

Identification of Ikaite ($\text{CaCO}_3 \times 6 \text{H}_2\text{O}$) in Antarctic Sea Ice by Low Temperature Synchrotron X-ray Diffraction

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Hyper-saline brines are generated within the ice matrix when sea ice forms. They undergo physicochemical changes at sub-zero temperatures resulting in mineral precipitation. Ca containing crystals ($< 5 \mu\text{m}$ to $600 \mu\text{m}$ in size) were detected in Antarctic sea ice cores. Ikaite ($\text{CaCO}_3 \times 6\text{H}_2\text{O}$, monoclinic) precipitation had previously been discussed in literature but has until recently not been observed. Because ikaite decomposes above 4°C mineralogical phase identification requires sample processing, transport, and measurement in a cryogenic environment. Thus, the temperature throughout the sample preparation was kept below 4°C . Ice cores were either melted on board ship or returned to the home laboratory at -18°C and melted there. Temperature of the melt water never exceeded 0°C . Crystals were stored in ethanol solution and frozen at -18°C for later phase identification. From both sets, crystals were selected and mounted on glass fibres or filled in capillaries in a cold room (about 1°C) at the Alfred Wegener Institute for Polar Research in Bremerhaven, and transported to the synchrotron radiation source ANKA of the Forschungszentrum Karlsruhe. During transport, samples were kept at temperatures below 2°C . Analyses were done by X-ray diffraction at the X-ray beam-line of the Synchrotron Laboratory for Environmental Studies (SUL-X). Diffracted intensities were collected from the crystals in a cryogenic N_2 gas stream with a focussed ($150 \text{ mm} \times 150 \text{ mm}$) monochromatic X-ray beam (14 keV), using a CCD detector with a fibre-optic taper (XDI-VHR, Photonic Science). Diffraction angles were calibrated with silicon and integrated to one dimensional background corrected powder diffractograms. X-ray diffraction (XRD) patterns of the crystals from Antarctic sea ice matches that of ikaite precipitated as reference material in the laboratory according to Marland (1975), and that calculated for ikaite. Since all crystals extracted from the ice cores had the same morphology, it is most likely that the XRD identification as ikaite is representative for all crystals used in this study. Ikaite crystals were found throughout the sea ice and in all ice types but were not distributed uniformly with depth in the ice cores. Up to 19.4 mg of ikaite were determined per liter of melted sea ice. Little is known on the spatial and temporal occurrence of ikaite precipitates in polar sea-ice, so far. But, this is crucial since the precipitation of CaCO_3 is implicated in many processes of global significance, such as the sea ice-driven carbon pump and the global carbon cycle.

References

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