IODP Exp. 325 Great Barrier Reef Environmental Changes (GBREC)
QAQC - Documentation - Analytics

Interstitial Water (IW) - Major Elements (ME)

Method and measurement documentation for the analysis of the IW - ME ; calibration solutions, internal and certified standards, IODP reference and core samples

compiled by L. Schnieders

Content:

appendent:

MARUM
2010

IODP_325_QAQC_IW - ME

Chapter 1: Method and measurement documentation

Date:	05.07.2010 - 06.07.2010
Location:	Onshore Science Party (OSP) at IODP Bremen Core Respositiry, MARUM, University Bremen, Germany
Device:	ICP-OES PerkinElmer Optima 3300 R
Nebulizer:	GemTip™ Cross-Flow II Ryton Nebulizer Assembly for ICP-OES

Interstitial Waters:

IW aliqout:	Measurements were conducted with IODP cation split (IW aliquot acidified with 1% HNO_3)
IW core sample (origin/ID):	IODP IW core samples; referred to as "samples" and therefore marked with "S" + consecutive number (S1, S2, etc.) in sample info file of the device as well as in overview table of the analysed IODP samples **{**5.: **IODP_325_QAQC_IW_sample overview II}.**
IW reference sample (origin/ID):	IODP IW reference samples (e.g. drill mud, seawater from locations, etc.); referred to as "references" and therefore marked with "R" + consecutive number (R1, R2, etc.) in sample info file of the device as well as in overview table of the analysed IODP references {4.: **IODP_325_QAQC_IW_sample overview I}.**
Remark on non measured samples:	IODP samples "S" / "R" that exist but (i) do not have CATION split / enough material or (ii) material that got contaminated (e.g. reference samples R17, R18) were not analyzed (cf. Chap. 4. and 5. - Remarks in overview tables)
Samples/References Overview:	For complete sample and reference list see Chapter 4. and 5.; ► {IODP_325_QAQC_IW_sample overview I} & {IODP_325_QAQC_IW_sample overview II}
IW ME data files:	► **Al, B, Ba, Ca, Fe, K, Li, Mg, Mn, Na, P (2xλ), S, Si, Sr, Ti** ► data files to be downloaded from zip folder as [.txt] (cf. below '*Data location*') FYI: raw data, not modified,with all three replicates (device internal) to calculate Std deviation FYI: please note, negative value e.g. '-0,01' in wash and blank means => signal below detection limit / background noise => no contamination; value can be set to Zero for calculations

Method:

Sample handling:	FYI: both, core and reference samples were handled the same way during the entire sampling and analytical process to **guarantee consistency in treatment**
Work solution:	MilliQ (3x purified water, UV treated) acidified with 1 Vol% HNO3 (=> 0.65 weight%) was the work solution both for flushing the ICP-OES device while running and all the dilutions and blanks used during the analytical process.
Matrix:	As most of the samples have a seawater-like matrix, a seawater matrix is also implied to the Multi-element standards to ensure similar conditions in the plasma torch for both standards and samples. The only exception are the single Na-Standards and the Na-Blank.
Matrix solution:	$NaCl_{solid}$ / Work Solution <=> 5.6 g / 200 ml (consistent with seawater concentration refering to IAPSO (CRM) provided by OSIL®).
Sample dilutions:	1:100 for Na and ME (Na and Al, B, Ba, Ca, Fe, K, Li, Mg, Mn, P, S, Si, Sr, Ti) 1: 10 for ME (Al, B, Ba, Ca, Fe, K, Li, Mg, Mn, P, S, Si, Sr, Ti)
Approach:	► **first "test / optimizing" run**: all elements calibrated with different specific standards respectively => to find the highest content of each element in all samples and then eliminate the higher standards of those elements where the standard concentrations were out of the element content range. This enhances the sensitivity of the calibration for each element individually. ► **second "measurement" run:** (with optimized CalibSolution range); after successfull calibration (cf. screenshot of calibration **IODP325W ME** 05.Juli 2010 in Chapter 3 **IODP_325_QAQC_IW-ME_Calib II**) both references "R" and samples "S" were measured in one run starting with 1:100 dilution followed by the 1:10 dilution.

