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# Data report: carbon stable isotope ratios of dissolved inorganic carbon in interstitial waters from IODP Expedition 303 Sites U1305, U1306, and U1307 (Eirik Drift)<sup>1</sup>

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## Abstract

In this data report, we present carbon stable isotope ( $\delta^{13}\text{C}$ ) analyses of dissolved inorganic carbon (DIC) in interstitial water samples from Integrated Ocean Drilling Program Expedition 303 Sites U1305, U1306, and U1307 on the Eirik Drift in the Labrador Sea. A total of 84 interstitial water samples extracted on board were analyzed postcruise in this study. The low- to intermediate-resolution (from ~3 to 30 m interval) data allow us to identify downhole trends in  $\delta^{13}\text{C}_{\text{DIC}}$ . Our results show a downhole decreasing trend in  $\delta^{13}\text{C}_{\text{DIC}}$  in the upper ~50–80 meters below seafloor (mbsf) at the three sites studied. The most depleted  $\delta^{13}\text{C}_{\text{DIC}}$  values occur at 51.4, 85.8, and 79.2 mbsf at Sites U1305, U1306, and U1307, respectively. Below the depths of  $\delta^{13}\text{C}_{\text{DIC}}$  minima,  $\delta^{13}\text{C}_{\text{DIC}}$  values generally increase toward the bottom of the cored interval.

## Introduction

Carbon stable isotopic composition ( $\delta^{13}\text{C}$ ) of dissolved inorganic carbon (DIC) in interstitial waters, as well as concentrations of other pore fluid chemical species, have been widely used to infer rates of subseafloor metabolic activities and carbonate diagenesis (Claypool et al., 2006; D'Hondt et al., 2004). Shipboard analyses of interstitial water chemistry and headspace gas at the Eirik Drift sites exhibit downhole sulfate and methane concentration profiles that indicate the presence of the sulfate/methane interface (see the “[Expedition 303 summary](#)” chapter). To further explore chemical signatures of subseafloor metabolisms and early diagenesis, we measured  $\delta^{13}\text{C}$  of DIC in interstitial waters from the Eirik Drift Sites U1305, U1306, and U1307 (Fig. [F1](#)). This report provides results of our shore-based analysis of interstitial waters from the three sites.

## Methods and materials

Samples for interstitial waters were extracted from either (1) 5 cm long whole-round sediment sections that were cut and capped immediately after core retrieval on deck or (2) small plug sediment samples of ~10 cm<sup>3</sup> taken with a syringe from the ends of cut sections, also immediately after core retrieval. In the shipboard laboratory, whole-round sediment samples were removed

<sup>1</sup>Ennyu, A., and Malone, M.J., 2009. Data report: carbon stable isotope ratios of dissolved inorganic carbon in interstitial waters from IODP Expedition 303 Sites U1305, U1306, and U1307 (Eirik Drift). In Channell, J.E.T., Kanamatsu, T., Sato, T., Stein, R., Alvarez Zarikian, C.A., Malone, M.J., and the Expedition 303/306 Scientists, *Proc. IODP, 303/306*: College Station, TX (Integrated Ocean Drilling Program Management International, Inc.). doi:10.2204/iodp.proc.303306.207.2009

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from the core liner and the outside surfaces of the samples were carefully scraped off with spatulas to minimize potential contamination with drill fluids. Plug sediment samples were expelled from the syringe directly into squeezers. Fluids were extracted from sediments in Manheim titanium squeezers at ambient temperature with a Carver hydraulic press (Manheim and Sayles, 1974). Interstitial water samples discharged from the squeezer were passed through 0.45  $\mu\text{m}$  polyethersulfone membrane filters and collected in plastic syringes. Aliquots for shore-based inorganic carbon isotope analysis were poisoned with saturated mercuric chloride solution and stored in 2  $\text{cm}^3$  glass vials sealed with butyl rubber septa and plastic screw caps. The glass vials were kept refrigerated until they were processed for isotopic analysis at a shore-based laboratory.

Interstitial water samples were analyzed for  $\delta^{13}\text{C}$  content of DIC at Oregon State University using the method described in Torres et al. (2005). In summary, this method loads ~0.15–0.7 mL of sample into a Thermo Fisher GasBench-II headspace sampler, which is online with Thermo Fisher Delta V Plus isotope ratio mass spectrometer. Replicate measurements during these analyses indicate precision to be better than  $\pm 0.15\text{‰}$  ( $1\sigma$ ).

