NGU Report 2001.069

Fluorine in sediments and porewaters in the subduction zone offshore Costa Rica, ODP 170.

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REPORT

Report no.: 2001.069 | ISSN 0800-3416 | Grading: Open

Title:
Fluorine in sediments and porewaters in the subduction zone offshore Costa Rica, ODP Leg 170

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County:

Commune:

Map-sheet name (M=1:250.000)

Map-sheet no. and -name (M=1:50.000)

Deposit name and grid-reference:

Number of pages:
Map enclosures:

Price (NOK): 40,-

Fieldwork carried out:	Date of report:	Project no.:	Person responsible:
16 Oct-17 Dec, 1996	29. juni 2001	272101	Jan T. Kramer

Summary:

The results presented here are based on samples collected by the author during ODP Leg 170, 16. October- 17. December, 1996, and subsequent analyses at the laboratories of the Geological Survey of Norway in Trondheim, Norway. The main objectives for thoroughly documenting the chemistry of the pore fluids and solids are:
1) to identify the distribution of key elements and isotope ratios between pore fluids and various solid phases,
2) to quantify the fluxes of these key elements and isotopes into the subduction zone, and
3) to utilize these data for evaluating geochemical and material mass balances, as well as constraining the importance of underplating (Kimura et al. 1997, p. 74). Ultimately it is the concentration data of the key elements in the sediments and the pore fluids which are necessary for answering the question of how much material is subducted. Fluorine in pore fluids and in solids is one of the key elements in this setting.
In the reference section, borehole 1039, sediments in the upper 130 m contains on average 4500 mg/kg F. The lower biogenic ooze from about 200-380 mbsf contains on average 900 mg/kg F. The gabbro at the base of the borehole contains about 4000 mg/kg F. An attempt to identify which minerals contain the F has been only partially successful. Grains containing 2400 to 6200 mg/kg f have been spotted on the SEM by wave-length dispersive XRF. It is evident that F is associated with detrital minerals diagnostic of the ash debris. These could be amphiboles, volcanic glass or tephra. However, F does also seem to be a component of the biogenic fraction in the lower section since it is only partially diluted by the carbonates. In borehole 1040 the upper 500 m contains about 3000 mg/kg F. A slight increase is observed below the decollement at 330-360 mbsf. Below 500 mbsf the average F-concentration drops to 700 mg/kg.
In the reference section, borehole 1039, pore fluids contain less than 1.0 mg/L F down to 160 mbsf. Between 160 mbsf and 320 mbsf there is an almost symmetric peak with maximum 2.0 mg/L F at 220 mbsf. A similar peak is identified at a stratigraphically equivalent position in borehole 1040 where the peak has a maximum of 2.7 mg/L F.
The consistently high concentration of F in the diatomaceous ooze is not matched by a similar higher concentration in the pore fluids. The maximum concentration of F in these are found in the biogenic zone, and the peak is more accentuated in borehole 1040 below the decollement than in the reference section in 1039 west of the trench. This could suggest a source of these waters in the subsurface to the east. The average flux of fluorine subduction in borehole 1040, sections U1, U2, and U3 is estimated to be 181, 193 and 136 g/yr/cm arc length, respectively.

Keywords: fluorine	sediments	porewaters
marine geology		

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1 INTRODUCTION

The results presented here are based on samples collected by the author during ODP Leg 170, 16. October- 17. December, 1996, and subsequent analyses at the laboratories of the Geological Survey of Norway in Trondheim, Norway. The stratigraphy of each borehole is described in detail in Kimura et al. 1997, and the exact position of the samples are given by their depth in meteres below the seafloor (mbsf).

The main objectives for thoroughly documenting the chemistry of the pore fluids and solids in a subduction zone are:

- to identify the distribution of key elements and isotope ratios between pore fluids and various solid phases,
- to quantify the fluxes of these key elements and isotopes into the subduction zone, and
- to utilize these data for evaluating geochemical and material mass balances, as well as constraining the importance of underplating (Kimura et al. 1997, p. 74).

The concentrations of the key elements in the sediments and pore fluids are needed to answer the question of how much material is subducted. Fluorine in the pore fluids and solids is one of those key elements in this setting.

Sediments and pore waters collected on Leg 170 and later analysed for fluorine, represent a reference section through the sedimentary section west of the subduction zone in borehole 1039 (430 m deep) and a section through the accretionary prism and the underlying subducted sediments east of the trench in borehole 1040 (660m deep)(Fig. 1).

2 METHODS

The concentration of F has been measured using an ion-selective electrode (Adriano & Doner, 1982) in 36 sediment and porewater samples from borehole 1039 and 65 sediment and porewater samples from borehole 1040 (Fig. 2, 3, 4 and 5). Details of this procedure are as follows:

1)Determination of fluoride in marine sediment samples using ion-selective electrode after digestion by alkali fusion.

a) Alkali fusion (after McQuaker & Gurney 1977, in Page et. al. 1982)
The samples were first freeze-dried and milled to a fine powder in an agate mill about 0.25 g sample was accurately weighed and transferred into a nickel crucible. The sample was wetted with a small amount of de-ionised water, 3.0 ml 17N NaOH added and the crucible placed in a drying oven at 150°C for approximately 1 hour, until the NaOH had solidified. The crucible was then transferred to a muffle furnace at 300°C and the temperature was gradually raised to 600°C. The sample-alkali mixture was allowed to fuse for 30 minutes at this temperature. The crucible was then removed from the muffle furnace and allowed to cool to room temperature. The NaOH cake was dissolved by adding 5 ml de-ionsed water and gentle warming. 4.0 ml concentrated HCl was then added , under stirring, to adjust the pH to 8 - 9, the pH being checked using pH paper. The acidified sample was then cooled, transferred to a 100 ml volumetric flask, diluted to 100 ml with water and filtered through "Schleicher & Schüll" black band filter paper. Acidification and filtration of the sample removes Al and Fe which

may be present and which would interfere with the subsequent determination of the fluoride using an ion selective electrode.

b) Determination of fluoride in the digested sample

A sample of 10 ml was added to 10 ml TISAB (Total Ionic Strength Adjusted Buffer) solution. The potential was read after stirring 10 minutes - as for water samples - and compared to a calibration curve prepared using the «single solution technique» (McQuaker & Gurney, 1977).

2)Determination of fluoride in porewater samples using ion-selective electrode

Determination of fluoride in the porewater samples was carried out using a modified version of the Norwegian standard method NS 4740 (NSF, 1975).

The water samples were thawed and diluted 1:10 with Milli-Q® water. A 10 ml diluted sample was added to 10 ml TISAB buffer solution, pH 5.4, prepared according to NS 4740*, and the potential read after stirring for 10 minutes on a magnetic stirrer. Fluoride concentration was determined by comparison of the potential reading with a standard curve, prepared using standard NaF solutions. Ten milliliters of the standard solution was added to 10 ml EDTA buffer solution, pH 5.4 and the potential was read after stirring for 10 minutes.

