Nautical Bottom Sediment Research

Sub report 8:
Sediment parameter evolution in the STT.

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Van Hoestenberghe, T.; Claeys, S.; Vanlede, J.; De Schutter, J.; Verwaest, T.; Mostaert, F.

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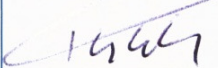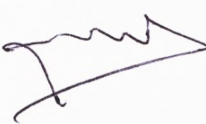
Berchemlei 115
B-2140 Antwerp
Tel. +32 (0)3 224 60 35
Fax +32 (0)3 224 60 36
E-mail: waterbouwkundiglabo@vlaanderen.be
www.waterbouwkundiglaboratorium.be

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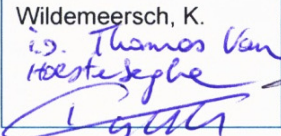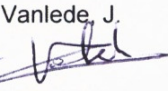| Title:	Nautical Bottom Sediment Research: Sub report 8: Sediment parameter evolution in the STT.			
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Abstract

The Sediment Test Tank (STT) has been built on the terrain of Flanders Hydraulic Research (FHR) to host in-situ sediment for sediment studies. After delivery (16/04/2009) of 50 m³ of sediment from the harbour of Zeebrugge at a density of 1.14 m³/s, sedimentation and consolidation created vertical differences in the sediment in the STT. The measurements started on 21/04/2009. This report deals with the measurements until 26/05/2010.

A whole range of parameters have been measured over time and depth to monitor the sediment characteristics as much as possible. Some parameters are measured in situ in the tank, e.g. measurements by the YSI-multi-parameter probe and pore pressure measured by piezometric tubes. Other parameters are measured on undisturbed samples, e.g. density, rheological parameters and thermo-gravimetric analysis.

This report gives the findings of the measurements and recommendations for further measurement campaigns in the STT.

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1   Introduction

The harbour in Zeebrugge experiences a lot of sedimentation of fines. To ensure a safe passage for the vessels, the depth of the harbour needs to be kept at a target depth. A sediment layer of up to 4 meter can occur. Because of the differences in behaviour in the sediment, from the top to the hard bottom, only a part of sediment layer needs to be dredged.

Relations between the shear stress and density can be obtained for different locations in the harbour. These relations are influenced by the composition of the highly concentrated sediment. The composition of the fluid and consolidated mud is very complex. Even sediment mixtures with different proportion of compounds can have the same density, but will react differently to deformation, e.g. by ships.

Rheology parameters, representing the related forces that a vessel will experience when sailing in or just above the mud layer, are to be found and defined. A measurement method needs to be found to determine these rheology parameters (Claeys et al., 2009).

A lot of effort has been put in trying to test sensors in-situ. Laboratory analysis on undisturbed mud samples analyses is used as reference for evaluating the obtained data.

There has been much research (in-situ and laboratory tests) conducted on the relationship of the sediment characteristics and the impact on the manoeuvrability of ships.

To avoid problems during comparison surveys, a platform was constructed to allow controlled profiling of the sensors. The Sediment Test Tank (STT) has been built on the terrain of Flanders Hydraulic Research (FHR) to host in-situ sediment for sediment studies.

The execution of this research is part of the project plan (offer Gems International) related to the tender 16EB0601. This report refers to paragraph "*4.3.5 Keuze van de onderzoeksparameters en uitvoeringmodaliteiten*" of the project plan.

This report is part of a series of sub reports. The full list of reports is given below in Table 1.

Report id (version)	Report Name
WL2010R751_1a_rev2_0	Comparison of in-situ rheological based instruments in the Sludge Test Tank
WL2011R751_1a_rev2_0	Comparison of in-situ rheological based instruments at Zeebrugge
WL2012R751_1a_3rev3_0	Sub report 3: Individual trials of in-situ rheological based instruments in the Sludge Test Tank "DRDP"
WL2013R751_1a_4rev3_0	Sub report 4: Individual trials of in-situ rheological based instruments in the Sludge Test Tank "Mechanical In-situ Rheometer (MIR)"
WL2013R751_1a_5rev3_0	Sub report 5: Individual trials of in-situ rheological based instruments in the Sludge Test Tank "Rheotune"
WL2013R751_1a_6rev3_0	Sub report 6: Individual trials of in-situ rheological based instruments in the Sludge Test Tank "Rheocable & Accelero Probe"
WL2013R751_1a_7rev3_0	Sub report 7: Individual trials of in-situ rheological based instruments in the Sludge Test Tank "Graviprobe"
WL2013R751_1a_8rev3_0	Sub report 8: Beheersvraag_AMT_Density
WL2013R751_1a_9rev3_0	Sub report 9: Analysis of clay minerals of Zeebrugge mud stored in the STT
WL2013R751_1a_10rev3_0	Sub report 10: Sediment parameter evolution in the STT
WL2013R751_1a_11rev3_0	Sub report 11: Statistical Analysis
WL2013R751_1a_12rev3_0	Sub report 12: Theoretical Review of Cohesive Sediments Rheology, General Guidelines for Numerical Modelling and Laboratory Measurements
WL2013R751_1a_13rev3_0	Sub report 13: Cohesive Sediments Dimensional Analysis

Table 1: Sub reports in the 'Sediment Related Nautical Bottom Research Phase I: Measuring Techniques' project.

2   Sediment test tank

The Sediment Test Tank (STT) was built on the terrain of Flanders Hydraulic Research (FHR) to host in-situ sediment for sediment studies.

Sediment from the Wielingendok (Zeebrugge) was delivered in the STT on 16/04/2009 at a density of 1140 kg/m³.The sediments were diluted to this density to pump the material into the STT.

A full description of the dredged sediment at Zeebrugge and the test tank is given in the report 'Nautical Bottom Sediment Research: Comparison of in-situ rheological based instruments' (Claeys et al., 2009).

In Figure 1 a side view of the sediment test tank (STT) is given. The STT is divided in 3 mayor compartments: a test tank, a sediment selection tank and a pumping container. The sediment of the Wielingendok is stored in the test tank compartment. A mobile bridge can be moved over the tank to perform in situ measurements and take undisturbed samples.

Figure 2 gives a top view of the test tank, with dimensions 9,64 x 2,44 x 3,11 m. To avoid profiling on the same (disturbed) location, the positions of the profiles were mapped. Marks have been added to the STT nearby the mobile bridge. Also the plates in the mobile bridge were numbered. A taken profile is numbered according the following procedure: '*D6profiel2;1C2"*:

-    'D6' (day after sediment delivery)
-    'Profiel2' (profile number of the day)
-    '1' (location of the bridge)
-    'C2' (position on the bridge).

Sedimentation and consolidation creates vertical differences in the sediment. A whole range of parameters have been measured over time to monitor these differences.
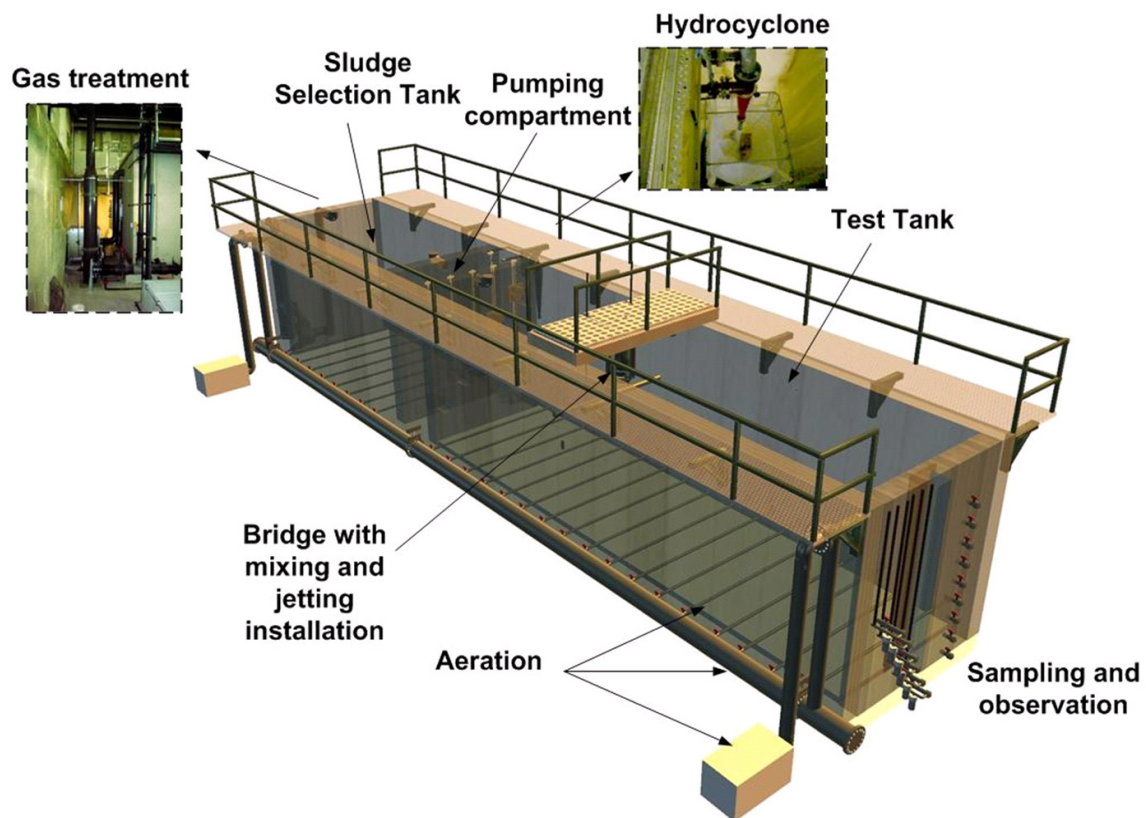
Figure 1: Sediment test tank - side view

Figure 2: Test tank compartments of the sediment test tank –top view.
The blue box represents the 5B4th position: 5 is the location of the bridge (vertical numbering: 1-9) and B is the position on the bridge. In the B (horizontal numbering: C-B-A) position 4

3   Measurement methods

3.1   General

Sedimentation and consolidation create vertical differences in the sediment in the STT. A whole range of parameters have been measured over time and depth to monitor the sediment characteristics as much as possible.

Lateral, little difference can be expected in the sediment characteristics because of the profound mixing of the sediment immediately after delivery in the STT. The presence of lateral differences is checked by comparing measurements at different locations in the test tank.

3.2   Measurement period

Delivery of the sediment took place on Thursday 16/04/2009. The measurements, however, only started on Tuesday 21/04/2009. Between Thursday and Tuesday the sediment was aerated to avoid consolidation. The aeration caused a profound mixing of the sediment, which reduced the lateral differences of sediment properties. The effect of aeration on the consolidation process is however unknown and will be analysed in phase two of the project. The measurements started on 21/04/2009 with completely mixed and aerated sediment, except at the bottom where consolidated mud ripples with a maximum height of 14 cm are present.

This report deals with the measurements until 26/05/2010. That is 406 days after the delivery of the sediment ('day 406'). In this report we only focus on 'cycle 1' because during these 406 days the mudlayer has not been stirred up. However, not all parameters are measured every monitoring day. In table 1 the dates and measured parameters are given. On some days, different profiles were taken. Sediment- and water level are always measured on the dates mentioned in table 1. A cross (X) means that the mentioned parameter was measured for that day. When empty the parameter was not measured at that time. The sediment height en water level were always measured and is therefore not included in the table.

Table 2: Dates and measurement types

Date	Day number (from 15/04/09 on)	Pore pressure	Undisturbed soil sample**	YSI-measurement*	Number of profiles
21/04/09	6		X		6
22/04/09	7		X	X	6
23/04/09	8	X	X	X	4
24/04/09	9	X	X	X	4
27/04/09	12	X	X	X	2
28/04/09	13	X	X		2
29/04/09	14	X	X		2
30/04/09	15	X	X	X	2
04/05/09	19	X	X	X	2
07/05/09	22	X	X	X	2
11/05/09	26	X	X	X	2
15/05/09	30	X	X	X	2
20/05/09	35	X	X	X	2
27/05/09	42	X	X	X	2
04/06/09	50	X	X	X	1
16/06/09	62	X	X	X	1
17/06/09	63		X		1
18/06/09	64		X	X	3
19/06/09	65		X	X	1
02/07/09	78	X	X	X	1
16/07/09	92	X	X	X	1
24/07/09	100				1
01/09/09	139				1
03/09/09	141	X	X	X	1
08/09/09	145				1
16/09/09	154	X	X		1
29/09/09	167	X	X	X	1
22/10/09	190	X	X	X	1
26/10/09	194			X	1
10/11/09	208	X	X		1
26/11/09	225	X			1
01/12/09	230	X	X	X	1
14/12/09	243	X	X	X	1
24/12/09	253	X			1
08/01/10	268	X	X		1
29/01/10	289	X	X		1
12/02/10	303	X			1
19/02/10	310	X	X		1
05/03/10	324	X	X		1
25/03/10	344	X	X		1
02/04/10	352	X		X	1
15/04/10	365	X	X	X	1
28/04/10	378	X	X		1
12/05/10	392	X	X		1
26/05/10	406	X	X	X	1

*temperature, pH, Oxido-Reduction Potential, turbidity, conductivity, Dissolved Oxygen

**Analysis on undisturbed soil sample are: density in lab-condition, thermo-gravimetric analysis (dry matter, organic matter and carbon content), rheology parameter, grain size distribution

3.3   In situ measurements

Some parameters are measured in situ in the tank:

- Sediment height & water level
- Temperature, pH, Oxido-Reduction-Potential, turbidity, (specific) conductivity and dissolved oxygen (YSI-multi-parameter probe)
- Pore pressure is measured by a set of filters connected to the tank

3.3.1   Water/sediment level

The sediment height (height from the bottom of the tank to the interface level of sediment and water) and water level can easily be monitored in the tank trough a glass window. Sediment height is a first measure for consolidation. Monitoring of the water height is needed among others for calculating the water overpressure in the sediment. Figure 3 shows the glass window in the centre of the tank. On the left side are the pore pressure tubes. Figure 4 illustrates the easy readings through the glass window.
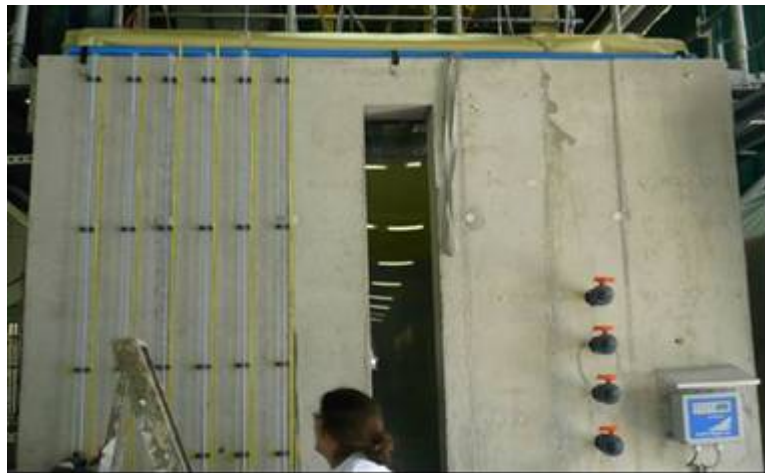
Figure 3: Glass window in the tank where sediment height and water level can be monitored easily.
On the left hand of the glass window are the pore pressure tubes
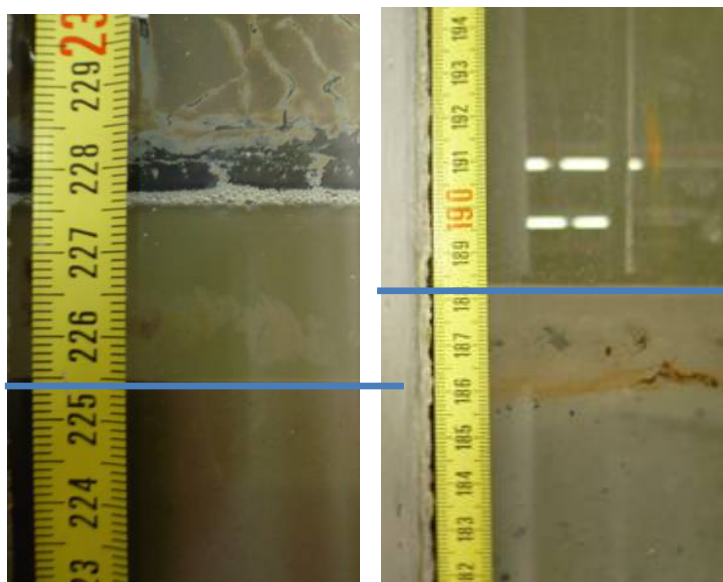
Figure 4: Sediment height observed through the glass window in the tank,
day 8 (23/04/2009) left and day 64 (18/06/2009) right (the blue line is the interface level between the sediment and the 'clear' water. A very clear contrast can be observed between the water column and mud column)

3.3.2 Pore pressure

The pore pressure evolution is essential in the analysis of the sediment consolidation. Pore pressures are measured by piezometric tubes, inserted on different heights from the bottom of the test tank till 120 cm above the bottom of the tank. The pore pressure can be read by riser tubes, connected to the piezometric tubes with a filter (Figure 5). The filter prevents the intrusion of sediment in the riser tubes. Disadvantage of the filter system is possible blocking of the filter by sediment, which can disturb the pressure readings. Filters are polyester membranes. Rising tubes have a diameter of 2 cm, so capillarity is negligible.

The tubes were filled with water immediately after filling the tank and before opening of the valves (Figure 5), for achieving the equilibrium in the piezometers as fast as possible. Osmotic pressure is not a possible issue because of the polyester membrane (20 µm).

Figure 5: Piezometric tubes and filters (left) were immediately filled up with water after filling up the tank (right)

3.3.3 YSI-profiling

Parameters

Temperature, pH, Oxido-Reduction-Potential (ORP), turbidity, (specific) conductivity and dissolved oxygen (DO) are all measured by a multi-parameter probe of the mark 'YSI'. From the conductivity measurement the salinity can be deducted. The YSI-probe is of the type '6920V2-M' (Figure 6).

Figure 6: Multi-parameter probe '6920V2-M' of the mark 'YSI'.

A pressure sensor is inserted in the probe (Figure 7), which measures the depth of the probe under a liquid. The following (discrete) sensors are mounted on the multi-parameter probe:

- 6150 Dissolved Oxygen sensor (Figure 8)
- 6136 Turbidity sensor (Figure 8)
- 6560 conductivity/temperature sensor (Figure 9)
- 6565 pH/ORP sensor (Figure 9)

Figure 7: Pressure probe and different sensors inserted in the multi-parameter probe '6920V2-M'

The dissolved oxygen and turbidity sensor measure at a distance of 18 cm from the pressure sensor. The conductivity/temperature sensor and pH/ORP sensor measure at a distance of 13 cm from the pressure sensor (Figure 7).

Figure 8: Dissolved Oxygen sensor (left) and turbidity sensor (right)

The measuring method for dissolved oxygen (Figure 8) is based on the reduction of the present O_2 in the aqueous solution to OH^-ions by polarised sensors with a negative tension. This tension is converted to a current by 2 electrodes that are correlated with the concentration of dissolved O_2 in the water. The effect of temperature on dissolved oxygen is significant: +- 3% per °C. This dependence on temperature is reduced to 1% by the measuring system, which in return is corrected by the YSI-software after measuring.

The turbidity is influenced by the content of suspended solids in water and is measured by the amount of light that is scattered by the particles. The light source in the YSI-system is a LED that produces radiation in the near infrared region of the spectrum. Because of the high concentration of the mud, the turbidity sensor will experience a full multiple scattering. No light is scattered back to the sensor and gives low erroneous values for high concentrated sediment. Therefore the zone between the suspended solids and the forming of a mud layer cannot be distinguished. The turbidity measurements were useless in the sediment test tank, and are not further analysed nor discussed. Still the turbidity sensor could be used for further sedimentation research, if needed.

Figure 9: Conductivity/temperature-sensor (left) and pH/ORP-sensor (right)

The conductivity sensor (Figure 9) measures the conductivity of solutions of ionized substances by the level of voltage drop between four pure nickel electrodes. The measured voltage drop is then converted to a conductance value in milli-Siemens. The conductivity highly depends on the temperature. Because of this dependence, the YSI-software calculates the 'specific conductance', which is the conductivity corrected to a reference temperature of 25° C. The salinity of the solution is automatically deducted from temperature and conductivity measurements according to algorithms found in Standard Methods for the Examination of Water and Wastewater (www.standardmethods.org, ed. 1989).

The temperature is measured by a thermistor of sintered metallic oxide that changes in resistance with temperature variation. Temperature and conductivity sensors are integrated in the same sensor housing (Figure 9).

The ORP sensor measures the redox potential, which is the tendency of a chemical substances to acquire electrons and will thereby be reduced. The more positive the potential, the greater the substances' affinity is for electrons and tendency to be reduced. Because the absolute potentials are difficult to measure accurately, reduction potentials are defined relative to a reference electrode (difference in potential between an inert electrode and a reference electrode). The measurements are not compensated for temperature, neither in the sensor nor in the software. The dependence is illustrated by the difference of ORP for a same solution at 15°C (244 mV) and 25°C (231 mV).

The pH is measured by means of a combined glass electrode, which measures the potential difference between an electrode sensitive to the hydrogen ion activity and a reference electrode. The pH is influenced by the temperature: as temperature increases the solution becomes more acidic. This dependence is compensated in the measurements. The pH and ORP sensor are integrated in the same sensor housing (Figure 9).

Profiling

YSI-measurements are depth-referenced in a liquid by a pressure sensor (the depth reading in the watercolumn was accepted as correct (manufacturer calibration); no calibration carried out). To reference the YSI-measurements in sediment, a clear correlation must exist between the measured YSI-pressure and the height of the sensor in the sediment column (measured by measuring tape). For a number of YSI-profiles for days D64 (3 profiles), D194 and D365 (1 profile) the height of the YSI-sensor were recorded with an interval of 10 to 20 cm. The results of these reference profiles are given in Figure 10 and Figure 11. Because of maintenance manipulation, the YSI-sensor for D365 has different configuration settings for the pressure sensor than the YSI-sensor for D64 and D194, what causes the YSI-readings to differ considerably from the measurements on D64 and D194.

The reference profiles for YSI-measurements in sediment and water are given in Figure 10 for days D64, D194 and D365. In this figure the measurements in both the sludge and water are given together with a trend line to state the linearity. A first linear relationship is noticed when in the sediment and a second and different relationship noticed when in the water. This is very clear for day 365. The figure hence illustrates that the pressure readings are correlated with the height above the bottom by means of linear relation, but that this relationship differs for sediment and water.

MEASUREMENTS IN SLUDGE & WATER
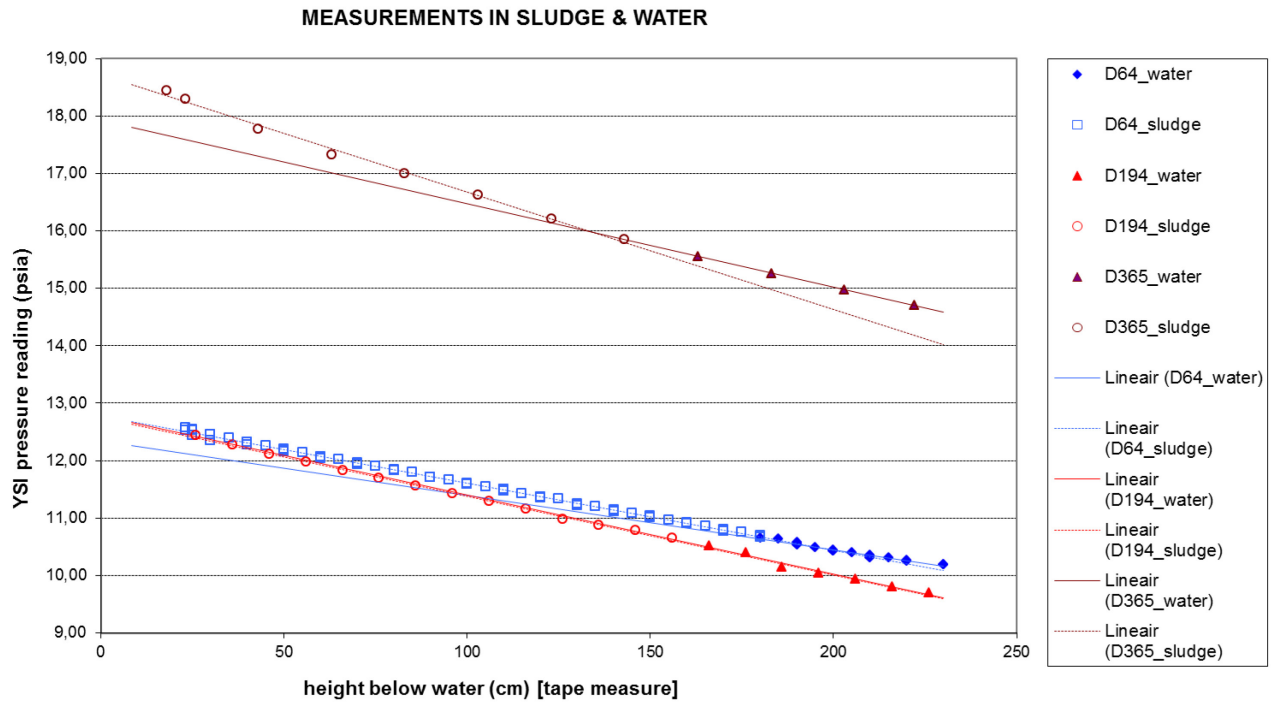
Figure 10: Reference profiles for YSI-measurements in sediment and water for days D64 (4 profiles), D194 and D365. Pressure measurements by the YSI-sensor are given on Y-axis.
In this figure the measurements in both the sludge and water are given together with a trend line to state the linearity

MEASUREMENTS IN SLUDGE
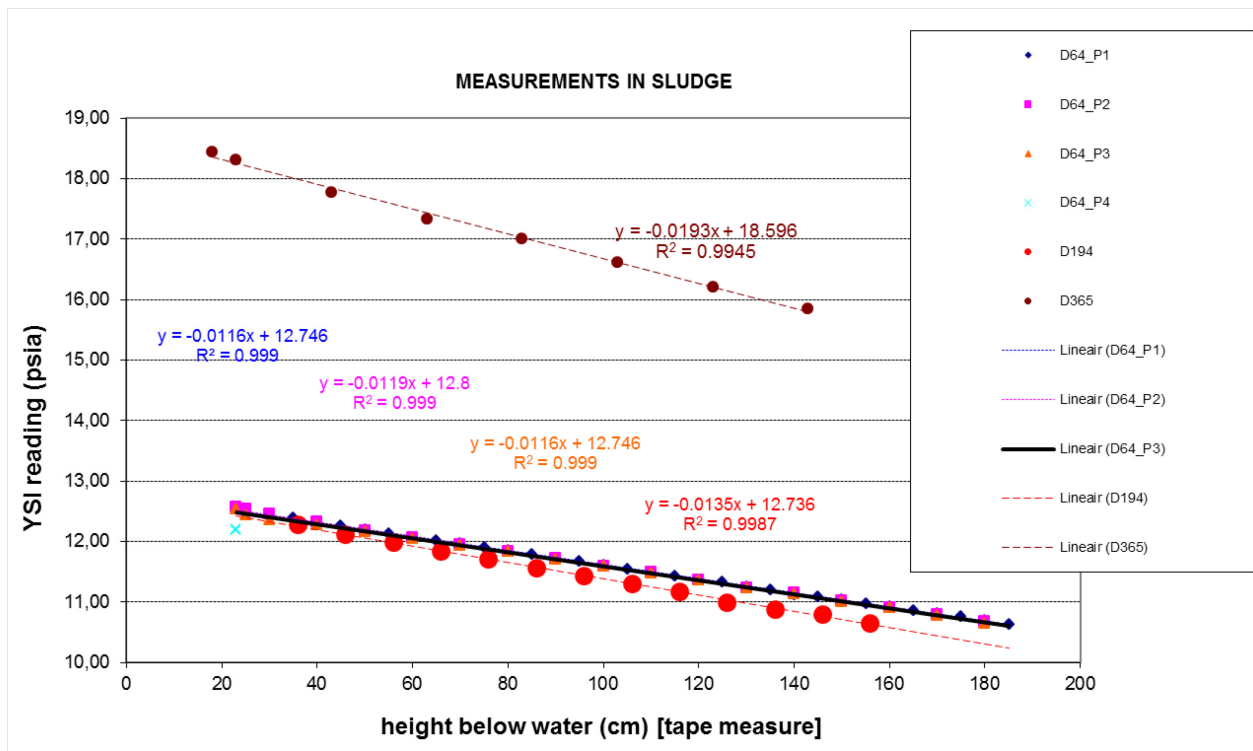
Figure 11: Reference profiles for YSI-measurements only in sediment for days D64 (4 profiles), D194 and D365. Pressure readings (Y-axis) are linear correlated with height above bottom (X-axis)

In Figure 11 only the measurements in the sediment column are presented. For each profile (4 for D64, 1 for D194 and 1 for D365) a linear regression is fitted. The percentage of variance in YSI readings explained by the height readings is at least 99.5% for every profile. Although the YSI-sensor has identical configuration settings, regression relations between the profiles of D64 and D194 differ significantly, because of the change in sediment characteristics (see Figure 36). Most important for depth referencing of the YSI-measurements is that the relation is linear for each profile in the sediment column.

