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1. SAANICH INLET EXPLANATORY NOTES[1]

Shipboard Scientific Party[2]

INTRODUCTION

In this chapter, we outline the procedures undertaken during Ocean Drilling Program (ODP) Leg 169S, as well as during the initial description and sampling of cores at the Pacific Geoscience Centre (PGC), Sidney, British Columbia, immediately following drilling, and at the Gulf Coast Core Repository (GCR) in College Station, Texas, about 80 days after Leg 169S was completed. Unlike conventional drilling legs, only one hole per site was opened during drilling; cores from the remaining holes were opened in College Station. The information contained in this volume is derived from investigations undertaken by the shipboard scientific party during the leg and by post-Leg 169S investigators.

NUMBERING OF SITES

Numbering of sites, holes, cores, and samples, determination of seafloor depth (meters below sea level) and core depths (meters below seafloor), and curation of cores all followed standard ODP procedures (e.g., Westbrook, Carson, and Musgrave, et al., 1994).

CORE HANDLING

Standard core handling and sampling procedures were followed, except for the following. Cores in the upper part of the sediment section were assessed for hydrogen sulfide content as they arrived on the catwalk. Microbiologic samples were taken by syringe on the catwalk from the ends of the cut sections, and immediately stored in a liquid N_2 dewar. Twice a day, these frozen samples were taken off the ship by a launch to a lab at the Institute of Ocean Sciences in Sidney, B.C.

Interstitial water samples were taken as $^1/_4$-round scoops from the working half of each core, were immediately squeezed, and the water was stored in vials for subsequent analyses. Whole-round samples were not allowed because preservation of the entire record of laminations was of the highest priority for the leg. Owing to lack of time, only alkalinity was determined during Leg 169S. The remaining standard shipboard interstitial water analyses were done during Leg 169.

Headspace gas samples were taken on the catwalk and analyzed during Leg 169S for safety monitoring.

Because Cores 169S-1033A-1H and 1034A-1H did not recover the mudline, they were not split during the leg. Cores from Holes 1033B and 1034B were run through the shipboard multisensor track (MST) prior to splitting. No thermal conductivity measurements were made.

Index properties samples were taken aboard ship, stored, and taken to PGC after the leg for analysis. The working half of each section was then wrapped in plastic wrap and stored in d-tubes.

The archive halves of each section were analyzed for color using a Minolta spectrophotometer as soon as the cores were split. Each core was then photographed, wrapped in plastic wrap, and stored in d-tubes. The B-hole cores from Sites 1033 and 1034 were taken off the ship for temporary cold storage at PGC. A description and sampling party was held at PGC for the 3 days immediately after the leg. The B-hole cores remained at PGC for 1 yr post-cruise, and then were sent to the Gulf Coast Repository (GCR) in College Station, where the remaining cores from this leg are stored.

Cores from Holes A, C, D, and E from Sites 1033 and 1034 remained unsplit on ship and were run through the MST during Leg 169. After Leg 169, these cores were shipped to the GCR, where a description and sampling party was held in November, 1996.

SCIENTIFIC PROCEDURES

Lithostratigraphy

Sedimentological description and lithostratigraphic classification of cores recovered during Leg 169S follow those adopted for the description of Site 893 (Shipboard Scientific Party, 1994). Laminated sediments were described as well laminated, intermittently laminated, or as containing traces of laminae.

Radiocarbon Dating

Samples of wood, charcoal, plant material, and whole and broken shells were extracted with stainless steel tweezers, rinsed with distilled water, and dried at room temperature under a fume hood.

Of the 71 samples submitted for dating, 32 were sent for identification. Terrestrial samples were identified by G.D. Jensen at the Pacific Forestry Centre, Victoria, B.C., and shell samples, by R.G.B. Reid at the University of Victoria (Canada).

Samples were sent for accelerator mass spectrometry (AMS) radiocarbon analysis at the Center for Accelerator Mass Spectroscopy, Lawrence Livermore National Laboratory, California. The selection of samples was based on a priority system. The first priority was to choose samples that could represent major stratigraphic breaks or boundaries of significant sedimentary units. The second priority was to obtain evenly distributed samples that would reflect the complete sediment record. The third priority was the type of sample: (1) large terrestrial samples (e.g., charcoal or wood fragments a few centimeters long), because there is no need for marine reservoir correction; (2) whole marine (e.g., paired bivalves) samples that reflect in situ growth within the sediments; (3) marine and terrestrial pairs found at the same stratigraphic location to aid in confirming the reservoir age determined for the area; and (4) small terrestrial or marine organic fragments.

