates that one must be very careful in relating such results to deposition of plasma impurities. It is worthwhile noting, that although the PIXE-yields (Fig. 9) are mostly comparable to or smaller than 'confidence limits', useful information was gathered from these anyway.

4.4 Oxygen and sulphur fluxes

i) The measurement of oxygen fluxes with collector probes has been attempted quite frequently /4, 12-20, 24-26, 56/, and would indeed be of great interest /5/ if possible. However, serious difficulties are encountered:

- 1) "Sticking coefficients" for low energy oxygen ions and neutrals incident on relevant probe materials are unknown. Furthermore, the probe may become saturated during exposure /16/. Oxygen fluxes are therefore usually only reported as lower limits /4, 14, 16/.

- 2) Sputtering by the plasma may result in substantial surface roughness and thus in a strongly enlarged effective probe surface area. This again would allow for additional adsorption e.g. of water from residual gases or the air. Most probe analysis techniques do not distinguish the chemical state of the oxygen.

- 3) The collector probe is often exposed to air before analysis. Even when this is not the case, the strongly increasing residual gas pressure at the end of a plasma discharge may, particularly in machines with leaks, lead to an oxidation of metal deposits. It is often argued, that an oxygen-to-metal ratio of 4 or more excludes a significant contribution from metallic oxides /12, 15/. However, if sufficient amounts of carbon and various other elements are available considerably larger ratios may be realized in chemical compounds such as metal carbonyls or more complicated mixed species /57/.

- 4) The surface of a carbon probe might also itself oxidize under the catalytic influence of Fe or another metal /58/, or because it has been activated by irradiation during plasma exposure ("dangling bonds"). This, of course, is only important if the oxide remains somehow bound to the substrate.

The effect of irradiation and subsequent air exposure was briefly tested: A graphite sample was irradiated with 1 keV deuterons on 3 different spots to various fluences of the order of 10^{18} cm^{-2} . The sample was then exposed to air before RBS-analysis. Figure 10 shows the resulting oxygen coverage as a function of target position. The implanted spots, indicated in the Figure, each had a diameter of 1.5 mm. The horizontal "error" bars represent the size of the analysis beam. Outside of the irradiated region we find no more than $\sim 2 \times 10^{15} \text{ O-atoms/cm}^2$, in good agreement with the findings of Section 4.1. In contrast, irradiation led to an additional surface coverage of $\sim 10^{16} \text{ cm}^{-2}$, a number not significantly smaller than the maximum of typical oxygen distributions found on collector probes /12-20, 22, 23, 26-28/.

It should be stressed, that the above problems are not specific for a particular analysis technique. Obviously, other collector materials may behave differently. However, as it is known that wall surfaces activated during a plasma discharge subsequently pump oxygen very efficiently from the residual gases, even during deliberate oxygen inlet /59/, metal probes /25, 26/ are not expected to be any more reliable for the determination of oxygen fluxes.

ii) The deposition of other reactive species such as chlorine or sulphur /20-22, 28, 30, 56, 60/ is probably not easier to measure: For instance, sulphur is already present in the bulk of many probe materials. In particular, most graphites contain significant S- and Cl-concentrations (see also Sect. 4.1). As expected, heating of an uncovered carbon probe may lead to a significant desorption of both. However, a layer of Fe evaporated onto the surface seems to form a segregation barrier (perhaps an Fe-S

compound), so that heating results instead in S-enrichment at the surface /61/. Of course, it is also possible that metal coverage strongly influences the "sticking coefficient" for incident sulphur. It may be significant, that concentrations observed in collector probe experiments /11, 20/ are in good agreement with the assumption of a sulfide. Local heating of the probe during exposure (perhaps even correlated with metal deposition) may thus totally falsify deposition measurements. Anyway, a correlation between sulphur and metal distributions /11, 20/ is no argument for the former being determined by incident flux distributions. In particular, the measurement of sulphur fluxes with carbon probes may be inherently impossible.

5. CONCLUSIONS

In many collector probe experiments a rapid accumulation and analysis of large numbers of data is required. For this purpose only RBS, PIXE and perhaps EIX are really suitable. For all three methods we should prefer a pure low-Z probe material. Carbon here seems to be one of the most practical candidates, although for the most handy version (Papyex) sensitivities are severely limited by the heavy bulk impurities present in the 'virgin' material. The use of higher purity carbon may complicate handling of the probes considerably, but will certainly improve data evaluation. In general, there does not seem to be much gained by using Be instead. For PIXE-measurements the use of high-purity silicon may be reasonably fast when using a suitable absorber in front of the detector, but for RBS measuring times become considerably larger.

The experimental 'confidence limits' are generally higher than optimum 'detection limits', due to diffusion, segregation, original impurities in the collector material and simultaneous deposition of several elements. The effect of original impurities is included in the confidence limits for RBS and PIXE given in Table 1, but in reality we expect the limits to be even larger, in many cases. We have seen examples illustrating, that the values in Table 1 are indeed not too pessimistic, at least for Papyex. For a high-purity carbon Table 1 suggests almost 'ideal' values, but the original impurities may not be the only ones obscuring results. If deposited material is only present at the surface, and has not agglomerated into 'islands', considerably lower confidence limits are obtained with EIX using electron beams of a few keV only /62/. Unfortunately, this is often not the case.

For RBS measurements a visual inspection of the individual spectra is mandatory, for PIXE-measurements perhaps less necessary but desirable.

Even with a careful data analysis the application of PIXE alone has been seen to be at best risky, particularly when bulk impurity concentrations are substantial. Diffusion into the bulk, or agglomeration into 'islands', may significantly distort results, and even relative distributions may be wrong /20/. In contrast, the sole application of RBS is much easier controlled and understood, but obviously a combination of the two techniques would be preferable. For the sake of fast analysis, it is therefore of interest whether one can simultaneously use the same beam for both types of measurement. It is generally recommended to optimize the beam for RBS, and then simultaneously record the resulting X-rays.

Hence, the validity of fast routine collector probe measurements in plasma experiments seems questionable. Instead, we should prefer to concentrate on a single, well defined problem, e.g. temporal behaviour of a dominant impurity deposition, and optimize collector material and analysis technique accordingly. If more time and effort is invested, other surface analysis techniques are available, perhaps in combination. However, potential problems, such as the effect of a possible distribution of deposits to large depths, must still be carefully considered.