In Figure 10 and Figure 11 the YSI reading is given in pounds per square inch absolute (PSIA). A conversion to kPa is made by multiplying the YSI reading by 0.1449 (YSI incorporated, 2010).

The relation between YSI-readings and height above the bottom of the tank is slightly different in the more consolidated sediment at the bottom of the profile for D194 and (more pronounced) for D365. However, the effect of the more consolidated sediment layers does not influence depth readings to that extent that it has to be corrected.

With the linear relations, the YSI-measurements (pH, conductivity …) can be depth-referenced by linear interpolation of the pressure readings between top and end of the sediment column. The top and bottom of the sediment column can be obtained easily from the different YSI-measurements (e.g. turbidity). The end of the profile is always 18 cm above the bottom of the tank, because the pressure sensor is integrated 18 cm above the top of the YSI-sensor (Figure 7), and the YSI-sensor is always profiled till the bottom of the tank.

The reference profiles have proven that the depth of the probe can be measured by the pressure sensor in sediments as well, but that a different relationship has to be established for water and sediment measurements (see Figure 10). When the mud is more consolidated, it has to be verified whether the relationship between pressure reading and depth does not differ too much with the relationship in the less-consolidated layers. In the STT, this is not the case.

3.4 Undisturbed soil sample

The references for all experiments are the samples taken with the Beekersampler. The improved version of the Beekersampler is used, under the name 'Sediment core sampler, type Beeker'. The Beekersampler takes an uncompressed sample of the bulk sediment, but cannot sample the lowest 10 cm because of the limitation of the sampling head. When sampling a sediment column more than 2 m high, the whole column had to be divided into two different soil samples.

The undisturbed soil sample can be partitioned in a controlled way, by the dividing system (Figure 12). Sub sample every 10 cm were taken, and treated the same day in the sediment laboratory of the FHR. Stirring of the subsamples (for degassing) did not influence the density in a significant way. A few, not registered, stirring tests have been executed to test a possible difference. Splitting the sub sample allowed us to take an undisturbed soil sample every 5 cm. They have been analysed for the following parameters:

- Density
- Grain size distribution
- Rheological parameters (shear rate and shear stress)
- Thermo-gravimetric analysis (dry matter, organic matter and carbonate content)
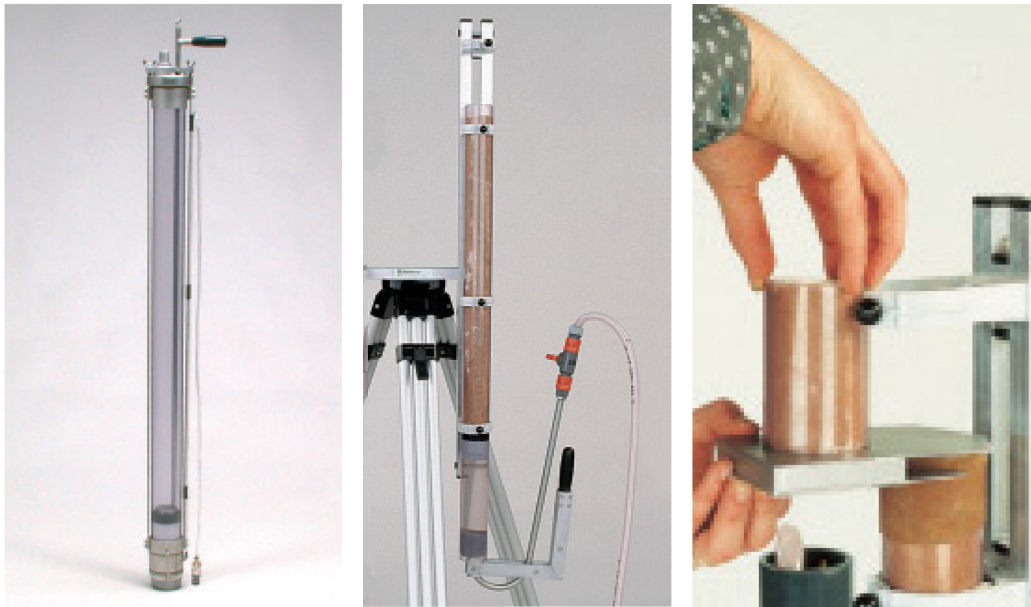
Figure 12: Beeker sampler ready for use (left), dividing system for taking sub samples (middle and right)

No samples were taken close to the walls of the tank, because of possible inhomogeneous mixing by aeration nearby the walls after delivery of the sediment.

Density measurements

The density meter 'DMA 38' from Anton Paar has been used for density measurement. The DMA38 uses the oscillating U-tube method. Sediment is injected in the tuning fork system by an injection tube (Figure 13). The density meter has following specifications:

- Density range 0 to 3 g/cm³ ± 0,001 g/cm³ (=1 kg/m³)
- Temperature range 15°C tot 40°C ± 0,2°C
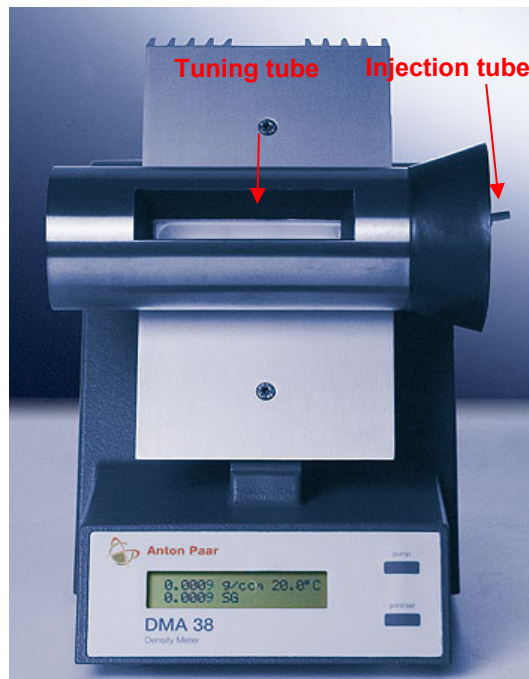
Figure 13: Density meter 'DMA 38' from Anton Paar.
The sediment is injected in the tuning fork system by an injection tube (on the right side)

When gas bubbles are present in the samples, an error is displayed on the instrument screen: "please fill". Besides difficulties for pumping the highest density sediments (1300 kg/m³) into the small tuning tube, density measurements were executed without problems. The technical sheet is in Annex 1: comparison measurement data.

Grain size distribution

The grain size distribution was measured with the Particle Size Analyser 'Mastersizer 2000'. The Mastersizer uses laser diffraction for particle sizing. Particles are passed through a focused laser beam. These particles scatter light at an angle that is inversely proportional to their size. The angular intensity of the scattered light is then measured by a series of photosensitive detectors. The number and positioning of these detectors in the Mastersizer 2000 makes a 0.02-2000μm measurement range possible. More information can be found in Annex 3: technical sheet of the Particle Size Analyser 'Mastersizer 2000'.

Rheological parameters

The rheometer Anton Paar Physica MCR 301 has been used to determine the rheological parameters from the sediment. The MCR 301 is a shear stress and shear rate controlled device. The rheometer automatically dilutes until a good laser obscuration (10- 15%) is reached. Flocculation is hindered by using ultrasonic sound waves. More information can be found in Annex 4: technical sheet of the Anton Paar DMA 38.

In order to avoid slip of the bob in the sediment sample, no cylinder but a vane system is used. To compare this study with other research (Toorman 1994; Kelessidis 2010), a shear-rate controlled test is chosen. The applied shear rate increases logarithmical from 0.1 1/s to 1000 1/s. To visualise the yield-stress part of a rheogram, a focus on the lower shear-rate part was required. To find the equilibrium stage of the rheogram, enough high shear-rate points were also needed. In total, 14 measuring points were chosen: 0.09999; 0.2031; 0.4124; 0.8376; 1.701; 3.455; 7.017; 14.25; 28.94; 58.78; 119.4; 242.5; 492.4; 1000 1/s. More details on the measurement method and the 'shear-rate versus shear stress' flow-charts can be found in Claeys et al., 2009. In a later phase of the research, it became clear that the applied shear rates higher than 600 1/s gives erroneous data due to the creation of a vortex into the cup. This means that the measurement protocol needs to be changed. An adapted protocol is being used for the future analyses. For the data of this report, all the data is used. A new processing would advisable for fine-tuning the results. As the relative evolutions (trends, correlations) were not affected by the high shear rates, the data was still usable without filtering.

Thermo-gravimetric analysis

Dry matter, organic matter and carbonate content are measured by drying and ignition with the Prepash 229. Up to 29 specimens and a reference pan can be dried and ashed in one working cycle (Figure 14).

Figure 14:Thermo-gravimetric analyser 'Prepash' from Anton Paar

The samples are measured by an analytical balance while increasing the temperature. Weighing curves are recorded over time for each individual test sample. This permits an automatic end-point determination and control during the entire drying and ashing process. The dry matter content is measured at a temperature of 105°, the organic matter content at a temperature of 550° and that of carbonate at a temperature of 800°C.

The organic matter content can be derived from the measurements:

$$\%OM\ (550) = 1 - \%LOI\ (550).\frac{100-\%moisture}{100}$$ *Equation 1*

Where:

- LOI = loss on ignition, value given by thermo-gravimetric analyser (%)
- % moisture = loss on ignition at 100°C

The organic carbon content can be derived by assuming an average content of 56% carbon content in organic matter (Vanhoof, C. and Tirez, K.2003):

$$\%OC\ (550) = \frac{\%OM(550)}{1.8}$$ *Equation 2*

Some sediment samples were not heated long enough at 105°C to release all the moisture from the sample. This can be seen by a sudden decrease in the weight of the samples shortly after raising the temperature beyond 105°C. In Figure 15 the two upper curves have not released all their moisture content at 105°C. Temperature had to be risen to +- 200°C for measuring dry content. This was mainly so for bigger samples or samples with a high dry matter content. The extrusion speed of water is reduced due to an increased mass.
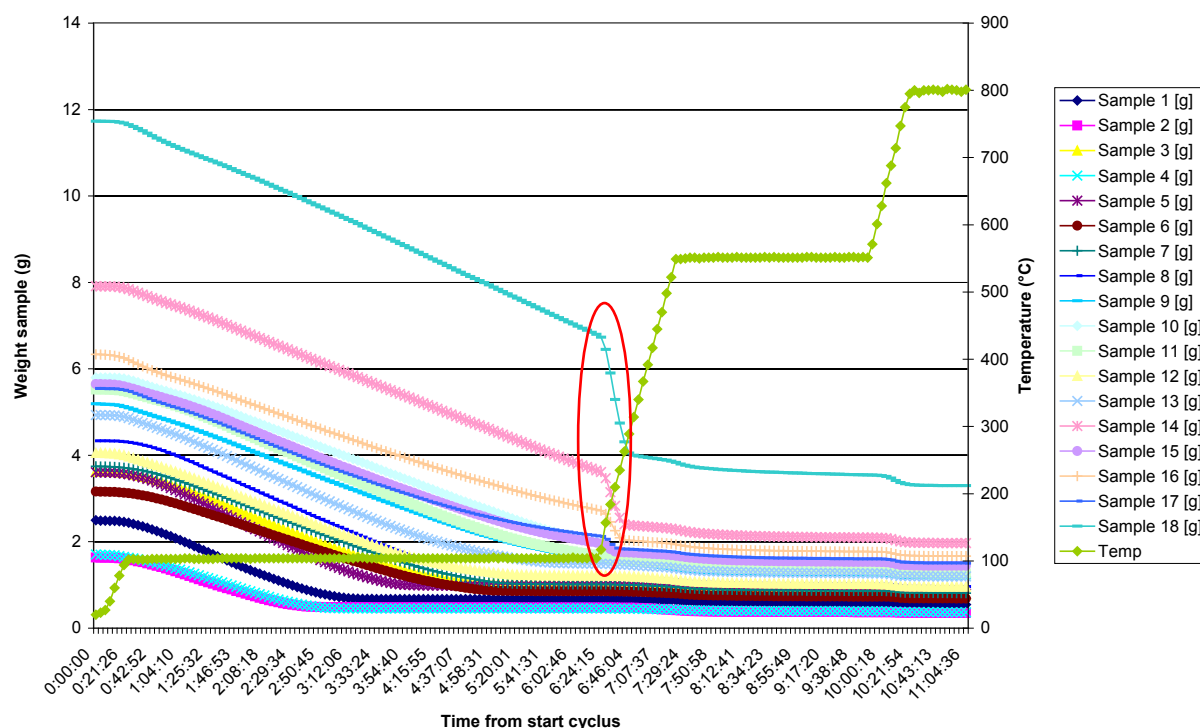
Figure 15: Weighing curves for subsamples from the STT on 16/9/2009 (D154).
The temperature is the green curve. For sample 9 (upper blue curve) and sample 14 (upper pink curve) 105°C was not enough to release all the moisture from the sample. The temperature had to be increased to +- 200°C for measuring the actual dry content (red circle)

4   Results

4.1 Sediment height and water level

In Figure 16 the evolution of the sediment height and water level is shown for the period 21/4/2009 (d7) till 28/4/2010 (d378). The height of the mud layer was observed at the observation window, but also monitored with a measure tape and disk over the whole length of the STT. There is lateral (over the STT) almost no difference measured (< 1cm) of the mud level during consolidation (cycle I).

The water height increased a little in the beginning, possibly because of gas creation. The overall water level decreases in time because of evaporation. Also, because of an observation error, the waterlevel is fluctuating. In Figure 17 the consolidation speed is given. The consolidation speed is not constant, but decreases with time. Immediately after the aeration phase the consolidation speed reaches 2 cm/day, but slowed down quickly to a speed of 0.5 cm/day in June 2009. In June/July 2009, when the temperature of the sediment increased strongly (Figure 16), the consolidation speed increased to 1 cm/day. It is not clear if this value should be classified as an outlier or not. The most plausible explanation of the sudden increase in consolidation speed is a lower viscosity of the sediment with increasing temperature (De Schepper et al., 1996). After this short increase, the consolidation speed decreased further to +/- 0.1 cm/day in 2010.

When analysing the relationship between temperature (of the water column above the sediment layer) and consolidation speed (as shown in Figure 17), no clear correlation can be found between the two parameters. Influences of other factors are probably masking the expected positive correlation between temperature and consolidation speed (De Schepper et al., 1996, Peeters, 1990).

Figure 16: Evolution of sediment height and water level for the period 21/4/2009 till 28/4/2010
(Note the sudden decrease in sludge level halfway June (blue circle))
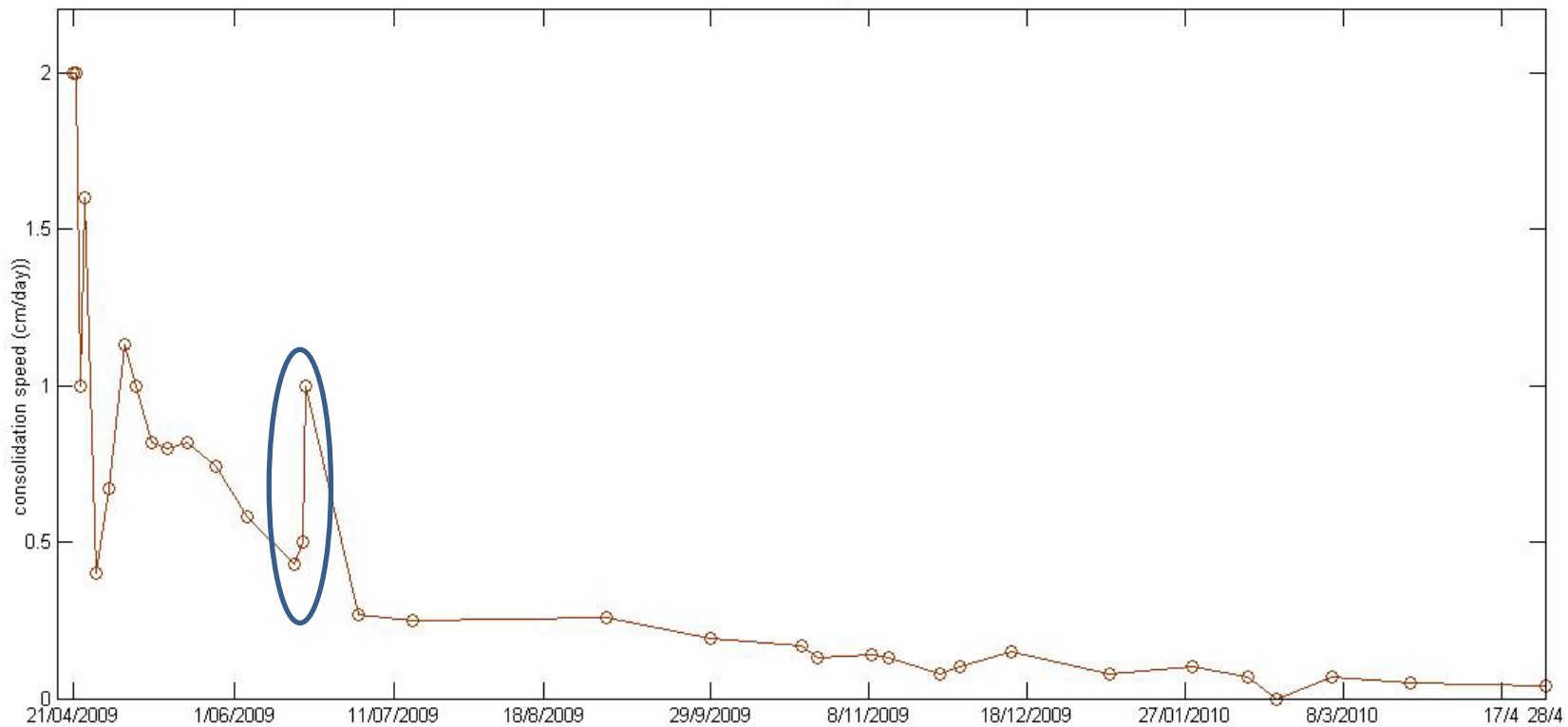
Figure 17: Consolidation speed for the period 21/4/2009 till 28/4/2010
(note the sudden increase in consolidation speed halfway June (blue circle))

4.2 YSI

4.2.1 Comparative measurements

The YSI-sensor used in this study is designed to measure in aqueous solutions, not in sediment. It had to be tested whether the measurements in sediment are comparable with the measurements in the aqueous solution resulting from the centrifuged pore water. The amount of deviation between measurements in sediment and measurements in the aqueous solution are an indication for the quality of the measurements in the sediment. Large differences, and hence lower quality of measurements, are particularly expected for the higher densities. For this objective, 26 sediment samples were prepared with a density ranging from 1.05 till 1.3 g/cm³. The different YSI-parameters were first measured in the sediment samples (Figure 18). After measuring, the sediment samples were centrifuged at a speed of 7000 rpm until enough aqueous solution was available for measurements with the YSI probe. First, the stabilisation time of the measurements in sediment (in the sediment test tank) and water was investigated. For pH, temperature and conductivity the measurements stabilised after less than 10 seconds. For the other parameters (oxide-reduction-potential and dissolved oxygen) the recommended stabilisation time is 120 seconds. These recommended stabilisation times are valid for measurements in the sediment (and hence without centrifugation). The deviation of the measurements in sediment from the measurements in the aqueous solution after centrifugation of the sediment was investigated for different densities. Measurements were done with a YSI multi-parameter probe (type 6920V2-M, Figure 6). The sensors on the probe are at least calibrated once a month. The relative errors for YSI-measurements in sediment samples compared to YSI-measurements in centrifuged pore water are given in Table 3. The relative errors are calculated as the ratio of the absolute values of the difference between sediment and pore water measurements and the water measurement.

In [Annex 1: comparison measurement data] the measurement results are given in more detail. From it is clear that the pH and temperature measurements are similar for sediment and pore water. For the conductivity measurements there seems to be a relationship between density and difference between sediment and pore water measurements. This correlation is shown in Figure 19. For most sediment samples with density lower than 1.2 g/cm³ the difference between sediment and pore water measurement is smaller than 30%. For densities higher than 1.2 g/cm³ the difference is almost always above 50%. Figure 18 (at right) shows a possible explanation. For higher densities, the sediment does not fill the vent hole of the conductivity sensor. This prevents the electrodes to be completely inserted in the sediment which gives a systematic underestimation of the conductivity of the pore water. For densities higher than 1.2 g/cm³, this underestimation is nearly constant at 60-70%.

For the dissolved oxygen (DO) and oxide-reduction potential (ORP) measurements, the comparison between the sediment measurement and the pore water measurement is difficult. The processes of centrifugation and manipulation of the sample will enrich the pore water with oxygen, which causes the DO and ORP content of pore water to be systematically higher than the content of the sediment sample. Therefore, the comparison between sediment and pore water is not straightforward.

Measured profiles in the STT however show that DO and ORP sensors are measuring an expected decrease in ORP and DO. A measured YSI-profile is shown in Figure 20for 2/4/2010 (D352). The probe was kept steady for 120 seconds every 20 cm (Parameter 'Pressure' = depth). The probe is inserted in the water layer above the sediment at 15:24 and reaches the sediment at 15:30. At 15:44 the probe reaches the bottom of the tank. The ORP reduces slowly from more than 200 mV in the water to close to -100 mV at the bottom sediment layer, which seems to be normal values for these conditions (Schöring et al., 1999). The reduction in DO towards the bottom of the tank is faster than the reduction in ORP, but also these values (from 60% at the top of the sediment to 2% at the bottom) are in the range of the expectations (De Lange et al., 2006). Therefore, the ORP and DO parameters will be analysed as well in this report.

In contrast to the profile of Figure 20, the measurements analysed in this report (for the year 2009) had a stabilisation time of only 5 seconds. For the pH, temperature and conductivity measurements this has no effect on the readings. But as stated before in this paragraph, the recommended stabilisation time for DO and ORP is 120 seconds. Therefore, the DO and ORP vertical trend might not be representative for the ORP and DO content in the sediment or pore water. It will hence be impossible to make solid conclusions for these parameters, but this will at least give an idea about the real parameter values in the sediment or pore water. It should be mentioned as well that the conditions in the pore water were

different from those in the sediment and hence it was impossible to compare and validate the obtained measurements.

A new pH/ORP sensor (Figure 9) was installed on the probe on January 2010. The membrane from the dissolved oxygen sensor (DO) was renewed in March 2010. To see the difference in measurement results, the average error results for 2009 and 2010 were distinguished in . The change of sensors had little impact on the relative errors between sediment and pore water measurements. This doesn't allow to analyse the possible difference in quality of both sensors in 2009 and 2010. But because of the (at least) monthly calibration (see § 4.2.1), the quality of the readings for both pH/ORP en DO-sensors can be considered as not influenced by the change of sensors.

Figure 18: Laboratory setup for comparative measurements with the YSI-probe.