## Results

Results of carbon stable isotope analyses of interstitial waters from Sites U1305, U1306, and U1307 are shown in Figure F2 and are reported in Table T1. Our results indicate a downhole decreasing trend in  $\delta^{13}\text{C}_{\text{DIC}}$  in the upper ~50–80 meters below seafloor (mbsf) at the three sites studied. The most depleted  $\delta^{13}\text{C}_{\text{DIC}}$  value of approximately  $-34.1\text{‰}$  is recorded at 79.15 mbsf at Site U1307. At Sites U1305 and U1306, the  $\delta^{13}\text{C}_{\text{DIC}}$  minima occur at 51.4 and 85.8 mbsf, respectively. Below the depth of  $\delta^{13}\text{C}_{\text{DIC}}$  minima, the  $\delta^{13}\text{C}_{\text{DIC}}$  values generally increase to approximately  $-3\text{‰}$  toward the bottom of the cored interval.

Also presented in Figure F2 are dissolved sulfate ( $\text{SO}_4^{2-}$ ) and headspace methane ( $\text{C}_1$ ) concentration profiles from shipboard measurements (see the “Site U1305,” “Site U1306,” and “Site U1307” chapters). The depth at which the most depleted  $\delta^{13}\text{C}_{\text{DIC}}$  value is recorded at each site typically coincides with the sulfate/methane interface inferred from the shipboard  $\text{SO}_4^{2-}$  and  $\text{C}_1$  profiles (Fig. F2).

## Acknowledgments

We thank Marta Torres for comments that helped to improve the manuscript and Alan Mix for providing the analyses. This research used samples and data provided by the Integrated Ocean Drilling Program (IODP). Funding for this research was provided by U.S. Science Support Program to M.J.M. Travel funds were provided to A.E. by Japan Agency for Marine-Earth Science and Technology (JAMSTEC) to participate in Expedition 303 and the 2nd postcruise meeting.

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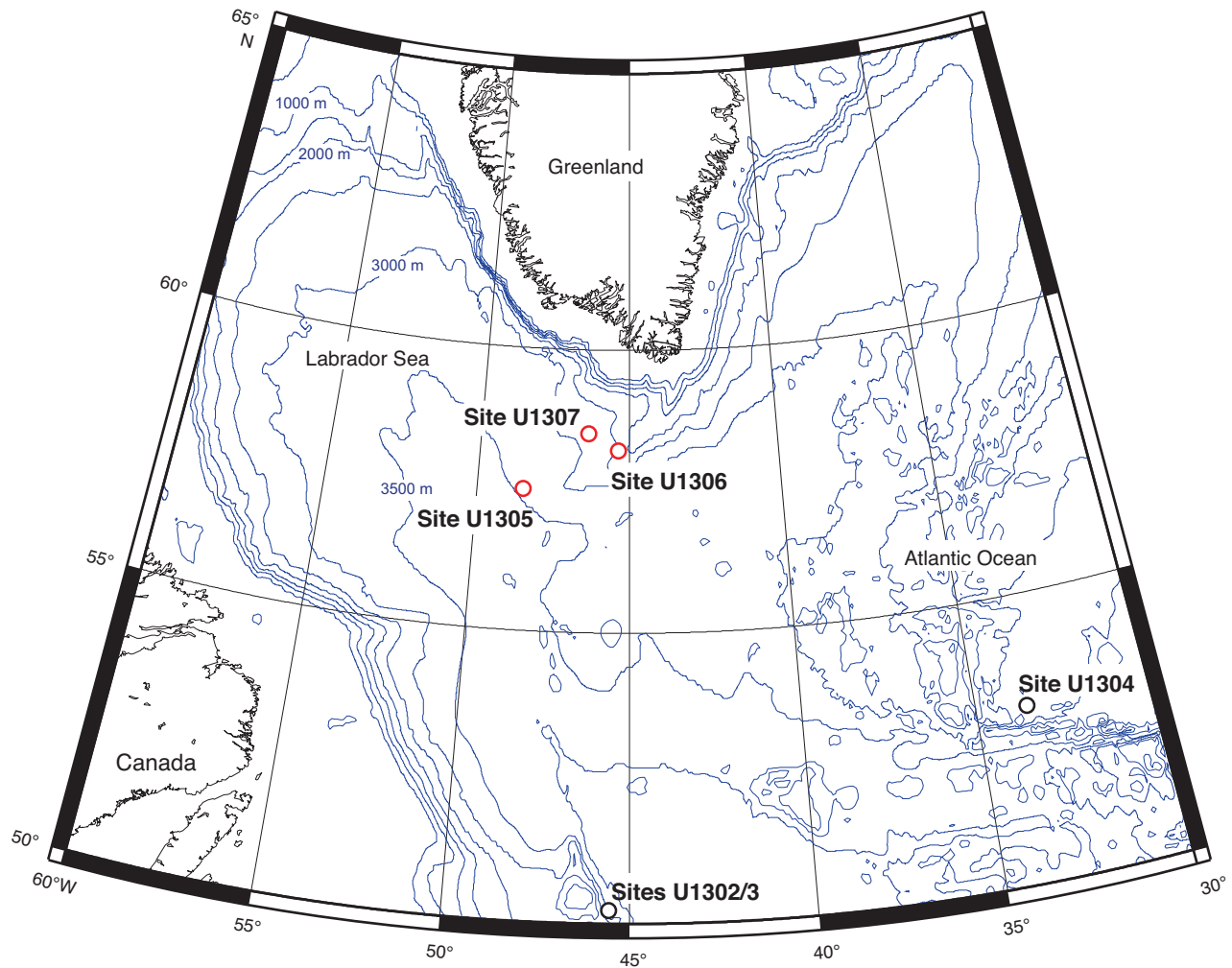
**Initial receipt:** 9 May 2008