A couple of serious problems remain, almost independently of analysis technique and effort: In general, oxygen fluxes - or even lower limits - may not be measured with carbon probes, and probably also not with metal probes. It would appear, that the same is true for sulphur and perhaps chlorine fluxes. It remains to be documented, that similar problems do not occur for other collector materials. A determination of the chemical state of the measured element might here be helpful.

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Figure Captions

1. Experimental principle, RBS.
2. Idealized RBS-spectrum.
3. Exp. RBS-spectrum for 2.5 MeV $^4\text{He}^+$ -ions incident on a carbon-limiter probe.
4. Exp. PIXE-spectrum for 1.5 MeV protons incident on a carbon-limiter probe.
5. Detected areal densities versus position for Papyex stripe exposed both as stationary and time resolving collector probe.
 - a) Sum of Fe- and Ti-amounts. RBS-result
 - b) Fe-amount. PIXE-result.
6. Detected areal densities versus position for Papyex stripe exposed as time resolving collector probe. RBS-result. Broken lines denote confidence limits (out of scale where not shown).
7. PIXE-result corresponding to Fig. 6.

8. Detected areal densities versus position for graphite-block exposed as stationary collector probe. RBS-result. Broken lines denote confidence limits (too low to be visible for Ta).

9. PIXE-result corresponding to Fig. 8.

10. Areal density versus position, for oxygen on graphite sample after D-irradiation and air exposure. Positions 2.2 - 3.7, 4.2 - 5.7, and 6.2 - 7.7 mm were irradiated. Horizontal bars indicate diameter of analysis beam.

Table 1

Confidence limits for routine analysis of Papyex and a high purity carbon (after handling) with PIXE and RBS. For RBS also the empirical estimate (Eq. 5) for pure carbon.

ELEMENT	CONFIDENCE LIMITS (Atoms/cm ²)				
	PIXE		RBS		
	Papyex	Pure C	Papyex	Pure C	Eq. 5
Si			$2 \cdot 10^{15}$	$3 \cdot 10^{14}$	$2 \cdot 10^{13}$
S	$1 \cdot 10^{17}$	$5 \cdot 10^{15}$	$1 \cdot 10^{15}$	$2 \cdot 10^{14}$	$1.5 \cdot 10^{13}$
Cl	$3 \cdot 10^{15}$	$5 \cdot 10^{14}$	$5 \cdot 10^{14}$	$1 \cdot 10^{14}$	$1.2 \cdot 10^{13}$
K	$2 \cdot 10^{15}$	$5 \cdot 10^{13}$	$5 \cdot 10^{14}$	$5 \cdot 10^{13}$	$1 \cdot 10^{13}$
Ca	$1 \cdot 10^{16}$	$5 \cdot 10^{13}$	$5 \cdot 10^{14}$	$3 \cdot 10^{13}$	$9 \cdot 10^{12}$
Ti	$5 \cdot 10^{14}$	$5 \cdot 10^{13}$	$3 \cdot 10^{14}$	$2.5 \cdot 10^{13}$	$7 \cdot 10^{12}$
Cr	$5 \cdot 10^{15}$	$1 \cdot 10^{14}$	$2.5 \cdot 10^{14}$	$2 \cdot 10^{13}$	$6 \cdot 10^{12}$
Fe	$5 \cdot 10^{15}$	$5 \cdot 10^{14}$	$1.5 \cdot 10^{14}$	$1.5 \cdot 10^{13}$	$5 \cdot 10^{12}$
Ni	$1 \cdot 10^{15}$	$2 \cdot 10^{13}$	$3 \cdot 10^{13}$	$8 \cdot 10^{12}$	$4.5 \cdot 10^{12}$
Zn	$2 \cdot 10^{16}$	$5 \cdot 10^{13}$	$1 \cdot 10^{14}$	$7 \cdot 10^{12}$	$4 \cdot 10^{12}$
Br	$1 \cdot 10^{14}$	$1 \cdot 10^{14}$	$3 \cdot 10^{13}$	$5 \cdot 10^{12}$	$3 \cdot 10^{12}$
Mo	$1 \cdot 10^{15}$	$2 \cdot 10^{14}$	$1 \cdot 10^{13}$	$4 \cdot 10^{12}$	$2 \cdot 10^{12}$
Ta	$1 \cdot 10^{15}$	$5 \cdot 10^{14}$	$1.5 \cdot 10^{12}$	$1.5 \cdot 10^{12}$	$7 \cdot 10^{11}$
Au	$2 \cdot 10^{15}$	$7 \cdot 10^{14}$	$1 \cdot 10^{12}$	$1 \cdot 10^{12}$	$6 \cdot 10^{11}$