Calibration:

Calibration Stds Material I:	**Multi-element standard solutions** made from certified reference material (CRM) => high-purity single element standard solutions (Spectrascan by Teknolab, Norway) ascribable to standard reference material (SRM) from NIST® (labeled: **MK konz**, **MK VF2**,**MK VF10** and **MK VF100** refering to dilution factor "VF" of pre-stock solution and highest standard solution MK konz) ► Al, B, Ba, Ca, Fe, K, Li, Mg, Mn, P, S, Si, Sr, Ti ► all elements measured both in 1:100 and 1:10 dilution of the IW aliquot to find ideal measurement/concentration range (cf. data tables availabe for details)
Calibration Stds Material II:	**Single-element Standard solution Na** made from certified reference material (CRM) => high-purity single element standard solution (Spectrascan by Teknolab, Norway) ascribable to SRM from NIST® ► Na ► Na data available measured in detectable range only in the 1:100 dilution (1:10 dilution: due to seawater-like matrix of samples, average Na concentration often exceeds detactable range)

Measurement:

Calibration solution order:	► Calib Blank Na ► 250 Na ► 125 Na ► 25 Na ► Calib Blank MK ► MK konz ► MK:2 ► MK:10 ► MK:100
Sample order:	► 1:100 dilutions ► IAPSO ► CRM ► IW references ► IW samples ► 1:10 dilutions ► IAPSO ► CRM ► IW references ► IW samples

Quality assurance:

Certified reference material (CRM):	► IAPSO Standard seawater provided by OSIL® ►CRM-SW (certf. reference material-seawater) provided by HighPuritySTANDARDS®
Standard Reference material (SRM):	► Calibration solutions made from high-purity single-element standards (Spectrascan by Teknolab, Norway) were used as internal quality control (cf. above information on calibration)
	=> both CRM and SRM used for this method are ascribable to SRM from NIST® => CRM was measured two times, together with the 1:100 and the 1:10 dilutions respectively => SRM was remeasured as a sample every 30 IW samples

Data:

Data information:	Results of measurement - element specific Choosen sample dilution, underlying the element data available, is element specific. The choice of sample dilution is dependent on the average element concentration and therefore the ideal concentration/measurement range.
Data location:	► data files to be downloaded from zip folder **{IODP325QAQC_IW-ME_Data.zip}** as [.txt]

IODP_325_QAQC_IW - ME

Chapter 2: **Calibration - preparation of standard solutions**

IODP_325_QAQC_IW - ME_Calib docu I

► Preparation of calibration solutions for the analysis of the IW - ME
► Calibration Standard Material I: Multi-Element calibration solutions for all elements

Multi-element solutions made from certified reference material (CRM): High-purity single element standard solutions Spectrascan® by Teknolab, Norway, ascribable to SRM from NIST®. Labeled: **MK konz**, **MK VF2**,**MK VF10** and **MK VF100** refering to dilution factor "VF" of pre-stock solution and highest standard solution MK konz.

MK Konz	**stock solution**	**target**	**VF (dilution factor)**	**Aliquot in _ 100 _ml final solution**
Element	[ppm]	[ppm]	[=stock/target]	[= Volume final solution/VF]
Al	**1000**	**1**	**1000**	**0.1**
B	**1000**	**2.5**	**400**	**0.25**
Ba	**1000**	**1**	**1000**	**0.1**
Ca	**10000**	**50**	**200**	**0.5**
Fe	**1000**	**1**	**1000**	**0.1**
K	**10000**	**50**	**200**	**0.5**
Li	**1000**	**1**	**1000**	**0.1**
Mg	**10000**	**150**	**66.66666667**	**1.5**
Mn	**1000**	**1**	**1000**	**0.1**
P	**1000**	**5**	**200**	**0.5**
S	**10000**	**150**	**66.66666667**	**1.5**
Si	**1000**	**5**	**200**	**0.5**
Sr	**1000**	**2**	**500**	**0.2**
Ti	**1000**	**1**	**1000**	**0.1**
MK Konz	► MK Konz + 10 ml NaCl Solution ("seawater matrix" 5.6 g/200 ml) filled up with purified H_2O (acidified 1% HNO_3) to 100 ml pre-stock solution in volumetric flask			
MK VF 2	► 25 ml MK konz + 2,5 ml NaCl Solution ("seawater matrix") filled up with purified H2O (acidified 1% HNO3) to 50 ml of final solution in volumetric flask			
MK VF 10	► 5 ml MK konz + 4,5 ml NaCl Solution ("seawater matrix") filled up with purified H2O (acidified 1% HNO3) to 50 ml of final solution in volumetric flask			
MK VF 100	► 0.5 ml MK konz + 4.95 ml NaCl Solution ("seawater matrix") filled up with purified H2O (acidified 1% HNO3) to 50 ml of final solution in volumetric flask			
Blank MK	► 10 ml NaCl Solution ("seawater matrix") filled up with purified H2O (acidified 1% HNO3) to 100 ml of final solution in volumetric flask			

IODP_325_QAQC_IW - ME

Chapter 2: **Calibration - preparation of standard solutions**

IODP_325_QAQC_IW - ME_Calib docu II

► Preparation of calibration solutions for the analysis of the IW - ME
► Calibration Standard Material II: Single-Element calibration solution for Na

