

Environmental Research

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1110 Near Surface and Depth Profiling Analysis Facility (SIBA2)

XYZ Target Manipulator - UHV target chamber with charged particle detectors for depth profile measurements of light elements such as hydrogen, carbon and oxygen, using forward recoil and nuclear reaction techniques. This has a target positioning stage with precise 3D X, Y and Z movement. These are used for study of water turnover in desert lizards using O¹⁸ as a biological tracer, depth profiling of ion implanted species, metallised contacts on semiconductors near surface hydrogen profiling in solar cells. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

1120 Multiple Surface Analysis Facility (SIBA1)

Target chamber and data collection facility for simultaneous PIXE (proton-induced X-ray emission), PIGE (proton-induced gamma-ray emission) and RBS (Rutherford backscattering). Samples are measured in vacuum and measurement of samples is can also be automated. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE. These techniques allow bulk element determinations to the parts per million concentrations, and the depth profiling of surface layers. The range of analyses possible covers elements from hydrogen to uranium. Applications include pigment composition of paintings; studies of Aboriginal and Polynesian artefacts to identify the origin of samples and migratory history; composition of geological samples as an aid to mineral ore prospecting. This service includes spectrum evaluation and data interpretation.

1130 Aerosol Measurement Facility (SIBA1)

Determination of environmental pollutants by PIXE analysis of particulate matter on filter papers from air samples. A complete service is provided, including PIXE analysis using the STAR Tandedron facility, and data evaluation. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

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Description

The 10MV Tandem Accelerator was commissioned in 1991. It provides ion beams with energies in the range of approximately 5 to 100 MeV, depending on the ion species, with beam intensities up to a few microamps. Virtually any naturally occurring isotope can be produced as a beam, free from contamination by other elements, isotopes or molecular ion species. A range of experimental facilities and analytical services are under continual enhancement on this facility and are described in the next three sections.

Support Services and Facilities

Operation of facility, construction and arrangement of experimental equipment, data acquisition and analysis, sample preparation.

Applications

Accelerator mass spectrometry, including high precision C¹⁴ dating; studies of basic nuclear, atomic and solid state processes; high sensitivity elemental composition analysis; high resolution depth profiling. Heavy ion microprobe applications.

Availability

Accelerator time is shared with other users, and requests for use must be made well in advance. Availability is normally between 8am – 8pm, however, upon request, consideration will be given to operations outside these hours. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

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Description

The Ion Beam Analysis group has a variety of beamlines on the ANTARES Tandem Accelerator for the study of solid state, atomic and nuclear processes. Ion beams of almost all elemental isotopes can be produced and accelerated to high energies. Signals from target chamber charged particle, gamma and X-ray detectors are collected and sorted by a multi-parameter data acquisition system, and analysed off-line by graphical interface software permitting display and analysis of data in either one, two or three dimensions. The Tandem Accelerator, ancillary facilities and data acquisition system are computer controlled, permitting a variety of automatic irradiation and measurement cycles to be pre-programmed and a broad range of analysis methods to be applied to a range of problems.

Support Services and Facilities

Operation of facility, data acquisition and analysis, sample preparation.

Applications

Microanalysis of the first few microns of the surface region of materials by ion beam analysis can provide good elemental sensitivity and depth resolution. The quantitative determination and localisation of elements in modern high technology materials and devices such as semiconductors, optoelectronics and sensors, critically depends on the composition and interactions of multi-layer thin film coatings. The use of high energy, heavy ion beam techniques permits analysis of these structures, which are often impossible to characterise satisfactorily by other, low energy methods. Examples include metal-polymer, metal-ceramic adhesion studies, optical coatings on glass, high speed semiconductor memory chips and the characterisation of surface modified materials produced by plasma processing, chemical or ion implantation. The use of high energy light ion beams permits a wide range of nuclear reactions and techniques to be used for element specific studies, such as hydrogen and oxygen in corrosion, lithium in aluminium-lithium alloy materials, carbon from lubricants in wear, and nitrogen in nitrided cutting tools.