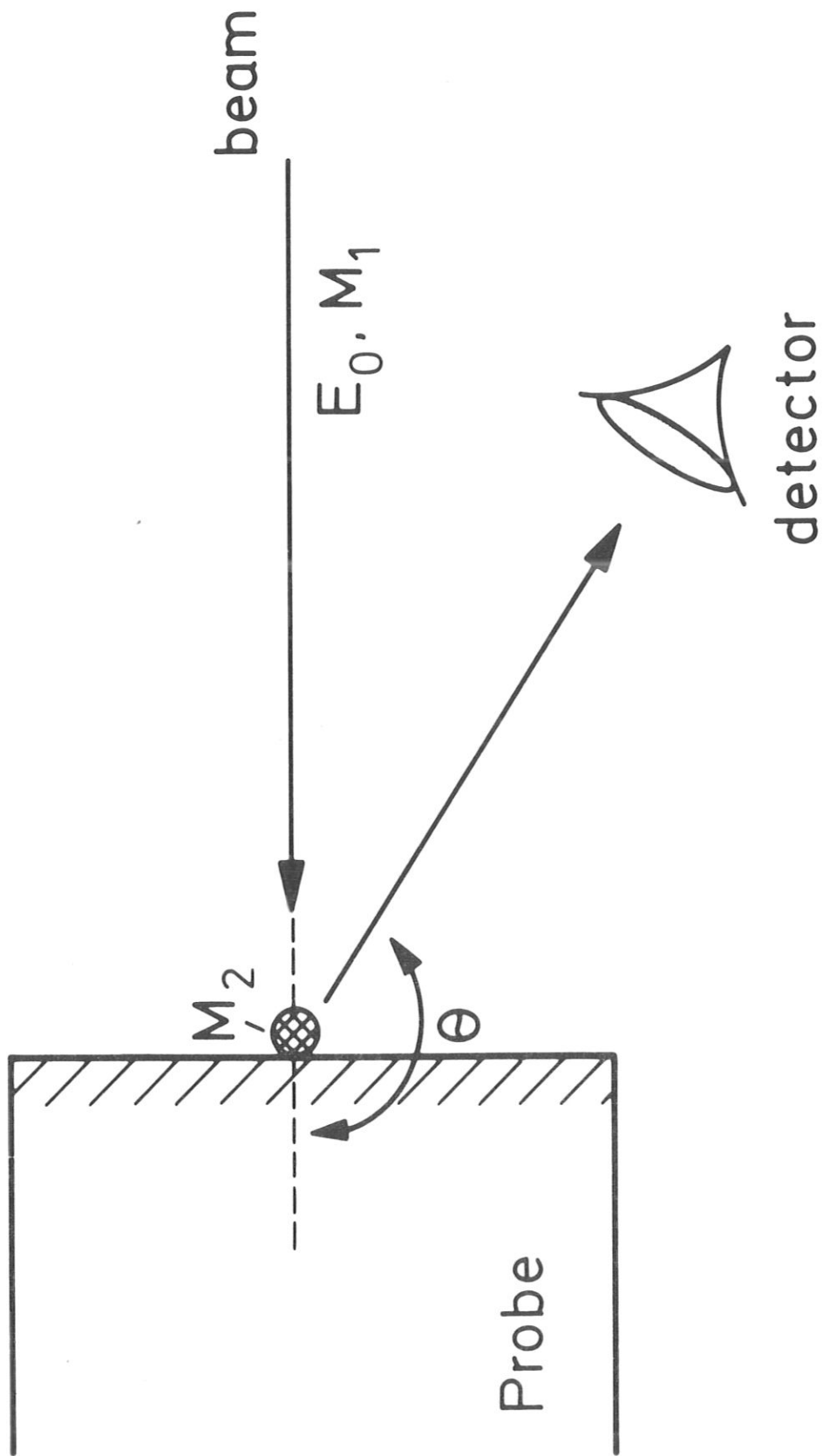
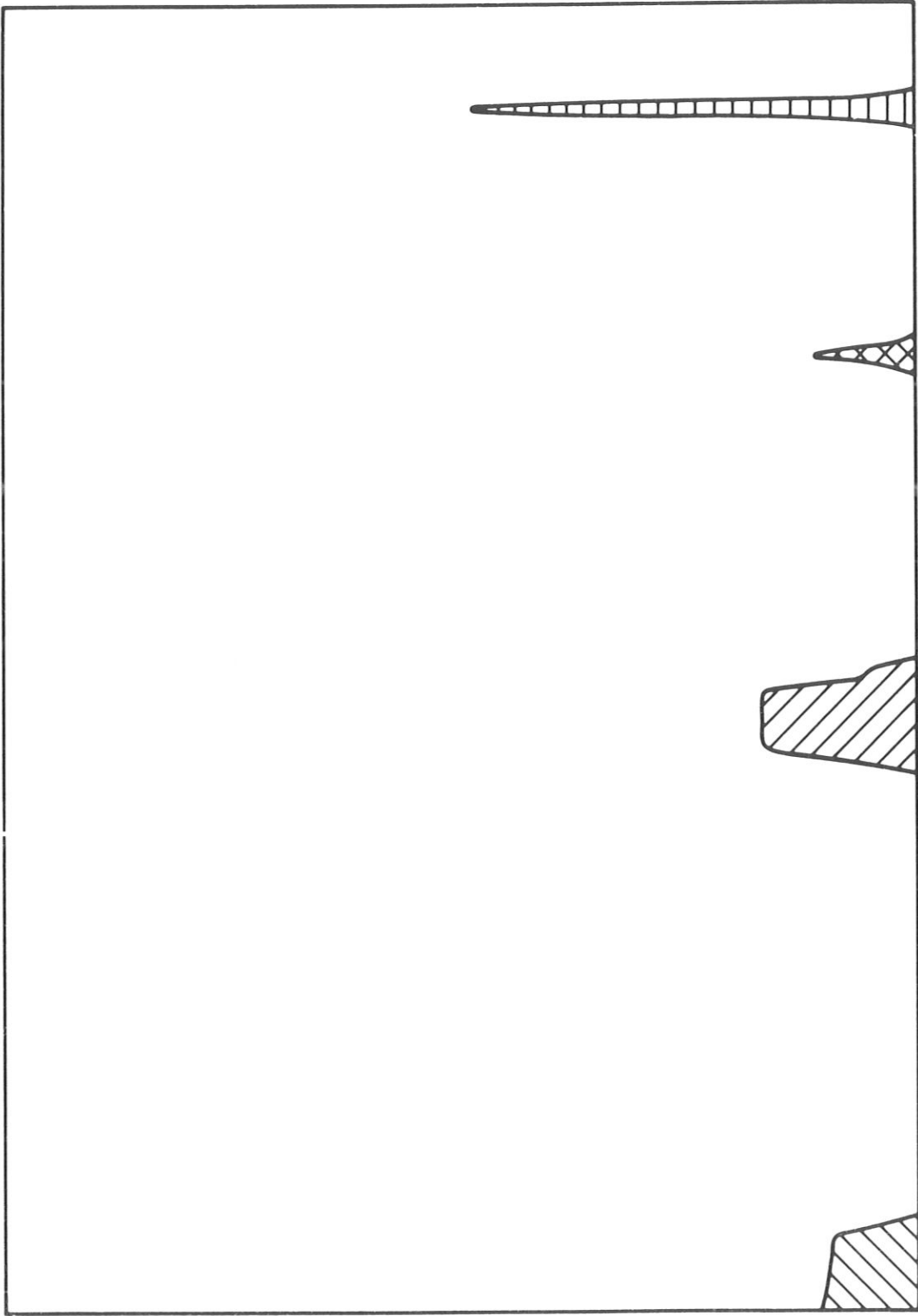


