A Long Road: from Greenland to the Lab in Zurich

Conclusions can be drawn about past environmental changes from telltale footprints left in environmental records. We are following one such hot lead in our investigations of the 3-km-long Greenland ice core. Layer for layer, the ice is examined to determine the concentrations of the radionuclide beryllium-10 (^{10}Be). From such data it is possible to obtain information about past climate changes. About 10,000 ice samples have been prepared by the EAWAG laboratory in recent years for measurement in the ETHZ/PSI accelerator mass spectrometer.

Each year, rain and snow transport around 1 million atoms of the radionuclide beryllium-10 (^{10}Be) from the atmosphere on to each square cm of the earth's surface. Some of these ^{10}Be atoms are frozen, layer by layer, into the polar ice. Taking the global annual mean precipitation to be 1 m, this means that 1 kg of ice contains approximately 10 million ^{10}Be atoms. At first glance this would appear to be many. 10 million ^{10}Be atoms, though, weigh just 10^{-15} g, the equivalent of a single raindrop in Lake Constance. To detect such a low concentration is a challenge to analytical science. To keep pace with the development of the accelerator mass spectrometer, over the last 15 years EAWAG has developed an elegant sample preparation method (see box). This article provides an insight into the routine work carried out in our laboratory and describes how ^{10}Be atoms are extracted from the ice core and prepared for measurement in the ETHZ/PSI accelerator mass spectrometer at Hönggerberg, Zurich.

From the Arctic to the EAWAG Laboratory

The ice cores from Greenland (55-cm-long sections, maximum a quarter of the total cross section) are sealed in plastic bags and packed in styrofoam boxes before being sent to Dübendorf, where they are stored in a refrigerated room at –20 °C until required for preparation (Photo 1). Some of the ice samples are cut to smaller size with a bandsaw (Photo 2) to obtain a higher temporal resolution. Prior to the actual preparation procedure, each sample is washed with high purity water to remove any residual bore fluid and other external contaminants. Finally, the ice samples are placed in melting trays, weighed, and an exactly known amount of ^{9}Be carrier added (Photo 3). The ice is then melted in a conventional microwave oven (Photo 4) and afterwards passed drop by drop through a cation exchange column (Photo 5), which retains the beryllium ions. The cation resin with the beryllium ions can be easily stored for months till the next preparation stage.

In the next step, the beryllium ions are eluted out of the cation resin with acid, precipitated out with ammonia as beryllium hydroxide [$Be(OH)_2$], separated from the solution, and redissolved in acid. For Be to deliver negative ions to the ion source of the accelerator mass spectrometer, it needs to be laced with a conducting metal. We use silver, added as silver nitrate solution. Silver and beryllium now precipitate out together in hydroxide form. For the spectrometric measurement, the $Be(OH)_2$ must now be oxidized. For this, the samples are dried briefly under a UV lamp and put into a muffle kiln (Photo 6), where, at 850 °C, $Be(OH)_2$ oxidizes to BeO within 2 hours. Finally, the samples are pressed into a small hole of 1 mm diameter in the copper target (Photo 7). After about 24 hours, the beryllium samples are ready at last (Photo 8) for measurement in the accelerator mass spectrometer (Photos 9 and 10).

From EAWAG to the Lab at Hönggerberg

The isotope ratio in our samples is extremely low – on the order of 10^{-13}. This is outside the range of detection of a conventional

Extraction and Detection of ^{10}Be

Traditionally, radionuclides are detected by means of their radioactive decay. However, this method is only of use if the radionuclide being investigated has a sufficiently short half-life. Within a reasonable measuring period – several days to several weeks at most – enough atoms have to decay so that they can be detected without excessive measuring error. It is precisely here that the difficulty lies with ^{10}Be. Its half-life of 1.5 million years is far too long. Per year, only about 5 ^{10}Be atoms of the approximately 10 million atoms of ^{10}Be present in a kilogram of ice undergo radioactive decay. The detection of ^{10}Be by means of its radioactive decay is therefore not a viable technique.

The mass spectrometer offers an alternative method. This instrument exploits the fact that most elements are found in a range of isotopes of different masses. Beryllium is no exception. Along with the radioactive isotope ^{10}Be, with an atomic mass of 10, there is also the lighter, non-radioactive isotope ^{9}Be, with an atomic mass of 9. Mass spectrometry can determine the ratio of two isotopes – in our case ^{10}Be/^{9}Be – so that the number of ^{10}Be atoms can be calculated. Since the ice samples contain only tiny trace amounts of the ^{9}Be isotope, a known quantity of this isotope (typically 0.2 mg) must be added for the measurement. The added ^{9}Be acts as a so-called carrier and ensures that during the chemical extraction, the few ^{10}Be atoms present in the ice are also extracted.