Single-element solution made from certified reference material (CRM) => high-purity single element standard solution (Spectrascan by Teknolab, Norway) ascribable to SRM from NIST®

Na	**stock solution**	**target**	**VF (dilution factor)**	**Aliquot in _ 100 _ml final solution**
Element	[ppm]	[ppm]	[=stock/target]	[= Volume final solution/VF]
Na	**10000**	**250**	**40**	**2.5**
Na	**10000**	**125**	**80**	**1.25**
Na	**10000**	**25**	**400**	**0.25**
Na	► MK Konz + 10 ml NaCl Solution ("seawater matrix" 5.6 g/200 ml) filled up with purified H_2O (acidified 1% HNO_3) to 100 ml pre-stock solution in volumetric flask			
250 Na	► 2.5 ml of stock solution without "seawater matrix" filled up with purified H_2O (acidified 1% HNO_3) to 50 ml of final solution in volumetric flask			
125 Na	► 1.25 ml of stock solution without "seawater matrix" filled up with purified H_2O (acidified 1% HNO_3) to 50 ml of final solution in volumetric flask			
25 Na	► 0.25 ml of stock solution without "seawater matrix" filled up with purified H_2O (acidified 1% HNO_3) to 50 ml of final solution in volumetric flask			
Blank Na	► purified H_2O (acidified 1% HNO_3)			

Chapter 3: **Calibration - standard solutions, λ, quality control**

IODP_325_QAQC_IW - ME_Calib I

► Calibration solutions (standards) prepared for the analysis of the IW - ME
► wavelength, concentrations

MK	wavelength [λ]	MK konz [mg/L]	MK VF 2 [mg/L]	MK VF 10 [mg/L]	MK VF 100 [mg/L]
Na	589,592	1	0.5	0.1	0.01
Al	396,153	2.5	1.25	0.25	0.025
B	249,677	1	0.5	0.1	0.01
Ba	455,403	1	0.5	0.1	0.01
Fe	238,204	50	25	5	0.5
K	766,490	1	0.5	0.1	0.01
Li	670,784	150	75	15	1.5
Mg	280,271	1	0.5	0.1	0.01
Mn	257,610	5	2.5	0.5	0.05
P*, P**	213.617*/178.221**	5	2.5	0.5	0.05
S	181,975	150	75	15	1.5
Si	251,611	5	2.5	0.5	0.05
Sr	407,771	2	1	0.2	0.02
Ti	334,940	1	0.5	0.1	0.01
Ca	317,933	50	25	5	0.5
Na	**wavelength [λ]**	**250 Na [mg/L]**	**125 Na [mg/L]**	**25 Na [mg/L]**	
Na	589,592	250	125	25	-

