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Preparation of water and ice samples for ^{39}Ar dating by atom trap trace analysis (ATTA)

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Atom trap trace analysis (ATTA) is a new and promising method to measure very rare noble gas radioisotopes in the environment. The applicability of this method for the dating of very old groundwater with ^{81}Kr has already been demonstrated [1]. Recent developments now show its feasibility also for the analysis of ^{39}Ar [2,3], which is an ideal dating tracer for the age range between 50 and 1000 years. This range is of interest in the fields of hydro(geo)logy, oceanography, and glaciology. We present preparation (gas extraction and Ar separation) methods for groundwater and ice samples for later analysis by the ATTA technique.

For groundwater, the sample size is less of a limitation than for applications in oceanography or glaciology. Large samples are furthermore needed to enable a comparison with the classical method of ^{39}Ar detection by low-level counting. Therefore, a system was built that enables gas extraction from several thousand liters of water using membrane contactors. This system provides degassing efficiencies greater than 80 % and has successfully been tested in the field. Gas samples are further processed to separate a pure Ar fraction by a gas-chromatographic method based on Li-LSX zeolite as selective adsorber material at very low temperatures. The gas separation achieved by this system is controlled by a quadrupole mass spectrometer. It has successfully been tested and used on real samples. The separation efficiency was found to be strongly temperature dependent in the range of -118 to -130 ˚C.

Since ATTA should enable the analysis of ^{39}Ar on samples of less than 1 ccSTP of Ar (corresponding to about 100 ml of air, 2.5 l of water or 1 kg of ice), a method to separate Ar from small amounts of gas was developed. Titanium sponge was found to absorb 60 ccSTP of reactive gases per g of the getter material with reasonably high absorption rates at high operating temperatures ($\sim$ 800 ˚C). Good separation (higher than 92 % Ar content in residual gas) was achieved by this gettering process. The other main remaining component is H_2, which can be further reduced by operating the Ti getter at lower temperature. Furthermore, a system was designed to degas ice samples, followed by Ar separation by gettering. Ice from an alpine glacier was successfully processed on this system.

References:
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