Fig. 1



YIELD

ENERGY

Fig. 2

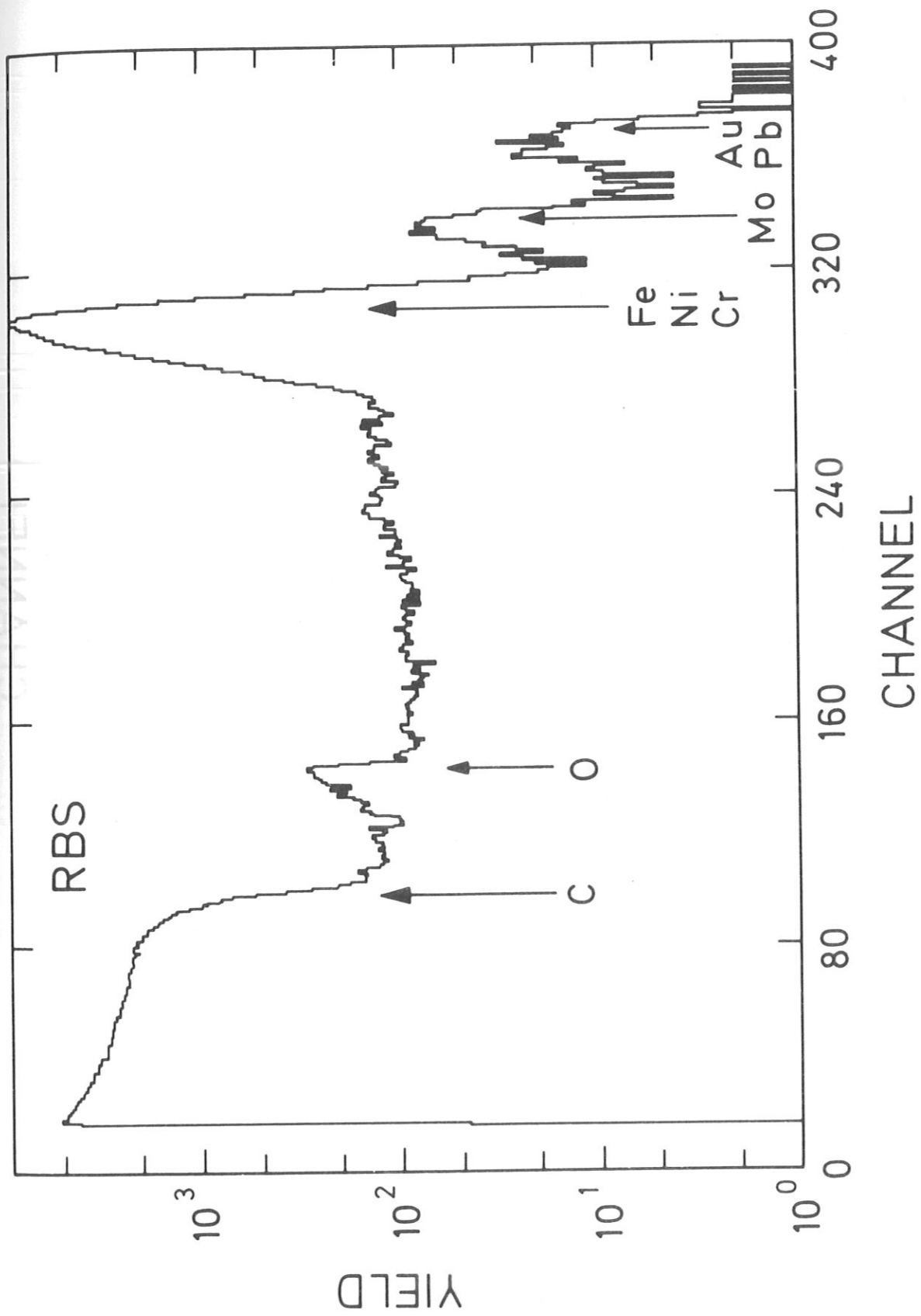


Fig. 3

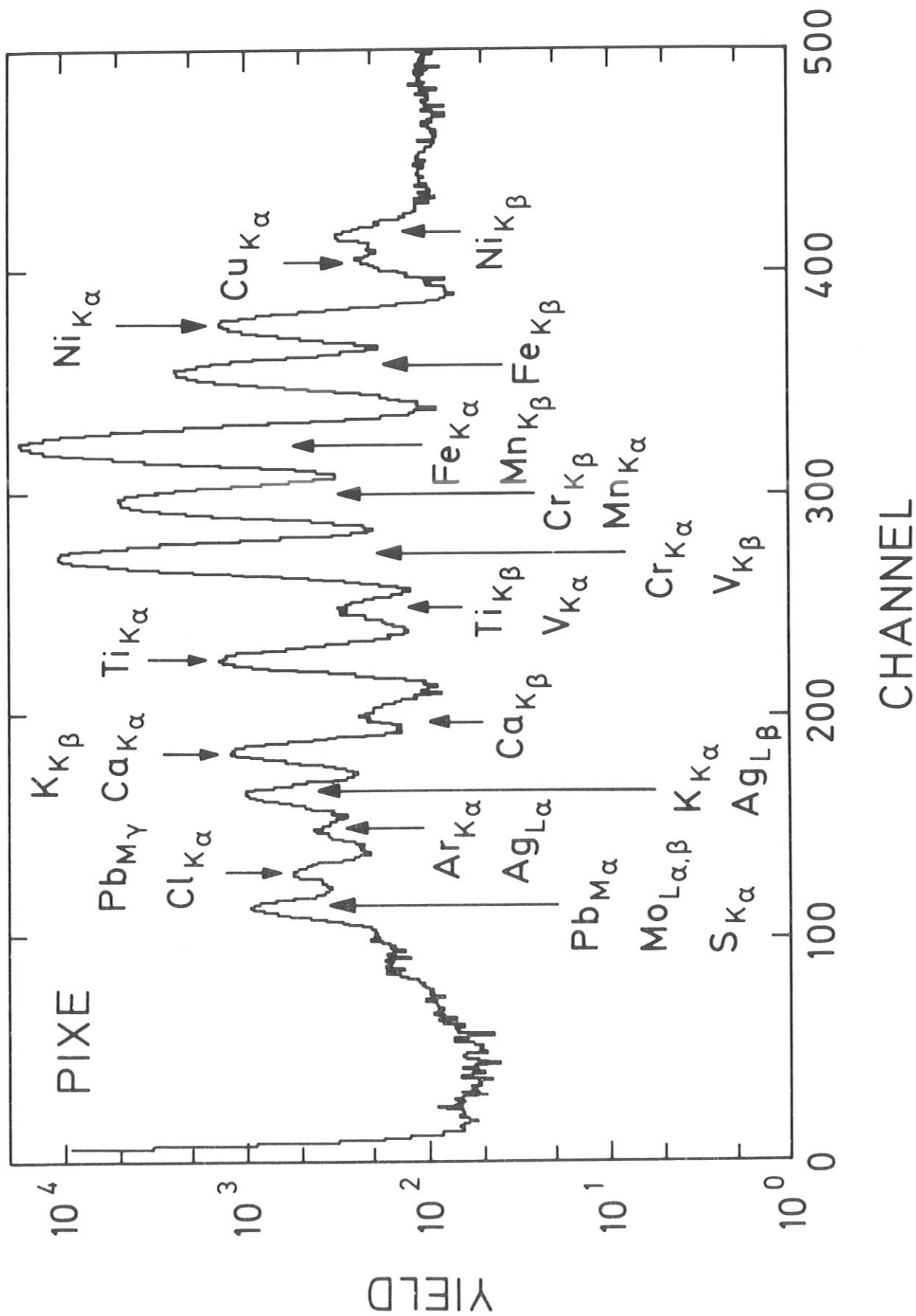


