**Quantitative determination of methane and its isotopologues in deep marine sediments at IODP 370 site C0023, off Muroto, Japan**

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Methane (CH₄) geochemistry and cycling are of particular interest in regards to deep microbial life because methanogenesis is a terminal process of organic matter decomposition such that it is expected to represent the deepest microbial metabolisms. Standard methods of extracting and determining dissolved CH₄ in sediment cores (e.g. headspace analysis) fail in poorly-competent and gassy sediments due to the formation of gas pockets during core retrieval. Further, no method has been described for extraction of CH₄ from sediments for isotopologue analysis.

During IODP Expedition 370, the Scientific Party collected a set of whole-round core slice samples for gas analysis, which were immediately frozen and maintained at -80°C[1]. Here, we report a method to quantitatively extract CH₄ and other gases from the core slices. Dissolved gases (CH₄, N₂, Ar, O₂) released from sediment core samples upon thawing are measured using a quadrupole gas analyzer. The in situ CH₄ concentration can then be estimated by applying corrections to account for both depressurization-induced gas loss during core retrieval, and entrainment of air during subsampling and initial freezing aboard D/V Chikyu; this method is similar to one previously developed to determine in situ CH₄ concentrations from depressurization-associated gas pockets [2].

For samples that yield enough gas for clumped isotopologue analysis (ca. >1 mL, STP), the ¹³CH₃D abundance, as well as apparent equilibrium temperatures calculated from the relative abundance of four methane isotopologues (¹³CH₄, ¹₂CH₃, ¹²CH₂D, and ¹³CH₂D), will be reported. Clumped isotopologue analysis can potentially target whether methane found in sediments was formed in situ by microbial methanogenesis, or at greater depth by thermal cracking of organic materials, and migrated upward.