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Sampling and analytical methods of stable isotopes and dissolved inorganic carbon from CO2 injection sites

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The isotopic composition (δ^{13}C) of dissolved inorganic carbon (DIC), in combination with DIC concentration measurements, can be used to quantify geochemical trapping of CO_2 in water. This is of great importance in monitoring the fate of CO_2 in the subsurface in CO_2 injection projects. When CO_2 mixes with water, a shift in the δ^{13}C values, as well as an increase in DIC concentrations is observed in the CO_2-H_2O system. However, when using standard on-site titration methods, it is often challenging to determining accurate in-situ DIC concentrations. This may be due to CO_2 degassing and CO_2-exchange between the sample and the atmosphere during titration, causing a change in the pH value or due to other unfavourable conditions such as turbid water samples or limited availability of fluid samples.

A way to resolve this problem is by simultaneously determining the DIC concentration and carbon isotopic composition using a standard continuous flow Isotope Ratio Mass Spectrometry (CF-IRMS) setup with a Gasbench II coupled to Delta *plus*XP mass spectrometer. During sampling, in order to avoid atmospheric contact, water samples taken from the borehole-fluid-sampler should be directly transferred into a suitable container, such as a gasbag. Also, to avoid isotope fractionation due to biological activity in the sample, it is recommended to stabilize the gasbags prior to sampling with $HgCl_2$ for the subsequent stable isotope analysis.

The DIC concentration of the samples can be determined from the area of the sample peaks in a chromatogram from a CF-IRMS analysis, since it is directly proportional to the CO_2 generated by the reaction of the water with H_3PO_4. A set of standards with known DIC concentrations should be prepared by mixing $NaHCO_3$ with DIC free water. Since the DIC concentrations of samples taken from CO_2 injection sites are expected to be exceptionally high due to the additional high amounts of added CO_2, the DIC concentration range of the standards should be set high enough to cover the sample concentrations. In order to assure methodological reproducibility, this “calibration set” should be included in every sequence analysed with the Gasbench CF-IRMS system. The standards, therefore, should also be treated in the same way as the samples. For accurate determination, it is essential to know the exact amount of water in the vial and the density of the sample. This requires weighing of each vial before and after injection of the water sample. For stable isotope analysis, the required signal height can be adjusted by the sample amount. Therefore this method is suitable for analysing samples with highly differing DIC concentrations. Reproducibility and accuracy of the quantitative analysis of the dissolved inorganic carbon need to be verified by independent control standards, treated as samples.

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