

Institution: University of Glasgow (UofG)		
Unit of Assessment: 10 Mathematical Sciences		
Title of case study: Maintaining standards: Producing the reference materials underpinning all		
radiocarbon dating measurements		
Period when the underpinning research was undertaken: 2003-present		
Details of staff conducting the underpinning research from the submitting unit:		
Name(s):	Role(s) (e.g. job title):	Period(s) employed by
		submitting HEI:
Prof. Marian Scott	Professor of Environmental Statistics	1983-present
	Professor of Environmental	
Prof. Gordon Cook	Geochemistry	1978-present
Period when the claimed impact occurred: Pre-2013 to present		
Is this case study continued from a case study submitted in 2014? N		
1. Summary of the impact		

Each year, cultural and environmental organisations around the world use accelerator mass spectrometry (AMS) to determine the age of items containing organic (carbon-based) material, a process known as radiocarbon (¹⁴C) dating. Within the sector globally, >GBP17.5 million/annum is spent on procuring radiocarbon dates. UofG research has developed the extensive set of radiocarbon reference materials to ensure the consistency of ¹⁴C measurements made in laboratories around the world. UofG's research designed the experiments and developed the statistical models that allowed the formal characterisation of these unique reference materials. These are now recognised as the international standards, underpinning the laboratory accuracy and precision of radiocarbon dating in support of heritage and environmental investigations around the globe.

2. Underpinning research

The long half-life of radiocarbon has proved revolutionary in cultural and environmental heritage activities such as archaeology and forensic science, by facilitating measurement of the age of an artefact based on its radiocarbon content measured through a complex process. Quality assurance and quality control are vital aspects to ensure the accuracy and precision of any measured radiocarbon date. Prior to UofG research, no set of natural reference materials existed for the radiocarbon dating community. Our research has focussed on contributing natural reference materials, characterising them through the statistical design of global experiments and providing a full quantification of uncertainties: these reference materials now underpin global radiocarbon measurement [3.1, 3.4] and are fundamental to good laboratory practice.

UofG statistical research has led every step of the process in creating the radiocarbon reference materials, including: identifying plausible natural materials for testing: preparing and designing the testing protocol (with the UofG Radiocarbon Laboratory) through a series of statistically designed, hierarchical inter-comparisons (3 global inter-comparisons since 2000) [3.1, 3.2, 3.4]) involving laboratories based in more than 50 countries with each inter-comparison involving between 60 and 75 laboratories. On receipt of the radiocarbon results from the participating laboratories, UofG statistical researchers developed novel hierarchical random effects models to characterise each material. These take into account laboratory estimated variation and biases before estimation of the consensus age and associated uncertainty. UofG contribution to measurement standardisation is based on two aspects, the sampling and design of experiments to identify and characterise these reference materials and the full statistical uncertainty quantification associated with their measurement.

Using the database of the entire set of intercomparison results, statistical research based on random effects models has delivered a full accounting of uncertainty as a function of material and age (>45,000 years to modern) which has been used in the global calibration curve to specify *a priori* the variability [3.4, 3.5]. Each past inter-comparison— the fourth (FIRI, 1997–2002), fifth (VIRI, 2004–2008) and sixth (SIRI, 2013–2017) [3.6] —has expanded the repository of reference materials that are available for laboratories (on request) for quality assurance purposes. This unique database has allowed the UofG contribution to the Global international calibration curve (IntCal20) which is used to calibrate all radiocarbon measurements to a common, calendar timescale. During the calibration curve estimation, prior specification of sources of uncertainty are required (based on replicate measurements). UofG analysis of results from VIRI and SIRI were used to address prior uncertainties in the development of IntCal20 [3.5, 3.6].

UofG statistical research has been fundamental to the development of metrological principles and good practice for Radiocarbon laboratories and users. We have led the way in the development of statistical models for the complex data generated in laboratory inter-comparisons, and specifically how the material consensus value is evaluated with its associated uncertainty. These approaches have since been adopted in other cosmogenic isotope communities, including beryllium and aluminium, which are key in geological dating [CRONUS-EARTH 3.7]. UofG statistical research has demonstrated several key findings for end users such as archaeologists (in national museums, in rescue excavations, in cultural heritage), geologists, the antiquities and nuclear industry sectors [3.3]. Findings included:

- The evidence for significant laboratory biases and their magnitude
- That laboratory quoted errors are frequently underestimated, and how they should be adjusted to be realistic using error multipliers
- The statistical quantification of uncertainty, due to both natural materials and laboratory procedures [3.1, 3.2]

