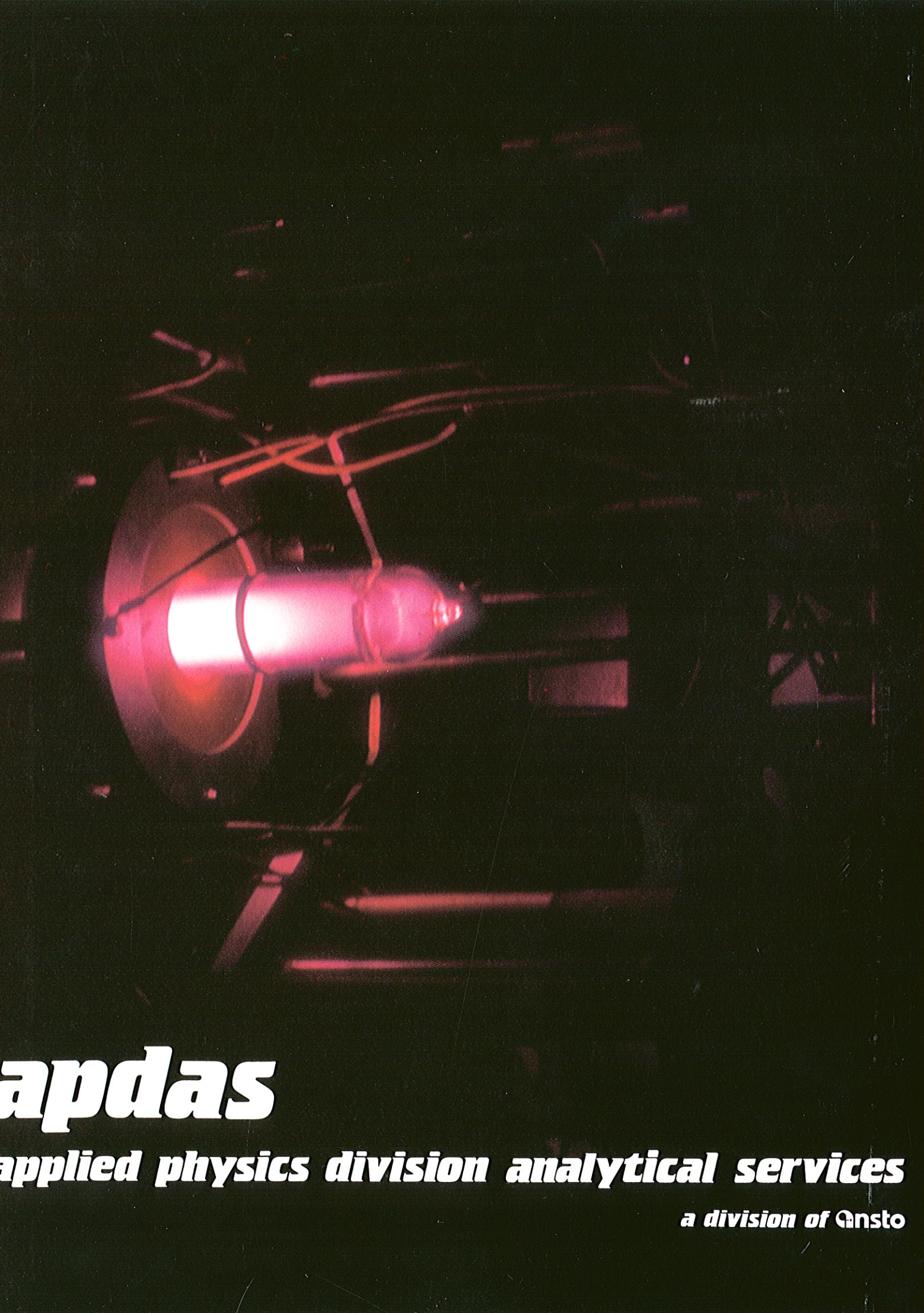

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Applied Pnysics Division Analytical Services (APDAS) is a new initiative within the Australian Nuclear Science and Technology Organisation. This government-funded organisation, Ansto, is fully committed to building and maintaining strong links with industry through the provision of a range of scientific and technical goods and services.

Because of its background and achievements in high-tech research, APDAS can provide solutions to many of the problems that arise in Australian industries.

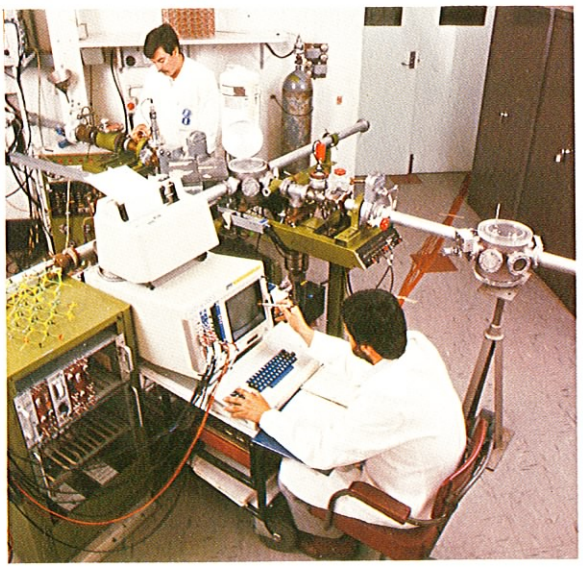
One of the facilities available to APDAS is a positive ion particle accelerator. This enables any positive ion in a gaseous medium to be accelerated to energies ranging from a few hundred thousand to three million electron volts for single charge states.

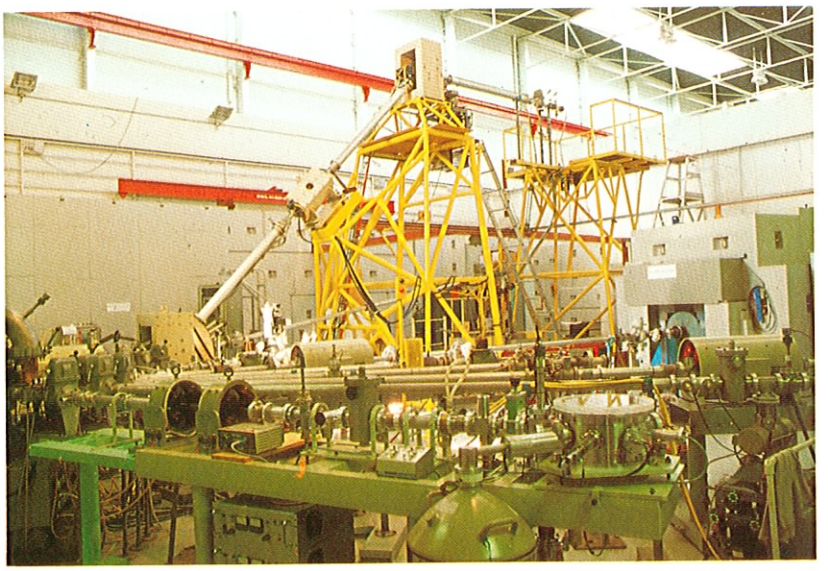
The accelerated ions strike a target causing nuclear reactions which allow several types of analyses including elemental analysis to be performed.