Availability

Accelerator time is shared with other users, and requests for time must be made well in advance. Availability is normally between 8am - 8pm, however, upon request, consideration will be given to operations outside these hours. The number of running days required to successfully complete each project should be discussed with the ANSTO contact scientist or collaborator before the project is submitted to AINSE.

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1310 Heavy Ion Time-Of-Flight Spectrometer and Surface Analysis Facility

Simultaneous element depth profiling of the composition of multi-layered thin films, and of the near surface region of bulk materials using high energy, heavy ion forward recoil spectrometry. A heavy ion beam incident on a target material ejects atoms from the surface region, which are subsequently mass identified by a measurement of particle energy and velocity. Mass separated particle yield vs energy profiles are obtained which are translated to concentration vs depth profiles. Applications include interfacial stoichiometry in semiconductor devices, solar cells, optical coatings and metallised polymers. Facilities include ultra high vacuum chamber (UHV) with XYZ target manipulator, cryogenic vacuum pumping, multiparameter data acquisition system and graphical analysis software and data interpretation. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

1320 Ion Beam Analysis Facility

Ion beams from protons to gold are available for materials analysis, nuclear and atomic physics studies. Ion beam energies up to 100 MeV are available, with atoms having either partially or fully stripped electron shells. Ion energy and charge state depends on ion species and accelerator terminal voltage. Facilities include UHV vacuum chamber with 3D XYZ target manipulator, ability to cool and heat targets during analysis, range of charge particle detectors, and gamma and X-ray spectrometers. Applications include measurement of carbon, nitrogen and oxygen by inelastic proton scattering, near surface profiling, hydrogen depth profiling and nuclear structure studies. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

1330 Ion Beam Irradiations

Facilities are available for high energy and heavy ion implantations, charged particle surface activations for wear and corrosion studies, and the production of some neutron deficient radioisotopes using (p,xn) reactions. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

1340 High Energy Heavy Ion Microprobe Analysis Facility

This microanalysis tool employing various IBA techniques for materials, environmental and geological analysis is available on the tandem accelerator. Microanalysis with spot sizes down to a few μm is readily available. With the heavy ion microprobe ions from hydrogen up to iodine can be focussed and used for analysis as well as ion irradiation. This allows the use of a wide range of ion beam techniques from Rutherford Back Scattering (RBS), Particle Induced X-Ray Emission (PIXE) to Elastic Recoil Detector Analysis (ERDA). This service includes spectrum evaluation and data interpretation. The number of running days required to successfully complete each project should be discussed with the ANSTO contact officer or collaborator before the project is submitted to AINSE.

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1500 – 1550 ANTARES AMS Facility

Description

Accelerator Mass Spectrometry (AMS) is an ultra-sensitive analytical technique based on the use of an ion accelerator as a powerful mass spectrometer. The element of interest is chemically separated from the original material and loaded as a target in the ion source of the tandem accelerator. The ion beam produced from it is accelerated and isotopically analysed. Selected isotopes are identified and counted individually with ion detectors. Isotopic concentrations at the level of 1 part in 10^{15} can be measured for long-lived radioisotopes such as ^{14}C , ^{10}Be , ^{26}Al , ^{129}I , ^{236}U and Pu isotopes, which have extensive applications as chronometers and tracers in a wide range of disciplines.

Only small quantities of the specific element are needed, often less than 1mg. The ANTARES AMS facility is designed for multi-isotope analysis with high precision and is being utilised to analyse the above isotopes in unknown samples from a variety of projects in quaternary science, global climate change, nuclear safeguards and other applications.

The AMS group provides assistance in sample selection and treatment, data interpretation, radiocarbon calibration and development of new applications of long-lived radioisotopes (in geomorphology, biomedicine and other disciplines).

