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Research and measurement program at the ANTARES AMS facility

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Abstract

The ANTARES AMS facility is fully operational and supports a research program in environmental science, with emphasis on global climate change and nuclear safeguards. A measurement program for external projects involving Australian and overseas institutions is also carried out, mainly in Quaternary science studies.

1. Introduction

An Accelerator Mass Spectrometry (AMS) facility has been under development during the past six years based on the FN tandem accelerator ANTARES at the Lucas Heights Science and Technology Centre. The long-lived radioisotopes ^{14}C, ^{26}Al, ^{36}Cl, ^{10}Be and ^{129}I can presently be analysed, with the development of an AMS measurement capability for actinide isotopes under way. Having accomplished the task of transforming the old nuclear physics accelerator from Rutgers University into a world-class AMS spectrometer, the AMS group at ANSTO is now involved in developing a research program in global climate change and nuclear safeguards. External projects in archaeology, environmental science and biomedicine are also supported by the ANTARES facility. Most internal and external projects are based on collaborations with Australian universities, the Commonwealth Scientific and Industrial Research Organisation (CSIRO), Australian Antarctic Division (AAD) and other research agencies. Funding is provided mainly by the Australian Research Council (ARC), the Australian Institute of Nuclear Science and Engineering (AINSE), the National Greenhouse Advisory Committee (NGAC) and the Australian Safeguards Office (ASO). Collaborative research is being carried out with institutions from the European Union and Japan.

2. The ANTARES facility

2.1. AMS spectrometer

The ANTARES AMS spectrometer provides high precision and high throughput analyses of long-lived radioisotopes spanning a wide range of masses [1,2].

The key components of the AMS system are:

(i) a 59-sample high-intensity ion source, model 846B, High Voltage Engineering Europa [2];

(ii) a fast isotope switching system [3];

(iii) two AMS beamlines in the high-energy section, one dedicated to ^{14}C and other light radioisotopes [4] and one dedicated to ^{129}I and other medium-heavy radioisotopes [5].

The gas stripper incorporates a retractable tube and a recirculating turbomolecular pump in the terminal. The gas scavenged from the stripper box by the pump is returned to the stripper tube directly from the foreline and additional gas can be added from a reservoir using an externally controlled thermo-mechanical leak. A clear improvement in the beam transport and beam size was observed after the installation of the gas stripper, which is essential for high-precision AMS measurements and for the AMS analysis of ^{129}I and actinides. In particular, the beam diameter at the image of the high-energy 90° analysing magnet was reduced from 4 to 2 mm with a 100% transmission through the switching magnet to the final ion detector.

The second and third High Voltage accelerator tubes have been recently replaced with Dowlish spirally-inclined electrode tubes. The final Dowlish tube and a Pelletron charging system will be installed within the next 12 months.

2.2. The actinide beamline

A heavy-ion beamline has been designed that is expected to be capable of measuring rare heavy radionuclides, such as ^{236}U, $^{229,230}Th$ and ^{244}Pu, in natural samples with ultra-high sensitivity. An electrostatic quadrupole doublet immediately following the accelerator will provide mass independent focussing of the beam at an external gas stripper, where the stripping to higher charge states will allow rejection of molecular fragments having similar M/Q. Momentum and E/Q analysis will be performed

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with a new analysing magnet (mass-energy product = 250 MeV.amu and an electrostatic analyser (ESA).

The first component of this spectrometer, a 90° spherical ESA manufactured by Danfysik has been delivered and is being tested. This unit has a mean radius of 2.5 m, a plate separation of 25 mm, a nominal maximum rigidity of $E/Q = 7.6$ MV and an energy dispersion of 5000 in the image plane. The analyser has been voltage conditioned beyond ± 80 kV. The ESA has been installed on the 15° port of the switching magnet to assess its energy dispersive properties. A preliminary test of this device involved using it for ^{129}I measurements.

2.3. The chemistry laboratories

The laboratories for target preparation are an integral part of the AMS facility. To date these laboratories have concentrated on setting up the procedures for processing carbon samples for ^{14}C AMS measurement. Graphite targets are prepared by the reduction of carbon dioxide over an Fe catalyst using Zn as the reducing agent. Samples containing 0.2 mg or more of original carbon are processed routinely for radiocarbon analysis. The current ^{14}C chemistry background for 1 mg carbon is about 0.2–0.3% of modern carbon. With the increasing demand for measurement of extremely small samples, we are currently developing methods for the preparation of targets containing tens of micrograms of carbon. Several unknown samples with masses as low as 50 μg carbon have been recently analysed. The background due to combustion and graphitisation for 50 μg carbon samples is 2% of modern carbon. A dilution technique in which the sample is diluted with dead CO_2 has been investigated. Samples containing between 40–80 μg of modern carbon were diluted to 200–300 μg carbon. The precision of this method (dilution and measurement combined) ranged between 2–3%.