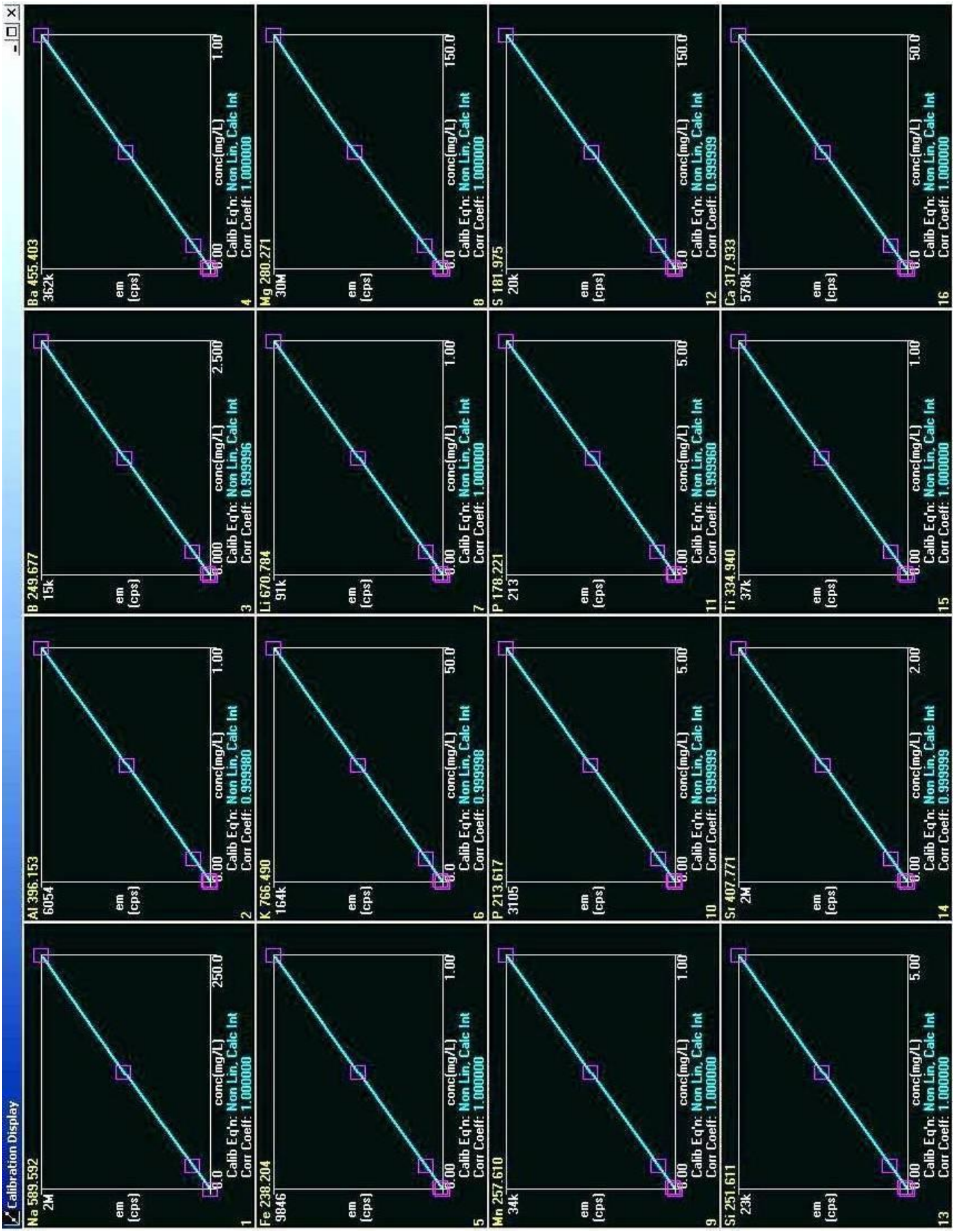
*** P 213.617**
**** P 178.221**

► Cu interference existent, but negligible
► less sensitive, examination of data set showed Iode interference on λ178 (which is expectable for seawater-like samples)

=> therefore λ213 has been deployed for the analysis

IODP_325_QAQC_IW - ME_Calib II

► Screenshot of the calibration IODP325IW ME 05.Juli 2010



Chapter 4: **IODP Exp. 325 reference sample overview**

IODP_325_QAQC_IW_sample overview I
IODP reference samples "R"
All

	=>	relevant Cation split for ICP-OES IW-ME analysis
x/0,7	=>	yes; analysis conducted / IW amount originally sampled
-	=>	no; analysis not conducted / no sample