Support Services and Facilities

The sample preparation section is an integral part of the AMS facility and includes three laboratories for ^{14}C sample preparation – a pre-treatment laboratory, a carbon dioxide/stable isotopes laboratory and a graphitisation/target pressing laboratory. Sample preparation for the cosmogenic isotopes (^{10}Be , ^{26}Al) is performed in two laboratories with a special dedicated laboratory for the actinide/ ^{129}I sample preparation.

Radiocarbon dating

Radiocarbon dating is performed on a variety of sample types, optimum sample sizes are listed in Table 1.

Table 1. Optimum sample sizes for AMS Radiocarbon analysis

Material	Quantity	Material	Quantity
Charcoal	10 – 50 mg	Bone ^(a)	1-2 g
Wood	20 – 30 mg	Paper, textiles	20 – 30 mg
Cellulose from wood	50-100 mg	Seeds, leaves	20 – 40 mg
Carbonates (shell, coral, stalagmites)	20 – 30 mg	Water	0.5 – 1 litre

^(a) - The sample size depends on the degree of preservation.

The facility to choose depends on the sample type, or the form in which you wish to send the sample. There is an additional charge for high precision analysis; this is also listed as a separate facility.

Choices for radiocarbon dating are:

1502 C-14 AMS analysis - graphite

To be used when you wish to pre-treat and process the sample yourself, and send the resulting graphite for analysis.

1504 C-14 AMS analysis - carbon dioxide

Carbon dioxide is sent for conversion to graphite and subsequent AMS analysis.

1506 C-14 AMS analysis - pre-treated

To be used when you wish to pre-treat the sample yourself, and send the cleaned sample for us to isolate and purify the carbon dioxide, convert it to graphite and perform AMS analysis.

1508 C-14 AMS analysis - standard treatment

This is for routine samples that require simple pre-treatment (e.g. the AAA treatment). Sample such as charcoal, wood, shell and corals, water, peat, sediment and fabrics are included as requiring standard treatments. We will pre-treat the samples, isolate the carbon dioxide and convert it to graphite prior to AMS measurement.

1510 C-14 AMS analysis - complex treatment

Samples needing isolation of pollen from sediments, collagen from bone and cellulose from tree-rings require a more extensive pre-treatment. Collagen from bone also includes stable isotope analysis to ascertain the quality of the bone and of the collagen isolated from the bone. These results are also reported with the final AMS measurement. Bone samples which are deemed to be too degraded for analysis will be returned without further processing.

1512 C-14 high precision analysis (<0.5% precision)

Requests for high precision measurements must also include this facility in addition to any of the above facilities.

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1514, 1545 – 1547 Stable Isotope Analysis

The AMS group also operates an Elemental Analyser / Isotope Ratio Mass Spectrometer which is routinely used to measure $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and C:N ratio, as part of the radiocarbon sample preparation and measurement process. Access to this instrument is available for stable isotope measurements ($\delta^{13}\text{C}$, $\delta^{15}\text{N}$) in a range of sample types (such as vegetation, soils, organic matter etc.). We require that samples submitted for these analyses are:

- suitable for measurement i.e. dry, finely ground and well homogenised
- %C and/or %N is provided; or
- %C and/or %N facility is requested if you are unable to perform these measurements.

Choices for stable isotope analysis are:

1514 Stable isotope, delta C13 analysis

1545 Stable isotope, delta N15 analysis

1546 Stable isotope, %C analysis

1547 Stable isotope, %N analysis

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1516-1528 Cosmogenic Isotope Dating

The longer lived isotopes of ^{10}Be and ^{26}Al are now used extensively for determining the exposure history of geomorphic formations and surfaces and applications to landscape evolution. Dating can be performed on quartz bearing rocks, sediments or ice. The facility to choose depends on the sample type and the isotope(s) of interest.

