The Erlangen AMS-facility: status, applications and the prospect of ^{14}C measurements of microgram samples

Wolfgang Kretschmer

Physikalisches Institut, Universität Erlangen, Germany

The Erlangen AMS facility consists of a negative ion source, an injection spectrometer, a 6 MV EN tandem accelerator, an analyzing spectrometer and a heavy ion detector. Negative ions are generated from solid or gas samples in a hybrid sputter ion source from NEC, pre-accelerated to 50 keV and injected into the accelerator by a combination of a 90° electrostatic deflector and a 90° magnet with fast isotope switching for the sequential injection of ^{14}C, ^{13}C and ^{12}C ions. At 5 MV terminal voltage the most abundant positive charge state is 4+, which is analyzed in a mass spectrometer consisting of a 15° electrostatic deflector and a 55° magnet. Finally the stable ions ^{13}C and ^{12}C are measured in Faraday cups and ^{14}C is detected in a ΔE-E gas detector, which serves as a last filter for the remaining background. The beam transport between injection- and analyzing magnet is completely performed by electrostatic devices, which ensures identical ion optics for the different carbon isotopes. The machine background obtained from unprocessed graphite samples is 0.02 percent modern carbon (pMC) corresponding to an apparent age of about 69.400 years.

The conversion of raw samples to sputter targets suitable for an efficient formation of negative carbon ions usually involves three steps. First, the samples are chemically pre-treated to remove carbon compounds which are not representative for the age, e.g. carbonates and humic acids in soil or conservation material in artefacts. Second, the remaining material with a typical carbon content of about 1 mg is oxidized to CO_2, which is performed at Erlangen in an elemental analyzer (EA), followed by a stable mass spectrometer for precision measurement of $\delta^{13}C$ from 1% of the CO_2. Third, CO_2 is catalytically reduced to carbon at high temperature. The use of an EA has the big advantage that both combustion and reduction are connected in a closed vacuum line with minimum contamination from outside and that both processes can be performed in a fully automated and time saving way. For a typical 1 mg sample the background from procession and machine is about 0.08 pMC corresponding to an apparent age of 57.300 years. To extend the range of applications beyond our standard samples (charcoal, soil, pollen, bones, mussels) attempts have been made for the radiocarbon dating of iron artefacts and lime mortar, which involves different pre-treatment techniques.

Radiocarbon dating of ancient iron artefacts is possible due to their slight carbon content (0.1-3.5%) that originates from the iron smelting process, which has been powered by charcoal until about the 18th century. Two alternative methods have been tested, direct sputtering from the pre-cleaned sample and the extraction of carbon via HCl. Both approaches give reliable results, but direct sputtering is only reasonable for samples with high carbon content, since otherwise the source current is too small. Radiocarbon dating of lime mortar is often done by using pieces of wood or charcoal incorporated in the mortar. But, since origin and age of the wood at the time of incorporation is unclear, another method was tested using calcium carbonate produced from slaked lime and the CO_2 from the air during the setting of mortar. Problem with this method is the possible contamination of lime mortar with old calcium carbonate from other sources. Separation of the different carbonates seems to be possible due to their different microscopic structure and their different reaction times with phosphoric acid used to produce CO_2 for AMS measurements. First results for both methods are promising and will be discussed. Some interesting archaeological applications of AMS will be shown, e.g. the ^{14}C dating of a “Persian mummy”.

For some applications especially in environmental studies the amount of carbon for radiocarbon measurements is extremely low even if a large amount of natural sample (rain water, soil, air) is collected. Therefore an extension of the carbon mass range down to microgram is highly desirable, but this involves new and elaborate techniques, since with the normal solid targets the ^{14}C-background rises roughly inversely proportional to the carbon mass. For a carbon mass less than 100 μg the EA is directly coupled to the gas ion source via a fully automated gas handling system, thus avoiding the usual reduction to carbon and pressing the iron-carbon mixture into target holders. With this method calibration samples with carbon mass less than 5 μg have been measured with reasonable results. As an application the origin of trichloroacetic acid (TCA) in Bavarian forest soil has been investigated via their ^{14}C content. The soil has been chemically pre-treated for an injection into a two-dimensional preparative gas chromatograph (GC), resulting in a well separated TCA peak containing a carbon mass of 1.5 μg, the blank sample with the same chemical procedure and GC separation, but without soil, had a carbon mass of 0.21 μg. The ^{14}C content for the soil sample of 2.2 ± 0.2 pMC indicates anthropogenic origin of TCA in forest soil. We are currently working on a direct coupling of GC, EA and AMS facility to minimize any carbon contamination.