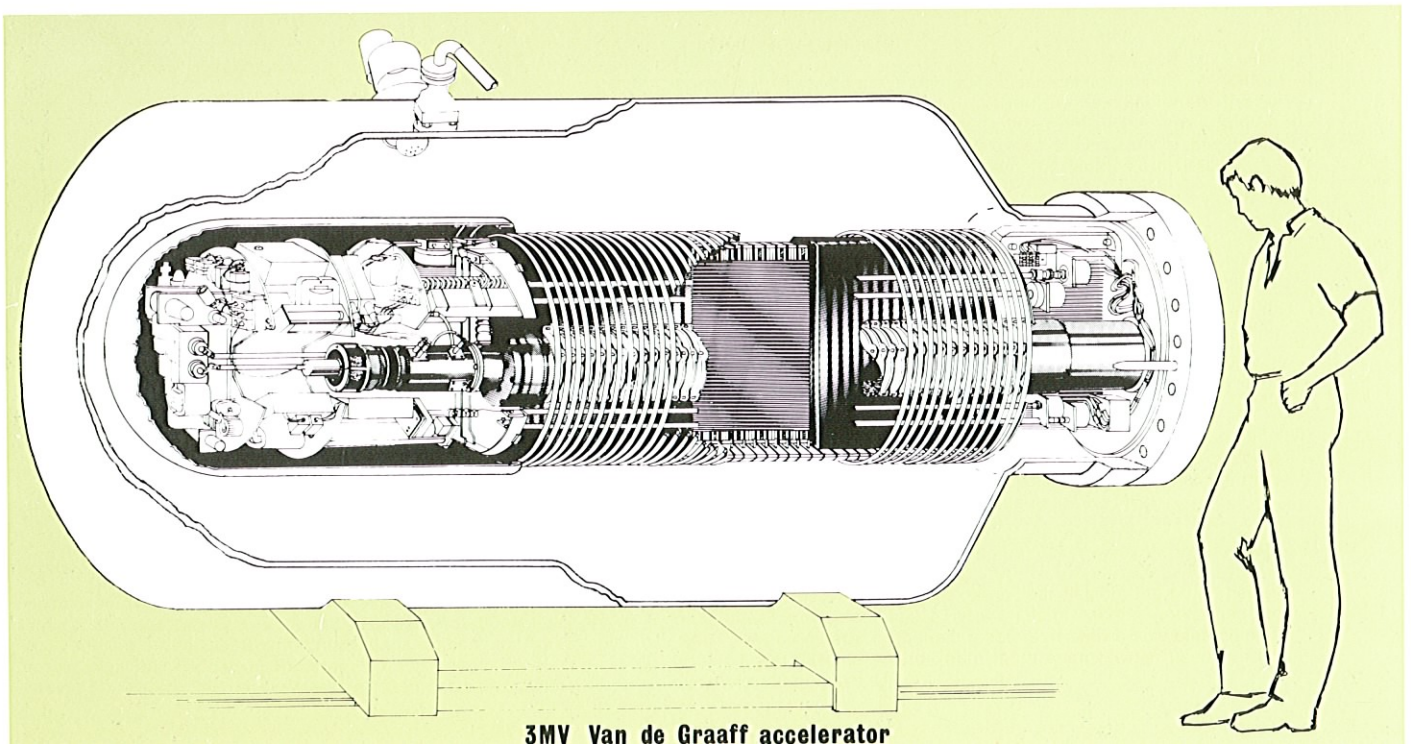
In this APDAS facility, ion beams can be steady-state or pulsed with pulse durations as low as three nanoseconds. Target preparation and fully automated data recording are also available.

ACCELERATOR-BASED SERVICES PRESENTLY AVAILABLE

- Proton Induced X-ray Emission (PIXE)
- Proton Induced Gamma-ray Emission (PIGME)
- Standard Neutron Irradiation Facility (SNIF)
- Oxygen-18 Analysis
- Surface Analysis
- Valuable Specimen Analysis
- Analysis Software Packages
- Nuclear Reaction Profiling







3MV Van de Graaff accelerator

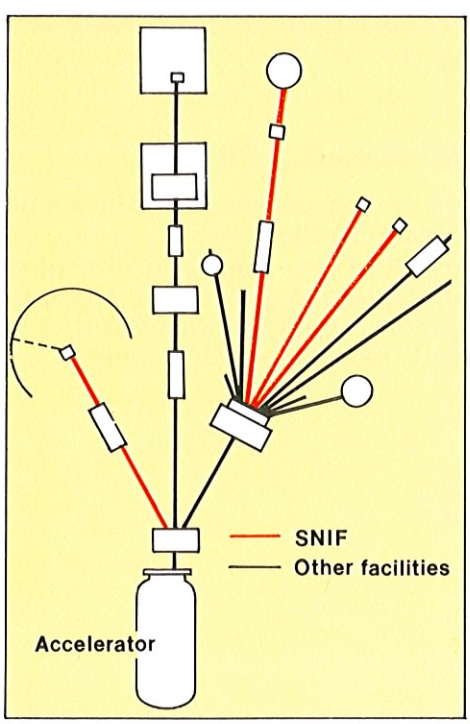
ACCELERATOR BASED TECHNIQUES

Printed at the
Lucas Heights Research Laboratories
Designed by Laura Edwards

STANDARD NEUTRON IRRADIATION FACILITY – SNIF

WHAT IS SNIF?

SNIF was established in 1984 within the Applied Physics Division at the Lucas Heights Research Establishment. It is several beamlines on the 3 MV Van de Graaff accelerator dedicated to producing neutron beams of known energies and known intensities. These neutrons are produced by bombardment of light solid targets, such as ^{7}Li, ^{9}Be, or gas targets such as ^{2}D, ^{3}T, by light ions such as protons, alpha particles or deuterons. The kinematics of these light ion reactions determines the neutron energies and fluxes produced, and these may be tailored to some extent to meet user demands.



PLAN VIEW – ACCELERATOR BEAM LINES

NEUTRON BEAMS AVAILABLE

Using neutron producing reactions such as:

^{7}Li(p,n)^{7}Be
^{9}Be(d,n)^{10}B
^{2}D(d,n)^{3}He
^{3}T(d,n)^{4}He

we can supply up to approximately 3 x 10^{8} neutrons cm^{-2} s^{-1} with energies from tens of keV to several MeV. The upper neutron energy limit is presently set by the maximum voltage of 3 MV obtainable on the Van de Graaff accelerator. The maximum neutron flux tends to be set by target heating effects of the bombarding ions or by the radiation shielding problems associated with large neutron flux production.

The neutrons are generally produced in air at the end of the beamline which means that geometrical restrictions on sample sizes are minimal. Sample sizes from sub-millimetre to around 100 cm diameter are possible, depending on the neutron flux distributions required. Solid, liquid or gas samples are also possible.

