# 49. MOLECULAR AND ISOTOPIC ANALYSIS OF CORE GASES AND GAS HYDRATES, DEEP SEA DRILLING PROJECT LEG 96[1]

Ronald C. Pflaum, James M. Brooks, H. Benjamin Cox, Mahlon C. Kennicutt II, and Der-Duen Sheu,
Texas A&M University[2]

## ABSTRACT

Molecular and isotopic compositions of void gases from Sites 618 and 619 were determined. The gases were predominantly methane with the $C_1/C_2$ ratios averaging 4,000 and 26,000 at Sites 618 and 619, respectively. The $\delta^{13}$C-$C_1$ values at Site 618 were nearly constant, ranging from −70.1 to −73.7‰. $\delta^{13}$C-$CH_1$ values for Site 619 between 76 and 178 meters sub-bottom ranged from −94.8 to −70.8‰, becoming progressively heavier with depth. The molecular and isotopic compositions at both sites are characteristic of biogenic methane.

Small spheres (~1–2 mm diameter) of gas hydrates were observed in the Orca Basin between 20 and 40 m sub-bottom and were often associated with sandy sections. The hydrate was predominantly methane with a $\delta^{13}$-$C_1$ of −71‰ and trace amounts of ethane, propane, and carbon dioxide. The isotopic value for the methane was similar to that of the nearest gas pocket, indicating that hydrate was formed from predominantly biogenic methane without isotopic fractionation.

## INTRODUCTION

Gaseous hydrocarbons are ubiquitous in marine sediments and are derived from biologically mediated processes and/or thermogenic cracking of organic matter (Bernard, 1977). In most near-surface sediments, the primary processes affecting gaseous hydrocarbon levels are biological. In the oxygenated zone, $CO_2$ is produced from organic matter during aerobic bacterial respiration. Below the aerobic zone, organic matter continues to be oxidized to $CO_2$ during anaerobic bacterial respiration utilizing dissolved sulfate as the major electron acceptor (ZoBell and Rittenberg, 1948). Below the zone of sulfate reduction, bacteria consume bicarbonate-derived $CO_2$ and produce methane ($C_1$). This reduction of $CO_2$ to $C_1$ may occur at considerable depths in the sediment column (Claypool and Kaplan, 1974). As pressure and temperature increase with depth, chemical diagenetic reactions become increasingly important. $C_1$ can be produced by carbon-bond cleavage in the thermogenic reduction of organic matter. Small amounts of higher hydrocarbons can also be produced during the initial cracking of organic matter (Whelan et al., 1980; Claypool and Kvenvolden, 1983). As sediment temperatures increase above ~50°C, thermogenic decomposition of organic matter generates larger amounts of methane and higher molecular weight hydrocarbons. If migration pathways exist (e.g., faulting and/or fracturing), the upward migration of these thermogenic hydrocarbons will influence the gas content of surface sediment.

The origin of the gaseous hydrocarbons can be differentiated on the basis of the carbon-isotope ratio of methane ($\delta^{13}$C-$C_1$) and the ratio of methane to ethane plus propane [$C_1/(C_2 + C_3)$]. Bernard (1977) found that a $\delta^{13}$C-$CH_1$ less than −60‰ and a $C_1/(C_2 + C_3)$ ratio of greater than 1000 were diagnostic of gas of a microbial origin and that a $\delta^{13}$C-$C_1$ greater than −50‰ and a $C_1/(C_2 + C_3)$ ratio less than 50 indicated gas of a thermogenic origin. Intermediate values may result from mixing and/or oxidation.

Sites 618 and 619 are located in intraslope basins formed between diapirs of an underlying salt layer of Louann age (Jurassic). The Orca Basin (Site 618) is located in ~2400 m of water and is an interdomal basin resulting from the coalescing of a group of upward moving diapirs (Bouma, 1983; Intraslope Basin Introduction and Summary, this volume). The basin is unique in that it contains 200 m of anoxic, hypersaline brine. Shokes et al. (1977) reported that the source of the brine was dissolution of a near surface salt diapir and that the brine was entering laterally rather than by diffusion upward through the sediments. The relative stability of the brine has allowed high concentrations of methane from the underlying sediments to accumulate in the brine layer.