Ref No°	Sample ID Exp.	Sit	Hol	Cor	Ty	Sec	IW Rhiz SecDepth [cm]	IW total [mL]	Aliquots offshore* Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	d13C [mL]	d18O [mL]	d34S [mL]	DOC [mL]	Sal [mL]	Analysis offshore / onshore Sal [mL]	Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	Req 776IODP Mg-Isotope** [mL]	Req 776IODP Sulfur-Isotope d34S-split [mL]	Req 776IODP Sulfur-Isotope d13C-split [mL]	Remarks
R1	325	34	A	Blank-20-02-10			-	20.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R2	325	40	A	Blank-06-03-10			-	20.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R3	325	-	-	SterileBlank-03-03-10			-	16.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	shows NO3 contamination in IC chromatogramm
R4	325	34	A	Tap-15-02-10			-	12.0	-	-	3	6	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R5	325	34	A	Tap-Cabin			-	13.0	-	-	2	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R6	325	-	-	Rain-23-03-10			-	25.4	0.7	0.7	8	8	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	Collected between 6am-noon. 500mL.
R7	325	-	-	Rain-01-04-10			-	14.4	0.7	0.7	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	Rain sample can have salt contamination due to seaspray on collecting surface
R8	325	-	-	Rain-04-04-10			-	25.4	0.7	0.7	8	8	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	100mL during 19h between 0am-19pm at Site 57A
R9	325	Tville	-	FSW-13-03-10			-	16.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R10	325	Tville	-	FSW-18-03-10			-	16.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R11	325	34	A	FSW-20-02-10			-	14.0	-	-	8	7	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R12	325	40	A	FSW-05-03-10			-	14.0	-	-	8	7	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R13	325	42	A	FSW-08-03-10			-	16.0	-	-	8	8	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R14	325	46	A	FSW			-	14.0	-	-	8	7	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R15	325	52	A	FSW-27-03-10			-	23.4	0.7	0.7	8	7	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R16	325	54	A	FSW-27-03-10			-	23.4	0.7	0.7	8	7	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R17	325	55	A	FSW-01-04-10			-	29.4	0.7	0.7	10	10	2	2	2	2	-	-	x	x	-	-	-	2.0 ml	2.0 ml	Cat+Anion: contaminated; mouldy/cloudy solution
R18	325	57	A	FSW-03-04-10			-	29.4	0.7	0.7	10	10	2	2	2	2	-	-	x	x	-	-	-	2.0 ml	2.0 ml	Cat+Anion: contaminated; mouldy/cloudy solution
R19	325	34	A	ASW			-	8.0	-	-	1	4	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R20	325	-	-	MW-splash			-	8.0	-	-	2	3	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R21	325	-	-	MW-Cooroy			-	6.0	-	-	2	3	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R22	325	Mud 0mL/20 mL FSW-13-03-10					-	1.7	0.7	1	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	Mar. 13 Mud test;FSW=Mar13,2010 / no CATION + ANION split
R23	325	Mud 0.1mL/20 mL FSW-13-03-10					-	1.7	0.7	1	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	FSW=Mar13,2010 / no CATION + ANION split
R24	325	Mud 0.01mL/20 mL FSW-13-03-10					-	1.7	0.7	1	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	FSW=Mar13,2010 / no CATION + ANION split
R25	325	Mud 0.05mL/20 mL FSW-13-03-10					-	1.7	0.7	1	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	FSW=Mar13,2010 / no CATION + ANION split
R26	325	Mud 0mL/20 mL FSW-19-03-10					-	25.4	0.7	0.7	7	8	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	Mar. 19 Mud test;FSW=Mar18,2010
R27	325	Mud 0.0001mL/20 mL FSW-19-03-10					-	25.4	0.7	0.7	7	4	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	FSW=Mar18,2010
R28	325	Mud 0.001mL/20 mL FSW-19-03-10					-	25.4	0.7	0.7	7	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	FSW=Mar18,2010
R29	325	Mud 0.01mL/20 mL FSW-19-03-10					-	17.4	0.7	0.7	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	FSW=Mar18,2010
R30	325	Mud 0.1mL/20 mL FSW-19-03-10					-	13.4	0.7	0.7	0.5	1	2	2	2	2	-	-	x	x	x	x	0.5 ml	2.0 ml	2.0 ml	FSW=Mar18,2010
R31	325	Mud 0.5mL/20 mL FSW-19-03-10					-	7.4	0.7	0.7	-	0.1	-	-	-	2	-	-	x	x	-	-	-	-	-	FSW=Mar18,2010 / not enough CATION + ANION split
R32	325	Mud 1mL/20 mL FSW-19-03-10					-	6.1	0.4	0.7	-	0.1	-	-	-	1	-	-	x	x	-	-	-	-	-	FSW=Mar18,2010 / not enough CATION + ANION split
R33	325	31	A	Mud-15-02-10			-	6.0	-	-	2	3	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R34	325	33	A	Mud-18-02-10			-	13.0	-	-	4	6	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R35	325	41	A	Mud-06-03-10			-	7.0	-	-	2	3	2	-	2	-	-	-	x	x	x	x	1.0 ml	2.0 ml	2.0 ml	
R36	325	Mud 0.5mL/20 mL FSW-20-03-10					-	1.0	-	-	0.1	0.1	-	1	-	-	-	-	x	x	-	-	-	-	-	FSW=Mar18,2010 / not enough CATION + ANION split
R37	325	Mud 1mL/20 mL FSW-20-03-10					-	2.0	-	-	0.1	0.1	-	2	-	-	-	-	x	x	-	-	-	-	-	FSW=Mar18,2010,d18O collect21.03.10 / not enough CATION + ANION split
																					27	27	27	29	29	

Please note:

Requested samples have been shipped off to requester
*** Please note, table shows only IW amount initially sampled and stored, after analysis some aliquots can be used up**
**** Please note, Mg-Isotope splits are from cation splits (per 1ml sample/10µl HNO3 = 1 Vol% = 0,65 weight% acidification)**

Chapter 5: **IODP Exp. 325 core sample overview**

IODP_325_QAQC_IW_sample overview II
Interstitial water samples "S"
All

	=>	relevant Cation split for ICP-OES IW-ME analysis
x/0,7	=>	yes; analysis conducted / IW amount originally sampled
-	=>	no; analysis not conducted / no sample