APPLICATIONS OF SNIF

The possible applications of tailored neutron beams are only limited by the user's imagination. However some typical applications to date include:

(a) Bulk sample analysis — using inelastic neutron scattering, particularly good for elemental analysis of such elements as Li, Na, Al and Au.
(b) Resonance Neutron Radiography — a non-destructive technique for measuring and imaging the isotope distributions within samples.
(c) Determination of sample moisture content by neutrons and γ-ray transmission measurements. This has been applied in industry to measure moisture content to coke, coal, wood, soil and even meat.
(d) Neutron Radiobiology
- fast neutron therapy for the treatment of cancers;
- neutron damage studies in biological systems (DNA etc.);
- gene cloning studies for radioresistance;
- in vivo neutron diagnosis.

NEUTRON DETECTORS

Currently there are several types of neutron detector systems available within SNIF, these include

Long Counters
Plastic/Liquid Scintillators
BF_3 Counters and
Li Glass Scintillators

which adequately cover the available neutron energy ranges.

NEUTRON DOSE CALCULATIONS

For many biological applications it is necessary to convert neutron energy/flux parameters to total sample dose (grays) received. This is possible using a variety of computer codes and further work is proceeding on SNIF to expand these dose calculations to the widest possible range of samples.

An example of the dose received by a phantom tissue sample for various neutron energies is given in Fig. 1.

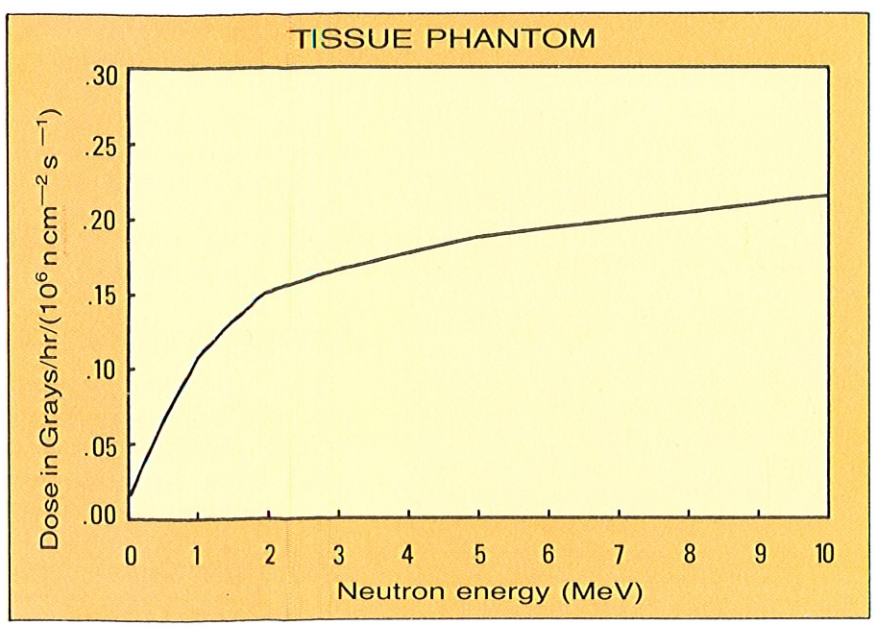


Fig. 1

TYPICAL COSTS AND AVAILABILITY

The cost to outside users of the accelerator time and technical support is subject to negotiation.

SNIF can be made available to Australian tertiary institutions through the Australian Institute of Nuclear Science and Engineering which supplies yearly grants to these institutions to use the Lucas Heights facilities.

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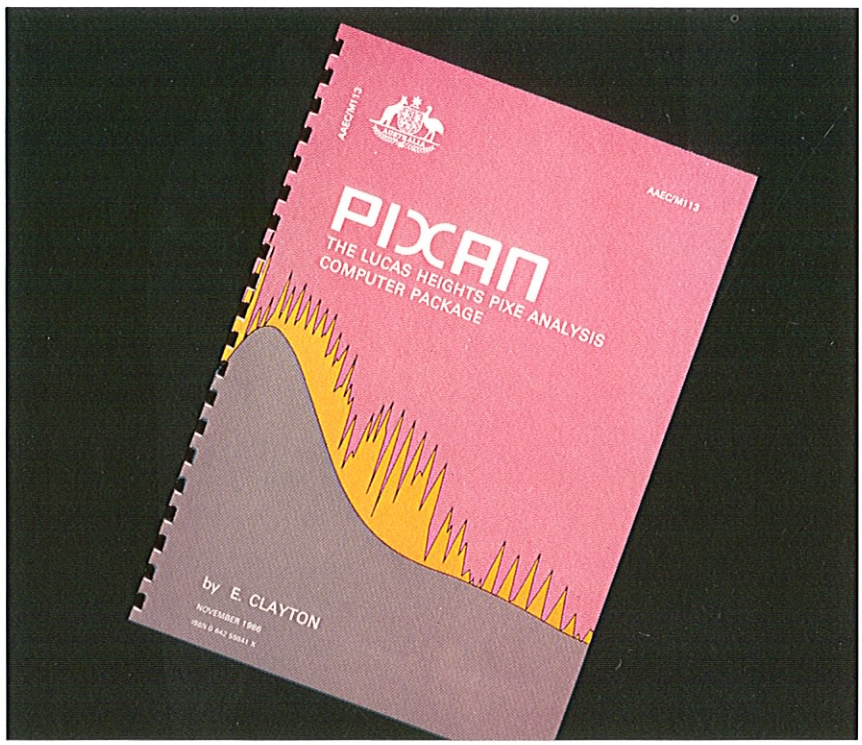
PIXAN – THE LUCAS HEIGHTS PIXE ANALYSIS PACKAGE



There are two programs in the package: BATTY, to calculate peak areas in PIXE spectra; and THICK which calculates X-ray yields for thick, thin and intermediate thickness samples. Both programs are suitable for running on VAX/VMS systems. They are written to be portable and have run on IBM mainframes, such as ANSTO's central computer system, VAX 11/780s and 11/785s, Norsk Data and Data General, as well as Unix-based systems such as the Pyramid.

The package is available on 9 track 1600 bauds/inch magnetic tape. The tape consists of the following files:

1. BATTY — the Fortran 77 source for the peak analysis program.
2. DSET 1 — the control dataset for BATTY.
3. DSET 2 — contains element X-ray energies and relative intensities.
4. DSET 3 — contains attenuation and stopping power data.
5. SPECTR — a measured X-ray spectrum for the test problem.
6. THICK — the Fortran 77 source of the X-ray yield program.
7. DSET 6 — the control dataset for THICK.
8. DSET 4 — contains element X-ray data (fluorescence yields, energies, etc.).
9. DSET 5 — a table of ECPSSR cross sections for protons (0.1 to 6 MeV).

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PIXE ELEMENT ANALYSIS

Proton Induced X-ray Emission (PIXE) is a fast simultaneous multi-element surface analysis technique. Samples can be solids, soils, powders and even aerosol particulates collected on filter paper.

In conjunction with PIGME (Proton Induced Gamma-ray Emission), the APDAS automated PIXE facility (Fig. 1) provides a unique analytical capability.

FEATURES

In 5 minutes, X-ray data can be collected from up to 20 elements. The emitted X-rays are, like a fingerprint, characteristic of the individual element.

The abundance of each element in the unknown substance can be obtained from a computer analysis of the X-rays emitted by that element. Major components and trace elements from aluminium to uranium, down to 1 mg/kg, can be determined in a single measurement.