Pigmy Basin (Site 619) is northwest of Orca Basin at a water depth of ~2270 m. Pigmy Basin is a blocked-canyon intraslope basin formed by blockage of the thalweg of a submarine channel by diapiric activity (Intraslope Basin Introduction and Summary, this volume). The basin shows a normal sedimentation sequence which is not extensively affected by slumping thereby differing from the Orca Basin sediments (see site chapters, this volume).

### METHODS

Visible gas pockets in the cores were sampled using a dual needle and septum device with a valve to prevent contamination of the sample with atmospheric gas when the core liner was punctured. The samples were collected in vacutainers. Analyses of gases were performed on a Hewlett-Packard (HP) 5710A gas chromatograph equipped with a single flame ionization detector (FID) and a 9 ft. × 1/8″ outside di-

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[1] Bouma, A. H., Coleman, J. M., Meyer, A. W., et al., *Init. Repts. DSDP*, 96: Washington (U.S. Govt. Printing Office).

[2] Addresses: (Pflaum, Brooks, Cox, Kennicutt II) Department of Oceanography, Texas A&M University, College Station, TX 77843; (Sheu) School of Geology and Geophysics, 830 Van Vleet Oval, University of Oklahoma, Norman, OK 73019.

ameter (OD) Porapak Q column. Measurements of carbon dioxide were quantified on board ship using a Carle Model 800 gas chromatograph with a thermal conductivity detector and a column (5 ft. × 1/8" OD) packed with 8% Carbowax 1540 on 90–100 mesh Anachrom ABS.

Gas hydrates collected at Site 618 were stored in a cryogenic dewar containing liquid nitrogen (−196°C) for laboratory analysis at Texas A&M University. In the laboratory, the frozen section was opened and hydrate crystals were placed in a 23-cm$^3$ Parr bomb pressure vessel (Kvenvolden and Barnard, 1983), sealed, and allowed to decompose. Aliquots of gas for molecular and isotopic analysis were withdrawn through a septum on the block assembly using a gas-tight syringe.

All hydrate molecular analyses were quantified on HP 5790A gas chromatographs. The fixed gases were determined by thermal conductivity with a ten-port valve system using a 2-m molecular sieve (5 Å) column and a 2-m Porapak-Q column. Hydrocarbons were detected by flame ionization using a 2-m HP DC-200 column.

Stable carbon-isotope ratios were obtained on $C_1$ after separation from the other components by fractional distillation at liquid nitrogen temperature and combustion to $CO_2$ in a method analogous to that of Sackett et al. (1970). The $\delta^{13}C$ of the resultant $CO_2$ was measured on a Nuclide RMS 60 ratio mass spectrometer and the results reported relative to the PeeDee belemnite (PDB) standard with a precision of ±0.2‰.

## RESULTS

The molecular and isotopic composition of the void gases from DSDP/IPOD Leg 96 are reported in Table 1. Methane was the dominant hydrocarbon at all sites, with only trace amounts of ethane and carbon dioxide present. The $C_1/(C_2 + C_3)$ ratios (reported in the tables as $C_1/C_2$ since propane was not detected) as well as the carbon-isotope ratio of the methane ($\delta^{13}C$-$C_1$) were consistent with a predominantly biogenic methane source in these cores. The $\delta^{13}C$-$C_1$ values for Site 618 from 3 to 92 m sub-bottom remained nearly constant, ranging from −70.1 to −73.7‰. $\delta^{13}C$-$C_1$ values for Site 619 from 76 to 178 m sub-bottom depth became progressively heavier (more positive) with increasing depth, ranging from −94.8 to −70.8‰. The systematic increase in $\delta^{13}C$-$C_1$ with depth at Pigmy Basin is consistent with most void gas profiles (Brooks et al., 1983). This trend may be the result of either upward migration of thermogenic gas or substrate depletion resulting in heavier methane. The reason for the absence of such a trend in Orca Basin is unclear. It could result from the shallow depth penetration of the hole, and/or the abundance of reworked sediments from localized mass movement processes and the rapid sedimentation rates in the basin, which tend to homogenize the section.