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1518 Be-10 target preparation, ancillary & AMS analysis - ice, snow, filters

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1530-1538 Heavy Isotope Analysis

The ANTARES AMS Facility also has the capability to analyse the heavier isotopes ^{129}I , ^{236}U and Pu isotopes.

Applications

The Actinide beamline on the ANTARES AMS Facility is used to analyse heavier long-lived radioisotopes with high sensitivity. ^{129}I , ^{236}U and Pu isotopes are routinely measured. These are of interest in nuclear monitoring and dating applications. The system can also be adapted to measure a range of other radioisotopes, with sensitivity of around 100,000 to 1 million atoms.

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1600 Environmental Radiochemistry - Geochronology

The facility specialises in low level measurement of radioactivity in environmental samples. Sensitive radioanalytical techniques have been developed to measure a range of radioisotopes including those in the natural uranium and thorium decay series to the lowest levels of radiometric detection. Sensitive radioanalytical instrumentations are used and dedicated to measure only low level activities to avoid cross contaminations. The facility has assisted numerous AINSE collaborators in ^{210}Pb and ^{137}Cs sediment dating by alpha and/or gamma spectrometry.

Applications

Applications are in the areas of Late Quaternary geochronology, sedimentology and biochronology.

1610 – Uranium/Thorium analysis by alpha spectrometry

Low level activities of uranium and thorium isotopes in environmental samples can be determined by alpha spectrometry. Sensitive radioanalytical methods have been developed to isolate uranium and thorium from environmental sample matrices. The activities of the uranium and thorium isotopes can then be quantified by alpha spectrometry. The disequilibrium of uranium and thorium isotopes in suitable environmental samples can be used in quaternary geochronology.

1620 – Pb-210 dating by alpha spectrometry

The geochronology of a sediment core can be determined by the ^{210}Pb dating method. The technique uses the decay of excess ^{210}Pb activity (half life = 22 years) to determine the rate of sediment accumulation. About 5 g of dried sample, from various depths of the sediment core, is required. The samples are processed to prepare ^{210}Po and ^{226}Ra alpha sources. The activities of these sources are determined by alpha spectrometry to calculate excess ^{210}Pb activities. This technique is more sensitive than gamma spectrometry, suitable when small quantity of sample is available for analysis. However, the method is destructive and other radionuclides such as ^{137}Cs will need to be analysed by a different technique such as gamma spectrometry. ^{137}Cs activities in sediment cores are analysed to verify ^{210}Pb chronologies.

1630 – Pb-210 dating by gamma spectrometry

^{210}Pb dating can be undertaken by gamma spectrometry technique. 20-50 g of dried sample is required. There is no radiochemical processing required with this technique. The samples are simply packed into petri dishes, left to equilibrate for 3 weeks and then counted on a High Purity Germanium (HP-Ge) gamma spectrometry system. Radionuclides such as ^{210}Pb , ^{226}Ra , ^{137}Cs , ^7Be , ^{228}Ra , ^{228}Th , ^{238}U and ^{40}K can be measured simultaneously. This technique is non-destructive.

1640 – Grain size analysis

It has been shown that preferential adsorption of radionuclides such as ^{137}Cs and ^{210}Pb to finer grain size can occur in sediment samples. For this reason, the grain size distribution of sediment samples for ^{210}Pb dating should be investigated. Grain size analysis is performed on an instrument which uses laser diffraction to determine the particle size distribution of solid materials. About 1 cm³ of wet sample is required to perform the analysis.

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1650 Environmental Chemistry - Tritium

The facility is dedicated to the determination of environmental levels of tritium.

Applications

Applications are in the areas of water resource management and planning, and tracing of groundwater flows. The laboratory is equipped with large and small volume distillation apparatus, 32 electrolysis units and 3 liquid scintillation counters (LSC).

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1670-1690 Radon/Radium

The atmospheric radioactivity laboratory is equipped with analytical instrumentation for measuring an extended range of radon and radon progeny concentrations and radon emanation and exhalation rates in gaseous, liquid, solid and mixed phase samples. The analytical laboratory instrumentation includes a radon calibration chamber, a radon rig, and liquid scintillation counter and gamma spectrometers. There is also field instrumentation for monitoring radon concentration in air and determining radon and thoron flux density.