Samples examined include environmental aerosols, freeze-dried blood and hair, archaeological pottery and natural glass, coal, oil shale and ores.

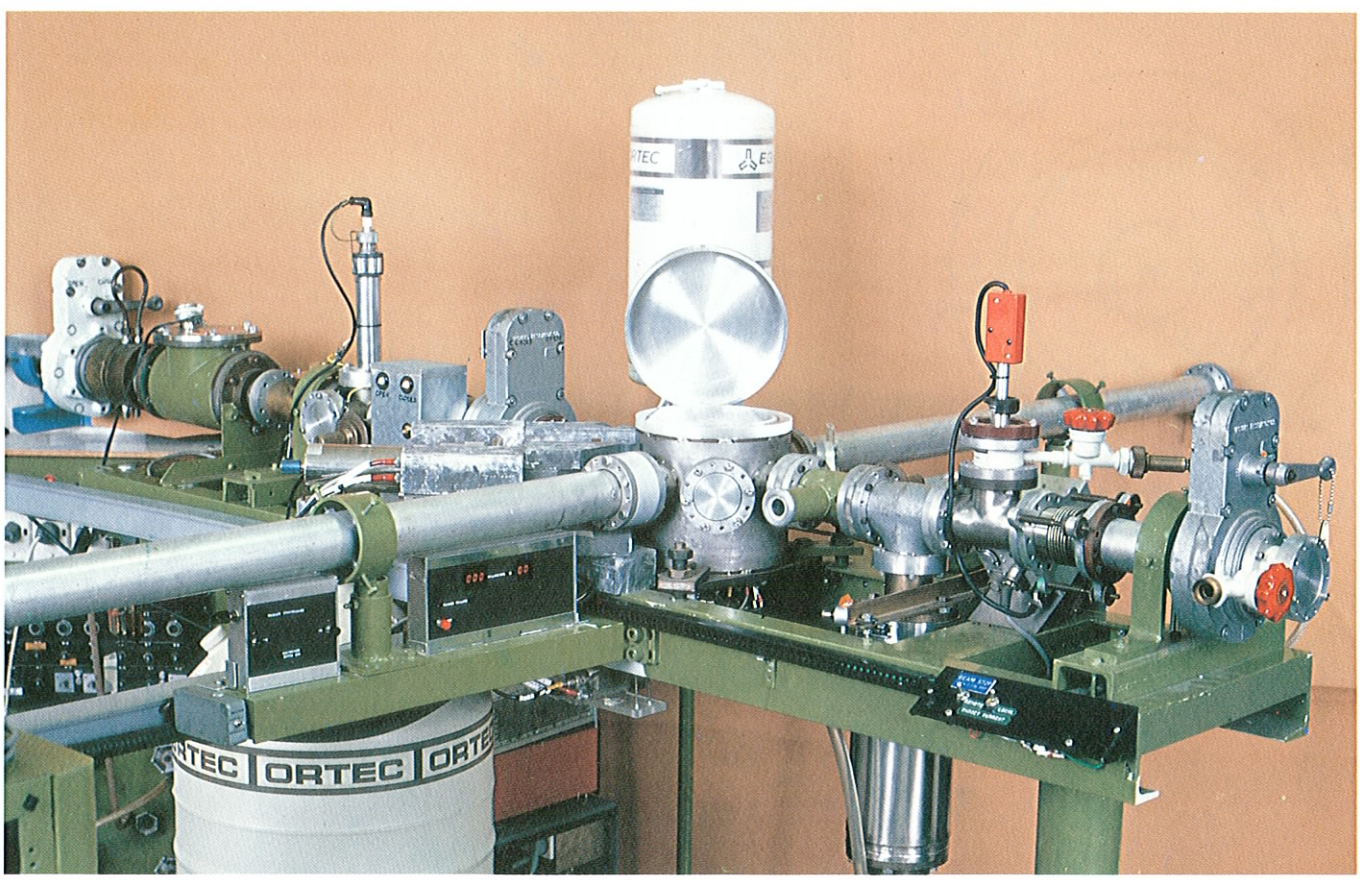


FIG. 1
PIXE AUTOMATED TARGET STATION AND ANALYSIS SYSTEM

PROCEDURE

Up to 60 samples can be loaded on a target stick for analysis in a vacuum chamber where they are bombarded with high energy proton beams (2-3 MeV) produced by a 3 MV Van de Graaff accelerator.

X-rays are produced by ionisation processes which occur during the slowing down of the proton in the sample.

The Coulomb interaction between the ion and the target nucleus results in the ejection of an inner shell (K, L etc.) electron (A). The resulting inner shell vacancy is filled by an outer electron (B) and an X-ray (C) carries off the excess energy. These X-rays are characteristic of the excited atoms (D). (See fig. 2).

X-ray spectra similar to that shown in figure 3 are stored in an on-line computer.

The energy of the X-rays is characteristic of the elements from which they originate and the number of X-rays is proportional to the elemental concentration.

PIXAN, and APDAS-produced computer package for the analysis of PIXE spectra, is then run for the final data reduction which produces for you a report tabulating measured elemental data, such as element concentration, and minimum detection limits.

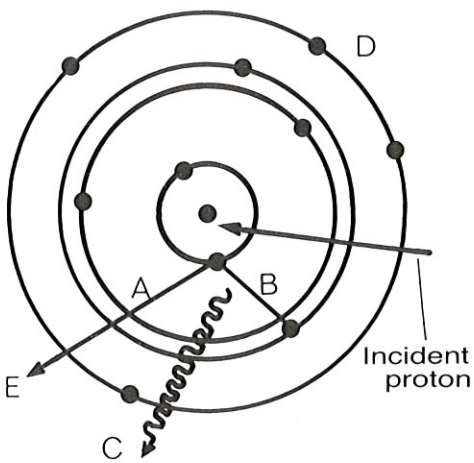
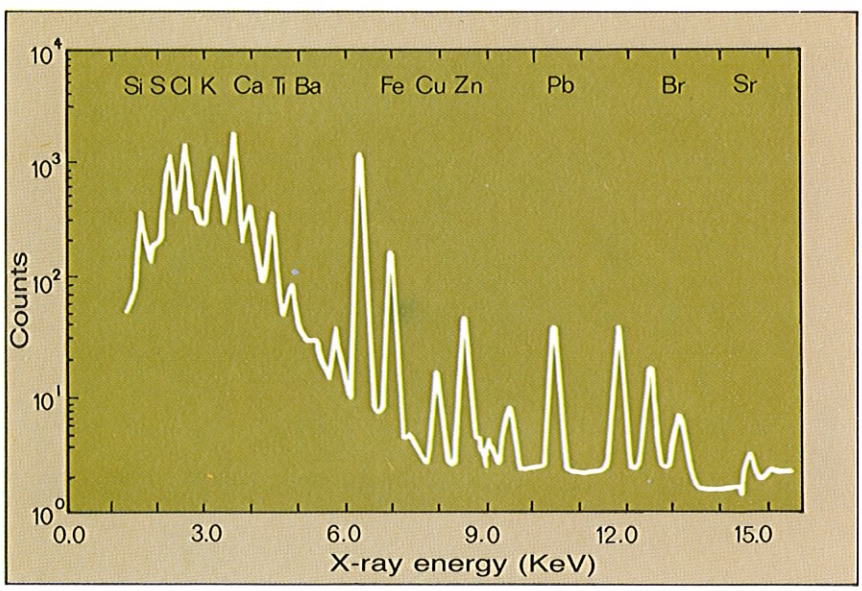


FIG. 2



PIXE ANALYSIS OF COARSE PARTICULATE MATTER IN A SAMPLE OF AIR

FIG. 3

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applied physics division analytical services

SURFACE ANALYSIS AND DEPTH PROFILING USING ION BEAMS

The study of material surfaces has been revolutionised recently by the development of a number of accelerator-based analytical methods. Through these analytical methods information is gained on the quantitative depth profiles of constituent elements in the surfaces of samples.