We are presently expanding the capabilities of our chemistry laboratories to encompass the preparation of ^{26}Al, ^{36}Cl, ^{10}Be and ^{14}C targets for in-situ studies. We are also setting up procedures for the extraction of ^{14}C from uranium minerals to study exotic radioactivity from nuclei in the trans-lead region.

The difficulties associated with the handling and digestion of iodine samples is well known. We are testing procedures for the extraction of iodine from a variety of environmental samples, including water, sediments, soils and biota.

A laboratory in a separate building is to be used to prepare ^{14}C samples for a biomedical tracer experiment, using the sealed-tube technique for combustion and graphitisation.

Sample processing to support AMS analysis at ANTARES is being carried out also at the Quaternary Dating Research Centre (Australian National University) and is being developed at other universities and research institutions. A system for the extraction of CO_2 from ice samples has been developed at the Division of Atmospheric Sciences of the CSIRO. An automated system for processing seawater for radiocarbon analysis has been recently developed at the Division of Oceanography of the CSIRO.

3. Quaternary science

A national research program in Quaternary science based on the AMS analysis of ^{14}C, ^{10}Be, ^{26}Al, ^{36}Cl and ^{129}I started at Lucas Heights in 1993. This program, funded by ARC and AINSE, involves collaboration with 32 Australian universities and AMS analysis of nearly 3000 samples from more than 100 projects covering a wide range of disciplines: archaeology, geomorphology, hydrology, palynology, glaciology and paleoclimatology. Some selected projects are described below to give the flavour of this research program.

3.1. Carbon cycle and palaeoclimatology

3.1.1. Biological uptake of atmospheric CO_2 in the Antarctic Ocean (with Antarctic CRC, University of Tasmania and Australian Antarctic Division)

The aim of this project is to assess the relative importance of air–sea exchange and deep convection as carbon sources to Antarctic oceanic phytoplankton. This is important in estimating the effective transfer of atmospheric CO_2 to the deep ocean via photosynthetic carbon fixation and organic matter settling. If most of the carbon utilised by the phytoplankton is actually derived from deep ocean waters, then little uptake of atmospheric carbon will occur despite the relatively high biological productivity of the Antarctic coastal margins. ^{14}C measurements are the method of choice because they can readily distinguish carbon from deep ocean and from the atmosphere.

^{14}C analyses of monthly seawater samples of dissolved inorganic carbon (DIC) from a nearshore Antarctic site show that surface water carbon is dominated by input from deep seawater, with little (less than 25%) re-equilibration with the post-bomb atmosphere. It also appears that complete replacement of surface waters by deep waters occurred several months after the formation of the winter ice cover.

3.1.2. Dating stick-nest rats (with University of New South Wales)

^{14}C is being used to give the essential framework for interpreting information locked in the ancient dwellings of stick-nest rats which are built in arid areas from local materials and preserve plant and animal material for up to 10000 a. The material found in nests from Western Australia, Northern Territory and South Australia includes bones and hair from extinct species and pollen from plants growing thousands of years ago in the arid centre of the

continent. AMS dating is being used to reconstruct the past distribution of Australia's arid zone mammals and provide much needed information about the last few thousand years.

3.1.3. AMS single-ring analysis of a late Pleistocene Tasmanian celery-top pine (with University of Sydney)

Ancient conifer logs exhumed from the bed and banks of the Stanley River in western Tasmania have been sampled for dendrochronology and atmospheric ^{14}C studies. They range in age from ~ 35 ka to the present. We recently completed a series of high precision ^{14}C measurements from 90 consecutive, single-ring samples from a celery-top pine log dating from ca. 12700 ^{14}C years BP. The rings were separated by careful shaving, then milled and pretreated to isolate alpha-cellulose before combustion to CO_2 and graphitisation. These data provide a short but precise high-resolution annual record of atmospheric ^{14}C when plotted against ring numbers providing insights into production variations and ocean CO_2 release during the last glacial transition.