Field instrumentation is a very important component of the radon group facilities. Instrumentation developed by ANSTO includes a series of radon detectors and a radon and thoron emanometer. The detectors have been designed to monitor radon concentration in air with a time resolution of less than one hour in harsh environments and without any maintenance for prolonged periods. The high sensitivity has a lower limit of detection of only 10 mBq m⁻³. The emanometer is used for determining radon and thoron flux density at environmental levels. Sampling time can be as low as one hour. The field instruments are supported and complemented by radon analytical facilities

- 1670 Radon/radium measurement**
- 1680 Radon/thoron emanometer**
- 1690 Radon/radium liquid scintillation counter**

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1700

SIMS - Secondary Ion Mass Spectrometry - Cameca IMS 5f

Located at the University of Western Sydney's Hawkesbury Campus, Richmond NSW.

The Cameca IMS 5f magnetic sector SIMS instrument is used for elemental surface analysis and depth profiling of a wide variety of materials. It can be operated in either microprobe or microscope mode enabling a unique combination of in-situ analysis, depth profiling and imaging. Analyses and images of masses between 1 – 500amu can be made in areas up to the size of 500 μ m x 500 μ m. Concentrations in the ppm/ppb range can be detected under favourable conditions. Depth profiling can be performed to depths of around 10 μ m.

The instrument is routinely used for isotopic analysis, studies of surface contaminants, imaging of materials and the analysis of trace and rare earth elements in samples. Samples are to be supplied suitable for mounting. Specimens should be less than 2.5cm in diameter and less than 1cm deep. Where possible the upper surface should be as flat as possible.

Support services and facilities

Sample preparation is a critical step in SIMS analysis and all samples must be fully prepared prior to arriving at UWS. It is strongly recommended that the SIMS Instrument Scientist is consulted for advice in this matter. The SIMS Instrument Scientist is available for consultation on matters relating to data interpretation following SIMS analysis.

Availability

The SIMS instrument has periods of down time while routine maintenance is performed. To maximise operational efficiency the instrument is operated in a single mode for scheduled periods. Accordingly, successful grant applicants will be allocated scheduled instrument time by the SIMS Instrument Scientist. The projects and instrument time required must be agreed with the SIMS Instrument Scientist prior to the submission of an application.

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Environmental Chemistry – Other Facilities

The Institute for Environmental Research maintains a range of equipment and facilities which are not unique to ANSTO. While ANSTO welcomes university researchers to use these facilities, AINSE as a general rule does not assist in providing the necessary funding support.

Facilities are available for trace element determination in the environment, the measurement of metal-ligand formation constants, dissociation constants, solid surface adsorption constants and pH-stat titrations with associated numerical analysis software.

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1810

Chemical analysis

The analytical laboratory provides a range of analytical services. It is equipped with sophisticated instrumentation such as quadrupole inductively coupled plasma - mass spectrometer (ICPMS); a simultaneous inductively coupled plasma atomic emission spectrometer (ICPAES); anion & cation chromatographs (IC) and isotope ratio mass spectrometer (IRMS).

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Environmental Radiotracer Facility

ANSTO's Institute for Environmental Research can use radioisotope tracer techniques to study the movement of fluids, particulates and contaminants in aquatic and terrestrial environments. ANSTO can apply radiotracers to study the fate and behaviour of water, contaminants and particulates in groundwater, rivers, estuaries and coasts with the objective of validating numerical transport models. Radiotracers may also be applied in field and laboratory environments to study the uptake and transfer of contaminants and nutrients by plants and aquatic organisms.

