New horizons for archaeological science in Ireland

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Major QUB Investment Presents New Horizons for Archaeological Science in Ireland

P Gleeson, P. Reimer, M. Blaauw, E. Murphy and G. Barrett

A major recent investment has created a new cutting-edge facility with state-of-the-art equipment at Queen's University Belfast. The new Institute for Heritage and Environmental Science, funded by a £3m grant from the UK Research and Innovation and Arts and Humanities Research Council Capability for Collections scheme and World Class Labs Fund, complements a £2.1m equipment upgrade (including new accelerator mass spectrometer) for the ^{14}CHRONO Centre for Climate, the Environment and Chronology in 2021. This has created a globally unique, world-leading facility to support archaeological science on the island of Ireland and worldwide in a new decade. It places Archaeology and Palaeoecology at QUB at the centre of some of the most transformative technological developments and scientific applications in archaeological science in recent years, and underlines its prestigious history as a leader in this field over eight decades.

History of Archaeology and Palaeoecology at QUB

Archaeology at Queen's had its genesis in 1908, the year Queen's transitioned from College to University, since a decision had been made at this time to create a lectureship in Ancient History and Archaeology within the Classics Department. The post was first occupied by K. T. Frost (1909-14) who was tragically killed in Mons, Belgium, at the start of the First World War (Dunlop 2000). While Ian Richardson next took up the post (1926-30), it was the succeeding post holder, Oliver Davies (1932-48), working alongside Estyn Evans in Geography (1928-1968) who paved the way for the establishment of a Department of Archaeology. The then Vice Chancellor of Queen's, Sir David Keir, radically supported the designation of archaeology as a science rather than an arts discipline and archaeology at Queen's was destined to be the first archaeology department set within a Science Faculty in a UK University. Martyn Jope, who had an interest in both biochemistry and medieval archaeology, was appointed as the first lecturer in this new department in 1949. By the late 1960s, the Archaeology department located at 17 University Square had four members of lecturing staff, with a Conservation laboratory, jointly run with the Ulster Museum, and a Palaeoecology Laboratory (established 1968) devoted to the understanding and dating of past environments. Palaeoecology was founded in QUB with a Nuffield Foundation grant which enabled a Nuffield Quaternary Research Unit to be set up with a team of four, two botanists (Alan Smith and Michael Morrison), soils specialist V. B. Proudfoot, and Margaret Jope, who was a leading expert on faunal remains in archaeology, especially birds.

Alan Smith researched and taught at QUB from 1954, leading the Nuffield Quaternary Research Unit there, becoming Co-Director of the Palaeoecology Laboratory from 1964. One of the interests of the team was the potential provided by the peat and tree remains in Irish bogs for the study of climate history. Radiocarbon dating, then a relatively new technique, provided a means to date the enormously long sequences of pollen data, though far greater precision was required than it could then provide. Jope and his team made the case for the establishment of a radiocarbon laboratory in QUB that would focus on precision dating. This was achieved through dendrochronological dating of the long tree-ring sequences obtained from trees recovered from Irish bogs and elsewhere. A small but effective laboratory was created in garages behind the Department of Archaeology. This was led by Gordon Pearson, formerly a senior technician at Windscale (better known today as Sellafield Nuclear Power Station), and

standards of precision were obtained equal to those of the best radiocarbon laboratories in the world. At this time Belfast and Minze Stuiver's radiocarbon laboratory in Seattle were recognised as world leaders in precision dating and calibration. These initiatives enabled botanist Jonathon Pilcher, and subsequently Mike Baillie and David Brown, to develop Queen's as a global leader in high-precision dendrochronology (Addyman 2016). Jope retired from Queen's in 1981 and was succeeded as Head of Archaeology by Basil Wilson, a Classical archaeologist by training, who secured funding for the establishment of the Palaeoecology Centre on Fitzwilliam Street, which was just completed before his untimely death due to illness in 1986. The Centre included a Radiocarbon Laboratory which used the then state-of-the-art liquid scintillation method. In 1987 Archaeology moved from 17 University Square to the top floor of the Elmwood Building on Elmwood Avenue – just across a carpark from the Palaeoecology Centre. Under the leadership of Gerry McCormac, then Head of the School of Archaeology and Palaeoecology, the ground-breaking, pioneering and internationally renowned quality of the research undertaken by the Palaeoecology Centre earned QUB a Queen Elizabeth II Queen's Anniversary Award in 2000.

