Extraction of gases and dissolved and particulate matter from ice in deep boreholes

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Abstract. An extraction probe has been developed which allows vacuum melting of several tons of ice in boreholes down to a depth of 400 m. It mainly consists of an electrical heater and a rubber packer to isolate the section of the borehole in which the extraction takes place. During melting the evolving gases are pumped and collected continuously. The meltwater can be circulated through ion exchange resins or a filter, for collection of dissolved and particulate matter. From the gas samples CO_2 is extracted for ¹⁴C-dating, Ar for ³⁹Ar-dating. From the meltwater the ³²Si activity is measured. Pollen and cosmic dust studies can be performed on filter samples.

L'extraction des gaz, des substances dissoutes et des particules de la glace dans des forages profonds

Résumé. Nous avons développé une sonde d'extraction permettant de fondre sous vide plusieurs tonnes de glace dans des forages d'une profondeur qui peut atteindre 400 m. Elle comprend essentiellement un système de chauffage électrique aussi qu'un joint d'étanchéité qui isole la portion du trou de sonde dans laquelle on fait l'extraction. Pendant la fonte, les gaz dégasés sont aspirés et recueillis de façon continue. Il est également possible de recueillir certaines substances dissoutes en faisant circuler l'eau de fonte à travers des résines échangeuses d'ion ou à travers un simple filtre. Des échantillons de gaz on extrait le $CO₂$ et l'argon pour datation par ¹⁴C et ³⁹Ar. L'activité du ³²Si est mesurée à partir de l'eau de fonte. L'analyse des dépôts sur le filtre permet l'étude du pollen et des poussières cosmiques.

INTRODUCTION

The content of dissolved and particulate traces, the content and composition of occluded gases as well as the isotopic composition of oxygen and hydrogen in ice show variations which reflect the environmental conditions at the time the ice was formed. For the investigation of most of these historical records, ice core samples of only a few grams up to ¹ kg of ice are needed. One important task of ice core studies is to establish a reliable time scale based on ice dynamic considerations, stratigraphic analyses and measurements of radioisotopes.

Radioactive ice dating methods in boreholes in the age range of 100-25 000 years are based on the cosmic ray produced isotopes ³²Si, ³⁹Ar and ¹⁴C. Even using specially developed low level counting methods, it is required to extract the Si, Ar and $CO₂$ from 2-5 tons of ice to obtain samples which can be analysed with satisfactory accuracy (Oeschger *et al.,* 1966). This motivated us to develop an *in situ* extraction method for gases, particulate and dissolved matter in boreholes down to a depth of 400 m (dry boreholes).

Till now ¹⁴C and ³⁹Ar-samples from Byrd station (Antarctica), from Dye ³ (south Greenland), and from Devon Island (Canada) have been analysed. The given results are preliminary because an important check for contamination, the analysis of ⁸⁵Kr, has not yet been applied. This extraction technique offers possibilities for many other studies. Until now we used e.g. the remaining meltwater for Si-extraction (32Si-dating), for Cl-extraction (36Cl-dating) and for extraction of particulate matter by filtering the meltwater.

EXTRACTION PROCEDURE

The extraction method is based on a melting probe which can be lowered down to 400 m in boreholes with 135-165 mm diameter (Oeschger *et al.,* 1976). For

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melting and vacuum extraction at the desired depth, a portion of the borehole is sealed vacuum tight.

The upper sealing is an inflatable rubber packer. A special low temperature Neoprene hose is drawn over a metal cylinder and fixed on both ends. The rubber hose is inflated with pure N_2 at a pressure of 2 b, pressing it over its whole length against the wall of the borehole. The metal body is provided with ceramic insulated electrical connectors and gas inlet tubes.

Using a similar rubber packer for the lower sealing caused problems because it got stuck. Therefore we are now using a special probe to place a lower seal before lowering down the extraction probe. A rubber balloon is filled with 40°C warm water which melts the balloon slightly into the wall of the borehole. After a day the water in the balloon is frozen. The water tank is then pulled up and the balloon remains in place as a vacuum tight plug.

After tests to insure the sealing system is tight, the desired amount of ice is melted by a 10-kW electrical heater. It consists of several 1.7-m long heating elements which are mantled by stainless steel. It is designed for operation in vacuum without overheating.

The released gases are pumped off continuously during the melting process to minimize their solution in the meltwater. The gas flows outside the probe and along the cold wall of the borehole, where a part of the water vapour condenses. The remaining humidity is adsorbed by passing the gases through a stainless steel column with an active length of 1.7 m, filled with 300 g molecular sieve 3A in pellets. Then the gas flows through the $CO₂$ extraction column which has a length of 50 cm and is filled with 20 g of molecular sieve 5A in pellets. It is placed above the rubber packer where the temperature is lower to increase the adsorption efficiency of the molecular sieve. The remaining gases are pumped to the surface, where they are compressed and stored in steel cylinders.