**Acceptance:** 30 March 2009

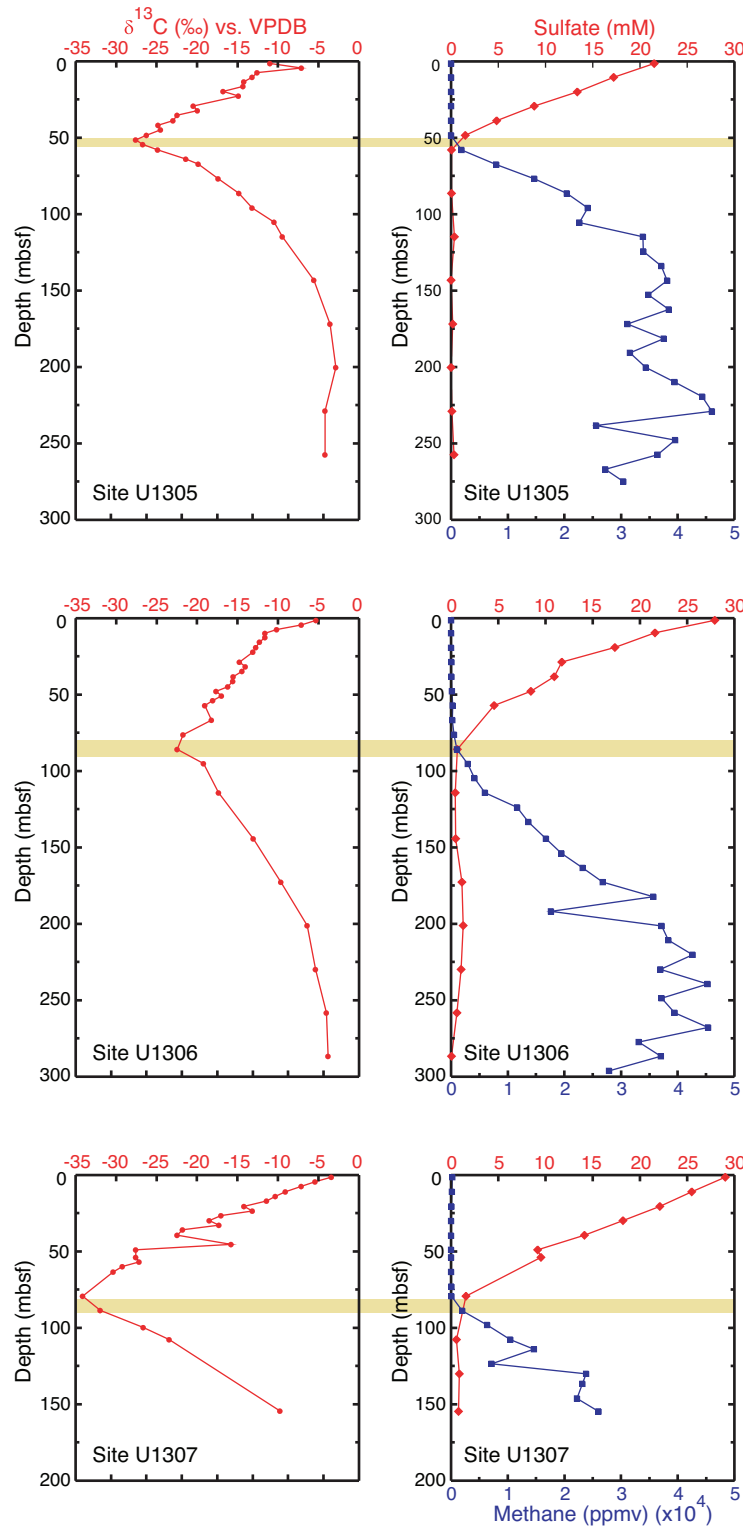
**Publication:** 19 June 2009

**MS 303306-207**

**Figure F1.** Map showing locations of sites used in this study that were drilled during Expedition 303. Red circles = sites investigated for interstitial water carbon stable isotopes in this report. Bathymetric contours are in meters below sea level. This map was generated using Generic Mapping Tools (Wessel and Smith, 1998).



**Figure F2.** Carbon stable isotope ( $\delta^{13}\text{C}$ ) records of dissolved inorganic carbon (DIC) from Sites U1305, U1306, and U1307. Also plotted are sulfate and methane concentration profiles measured on board during Expedition 303 (see the “Site U1305,” “Site U1306,” and “Site U1307” chapters). Light khaki bars = sulfate/methane interfaces inferred from the downhole chemical compositions at each site. The  $\delta^{13}\text{C}$  data are reported in Table T1. VPDB = Vienna Pee Dee belemnite standard.



**Table T1.** Carbon stable isotopes of pore water dissolved inorganic carbon, Sites U1305, U1306, and U1307. (See table notes.)