This distinguished history of pioneering research in archaeological science entered a new era in 2003, when the ^{14}CHRONO Centre for Climate, the Environment and Chronology was established through funding from Atlantic Philanthropies and Queen's University Belfast (SPURII). It saw a new building constructed as an extension to the Palaeoecology Centre for an Accelerator Mass Spectrometry (AMS) radiocarbon dating facility, as well as offices and laboratories for the Archaeology and Palaeoecology staff, at 42 Fitzwilliam Street, Belfast, still the home of Archaeology and Palaeoecology today. This AMS system was installed and tested in early 2007, with the laboratory identification UBA- henceforth used to designate samples analysed in Belfast by AMS. The first reported AMS ^{14}C date from the laboratory was a birch twig from Beldrigg, Co. Mayo (UBA-7591, 4732 ± 30 BP) as part of a project funded by the Royal Irish Academy (Warren 2009). Since then, it has been central to radiocarbon dating on the island of Ireland as well as globally, processing 2500-3000 samples a year for researchers working in the academic and commercial sectors of archaeology, environmental and climatic studies, and on samples from every continent. The Centre also continues to sponsor awards for radiocarbon dates through the Royal Irish Academy and the Irish Quaternary Association to support research specifically in Ireland.

As with all laboratories and apparatus, equipment that was state-of-the-art at its inception can quickly become superseded and require increasing maintenance. The AMS secured in 2004 began to near the end of its natural life from 2020 and required increasing amounts of downtime. Similarly, despite periods of sporadic investment and equipment renewal, other formerly state-of-the-art equipment for tephrochronology, elemental analysis and archaeomaterials research also required upgrades. Therefore, in 2021 the staff of Archaeology and Palaeoecology and the ^{14}CHRONO Centre set about addressing these issues, the long-term future of archaeological science at QUB, and the need for state-of-the-art facilities to support research on the island of Ireland and further afield. A series of targeted applications have now delivered an unprecedented level of investment to deliver a globally unique facility with state-of-the-art equipment and capabilities, which secures the long-term future of both archaeological science and radiocarbon dating at QUB.

New Investment

At the start of 2021, a team led by Dr Maarten Blaauw sought funding for the major upgrade of the AMS to a state-of-the-art IonPlus MICADAS, securing £2.1 million funding from the Department for Employment, Northern Ireland, and QUB. This new equipment from Ionplus has improved precision over our previous AMS, can handle smaller samples (down to 0.2 mg carbon), and uses around four times less energy. Automatisation of parts of the sample preparation process also enable enhanced reproducibility, and the more user-friendly equipment means that visiting researchers and research students are now able to perform parts of the measurement process themselves under guidance by ^{14}CHRONO technicians and researchers. Now housed at 42 Fitzwilliam Street, the MICADAS system is veritably table-top in size by comparison to the older AMS, which the building itself was built to house in 2007.

Besides radiocarbon measurements, the ^{14}CHRONO Centre has long provided world-leading tools to assist with interpreting radiocarbon dates and isotopes. The international IntCal group which produces and releases ratified calibration curves, a critical tool used by archaeologists and palaeoecologists worldwide to convert radiocarbon dates into calendar ages, has been led by ^{14}CHRONO's Prof. Paula Reimer from 2001 to 2020 (Reimer et al. 2020), while ^{14}CHRONO also hosts the on-line calibration tools Calib, CALIBomb and the Marine Radiocarbon Reservoir Correction database at calib.org. Alongside these, Dr. Maarten Blaauw maintains open-source age-modelling R packages such as *clam* (Blaauw 2010), *rbacon* (Blaauw and Christen 2011) and *rplum* (Aquino-Lopez et al. 2018).

The ^{14}CHRONO radiocarbon laboratory upgrade was thereafter complemented by a further £3 million awarded to QUB to fund a major upgrade of the Archaeology and Palaeoecology laboratories, and additional elements of the ^{14}CHRONO Centre, in order to help transform it into a world-leading institute in heritage and environmental science. This bid was led by Dr Patrick Gleeson and Prof. Paula Reimer, securing investment from the UK Research and Innovation (UKRI) was part of the Arts and Humanities Research Council's (AHRC) Capability for Collections (CapCo) fund. The CapCo fund represents a landmark investment in heritage science for the UK arts and humanities sector, aimed at building regional capacities and safeguarding the future of the galleries, libraries, archives and museums (GLAMS). As part of UKRI's World Class Labs funding scheme, CapCo was one element in a larger £213m investment for all disciplines from physical sciences to arts and humanities, made through eight research councils. The purpose of the funding scheme was to expand and upgrade existing research infrastructure to help UK researchers tackle major challenges. As such, our QUB bid was supported by colleagues in the National Museums of Northern Ireland, National Museum of Ireland, and the Historic Environment Division of Northern Ireland, in order to secure an open and accessible state-of-the-art facility for conservation, heritage and environmental science.

