21. CARBON AND CARBONATE ANALYSES, LEG 38

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Leg 38 $CaCO_3$ samples were collected onboard ship in 3-cc vials at the time the core was split open. On shore the samples were dried and ground to a homogeneous powder. The ground sediment was redried at 105°-110°C and two samples from each vial. a 0.1-g and 0.5-g sample, were then weighed into LECO crucibles. The 0.5-g sample was acidified with 10% HCl, washed with distilled H_2O, redried and analyzed for acid insoluble (organic) carbon using a LECO WR-12 analyzer. The 0.1-g sample was treated only with distilled water to cake the sample before analysis for total carbon. If the results showed less than 10% $CaCO_3$, an additional 0.5-g sample was analyzed for greater accuracy. The $CaCO_3$ percentages were calculated as follows: (% total C - % organic C) × 8.33 = % $CaCO_3$. Although other carbonates may be present, all acid-soluble carbon was calculated as $CaCO_3$. All weights are given in weight percent.

Detailed descriptions of technique and theory are in Volume 4 of the Initial Reports of DSDP (Bader, Gerard, et al., 1970).

For control purposes a sediment standard was made up from Deep Sea Drilling material and analyzed for total carbon at predetermined intervals with the regular samples. The carbonate content of these standards was determined by commercial laboratories.[1] Listed below are the statistical data for these standards.

DSDP Std.	No. of Samples	Totals as $CaCO_3$ (%)	Standard Deviation (%)	Maximum Range (%)
7	13	51.13	0.39	1.33
9	23	26.79	0.28	1.13

These data indicate the precision of the mechanical aspects of LECO analysis and do not necessarily reflect the precision of the total analytical procedure. This may be affected by factors such as sample homogeneity or contamination during sample preparation.

The carbon determinations were made using a LECO carbon analyzer which is described in detail in the LECO manual.

The LECO WR-12 selectively absorbs CO_2 on a molecular sieve. Later, through heating, the CO_2 is released and measured by its thermal conductivity. A sample of known amount is placed in a ceramic vial to which is added one or more combustibles such as ... (copper and iron chips). The crucible containing the sample is placed into a LECO high-frequency induction furnace within a combustion tube through which O_2 is passed.

[1]Schwarzkopf Microanalytical Laboratory.

TABLE 1
Carbon and Carbonate Analyses, Leg 38

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Site 336				
2-4, 70	12.2	1.5	0.1	11
3-2, 110	19.1	1.4	0.9	4
5-1, 40	35.9	1.3	0.6	5
5-6, 40	43.4	0.9	0.6	3
8-1, 30	64.4	0.5	0.4	1
8-5, 86	71.0	0.3	0.3	0
9-2, 78	75.8	0.2	0.2	0
9-5, 75	80.3	2.3	0.5	15
10-2, 69	94.7	0.2	0.2	0
11-3, 80	115.3	0.2	0.2	0
12-2, 83	132.8	0.3	0.3	0
15-1, 80	169.3	0.9	0.2	6
16-5, 60	184.6	0.8	0.7	1
18-2, 30	198.8	0.3	0.3	0
18-3, 20	200.2	0.4	0.4	0
22-6, 0	243.0	0.4	0.4	0
23-3, 48	248.0	0.5	0.4	0
24-3, 90	258.3	0.5	0.4	1
25-3, 73	267.6	0.4	0.4	0
26-3, 78	277.3	0.3	0.3	0
27-3, 81	286.8	0.4	0.0	3
29-2, 70	313.2	0.4	0.0	4
30-1, 30	330.7	0.5	0.5	1
30-3, 46	333.9	0.5	0.5	0
30-3, 54	334.0	3.6	0.4	27
30-3, 60	334.0	7.3	0.2	59
35-3, 63	428.6	0.8	0.8	0
36-3; 18	437.7	0.9	0.7	1
36-6, 123	443.2	0.5	0.5	0
38-1, 138	468.9	0.1	0.1	0
39-1, 83	473.3	0.1	0.0	0
39-3, 79	476.3	0.1	0.0	0
39-5, 67	479.2	0.1	0.1	0
Site 337				
1-2, 44	1.9	3.4	0.2	27
1-5, 47	6.5	1.1	0.3	6
2-3, 22	12.5	0.9	0.1	6
3-2, 110	21.6	0.4	0.2	1
4-2, 50	30.0	0.2	0.2	0
5-2, 122	40.2	6.5	0.1	53
5-4, 125	43.3	0.1	0.0	1
6-2, 83	49.3	0.1	0.1	0
6-2, 101	49.5	0.1	0.0	0
10-3, 39	88.6	0.1	0.0	1
11-3, 99	98.5	0.1	0.0	1
12-3, 30	107.3	0.1	0.0	1
Site 338				
1-1, 132	1.3	0.9	0.5	3
1-3, 78	3.8	1.8	0.2	13
2-3, 119	13.7	1.9	0.3	13
3-2, 105	21.6	1.1	0.8	2
4-4, 129	34.3	1.9	0.2	14
5-5, 86	44.9	0.3	0.2	1
6-5, 50	54.0	0.3	0.2	1
7-1, 105	67.6	1.4	1.3	1