It is significant that void gases were not encountered until 76 m sub-bottom depth at Site 619 compared to only 3 m sub-bottom depth at Site 618. This correlates well with the rates of sulfate reduction estimated from the concentration gradients of interstitial sulfate in the cores (see Kennicutt et al., this volume). In Orca Basin, sulfate was present at 3 m but absent at 12 m, whereas sulfate-free cores were not encountered until 71 m sub-bottom depth in the Pigmy Basin. The Orca Basin finding is somewhat at odds with other data (Wiesenburg, 1980) which show very slow rates of sulfate reduction in shallow Orca Basin sediments resulting from the high ionic composition of the brine. The reason for the high rate of sulfate reduction and subsequent methanogenesis in the near-surface sediments at Site 618 is unclear.

Table 1. Molecular and carbon-isotopic compositions of void gas from DSDP Leg 96.

| Section | Sub-bottom depth (m) | $C_1$ (%) | $C_2$ (%) | $CO_2$ (%) | $C_1/C_2$ | $\delta^{13}C$-$C_1$ (‰) |
|---|---|---|---|---|---|---|
| Orca Basin | | | | | | |
| 618-1-2 | 3 | 28.41 | 0.009 | ND | 3100 | −70.1 |
| 618-1-3 | 4.5 | 51.73 | 0.015 | ND | 3400 | −73.2 |
| 618-2-3 | 12 | 10.55 | 0.004 | 0.010 | 2400 | — |
| 618-3-5 | 24 | 86.18 | 0.025 | 0.010 | 3400 | −72.0 |
| 618-3-6 | 25 | 68.44 | 0.017 | ND | 4000 | −71.6 |
| 618-4-2 | 29 | 76.38 | 0.004 | 0.118 | 18,400[a] | −71.3 |
| 618-4-6 | 34 | 80.21 | 0.020 | ND | 4000 | −71.2 |
| 618-5-4 | 42 | 82.66 | 0.025 | 0.060 | 3300 | −71.1 |
| 618-5-7 | 45 | 74.31 | 0.023 | ND | 3200 | −71.0 |
| 618-6-3 | 49 | 89.49 | 0.024 | 0.042 | 3700 | −71.1 |
| 618-7-3 | 54 | 83.08 | 0.022 | 0.035 | 3800 | −71.2 |
| 618-8-5 | 64 | 75.77 | 0.019 | 0.119 | 4000 | −71.2 |
| 618-8-6 | 65 | 81.02 | 0.021 | ND | 3800 | −71.8 |
| 618-9-2 | 73 | 76.98 | 0.020 | 0.169 | 3800 | −71.3 |
| 618-10-5 | 80 | 63.91 | 0.011 | 0.146 | 5900 | −73.7 |
| 618-11-2 | 92 | 84.63 | 0.010 | 0.274 | 8400 | −73.6 |
| Pigmy Basin | | | | | | |
| 619-10-2 | 76 | 40.40 | 0.002 | 0.031 | 22,600 | −94.8 |
| 619-12-3 | 102 | 44.65 | 0.002 | 0.090 | 28,000 | −87.0 |
| 619-13-4 | 114 | 5.83 | <0.001 | 0.042 | — | — |
| 619-14-2 | 120 | 7.08 | <0.001 | 0.074 | — | — |
| 619-15-3 | 130 | 72.34 | 0.002 | 0.077 | 31,500 | −80.6 |
| 619-16-5 | 142 | 83.21 | 0.003 | 0.130 | 27,200 | −76.6 |
| 619-17-3 | 150 | 17.49 | 0.001 | 0.069 | 27,400 | −72.1 |
| 619-18-1 | 158 | 85.45 | 0.004 | 0.173 | 22,700 | −71.5 |
| 619-19-2 | 168 | 79.85 | 0.003 | 0.307 | 22,900 | −70.8 |
| 619-20-2 | 178 | 88.70 | 0.003 | 0.189 | 26,300 | −72.1 |
| Mississippi Fan | | | | | | |
| 620-41-3 | 380 | 30.73 | 0.001 | ND | 28,300 | −82.4 |
| 620-42-2 | 388 | 25.34 | 0.001 | ND | 42,000 | −77.2 |
| 621-4-2 | 27 | 24.90 | <0.001 | 0.200 | — | −71.8 |
| 621-5-2 | 35 | 78.53 | 0.002 | 0.240 | 36,500 | −70.6 |
| 621-6-2 | 45 | 4.43 | <0.001 | 0.040 | — | −72.0 |
| 621-7-2 | 49 | 13.42 | <0.001 | 0.043 | — | −69.4 |
| 621-19-1 | 125 | 34.02 | 0.001 | 0.086 | 29,900 | −79.5 |
| 621-27, top | 177 | 27.09 | 0.001 | 0.076 | 25,100 | −75.4 |
| 621-34-6 | 228 | 1.29 | <0.001 | ND | — | — |