Particular applications of surface analysis techniques are the study of semiconductor devices for depth profiles of elements such as hydrogen and oxygen, surface contamination information, and thin film measurements of uniformity and depth.

The techniques are non-destructive, highly sensitive, have good accuracy and are applicable for depth in the range 10 nm-100 μm.

METHODS

APDAS can provide several techniques. The various types of interactions are illustrated in figure 1. In each of them an incident ion beam interacts with the sample at depths which are related to the ion's energy and mass. The ion may be scattered from the sample in a forward or backward direction and gamma rays or secondary ions may be produced.

The scattered ions or secondary radiations are measured in an appropriate detector and, from the measured intensities, quantitative data on the constituent elements or a profile of the element concentration to the maximum range of the incident ion in the sample can be obtained.

- Rutherford backscattering (RBS) can provide quantitative data on major light element components such as nitrogen or carbon, or multi-element depth profiles for heavier elements in lighter matrices, such as Au or Si or As in Si.
- Nuclear reaction analysis (NRA) provides depth profiles for individual elements such as oxygen, lithium, boron, fluorine and sodium.
- Forward recoil analysis (FRA) gives depth profiles for light elements — in particular hydrogen and deuterium.
- Particle induced X-ray emission (PIXE) provides simultaneous measurement of all elements, of atomic number greater than 10, in near-surface layers.
- Channelling of ions is used for lattice and impurity location in near-surface regions.

Each of these techniques are discussed in detail on separate sheets.

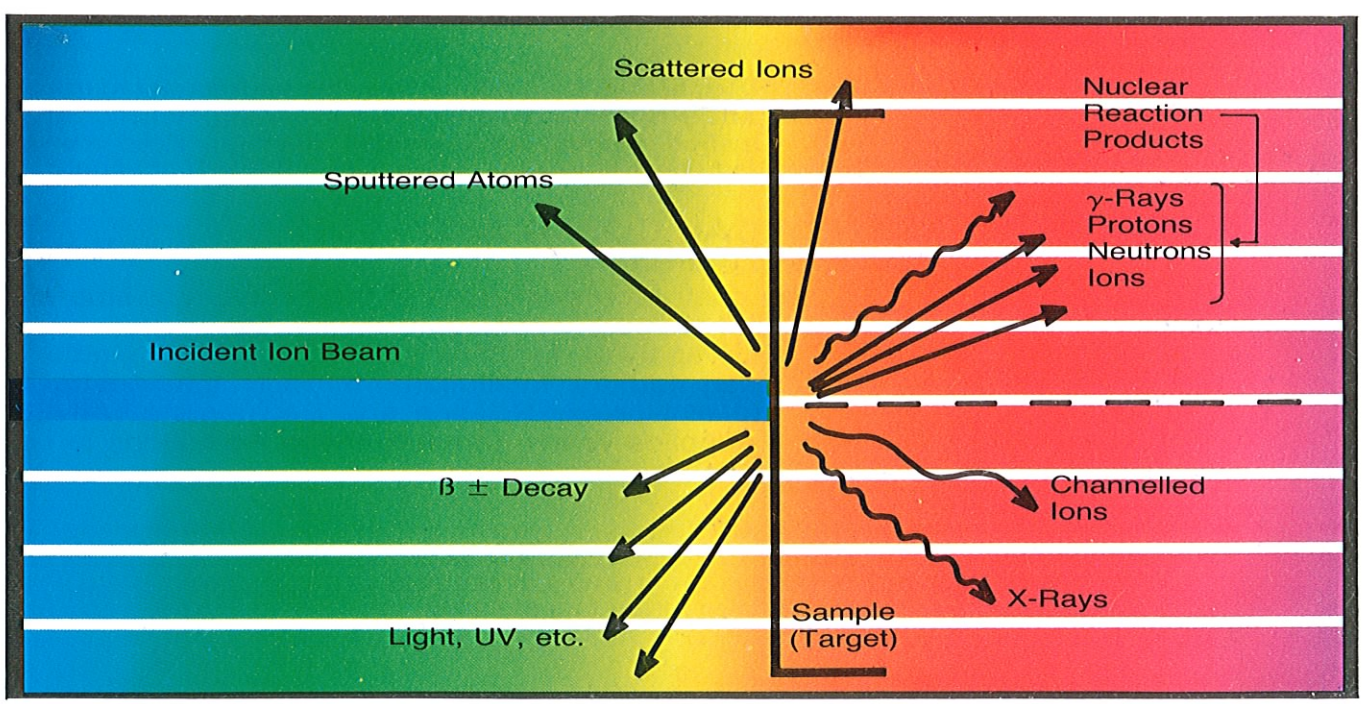

FIG. 1.
ION BEAM INTERACTIONS WITH MATTER

An example of the use of techniques is shown in figure 2, where the RBS spectrum resulting from a 15 nm layer of AuGe-Ni deposited on a GaAs substrate is shown. This technique was used to study the penetration of Au contact layers to GaAs crystals.

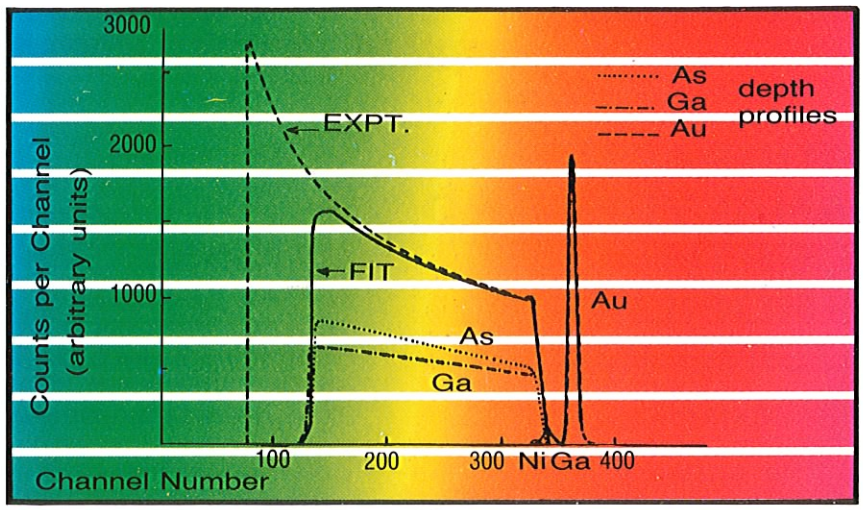

FIG. 2.