3.1.4. Dendrochronology of tropical trees (with Monash University)

Dendrochronological studies, and hence dendroclimatic modelling, for the tropics is hindered by the lack of clear annual growth cycles in most tree species. However, teak (*Tectona grandis*) is unusual in that it is deciduous and annual tree-rings can be identified and validated by careful cross-dating. We have measured bomb pulse ^{14}C in sections of teak trees from India and Java [7]. These measurements show a very close correspondence with atmospheric measurements, demonstrating that teak is a good archive of atmospheric carbon. No shifts or evidence of diffusion is seen. Hence there is potential to use bomb pulse measurements to estimate growth rates for other tropical species with less clear ring structures.

3.2. Geomorphology and landscape evolution

Long-lived cosmogenic radionuclides produced in-situ in exposed rocks and surfaces can be used as geochronometers and tracers of various geochemical and geophysical surface modification processes. A measurement program of in-situ radionuclides relating to the Australian and Antarctic environments is being developed at ANSTO in collaboration with a number of universities.

3.2.1. Glacial history of the Northern Prince Charles Mountains, Antarctica (with University of New England)

The Antarctic ice sheets have a fundamental influence on oceanographic processes and climate. Yet their histories are not well understood. The Lambert glacier is bordered by the Prince Charles Mountains and drains more than 15% of the East Antarctic ice sheet. Its expansion and contraction, as evidenced by its glacial moraine deposits and landscape sculpturing, records its historical volume. Geomorphic evidence points to a dynamic, multiphase glacial history during the late Pliocene. In-situ exposure age dating using ^{10}Be and ^{26}Al of glacially cut surfaces and large boulders studding the deposits will enable an age estimate of past glacial movements.

3.2.2. Glacial history of a pleistocene ice cap on the central plateau of Tasmania (with University of Tasmania)

The central Tasmanian Plateau shows distinct signs of glacial action with a series of defined moraine stages. Field observations have located two glacial advances relating to the Last Glacial maximum, about 10–12 ka ago, and to the Penultimate glaciation over 100 ka ago. Exposure age dating, using ^{36}Cl, of these moraine boulders will be used to determine the limits of glacial activity on the plateau and modelling of small-scale alpine ice-caps.

3.2.3. ^{10}Be analysis of deep sand regoliths in coastal plains of south west Western Australia (with Murdoch University and University of Western Australia)

The Victorian Plateau region of Western Australia contains extensive upstanding coastal reliefs of sand regoliths overlying sandstone bedrock. The formation process of this landscape may be explained either through an internal chemical modification process (laterization of pre-existing rock) or contrastingly, a deposition process of external material onto bedrock. The time scales for these two processes are believed to be quite different. In-situ ^{10}Be profiled from surface to bedrock may be able to distinguish between the alternatives. A profile of ^{10}Be as a function of depth should follow the known attenuation slope of cosmic rays in rock if the regolith was formed via laterization and moreover, the bedrock sample should be void of ^{10}Be.

3.3. Australian archaeology

Several archaeological projects based on radiocarbon dating have been funded by ARC, AINSE and the Australian Institute for Aboriginal and Torres Strait Island Studies. These projects include detailed research into the probable antiquity of the rock art of Chillagoe and Laura, North Queensland, the Kimberley, Western Australia, and Olary District, South Australia. A variety of materials are being analysed, including pigments, oxalate minerals, silica coatings, plant fibres, carbonised plant matter, fatty acids, beeswax and mud-wasp nests. Different sample processing techniques are being explored in these studies including low-pressure plasma techniques and laser extraction methods. Intercomparisons between ^{14}C dating and other dating techniques such as thermoluminescence and optically stimulated luminescence are carried out within some of the aforementioned projects.

3.3.1. Olary district (with Flinders University and Arizona State University)

The Olary District, in northeast South Australia contains a large collection of painted and engraved rock art. The aim of this project is to understand the reliability of radiocarbon dating of petroglyphs. Samples for radiocarbon dating were obtained from organic matter deposited underneath the rock coating or trapped within the weathering rinds of the underlying rock. Replicate AMS analyses of the organic matter encapsulated by rock coatings showed that intra-motif variability can reach 35% for 20–30 ka old petroglyphs.

3.3.2. Chillagoe (with University of Queensland)

The Chillagoe region of North Queensland has been under intensive archaeological scrutiny since the early 1980's. To date, a series of excavations have revealed cultural deposits dating back to at least 26000 years BP, with dramatic increases in the quantities of archaeological materials recovered during the mid to late Holocene. These increases appear to signify important cultural and demographic changes in past Aboriginal social systems, probably including a major population increase. The geographic distribution of rock art conventions through time in northern Queensland are being investigated showing that the earliest rock art is relatively homogeneous. The later rock art — estimated to date to the last 3500 years or so by indirect dating methods — however, is geographically heterogeneous, indicating a regionalisation of artistic, and by implication social, networks around that time. In order to test the chronological model constructed via indirect evidence, the ANTARES laboratories are directly dating several charcoal paintings and drawings from Chillagoe. Our initial results date the Chillagoe pictographs to the last 800 years or so, in support of the existing model of a mid to late antiquity for most of the regionalised art of the region.