* TISAB buffer solution: 57 ml glacial acetic acid + 58 g NaCl in 500 ml H_2O, + 4.0g EDTA ($C_6H_{10}(N(CH_2COOH)_2)$) , pH adjusted to 5.4 with 5M NaOH, finally diluted to 1000 ml with water

NOTE:
NGU-Lab is not accredited for these methods.
According to NS 4740, the lower detection limit for determination of fluoride in water by the ion-selective electrode method used here, is 0.1 ppm. The method also quotes results from a ringtest where analyses of a synthetic water sample by 111 laboratories gave a relative error of 0.7%, with a relative standard deviation of 3.6%. NGU-Lab is currently carrying out tests to check the presision and accuracy of the method as performed in our labs.
With respect to the analysis of fluoride in sediment samples, earlier trials at NGU-lab using a very similar, but not identical, method, and standard soil samples, gave a lowest recovery of 47% (for a Canadian calcareous C-horizon soil with 6% Al_2O_3) and a highest recovery of 88% (for a Canadian podzolic soil. With 15% Al_2O_3).

3 FLUORINE IN SEDIMENTS

In the reference section, borehole 1039, the upper 130 m of diatomaceous ooze with ash and silty clay with ash, contains on average 4500 mg/kg F (Table 1). The lower biogenic ooze from about 200-380 mbsf contains on average 900 mg/kg F. The fluorine content is inversely correlated with the calcium carbonate content (Fig. 2 and Kimura et al. 1997). The gabbro at the base of the borehole contains about 4000 mg/kg F.

An attempt to identify which minerals contain the F has been only partially successful. Grains containing 2400 to 6200 mg/kg F have been spotted on the SEM by wave-length dispersive XRF (Fig. 6, 7, 8 and 9). It is evident that F is associated with detrital minerals diagnostic of the ash debris. These could be amphiboles, volcanic glass or tephra. However, F does also seem to be a component of the biogenic fraction in the lower section since it is only partially diluted by the carbonates. Authigenic carbonate fluorapatite, the primary mineral phase of sedimentary phosphorite, is common on the Peru continental margin (Froelich et al. 1983),

but has not been identified in the sediments offshore Costa Rica. However, F could be a minor element in fish remains.

In borehole 1040, the upper 500 m contains about 3000 mg/kg F (Tab.1 and Fig.3). A slight increase is observed below the decollement at 330-360 mbsf. Below 500 mbsf the average F-concentration drops to 700 mg/kg. The F-content is inversely related to the content of calcium carbonate.

4 FLUORINE IN POREWATERS

The concentration of F in sea water is 1.3 mg/L (Krauskopf, 1979). In the reference section, borehole 1039, pore fluids contain less than 1.0 mg/L down to 160 mbsf. Between 160 mbsf and 320 mbsf there is an almost symmetric peak with maximum 2.0 mg/L F at 220 mbsf. This peak is independent of the F content of the sediments. Calcium in the pore fluids increases in an overlapping interval with a peak at 280 mbsf (Kimura et al. 1997). Alteration of the brown mafic glass-rich crystal-vitric mafic ashes in the calcareous section is most likely responsible for the very high Ca-values in this portion of the sedimentary section (Kimura et al. 1997, p. 74). This could explain the relatively high concentration of F in the pore fluids as well. A similar peak is identified at a stratigraphically equivalent position in borehole 1040 where the peak has a maximum of 2.7 mg/L F. This could suggest a source of these waters in the subsurface to the east.

5 ESTIMATION OF FLUORINE SUBDUCTION FLUX

The average flux of fluorine subduction in borehole 1040, section U1, is estimated to be 181.3 g/yr/cm arc length (Tab. 2). Similar estimates for section U2 and U3 are 193 and 136 g/yr/cm arc length. Minimum and maximum estimates are 88 and 302 g/yr/cm arc length.

6 DISCUSSION AND CONCLUSIONS

The consistently high concentration of F in the diatomaceous ooze is not matched by a similar higher concentration in the pore fluids. The maximum concentration of F in these are found in the biogenic zone, and the peak is more accentuated in borehole 1040 below the decollement than in the reference section in 1039 west of the trench. This could suggest a source of these waters in the subsurface to the east. Further details on fluid flow in this area are discussed in Silver et al. 2000a.

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TABLES

Table 1. Concentration of F (mg/kg) in the different stratigraphic units.

Section	$F_{Average}$	$F_{Maximum}$	$F_{Minimum}$
1039 (U1)	4300	5000	3360
1039 (U2)	4980	6000	3960
1039 (U3)	880	1200	500
1040 (U1)	3320	3520	3000
1040 (U2)	3293	3760	2840
1040 (U3)	774	1720	500

Tab. 2. Estimated average fluorine subduction flux* in borehole 1040 for stratigraphic units U1, U2 and U3.

Sec.	C_{avg} (g/kg)	C_{max} (g/kg)	C_{min} (g/kg)	Conv. rate (cm/yr)	Thickness (cm)	Bulk density (g/cm3)	Water cont (wt%)	Porosity (vol%)	Density solids I (g/cm3)	Density solids II (g/cm^3)	Solids flux avg (g/yr/cm arc-length)	Solids flux max (g/yr/cm arc-length)	Solids flux min (g/yr/cm arc-length)	Fluorine flux avg. (g/yr/cm
1039 (U1)	4.300	5.000	3.360	8.2	4970	2	33	66	3,94	3,94	690	803	540	-
1039 (U2)	4.980	6.000	3.960	8.2	5340	2	33	66	3,94	3,94	859	1035	683	-
1039 (U3)	0.880	1.200	0.500	8.2	16650	1.95	33,5	66	3,74	3,79	455	621	259	-
1040 (U1)	3.320	3.520	3.000	8.2	4970	2	33	66	3,94	3,94	533	565	482	181
1040 (U2)	3.293	3.760	2.840	8.2	5340	2	33	66	3,94	3,94	568	648	490	193
1040 (U3)	0.774	1.720	0.500	8.2	16650	1.95	33,5	66	3,74	3,79	400	890	259	136

*) Procedure for estimating subduction flux in 1039 and 1040.

The calculation of the general dry weight mass flux (F_s) is done by the equations (1) or (2), depending on whether the water content (w_w) or the porosity (n) is known;

$$F_s = v * h * \rho_t * [1-(w_w/100)] \quad (1)$$

$$F_s = v * h * [1-(n/100)] * \rho_s \quad (2)$$

v = Convergence rate (cm yr^{-1})
h = Sediment thickness (cm)
ρ_t = Bulk density (g cm^{-3})
w_w = Water content (wt%)
n = Porosity (vol%)
ρ_s = Specific density of the solid material (g cm^{-3})

The specific density (ρ_s) of the solid part of the sediment is calculated from bulk density and porosity:

$$\rho_s = [\rho_t - (n/100)] / (1 - n/100) \quad (3)$$

The final result of equations (2) and (3) is:

$$F_s = v * h * [\rho_t - (n/100)] \quad (4)$$

As an example the data for the section 1040 (U1) are given; v = 8.2 cm yr^{-1}, h = 4970 cm, ρ_t = 2.0 g cm^{-3} and n = 66 vol% and the equations (4) is calculated:

F_s = 8.2 cm yr^{-1} * 4970 cm * [2.0 - (66/100)] g cm^{-3} = <u>54610 g yr^{-1} per cm arc length</u> (1)

The calculation of the flux of the chemical component x (F_x) is then given by the equation;

$$F_x = F_s * C_x \quad (4)$$

C_x = Concentration of the chemical component x (ppm =mg kg^{-1} dry weight)

Using the average fluorine-composition of section 1040 (U1) C_F = 3.32 g/kg the fluorine-flux (F_F) is calculated:

F_F = 54610 * 3.32 = <u>181.3 g yr^{-1} per cm arc length</u>

Tab. 3. Concentration of F (mg/kg) in sediments measured at various depths (mbsf) in borehole 1039.