RBS spectrum for 2 MeV ^{4}He ions backscattered from 15 nm AuGe-Ni deposited on GaAs before alloying. Reaction angle 90°. Dashed curves: experimental spectra. Solid curves: computed spectra. Energy calibration ~ 5.2 keV/channel.

Subsequent measurements on a furnace alloy sample showed the Au alloyed region now extends down to about 200 nm into the GaAs. This is shown in Fig. 3 where the Au profile has changed from the sharp surface peak (15 nm) in Fig. 2 to the broader surface profile (200 nm) in Fig. 3.

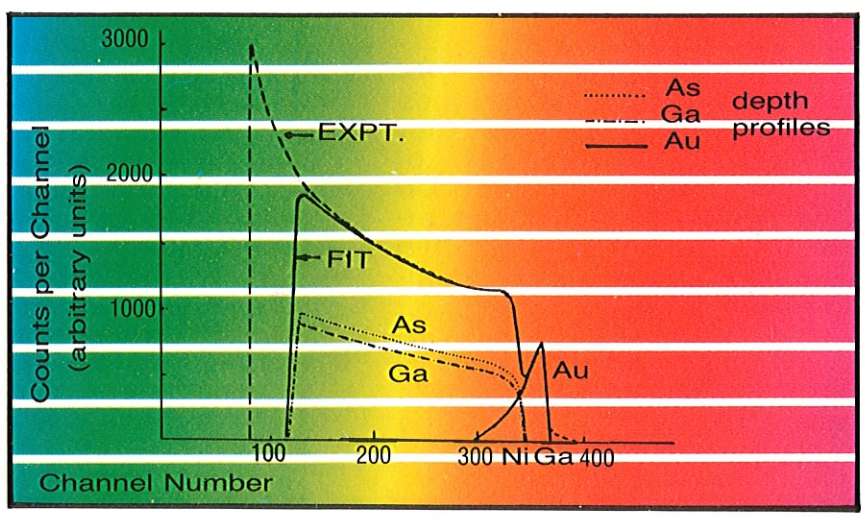

FIG. 3.

RBS spectrum from 2-MeV ^{4}He ions backscattered from 15 nm AuGe-Ni deposited after furnace alloying. Reaction angle 90°. Dashed curves: experimental spectra. Solid curves: computed spectra. Energy calibration ~ 5.2 keV/channel.

FEATURES

(i) Non-destructive.
(ii) High sensitivity and accuracy.
(iii) Probe depth 20 nm-1 μm.
(iv) Hydrogen and oxygen profiles.

APPLICATIONS

(i) Semiconductor studies.
(ii) Depth profiles.
(iii) Surface contamination.
(iv) Thin films.

TYPICAL COSTS AND AVAILABILITY

The cost to outside users of the accelerator time and technical support is subject to negotiation.

These analysis facilities can be made available to Australian tertiary institutions through the Australian Institute of Nuclear Science and Engineering (AINSE) which supplies yearly grants to these institutions to use the Lucas Heights facilities.

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OXYGEN ^{18} ANALYSIS

^{18}O is the most abundant stable isotope of oxygen after ^{16}O with a natural abundance of 0.204 atom % in atmospheric sources, 0.200 atom % in sea water and between 0.1889 and 0.2094 atom % in other sources. Oxygen is an important natural element; it occurs as oxides in most geological applications and is important in the metabolism of most living creatures. A technique that can analyse oxygen, or one of its stable isotopes, down to or below naturally occurring levels, is therefore most useful.

THE ANALYSIS

The 3D target manipulator shown in Fig. 1 has been set up to analyse ^{18}O samples using the $^{18}O(p,\alpha)^{15}N$ reaction. This reaction has a resonance, in its cross section, at a proton energy of 846 keV (see Fig. 2), which is broad enough (47 keV) to produce sufficient alpha particle yield to analyse for 0.1 atom % ^{18}O in just a few minutes of accelerator running time.

The cross section is angular dependent and factors of two in yield are obtained by going to forward reaction angles.

Angles between 10° in, 10° out and 50° in, 50° out are optimal. This is shown in the schematic of Fig. 3. The absorber foil in front of the detector allows the alpha particles (4 MeV) through but completely stops the scattered protons ($<$ 850 keV). Fig. 4 shows a typical alpha spectrum obtained using this technique for a 98 atom %, ^{18}O enriched Ta_2O_5 sample 0.23μm thick. The solid curve is a computer fit to the data and allows an ^{18}O depth profile to be obtained as well as its concentration.

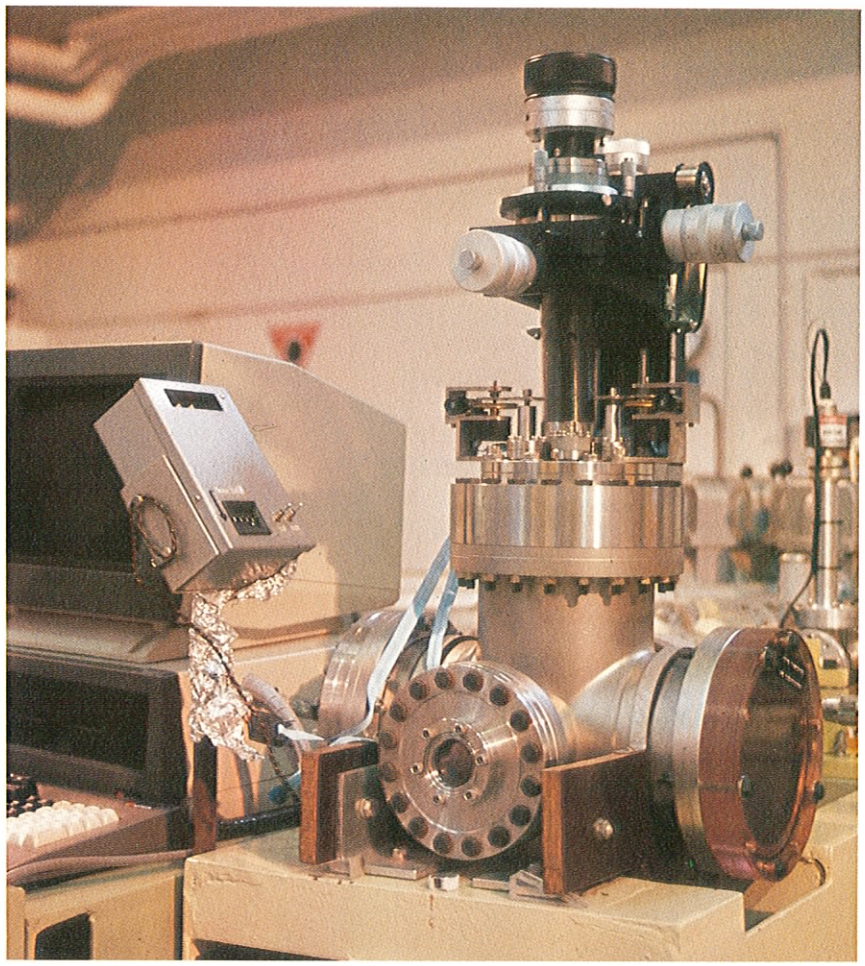

FIG. 1

TYPES OF SAMPLES

The manipulator shown in Fig. 1 is under vacuum and is currently capable of handling only solid targets from a few millimetres to several centimetres in diameter. Techniques have been developed to handle liquid samples by converting them to solid targets using standard anodising techniques on polished metal surfaces. ^{18}O enriched water samples as small as 150 μL have been successfully analysed in this way.