3.3.3. Kimberley (with University of New England and Australian National University)

The Kimberley rock art sequence is likely to be one of the longest and most complex in the world. On the basis of superimpositions and differential weathering, Grahame Walsh has constructed a very detailed rock art sequence of which the major phases include Pecked pits, Irregularly Infilled Animals, Bradshaws, Clawed Hand figures and Wandjinas [8]. This sequence depicts major changes in Aboriginal culture, ideology and local fauna over time. Fieldwork in the region started in 1994 with the aim of providing absolute dates for the Kimberley rock art sequence. Small samples of pigments, beeswax and associated mineral crusts have been collected. AMS dating of these samples has provided the first age estimates for the well-known Bradshaw painting style.

Mudwasps, which overlie or underlie Kimberley rock paintings, can be dated by Optically Stimulated Luminescence (OSL), providing minimum or maximum ages for rock paintings. We are providing AMS determinations for some of the same samples, thus allowing comparison between results from two very different dating techniques.

3.3.4. Mount Mulligan (with University of Queensland)

Ngarrabullgan or Mount Mulligan, located some 100 km northwest of Cairns, north Queensland, Australia, is a large table top mountain bordered by 300 m high cliffs along most of its periphery. A different vegetation can be noted on the top of Ngarrabullgan and in the savanna woodlands which surround the mountain. Ngarrabullgan Cave, on the top of Ngarrabullgan, is one of the earliest radiocarbon dated archaeological sites in Australia (37000 yr BP). The deposits at this site show very low erosion and a near-total absence of territorial vertebrate fauna. Our study shows that intensive use of the mountain started around 5000 yr BP, after 27 millennia of total abandonment. In this work we have also obtained the first paired ^{14}C/OSL determination for pre-30 ka archaeological deposits [9].

3.3.5. 30 ka record from 2 mm thick rock surface accretion (with James Cook University)

AMS was used to date the carbon bearing substances contained in a microstratigraphic sequence deposited on a boulder in a limestone rock shelter near Chillagoe, north Queensland. A time span of 30 ka was discovered in a rock surface accretion of about 2 mm. These results demonstrate that materials such as oxalate crusts and silica skins could provide new environmental archives for paleoclimate studies. This method was originally developed to identify ancient Aboriginal rock art concealed within encrustations.

4. Global climate change

The following projects on Global Climate Change have been funded by the National Greenhouse Advisory Committee.

4.1.1. Ice-core studies (with CSIRO, Atmospheric Division, ANU and Antarctic CRC)

Ice cores are providing the best source of preserved air from which to reconstruct levels of greenhouse gases over recent centuries to millennia. Ice cores from Law Dome, East Antarctica, characterised by high accumulation rates but minimal summer melting, provide an unparalleled time resolution through the Holocene and possibly beyond. In addition, air extracted from the firn permits direct comparison of entrapped trace gas concentrations with modern records. One of the problems is that recent CO_2 growth rate variations are difficult to interpret due to the smearing of ice-core signals induced by the diffusion of air in the firn. We recently succeeded in using the ^{14}C "bomb

spike'' to determine the age spread and age of CO_2 in Antarctic ice and firn [10].

Profiles of long-lived cosmogenic radionuclides, produced both naturally and anthropogenically, as a function of depth and thus age can provide vital information on past solar variability, production rate changes, and atmospheric transport and deposition mechanisms of radionuclides produced from the nuclear power industry. Analysis of the Law Dome Ice core for ^{10}Be, ^{36}Cl and ^{129}I will be a future development of this program.

4.1.2. Ventilation rates of the Southern Ocean (CSIRO, Oceanographic Division and Flinders University)

The aim of the project is to examine the role of the Southern Ocean as a source region for water masses, to determine its contribution toward the ventilation of the world ocean, and to quantify its performance as a sink for anthropogenically produced greenhouse gases. The database from oceanic field observations available for model validation purposes is sparse, and the role of the Southern Ocean within the global carbon cycle represents, to date, the largest unknown quantity in discussions of an anthropogenically enhanced greenhouse effect.