3. References to the research

Outputs (include DOIs or ePrints link):

- 3.1 Scott, E.M. (ed.), (2003). The third international radiocarbon inter-comparison (TIRI) and the fourth international radiocarbon inter-comparison (FIRI) 1990–2002: results, analyses, and conclusions, Radiocarbon, 45 (2), 135–408. https://www.cambridge.org/core/journals/radiocarbon/issue/CCE1D0E6A60A20AD87F53C47
- <u>D74EA92A</u> 3.2 Scott, E.M., Cook, G.T. and Naysmith, P. (2010) <u>The fifth international radiocarbon</u> <u>intercomparison (VIRI): an assessment of laboratory performance in stage 3</u>. Radiocarbon 52,
- intercomparison (VIRI): an assessment of laboratory performance in stage 3. Radiocarbon 52, 859–865. doi:10.1017/S003382220004594X.
- 3.3 Scott, E.M., Cook, G.T., Naysmith, P. (2017). <u>Should archaeologists care about 14C inter-</u> <u>comparisons? Why? A summary report on SIRI</u>. Radiocarbon, 59(5), 1589–1596. <u>doi:</u> <u>10.1017/RDC.2017.12</u>.
- 3.4 * Scott, E.M, Naysmith, P. and Cook, G.T. (2018) <u>Why do we need 14C inter-comparisons?:</u> <u>The Glasgow 14C inter-comparison series</u>, a reflection over 30 years. Quaternary Geochronology, 43, pp. 72–82. (doi:10.1016/j.quageo.2017.08.001)
- 3.5 Scott, E.M., Cook, G.T., Naysmith, P. and Staff, R. (2019) <u>Learning from the wood samples</u> in ICS, TIRI, FIRI, VIRI and SIRI. *Radiocarbon*, 61(5), 1293–1304. doi:10.1017/RDC.2019.12
- 3.6 * Heaton, T.J., Blaauw, M., Blackwell, P.G., Bronk Ramsey, C., Reimer, P.J. and Scott, E.M. (2020) The IntCal20 approach to radiocarbon calibration curve construction: a new methodology using Bayesian splines and errors-in-variables. Radiocarbon, <u>doi:</u> <u>10.1017/RDC.2020.46</u>



- 3.7 Jull, A.J.T., Scott, E.M. and Bierman, P. (2015) The CRONUS–Earth inter-comparison for cosmogenic isotope analysis. Quaternary Geochronology, 26, pp. 3–10. http://eprints.gla.ac.uk/229024/
- * = best indicators of research quality

4. Details of the impact

There are approximately 100 radiocarbon laboratories worldwide, each of which relies on fully characterised reference materials to quality assure the >50,000 radiocarbon determinations made annually. Radiocarbon laboratories globally operate a combined model of commercial contract work and applied research and development [5.1-5.4]. The reputation of this global community of laboratories is dependent on the use of the unique certified reference materials stemming from UofG research. Over GBP17.5 million worth of radiocarbon dating is commissioned globally each year, with many end-users working in rescue archaeology, geology (tracing gaseous leaks and hazards), environmental sciences such as tracing ocean currents, in the nuclear sector, and in forensic and material sciences. The UofG-led quality assurance research programme provides the underpinning certified reference materials for all of these analyses globally. These reference materials are key to ensuring measurement consistency and quality across all 100 labs worldwide and their commercial clients. Impact has been delivered in 4 key areas, detailed below.

1. Changing professional laboratory practice

UofG statistical research is recognised as providing global leadership in the design and organisation of radiocarbon inter-comparisons [5.1, 5.2], enabling standardisation of quality assurance and quality control processes around the world. *"Without exaggeration, the inter-comparison exercises that you have coordinated have been essential to the Quality Assurance /Control process in our (Woods Hole Oceanographic Institution) laboratory*". [5.3].

For large AMS laboratories such as the Keck AMS Facility at UC Irvine "The major impact of the Glasgow-based intercomparison studies for our laboratory has been that they provide a comprehensive set of internationally recognized standard materials ... [and] represent a unique resource for testing all stages of radiocarbon sample preparation and measurement including the initial chemical pretreatment" [5.4]. This laboratory undertakes in excess of 10,000 research unknowns per annum (two thirds of which are commercial samples, including 1,000 samples for the US National Oceanic and Atmospheric Administration's global program to monitor CO₂ sources and sinks).