Ref	Sample ID						IW Rhizone	IW	Aliquots offshore*								Analysis offshore / onshore					Req 776IODP	Req 776IODP	Req 776IODP	Remarks
No°	Exp.	Sit	Hol	Cor	Ty	Sec	SecDepth[cm]	total [mL]	Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	d13C [mL]	d18O [mL]	d34S [mL]	DOC [mL]	Sal [mL]	Sal [mL]	Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	Mg-Isotope** [mL]	Sulfur-Isotope d34S-split [mL]	Sulfur-Isotope d13C-split [mL]	
S1	325	31	A	10	R	1	15	8	0.3	0.5	0.1	0.1	2	0.8	2	-	0.3	x	x	x	x	x	-	2 ml	2 ml	
S2	325	32	A	4	R	1	17	0.1	-	-	-	-	-	-	-	-	0.1	-	-	-	-	-	-	-	-	no CATION + ANION split
S3	325	33	A	21	R	1	38	17	0.3	0.8	2	6	2	2	2	-	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S4	325	33	A	22	R	1	42	38	1	1	8	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	Anion double
S5	325	33	A	23	R	1	25	1	0.3	0.6	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S6	325	35	A	13	R	1	25,35	1.2	0.3	0.3	-	-	-	-	-	-	-	x	x	x	-	-	-	-	-	no CATION + ANION split
S7	325	36	A	4	R	1	75	3	not enough sample									x	x	x	-	-	-	-	-	no CATION + ANION split
S8	325	36	A	14	R	1	25	9	0.7	-	1	2	2	-	-	-	-	x	x	x	x	x	1.0 ml	-	2 ml	
S9	325	36	A	21	R	CC	6	7	-	-	0.2	1	2	-	2	-	-	x	x	x	x	x	-	2 ml	2 ml	
S10	325	37	A	1	R	1	30	30	1	1	8	8	2	4	2	4	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S11	325	37	A	1	R	2	15	30	1	1	7	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S12	325	37	A	7	R	1	8	1	not enough sample									x	x	x	-	-	-	-		no CATION + ANION split
S13	325	37	A	9	R	1	30	30	1	1	8	5	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S14	325	37	A	10	R	1	126	30	1	1	8	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S15	325	37	A	13	R	1	27	36	0.7	0.7	7	6	2	4	2	4	0.3	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S16	325	39	A	5	R	1	28	3	-	-	0.3	0.5	-	-	-	-	-	x	x	x	x	x	-	-	-	
S17	325	39	A	20	R	1	19	3	0.5	0.5	0.1	-	-	-	-	-	-	x	x	x	x	-	-	-	-	no ANION split
S18	325	39	A	21	R	1	18	30	0.7	0.7	7	6	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S19	325	40	A	1	R	1	75	30	0.7	0.7	7	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S20	325	40	A	1	R	2	21	28	0.7	0.7	8	7	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S21	325	40	A	10	R	1	36	14	0.7	0.7	2	2	2	2		2	-	-	x	x	x	x	1.0 ml	-	2 ml	
S22	325	40	A	12	R	1	92	32	0.7	0.7	7	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S23	325	41	A	1	R	1	27	22	0.7	0.7	7	5	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S24	325	41	A	8	R	1	42	30	0.7	0.7	8	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S25	325	41	A	10	R	1	72	30	0.7	0.7	8	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S26	325	41	A	11	R	1	60	30	0.7	0.7	8	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S27	325	41	A	12	R	1	29	30	0.7	0.7	7	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S28	325	41	A	12	R	1	130	30	0.7	0.7	7	8	2	2	2	2	-	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S29	325	42	A	13	R	1	36	4	0.5	0.5	0.5	0.2	-	-	-	-	-	-	x	x	x	x	0.5 ml	-	-	
S30	325	42	A	29	R	1	25	7	0.7	0.5	0.2	0.2	-	-	-	-	-	-	x	x	x	x	0.5 ml	-	2 ml	
S31	325	43	A	2	R	1	33	16	-	-	0.5	0.3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S32	325	43	A	10	R	1	26	2	0.3	0.3	0.2	0.2	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION analysis, not enough sample
S33	325	43	A	18	R	1	7	2	0.3	0.3	0.1	0.6	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION analysis, not enough sample
S34	325	43	A	18	R	1	15	20	0.7	0.5	3	1	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S35	325	46	A	13	R	CC	3	6	0.7	0.5	0.2	-	2	1	-	-	-	-	x	x	x	-	1.0 ml	-	2 ml	no ANION split
S36	325	46	A	13	R	CC	6	3	0.7	0.5	-	-	-	-	2	-	-	-	x	x	-	-	-	2 ml	-	no CATION + ANION split
S37	325	47	A	8	R	1	14	2	0.7	0.5	-	-	2	0.2	-	-	-	-	x	x	-	-	-	-	2 ml	no CATION + ANION split
S38	325	47	A	8	R	1	20	10	0.7	0.5	0.2	0.5	-	2	2	-	-	-	x	x	x	x	0.5 ml	2 ml	-	
S39	325	49	A	1	X	1	35	38	0.7	0.5	7	7	2	2	2	2	0.02	x	x	x	x	x	1.0 ml	2 ml	2 ml	
S40	325	50	A	1	X	1	45	10	0.7	0.5	0.5	1	2	2	2	-	-	-	x	x	x	x	0.5 ml	2 ml	2 ml	
S41	325	52	A	1	X	2	15	18	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S42	325	52	A	1	X	2	35	18	0.5	0.5	3	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S43	325	52	A	1	X	2	55	18	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S44	325	52	A	1	X	2	75	18	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S45	325	53	A	1	X	1	45	18	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S46	325	53	A	1	X	1	65	18	0.5	0.5	3	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S47	325	53	A	1	X	1	75	15	0.5	0.5	1	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S48	325	53	A	1	X	1	90	8	0.5	0.5	0.5	1	2	-	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S49	325	53	A	18	R	1	51+55	4	0.7	0.7	0.2	0.1	-	-	-	-	-	-	x	x	x	x	-	-	-	
S50	325	53	A	22	R	1	12	2	0.7	0.7	0.2	-	-	-	-	-	-	-	x	x	x	-	-	-	-	no ANION split
S51	325	53	A	22	R	1	20	15	0.7	0.7	2	0.3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S52	325	53	A	27	R	1	18	2	0.3	0.3	0.1	0.2	-	-	-	-	-	-	x	x	x	x	-	-	-	
S53	325	53	A	27	R	1	38	9	0.3	0.3	0.5	2	2	1	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
									Aliquots offshore*									Analysis offshore / onshore					Req 776IODP	Req 776IODP	Req 776IODP	