TABLE 1 – *Continued*

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Site 338 – *Continued*				
13-5, 50	130.0	1.1	0.9	2
15-3, 75	146.3	1.4	1.2	2
17-4, 39	166.4	1.4	1.1	2
18-1, 79	171.8	0.8	0.7	1
19-1, 137	181.9	1.0	0.8	2
19-5, 73	187.2	0.6	0.6	0
20-1, 142	191.4	1.2	0.4	7
20-5, 75	196.8	0.9	0.5	3
21-1, 125	200.8	0.3	0.4	0
22-1, 74	209.7	5.7	0.4	44
22-4, 85	214.4	2.0	0.5	13
22-6, 138	217.9	0.4	0.4	0
23-1, 73	219.5	4.3	0.2	34
23-6, 107	227.4	4.7	0.2	38
24-3, 60	231.6	6.1	0.1	50
26-3, 140	251.4	0.1	0.1	0
27-5, 89	263.4	0.6	0.5	1
30-4, 61	290.1	0.2	0.2	0
31-1, 80	295.3	0.4	0.4	0
32-4, 27	308.8	0.7	0.6	2
33-4, 50	318.5	0.5	0.4	1
35-2, 45	334.5	0.6	0.4	1
37-2, 64	353.6	0.7	0.5	2
Site 339				
1-5, 100	7.0	2.1	0.5	14
2-2, 49	10.0	2.2	0.5	14
2-4, 89	13.4	1.2	0.4	7
4-3, 69	30.7	2.1	0.6	12
5-2, 76	38.8	1.4	0.6	6
6-2, 90	48.4	2.0	0.7	10
7-2, 100	58.0	0.6	0.4	2
8-2, 60	67.1	1.5	0.6	8
8-5, 70	71.7	0.4	0.4	1
10-3, 95	88.0	0.3	0.0	3
12-3, 60	106.6	0.6	0.6	0
Site 340				
1-3, 93	3.9	2.9	0.4	21
6-2, 27	49.3	0.3	0.3	0
8-4, 50	71.5	0.2	0.2	0
9-3, 15	79.2	0.3	0.3	0
Site 341				
1-2, 8	1.9	0.9	0.3	5
6-2, 70	49.7	1.3	0.6	6
7-2, 30	58.8	0.6	0.5	1
8-4, 40	71.4	1.4	0.6	7
10-4, 69	90.7	1.5	0.4	9
11-1, 75	95.8	1.4	0.4	8
12-4, 10	109.1	1.5	0.4	9
16-1, 99	162.5	1.2	0.3	8
20-1, 120	238.9	1.3	0.3	8
21-2, 30	258.8	2.1	0.4	15
23-6, 55	312.3	0.4	0.4	1
16-3, 8	355.0	4.7	0.0	39
27-3, 82	374.8	2.0	0.0	17
28-3, 79	393.7	1.5	0.0	13
29-1, 120	400.5	1.7	0.0	14
29-5, 52	405.8	3.3	0.0	28
32-2, 113	430.4	1.6	0.0	13
33-3, 99	441.4	1.7	1.5	2
34-3, 80	450.6	1.8	1.8	0
Site 342				
1-4, 80	5.5	1.5	0.2	10