Fig. 4

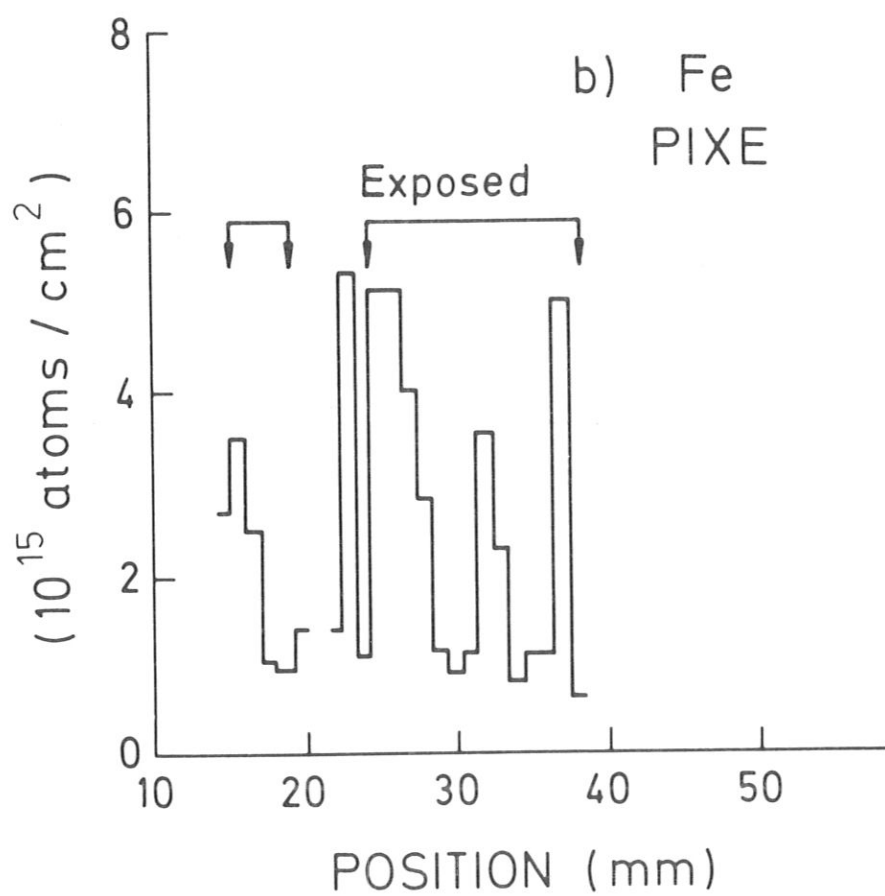
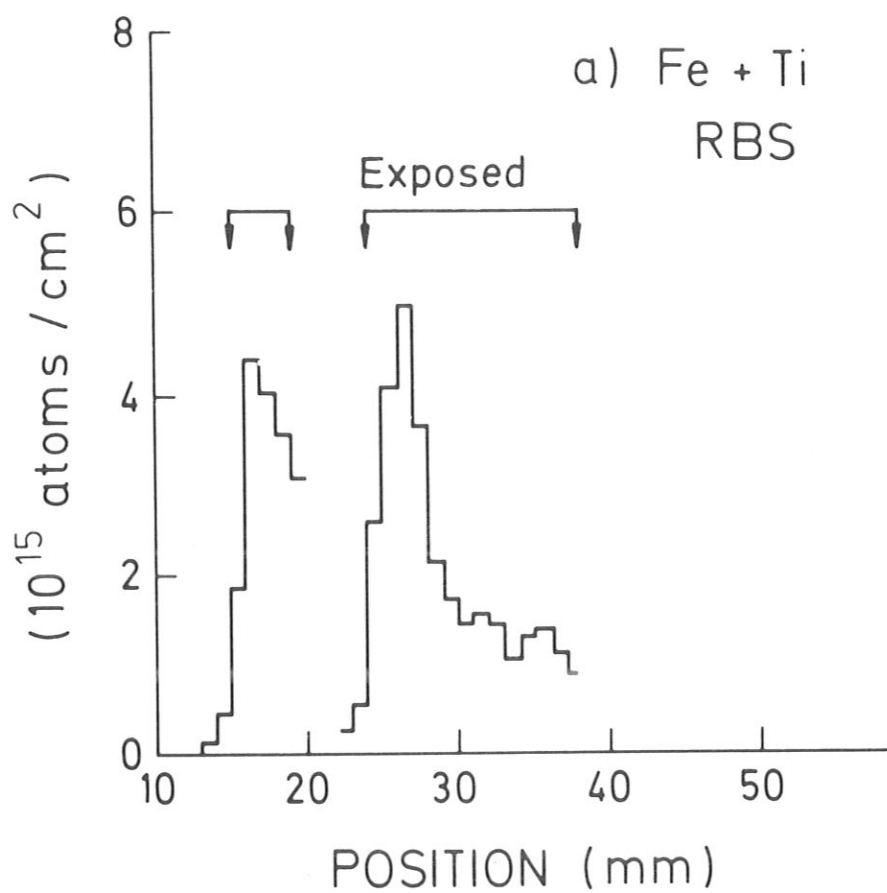