Ref No°	Sample ID Exp.	Sit	Hol	Cor	Ty	Sec	IW Rhizone SecDepth[cm]	IW total [mL]	Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	d13C [mL]	d18O [mL]	d34S [mL]	DOC [mL]	Sal [mL]	Sal [mL]	Alk [mL]	NH4 [mL]	Cat [mL]	An [mL]	Mg-Isotope** [mL]	Sulfur-Isotope d34S-split [mL]	Sulfur-Isotope d13C-split [mL]	Remarks
S54	325	53	A	30	R	1	10	**2**	0.3	0.3	0.1	0.2	-	-	-	-	-	-	x	x	x	x	-	-	-	
S55	325	53	A	31	R	1	20	**6**	0.5	0.5	1	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S56	325	53	A	32	R	1	10	**1**	0.5	0.5	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S57	325	53	A	32	R	1	25	**18**	0.5	0.5	3	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S58	325	54	B	9	R	1	32+43+65	**2**	0.4	0.4	-	0.5	-	-	-	-	-	-	x	x	-	x	-	-	-	no CATION split
S59	325	54	B	10	R	1	6	**1**	0.4	0.4	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S60	325	54	B	11	R	1	16+40	**1**	0.4	0.4	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S61	325	54	B	12	R	1	12+27	**1**	0.4	0.4	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S62	325	55	A	1	R	1	-		no sample									-	-	-	-	-	-	-		no sample taken
S63	325	58	A	1	X	1	20	**9**	0.5	0.5	0.5	2	2	2	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S64	325	58	A	1	X	1	40	**10**	0.5	0.5	0.5	2	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S65	325	58	A	1	X	2	20	**12**	0.5	0.5	0.5	1.5	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S66	325	58	A	1	X	2	40	**12**	0.5	0.5	0.5	1.5	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S67	325	58	A	1	X	2	60	**7**	0.5	0.5	0.5	2	2	-	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S68	325	58	A	1	X	2	80	**9**	0.5	0.5	0.5	2	2	2	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S69	325	58	A	1	X	2	100	**12**	0.5	0.5	0.5	2	2	1	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S70	325	58	A	2	X	1	20	**8**	0.5	0.5	0.5	2	2	1	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S71	325	58	A	2	X	1	40	**11**	0.5	0.5	0.5	2	2	-	2	1.8	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S72	325	58	A	2	X	1	60	**16**	0.5	0.5	2	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S73	325	58	A	2	X	1	80	**8**	0.5	0.5	0.5	2	2	1	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S74	325	58	A	2	X	2	20	**13**	0.5	0.5	0.5	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S75	325	58	A	2	X	2	40	**16**	0.5	0.5	2	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S76	325	58	A	2	X	2	60	**11**	0.5	0.5	0.5	2	2	-	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S77	325	58	A	2	X	2	80	**8**	0.5	0.5	0.5	2	2	1	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S78	325	58	A	2	X	2	100	**8**	0.5	0.5	0.5	2	2	1	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S79	325	58	A	2	X	2	120	**7**	0.5	0.5	0.5	2	2	-	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S80	325	58	A	3	X	1	20	**10**	0.5	0.5	0.5	1.5	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S81	325	58	A	3	X	1	40	**10**	0.5	0.5	0.5	2	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S82	325	58	A	3	X	1	60	**11**	0.5	0.5	0.5	2	2	2	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S83	325	58	A	3	X	1	80	**10**	0.5	0.5	0.5	2	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S84	325	58	A	3	X	1	100	**10**	0.5	0.5	0.5	2	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S85	325	58	A	3	X	1	120	**6**	0.5	0.5	0.5	2	-	1	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S86	325	58	A	3	X	1	140	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S87	325	58	A	4	X	1	20	**18**	0.5	0.5	3	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S88	325	58	A	4	X	1	40	**18**	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S89	