TABLE 1 – *Continued*

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Site 342 – *Continued*				
2-2, 90	39.9	0.3	0.2	0
3-2, 110	87.6	0.3	0.3	0
4-2, 84	125.3	0.7	0.0	6
6-2, 129	144.8	1.1	0.3	7
Site 343				
3-2, 75	52.8	1.7	0.5	10
4-2, 75	100.3	0.5	0.3	2
4-4, 70	103.2	2.4	0.1	19
5-2, 78	147.8	0.8	0.0	6
6-1, 110	194.1	1.1	0.0	10
7-3, 60	206.5	0.4	0.3	1
8-3, 30	215.3	1.2	0.4	7
10-2, 40	232.9	0.9	0.5	3
11-2, 70	242.7	0.4	0.3	0
15-2, 110	271.6	1.0	0.8	2
16-1, 123	279.7	1.2	0.0	10
Site 344				
2-2, 20	3.2	1.2	0.9	2
3-3, 50	14.5	1.3	1.1	2
5-3, 20	33.5	1.0	0.2	6
6-3, 110	43.6	0.8	0.6	1
7-4, 69	54.2	0.9	0.4	4
8-4, 55	63.6	1.1	1.0	1
9-3, 74	72.2	0.6	0.6	0
10-3, 9	80.6	0.6	0.6	0
12-3, 20	99.7	0.5	0.5	0
13-1, 60	106.6	0.6	0.6	0
14-1, 60	116.1	1.0	0.9	1
15-3, 60	128.9	1.2	1.0	2
16-2, 60	136.6	0.9	0.7	2
17-1, 70	144.7	1.1	0.8	2
18-1, 70	154.2	0.9	0.8	1
21-2, 70	184.2	1.5	1.2	2
23-2, 70	203.2	0.9	0.0	8
24-1, 69	211.2	0.9	0.8	1
25-1, 65	220.7	1.1	0.9	1
26-1, 85	230.4	1.2	1.1	1
27-2, 50	241.0	1.4	1.1	2
28-2, 60	260.1	1.1	0.9	2
30-1, 120	287.7	1.7	0.5	10
32-2, 74	345.7	1.0	0.9	1
33-2, 48	374.0	1.7	1.5	1
Site 345				
1-2, 60	2.1	2.4	0.3	17
2-3, 70	11.7	1.6	1.3	3
3-2, 30	19.3	4.6	0.2	36
4-2, 69	29.2	0.4	0.2	2
5-2, 130	39.3	0.7	0.7	0
6-2, 50	57.5	0.2	0.2	0
7-2, 82	76.8	0.3	0.3	0
8-3, 81	97.3	0.4	0.4	0
9-3, 85	116.4	0.4	0.4	0
10-3, 40	135.4	0.4	0.4	0
11-3, 40	163.8	0.4	0.4	0
14-3, 10	248.6	0.5	0.5	0
16-4, 35	335.9	0.6	0.0	5
17-3, 68	372.7	0.7	0.4	3
19-2, 75	447.8	0.4	0.3	1
20-3, 20	486.2	0.4	0.3	0
21-3, 8	524.1	0.4	0.3	0
23-4, 35	602.4	0.4	0.4	1
24-3, 35	638.8	0.4	0.3	1
25-2, 123	675.9	0.7	0.4	2