At the right: the sensor is removed from the sediment sample after measurement. For higher densities, the sediment did not completely fill the vent hole of the conductivity sensor, which can lead to erroneous data

Figure 19: Underestimation of pore water conductivity (%) by sediment conductivity measurement, in function of density (g/cm³)

Table 3: Relative error (%) of the YSI-measurements in sediment samples against the YSI-measurements in pore water.
(Measurements are sorted with increasing density)

Density (g/cm³)	DO	Spec Cond	Error (%) pH	ORP	T°	date measurement	Treatment sediment sample
1.05	50	2	1	26	3	1/12/2009	without further treatment
1.05	92	7	8	43	3	1/12/2009	Heated till +- 25°C
1.05	62	73	1	122	3	7/12/2009	without further treatment
1.1	15	99	7	129	10	1/12/2009	without further treatment
1.1	21	18	6	20	6	1/12/2009	Heated till +- 25°C
1.1	65	0	0	154	3	7/12/2009	without further treatment
1.1	5	20	0	85	1	April 2010	without further treatment
1.11	92	20	2	281	4	April 2010	without further treatment
1.12	91	24	2	310	5	April 2010	without further treatment
1.14	81	26	3	443	5	April 2010	without further treatment
1.15	59	63	4	38	3	1/12/2009	without further treatment
1.15	75	23	3	57	2	1/12/2009	Heated till +- 25°C
1.15	56	24	1	115	4	7/12/2009	without further treatment
1.16	91	29	2	2139	5	April 2010	without further treatment
1.19	76	58	1	1270	4	April 2010	without further treatment
1.2	91	33	3	737	5	April 2010	without further treatment
1.2	52	52	4	12	9	1/12/2009	without further treatment
1.2	61	76	4	40	0	1/12/2009	Heated till +- 25°C
1.2	0	64	2	14	1	April 2010	without further treatment
1.23	86	56	2	100	2	April 2010	Original sediment, 80-120 cm above bottom tank
1.25	79	66	3	10	0	April 2010	Acidified (+ 3M HCl)
1.25	91	66	2	272	0	April 2010	De-acidified (+ 3M NaOH)
1.25	90	66	1	254	2	April 2010	De-acidified (+ 3M NaOH)
1.26	87	68	2	94	0	April 2010	Original sediment, 40-80 cm above bottom
1.28	223	68	0	66	3	April 2010	without further treatment
1.29	92	69	4	303	1	April 2010	Original sediment, 0-40 cm above bottom
Average error	72	45	3	274	3	●	●
Av. error 2009	55	40	4	69	4	●	●
Av. error 2010	85	49	2	425	3	●	●

D352_PO1.DAT
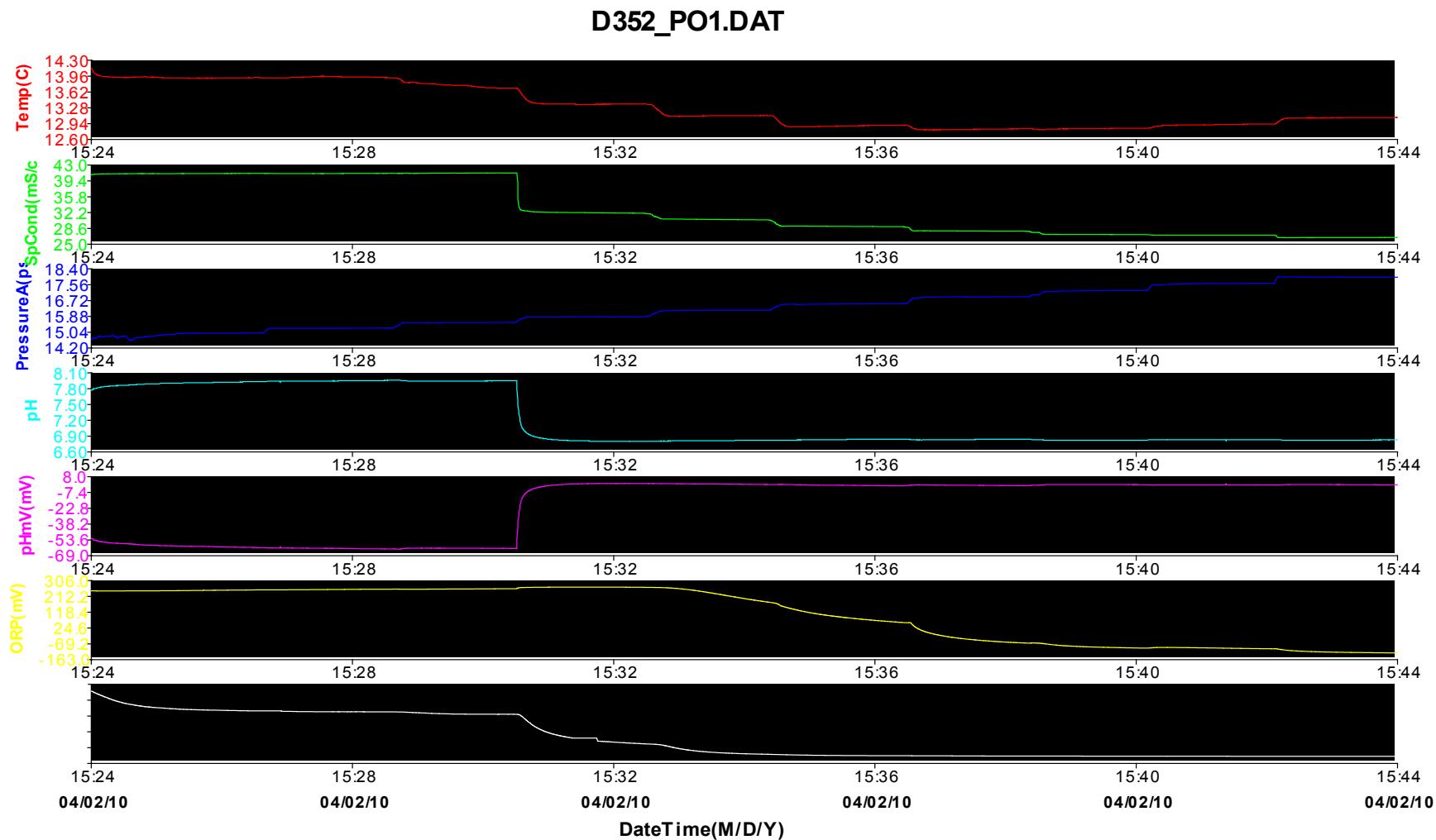
Figure 20: Profiling with YSI-probe on 2/4/2010 (D352) in the STT, with stabilisation time 120 seconds.
From 15:24 till 15:30 the YSI-sensors are measuring the water column above the sediment,
at 15:30 the sensors are penetrating the sediment till they reach the bottom of the tank at 15:44

4.2.2 Temperature

Results for temperature and other YSI-parameters will be shown and analysed for the measurements from 22/4/2009 (D7) until 26/10/2009 (D194). After 26/10/2009 there were technical problems with the probe (corrosion of an electronic connection) which may lead to erroneous data. As a consequence, these data are not presented here.

In Figure 21, Figure 22 and Figure 23 the temperature evolution is shown from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT. In Figure 21 all measurements are shown. More details are given for some reference days (Figure 22) and reference depths (Figure 23).

From Figure 21 it can be seen that the temperature of the sediment for the period 22/4/2009 till 26/10/2009 is between 15 and 21°C. Variations over the height in the tank are relatively small. During the summer season (d78 – d167) the temperature in the tank was at least 18.5°C.

Figure 22 shows the vertical variation for some reference days. Most of the days, the temperature of the top sediment differs 1 to 2°C from the bottom sediment. During summer season (d64, d92 and d141) the top sediment is warmer than the bottom sediment, due to higher air and water temperature. Water has a larger heat conductivity than sediment, what causes the water above the sediment to react faster to temperature changes than sediment. During autumn and spring the air and top sediment is colder than the sediment, what gives an increase in temperature towards the bottom. Because of the higher heat conductance of the concrete floor, the temperature decreases again near the bottom.

Figure 23 shows the temperature evolution for some reference depths trough the tank. The general evolution is for all depths the same, but due to the buffering capacity of the sediment the variation in temperature throughout the measurement period reduces towards the bottom. The temperature differences between depths for a given day are the highest in the beginning and at the end of the measuring period.
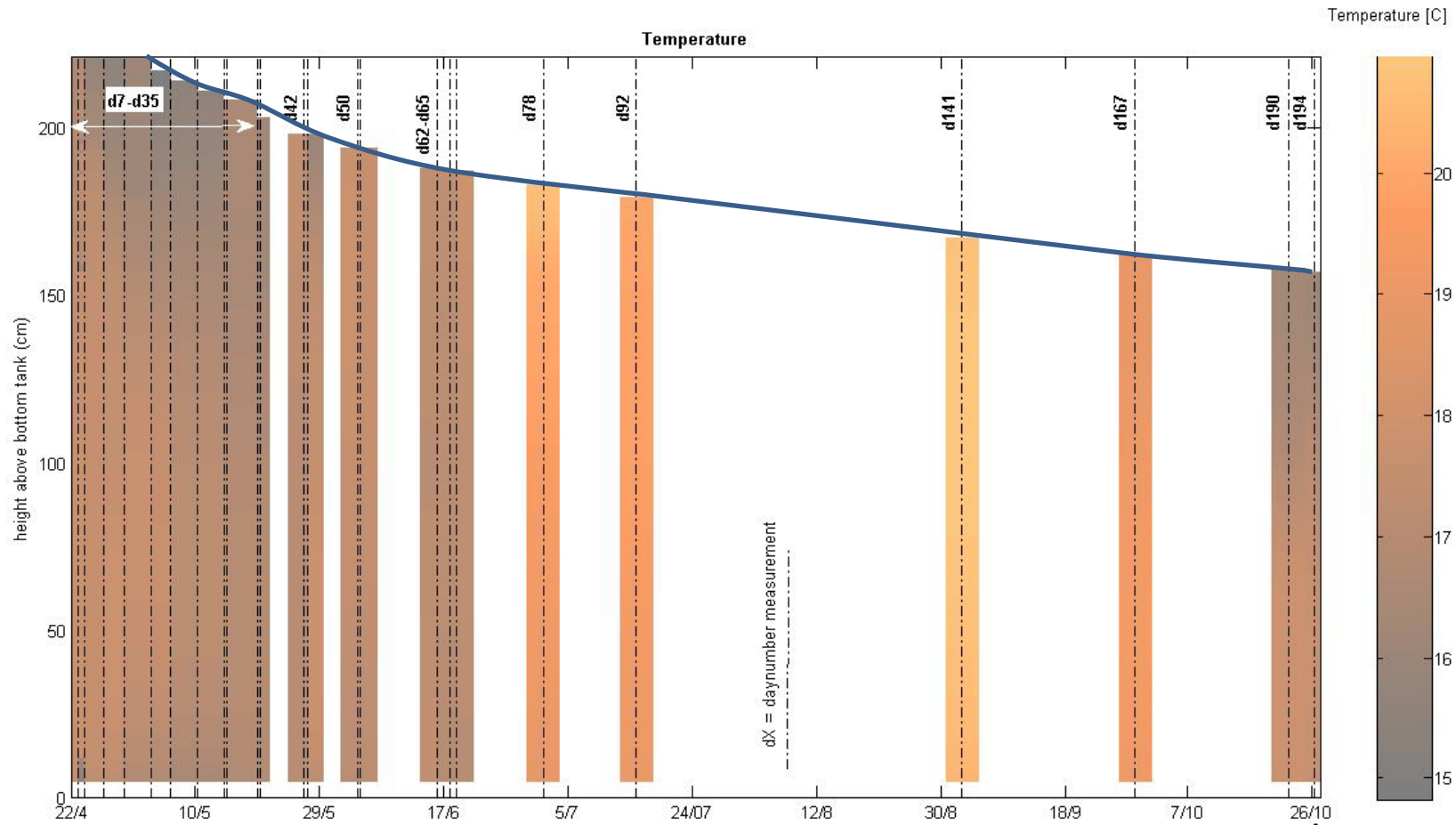
Figure 21: Temperature evolution from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT.
(The blue line is sludge/water interface. Hence above it is 'clear' water and under it the consolidating sludge)
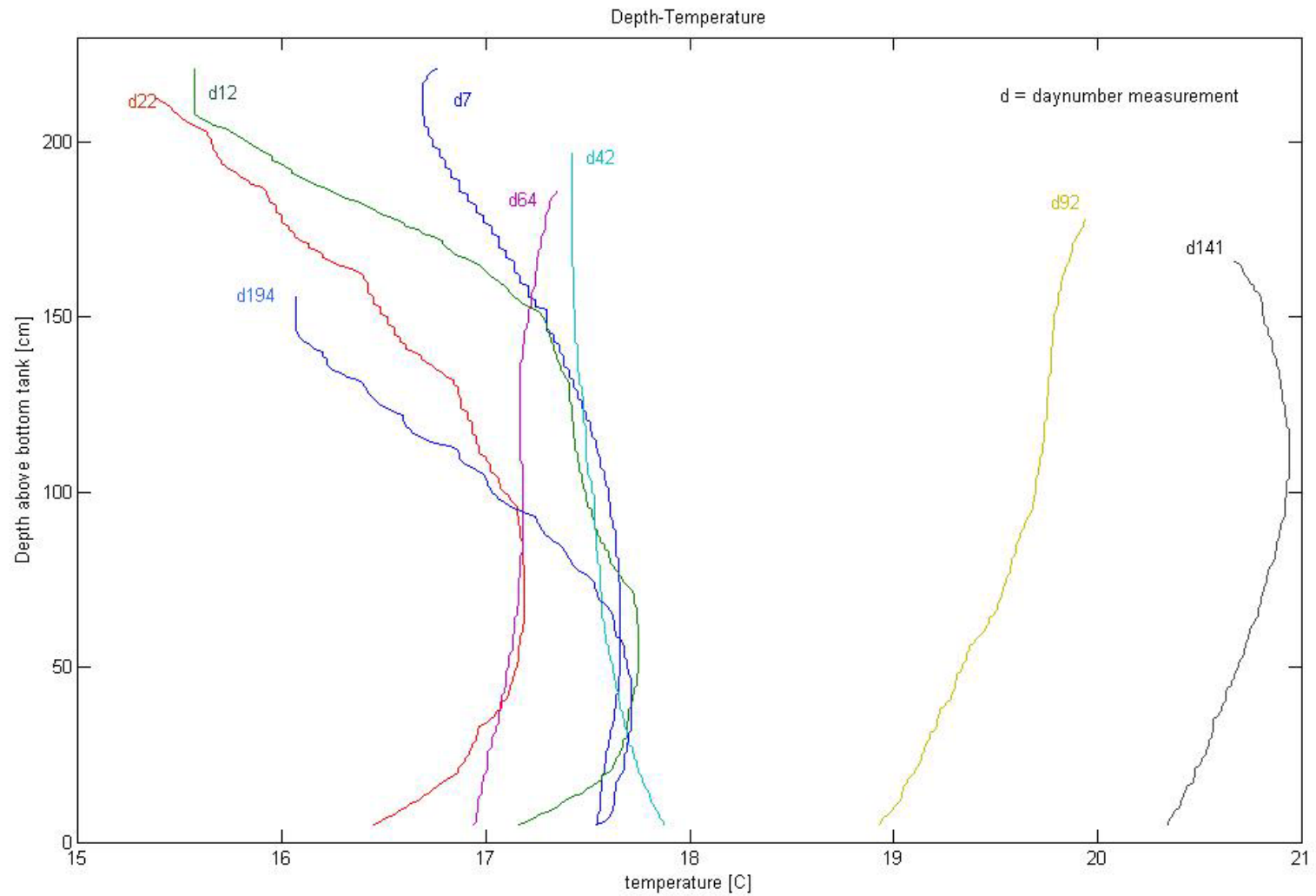
Figure 22: Vertical variation of temperature for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
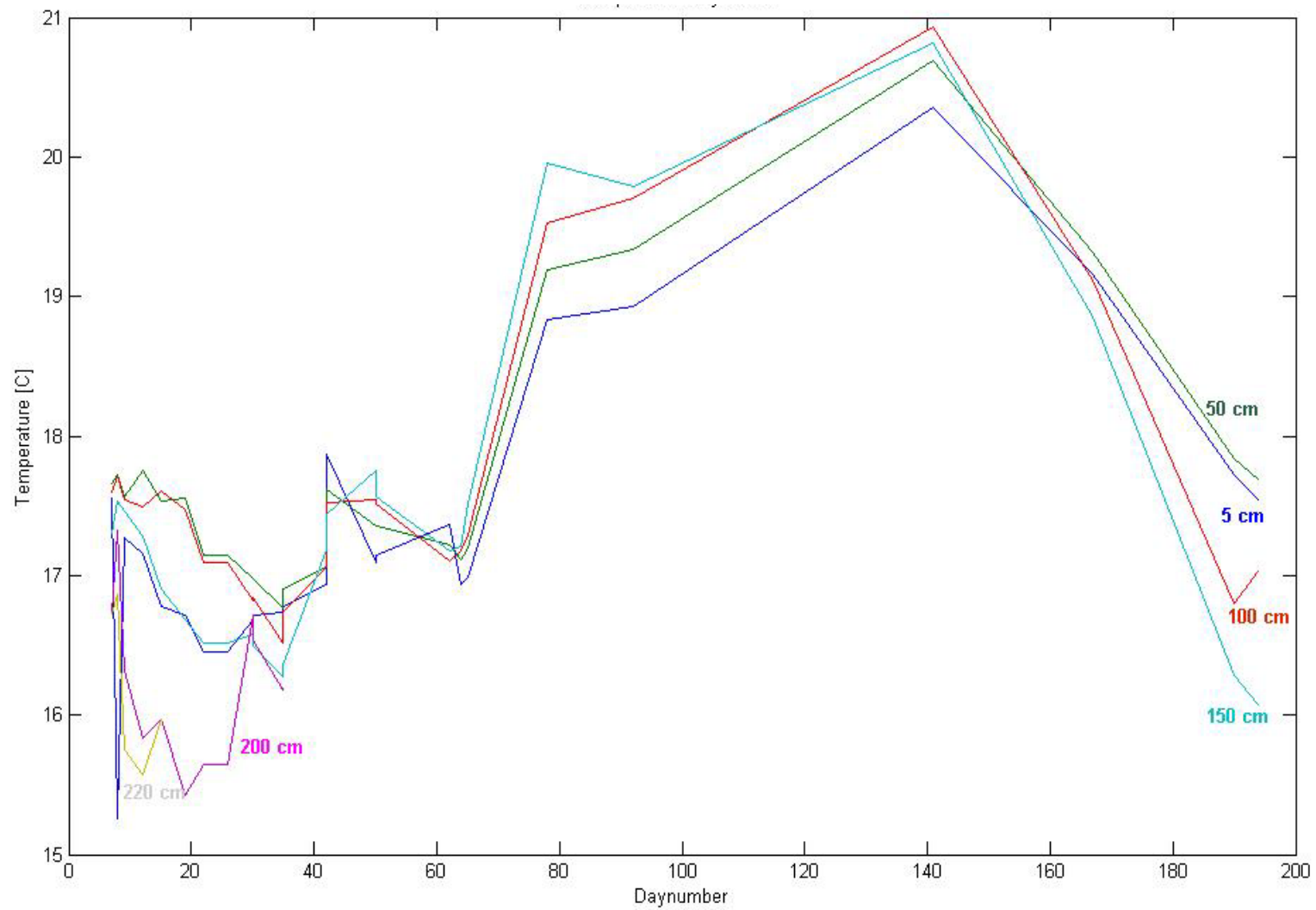
Figure 23: Temperature evolution for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm). Because of the reduction of sediment height, the graphs for the depths of 200 and 220 cm are limited.

4.2.3 pH

pH can have a significant influence on the viscosity of the sediment (De Schepper et al., 1996).Figure 24, Figure 25 and Figure 26 show the pH evolution in the STT from 22/4/2009 (D7) to 26/10/2009 (D194). In Figure 24 all measurements are shown. More details are given for some reference days (Figure 25) and reference depths (Figure 26).

From Figure 24 it can be seen that the pH of the sediment for the measuring period varies between 6.3 and 7.3. The pH decreases in summer season (d78 – d167), and stays low after summer season (d190/d194). The increase in temperature may have triggered biochemical reactions which causes a decrease of the pH. A pH of 7,28 in the pore water of in-situ mud samples from Zeebrugge was measured during the execution of environmental studies under the Mobag 2000 project (September 2002). The pH measured on the mud (CDNB) itself was 7.78.

Figure 25 shows the vertical variation for some reference days. The first weeks (d7, d12, d42) vertical variation in pH is small, except for d22. From that day on, the variation with depth increases with time, to reach a maximum on d194. The general trend is a decrease of pH towards the bottom. This trend is also clearly visible in Figure 24. Figure 26 shows the pH evolution for some reference depths through the tank. The general evolution is the same for all depths. In the beginning of the summer season, the pH suddenly decreases to +/- 6.4 for all depths.

One major influence in the pH evolution is the oxidation level of the sediment. When oxidized, pH can be lowered because of oxidation of sulphides according following equation:

$$H_2S + 4 \cdot H_2O \Leftrightarrow SO_4^{2-} + 10 \cdot H^+ + 8 \cdot e^-$$

Equation 3

According lab analyses in 2009 (Annex 5: lab results), a high concentration of sulphates (11 g/kg DM) and sulphides is present in the sediment, so the reaction stated in *Equation 3* seems to be plausible in the given conditions. But this implies that pH should increase towards to bottom, what is not confirmed by the measurements: except for the first weeks after aeration (d7, d12, d42, see Figure 25), pH decreases towards the bottom. So there are other biochemical reactions causing this decrease towards the bottom, where bicarbonate reactions are supposed to play a large role. This should be further investigated.
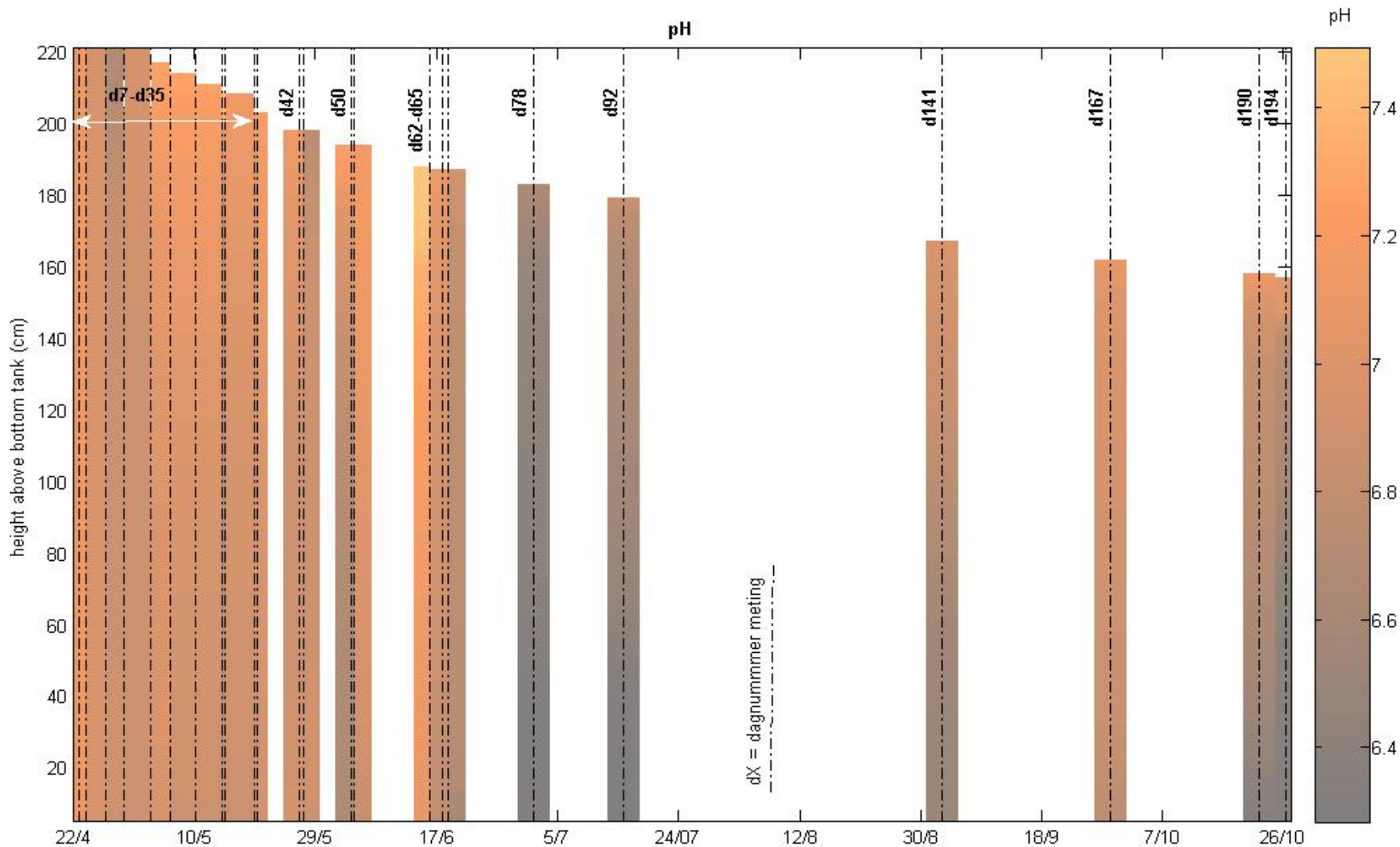
Figure 24: Evolution of pH from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT

Figure 25: Vertical variation of pH for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')

Figure 26: Evolution of pH for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm).
Because of the reduction of sediment height, the graphs for the depths of 200 and 220 cm are limited.

4.2.4 Dissolved oxygen (DO) and oxide-reduction potential (ORP)

The comparative measurements have indicated that the oxide-reduction potential (ORP) measurement in sediment cannot be compared to pore water ORP measurements. It was concluded in § 4.2.1 that the measurements might not be representative for the ORP and DO content in the pore water, but that they can serve as a first indication about the parameter values in the sediment. For that reason, only the overall figure of the measurements will be given in this report of DO (Figure 28) and ORP (Figure 29).

In the first weeks, the DO content is 100% at the top of the sediment till about 30% at the bottom of the tank. At the beginning of the summer season (d62-d65), the DO content ranges from about 50% at the top of the sediment layer till 10 to 20% at the bottom. These values stay the same until the end of the measuring period (D194). The relative high DO content near the bottom of the tank is not as expected, because visually one could see that the bottom sediment was in much reduced condition (deep black sediment). Only at the aeration tubes an oxidation layer of 10-20 cm at the bottom was found. In 2010, the DO content was measured with a larger stabilisation time (120 seconds instead of 5 seconds). The DO content near the bottom was around 2%, what is more as expected. An YSI-profile measured in 2010 with larger stabilisation time can be seen in Figure 20. This illustrates the possible errors of DO and ORP readings due to the lack of sufficient stabilisation time.

For some days (d50, d65) the DO-value stays around 20% for the whole sediment column, which is not as expected. Because of the difference with the other profiles – i.e. decrease from +/- 50% at the top till +- 15% at the bottom of the tank – these values might be erroneous and caused by blocking of the sensor with sediment.

The ORP values are given in Figure 29. At initialisation, the ORP values are at least 300 mV throughout the whole sediment column. After a month, the ORP values of the lowest half part of the tank change quickly: first a drop to 150 mV at the bottom of the tank (d22-26), soon after a drop to -50 mV to -100 mV. From day 42 to day 167, an ORP of -50 to -100 mV is most of the time measured for the lowest half part of the tank, except some (suspicious) measurements which are giving very high values (> 300 mV).

For the upper half part of the tank, ORP values are in general higher than in the lowest half part of the tank. The values mostly range between 150 and 300 mV. Exceptions are (suspicious) values of -50 to +50 mV for the upper part of the sediment column. This lower values for reduced conditions compared with higher values for more oxidised conditions are in accordance with the redox reaction of Equation 1, also shown in Figure 27. The reduced side in *Equation 3* corresponds with an ORP-value of -200 mV, which is in the range of the ORP-value measured most of the time in the lowest (most reduced) half part of the tank.

Day 167 and day 194, an almost constant ORP-value is measured along the whole depth. Day 167 the ORP was around 70 mV, day 190 the ORP was around 300 mV. These constant ORP-values for the whole sediment column are not logical, and perhaps caused by blocking of the sensor with sediment from the upper layer.