325	58	A	4	X	1	60	**18**	0.5	0.5	2	3	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S90	325	58	A	4	X	1	80	**10**	0.5	0.5	0.5	2	2	1	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S91	325	58	A	4	X	1	110	**6**	0.5	0.5	0.5	2	-	1	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S92	325	58	A	4	X	1	130	**7**	0.5	0.5	0.5	2	2	-	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S93	325	58	A	7	X	1	50	**6**	0.5	0.5	0.5	2	-	1	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S94	325	58	A	7	X	2	5	**6**	0.5	0.5	0.5	2	-	1	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S95	325	58	A	7	X	2	55	**4**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S96	325	58	A	7	X	3	25	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S97	325	58	A	7	X	3	75	**9**	0.5	0.5	0.5	2	2	2	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S98	325	58	A	7	X	3	125	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S99	325	58	A	8	X	1	50	**7**	0.5	0.5	0.5	2	2	-	-	-	-	-	x	x	x	x	1.0 ml	-	2 ml	
S100	325	58	A	8	X	2	5	**4**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S101	325	58	A	8	X	2	55	**13**	0.5	0.5	0.5	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S102	325	58	A	8	X	2	105	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S103	325	58	A	8	X	3	45	**6**	0.5	0.5	0.5	2	-	1	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S104	325	58	A	8	X	3	95	**14**	0.5	0.5	0.5	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S105	325	58	A	9	X	1	60	**18**	0.5	0.5	1	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S106	325	58	A	9	X	3	11	**12**	0.5	0.5	0.5	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S107	325	58	A	10	X	1	50	**18**	0.5	0.5	2	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S108	325	58	A	10	X	3	52	**18**	0.5	0.5	3	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S109	325	58	A	11	X	1	50	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S110	325	58	A	11	X	3	53	**5**	0.5	0.5	0.5	2	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
S111	325	58	A	12	X	1	25	**12**	0.5	0.5	0.5	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S112	325	58	A	12	X	2	73	**18**	0.5	0.5	2	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S113	325	58	A	13	X	1	50	**15**	0.5	0.5	1	2	2	2	2	2	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S114	325	58	A	13	X	2	50	**10**	0.5	0.5	0.5	2	2	2	2	-	-	-	x	x	x	x	1.0 ml	2 ml	2 ml	
S115	325	58	A	14	X	1	50	**18**	0.5	0.5	2	2	2	2	2	2		-	x	x	x	x	1.0 ml	2 ml	2 ml	
Ref	**Sample ID**						**IW Rhizone**	**IW**	**Aliquots offshore***									**Analysis offshore / onshore**					**Req 776IODP Mg-Isotope****	**Req 776IODP Sulfur-Isotope**	**Req 776IODP Sulfur-Isotope**	**Remarks**
									Alk	**NH4**	**Cat**	**An**	**d13C**	**d18O**	**d34S**	**DOC**	**Sal**	**Sal**	**Alk**	**NH4**	**Cat**	**An**				

No°	Exp.	Sit	Hol	Cor	Ty	Sec	SecDepth[cm]	total [mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	[mL]	d34S-split [mL]	d13C-split [mL]	
S116	325	58	A	14	X	2	50	**0.5**	0.3	0.3	-	-	-	-	-	-	-	-	x	x	-	-	-	-	-	no CATION + ANION split
S117	325	58	A	15	X	1	50	**2**	0.5	0.5	0.3	0.2	-	-	-	-	-	-	x	x	x	-	-	-	-	no ANION analysis, not enough sample
S118	325	58	A	15	X	2	60	**4**	0.5	0.5	0.5	1	-	-	-	-	-	-	x	x	x	x	1.0 ml	-	-	
																					102	**99**	**93**	**63**	**79**	

Please note:

Requested samples have been shipped off to requester

*** Please note, table shows only IW amount initially sampled and stored, after analysis some aliquots can be used up**

**** Please note, Mg-Isotope splits are from cation splits (per 1ml sample/10µl HNO_3 = 1 Vol% = 0,65 weight% acidification)**