TABLE 1 – *Continued*

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Site 345 – *Continued*				
27-2, 58	713.1	1.6	0.1	13
28-3, 52	724.5	0.3	0.3	0
30-5, 80	746.3	5.2	0.1	43
Site 346				
1-3, 65	3.7	1.1	0.1	8
2-3, 91	10.4	0.2	0.2	0
3-5, 30	22.3	0.7	0.4	2
4-3, 30	28.8	0.5	0.3	1
5-2, 35	36.9	0.2	0.2	0
6-3, 57	48.1	0.4	0.3	0
7-4, 121	59.7	0.4	0.4	0
8-3, 40	66.9	0.3	0.2	0
9-4, 64	78.1	0.9	0.0	7
10-3, 79	86.3	1.0	0.0	8
11-3, 77	95.8	0.8	0.6	1
12-3, 77	105.3	1.0	0.0	8
13-2, 76	113.3	1.1	0.0	9
14-2, 30	122.3	0.8	0.8	0
14-4, 70	125.7	0.4	0.4	0
15-2, 76	132.3	4.5	0.3	35
17-1, 72	149.7	0.3	0.3	0
18-3, 132	162.8	0.4	0.4	0
19-1, 104	169.0	1.2	0.4	6
20-2, 83	179.8	0.6	0.6	0
Site 347				
1-1, 118	1.2	1.6	0.2	12
1-2, 30	1.8	3.1	0.2	25
1-3, 128	4.3	4.3	0.4	33
2-2, 56	123.1	0.5	0.5	0
3-2, 58	130.1	0.5	0.5	0
Site 348				
1-1, 30	0.4	4.8	0.1	39
1-1, 76	0.9	4.2	0.2	34
1-1, 126	1.4	2.6	0.1	20
1-2, 26	1.9	1.3	0.2	10
1-2, 50	2.2	1.7	0.1	13
1-3, 27	3.4	1.8	0.2	13
1-3, 78	3.9	0.8	0.2	4
1-3, 82	4.0	0.9	0.2	6
1-4, 26	4.9	0.8	0.1	6
1-4, 76	5.4	3.8	0.1	31
1-5, 26	6.4	0.4	0.1	2
1-5, 78	6.9	0.8	0.6	2
1-6, 26	7.9	0.6	0.2	3
1-6, 76	8.4	0.4	0.2	1
2-1, 80	9.8	2.7	0.2	21
2-1, 116	10.2	0.9	0.1	6
2-1, 126	10.3	1.1	0.2	8
2-2, 26	10.8	0.3	0.2	1
2-2, 45	11.0	1.6	0.2	11
2-2, 126	11.8	0.7	0.5	2
2-3, 32	12.3	0.5	0.3	1
2-3, 82	12.8	0.4	0.3	1
2-4, 26	13.8	1.4	0.5	8
2-4, 76	14.3	1.0	0.7	3
2-4, 134	14.8	0.8	0.3	4
3-1, 26	18.9	0.4	0.3	1
3-1, 56	19.2	0.3	0.3	0
3-1, 125	19.9	0.4	0.2	2
3-2, 34	20.4	0.3	0.3	0
3-2, 82	20.9	0.3	0.2	1
3-2, 126	21.4	0.3	0.2	0
3-3, 26	21.9	0.4	0.4	0

TABLE 1 – *Continued*

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Site 348 – *Continued*				
3-3, 73	22.3	0.3	0.3	0
3-3, 126	22.9	0.4	0.3	0
3-4, 26	23.4	0.4	0.3	0
3-4, 82	23.9	0.5	0.4	1
3-4, 132	24.4	0.3	0.3	0
3-5, 26	24.9	0.3	0.2	0
3-5, 76	25.4	0.4	0.3	1
3-5, 118	25.8	0.3	0.2	0
3-6, 26	26.4	0.2	0.2	0
3-6, 82	26.9	0.4	0.4	0
4-1, 76	38.3	0.4	0.4	0
4-2, 26	39.3	0.4	0.3	0
4-2, 76	39.8	0.3	0.3	0
4-2, 123	40.2	0.2	0.2	0
4-3, 71	41.2	0.3	0.3	0
4-3, 126	41.8	0.2	0.2	0
5-2, 26	58.3	0.3	0.3	0
5-2, 76	58.8	0.4	0.3	0
6-2, 0	67.5	0.3	0.2	1
6-2, 0	67.5	0.3	0.2	1
6-3, 0	69.0	0.3	0.3	0
6-4, 0	70.5	0.3	0.2	0
6-5, 0	72.0	1.9	0.3	14
7-3, 0	78.5	0.3	0.3	0
8-3, 0	97.5	0.3	0.3	0
9-3, 0	116.5	0.3	0.2	0
11-3, 0	154.5	0.4	0.0	3
12-3, 10	164.1	0.5	0.4	1
14-4, 10	194.5	0.4	0.4	0
15-2, 45	210.5	0.6	0.6	0
16-3, 25	231.2	0.6	0.5	0
19-5, 15	271.7	0.2	0.2	0
20-2, 45	277.3	0.3	0.2	1
21-2, 90	287.4	0.3	0.2	0
23-3, 70	326.2	0.4	0.3	0
24-3, 56	345.4	0.7	0.6	1
25-4, 106	376.1	0.7	0.6	1
26-3, 14	401.6	0.7	0.6	1
27-2, 42	429.4	0.7	0.6	1
29-4, 16	489.2	0.9	0.8	1
30-3, 39	506.4	0.7	0.6	0
31-3, 68	516.6	0.4	0.3	0
Site 349				
1-3, 40	3.4	1.6	0.3	11
2-3, 44	57.2	0.3	0.2	0
3-2, 94	93.9	0.4	0.4	0
4-1, 85	101.9	0.3	0.3	0
5-3, 65	114.5	0.2	0.2	0
6-3, 60	123.6	1.3	1.0	2
7-3, 100	133.5	1.0	0.4	5
9-2, 37	150.4	1.0	1.0	0
10-4, 61	191.6	1.0	0.7	3
10-5, 7	192.6	1.1	0.9	1
11-2, 56	226.6	0.7	0.6	1
12-5, 128	269.8	0.9	0.7	2
13-3, 123	314.2	0.4	0.3	0
Site 350				
1-2, 65	2.2	3.5	0.1	28
2-3, 56	30.6	0.4	0.3	1
Hole 352				
1-1, 126	1.3	2.1	0.1	16
1-2, 126	2.8	1.3	0.1	10
1-3, 84	3.8	1.1	0.1	8