Fig. 5

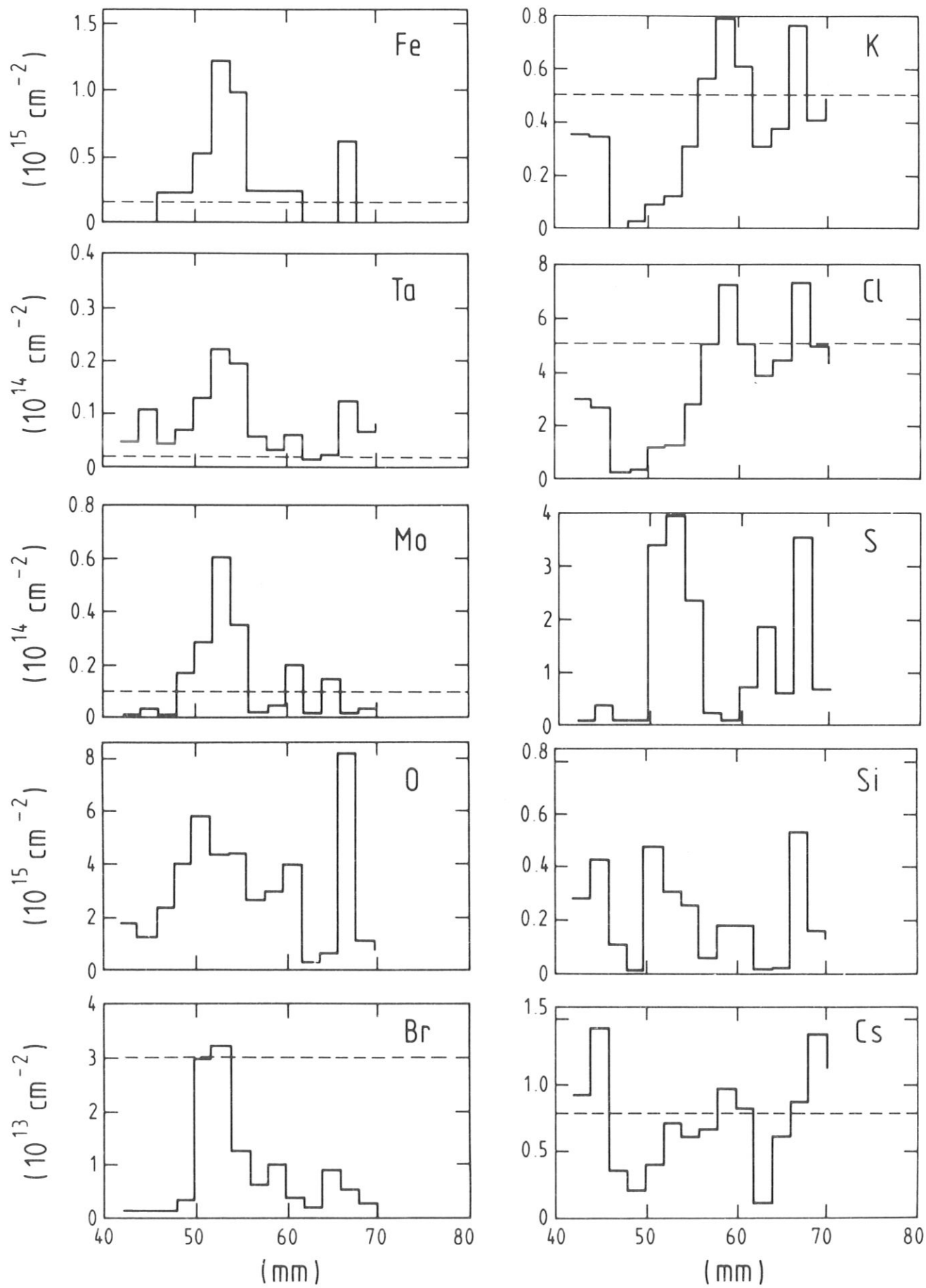


Fig.