1	6,44	170	1039	B	1	4480
2	23,85	170	1039	B	2	4480
3	28,36	170	1039	B	3	4200
4	36,35	170	1039	B	4	4200
5	45,41	170	1039	B	5	5000
6	55,35	170	1039	B	6	3360
7	64,83	170	1039	B	7	3960
8	74,36	170	1039	B	8	4560
9	83,86	170	1039	B	9	4400
10	93,33	170	1039	B	10	3960
11	102,85	170	1039	B	11	4240
12	118,96	170	1039	B	12	4720
13	128,56	170	1039	B	13	5280
14	136,76	170	1039	B	14	5680
15	147,85	170	1039	B	15	6000
16	157,46	170	1039	B	16	2120
17	168,38	170	1039	B	17	1480
18	177,87	170	1039	B	18	5000
19	187,7	170	1039	B	19	2240
20	197,01	170	1039	B	20	1120
21	206,79	170	1039	B	21	680
22	216,28	170	1039	B	22	880
23	226,21	170	1039	B	23	1120
24	245,28	170	1039	B	24	960
25	252,21	170	1039	B	25	1160
26	261,91	170	1039	B	26	1200
27	271,62	170	1039	B	27	1040
28	279,71	170	1039	B	28	600
29	289,47	170	1039	B	29	760
30	300,52	170	1039	B	30	1160
31	319,44	170	1039	B	31	760
32	332,32	170	1039	B	32	560
33	348,15	170	1039	B	33	1160
34	360,68	170	1039	B	34	<500
35	372,22	170	1039	B	35	missing
36	385,19	170	1039	C	36	640

Tab. 4. Concentration of F (mg/kg) in sediments measured at various depths (mbsf) in borehole 1040.

37	416,6	170	1040	C	37	3800
38	1,55	170	1040	C	38	3200
39	4,5	170	1040	C	39	3720
40	8,57	170	1040	C	40	3240
41	18,95	170	1040	C	41	3560
42	30,01	170	1040	C	42	4200
43	37,93	170	1040	C	43	3920
44	56,15	170	1040	C	44	2400
45	70,54	170	1040	C	45	3040
46	90,01	170	1040	C	46	2680
47	96,9	170	1040	C	47	3200
48	101,54	170	1040	C	48	3160
49	106,9	170	1040	C	49	3360
50	119,36	170	1040	C	50	3080
51	134,02	170	1040	C	51	3000
52	144,69	170	1040	C	52	3160
53	157,67	170	1040	C	53	3280
54	164,48	170	1040	C	54	3280
55	175,05	170	1040	C	55	3120
56	183,68	170	1040	C	56	missing
57	191,01	170	1040	C	57	2600
58	203,17	170	1040	C	58	2720
59	212,94	170	1040	C	59	2600
60	219,98	170	1040	C	60	2680
61	229,56	170	1040	C	61	2720
62	240,21	170	1040	C	62	2360
63	251,24	170	1040	C	63	2320
64	261,68	170	1040	C	64	2600
65	269,07	170	1040	C	65	2440
66	280,3	170	1040	C	66	2720
67	287,75	170	1040	C	67	2600
68	306,1	170	1040	C	68	2840
69	316,3	170	1040	C	69	2680
70	325,52	170	1040	C	70	2600
71	334,59	170	1040	C	71	2880
72	349,16	170	1040	C	72	2200
73	357,81	170	1040	C	73	3480
74	367,41	170	1040	C	74	2520
75	372,09	170	1040	C	75	2760
76	382,11	170	1040	C	76	3000
77	391,38	170	1040	C	77	3280
78	401,34	170	1040	C	78	3400
79	413,51	170	1040	C	79	3520
80	422,11	170	1040	C	80	3400
81	431,64	170	1040	C	81	3200
82	441,25	170	1040	C	82	3680
83	452,3	170	1040	C	83	3760
84	461,9	170	1040	C	84	3440
85	474,55	170	1040	C	85	2840
86	480,84	170	1040	C	86	2840
87	490,89	170	1040	C	87	4200
88	500,22	170	1040	C	88	1040
89	511,16	170	1040	C	89	560
90	519,81	170	1040	C	90	1120
91	530,49	170	1040	C	91	<500
92	540,19	170	1040	C	92	<500
93	551,31	170	1040	C	93	680
94	560,71	170	1040	C	94	880
95	564,5	170	1040	C	95	<500
96	577,16	170	1040	C	96	<500
97	585,48	170	1040	C	97	<500
98	595,34	170	1040	C	98	880
99	607,95	170	1040	C	99	1720
100	620,23	170	1040	C	100	960
101	626,82	170	1040	C	101	680
102	634,61	170	1040	C	102	<500
103	644,49	170	1040	C	103	920
104	653	170	1040	C	104	720

Tab. 5. Concentration of F (mg/kg) in porewaters measured at various depths (mbsf) in borehole 1039.

1	9,4	1039	B	1	< 1.0
2	23,9	1039	B	2	< 1.0
3	28,4	1039	B	3	< 1.0
4	36,4	1039	B	4	1,1
5	45,84	1039	B	5	< 1.0
6	55,4	1039	B	6	< 1.0
7	64,9	1039	B	7	< 1.0
8	74,43	1039	B	8	< 1.0
9	83,93	1039	B	9	< 1.0
10	94,9	1039	B	10	< 1.0
11	101,35	1039	B	11	< 1.0
12	117,45	1039	B	12	< 1.0
13	131,55	1039	B	13	< 1.0
14	136,55	1039	B	14	< 1.0
15	147,65	1039	B	15	< 1.0
16	157,25	1039	B	16	1
17	168,45	1039	B	17	1,4
18	177,95	1039	B	18	1,5
19	187,55	1039	B	19	1,6
20	197,15	1039	B	20	1,7
21	206,85	1039	B	21	1,8
22	216,45	1039	B	22	2
23	226,05	1039	B	23	2
24	245,35	1039	B	24	1,9
25	252,05	1039	B	25	1,8
26	261,75	1039	B	26	1,7
27	269,95	1039	B	27	1,7
28	279,5	1039	B		
29	289,2	1039	B	29	1,5
30	300,3	1039	B	30	1,4
31	319,5	1039	B	31	1,2
32	332,1	1039	B	32	1
33	348,3	1039	B	33	< 1.0
34	360,9	1039	B	34	< 1.0
35	372	1039	B	35	< 1.0
36	384,95	1039	C	36	< 1.0
37	416,98	1039	C	37	< 1.0

Tab. 6. Concentration of F (mg/kg) in porewaters measured at various depths (mbsf) in borehole 1040.