The correlation between DO and ORP content is further investigated in Sub report 11: Statistical Analysis.
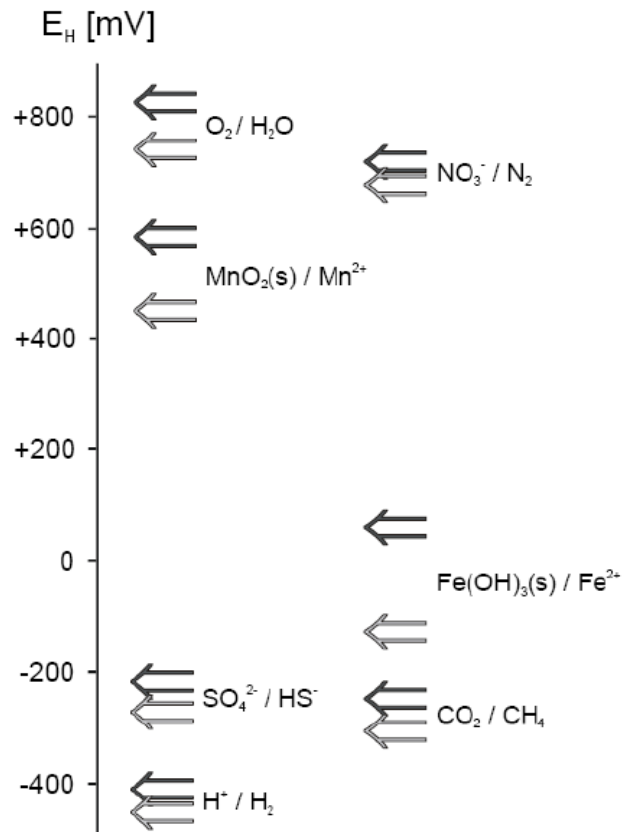
Figure 27: Redox potential for some redox reactions in natural water
(pH=7 (black arrows) and pH = 8 (light arrows))
(source: CMA procedure ORP measurements, VITO).
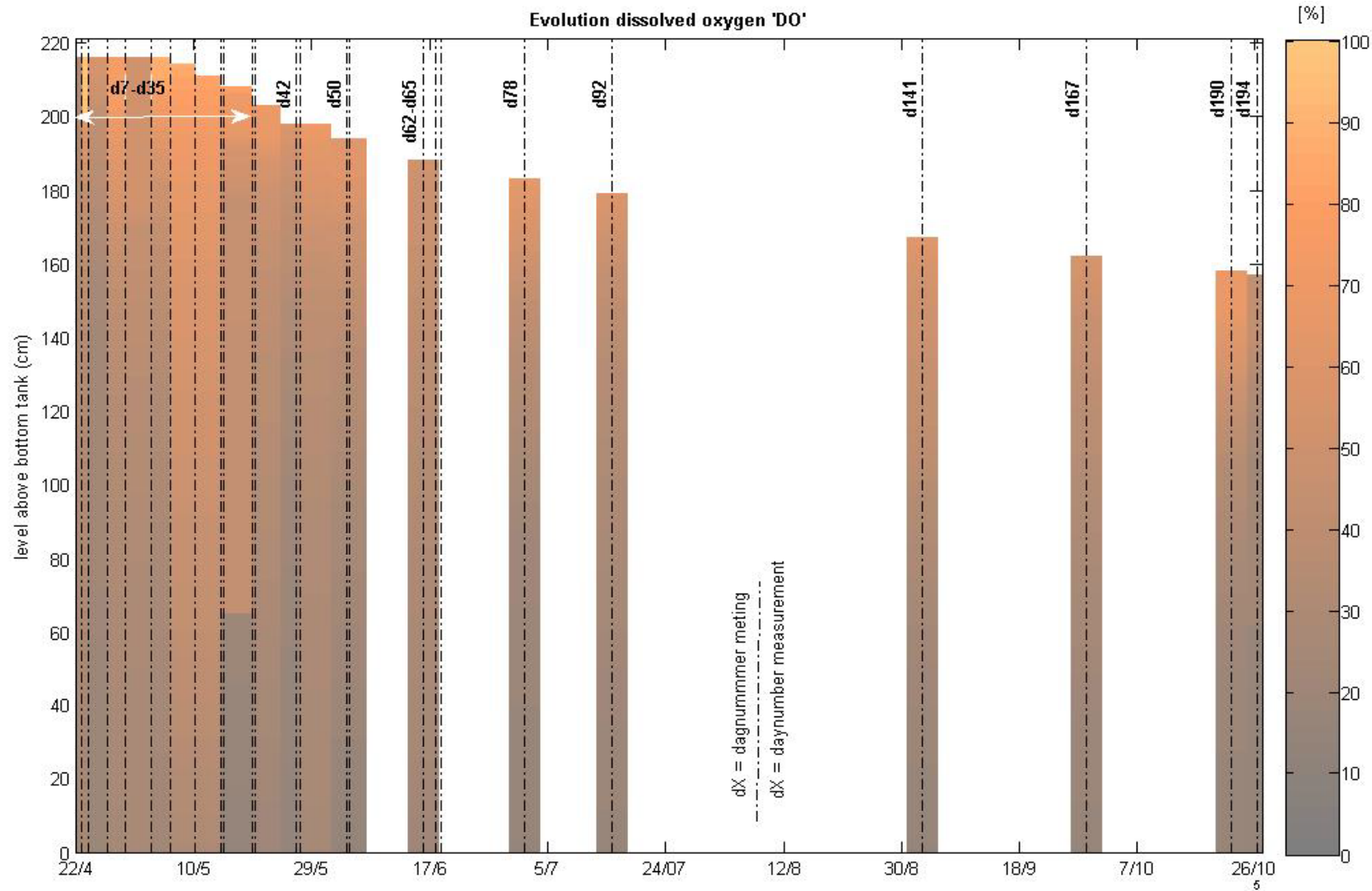
Figure 28: Evolution of dissolved oxygen from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT
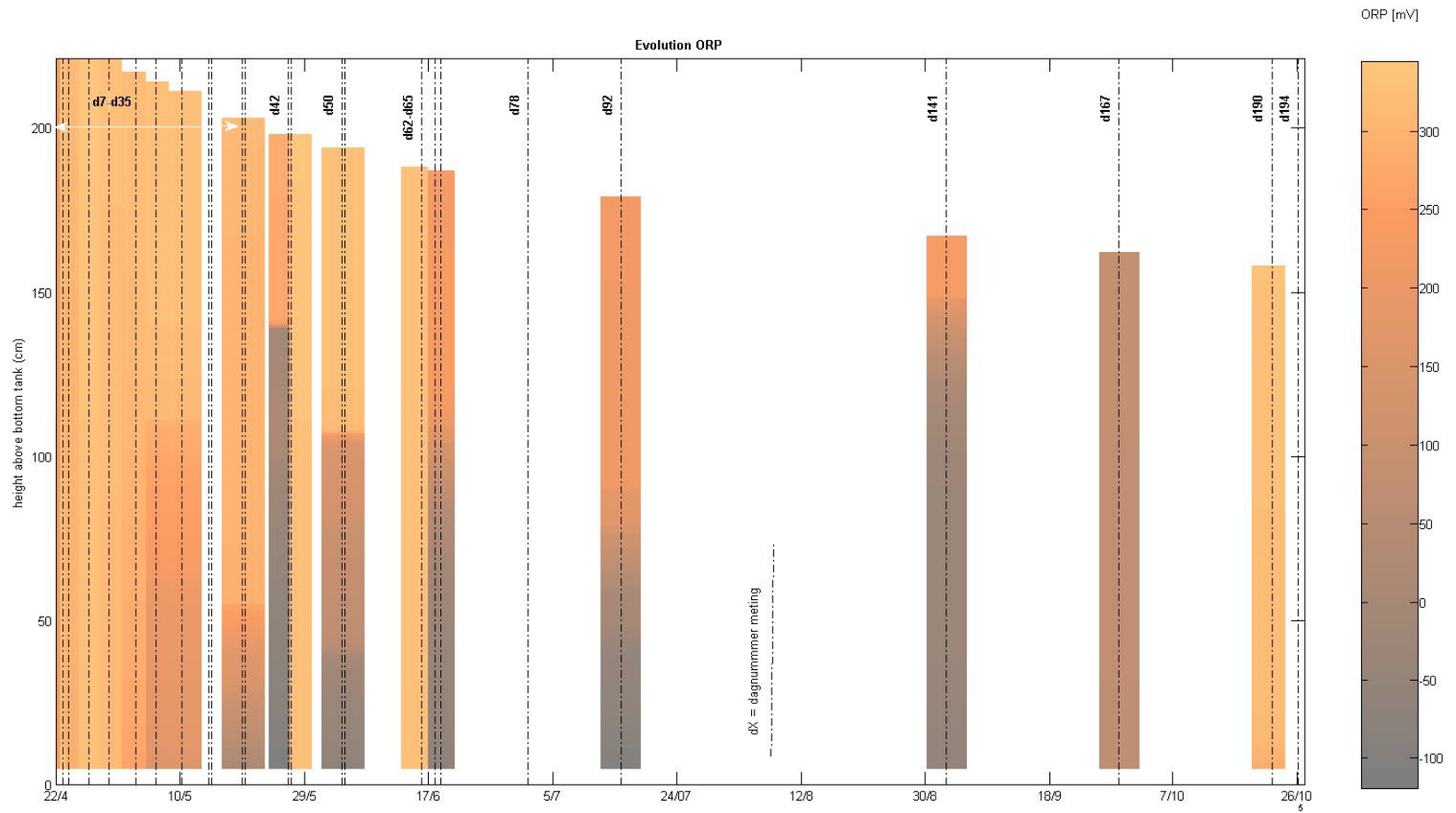
Figure 29: Evolution of ORP from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT

4.2.5   (Specific) conductivity

The presence of salts has an influence on the consolidation behaviour of the sediment. Salt ions in the pore water speed up coagulation and can enhance consolidation processes (De Schepper et al., 1996). With conductivity measurements, the influence of the salts on consolidation can be verified.

Only the evolution of the 'specific conductance' or specific conductivity, which is the conductivity corrected to a reference temperature of 25° C, will be reported.

In Figure 30, Figure 31 and Figure 32 the (specific) the conductivity evolution is shown from 22/4/2009 (D7) until 3/9/2009 (D141). The quality of the measurements between 29/9/2009 (D167) and 26/10/2009 (D194) is questionable, since the values are unrealistically low (D167) or high (D190, D194). These unrealistic values are probably due to the obstruction of the vent hole of the conductivity sensor. As a result, the data between 29/9/2009 (D167) and 26/10/2009 (D194) is not presented here. In Figure 30 all measurements are shown. More details are given for some reference days (Figure 31) and reference depths (Figure 32).

From Figure 30 it can be seen that the specific conductivity of the sediment for the measuring period varies between 25 and 40 mS/cm. The specific conductivity values stay more or less the same during the first months for the whole vertical column. When temperature increases during spring (d78 –d92), specific conductivity decreases. At the end of the summer season, specific conductivity decreases to 25 mS/cm, for the whole sediment column. A possible explanation for the decrease in specific conductivity is the influence of temperature on density (§ 4.3). Because the electrodes of the conductivity sensor cannot be inserted properly in the thick sediment (Figure 18), the conductivity of the pore water is not measured accurately. So until d65 the conductivity of the pore water could be measured by the sensor, but from d78 on, the pore water of the sediment cannot entirely immerse the conductivity electrodes any more. The comparative measurements have shown that for densities higher than 1.2 g/cm³, the conductivity of the pore water is systematically underestimated.

A more plausible explanation is that there is simply not enough pore water left in the consolidated sediment to immerse the conductivity sensor completely. This also results in an underestimation of the pore water conductivity.

Figure 31 shows the vertical variation for some reference days. The first weeks (d7, d12, d22, d42) vertical variation in conductivity is small. From d64, there is an important vertical variation in specific conductivity, with a decrease towards the bottom. Again the increase in density towards the bottom might have biased the (specific) conductivity measurements towards the bottom. For day 141, the higher densities are explaining the (erroneous) low conductivity values. From Figure 19, it can be seen that underestimation of pore water conductivity is constant for densities higher than 1.2 g/cm³. This explains the small variation in conductivity values for the lowest 100 cm for d141 (Figure 19: densities higher than 1.2 g/cm³).

Figure 32 shows the conductivity evolution for some reference depths through the tank. As long as the densities stay above 1.2 g/cm³, values are all +-35 mS/cm. From d64, densities start to reach 1.2 g/cm³, first 5 cm above bottom, later on towards the top (d92, d141). This correlates well with a drop in the specific conductivity values for the different reference depths.
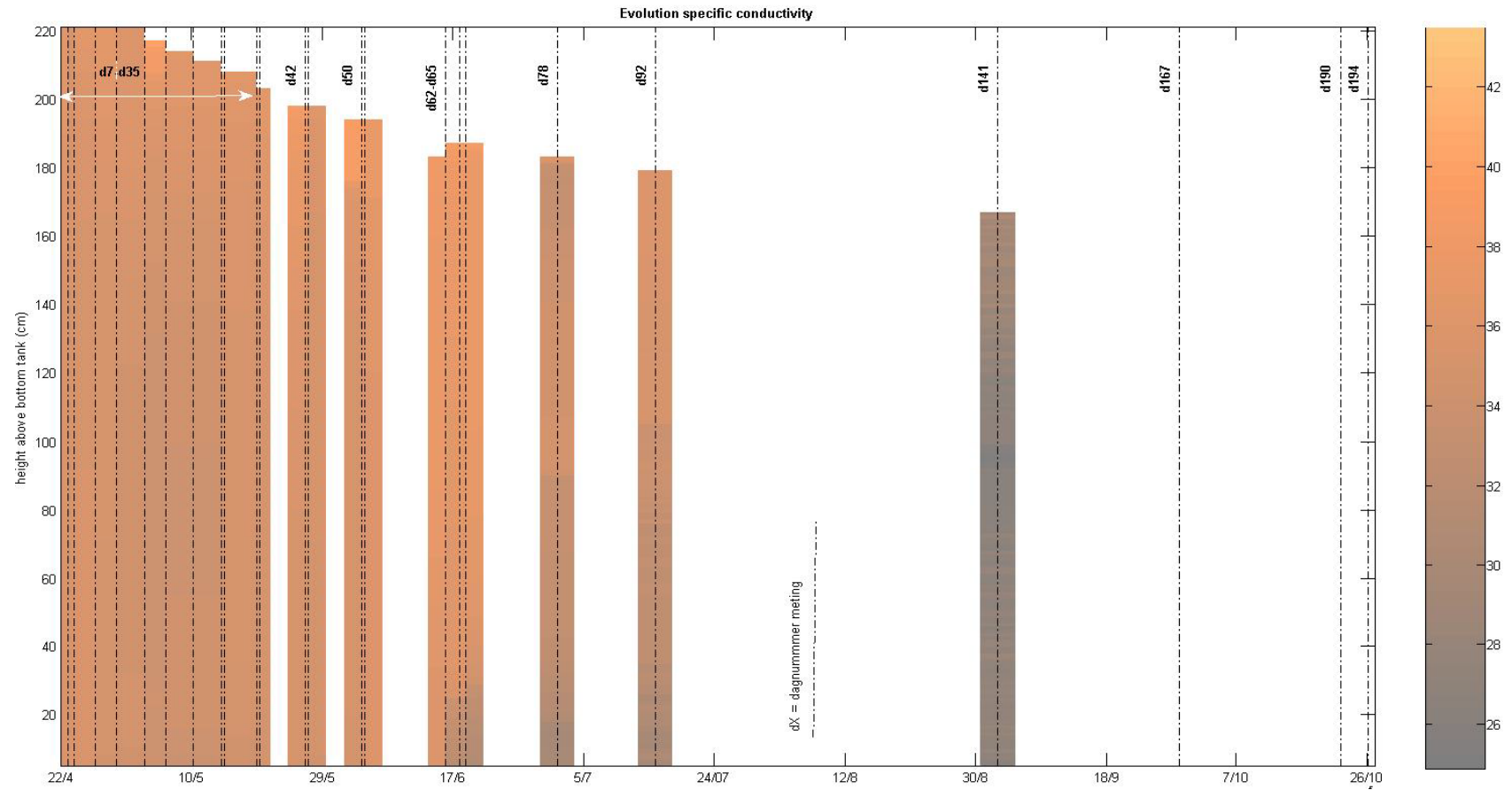
Figure 30: Evolution of specific conductivity from 22/4/2009 (D7) until 26/10/2009 (D194) in the STT
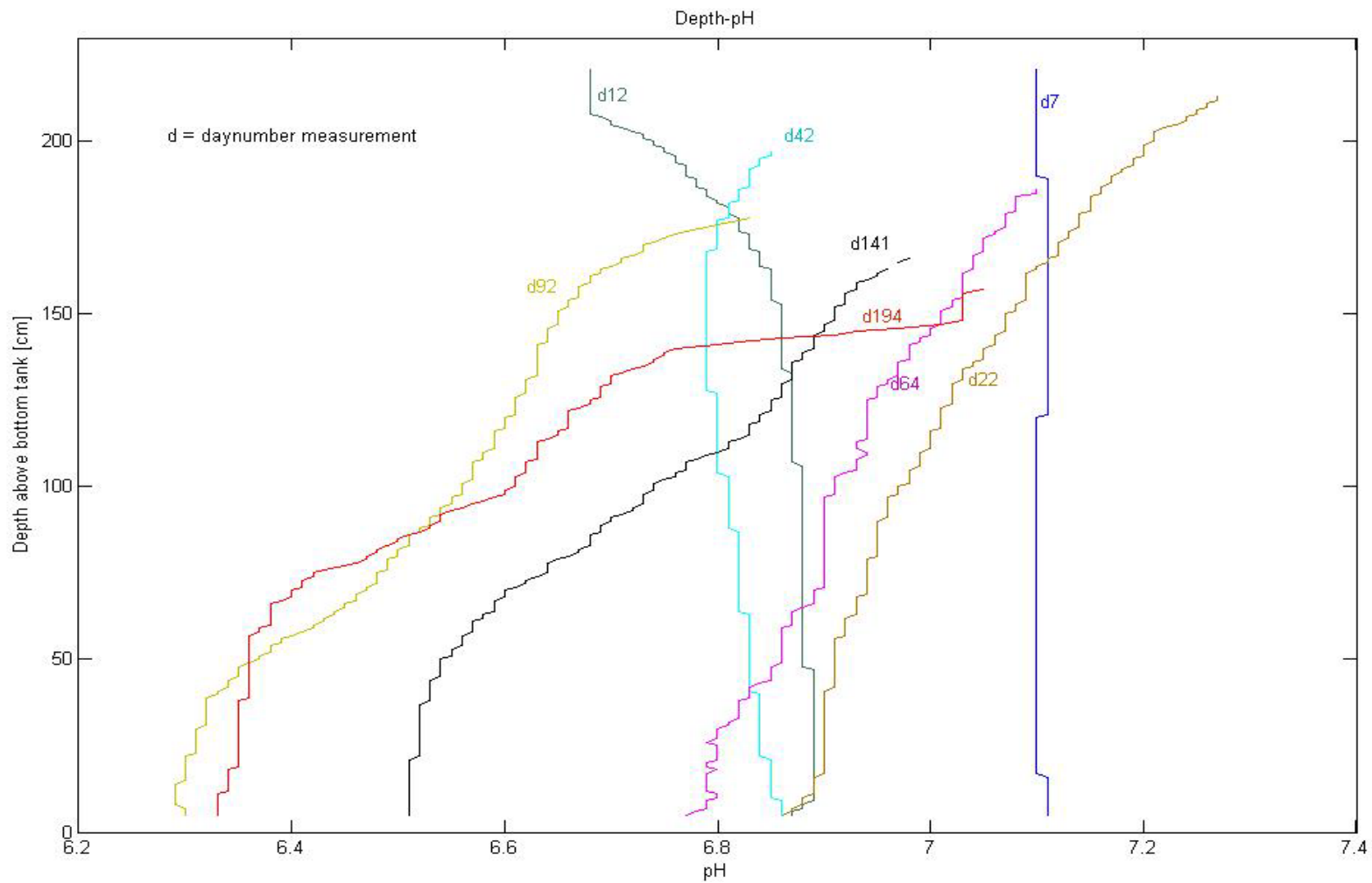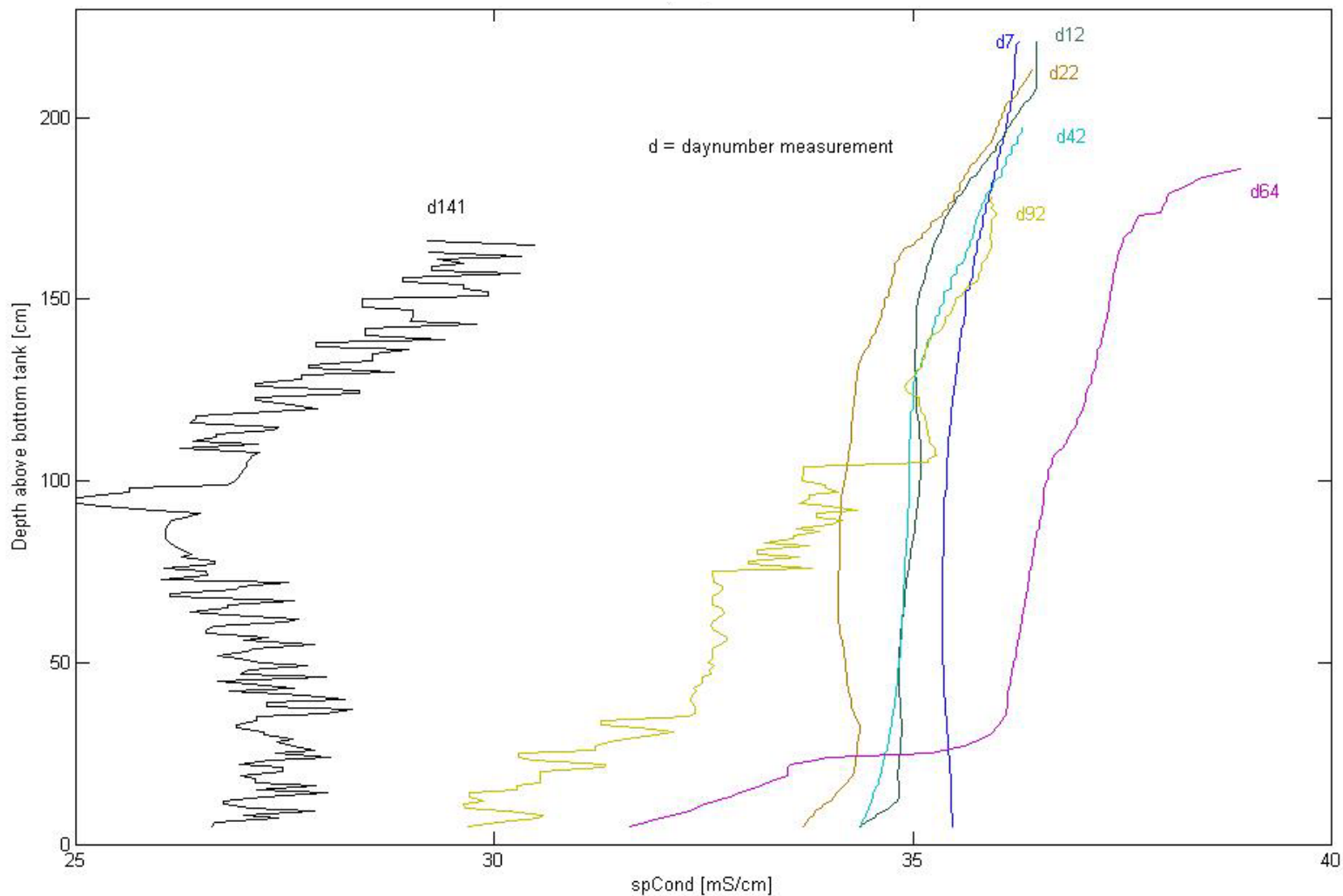
Figure 31: Vertical variation of specific conductivity for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
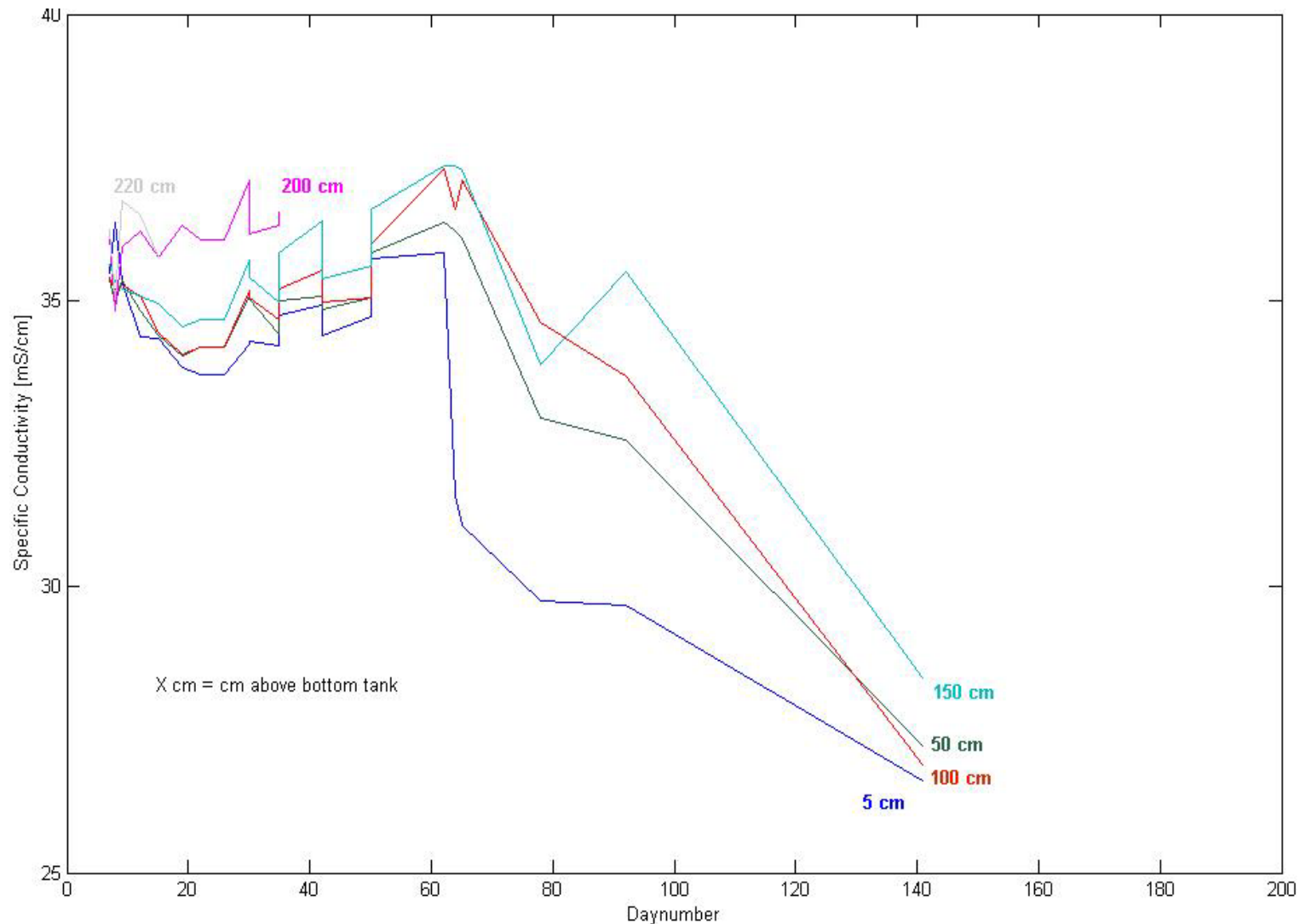
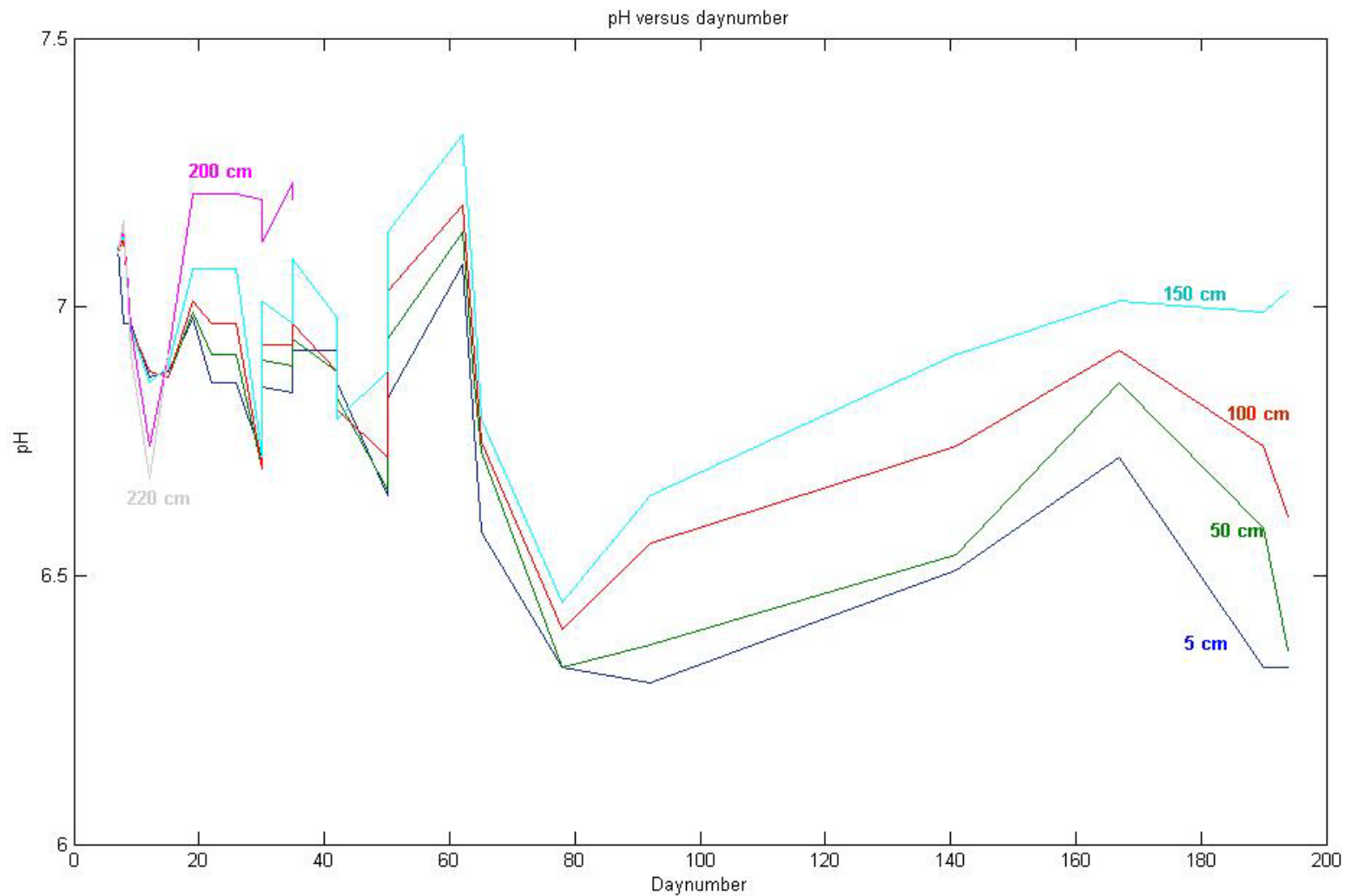Figure 32: Evolution of specific conductivity for some reference depths in the STT
Numbers refer to the depth above the bottom of the tank (cm).
Because of the reduction of sediment height, the graphs for the depths of 200 and 220 cm are limited.