Each UofG reference material is reported with a certified consensus value and uncertainty, evaluated using statistical models developed by UofG [3.1–3.2; 3.5–3.6], allowing each individual laboratory to assess their ongoing performance [5.3], and test any new laboratory developments. This delivers improved laboratory precision and accuracy for their commercial customers [5.4, 5.5]. "Any small interlaboratory offsets that may exist are no longer hidden in the scatter of less precise data, and distinguishing these effects from real regional 14C differences is becoming a significant concern" [Keck AMS Facility, 5.4]. Participation in the laboratory intercomparisons is used to demonstrate that the lab results meet international norms for compliance with national regulators [5.2;5.6]. The Director of the US National Science Foundation AMS Laboratory states: "It is important to have a continuous updating of the intercomparison materials, to try to test the new types of machines currently being developed (such as) gasion source AMS machines" [5.2] under development for the commercial market.

The UofG research has also been adopted by other communities in the characterisation of their reference materials. The basic statistical model [3.1] is used by the International Atomic Energy Agency in characterising their Reference Materials for Radionuclides [5.7], while the CRONUS intercomparison materials [3.7] for the study of cosmogenic ¹⁴C, ¹⁰Be and ²⁶Al [5.2] are used in geological hazard studies of volcanoes and earthquakes.

2. Archaeological Calibration

The radiocarbon community and all users use an internationally accepted and recognised calibration curve, which translates the measured radiocarbon age and its uncertainty to a calendar (or more specifically a range of calendar) ages. A major revision of the calibration curve was published in 2020 (IntCal20) [3.6], and in this revision, results from three of the UofG intercomparisons have been used to inform key statistical model parameters (prior specifications of variation) to produce the global calibration curve and uncertainty estimates [5.1].

3. Cultural heritage

Directly and indirectly, all users of ¹⁴C dates are impacted by this work, as it underpins the precision and accuracy of ¹⁴C dating. Radiocarbon dating is widely used in archaeological investigations. Historic England's National Head of Research stated: *"[T]he intercomparisons and the reference standards ... have enabled the construction of robust, reliable and readily comparable chronologies, ensuring clear value for money for the development industry which funds the great majority of archaeological investigation in England (a market currently worth an estimated £200M annually)"* [5.8]. The value of these studies to the field of archaeology has been acknowledged by Historic England through the provision of both knownage material and financial support for both VIRI and SIRI [5.8]. Radiocarbon dating has had a significant impact in changing our understanding of human history. The Principal Archaeological Research Curator for National Museums of Scotland states: *"The shocking discrepancy between the dates for a cemetery population obtained by the laboratories was a wake-up call to the whole audience about the necessity for rigorous inter-laboratory comparisons and the maintenance of the highest standards at all stages of radiocarbon dating"* [2017 Society for American Archaeology conference in Vancouver, 5.5)

4. Forensics and materials science

The world-leading analytical accuracy in bone dating, evidenced through these intercomparisons, of the UofG Radiocarbon Laboratory, has delivered critical age data on over 280 unidentified human remains [5.9] for forensic investigations in jurisdictions throughout UK, with international work in the Falkland Islands and Republic of Ireland since 2014. In Ireland our data provided significant assistance to the investigation into deaths of children at Mother-and-Baby homes from 1920s–1960s [5.10]). The research facilitates investigative decision-making and criminal convictions, reduces police time wastage and financial expenditure; and helps alleviate emotional trauma for victims' families by providing vital information needed for closure. This global reference material research has also underpinned the development of a specific whisky radiocarbon dating technique by the UofG Radiocarbon Laboratory [5.11] being used to authenticate claims of age.

5. Sources to corroborate the impact [PDFs made available]

- 5.1. Testimonial, International Steering Committee of the IntCal group
- 5.2. Testimonial, NSF AMS Laboratory, Arizona
- 5.3. Testimonial, National Ocean Sciences AMS Facility, Woods Hole Oceanographic Institution



- 5.4. Testimonial, Keck AMS facility, UC Irvine
- 5.5. Testimony, National Museums of Scotland
- 5.6. Testimonial, Ruđer Bošković Institute.
- 5.7. IAEA Reference Control Materials for 14C measurements. RS_IAEA-C1 to IAEA-C9.Rev.01 / 2014-03-24
- 5.8. Testimonial, Historic England
- 5.9. Database of police reports available on request from HEI
- 5.10. 5th Interim Report from <u>Mother and Baby Homes Commission of Investigation for Irish</u> <u>Government.</u>
- 5.11. Testimonial, Bonhams Auctioneers.