38	1,4	1040		
39	4,35	1040	39	< 1.0
40	8,35	1040	40	< 1.0
41	19,1	1040	41	< 1.0
42	29,85	1040	42	< 1.0
43	37,7	1040		
44	55,94	1040		
45	70,26	1040	45	< 1.0
46	89,8	1040		
47	97,85	1040		
48	101,8	1040	48	< 1.0
49	106,5	1040	49	< 1.0
50	119,05	1040	50	< 1.0
51	133,7	1040	51	< 1.0
52	144,9	1040	52	< 1.0
53	157,35	1040	53	< 1.0
54	164,1	1040	54	< 1.0
55	174,57	1040		
56	183,3	1040		
57	190,7	1040		
58	203,25	1040		
59	213	1040		
60	219,6	1040		
61	229,15	1040		
62	240,31	1040		
63	251,45	1040		
64	261,86	1040	64	< 1.0
65	269,25	1040		
66	279,95	1040		
67	287,4	1040		
68	306,4	1040		
69	315,95	1040		
70	325,65	1040		
71	335,25	1040		
72	349,35	1040		
73	357,45	1040		
74	367,05	1040		
75	372,15	1040	75	< 1.0
76	381,8	1040	76	< 1.0
77	391,45	1040	77	< 1.0
78	401,05	1040	78	< 1.0
79	413,65	1040	79	< 1.0
80	423,35	1040	80	< 1.0
81	431,25	1040	81	< 1.0
82	440,95	1040	82	< 1.0
83	452,05	1040	83	< 1.0
84	461,65	1040	84	< 1.0
85	474,2	1040	85	1
86	480,95	1040	86	1,1
87	490,69	1040	87	1,4
88	500,35	1040	88	1,9
89	511,32	1040	89	2,3
90	519,6	1040	90	2,4
91	530,55	1040	91	2,5
92	540,3	1040	92	2,7
93	551,43	1040	93	2,7
94	560,8	1040	94	2,6
95	564,7	1040	95	2,6
96	577,36	1040		
97	585,5	1040	97	2,4
98	595,1	1040	98	2,6
99	607,7	1040	99	2,5
100	620,4	1040	100	1,9
101	626,85	1040	101	1,7
102	634,9	1040	102	1,4
103	644,6	1040	103	1,1
104	652,75	1040	104	< 1.0

FIGURES

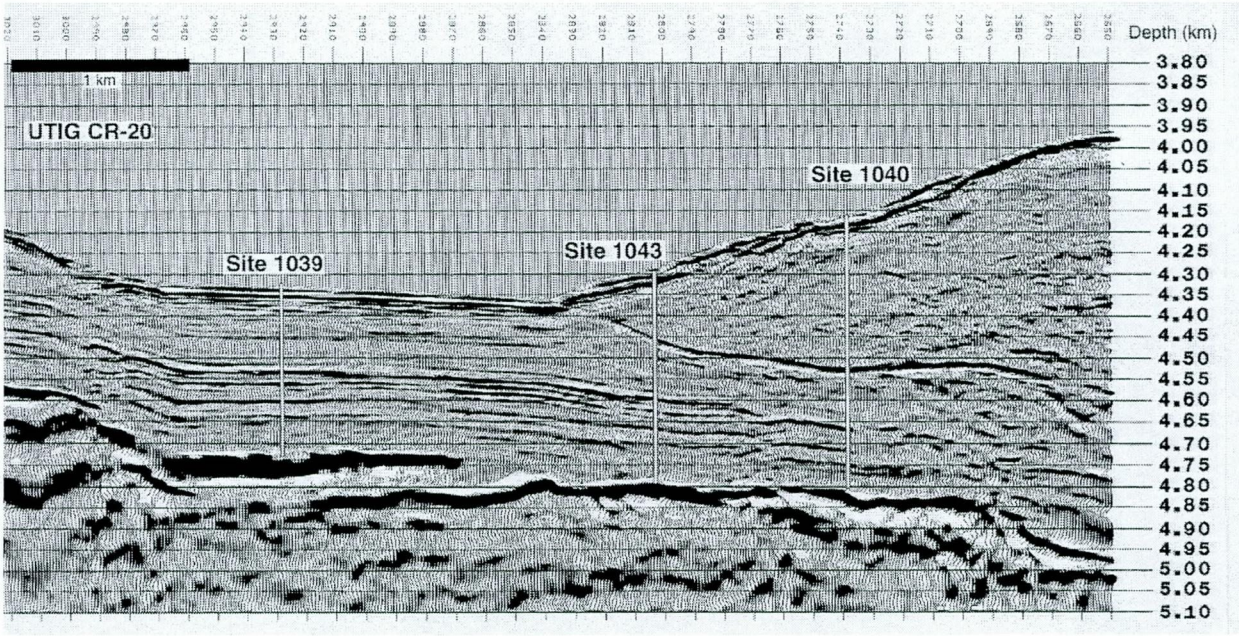


Fig. 1. Seismic section across the subduction zone west of Costa Rica showing the location of borehole sites 1039 and 1040.

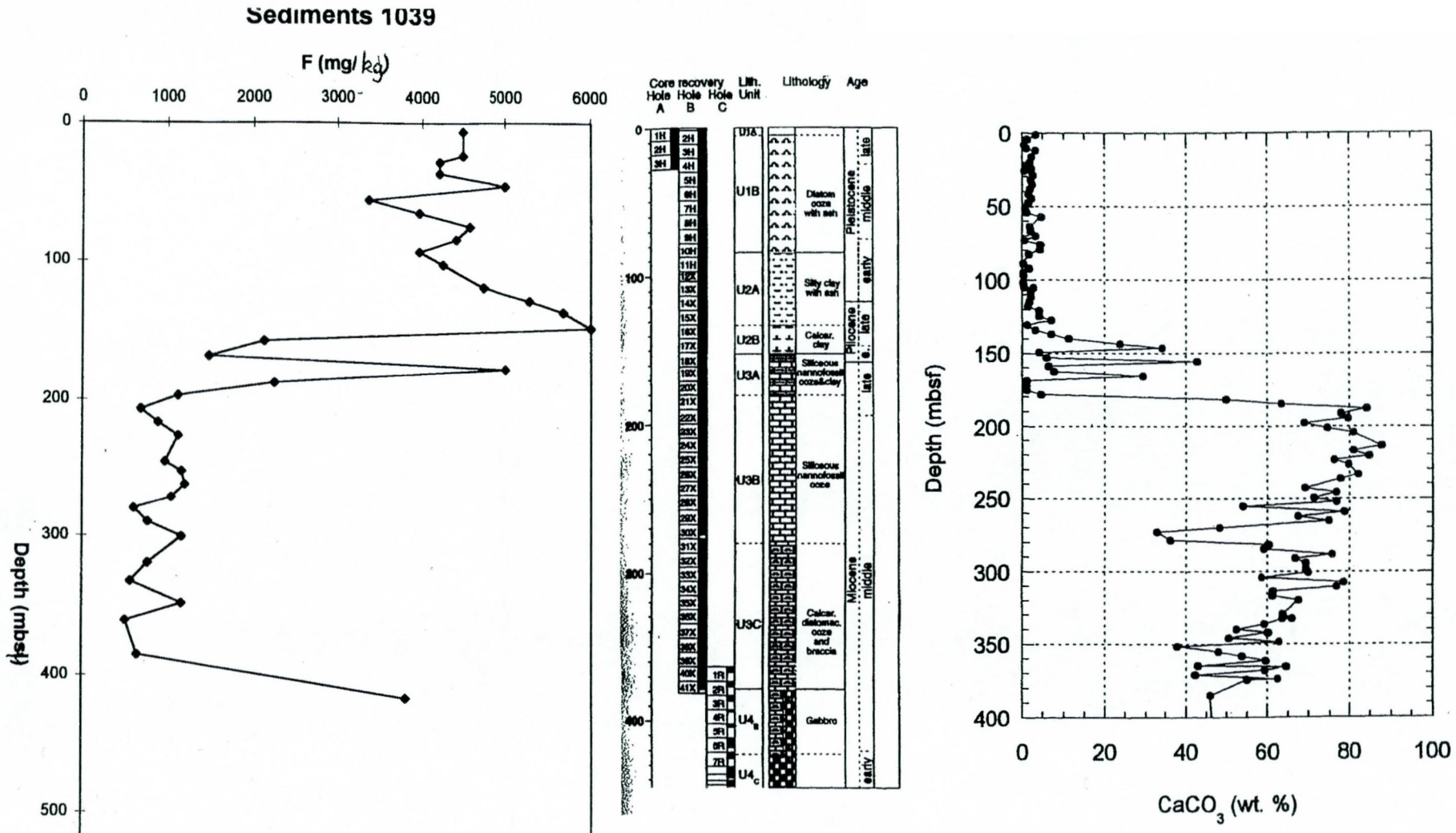


Fig. 2. Diagram showing the concentration of F (mg/kg) in sediments vs. depth (m) in borehole 1039