4.3 Density

Figure 33, Figure 34 and Figure 35 show the density evolution from 22/4/2009 (D7) until 19/02/2010 (D310) in the STT. In Figure 33 all measurements are shown. More details are given for some reference days (Figure 34) and reference depths (Figure 35). For some days, more than 1 sample was taken and analysed for density values by the lab. These different density values were averaged per day, for each depth.

As mentioned above, the accuracy of the density measurement is limited to +/- 0.02 g/cm³ and samples are taken every 5 cm (vertical resolution). Some spikes with deviations larger than 0.02 g/cm³ are observed for the higher densities. The difference between measurements of undisturbed and disturbed samples was analysed. Mixing the samples smoothens the vertical density trend in the mud column.

Large fluctuations in the vertical trend can be observed for d230 and d141. Presumable this is caused by the manipulation of the laboratory personnel/researcher. One must take precautions when filling the tube of the DMA-density instrument, preventing inclosing air bubbles and also one needs to wait for the measurements to stabilise.

From Figure 33 it can be seen that the density of the sediment for the measuring period varies between 1.1 and 1.35. The first weeks, density ranges from 1.1 at the top of the sediment to less than 1.2 g/cm³ at the bottom of the tank. After a month (d42), the density level of 1.15 g/cm³ is still present at the top, while the bottom sediment layers are reaching the 1.2 g/cm³ density. This 1.2 g/cm³ density limit stays constant for about 1 month. From d78, when sediment temperature starts to increase (Figure 21), the maximum sediment level that reaches the 1.2 g/cm³ increases at a constant speed, until at the end of the summer season (d154) the complete lower half of the tank has the 1.2 g/cm³ density. The level of 1.15 g/cm³ has disappeared. From d154 to d230 density passed the 1.2 g/cm³ level, but with more fluctuations. One must be careful to interpret these fluctuations as they may be generated by the limitations/accuracy of the measurement techniques, especially for high density samples. We can also observe lower densities below the 1.2 g/cm³ top layer. The creation of gas bubbles could cause these anomalies. .

The positive correlation between consolidation speed and temperature is well known (De Schepper et al., 1996). In fact, it is the viscosity of the pore water that shows an inverse correlation with temperature. A lower pore water viscosity increases the pore water drainage from the sediment, which causes faster consolidation. On the other hand, higher temperature increases the decomposition of organic matter into gasses, which can block the soil pores and cause a decrease in permeability and consolidation speed. In the STT, the effect of higher viscosity is clearly more important than the effect of organic matter decomposition.

At d230, the 1.2 g/cm³ density limit reaches the layer at 10 cm under the sediment top layer. From then on, the 1.2 g/cm³ density limit decreases slowly together with the top sediment level in the tank. At d310, the upper 10 cm of the sediment comes close to reaching the 1.2 g/cm³ density level. The deviation for the upper 10 cm of the sediment column might also be causes by manipulation during subsampling (dilution with water layer above sediment).

At d310, the density at the bottom of the tank increases to 1.35 g/cm³. Until 0.5 m above bottom level, density reaches 1.3 g/cm³. Until 1 m above bottom level, density reaches 1.25 g/cm³.

Figure 34 shows the vertical variation for some reference days. The fluctuations that can be seen (+- 0.015 g/cm³), especially for d141 and d230, are mainly because of the limited accuracy of the instrument (+- 0.02), and possibly small gas bubbles in the DMA38 instrument. The first two weeks (d7-d12), density variations around 1.15 g/cm³ across the whole sediment layer. From d22 to d64, the lowest sediment layers are near or just above the 1.2 g/cm³ limit. The density profiles for d92, d141 and d194 are shifting to higher values. This shift is more important for the lower half sediment layer than for the upper half of the tank. Because of this vertical differentiation, an exponential graph is obtained. In Figure 34, the value of 1.35 g/cm³ seems an outlier, but this high density value is confirmed by other measurements.

Figure 35 depicts the density evolution for some reference depths trough the tank (30/50/100/150/200/220 cm above bottom). From this figure it is clear that the 1.15 g/cm³ density limit is reached after a couple of weeks in the lowest half part of the tank. In the upper half of the tank, density

at 150 cm above the bottom seems to be a bit larger than the density at 100 cm above the bottom, but the difference remains small. After 2 months, the whole sediment layer has a density above 1.15 g/cm³. As already could be derived from Figure 33, there is a steep increase in density from d62 to d154 probably due to higher sediment temperature.

From Figure 34, it can be seen that the consolidation speeds of the sediment layers differs significantly. The first 100 days, the consolidation speed at 30 cm and 50 cm above the bottom is much faster than the upper sediment layers (100cm and 150 cm above bottom tank). As a consequence, the density curve has a rather exponential course from top to bottom (Figure 34) until d141. Around day 150, after summer, consolidation speed of the bottom layers is decreasing. In contrast with the bottom layers, the consolidation speed for the top layer (in particular 100 cm above bottom) is increasing. As a consequence, the density curve has again a more linear course from top to bottom from d194 until d310 (Figure 34).
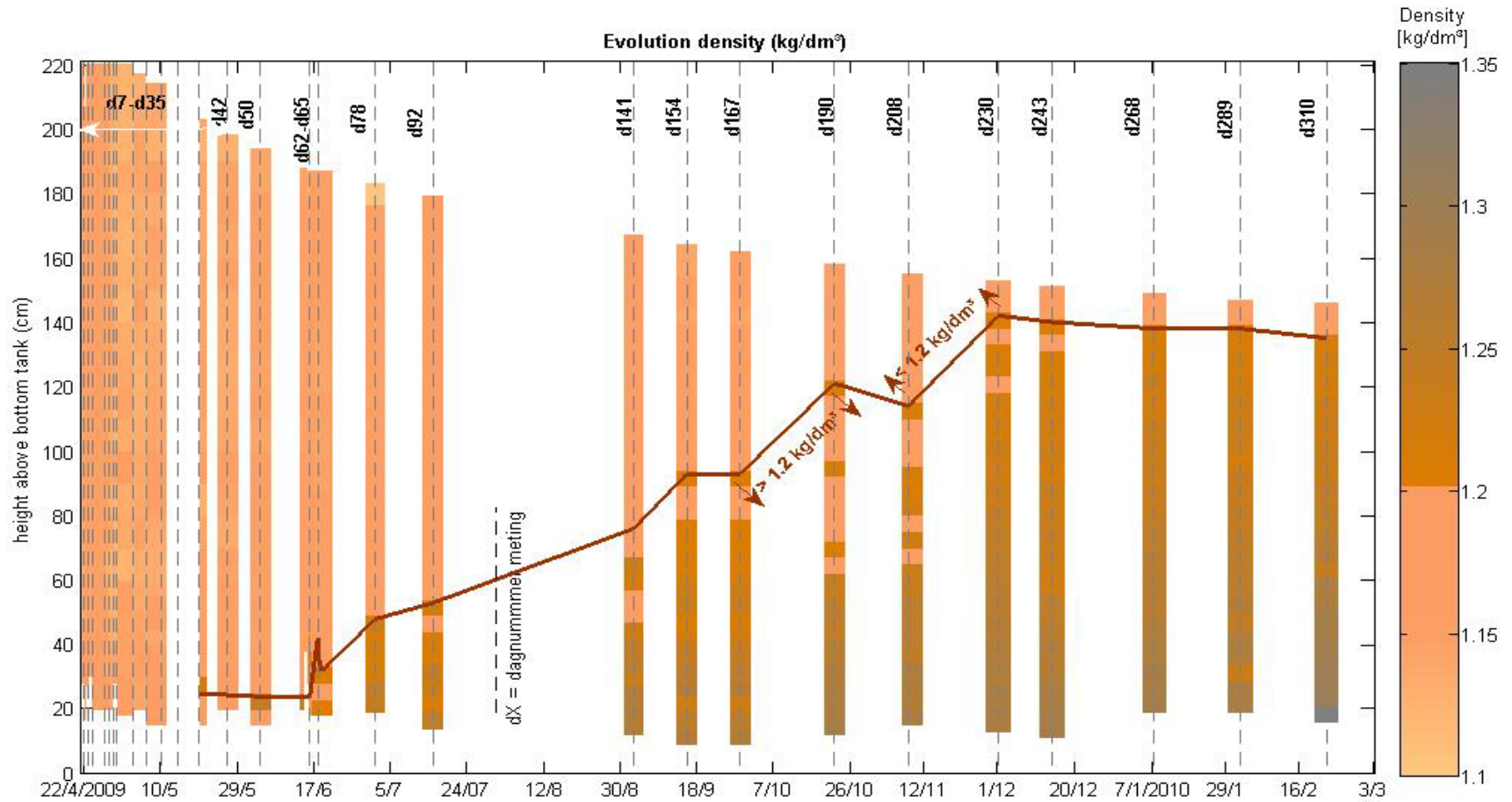
Figure 33: Evolution of density from 22/4/2009 (D7) until 19/02/2010 (D310) in the STT. The top density level of 1.2 g/cm³ is illustrated by the thick brown line

Figure 34: Vertical variation of density for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
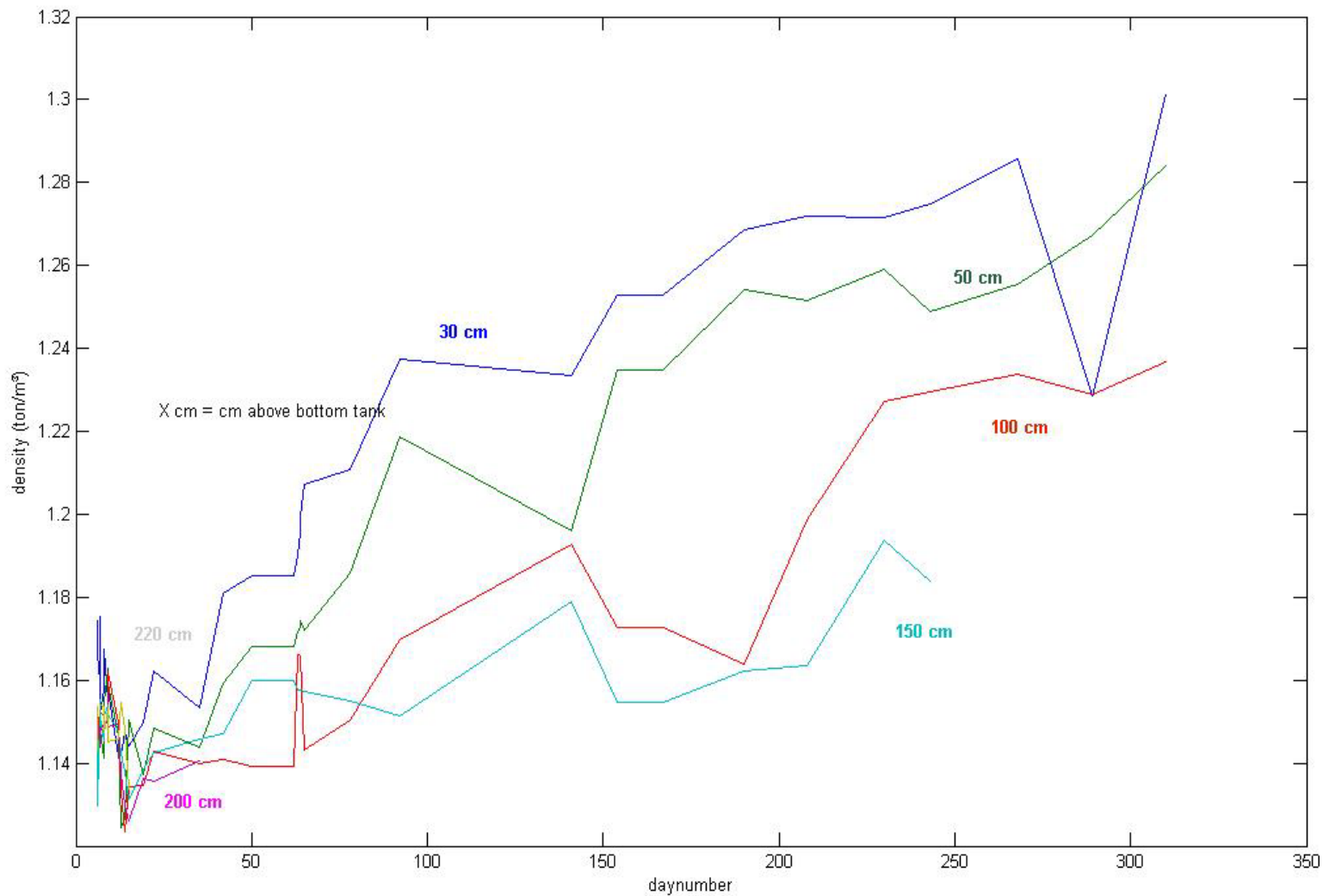
Figure 35: Evolution of density for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm). Because of the reduction of sediment height, the graph for the depths of 200 and 220 cm are limited

4.4 Rheological parameters

Every subsample of the undisturbed column taken with the Beeker-sampler was analysed with the Anton Paar rheometer 'Physica MCR 301' (§ 3.4). The rheometer is set-up to register the shear stress needed to generate a range of shear rates (0.09999; 0.2031; 0.4124; 0.8376; 1.701; 3.455; 7.017; 14.25; 28.94; 58.78; 119.4; 242.5; 492.4; 1000 1/s). The rheograms for the different shear rates are shown in [Annex 2: rheogram].

From the rheograms presented in [Annex 2: rheogram], it can be seen that the trends are similar for the different applied shear rates. At a certain density level (often at 1.25 g/cm³, depending on the composition of the sample), the rheological behaviour changes. For the low applied shear rates (smaller than 30 1/s) this level corresponds often with a ramp in the shear stress course when going from top to bottom.

Once the shear rate is greater than 30 1/s, graphs are shifting in parallel towards higher shear stresses without much further vertical differentiation due to the equilibrium increase in shear rate vs. shear stresses (see Sub report 2, figures 30 and 31 on page 23). In this stage, viscosity is not changing any more. This yields in a typical profile of yields stress over depth and is function of the sediment composition.

In Figure 36, the shear stresses for a shear rate of 30 1/s are shown for some reference days through depth. Trends which can be seen from Figure 36 and which are valid for all rheograms are:

- There is little vertical variation in shear stresses the first weeks

- After 3 weeks (d22), the shear stress (at a shear rate of 30 1/s) at the bottom starts to increase. After 3 months (d92), the bottom shear stress increased with a factor 6 (100 till 600 Pa), while the shear stress at the upper half of the sediment column only doubled.

- From d92 till d190, the (more or less) differentiation continues, but less pronounced. There is more a parallel shift to the higher stresses for the whole graph, but for the lower layers the increase in shear stresses is still somewhat higher

- After d190, the rheogram becomes more linear, having almost an linear course at d310

Both density (Figure 37) and shear stresses at applied shear rates (Figure 36) increase rather exponentially towards the bottom. For the shear stress however, the exponential increase towards the bottom is more pronounced than for density, in particular between d22-d190. An in depth correlation research between density and the rheology parameter is carried out in the first two sub reports as well as sub report 11.

Finally, the 'outlier' in density of 1.35 g/cm³ (d310 at the bottom of the tank) (Figure 34) is also confirmed by a high shear stress value for the same sample.

Figure 36: Evolution of rheology for some reference days in the STT. Shear stresses are shown for a shear rate of 30 1/s at different levels in the STT. Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')

4.5 Pore pressures

The evolution of pore water pressures is indicative for the consolidation processes in the sediment test tank. The pore water pressures are monitored by the installation of piezometric filters, installed at 0, 20, 40, 80, 100 and 120 cm above the bottom of the tank. Total water pressure is monitored by the direct readings of the water height in the riser tubes connected to the piezometric filters. Pore overpressure is calculated by subtracting the hydrostatic pressure in the tank from the filter readings.

4.5.1   Total pore water pressure

In Figure 37 and Figure 38 the total pore water evolution is shown from 22/4/2009 (D7) until 25/03/2010 (D344) in the STT. In Figure 37 the total pore water measurements are shown together with water and sediment levels. The first days the pore water levels are stabilising, and then they are decreasing in time. This decrease is only partially due to a decrease in water level. More important is the decrease in pore overpressure (§4.5.2). Vertically, the total water pressure decreases from the bottom to the top of the sediment column. There are some important variations in pore water evolution through time for some depths (0, 40, 80, 120 cm), particularly from d200 to d300. These variations are probably due to filter clogging. Around d230, filters were cleaned.

In Figure 38 the total water pressure evolution is given for some reference days. The same evolutions as seen in Figure 37 can be derived from Figure 38. At d8, all pore water levels have to stabilise. At d42 only the pore pressures at the bottom levels has not yet stabilised. After the stabilisation time, there is a temporal decrease of total water pressure and a vertical increase of total water pressure towards the bottom of the tank.

4.5.2   Pore overpressure

When sediment consolidates, the weight of the overlaying sediment and water column causes a pore overpressure in the underlying sediment layers. Because of the induced pore pressure gradient, soil water fluxes through the pores according to the permeability equation (Darcy's law). Because of the removal of soil water in the pores, the overpressure is converted into an increase of the effective stress of the soil grains. This increase in effective stress causes a re-arrangement of the soil grains (law of Terzaghi), which is the primary consolidation. After this primary setting, a secondary much slower consolidation can be caused by creep effects, viscous behaviour of the clay-water system and compression of organic matter. In this report, only the primary consolidation processes are discussed. The secondary consolidation can last for many years, especially for saturated clays with low hydraulic conductivity.

In Figure 39, Figure 40 and Figure 41, pore overpressure is illustrated for the different levels were piezometric filters were installed. The overpressure is calculated as:

$$Pore\ overpressure = total\ water\ pressure - hydrostatic\ pressure$$

The total water pressure is monitored from the piezometric filter readings (§4.5.1). The hydrostatic pressure is the height of the water and the sediment column above the filter.

From Figure 39, Figure 40 and Figure 41 the same temporal and vertical gradients can be seen for the pore overpressure as for the total pore water pressure. The pore overpressures decrease with time, and are higher at the bottom of the sediment column than at the top. The negative overpressure as seen in Figure 40 around d200 to d300 is theoretically not possible. They are probably due to clogging of the filters. For some levels, these disturbances around d200 to d300 give deviations from the global trend until the end of the monitoring (d344). For instance, the pore overpressure at the bottom of the sediment column ('0 cm') drops after d250 to the lowest overpressure levels at d350, which is in contradiction with the general trend of the other levels. Also the evaporation of the water in the riser tubes, over this long period, could play a role.

The decreasing temporal evolution in pore overpressure confirms former consolidation studies with similar sediment (De Schepper et al., 1996). At the end of the monitoring period, a small increase in overpressure can be observed for most levels from d300. However, this short increase will be compensated soon after, so that pore overpressure levels fluctuate around the same values of 5 to 10 cm until the end of the testing period (+- d400).