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Aquatic Monitoring

The aquatic monitoring capability in ANSTO's Institute for Environmental Research uses a range of divisional facilities as well as collaborative links with external researchers. The Geohydrosphere Program is capable of sampling and identifying biota including algae, macro-invertebrates and fish, measuring a variety of water quality parameters, and sampling for the detailed determination of radionuclides and general water chemistry. Univariate and multivariate statistical analyses are used to interpret the data and provide information on the relationships between biological and chemical parameters.

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Aqueous Radionuclide Geochemistry laboratory

The facility measures interactions between dissolved pollutants and solids such as soils, clays and barrier materials. Typical applications would be

- studies of pollutant uptake on harbour sediments by radiotracer techniques
- measurement of radionuclide distribution coefficients (K_d value) on soils, oxides and clays for radioactive waste disposal and water treatment studies

The use of specialist apparatus such as autotitration equipment and controlled atmosphere glovebox are available for use in these types of experiments as required.

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Description

Instrumental neutron activation analysis (INAA) is a very sensitive method of quantitative elemental analysis based on the nuclear activation of the chemical elements present in a sample. Activation is achieved by placing the sample in the neutron flux of the OPAL reactor, using the facilities described in the OPAL section of the AINSE User Guide. The specific activity of each radionuclide may be determined by measuring the characteristic gamma radiation emitted from the sample after it has been removed from the reactor. Two methods of standardisation are available, the relative (comparator) method and the k_0 -method. Around 65 elements can be measured by INAA. The irradiation time can be selected to optimise the detection limit for particular elements, making use of the short residence time (SRT) and long residence time (LRT) facilities, as indicated below.

Delayed neutron activation analysis (DNAA) can measure the uranium content of a sample at concentrations from 0.5 to 100,000 ppm. The technique determines the concentration of the isotope ^{235}U by measuring the rate of emission of delayed neutrons from the sample after it has been irradiated in the OPAL neutron flux.

Sample preparation

There are strict requirements on the handling and nature of samples that are to be irradiated in OPAL facilities. In some cases samples will need to be analysed by XRF prior to irradiation, for an additional fee. Samples can only be loaded into approved polythene irradiation containers at the Lucas Heights site by accredited ANSTO staff. Please contact ANSTO early in the process of project development to discuss target and canning aspects.

Samples should be dry, powdered and homogeneous. Samples for DNAA should be ground to less than 200 μm . Between 100 mg and 10 g of sample will be weighed into the irradiation containers that will be supplied by ANSTO. The actual amount of sample used will depend on the expected dose-rate from the sample at the end of the planned irradiation.

Sample storage and return

Unused samples will be stored for 6 months prior to disposal. If requested, this unused material can be returned at the client's cost.

In certain circumstances, it may be possible to return irradiated samples to the client, subject to ARPANSA regulations.

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A selection of elements that are best measured in the short residence time facility are shown in the following table. Detection limits (ppm) are also provided for guidance but they may depend on the bulk matrix of a sample, the elemental composition and the method of standardisation used.

Indicative detection limits (ppm) of selected elements:

Al	1000	Na	50
Ca	200	K	200
Ti	200	Br	1
V	10	In	1
Mn	0.1	I	5
Mg	1000	Ga	20
Ba	2		

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A selection of elements that are best measured in the long residence time facility are shown in the following table. Detection limits (ppm) are also provided as guidance but they may depend on the bulk matrix of a sample, the elemental composition and the method of standardisation used.

Indicative detection limits (ppm) of selected elements:

As	1	La	0.5
Sb	0.2	Ce	2
Cr	5	Sm	0.2
Fe	200	Eu	0.5
Sc	0.1	Yb	0.5
Th	0.5	Lu	0.2
U	2	Se	5
Zn	100	Au	1

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DNAA - uranium analysis

Subject to initial review by ANSTO, the uranium concentration of any dry, stable, non-corrosive powder may be determined using this method.

The precision of the analysis is 3% (one sigma) of the stated uranium concentration above 5 ppm and about 10% below 5 ppm.

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