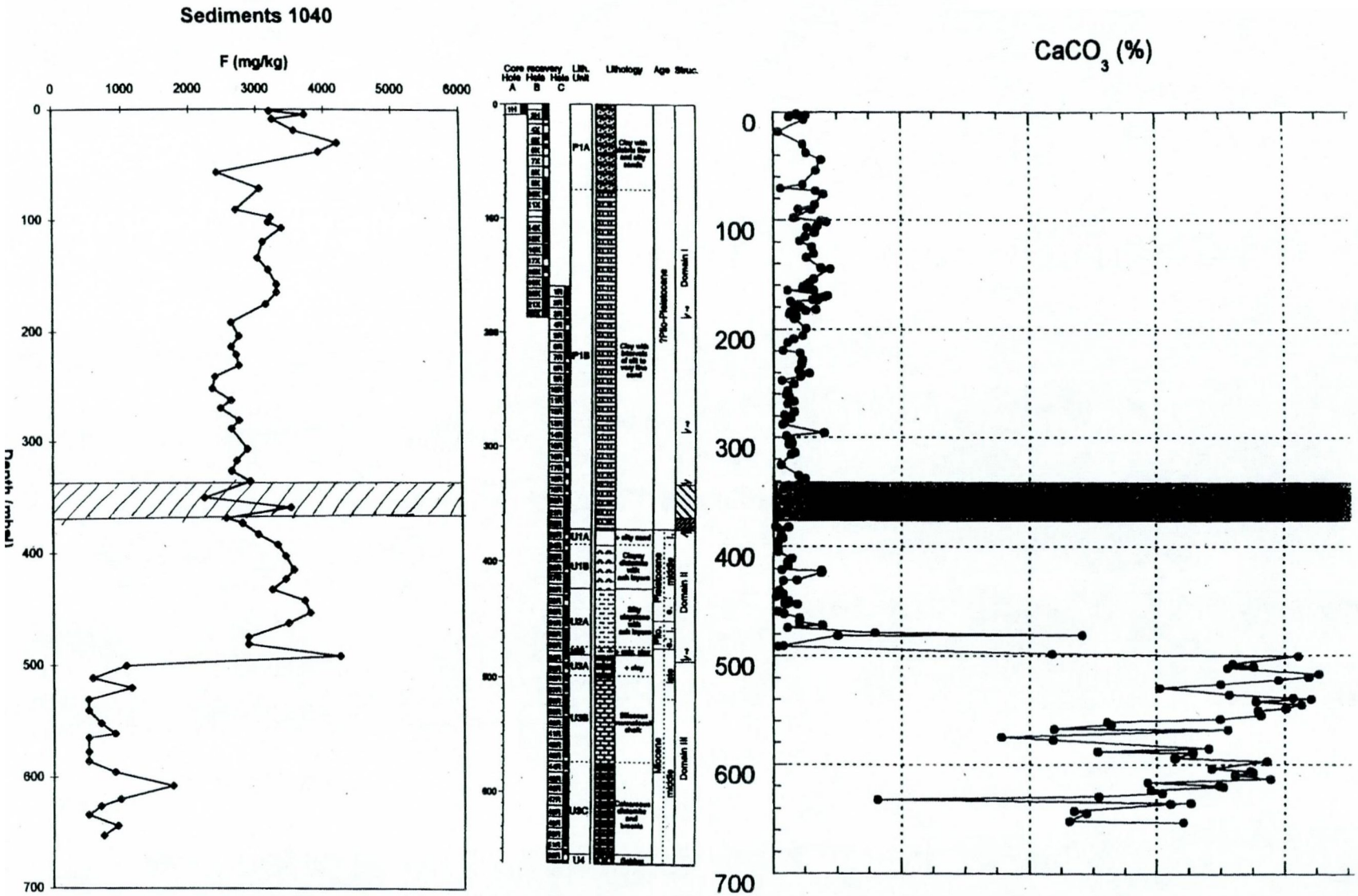


Fig. 3. Diagram showing the concentration of F (mg/kg) in sediments vs. depth (m) in borehole 1040