Note. ND = not determined; a dash (—) indicates a component was present in too small a concentration for the analysis or calculation to be performed.

[a] Gas hydrate crystals recovered from this section.

Sediments in both basins contain similar amounts of sedimentary organic carbon (see Kennicutt et al., this volume). Because of the anoxic layer of brine overlying the Orca Basin floor, it is possible that the organic matter reaching the seafloor is more labile and therefore more easily degraded by the sulfate-reducing bacteria. The slower rate of sedimentation in Pigmy Basin may also result in greater degradation of the labile organic matter at the seawater/sediment interface producing more refractory organic matter in the buried sediments.

Only small amounts of ethane and carbon dioxide were detected in the vacutainers. The $C_1/C_2$ ratios averaged ~4,000 and 26,000 for Sites 618 and 619, respectively. Ethane and carbon dioxide were not present in sufficient concentrations for carbon-isotope analysis. These compositions are consistent with similar studies by Schoell (1982) and Galimov and Kvenvolden (1983) in deep-sea sediments at similar sub-bottom depths from both the Atlantic and Pacific margins. At sub-bottom depths greater than those sampled at Leg 96 sites, ethane and other $C_2$–$C_5$ hydrocarbons were detected (Galimov et al., 1980; Schoell, 1982). In contrast, core gas-pocket studies in the Japan Trench (Whelan and Sato, 1980), California Basin (Galimov and Simoneit, 1982), and Middle America Trench (Jeffrey et al., 1985) found $C_2$–$C_5$ hydrocarbons at most depths where gas pockets were sampled. The processes controlling the amount of $C_2$–$C_5$ hydrocarbons in shallow sediments containing primarily biogenic gas are unclear. In these intraslope basins there is certainly little low-temperature diagenetic production of these gases or migration of deeper thermogenic gases into these shallower sediments even though there is evidence of thermogenic high molecular weight hydrocarbons at both sites (Kennicutt et al., this volume). The anomalous $C_1/C_2$ ratio measured in Section 618-4-2 (Table 1) may be related to the presence of the gas hydrates.

There appears to be significant horizontal variation in the $\delta^{13}C$-$C_1$ in Orca Basin. Sackett et al. (1979) analyzed hydrocarbons degassing from the pore water in a 5.6-m piston core taken in the northern sub-basin of Orca Basin near Site 618. Propane was present in those headspace samples and the $C_1/(C_2 + C_3)$ ratios averaged about 590. These $C_1/(C_2 + C_3)$ ratios appear to represent a mixture of thermogenic and biogenic gas (Bernard, 1977). The $\delta^{13}C$-$C_1$ values reported by Sackett et al. (1979) are extremely light, ranging from −85 to −105‰. Such a light $\delta^{13}C$-$C_1$ suggests that the reservoir of carbon from which the methane was generated is isotopically light. It is probable that these differences represent localized variability in the basin. Hole 618 was drilled on a minor topographic high in the center of the northern basin lobe and contained slumped material from the basin walls (see Site 618 chapter, this volume), whereas the Sackett et al. (1979) core was closer to the basin margin and the brine/seawater interface.

Only a few void gases were encountered in the Mississippi Fan sites visited during DSDP Leg 96. The data from these samples are shown in Table 1 and all represent biogenic gas.