When the desired amount of gases has been collected, pumping is stopped and sampling of particulate and dissolved matter starts. It can be performed either in the borehole (with a water circulation system in the probe) or at the surface. For surface extraction the probe is pulled up, while the lower seal and the meltwater remain in the borehole. A submersible pump is lowered into the hole and the meltwater is pumped to the surface for further sampling, e.g. by pumping through a filter or through an ion exchange column.

RADIO ISOTOPES USED FOR ICE DATING

We mainly discuss the application of three isotopes: ${}^{14}C$, ${}^{39}Ar$ and ${}^{32}Si$, which are produced in the atmosphere by cosmic radiation.

¹⁴C exists in the atmosphere as ¹⁴CO₂ and is therefore exchanging with the biosphere and the oceans. The half-life of ¹⁴C is 5570 years. The counting rate for 50 cm³ modern $CO₂$ (extracted from approximately 3 tons of ice) in a proportional counter with a background of 0.12 cpm is 0.32 cpm. For a counting time of 7 d ¹⁴C-ages with an accuracy are given in Fig. 1.

³⁹Ar has a half-life of 269 years. The counting rate of ⁵ dm³ modern Ar (extracted from approximately 5 tons of ice) is 0.36 cpm. The accuracy of ³⁹Ar-ages is given in Fig. 2.

The half-life of ³²Si is not well determined yet. A recent estimate is (295 \pm 25) years (Clausen, 1973). ³²Si has a residence time in the atmosphere of less than one year. It offers therefore a method to study short-term variations of isotope production by cosmic radiation in the atmosphere. More details on the ³²Si dating method are given by Dansgaard *et al.* (1966).

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FIGURE 1. Accuracy of ¹⁴C dating method. The calculated errors are based on a sample of 50 cm³ $CO₂$ and a measuring time of 10 000 min.

FIGURE 2. Accuracy of ³⁹Ar dating method. The calculated errors are based on a sample of 5 1. argon, a measuring time of 6000 min and a modern ³⁹Ar-activity of 0.12 dpm/L argon (Loosli and Oeschger, 1968).

RESULTS

During field projects at Dye 3 (Greenland) in 1971 and at Byrd station (Antarctica) in 1971-1972 we were able to extract samples at four different depths in each borehole. On average we melted 4 tons of ice and collected from each depth approximately 50 cm³ of CO_2 (for ¹⁴C-dating) and 260 dm³ of air for Ar-separation (39Ar-dating). In addition approximately 1.5 tons of water were used for Si-extraction (32Si-dating). The two projects were mainly thought as tests for the new ice dating techniques. The results are shown in Figs. 3 and 4.

At Dye 3 dating of ice by counting annual $\delta(^{18}O)$ -variations is possible. The agreement with the ages calculated from the ³⁹Ar and ³²Si results is satisfactory. The $CO₂$ samples from Dye 3 are contaminated by $CO₂$ degassing from plastic material; these results are not shown in Fig. 3.

The samples from Byrd station cover roughly the last 3000 years. For ³⁹Ar already the samples below 100 m are beyond the dating range. These samples therefore only yield minimum ³⁹Ar-ages. A contamination of some of the samples with modern air cannot yet be excluded. A check on the samples for the presence of ⁸⁵Kr, an isotope having been emitted into the atmosphere only during the last 20 years, will decide this. Of more interest are the ¹⁴C-ages which

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roughly agree with expectations except for the sample at a depth of 179 m. All samples were measured twice, except the sample from a depth of 179 m which got lost after the first measurement.

DISCUSSION

The radioactive ice dating methods described above can be considered as developed. They are an important contribution to glaciological studies in polar areas. Considering the great efforts involved in field and laboratory as well as the limited accuracy these methods should be applied in particular on occasions

FIGURE 3. Dye ³ (south Greenland) ages as a function of depth.

where other methods of age determinations fail. Examples are: regions with very irregular accumulation (no seasonal variations), ice profiles with discontinuities and ablation areas.

Improvements will mainly concern the radioactivity measurements. Lower backgrounds than reported here are obtained in an underground laboratory and a reduction of the statistical errors is expected.

Ice layers within the typical age range of the ¹⁴C-method are seldom reached in dry boreholes in accumulation areas. The extraction of $CO₂$ in liquid-filled boreholes is therefore desirable. This seems technically not impossible but it will be difficult to avoid contamination by the borehole fluid.

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FIGURE 4. Byrd station (Antarctica) ages as a function of depth.

Acknowledgements. This work was supported financially by grants from the US National Science Foundation, Office of Polar Programs, and the Schweizerischer Nationalfonds.

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