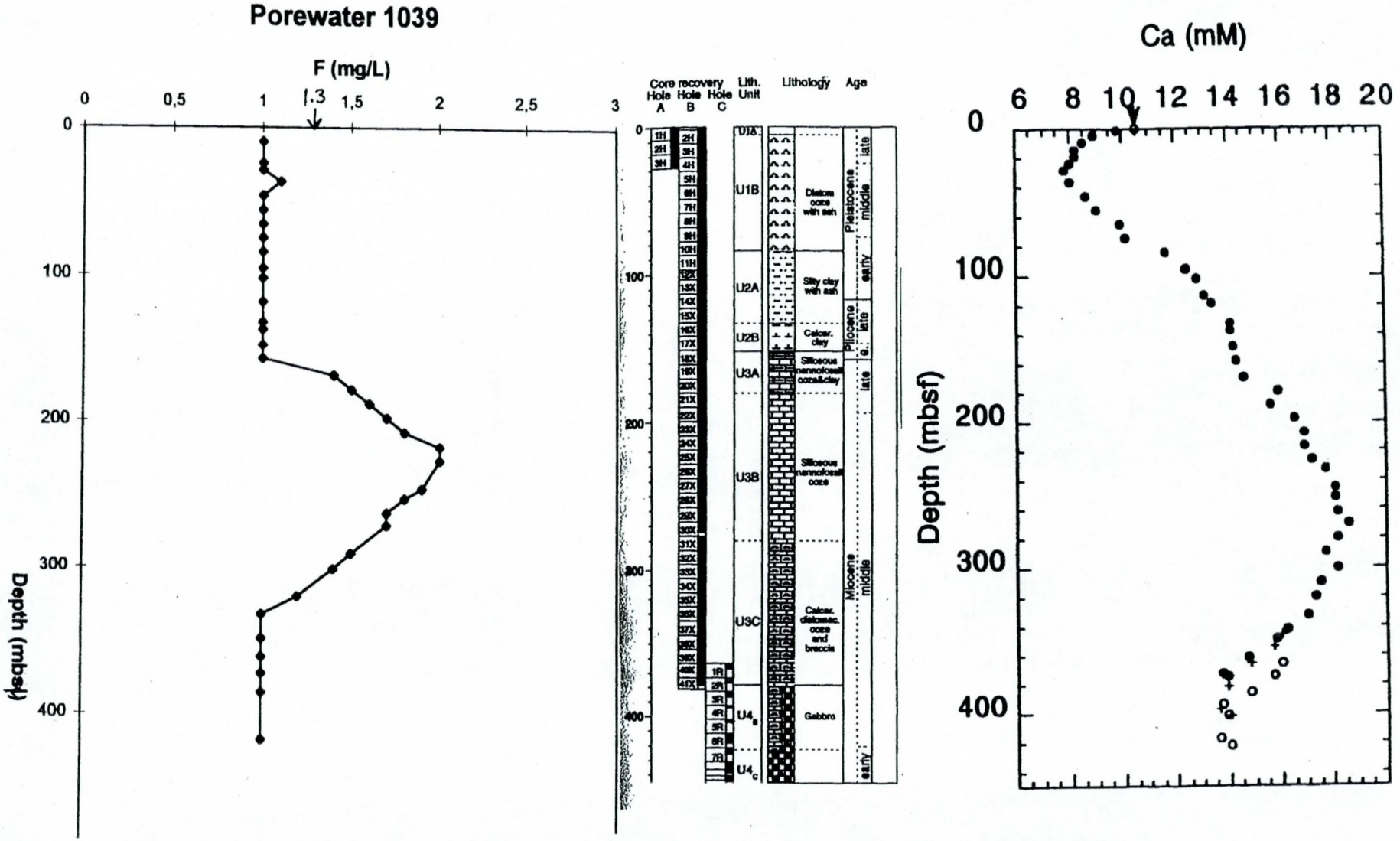


Fig. 4. Diagram showing the concentration of F (mg/kg) in porewaters vs. depth (m) in borehole 1039

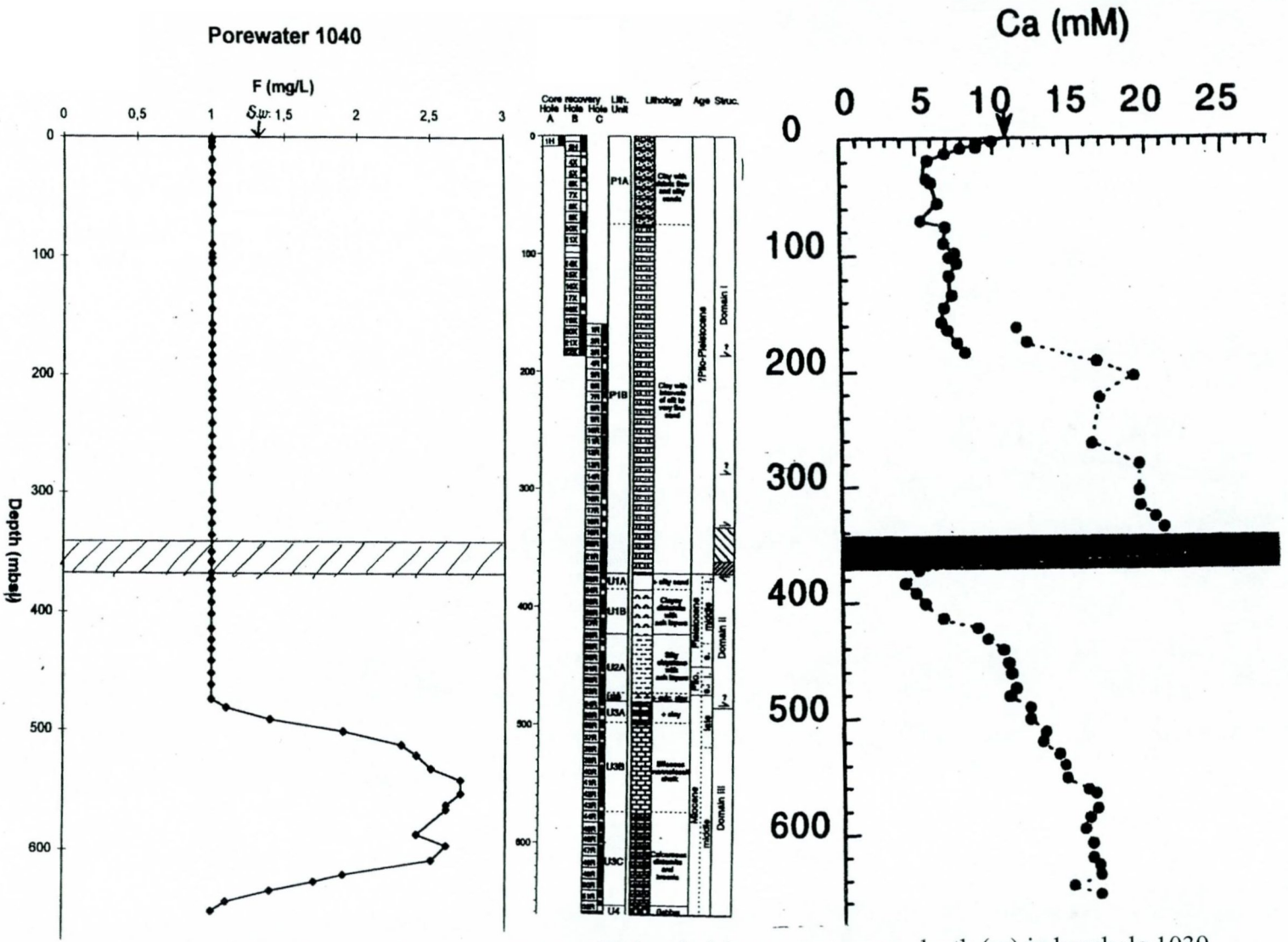
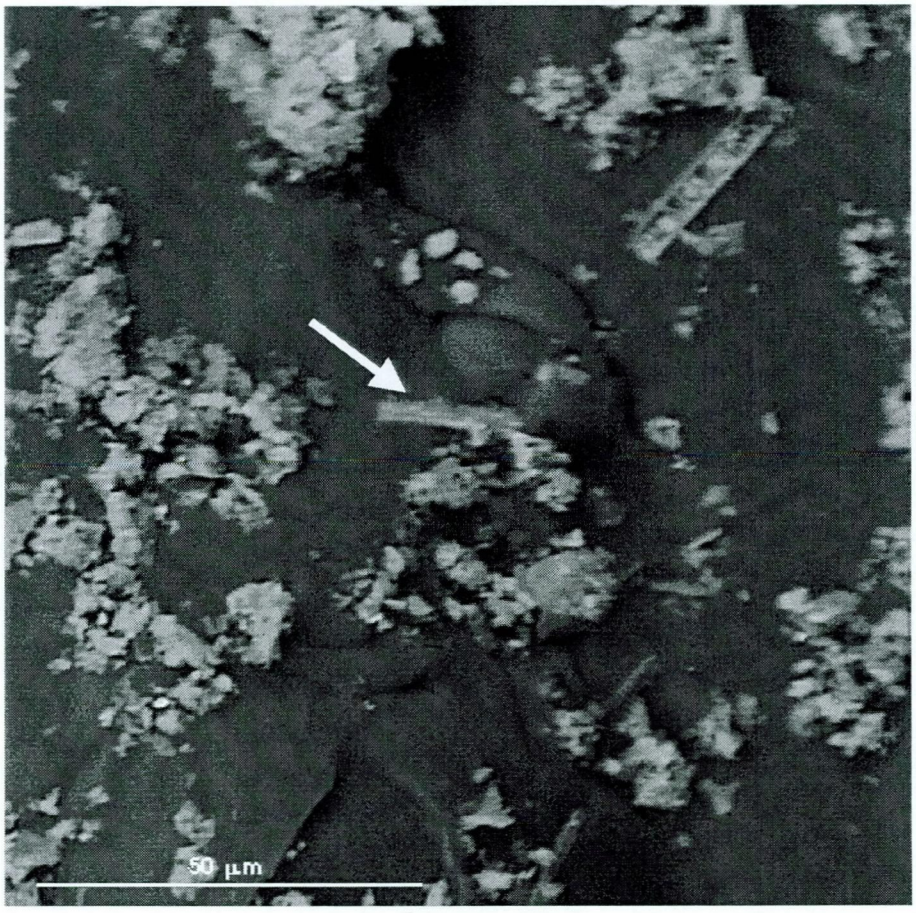