TABLE 1 – *Continued*

Sample (Interval in cm)	Depth (m)	Total Carbon	Organic Carbon	$CaCO_3$
Hole 352 – *Continued*				
1-4, 84	5.3	1.5	0.1	11
1-5, 84	6.8	1.7	0.2	12
1-6, 82	8.3	1.0	0.2	7
2-1, 84	9.3	0.7	0.2	4
2-3, 130	12.8	1.2	0.2	9
2-4, 60	13.6	0.9	0.2	6
2-5, 40	14.9	0.6	0.2	3
4-2, 93	30.3	0.8	0.3	4
4-3, 87	31.7	0.3	0.2	1
4-4, 84	33.2	0.4	0.1	2
4-5, 87	34.7	0.9	0.2	6
4-6, 84	36.2	0.4	0.2	2
5-2, 63	39.1	0.4	0.1	2
Hole 352A				
1-1, 142	95.4	6.3	0.1	52
1-2, 84	96.3	5.0	0.1	41
2-1, 129	104.8	5.3	0.1	43
3-1, 63	113.6	0.7	0.1	5
3-2, 71	115.2	7.1	0.1	58

The carbon in the sample is converted to CO_2 at temperatures in excess of 1000°C. Metal oxides either remain in the crucible or are filtered out in a series of dust traps while sulfur gases are absorbed in a trap containing manganese dioxide. A heated catalyst (containing rare earth and copper oxides) converts any CO to CO_2. Moisture is removed by an anhydrone trap.

Oxygen carries the formed CO_2 to a molecular sieve trap at room temperature when it is collected. Following collection of the CO_2, the trap is heated to approximately 600°C to release it. The second oxygen carrier system is directed through a system of solenoids at a flow rate of 500 cc/min to a measuring thermistor. The output of the thermal conductivity cell is integrated and read directly as %C on an electronic digital voltmeter. With pure oxygen in the system the thermal conductivity cell is balanced to yield 0.000 output.

The temperatures of the thermistors are always higher than ambient oven temperatures in which the cell is located. This virtually eliminates normal room temperature variations.

The two thermistors of the conductivity cell are mounted in a metal block. When oxygen in one thermistor cavity is replaced by CO_2, which has a lower thermal conductivity than oxygen, that thermistor becomes hotter and lower in electrical resistance. This electrical resistance change is calibrated to read percent CO_2 in oxygen. For detailed electronics of model 761-100, see the LECO manual.

Refer to the Initial Reports of the Deep Sea Drilling Project, Volume 9 (Boyce and Bode, 1972) for further information regarding precision testing.

REFERENCES

Bader, R.G., Gerard, R.D., et al., 1970. Initial Reports of the Deep Sea Drilling Project, Volume 4: Washington (U.S. Government Printing Office).

Boyce, R.E. and Bode, G.W., 1972. Carbon and Carbonate Analysis, Leg 9, Deep Sea Drilling Project. *In* Hays, J.D., et al., Initial Reports of the Deep Sea Drilling Project, Volume 9: Washington (U.S. Government Printing Office), p. 797.