| Core, section,<br>interval (cm) | Depth<br>(mbsf) | $\delta^{13}\text{C}$<br>(‰ VPDB) | Core, section,<br>interval (cm)                             | Depth<br>(mbsf) | $\delta^{13}\text{C}$<br>(‰ VPDB) |
|---------------------------------|-----------------|-----------------------------------|---|-----------------|-----------------------------------|
| 303-U1305A-                     |                 |                                   | 5H-6, 145–150   |                 |                                   |
| 1H-1, 145–150                   | 1.45            | -10.99                            | 6H-1, 145–150   | 44.92           | -16.20                            |
| 1H-3, 145–150                   | 4.45            | -7.11                             | 6H-1, 145–150   | 47.75           | -17.66                            |
| 1H-5, 145–150                   | 7.45            | -12.58                            | 6H-3, 145–150   | 50.75           | -16.96                            |
| 2H-1, 145–150                   | 10.35           | -13.21                            | 6H-5, 145–150   | 53.75           | -18.06                            |
| 2H-3, 145–150                   | 13.35           | -14.22                            | 7H-1, 145–150   | 57.25           | -19.02                            |
| 2H-5, 145–150                   | 16.35           | -14.30                            | 8H-1, 145–150   | 66.75           | -18.23                            |
| 3H-1, 145–150                   | 19.85           | -16.77                            | 9H-1, 145–150   | 76.25           | -21.72                            |
| 3H-3, 145–150                   | 22.85           | -14.88                            | 10H-1, 145–150  | 85.75           | -22.44                            |
| 4H-1, 145–150                   | 29.35           | -20.45                            | 11H-1, 145–150  | 95.25           | -19.18                            |
| 4H-3, 145–150                   | 32.35           | -19.95                            | 13H-1, 145–150  | 114.25          | -17.33                            |
| 4H-5, 145–150                   | 35.35           | -22.44                            | 17H-1, 145–150  | 144.25          | -13.06                            |
| 5H-1, 145–150                   | 38.85           | -22.95                            | 20H-1, 145–150  | 172.74          | -9.64                             |
| 5H-3, 145–150                   | 41.85           | -24.82                            | 23H-1, 145–150  | 201.25          | -6.38                             |
| 5H-5, 145–150                   | 44.85           | -24.52                            | 26H-1, 145–150  | 229.75          | -5.38                             |
| 6H-1, 145–150                   | 48.35           | -26.23                            | 29H-1, 145–150  | 258.25          | -4.02                             |
| 6H-3, 145–150                   | 51.35           | -27.56                            | 32H-1, 145–150  | 286.75          | -3.79                             |
| 6H-5, 145–150                   | 54.35           | -26.72                            | 303-U1307A-   |                 |                                   |
| 7H-1, 145–150                   | 57.85           | -24.85                            | 1H-1, 145–150   | 1.45            | -3.39                             |
| 7H-5, 145–150                   | 63.85           | -21.40                            | 1H-3, 145–150   | 4.45            | -5.40                             |
| 8H-1, 145–150                   | 67.35           | -19.83                            | 1H-5, 145–150   | 7.45            | -7.14                             |
| 9H-1, 145–150                   | 76.85           | -17.40                            | 2H-1, 145–150   | 10.95           | -9.10                             |