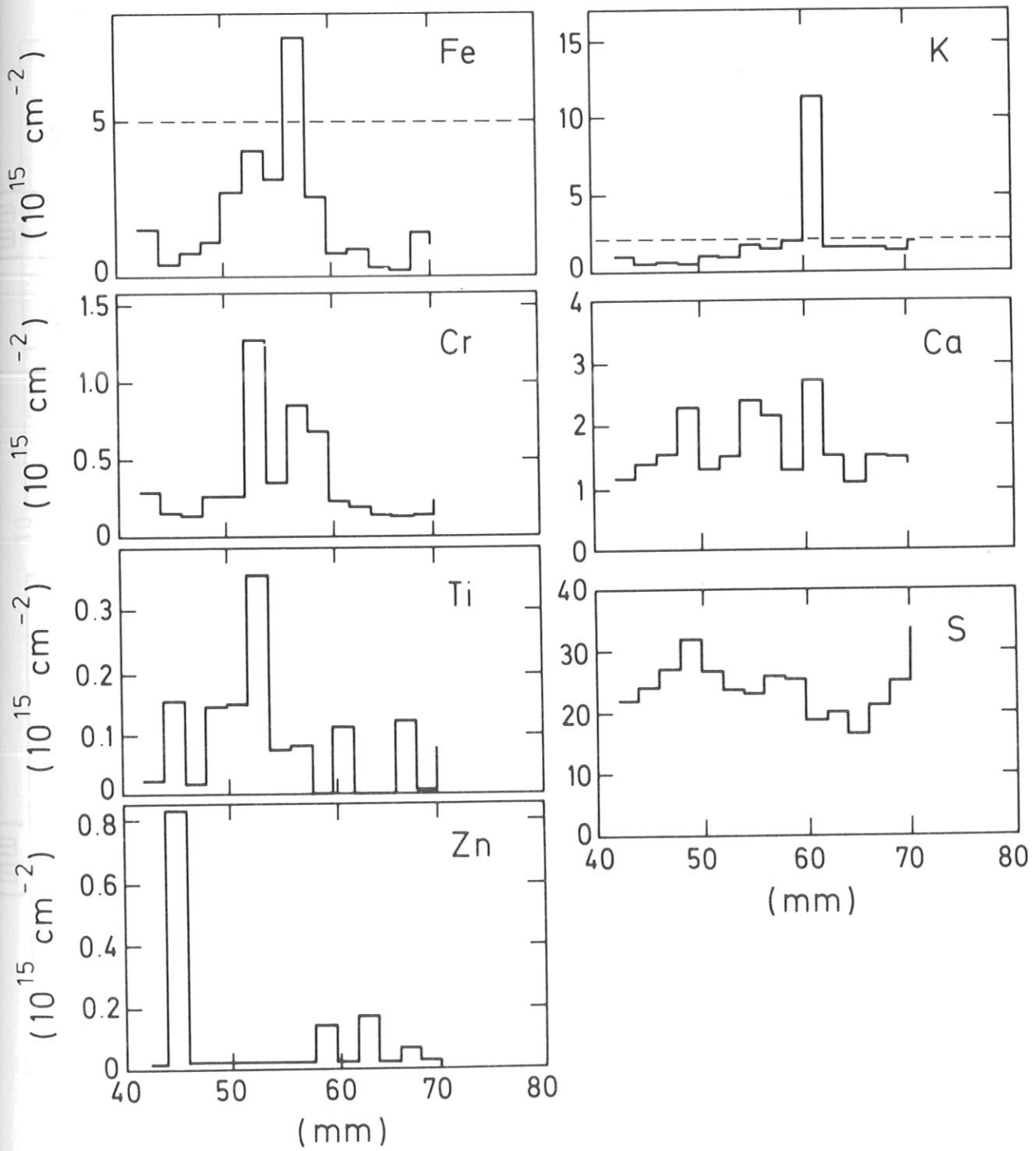


Fig. 7

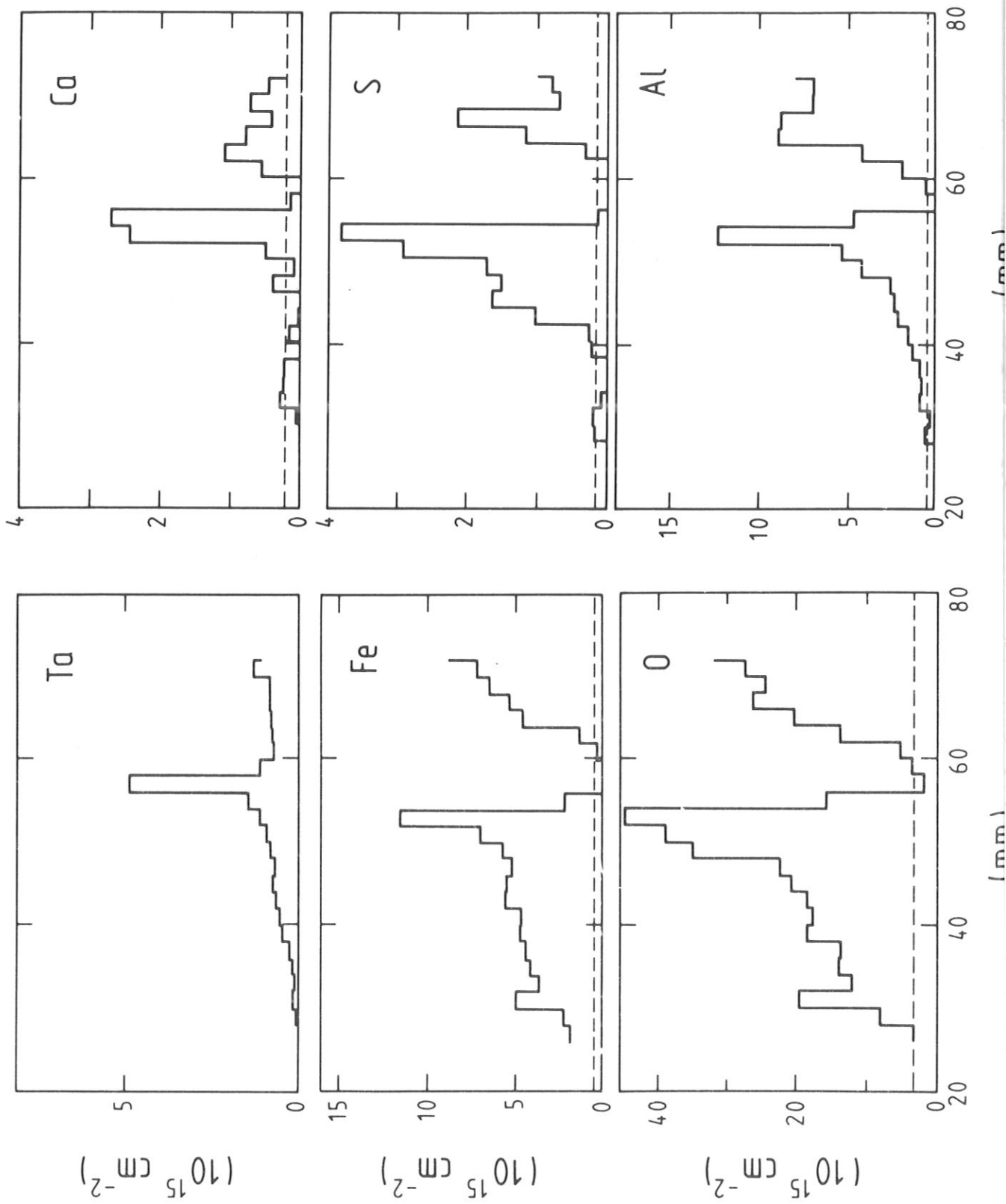


Fig. 8