APPLICATIONS

This analysis is a surface technique and is limited in depth to the range of 850 keV protons or 4 MeV alphas, which is typically just a few micrometers.To date the main application has been in following the metabolic rates of small animals, such as lizards and magpies. These animals are doped with known amounts of ^{18}O enriched water, allowed to run wild for a few days, caught and blood samples taken. The blood samples are then converted to solid targets and analysed. The ^{18}O consumption allows zoologists to assess the metabolic rates of these animals.

Other obvious applications are possible, for instance looking at oxidation processes on metal surfaces or tracing oxygen movements in Si or other important semiconductor materials.

TYPICAL COSTS AND AVAILABILITY

The cost to outside users of the accelerator time and technical support is subject to negotiation.

This analysis facility can be made available to Australian tertiary institutions through the Australian Institute of Nuclear Science and Engineering (AINSE) which supplies yearly grants to these institutions to use the Lucas Heights facilities.

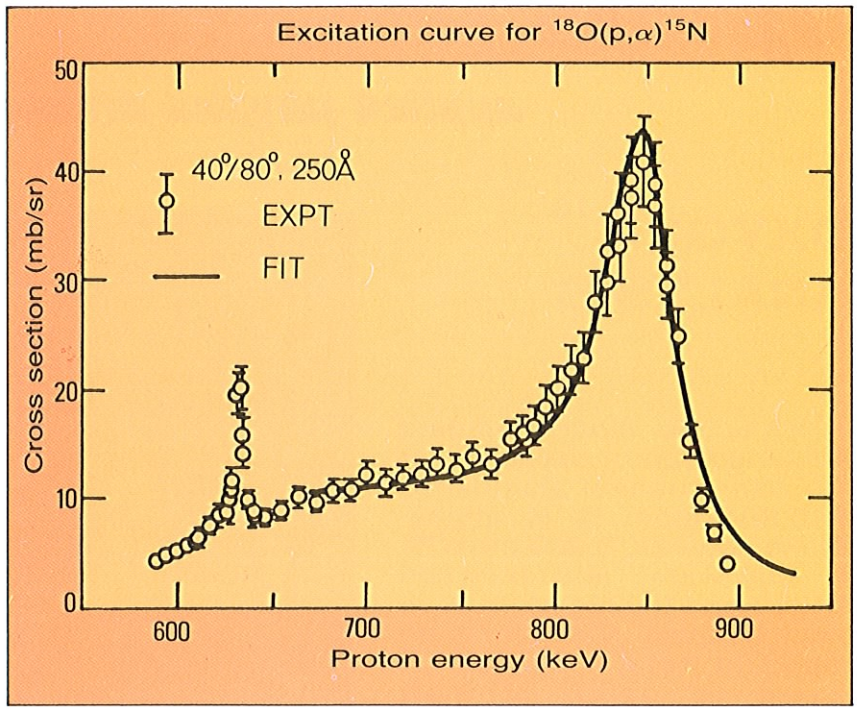

FIG. 2

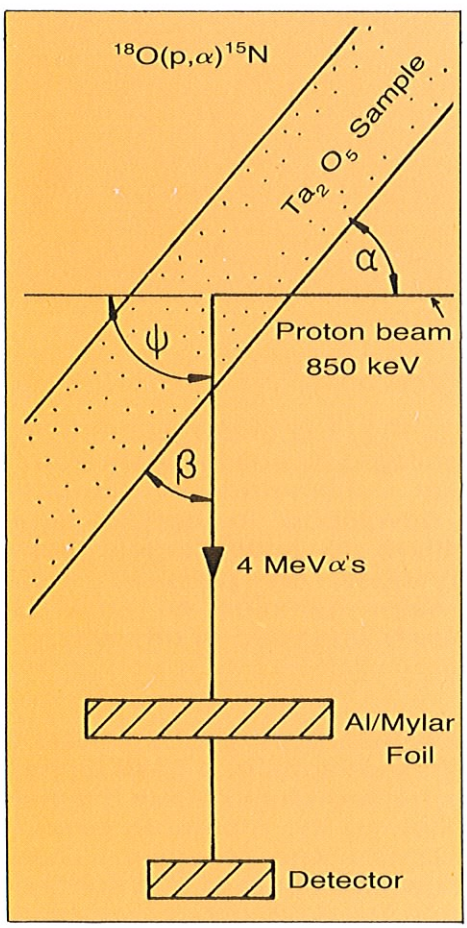

FIG. 3

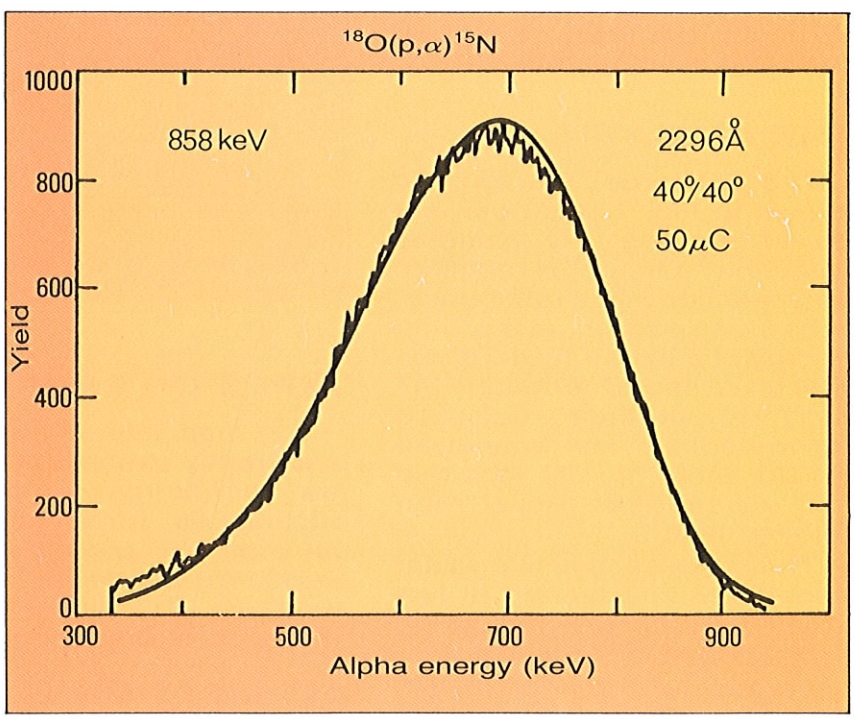

FIG. 4

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HYDROGEN ANALYSIS AND DEPTH PROFILING

The presence of hydrogen and its isotopes on the surface or in the near-surface regions of many materials can have dramatic effects on the physical, chemical and electrical properties of these materials. It affects the electrical properties of semiconductors and the efficiency of solar cells in the conversion of solar energy to electrical energy. Hydrogen resides on most surfaces in one form or another, it is an extremely mobile atomic species and it diffuses readily into most surface structures. A simple technique which is capable of analysing for hydrogen is therefore most useful.

