Radiocarbon dating in the 35-50 ka time range

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There are three primary sources of laboratory error in all ^{14}C analyses, regardless of sample age, size, or material type (organic or inorganic): (1) counting and statistical uncertainties associated with accelerator mass spectrometry (AMS) measurements, (2) incomplete removal of secondary carbon from the sample matrix, and (3) introduction of contaminant CO_2 during the extraction and/or graphitization of the sample. Errors associated with AMS measurements are typically quite small and largely unavoidable. At the University of Arizona's Desert Laboratory, we are working to minimize the impact of the latter two sources of error through the use of a more aggressive pretreatment technique, stepped combustion of samples, and a modified CO_2 extraction system based on a design by Michael Bird and co-workers at Australia National University.

The main obstacle to accurate ^{14}C dating in this time range is incomplete removal of secondary carbon, particularly humic acids. Humic acids are formed by the decay of organic material, are soluble in water, and therefore are present in nearly all ground- and surface-water systems. Humic acids sorb to primary organic material (living or dead) during transport and, because they are usually younger than the host material, their complete removal is critical for producing reliable ^{14}C dates for very old (>35 ka) samples. Even small amounts (<1%) of contamination makes very old samples appear substantially younger, producing a clustering of contaminated ^{14}C dates between 35 and 45 ^{14}C ka, which geochronologists variably refer to as the "black hole" of radiocarbon dating, the "^{14}C event horizon", and the "^{14}C barrier".

Standard pretreatment techniques (acid-base-acid or ABA) are effective at removing most secondary humic acids and are usually adequate for young samples. However, older samples must approach absolute purity in order to yield reliable ^{14}C ages. A new pretreatment protocol, known as acid-base-oxidation stepped-combustion (or ABOX-SC), has recently been developed that is more effective at removing secondary humic acids than the ABA treatment. The ABOX-SC treatment includes the standard ABA steps followed by strong chemical oxidation of non-elemental carbon, which leaves behind only elemental carbon (charcoal) residue. Samples are then subjected to a stepped combustion procedure in which any remaining labile organic matter (humic acids and other non-charcoal plant residue) is removed during the first combustion step and discarded, and CO_2 produced from the higher temperature steps is retained for ^{14}C dating. The use of the ABOX-SC treatment, in conjunction with a dedicated, low-level ^{14}C extraction system, appears to have pushed back the upper limit of reliable ^{14}C measurements to ~55 ka – a significant improvement over the current limit of ~35-40 ka. At the Desert Laboratory, we have recently designed and constructed a dedicated low-level ^{14}C extraction and graphitization system to confirm this upper limit, and to conduct ^{14}C dating in the 35-50 ka time range. While we anticipate our initial research efforts will be directed toward Old World archeological problems, we welcome the opportunity for interlaboratory comparison and calibration as part of the INQUA Drylands Dating Project, as well as future collaboration in arid lands, paleoclimate, and geochronologic research.