### Gas Hydrates

Gas hydrates are "icelike" crystalline structures in which methane (and other gases) are occluded inside a water lattice. These structures are typically only stable under the high pressures and low temperatures that can exist on the continental slope. Gas hydrates were observed and sampled in the Orca Basin in both Holes 618 and 618A between 20 and 40 m sub-bottom depth (Site 618 chapter, this volume). These hydrates consisted of scattered white crystals of a few millimeters to possibly a centimeter in diameter. Some of the hydrates appeared to be associated with sandy layers in the cores. Because of the very gassy nature of the entire Orca Basin core, it is possible that gas hydrates were scattered throughout the core and decomposed before retrieval on deck.

The molecular analysis of a decomposed gas hydrate recovered from Section 618-4-2 at approximately 27 m sub-bottom is reported in Table 2. The hydrate contained primarily methane with trace amounts of ethane, propane, and carbon dioxide representing predominantly a Structure I hydrate. The $\delta^{13}C$-$C_1$ for the hydrate was the same (−71.3‰) as for the gas collected in a vacutainer in the same core section (Table 1). The anomalous $C_1/C_2$ ratio for the section containing the hydrate may have resulted in part from the presence of the hydrate because the levels of ethane and propane, while low, are much higher than those found in the core gas pockets. These data suggest that hydrate formation does not involve isotopic fractionation of methane but may involve a preferential incorporation of the ethane and propane present. For comparison, data are included from the Green Canyon hydrate previously reported in Brooks et al. (1984). The Site 618 hydrate, having lighter $\delta^{13}C$-$C_1$ and low amounts of $C_2$–$C_4$ hydrocarbons, is primarily a biogenic gas hydrate as compared to the thermogenic Green Canyon hydrate.

Table 2. Comparison of molecular and isotopic compositions of the Orca Basin and Green Canyon Block 184 gas hydrates.

| | Orca Basin[a] | | Green Canyon[b] | |
|---|---|---|---|---|
| | Concentration (%) | $\delta^{13}C$ (‰) | Concentration (%) | $\delta^{13}C$ (‰) |
| Methane | 39.54 | −71.3 | 55.1 | −44.6 |
| Ethane | 0.13 | | 2.6 | −29.3 |
| Propane | 0.11 | | 14.4 | −18.6 |
| Isobutane | ND | | 4.4 | −28.6 |
| *n*-Butane | ND | | 0.2 | −28.6[c] |
| $CO_2$ | 0.09 | | 3.4 | +18.5 |
| $C_1/C_2$ | 295 | | 21.2 | |
| $C_1/C_2 + C_3$ | 159 | | 3.2 | |

Note. ND = not detected.

[a] Hydrate sample from Section 618-4-2, at approximately 27 m below seafloor.

[b] From Brooks et al. (1984).

[c] The butanes were collected and combusted together for isotope analysis.

## SUMMARY

Gases in core expansion pockets from Sites 618 and 619 are composed primarily of methane with small amounts of ethane and carbon dioxide. A gas hydrate from about 27 m sub-bottom depth at Site 618 was also primarily composed of methane with small amounts of ethane, propane, and carbon dioxide. $C_1/C_2$ ratios and $\delta^{13}C$-$C_1$ values indicate that biological activity is the primary source of the methane in the core gas pockets and gas hydrates. The composition of the gases in the core expansion pockets and hydrates do not show the effects of hydrocarbon production by low temperature chemical diagenesis or migration of hydrocarbons from more thermally mature sources even though studies on nonvolatile organics indicate that migration of thermogenic hydrocarbons is occurring (Kennicutt et al., this volume). Gas hydrate formation may involve a preferential incorporation of the ethane and propane at Site 618, but does not appear to involve any carbon-isotope fractionation in the methane incorporated into the hydrate.

### ACKNOWLEDGMENTS

We thank the shipboard scientists on the *Glomar Challenger* for their help during the sampling procedures. Research support for this study was provided by the National Science Foundation through Grant OCE-83-01538. Instrumentation support for this project was provided by the Center for Energy and Mineral Resources at Texas A&M University. This manuscript was reviewed by William Sackett and Patrick Parker.

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**Date of Initial Receipt: 17 January 1985**
**Date of Acceptance: 21 March 1985**