An estimate of the effective stresses and excess pore pressure is made in sub report 12.
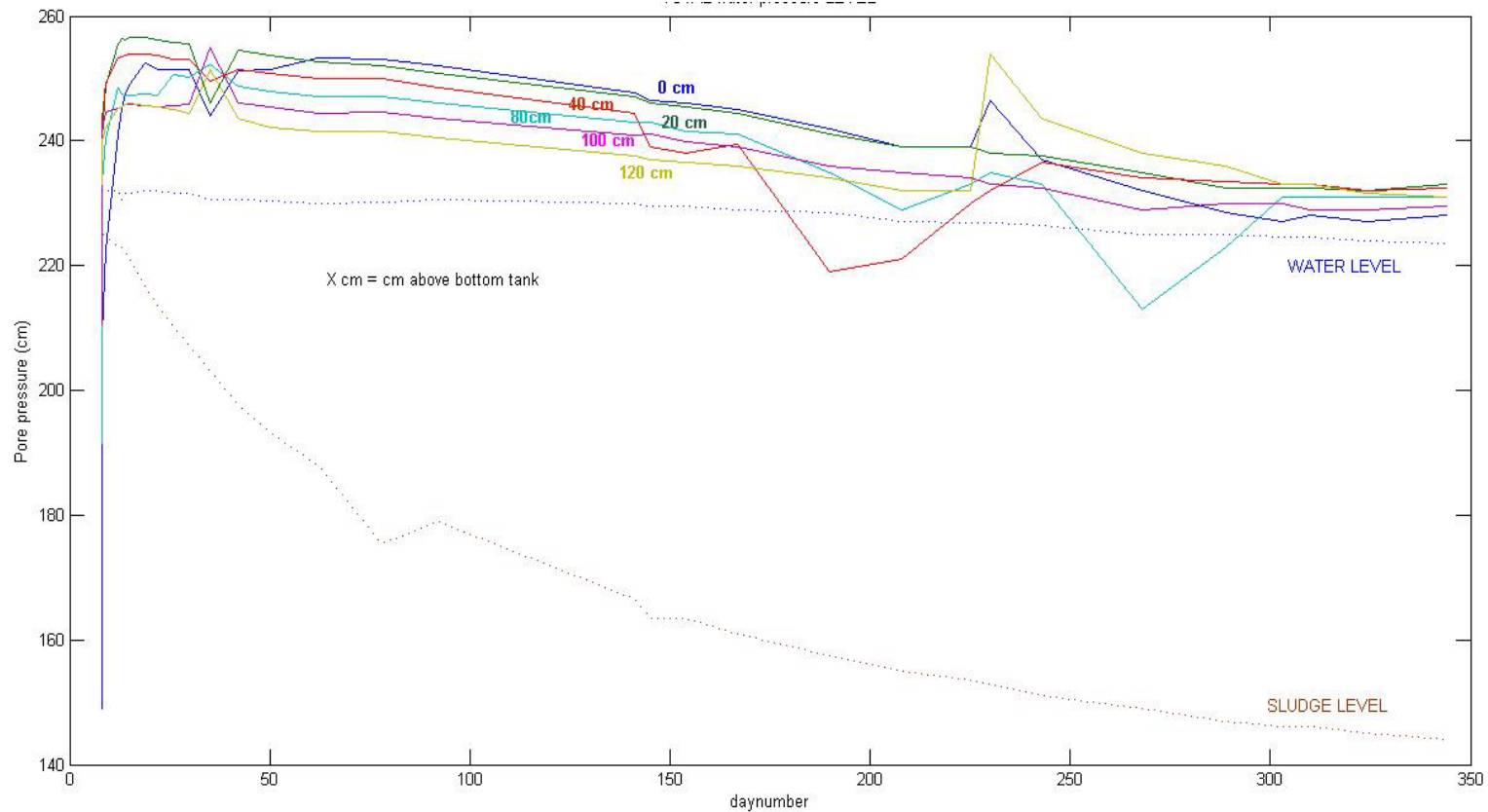
Figure 37: Evolution of total water pressure for the measured pore water levels in the STT from 22/4/2009 (D7) until 25/03/2010 (D344). The numbers 0, 20, 40, 80, 100 and 120 cm refer to the depth of the piezometric filters above the bottom of the tank (cm)
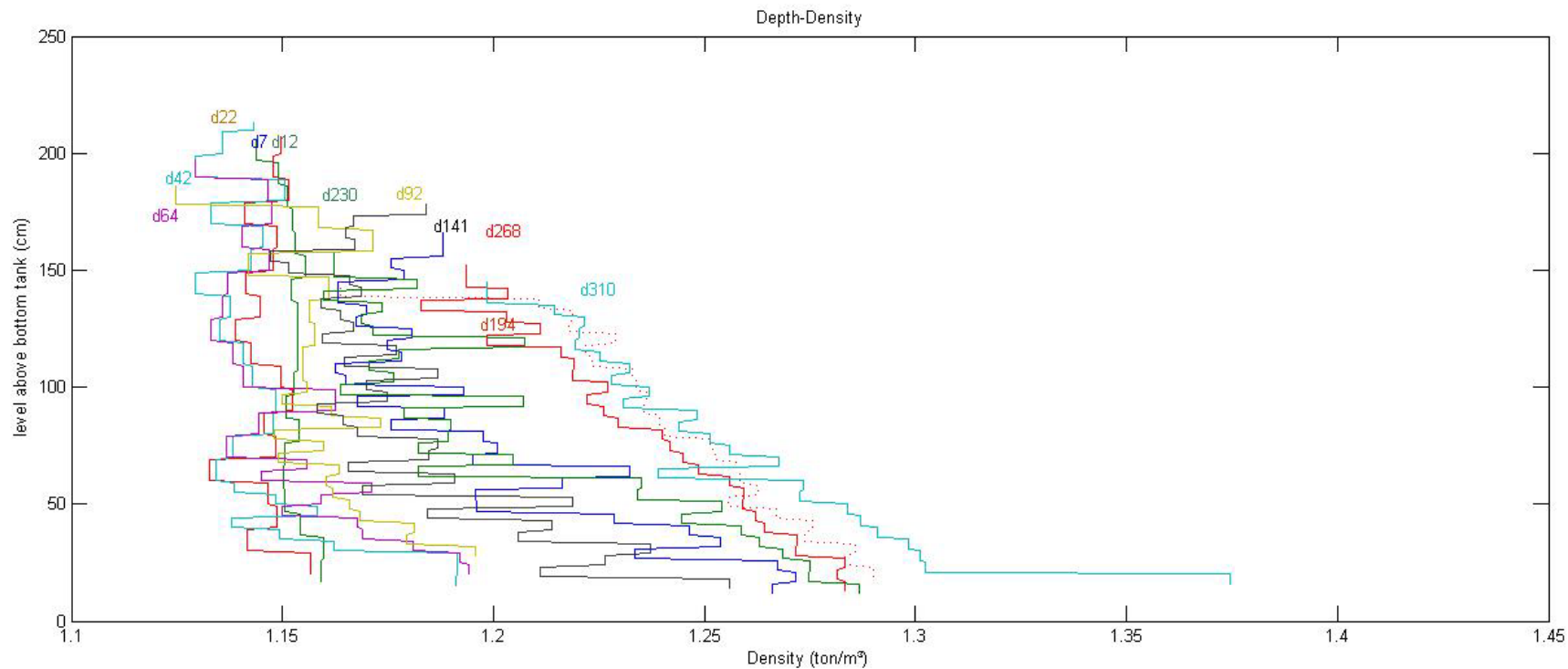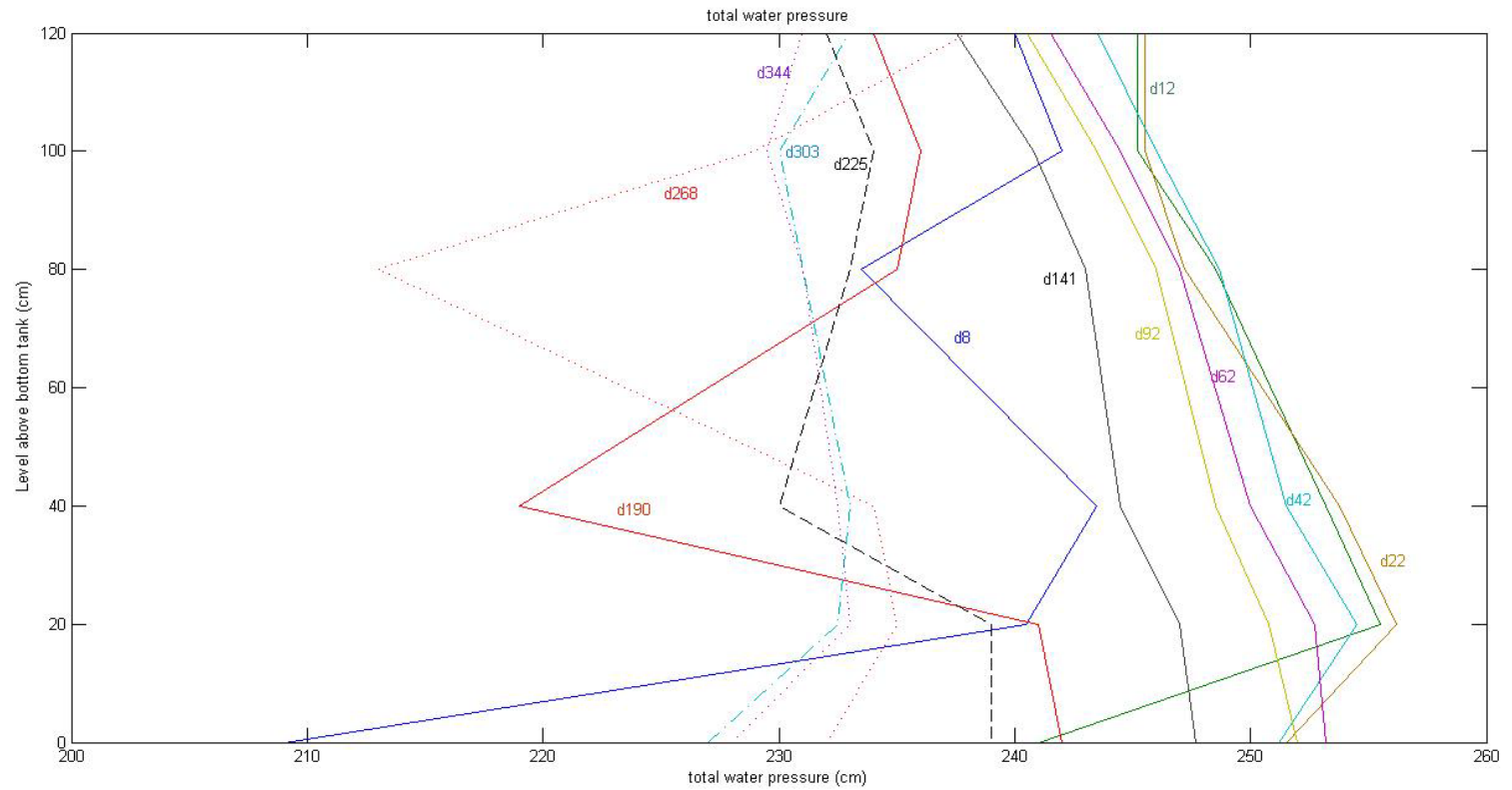
Figure 38: Evolution of total water pressure for some reference days in the STT.
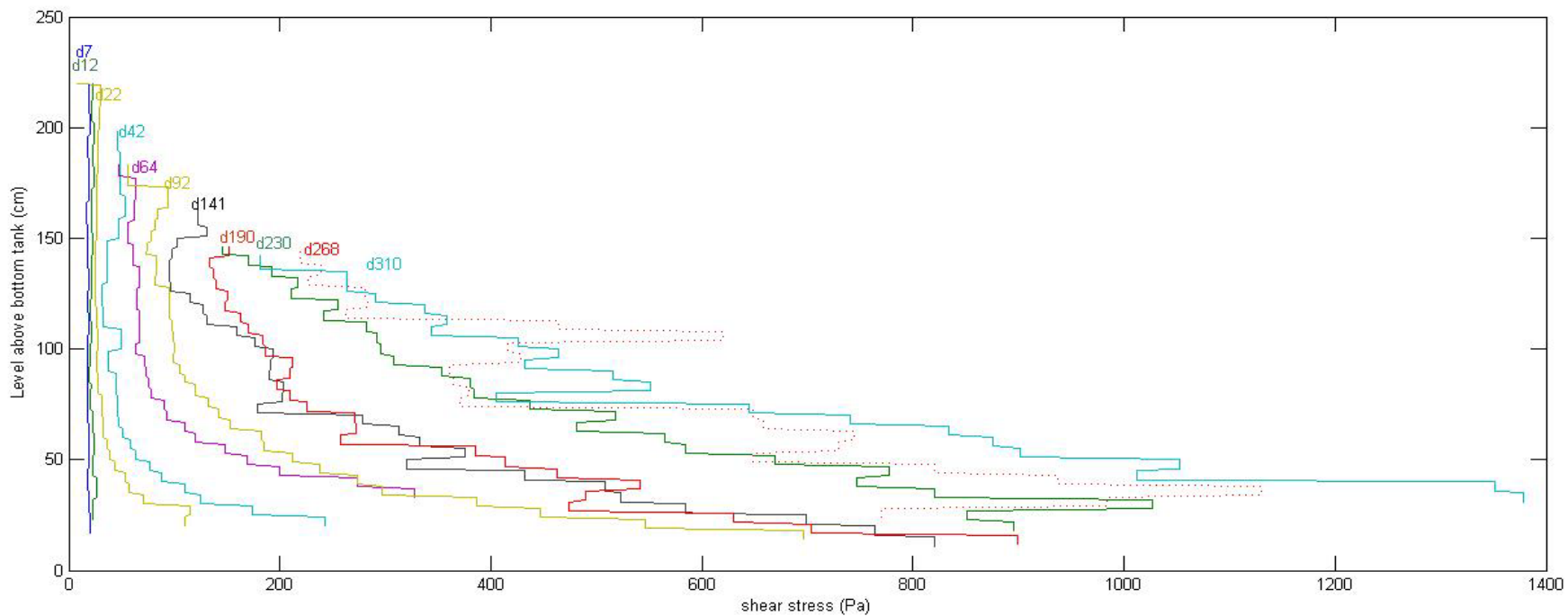Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
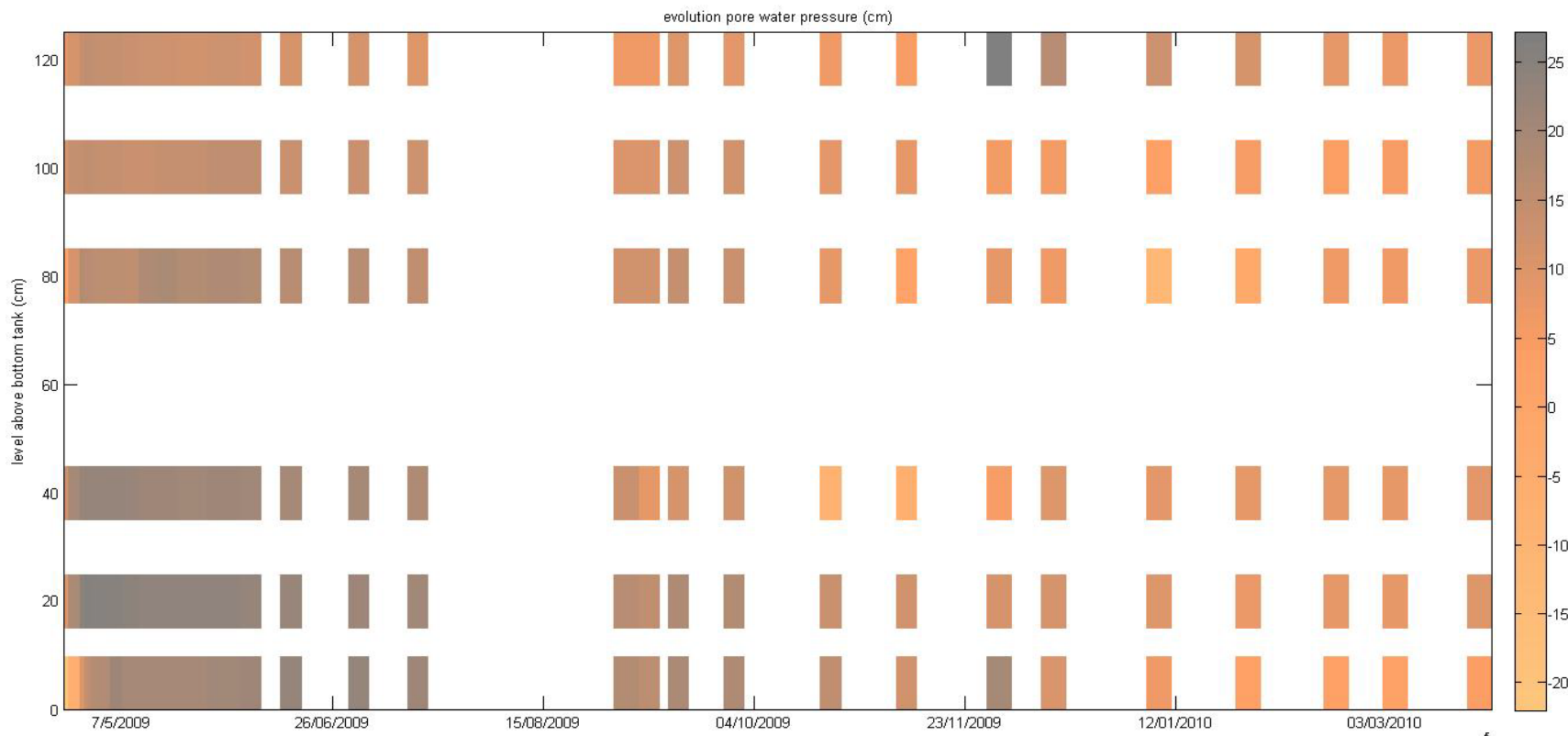
Figure 39: Evolution of pore overpressure at the piezometric filter levels in the STT
(0, 20, 40, 80, 100 and 120 cm above bottom tank) from 22/4/2009 (D7) until 25/03/2010 (D344)
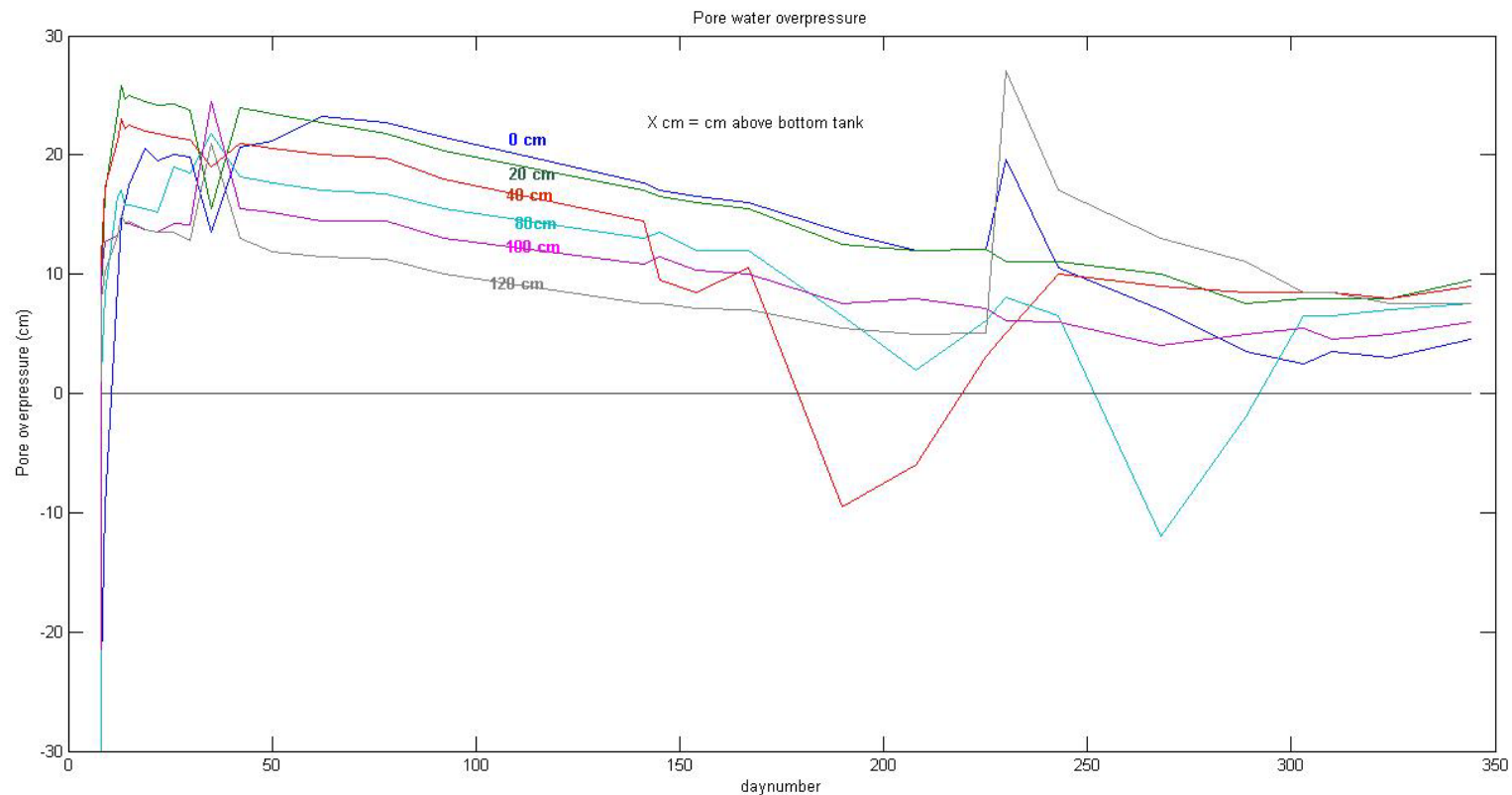
Figure 40: Evolution of pore overpressure for the measured pore water levels in the STT from 22/4/2009 (D7) until 25/03/2010 (D344). The numbers 0, 20, 40, 80, 100 and 120 cm refer to the depth of the piezometric filters above the bottom of the tank (cm)
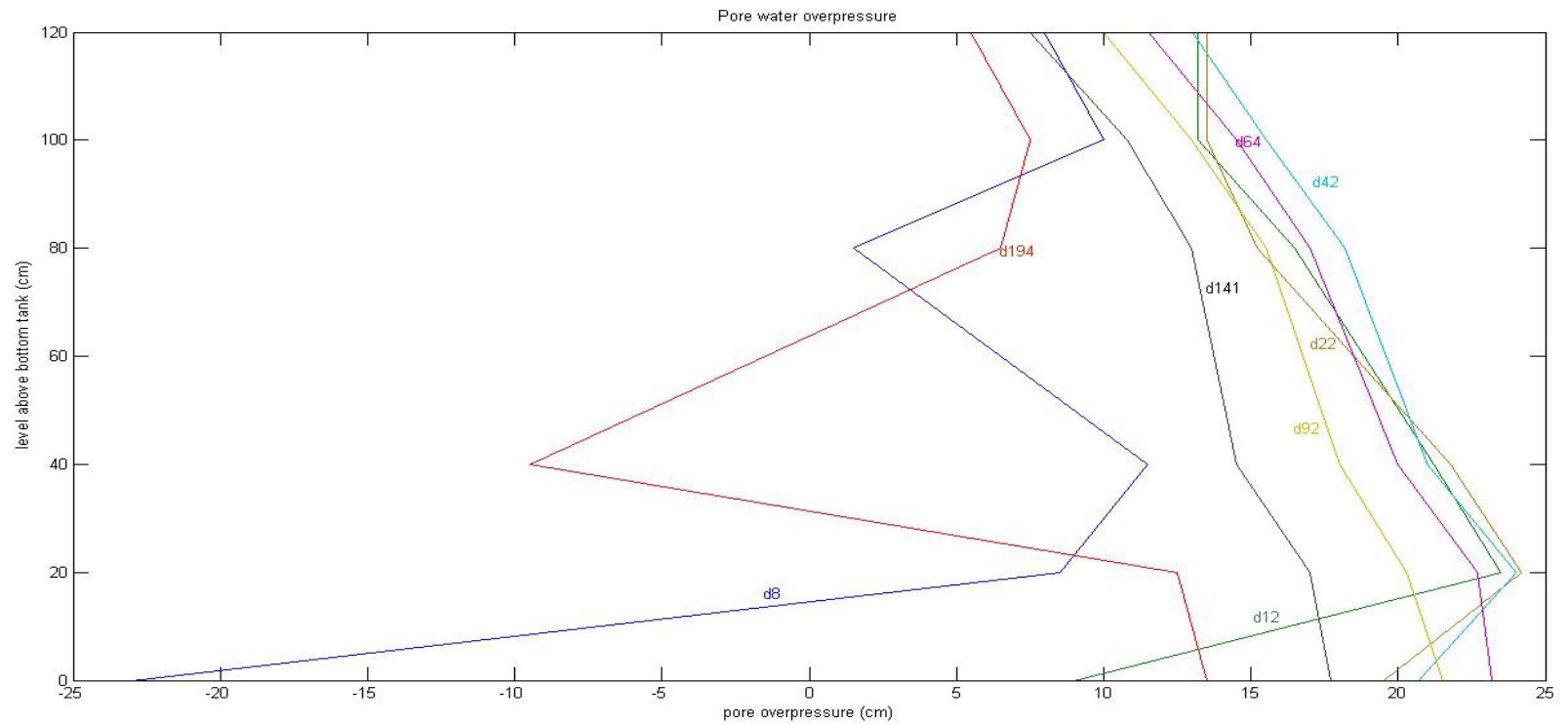
Figure 41: Evolution of pore overpressure for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')

4.6 Thermo-gravimetric analysis

Dry matter, organic matter and carbonate content are measured by drying and ignition with the Prepash 229 (§3.4). The dry matter content is measured after heating up to a temperature of 105° or more if 105°C is not sufficient to release all the moisture from the sample (Figure 15). The organic matter content is measured after heating up to a temperature of 550° and the carbonate content after heating up to a temperature of 800°C. Table 4 shows the full heating process as followed when performing the thermo-gravimetric analysis.

Step	Temp 1 [°C]	Temp 2 [°C]	Gas	Gas Flow [l/min]	Time [min]	Auto Stop [1/min]
1	20	105	Air	3	20	
2	105	105	Air	3	360	
3	105	550	Air	3	60	
4	550	550	Air	3	150	0,1 mg/10
5	550	800	Air	3	30	
6	800	800	Air	3	45	0,1 mg/10

Table 4: Prepash 229: stepwise heating

The content of dry matter and organic matter were analysed on a regular basis from d7 till d64 and from day 141 till d344. Between d64 and d141 no analysis was performed. Carbonates are measured from 16/09/2009 (d154). The analysis of the content of dry matter and organic matter happened on a subsample of the material that was used for deriving the previously mentioned parameters.

4.6.1 Dry matter

In Figure 43, Figure 44 and Figure 45 the evolution of dry matter content in the STT is illustrated for the period of 22/4/2009 till 19/02/2010 (d310). In Figure 43 all measurements are shown. More details are given for some reference days (Figure 44) and reference depths (Figure 45). From Figure 43 it can be seen that the dry matter content of the sediment for the measuring period varies between 20 and 36% weight of the original sample.

The dry matter content of the sediment and the density of the sediment are correlated by equation:

$$Density = (100 - DM) * \rho_{H_2o} + DM * \rho_{soil}$$

Where

DM = dry mater content of sediment (%)

ρ_{H2O} = density of water

ρ_{soil} = density of the dry soil in sediment (derived)

The correlation is also shown in Figure 42.
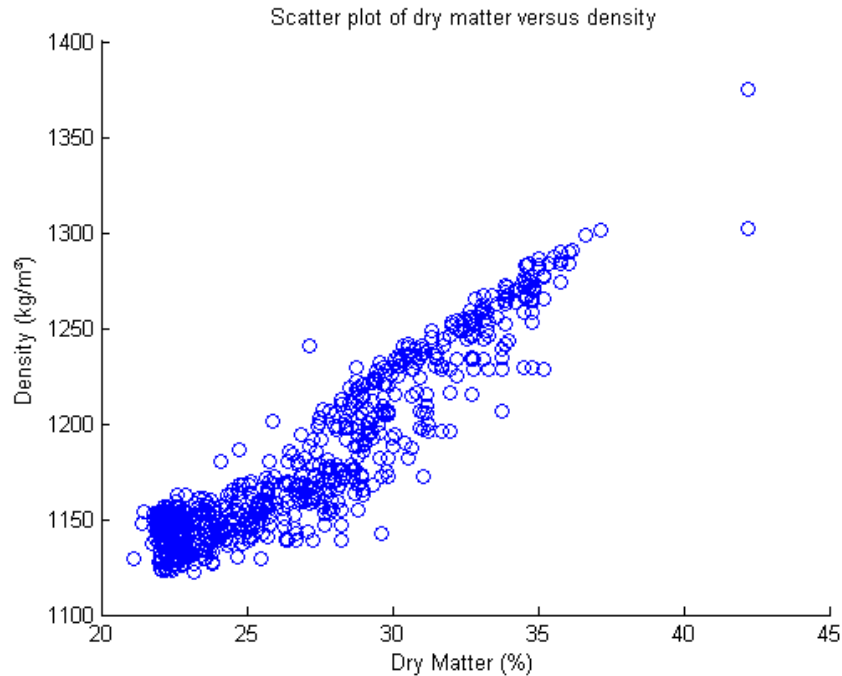
Figure 42: Scatter plot of the dry matter and the density measurements

The 1200 kg/m³ density limit is illustrated on Figure 43. This density limit corresponds roughly with a dry matter content of 29%.

Figure 44 shows the vertical variation for some depths trough the tank. Like density, dry matter content increases with time. Also the decrease in dry matter content from the bottom to the top of the sediment column is very consistent.

Figure 45 shows the dry matter evolution for some reference days. The first weeks, there is little vertical variation in dry matter content (d7-d12). After two months (d64), the dry matter content has rapidly increased, particularly for the lowest half a meter where it attained 30%. As for density, the shift in dry matter content is more important for the lower half part of the sediment column than for the upper half part, leading to a more exponential course.

After half a year (d190), the vertical gradient of dry matter content is more linear than exponential. The dry matter content at the top has not increased more than 2%, but at the bottom at least 5%. Six months later (d344), the whole dry matter profile increased some 1-2%, without further vertical differentiation.

4.6.2   Organic matter

The organic matter content as a percentage of the dry matter content is illustrated in Figure 46, Figure 47 and Figure 48. Figure 46 shows all measurements. More details are given for some reference days (Figure 47) and reference depths (Figure 48). From Figure 46 it is clear that the organic matter content of the sediment for the measuring period varies between 10 and 20% weight of the dry matter content, with some exceptional values above 20%.