Water samples from locations within the Australian sector of the Southern Ocean were obtained to determine their ^{14}C content. The determination of the ^{14}C distribution as a function of depth and location is necessary to validate predictions obtained from the tracer model which was developed for the world ocean region south of 24°S. Subsequently, this model is applied to integrate the observed ^{14}C distribution in space and time, and to establish a ^{14}C budget for the Southern Ocean.

First ^{14}C AMS measurements have been obtained and are consistent with the observations made during the Geochemical Ocean Sections Studies (GEOSECS) programme conducted in the late 1970s for the southeast Indian Ocean [11].

5. Safeguards

As a result of the increasing interest in ^{129}I as an environmental tracer and monitor of nuclear operations, ^{129}I AMS measurements have been developed at ANTARES. We have been funded by the Australian Safeguards Office to develop an ^{129}I measurement capability and measure anthropogenic ^{129}I as part of an international program under the auspices of the IAEA. This has required the development of techniques to extract iodine from a variety of sample materials including water, sediments and biological materials. ^{129}I has been recently analysed in samples of waters and sediments collected by IAEA inspectors at various distances from a nuclear reprocessing plant. ^{129}I concentrations were in the range 1×10^7–2×10^8atoms/ml and 1×10^8–2×10^9 atoms/g.

Pu and U isotopes are introduced into the environment as the result of releases from nuclear activities. ICP-MS and decay counting have severe limitations in the detection of actinide isotopes. We are involved in a program supported by the Australian Safeguards Office to develop AMS techniques for the detection of actinides in environmental samples.

6. External measurement program

6.1. Italian artefacts and works of art (Italian Ministry of Cultural Heritage, Opificio delle Pietre Dure, Florence, Italy, University of Milan, University of Bologna, Italy)

Following agreements with the Italian Ministry of Cultural Heritage, a number of interesting archaeological artefacts and works of art are being dated at ANTARES. Some examples are briefly discussed in the following.

We dated the resin used to repair a terracotta cherub made by Donatello (1386–1466 AD) for the Annunciazione Cavalcanti (Cathedral of Florence, Italy). The ANSTO results for the glue, 1331–1429 AD (1σ), prove that the restoration had been performed during the lifetime of the artist himself, after damaging the statue in the kiln. Thermoluminescence (TL) analysis of the clay gives the same age range.

The *Carte d'Arborea* are a set of ancient codes and parchments from the 14th–15th century which appeared around 1845 in Italy. They are a unique source of information encompassing many centuries of history for Sardinia. The authenticity of these documents has been discussed by eminent historians for the last 150 years. In particular, in 1870, an international committee of historians stated that the *Carte d'Arborea* were a fake. Our ^{14}C analysis yields a calendar age 1409–1436 AD (1σ) for these documents.

The age of a Byzantine micromosaic from the museum of Sassoferato in Italy representing San Demetrius has puzzled the Italian historians for the last 300 years and our date, 1279 ± 26 yr AD (1σ), ruled out the original claim that this object had been made during the times of the Emperor Justinian, the First.

6.2. Dating of charcoal from prehistoric copper production in North-Tyrol, Austria (University of Innsbruck, Austria)

The copper ore deposits in the Austrian Alps rank among the classic regions for archaeometallurgical studies in Europe. With the ore from these deposits the Bronze Age metal market of Central Europe was supplied with copper. The Institute of Prehistory and of Mineralogy and Petrography of the University of Innsbruck, has recently started a systematic field survey in this area, with the aim to locate and date prehistoric copper mines and smelting sites and to reconstruct the technology used by the early

miners and metallurgists to extract the copper from the ore. Some sites have been chosen for archaeological excavations, which furnished, among other materials, charcoal from firesetting as well as from metallurgical processes. So far, ten samples of charcoal have been dated, yielding ages from 1400 to 200 BC. These results, together with the archaeological evidence, give first indications that the main period of copper production in the area under investigation is related to the "Urnenfelder culture" from about 1200 to 800 BC.

6.3. The earliest neolithic population of Friuli, Italy (University of Trento, Italy)

The Neolithisation of Friuli (north-eastern Italy) is evident from archaeological findings in more than 20 early Neolithic sites. They include chipped and polished stones and pottery. Little is known about the origin of the Neolithic peoples in this region and of the relationship between the last Mesolithic bands and the first farmers. Dating has been performed in the past with conventional radiocarbon methods on charcoal samples. A burial site with some human remains was found in 1994. The remains belonged to a 5–6 year old girl, buried together with a large quantity of shells. Preliminary dates obtained at ANTARES, both on the skull and the shells confirm a chronology of 6–8 ka BP for these remains. This is the first direct dating of human bones from the Italian Neolithic.

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