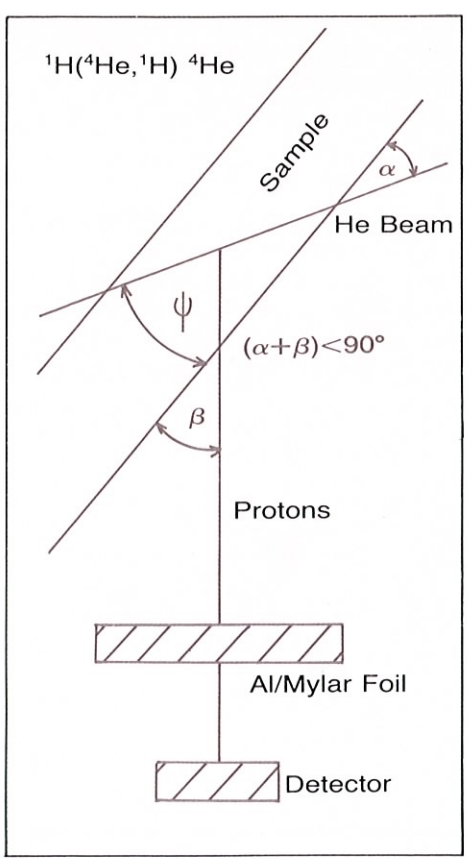

FIG. 1

THE ANALYSIS

Hydrogen is a most difficult atomic species to profile. Rutherford backscattering spectrometry (RBS) can be used to detect and profile most lighter elements, however, it is unsuitable for hydrogen analysis. A modification of the technique called elastic recoil detection (ERD) is used. This technique is based on the fact that a heavy ion elastically scatters a lighter atom from the surface regions of a sample and into a detector if the energy and the angle of incidence of the ion and the angle of emergence of the atom are correctly selected.

The experimental configuration used at Lucas Heights is illustrated schematically in Fig. 1. A helium beam (mass 4 amu) enters the sample at an angle α, between 5° and 40°, and the elastically scattered hydrogen atom (mass 1 amu) leaves the sample surface at an angle β, where $(\alpha+\beta)$ <90°. The Mylar foil (about 10 μm thick) prevents the scattered helium beam from reaching the detector. The hydrogen atoms are slowed down by this foil but not sufficiently to stop them from reaching the detection system. Typical helium beam energies obtainable at Lucas Heights on the 3 MV Van de Graaff accelerator are from 1 to 3 MeV, depending on the type of application.

Fig. 2 shows a typical hydrogen spectrum obtained for a thick Mylar sample using the ERD technique. Mylar contains 36 atom % hydrogen. The solid curve of Fig. 2 is a computer fit to the data (dashed curve) and allows not only the hydrogen concentration to be determined but also a depth profile to be obtained. The data are for $\alpha=\beta=10°$, for 2.5 MeV helium ions and the hydrogen energy scale of 0 to 0.9 MeV represents a depth of about 0.6 μm into the Mylar. The spectrum was acquired using a 10 nA helium beam for about 60 seconds, so data collection is very fast.

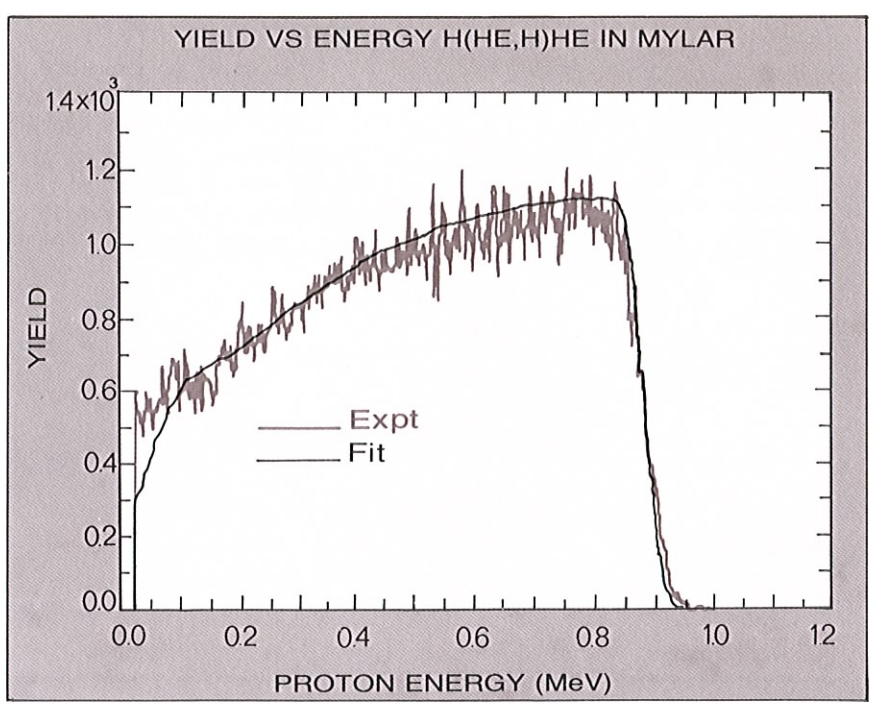

FIG. 2

Typically the depth resolution for the ERD technique is 400 to 800 A and the sensitivity is around 0.1 atom % for helium beams. Depths to about 2 μm into the sample may be analysed using the 3 MeV Van de Graaff accelerator.

TYPES OF SAMPLES

The three-dimensional manipulator shown in Fig. 1 of the Oxygen Analysis data sheet is also used for the hydrogen analysis. It is under vacuum and is only capable of handling solid targets from a few millimetres to several centimetres in diameter. Hydrogen is very mobile so care should be exercised that the analytical procedure does not alter the profiles obtained. To avoid this we may cool the targets with liquid nitrogen to freeze the hydrogen in place while taking the measurements. Samples measured so far include semiconductor silicon, obsidian glasses and a variety of polyesters.

APPLICATIONS

This type of analysis is a surface technique and is limited in depth to the range of the ion beam used in the sample, which is typically just a few micrometres.

An advantage of the ERD technique is that the hydrogen isotopes, ^{1}H, ^{2}D and ^{3}T, can be profiled simultaneously with 2 to 3 MeV helium beams. At a scattering angle of $(\alpha+\beta)=15°$ ^{1}H, ^{2}D and ^{3}T can be recoiled with sufficient energy to penetrate the Mylar foil with surface ^{2}D and ^{3}T both having a detected energy about twice that of surface ^{1}H. This type of capability is available for the measurement of hydrogen retention and isotopic exchange in first wall or coating materials subject to fusion reaction environments.

Other obvious applications are possible, for instance looking at hydrogen processes on metal surfaces or tracing hydrogen or deuterium movements in silicon or other important semiconductor materials.

TYPICAL COSTS AND AVAILABILITY

The cost to outside users of the accelerator time and technical support is subject to negotiation.

This analysis facility can be made available to Australian tertiary institutions through the Australian Institute of Nuclear Science and Engineering (AINSE) which supplies yearly grants to these institutions to use the Lucas Heights facilities.

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