From Figure 47, some temporal variation can be seen. Organic matter content stays quite constant around 15% for most depth levels until +-d150. At d150, when the sediment temperature increases to a maximum level (Figure 23), the organic matter content for all depths drops 2-3% . From then on, organic matter content is very stable and stays at a constant 12% level for all depths.

At the top sediment levels (100 and 150 cm), we can observe an increase in organic matter content during the period d50 till d150. However, this increase is only suggested by 2 to 3 measurements and cannot readily be explained by chemical or biological processes. As a consequence, this rise in organic matter should be neglected.

On the contrary, the drop in organic matter shortly after the summer period is confirmed by all depth levels. This drop is probably caused by increased mineralisation of the organic matter during the summer season (d50-d150), when sediment temperature increased by 3 to 4°C.

Figure 47 shows a shift in organic matter content from a mean 15% organic matter content before d150 to a mean 13% organic matter content after d150 . Also, there seems to be some layers in the sediment column where the organic matter content is higher than in the surrounding layers.

4.6.3   Carbonate

Carbonates are measured from 16/09/2009 (d154). During the first 150 days, no carbonate content was measured.

By increasing the temperature to 800°C, only the easy decomposable carbonates are measured. At 800°C carbonate is converted to carbon dioxide and air. The loss of carbon dioxide and air causes a measurable loss of weight. At 1000°C all carbonates are decomposed.

The carbon content as a percentage of the dry matter content is illustrated in Figure 49, Figure 50 and Figure 51. Figure 49 shows all measurements. More details are given for some reference days (Figure 50) and reference depths (Figure 51). From Figure 49 it is clear that the organic carbon content of the sediment varies most of the time around 5% of the dry matter content, with some exceptional values around 3 and 6%.

From Figure 50 and Figure 51 it can be seen that there is little temporal variation in carbon content. Except some outlier values and a general decrease around d230, the carbon content stays stable around 5% for all depth levels in the tank.
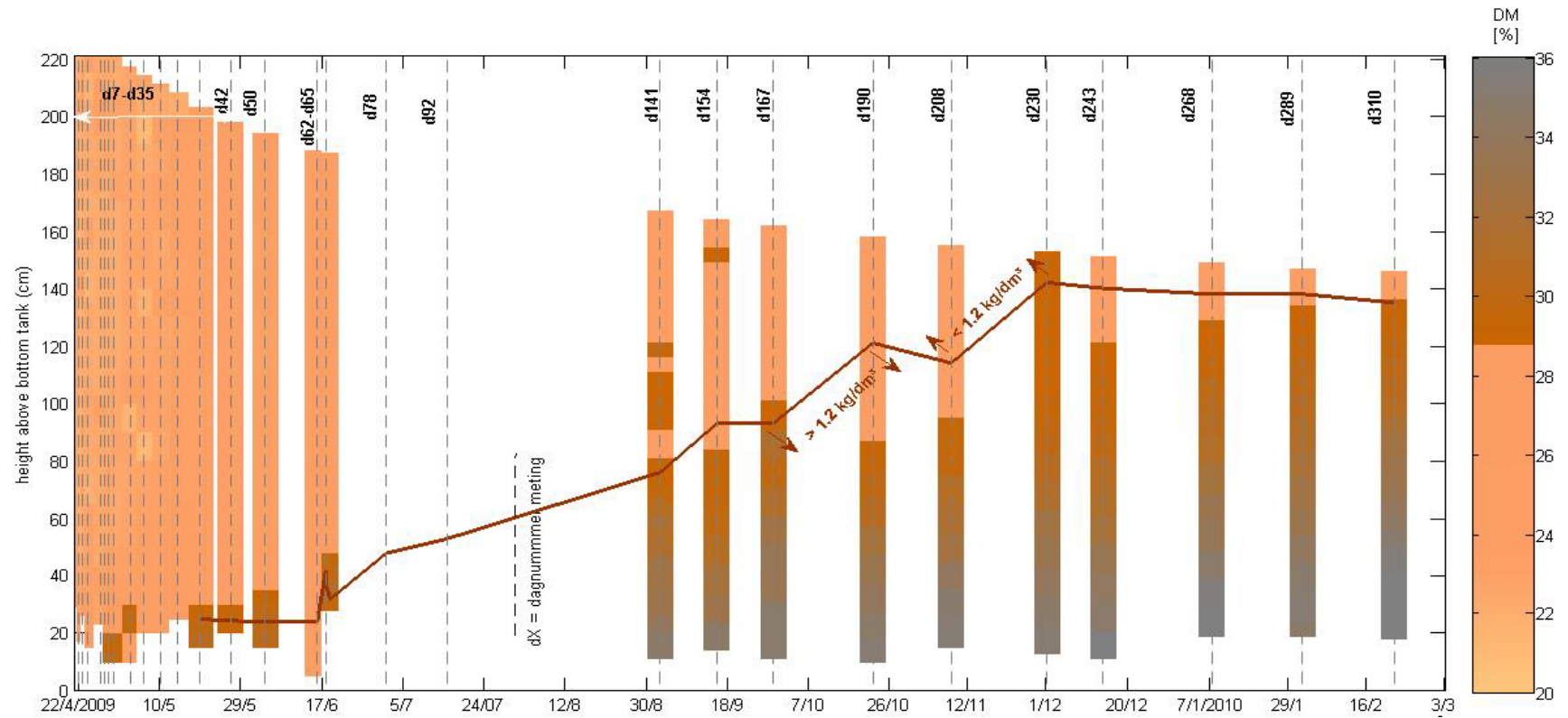
Figure 43: Evolution of dry matter content (%) in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7').
The density level of 1.2 g/cm³ is illustrated by the thick brown line
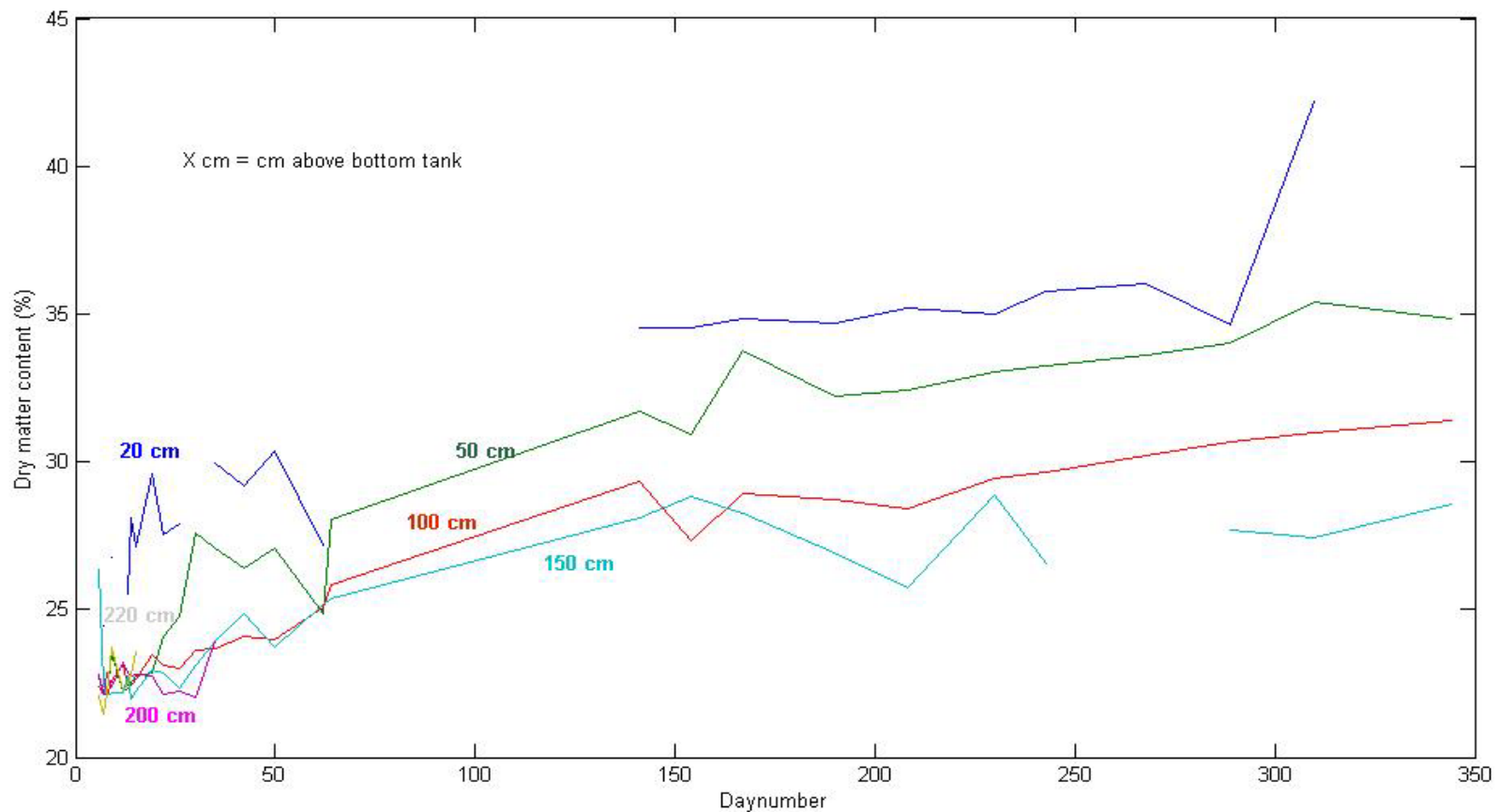
Figure 44: Evolution of the dry matter content for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm). Because of the reduction of sediment height,
the graphs for the depths of 200 and 220 cm are limited
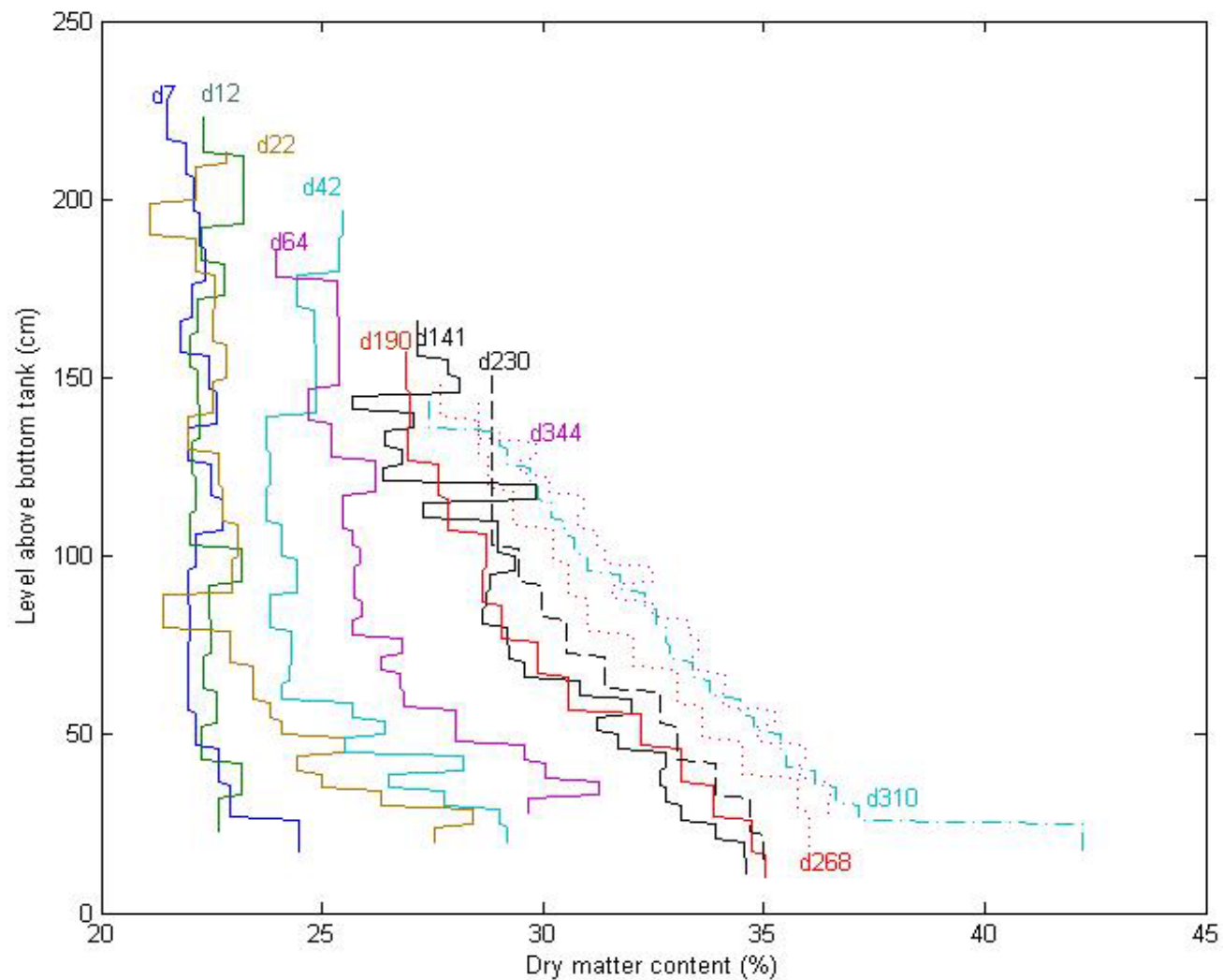
Figure 45: Evolution of dry matter content for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
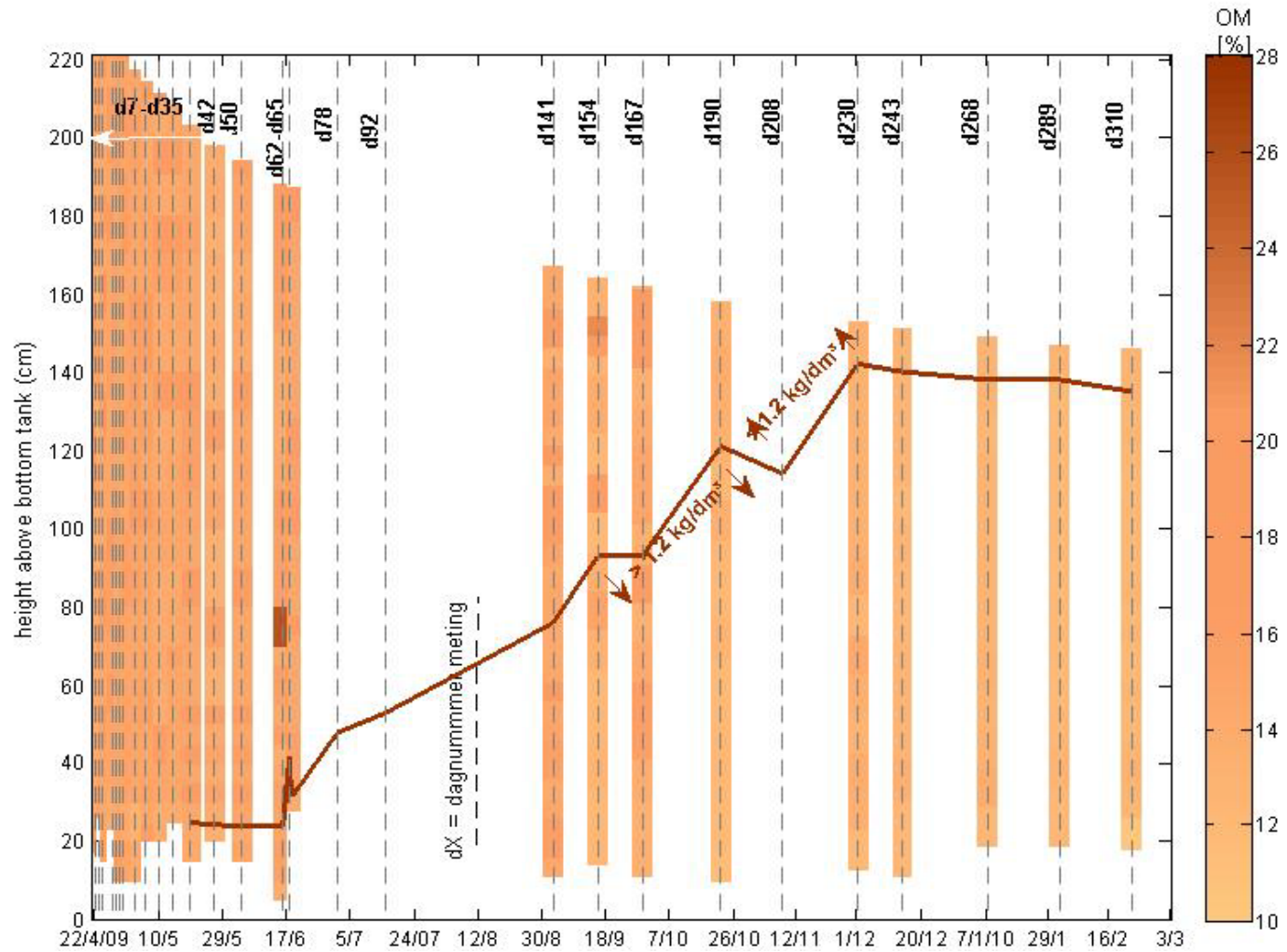
Figure 46: Evolution of organic matter content (as a percentage of dry matter) in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7').
The density level of 1.2 g/cm³ is illustrated by the thick brown line
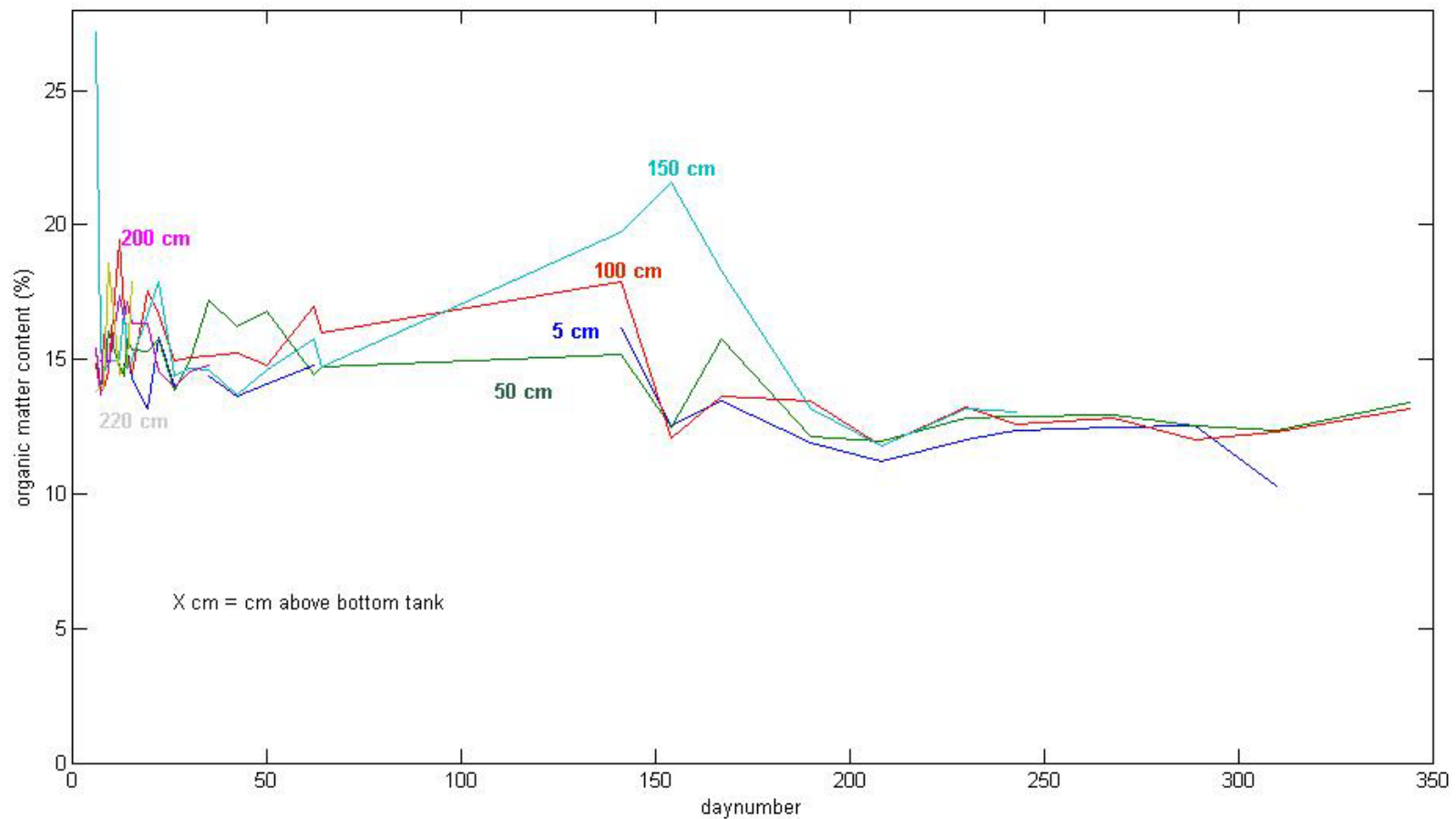
Figure 47: Evolution of organic matter content (as a percentage of dry matter) for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm). Because of the reduction of sediment height,
the graphs for the depths of 200 and 220 cm are limited
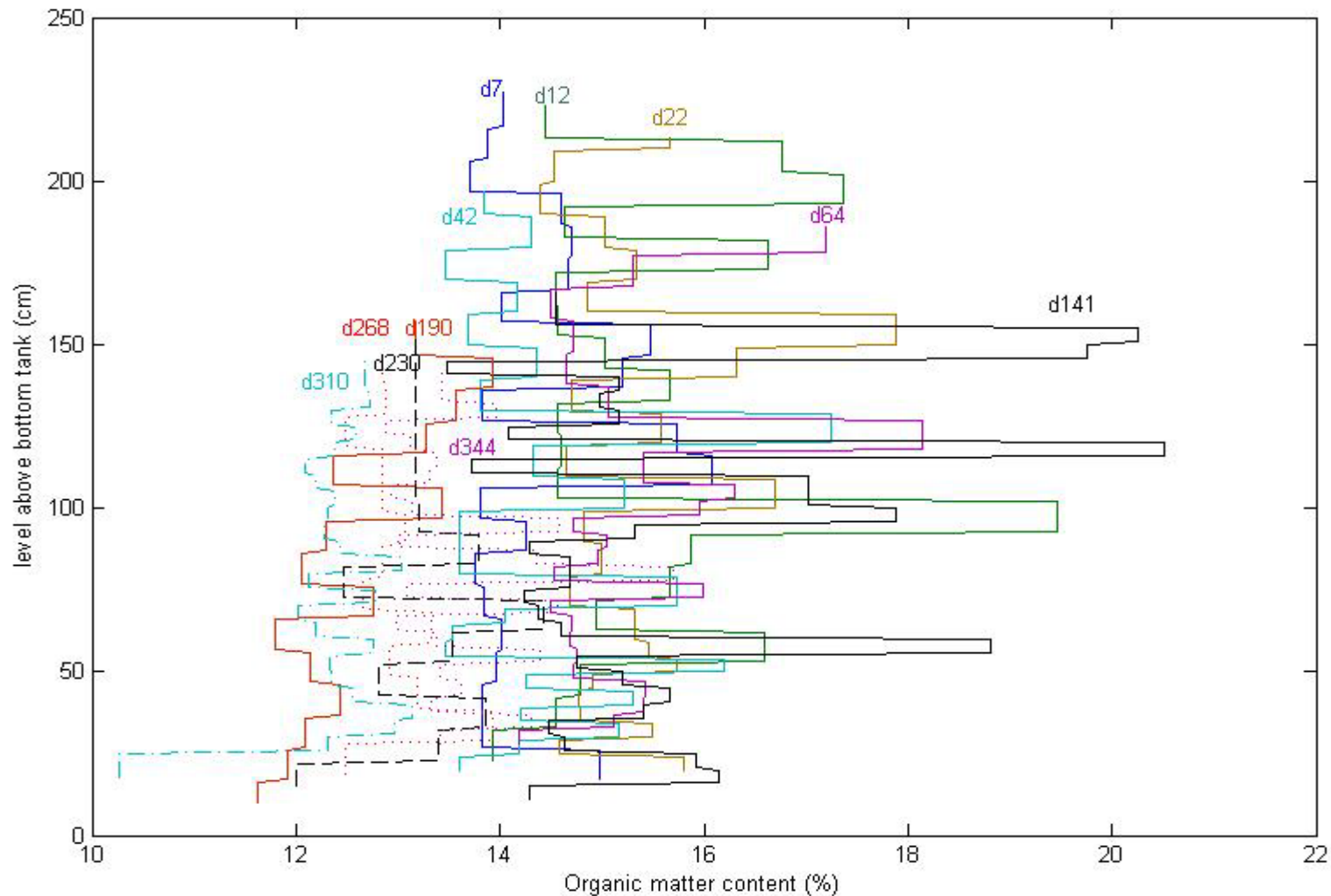
Figure 48: Evolution of organic matter content (as a percentage of dry matter) for some reference days in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
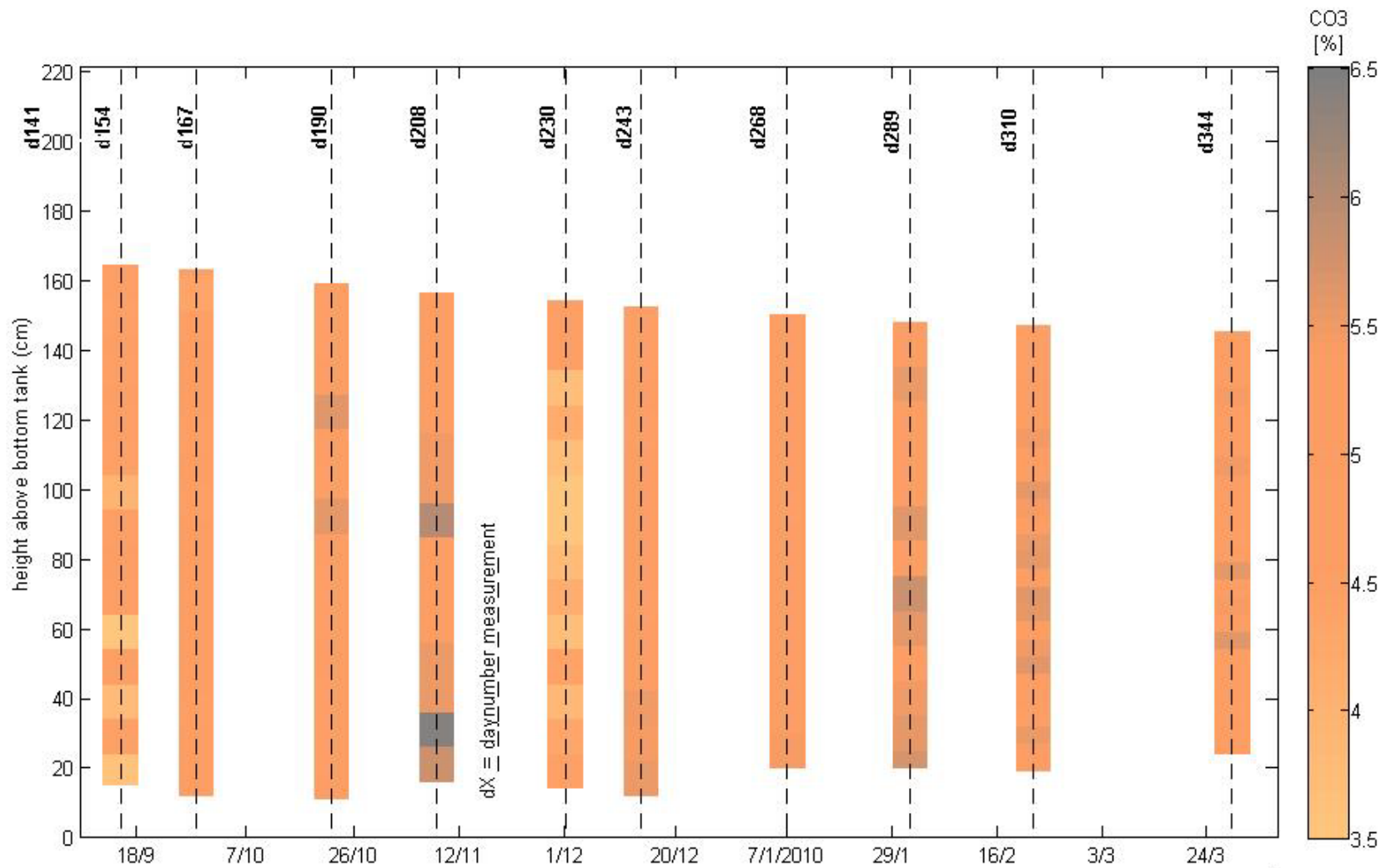
Figure 49: Evolution of carbonate content (as a percentage of dry matter) in the STT.
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7')
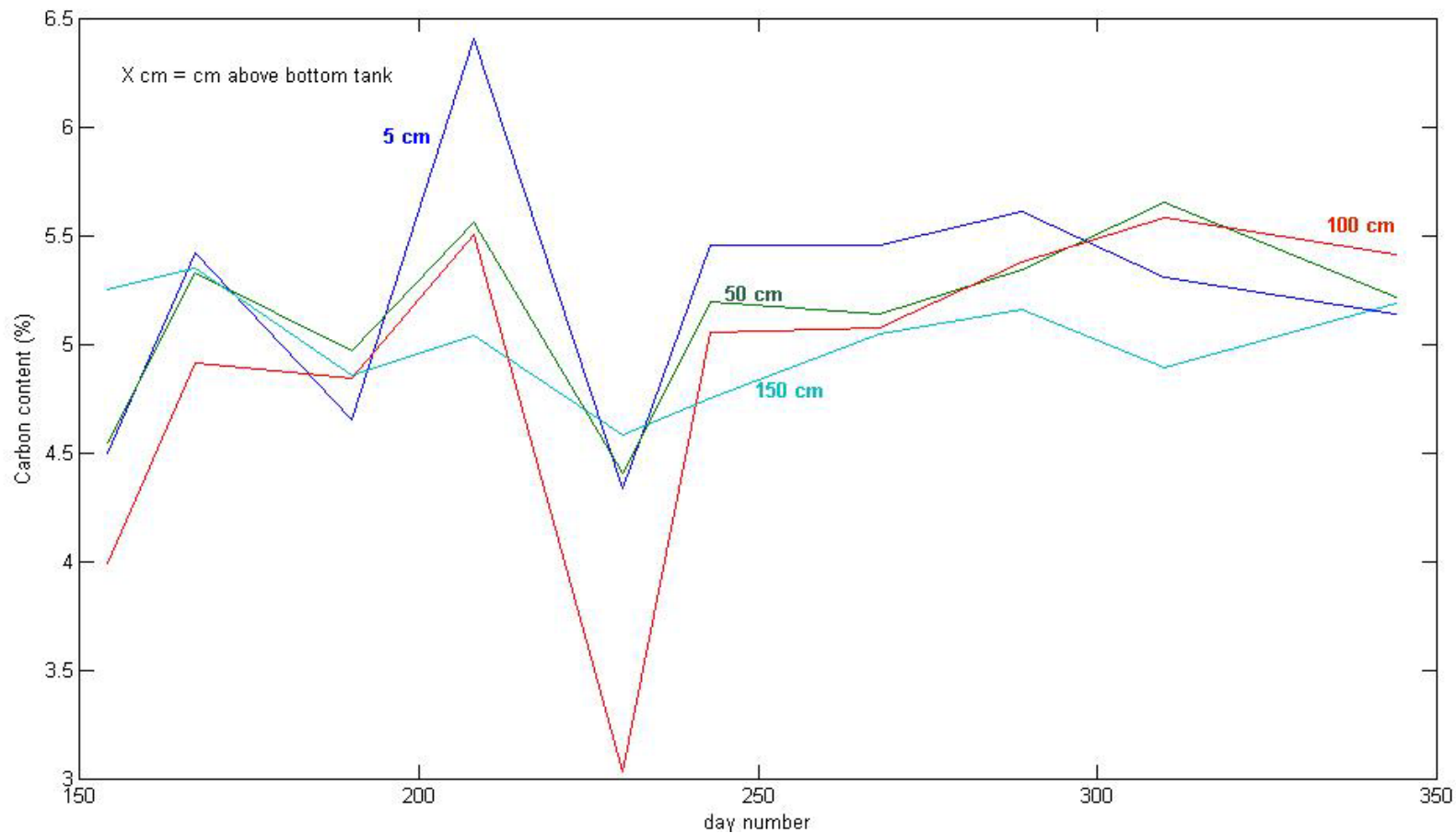
Figure 50: Evolution of carbon content (as a percentage of dry matter) for some reference depths in the STT.
Numbers refer to the depth above the bottom of the tank (cm). Because of the reduction of sediment height ,
no graphs for the depths of 200 and 220 cm are on the figure
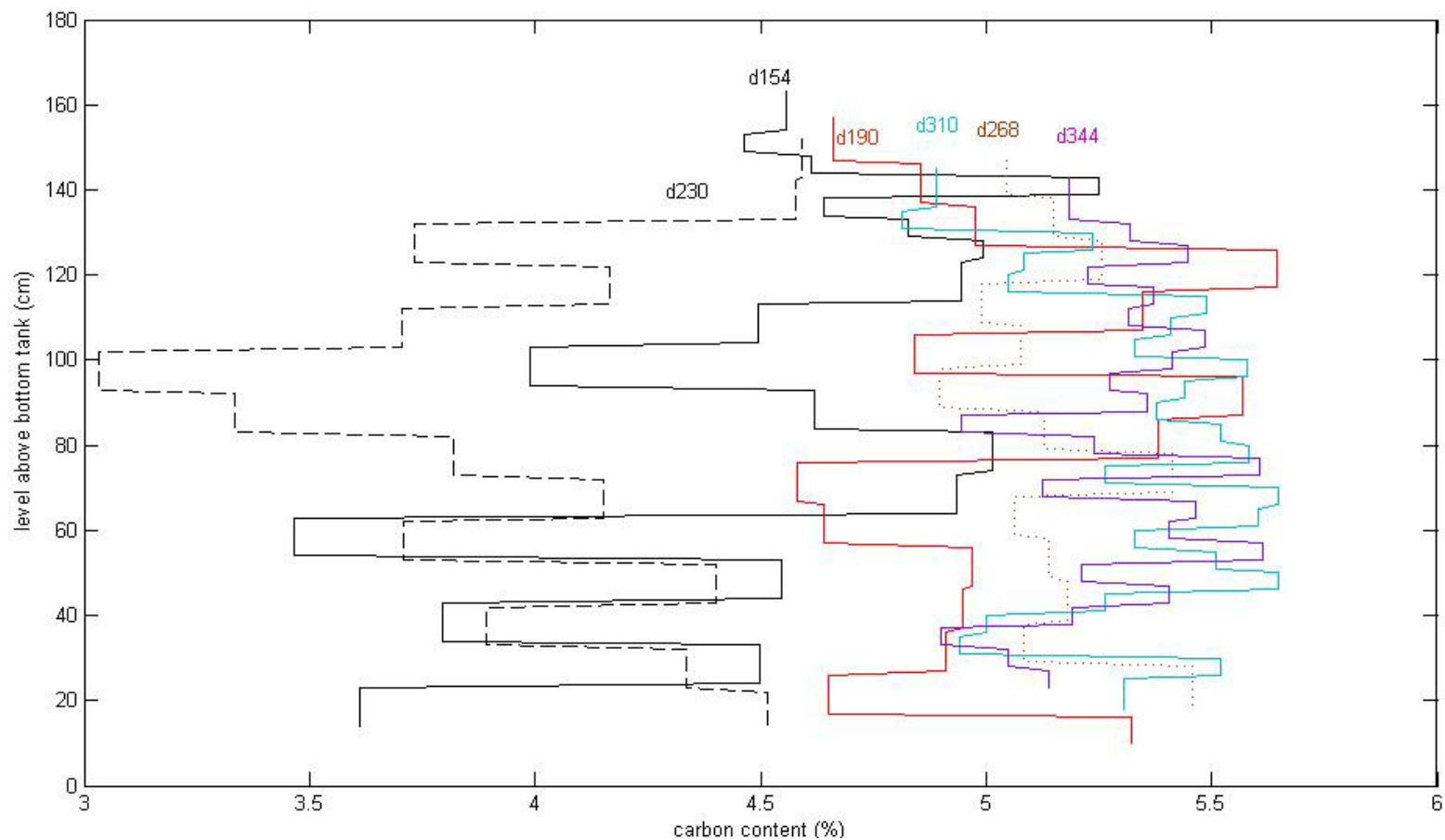
Figure 51: Evolution of carbon content for some reference days in the STT (as a percentage of dry matter).
Numbers refer to the day of measurement from 16/4/2009 on (e.g. 22/4/2009 is day 'd7') -OM in % van DM