Overall, the investment creates leading facilities in isotope analysis, materials characterisation and elemental mapping, conservation and wider archaeological science, intended to support the heritage and environmental science sector of Northern Ireland and beyond. It underlines QUB's recent investment in this area through the establishment of the Heritage HUB @ QUB, and is aimed at now offering an all-island hub for collections-based research and archaeological science on the island of Ireland and further afield. This new suite of equipment supports research across all of the Faculties at QUB, supporting colleagues in Maths and Physics, Biological Sciences and the Arts, Humanities and Social Sciences, and allows Archaeology and Palaeoecology at QUB to keep pushing the boundaries of understanding in past and current human-environment dynamics, collections research and conservation. The new state-of-the-art

facility will have a globally unique cluster of equipment, technical capabilities and identity, ensuring that Archaeology and Palaeoecology at QUB retains its legacy as a world-leading heritage science pioneer.

New Equipment

In total, this £5.1 million investment represents a landmark for archaeological science at QUB, and some of the equipment secured for the facility includes:

- Ionplus MICADAS : AMS system for radiocarbon dating.

- Ionplus Gas Interface: This interface is used for radiocarbon dating very small samples (e.g,. just 6 fibres of a canvas), and facilitates minimal damage to collections when researching date, provenance or authenticity.

- ELEMENTAR PrecisION IRMS: Linking an IRMS with ^{14}CHRONO's recently developed ramped pyrolysis/pyroxidation system (RPO) enables live $\delta\,^{13}C$ readings, allowing better interpretation of the ^{14}C measurements of different temperature fractions for mortar dating and separation of preservatives from museum objects.

- SERCON HS2022 IRMS: An EA-IRMS for measuring C, N, S H and O isotope values, and fundamental to assessing date reliability via C/N ratios.

- ThermoFisher iCAP LA TQ ICP-MS: The triple quadrupole (TQ) inductively coupled plasma mass spectrometer facilitates a wider range of isotope characterisation (e.g.^{204}Pb &^{87}Sr/^{86}Sr, down to three decimal places). Interfacing with Ion Chromatography and Laser Ablation, to enable quantification of key elements (Fe, S and Ti) and Rare Earths, for source fingerprinting.

- Ortec Alpha Counters: Alpha counters facilitates ^{210}Pb dating of recent horizons in environmental samples (i.e. last 150-200 years).

- ThermoFisher ATR FT-IR: Fourier-transform infrared spectrometry (FTIR) is more generally used to explore and carry out organic/inorganic characterisation of archaeological artefacts or associated residues. More specifically, it will be used to measure the crystallinity index of cremated bone to select suitable candidates for dating, to characterise mortar or other samples, or to identify conservation or other material on artefacts/samples that can affect radiocarbon dates.

- JOEL JXA-iHP200F Electron Probe Micro Analyser (EPMA): The EPMA allows geochemical fingerprinting, and offers the only regional dedicated facility for the geochemical characterisation and provenancing of meteorites, rocks and minerals from natural history collections, and stone, ceramic, glass and glass glazes from archaeology collections (additionally, this item has an SEM-EDX).

- BRUKER XRF: Helium Purge M4 Tornado Micro-XRF: facilitates X-Ray Fluorescence with spatial resolution, particularly 2D distribution of elements on samples (artefact, geological sample, or corrosion) key for research and conservation of collections. A large chamber allows samples up to 5 kg and 30 cm long, allowing large areas to be measured quickly.

- BRUKER Handheld Tracer 5 pXRF: The Tracer 5 pXRF has a graphene window with higher transmission of X-rays throughout the energy spectrum to improve the transmission for light elements: this makes for better readings and more accurate, quicker data.

- BRUKER Portable 2D Micro-XRF ELIO: A fully portable Micro-XRF mapper, which permits 2D elemental mapping of very large and/or stationary specimens that cannot be brought into a lab or moved.

- HOLOGIC X-Ray: The UltraFocusXL detector allows analysis of substantial objects (e.g. human bones, metal objects) within a 50 cm by 50 cm chamber.

This newly acquired state-of-the-art equipment complements other facilities in DNA analysis, mass spectrometry, isotopes, digital recording, data digitisation, remote sensing and GIS, available elsewhere in the School of Natural and Built Environment and QUB, through the Institute for Global Food Security, ASSET Laboratory, Core Technology Units, and the Centres for Data Digitisation and Analysis, GIS and Geomatics, and Community Archaeology. With our partners and colleagues in the GLAMs and wider heritage sector, we hope to expand these facilities with additional investment in expertise and equipment in coming years, further securing a bright future for heritage, environmental and archaeological science at QUB.