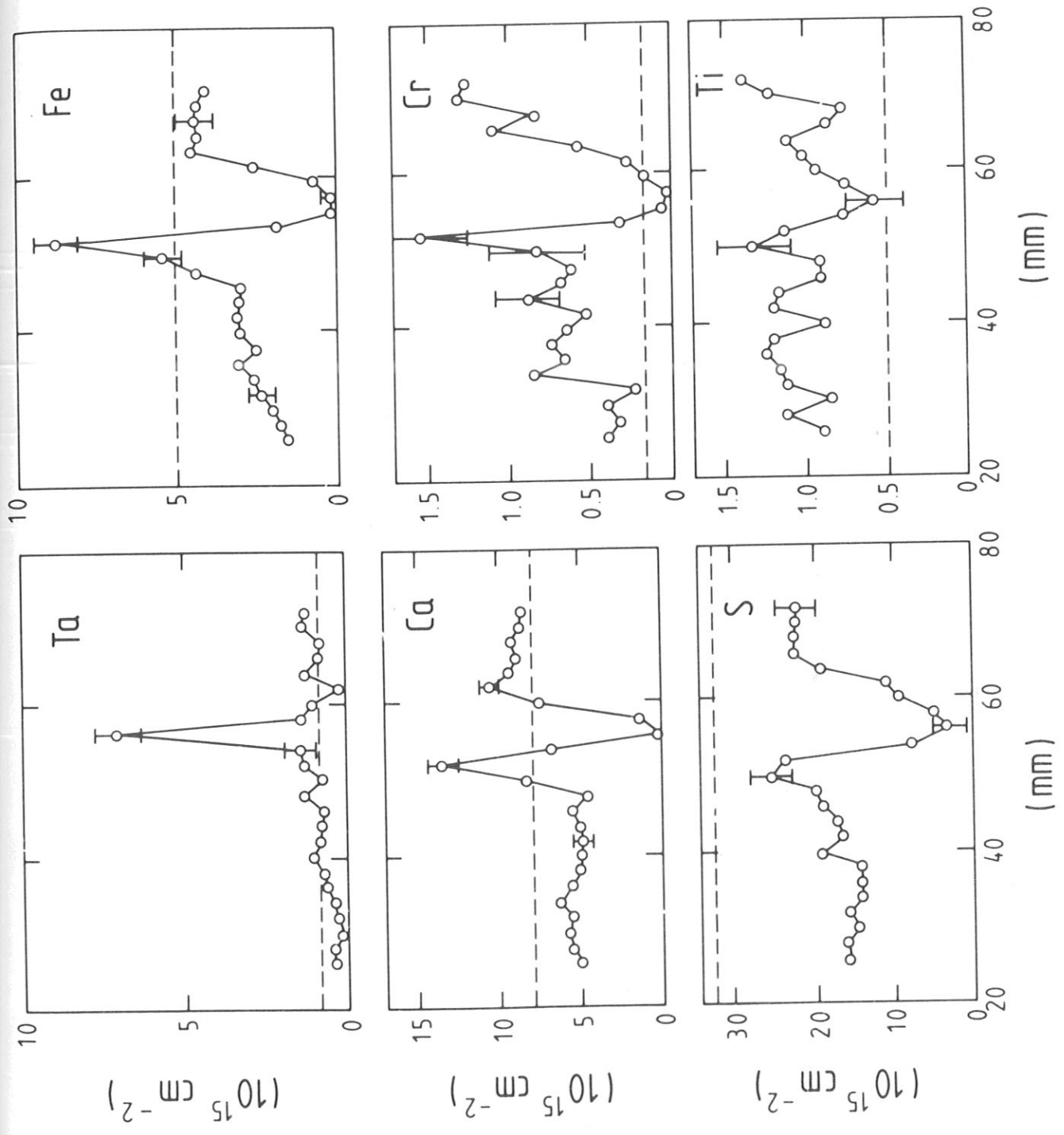


Fig. 9

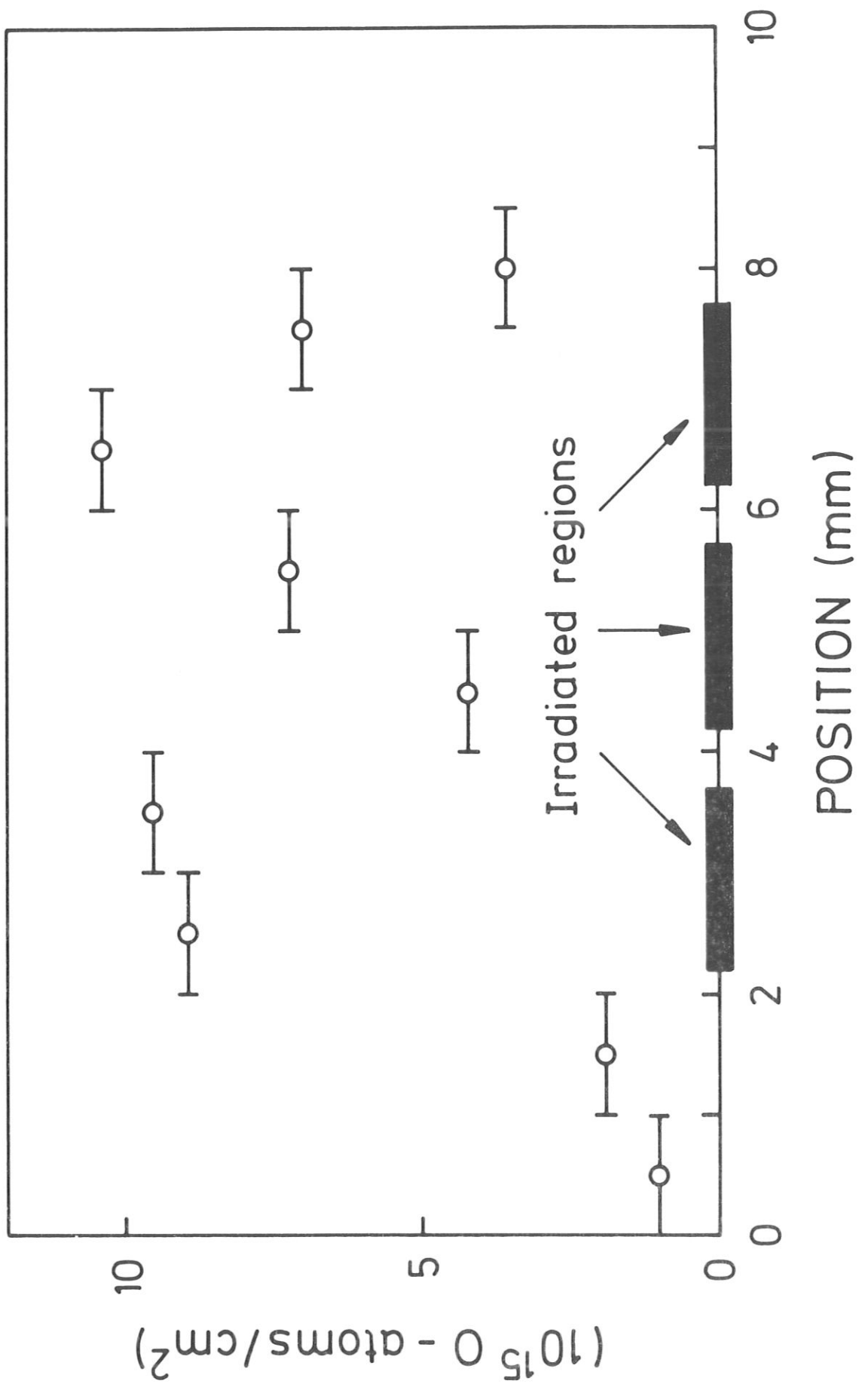


Fig. 10