ORGANIC GEOCHEMISTRY

The drilling objectives of Leg 169S included an extensive component of organic and inorganic geochemistry. Organic geochemical investigations were conducted to provide (1) real-time monitoring of

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[1] Bornhold, B.D., Firth, J.V., et al., 1998. *Proc. ODP, Init. Repts,* 169S: College Station, TX (Ocean Drilling Program).

[2] Shipboard Scientific Party is given in the list preceding the Table of Contents.

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light hydrocarbons gases, hydrogen sulfide, and other gases as part of the shipboard safety requirements; (2) an initial characterization of the types and amounts of sedimentary organic matter encountered; and (3) initial information on the diagenetic situation in the sediments of Saanich Inlet.

The C_1/C_2 ratio is generally used to obtain quick information about the origin of hydrocarbons; that is, to distinguish between biogenic gas and gas migrating from thermogenic hydrocarbons, typically from a deeper more mature source. Very high C_1/C_2 ratios generally indicate gas (C_1) formation by microbiological processes. On the other hand, the occurrence of major amounts of C_2 (to C_5) gas at shallow depths is associated with the occurrence of allochthonous thermogenic hydrocarbons.

The analytical program was also intended to provide preliminary geochemical information to assist in sample selection for additional or more detailed shore-based studies. The following instrumentation and procedures were used during Leg 169S to (1) measure the concentrations of hydrocarbon and other gases, such as sulfides and permanent gases (e.g., nitrogen and oxygen), and (2) determine the quantity and quality of organic matter in the sediments.

Sampling for Headspace and Vacutainer Gases

During Leg 169S, the compositions and concentrations of hydrocarbons and other gases were monitored in the sediments generally at intervals of at least one per core (typically mid-core at the liner breaks). Two methods were used, referred to as headspace (HS) and vacutainer (V) in the data tables.

In the HS method, gases released by the sediments after core recovery were analyzed by gas chromatography (GC) with the following technique: a modified (open-ended) 5-mL plastic syringe or a calibrated cork borer was used to obtain a measured volume of sediment from the end of a section of core, immediately after retrieval on deck. The sediment, usually ~5 cm^3, was placed in a 21.5-cm^3 glass serum vial that was sealed with a septum and metal crimp cap. When consolidated or lithified samples were encountered, chips of material were placed in the vial and sealed. The vial was then heated to 60°C in an oven and kept at this temperature for 30 min before gas analysis. A 5-cm^3 volume of the headspace in the vial was extracted with a standard glass syringe for each analysis by gas chromatography.

The vacutainer method of gas analysis was used when gas pockets or expansion voids occurred in cores as they arrived on deck. Vacutainers are pre-evacuated, septum-sealed glass tubes (20 cm^3). For the purpose of obtaining a gas sample, a special tool is employed to penetrate the core liner. This tool, equipped with a valve and needle, is used to transfer gas from the core into the vacutainer. Portions of gas in the vacutainer were analyzed by gas chromatography.

Analytical Procedure for Hydrocarbon and Other Gases

Compositions and concentrations of hydrocarbon and other gases were monitored using two different gas chromatographic systems. The first system, the Hewlett Packard 5890 Series II gas chromatograph, is applied by extracting gases using standard ODP headspace-sampling techniques (Kvenvolden and McDonald, 1986), referred to as HC. The second method employed the Hewlett-Packard 5890A, Natural Gas Analyzer, modified by John Booker and Company, Austin, Texas, and referred to as NGA.