The distinguished history of Archaeology and Palaeoecology and the ^{14}CHRONO Centre means that it has been at the forefront of some of the most exciting and impactful research of recent decades, as well as providing facilities to facilitate external research from colleagues further afield, advancing our understanding of Ireland and its place in the wider world. This includes many QUB-based and collaborative projects, including several major INSTAR-funded projects, particularly the *Early Medieval Archaeology Project* (led by Prof. Aidan O'Sullivan (UCD) and Dr Finbar McCormick (Kerr et al. 2015; McCormick et al. 2014); O'Sullivan et al. 2013 and the *Cultivating Societies Project* (led by Prof. Nicki Whitehouse focused on Neolithic agriculture, settlement and landscape in Ireland (Whitehouse et al. 2010; 2014);

Likewise, our facilities have supported major excavation and research projects stemming from infrastructure and development in recent years, including those with a focus on human osteoarchaeology, particularly the investigations of medieval burial grounds at Ballyhanna, Co. Donegal (McKenzie and Murphy 2018; McKenzie et al. 2015; 2020; 2022) and Ranelagh, Co. Roscommon (Delaney and Murphy in press), both funded by Transport Infrastructure Ireland and led by Prof. Eileen Murphy. Similarly, some of the most transformative and seminal discoveries of recent decades in Irish archaeology have involved samples processed in our laboratories at QUB, including the dating of a bear patella with cut marks from Alice and Gwendoline Cave forming the earliest evidence for human presence in Ireland, to date (Dowd and Carden 2016; first dated at the ^{14}CHRONO Centre (UBA-20194, 10,798 ± 71 BP) and calibrated to cal BP 12,860 – 12678 with IntCal20).

Ramped Pyroxidation

Continuing a long history of research and development of cutting-edge techniques for radiocarbon dating, ^{14}CHRONO is leading the way in the research and application of ramped

pyroxidation (RPO) (originally developed for application to environmental samples such as soils and sediments) to novel archaeological materials or those that are especially difficult to date using existing methods. This work is being pioneered by Dr. Gerard Barrett who, with colleagues, is currently exploring its application to a range of materials, including museum artefacts that have been heavily treated with preservatives/consolidants, as well as building mortars that are lime or carbonate-based. Recent work, in conjunction with staff of the National Museum of Ireland, has resulted in the successful dating of an early medieval wooden bowl, the *Cuillard Bowl* (Barrett et al. 2021); previously a reliable date was impossible due to the presence of preservatives from when the bowl was originally conserved. There has also been considerable progress in the dating of building mortars (Barrett et al. 2021), most notably, perhaps, with the discovery of a 'lost' Irish round tower in Derry. Developing upon this, with funding from the Royal Irish Academy, and in collaboration with the Historic Environment Division, the *Northern Irish Round Tower Project* (NIRT) is an on-going project examining the application of mortar dating and RPO to understand the chronology and lifespan of the medieval round towers of Northern Ireland.

New Horizons

We are delighted that this track-record of transformative and impactful research, whilst also supporting the wider ecology of research in archaeological and environmental science on the island of Ireland, has been recognised with renewed investment. This secures the future of archaeological science and the ^{14}CHRONO Centre at QUB to open up new horizons and exciting vistas for blue-sky thinking and frontier-led research into a new decade. In particular, the new Institute for Heritage and Environmental Science is intended to operate as an all-island facility, and to support researchers and practitioners outside QUB, in Northern Ireland, the Republic of Ireland and beyond. We are keen to facilitate this by continuing the long tradition of ^{14}CHRONO as an important element of the wider research infrastructure of our field through access, funded grant schemes, and collaborative research, including doctoral and post-doctoral work, in the coming years.

For queries about access, potential applications and further knowledge, please contact Dr Patrick Gleeson (p.gleeson@qub.ac.uk) or Dr Maarten Blaauw (m.blaauw@qub.ac.uk).

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Figures/Images

Figure 1: Jonathan Pilcher, Mike Baillie and Gordon Pearson by the original C14 dating machine (Image: B. Hartwell)

Figure 2: Original machine for C14 dating at Queen’s University Belfast (Image: B. Hartwell)

Figure 3: Paula Reimer and Gerry McCormac at the launch of the new Accelerated Mass Spectrometer in 2007 (Image: P. Reimer)

Figure 4: The SPURII-funded Accelerated Mass Spectrometer after installation in 2007 (Image: P. Reimer)

Figure 5: The Ionplus MICADAS after installation in 2021 at 42 Fitzwilliam Street. (Image: G. Barrett)

Figure 6: 42 Fitzwilliam Street, the home of Archaeology and Palaeoecology at QUB today. (Image: M. Blaauw)

Figure 7: New XRF suite in the Imaging Laboratory at 42 Fitzwilliam Street (Image: P Gleeson)

Figure 8: JOEL JXA-iHP200F Microprobe due for installation by at QUB by the end of 2022 (Image: J. Dalby, JOEL)

Figure 9: Ramped Pyroxidation (RPO) setup established by Gerard Barret at ^{14}CHRONO. (Image: G. Barrett)