Fig. 5. Diagram showing the concentration of F (mg/kg) in porewaters vs. depth (m) in borehole 1039

A



B

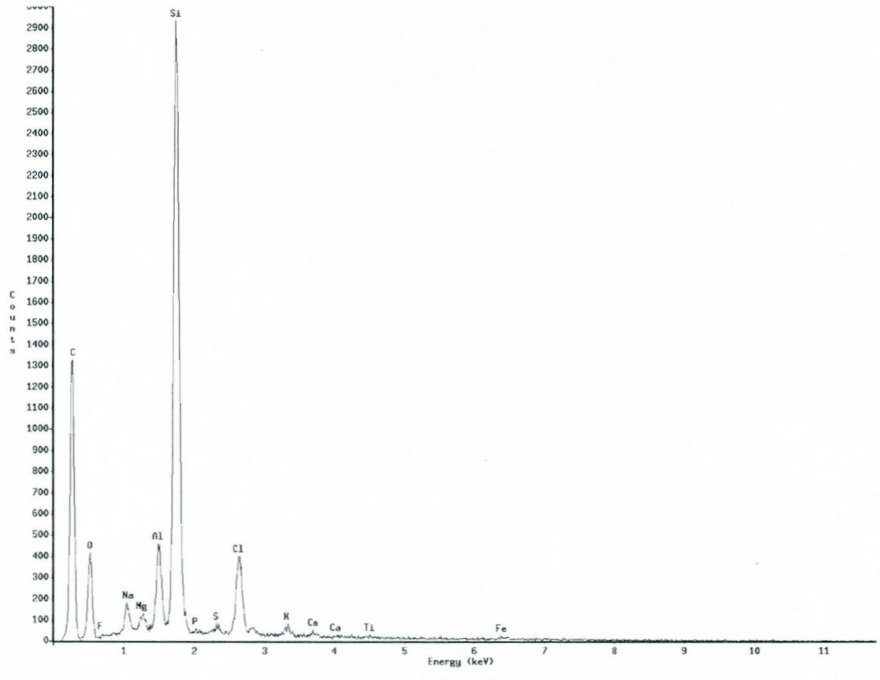


Fig. 6. A) SEM-photo of sediment particles at 23.85 mbsf in borehole 1039. B) EDAX-spectrogram of particle in center of A (F=2400 mg/kg).

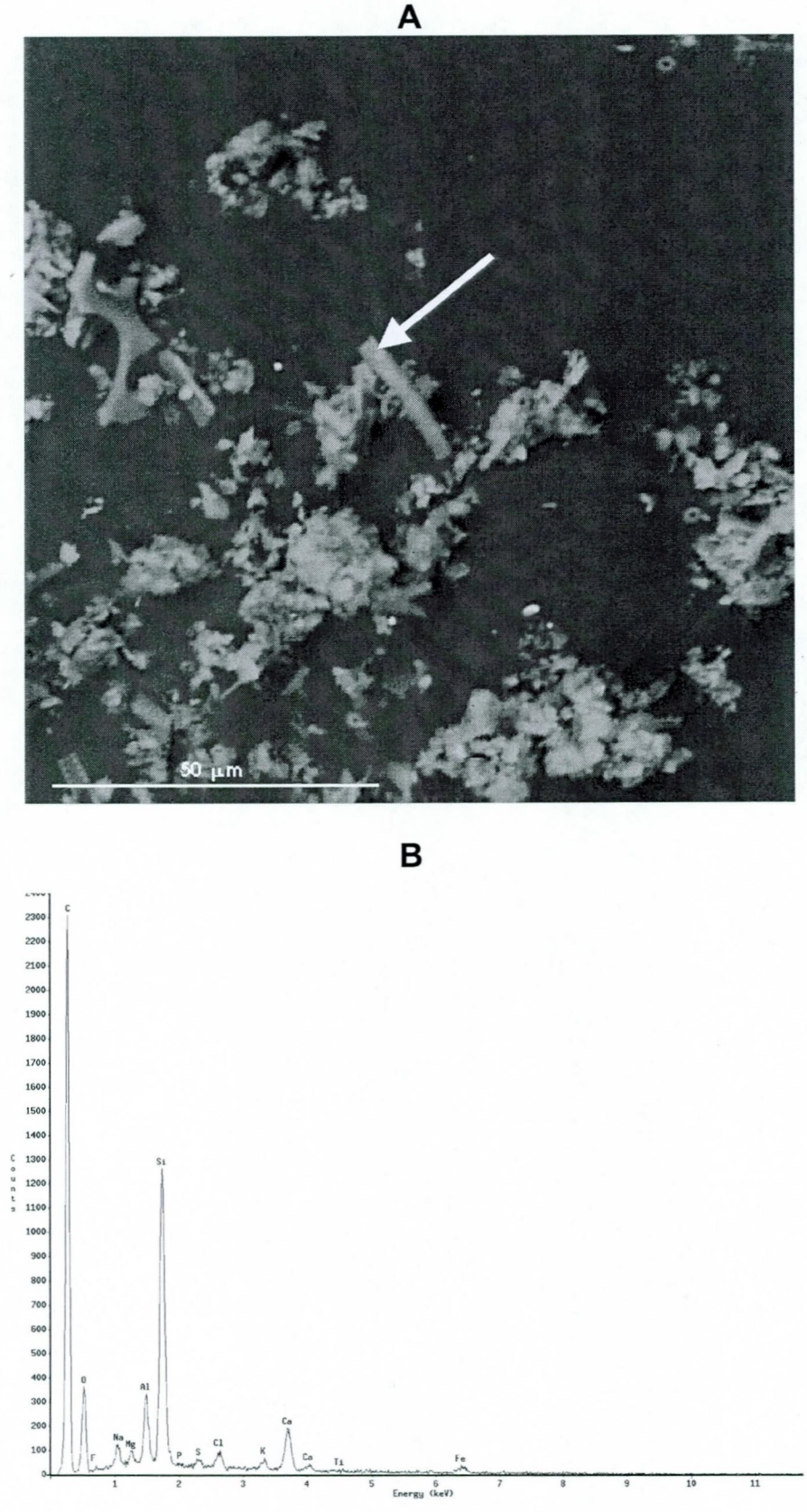


Fig. 7. A) SEM-photo of sediment particles at 23.85 mbsf in borehole 1039. B) EDAX-spectrogram of particle in center of A (F=6200 mg/kg).