Headspace Gas Analyses: Gas Chromatograph (HC)

The Hewlett Packard 5890 gas chromatography is a standard packed-column flame ionization detector (FID) GC, the output of which is attached to the Hewlett-Packard Chemstation integration and processing software that allows the single measurement of gas concentrations >6 orders of magnitude after appropriate calibration. The HC instrument is designed to measure accurately in ~7 min the concentrations of methane, ethane, and propane. Ethene is resolved from ethane and can also be quantified. The HC has the following characteristics: samples are introduced by means of a 1.0-cm^3 sample loop with manual column backflush; the chromatographic columns used were a 0.32 cm × 1.8 m stainless-steel tubing packed with 80% Porapak N and Porapak Q (80/100 mesh) and a 0.32 cm × 1.8 m stainless-steel tubing packed with 10% Carbowax 20M on Chromasorb W-HP (80/100 mesh). Only the first column was used for routine analyses. For detection an FID was used, and the chromatographic conditions were isothermal at 90°C, with helium used as the carrier gas.

Natural Gas Analyses: Gas Chromatograph (NGA)

The modified NGA is a modified multivalve, multicolumn gas chromatograph equipped with both a thermal conductivity detector (TCD) and an FID. The instrument is interfaced to the HP Chemstation software integration and processing package. This GC analysis system consists of automatic valve switching to direct flows through various sample loops and columns. Three GC columns were used sequentially to provide a rapid partitioning and measurement of N_2, O_2, CO_2, H_2S, CS_2, and C_1 through C_7 hydrocarbons. The modified NGA employs a multicolumn system composed of a 0.32 cm × 1.8 m stainless-steel column packed with a Porapak T (50/80 mesh) in line with a 0.32 cm × 0.9 m column packed with Molecular Sieve 13× (60/80 mesh), a 0.32 cm × 1.8 m stainless-steel column packed with 80/100 mesh Haysep (acid washed), and a 60 m × 0.32 mm capillary column coated with a 1-µm film thickness of DB-1 (J and W, Inc.). Samples were introduced by means of a 0.5-cm^3 sample loop with an automatic sample backflush. The chromatographic separation on the TCD portion of the NGA system was carried out isothermally at 80°C, whereas the hydrocarbon separation on the FID portion was carried out by programming from 80° to 100°C at 8°C/min and then to 200° at 30°C/min. Helium was used as the carrier gas. The TCD injector and detector temperatures were 80°C and 150°C, respectively, and the corresponding temperatures for FID were 150°C and 250°C, respectively. Chromatographic response was calibrated against preanalyzed standards and the gas contents are reported in parts per million by volume.

A more quantitative evaluation of the gas concentration was possible by relating the headspace volume and gas composition to the weight of the sediment sample. Weights were determined by difference with the preweighed vials.

Carbon, Nitrogen, and Sulfur Analyses

Sediments were analyzed aboard ship for inorganic carbon and for total nitrogen, carbon, and sulfur. The total organic carbon content (TOC) of the sediments was determined by the difference between carbonate carbon (inorganic carbon) and the total carbon value. A detailed description of the Coulometrics instrument and procedure can be found in the “Inorganic Geochemistry” section (ODP Leg 146, Westbrook, Carson, Musgrave, et al., 1994).

Briefly, the analyses were carried out on sediment left in squeeze cakes, from which most of the interstitial waters had been removed by pressing in the titanium squeezers. The sediments were freeze-dried before being analyzed.

Total inorganic carbon (TIC) was determined using a Coulometrics 5011 coulometer equipped with a System 140 Carbonate Carbon Analyzer. Approximately 5 mg of ground and weighed sediment was reacted in a 2N HCl solution. The liberated CO_2 was titrated in a monoethanolamine solution with a color indicator, while the change in light transmittance was monitored with a photodetector cell. Carbonate percentage is calculated from the inorganic carbon (IC) content, assuming that all carbonate occurs as calcium carbonate as follows:

$$CaCO_3 = IC \times 8.33.$$

Total nitrogen, carbon, and sulfur were determined using a NA 1500 Carlo Erba NCS analyzer. Bulk samples were combusted at 1000°C in an oxygen atmosphere with the addition of vanadium pentoxide, converting organic and inorganic carbon into CO_2 and sulfur into SO_2. These gases along with nitrogen were then separated by gas chromatography and measured with a thermal conductivity detector. TOC content is calculated by the difference between TC and inorganic carbon (IC):

$$TOC = TC - IC.$$

INORGANIC GEOCHEMISTRY

Methodologies used are from the routine program on *JOIDES Resolution* as described by Gieskes et al. (1991).

REFERENCES

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