

Deployment of method for analysis of $\delta^{13}\text{C}$ (n-alkanes C1-C5) in gas samples at low concentrations

^aMorales J R, ^aWagener A L, ^aScofield A L, ^aBaêta A P

The concentration and isotopic composition of trace hydrocarbon gas have provided useful data for the interpretation and estimation of interesting processes in oil exploration, climate change studies, degradation of oils and elucidation of the carbon cycle. The analysis of atmospheric and drilling samples is a challenge, since the low concentration limits the use of the conventional technique GC-IRMS and requires the use of large sample volumes.

Cryogenic techniques using a preconcentration system coupled to GC-IRMS system have been successfully tested in the analysis of gases such as CO_2 , N_2O , CH_4 and CO . The main goals of this study are to determinate the isotopic composition of n-alkanes C1-C5 in gas samples at low concentration using a cryogenic device for gas concentration (PreCon). This device collects the condensable gases (C_{2+} , CO_2 , Ar, N_2 , O_2 , N_2O , H_2O) in liquid nitrogen, while oxidizes the C_1 introduced, cryofocuses and injects onto a GC-IRMS system, using only a few milliliters of the sample.

Initially, was adjusted the isotopic analysis of n-alkanes by GC-IRMS (at high concentration), and was verified the absence of fractionation during the analysis done in the pre-concentrator (using standards at low concentration). The introduction of a PLOT column in the main trap, inside the PreCon, allowed us to check the separation of condensable gases and the quantitative collection of compounds of interests. The interferences (H_2O , CO_2 , N_2 , O_2 and olefins) were minimized by the introduction of a cleaning trap in the carrier gas and by using of I_2O_5 in the chemical trap. The determination of n-alkanes C_{2+} was made via oxidation at the GC-IRMS system. The methodology was tested analyzing the headspace of sediments and drilling samples (gases confined into Isotube® sampler) with low concentrations of C_1 - C_5 (2ppm-100ppm C_1). The determination of the isotopic composition of these samples confirmed the applicability of the methodology and system with a high analytical precision (<0.2‰).

^a Laboratório de Estudos Marinhos e ambientais LABMAM. Departamento de Química PUC-Rio (juliana_rozo@hotmail.com)