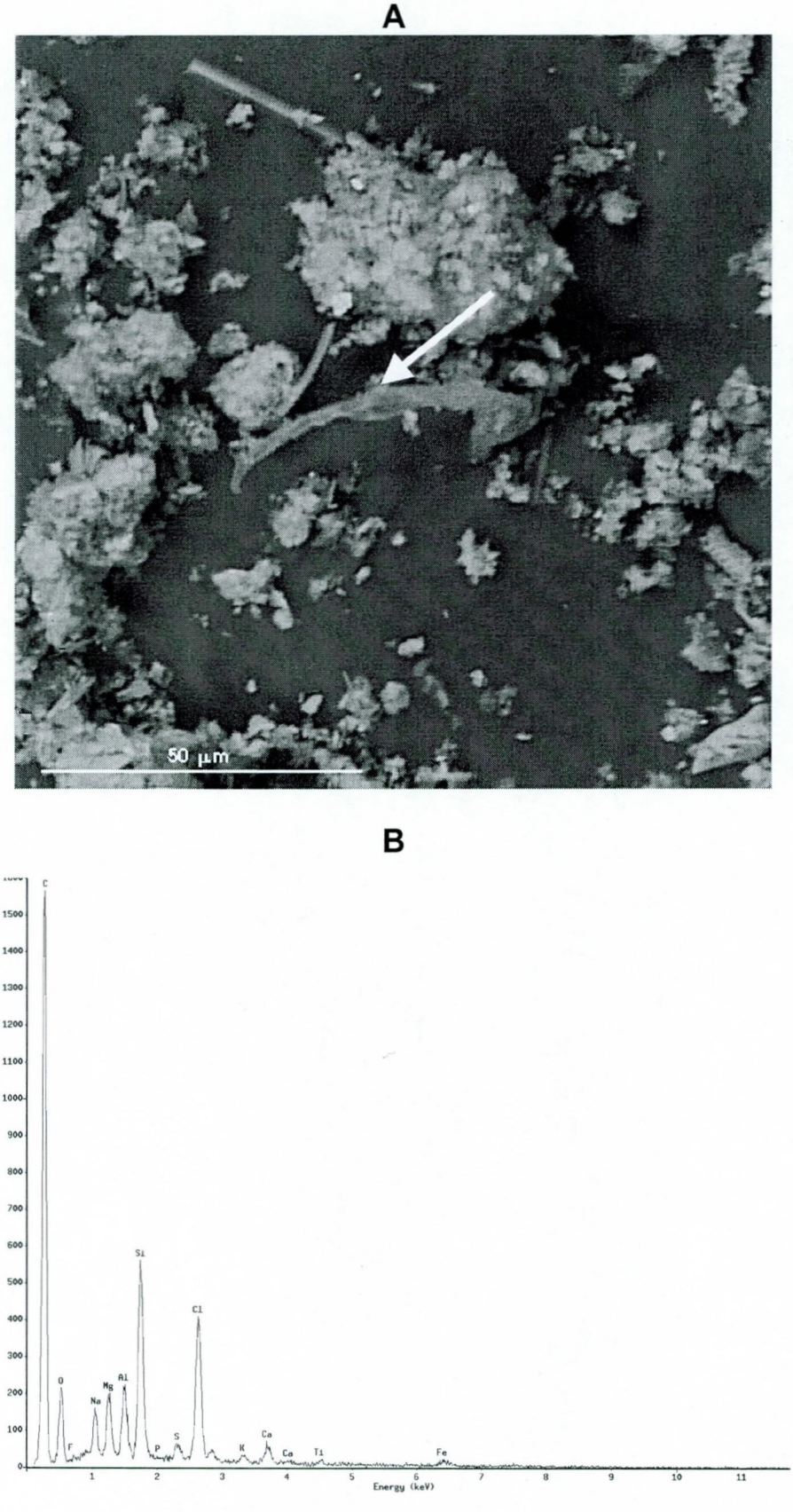


Fig. 8. A) SEM-photo of sediment particles at 23.85 mbsf in borehole 1039. B) EDAX-spectrogram of particle in center of A (F=5700 mg/kg).

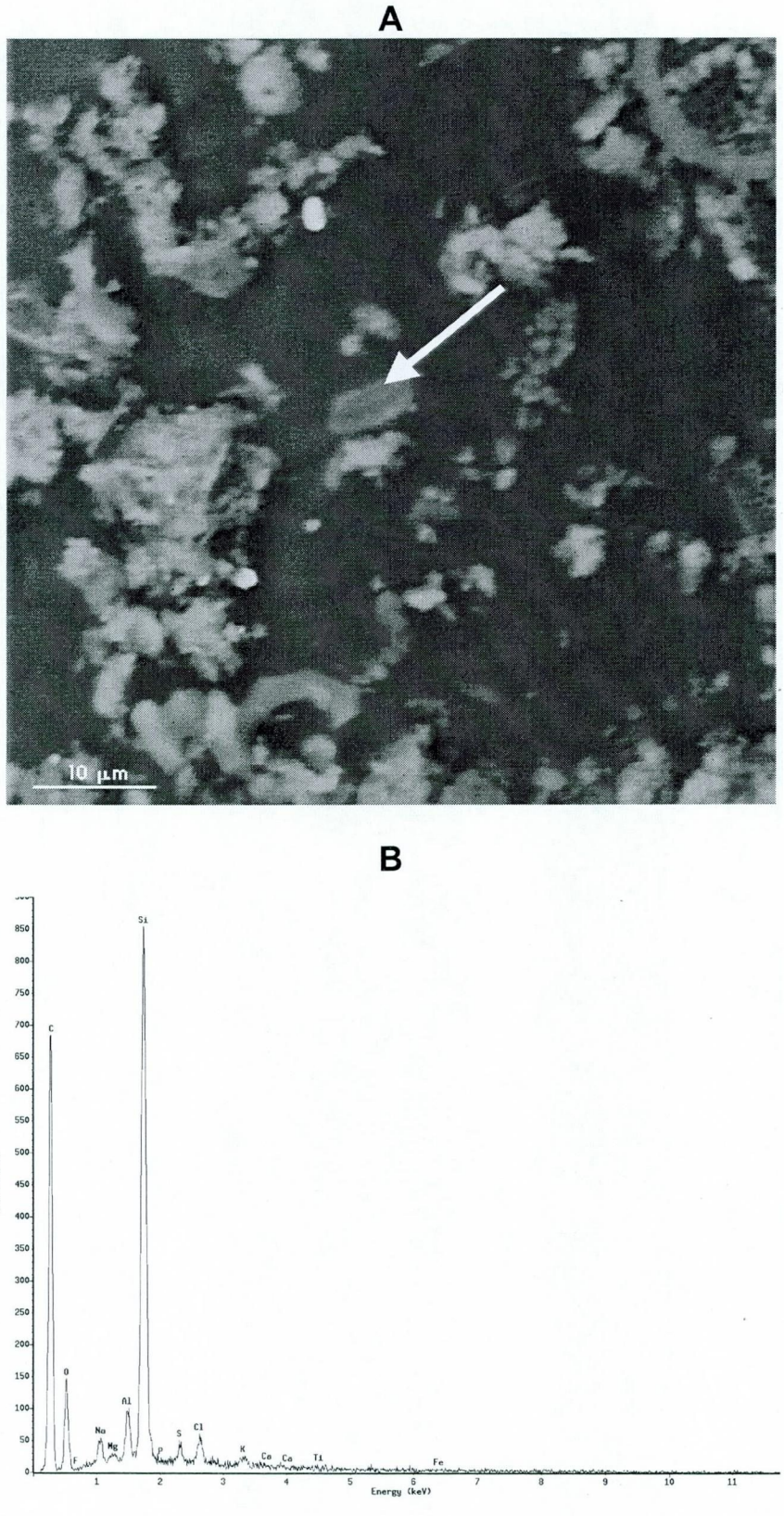


Fig. 9. A) SEM-photo of sediment particles at 23.85 mbsf in borehole 1039. B) EDAX-spectrogram of particle in center of A (F=5200 mg/kg).