| 10H-1, 145–150                  | 86.35           | -14.84                            | 2H-3, 145–150   | 13.95           | -10.32                            |
| 11H-1, 145–150                  | 95.85           | -13.21                            | 2H-5, 145–150   | 16.95           | -11.39                            |
| 12H-1, 145–150                  | 105.35          | -10.48                            | 3H-1, 145–150   | 20.45           | -14.17                            |
| 13H-1, 145–150                  | 114.85          | -9.47                             | 3H-3, 145–150   | 23.45           | -13.14                            |
| 16H-1, 145–150                  | 143.35          | -5.59                             | 3H-5, 145–150   | 26.45           | -17.02                            |
| 19H-1, 145–150                  | 171.85          | -3.58                             | 4H-1, 145–150   | 29.95           | -18.47                            |
| 22H-1, 145–150                  | 200.35          | -2.85                             | 4H-3, 145–150   | 32.95           | -17.27                            |
| 25H-1, 145–150                  | 228.85          | -4.19                             | 4H-5, 145–150   | 35.95           | -21.79                            |
| 28H-1, 145–150                  | 257.35          | -4.17                             | 5H-1, 145–150   | 39.45           | -22.45                            |
| 303-U1306A-                     |                 |                                   | 5H-5, 145–150   | 45.42           | -15.81                            |
| 1H-1, 145–150                   | 1.45            | -5.32                             | 6H-1, 145–150   | 48.95           | -27.55                            |
| 1H-3, 145–150                   | 4.45            | -7.10                             | 7H-1, 145–150   | 53.95           | -27.56                            |
| 1H-5, 145–150                   | 7.45            | -10.16                            | 7H-3, 145–150   | 56.95           | -27.13                            |
| 2H-1, 145–150                   | 9.75            | -11.59                            | 7H-5, 145–150   | 59.95           | -29.19                            |
| 2H-3, 145–150                   | 12.75           | -11.62                            | 8H-1, 145–150   | 63.45           | -30.35                            |
| 2H-5, 145–150                   | 15.75           | -12.27                            | 10H-1, 145–150  | 79.15           | -34.09                            |
| 3H-1, 145–150                   | 19.25           | -12.74                            | 11H-1, 145–150  | 88.65           | -31.94                            |
| 3H-3, 95–100                    | 22.25           | -13.09                            | 12H-2, 145–150  | 99.65           | -26.63                            |
| 4H-1, 145–150                   | 28.75           | -14.75                            | 13H-1, 145–150  | 107.65          | -23.43                            |
| 4H-3, 145–150                   | 31.75           | -14.03                            | 16H-3, 145–150  | 130.15          | ND                                |
| 4H-5, 145–150                   | 34.75           | -14.42                            | 19H-1, 145–150  | 154.55          | -9.75                             |
| 5H-1, 145–150                   | 38.25           | -15.53                            | Notes: VPDB = Vienna Peedee belemnite. ND = not determined. |                 |                                   |
| 5H-3, 145–150                   | 41.25           | -15.56                            |   |                 |                                   |