4.7 Grain size distribution

The grain size distribution of the sediment is given in Figure 52 for 21/04/2009 (d6, full line) and 8/1/2010 (d268, dotted line). The figure illustrates the almost unchanged grain size distribution during consolidation during more than 8 months. So no sorting of the sediment took place during the measurement period. Median grain size is 7-9 µm, d_{10} is around 1-2 µm and d_{90} around 40-60 µm.
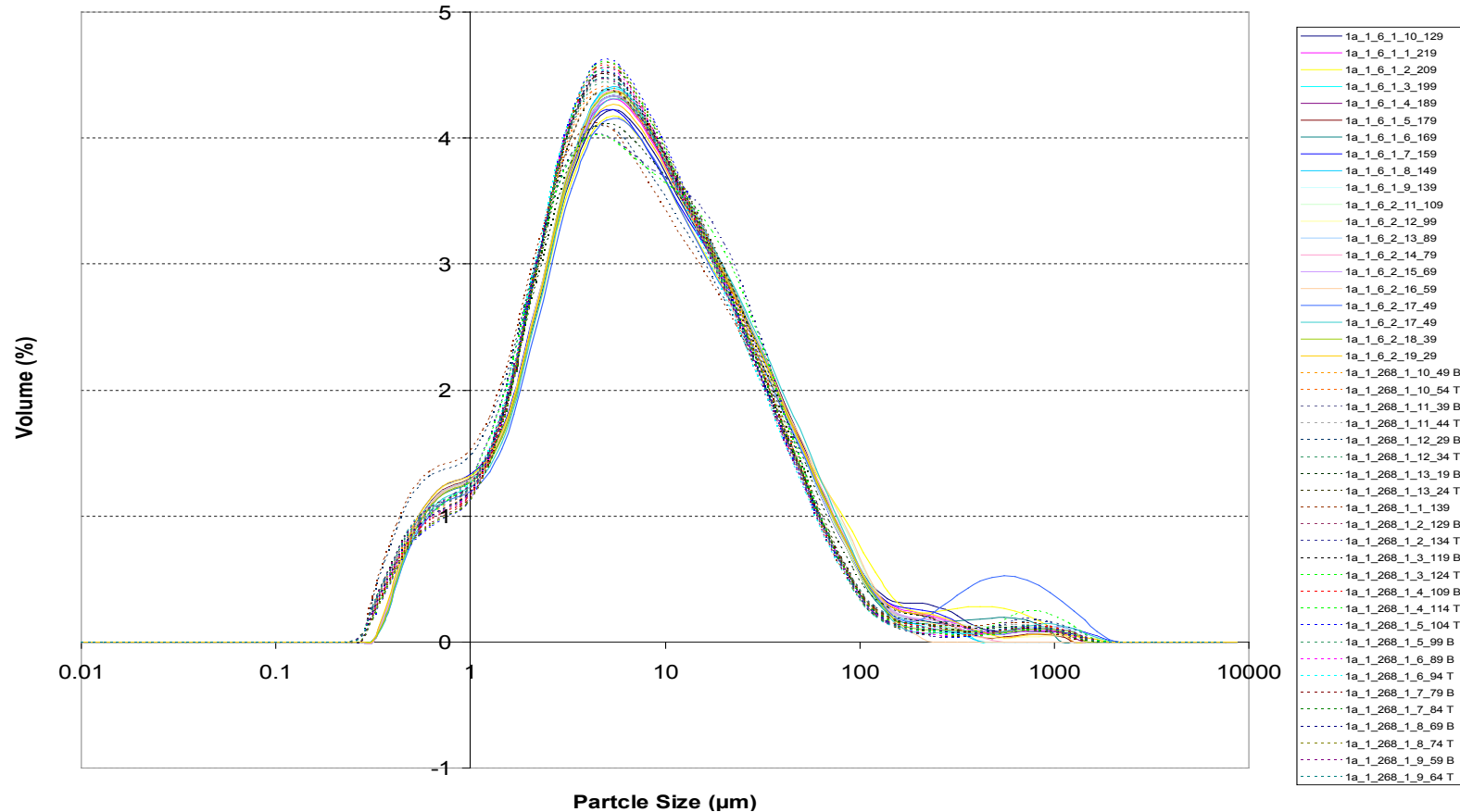
Figure 52: Grain size distribution of sediment in the STT for 21/04/2009 (d6, full line) and 8/1/2010 (d268, dotted line)

5 Discussions

5.1 Measured parameters

This chapter summarizes the measurements and findings for the measurements that were carried out in the Sediment Test Tank in Flanders Hydraulic Research from 21/04/09 till 26/5/2010. The structure in this chapter corresponds to that of chapter 4.

5.1.1 YSI

- Comparative measurements: research was conducted into 1) stabilisation time of the measurements in sediment and water and 2) deviation of the measurements in sediment from the measurements in the aqueous solution. Following conclusions could be made for <u>stabilisation time</u>:
  - o Parameters pH, temperature, specific conductivity: stabilisation time is less than 10 seconds. The here mentioned stabilisation time is valid for measurements in the sediment and not in the pore water.
  - o Parameters oxide-reduction-potential, dissolved oxygen: recommended stabilisation time is 120 seconds.

Following conclusions can be made for deviation of the measurements in sediment from the measurements in the aqueous solution, which is a measure of the <u>reliability of the measurements in sediment</u> (see also Table 3):
  - o For the parameters pH and temperature, there are no significant differences between measurements in sediment and measurements in the aqueous solution (average difference over all measurements is 3%). For these parameters, the YSI can be used (without adjustments) in water and sediment.
  - o For the parameter conductivity, the difference between sediment and pore water parameter measurement is smaller than 30% for most sediment samples with density lower than 1.2 g/cm³. For densities higher than 1.2 g/cm³, the conductivity measurement in sediment underestimates the conductivity measurement in water with +- 70%. Conductivity can be measured in sediment with lower density values, but for the higher values the measurements are not reliable.
  - o For the parameters oxide-reduction-potential and dissolved oxygen, differences between measurements in sediment and measurements in the aqueous solution are on average respectively 72 and 274%. Measurements with the YSI in sediment are not reliable for oxide-reduction-potential and dissolved oxygen values.
- Regarding evolution of <u>temperature</u>, following conclusions can be made:
  - o Temperature varies between 15 en 21°C for the whole measurement period. During summer season a temperature of at least 18.5°C was recorded.
  - o In general: the temperature of the top sediment differs 1 to 2°C from the bottom sediment.
  - o Due to the buffering capacity of the sediment, the variation in temperature throughout the measurement period reduces towards the bottom.
- Regarding evolution of <u>pH</u>, following conclusions can be made:
  - o The pH varies between 6.3 and 7.3 through the measurement period
  - o pH decreases in summer season (d78 – d167), and stays low after summer season (d190/d194). The increase in temperature seems to have triggered biochemical reactions.
  - o Vertical variation in pH is small at initialisation and increases with time.
  - o With the high level of sulphates in the sediment, pH is expected to decrease when oxidation level increases (due to oxidation of sulphates). This is not confirmed by the measurements in the STT, since pH decreases from the top (high oxidation level) to the bottom (low oxidation level) of the tank. Further investigation to the underlying processes is needed.

5.1.2 Density

- The accuracy of the density measurement is limited to +/- 20 kg/m³, but some spikes with deviations larger than 20 kg/m³ are observed for the higher densities.
- The sediment is brought into the STT at a density of 1140 kg/m³. After a month, the density of the bottom layer (maximum 0.5 m) increased to 1200 kg/m³ near the bottom. At the top, density is still 1150 kg/m³.
- The height of the sediment column that has a density of at least 1200 kg/m³ is increasing strongly during summertime. After summer, the complete lower half of the tank is consolidated to 1200 kg/m³. At day 230 after filling the tank, sediment of almost the total tank (except top layer) reaches 1200 kg/m³. At day 310 after filling the tank, sediment at the bottom of the tank reaches densities more than 1300 kg/m³.

5.1.3 Rheological parameters

- Rheograms are obtained on subsamples with the Anton Paar rheometer 'Physica MCR 301'. Shear rates of 0.09999 till 1000 1/s were applied.
- Little vertical variation in shear stress, needed to apply the chosen shear rates, can be observed the first weeks. After a couple of weeks, shear stresses at the bottom start to increase. Until d190, bottom shear stresses are increasing much more than the shear stress of the upper half of the sediment column. After d190, all shear stresses are increasing more in parallel. After 300d, rheogram are follow a linear trend.

5.1.4 Pore pressure

Concerning total water pressure, following conclusions can be drawn:

- Total water pressure is monitored by the direct readings of the water height in the riser tubes connected to the piezometric filters installed at 0, 20, 40, 80, 100 and 120 cm above the bottom of the tank. The first days the total pore levels are stabilising. After a couple of days pore levels are decreasing in time. This decrease is only partially due to a decrease in water level. More important is the decrease in pore overpressure
- Total pore water pressure: After the stabilisation time, there is a temporal decrease of total water pressure. On any given day the total water pressure increases with depth.
- There are some important variations in pore water evolution through time for some depths (0, 40, 80, 120 cm), particularly from d200 to d300. These variations are probably due to filter clogging. Around d230, filters were cleaned.

Concerning pore water pressure, following conclusions can be drawn:

- Pore overpressure is calculated by subtracting the height of the water column (hydrostatic pressure) in the tank from the filter readings.
- The pore overpressure decreases with time, and are higher at the bottom of the sediment column than at the top
- The same temporal and vertical gradients can be seen for the pore overpressure as for the total pore water pressure: pore overpressures decrease with time, and are higher at the bottom of the sediment column than at the top.
- Negative overpressures or deviations from the global trends are probably due to clogging of the filters or evaporation of the water in the riser tubes
- In general, conclusions of former consolidation studies with similar sediment are confirmed by the results
- After 400 days, the consolidation processes has not ended since the pore overpressures are still positive.

5.1.5 Thermo gravimetrical analysis

Concerning dry matter content, following conclusions can be drawn:
- Dry matter content varies between 20 and 36% weight of the original sample
- This density limit (1.2 g/cm³) corresponds roughly with a dry matter content of 29%.
- A consistent decrease in dry matter from bottom to top can be observed.
- During the first weeks, little variation can be observed.
- After two months, dry matter content increased to 30% in the lower half part of the STT
- After half a year, the vertical gradient is more linear than exponential. The increase at the top is smaller than 2%; near the bottom the increase is larger than 5%.
- After a year there is a further increase of 1-2%, without vertical differentiation.

Concerning organic matter, following conclusions can be drawn:
- In general: varies between 10 and 20% weight of the dry matter content
- The organic matter content is more or less constant at 15% until d150
- At d150 the organic matter content decreases to 12% and remains constant. Probably this happens because of mineralisation of the organic matter during the summer season

Concerning carbonate, some 5% of the dry matter content was observed for all depths starting from d150, with some exceptional values around 3 and 6%.

5.1.6 Grain size distribution

Median grain size is 7-9 µm, d_{10} is around 1-2 µm and d_{90} around 40-60 µm. Because there is no change in grain size distribution over the first 8 months, and no variation in grain size distribution over depth, one can conclude that there is no sorting of the sediment during the measurement period.

6   Conclusions

A whole range of parameters have been measured over time and depth to monitor the sediment characteristics as much as possible. Some parameters are measured in the Sediment Test Tank, e.g. measurements by the YSI multi-parameter probe and pore pressure measured by piezometric tubes. Other parameters are measured on undisturbed samples taken from the Sediment Test Tank, among others: density, rheological parameters and thermo-gravimetric analysis.

Some problems and solutions (measuring protocol) to apply the YSI multi-parameter probe in a cohesive sediment environment are given. The parameters pH and temperature measured with the YSI can be used in without restriction in the (fluid, consolidated) cohesive sediment layer.

For the parameters conductivity, oxido-reduction-potential and dissolved oxygen, measured with the YSI in the sediment layer, no solid conclusions can be drawn. The recommended stabilisation time, when measuring in cohesive sediment with a maximum density of 1200 kg/m³, is 120 seconds. When the density exceeds 1200 kg/m³ an underestimation up to 60% might be noticed (see 4.2.1).

The above mentioned recommendation is not applicable for DO and ORP.

Besides of the problems with the YSI-probe, also other measurement problems are discussed. Negative overpressures are an indication of filter clogging or evaporation of the water in the riser tubes. The monitoring setup revealed some errors due to sample handling or inaccuracy of the measurement devices. These problems are to be tackled in the next phases of the research.

For most physico-chemical parameters a clear evolution in time and depth could be monitored by the YSI multi-parameter probe and by measurements on undisturbed samples in the sediment test tank. The parameter trends revealed that evolution of the consolidation and related processes are little, but clearly, influenced by seasonal conditions.

We can distinguish quick physical consolidation processes (seconds-weeks) and slower bio-chemical processes (months- years). The quick physical processes (dewatering, transport channels, restacking of particles,…) will mask or influence the occurring (bio) chemical processes. When the general drainage stops, the further dewatering will be triggered by diffusive processes. At that time other (bio) chemical processes will occur in the enclosed liquid and between the particles.

The increase in temperature seems to trigger (bio) chemical reactions, for example a sudden decrease in pH in the beginning of the summer. Most of the findings can be explained by theoretical concepts and are consistent with former studies: influence of temperature, trends of dissolved oxygen and ORP, increase of consolidation with higher temperature, decrease of overpressure in time and mineralisation of organic matter.

But some measurement trends remain unclear and deserve further investigation, for example: decrease of pH from the top to the bottom of the tank.

A variation in consolidation speed between sediment layers can be observed. The first 100 days, the consolidation speed of the bottom layers is higher than the upper sediment layers. After the summer, the consolidation speed of the top layer is higher than the lower sediment layers. Both density and shear stresses at applied shear rates increase rather exponentially towards the bottom. But for the shear stress, the increase towards the bottom is more pronounced than for density. A non-uniform relationship between density and rheology (yield stress) can be stated. This is also hinted in sub reports 1, 2 and 11.

7   List of references

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Annex 1: comparison measurement data

Density (kg/m³)	date measurement	Treatment sediment sample	Dissolved oxygen (%)		
			sediment	water	**difference**
1050	1/12/2009	without further treatment	17.6	35	**50**
1050	1/12/2009	Heated till +- 25°C	3	37.5	**92**
1050	7/12/2009	without further treatment	16	42	**62**
1100	1/12/2009	without further treatment	24	28.4	**15**
1100	1/12/2009	Heated till +- 25°C	41	34	**21**
1100	7/12/2009	without further treatment	12.8	37	**65**
1100	April 2010	without further treatment	6.5	6.2	**5**
1110	April 2010	without further treatment	4.5	54	**92**
1120	April 2010	without further treatment	4.8	52	**91**
1140	April 2010	without further treatment	8.4	44	**81**
1150	1/12/2009	without further treatment	15	37	**59**
1150	1/12/2009	Heated till +- 25°C	13	52	**75**
1150	7/12/2009	without further treatment	12.5	28.3	**56**
1160	April 2010	without further treatment	3.8	42	**91**
1190	April 2010	without further treatment	9	38	**76**
1200	April 2010	without further treatment	3.5	40.1	**91**
1200	1/12/2009	without further treatment	13	27	**52**
1200	1/12/2009	Heated till +- 25°C	16	41	**61**
1200	April 2010	without further treatment	4	4	**0**
1230	April 2010	Original sediment, 80-120 cm above bottom tank	3.6	25	**86**
1250	April 2010	Acidified (+ 3M HCl)	4.4	21	**79**
1250	April 2010	Deacidified (+ 3M NaOH)	4.1	47	**91**
1250	April 2010	Deacidified (+ 3M NaOH)	3.8	36.6	**90**
1260	April 2010	Original sediment, 40-80 cm above bottom tank	3.3	26	**87**
1280	April 2010	without further treatment	12.9	4	**223**
1290	April 2010	Original sediment, 0-40 cm above bottom tank	3.2	41	**92**
Average			10	34	**72**
Average_2009			17	36	**55**
Average_2010			5	32	**85**
SD			9	14	**42**
Range			0-500 (air saturation)		
Accuracy			+- 2 % of reading		

date measurement	density (kg/m³)	Specific conductivity (mS/cm)			pH (/)		
		sediment	water	difference (%)	sediment	water	difference (%)
1/12/2009	1050	42	43	**2**	7.45	7.4	**1**
1/12/2009	1050	41.5	44.8	**7**	7.98	7.41	**8**
7/12/2009	1050	10.93	6.33	**73**	7.57	7.67	**1**
1/12/2009	1100	0.57	44	**99**	7.75	7.25	**7**
1/12/2009	1100	36.5	44.7	**18**	7.6	7.2	**6**
7/12/2009	1100	20.32	20.3	**0**	7.4	7.37	**0**
April 2010	1100	25.72	32.23	**20**	7.65	7.64	**0**
April 2010	1110	30.43	38.25	**20**	7.41	7.59	**2**
April 2010	1120	33.92	44.46	**24**	7.38	7.55	**2**
April 2010	1140	38.59	52.08	**26**	7.24	7.5	**3**
1/12/2009	1150	15	40.9	**63**	7.6	7.3	**4**
1/12/2009	1150	32	41.3	**23**	7.5	7.31	**3**
7/12/2009	1150	24.54	32.18	**24**	7.3	7.2	**1**
April 2010	1160	37.48	52.67	**29**	7.35	7.47	**2**
April 2010	1190	23.6	56.01	**58**	7.43	7.47	**1**
April 2010	1200	34.53	51.42	**33**	7.24	7.43	**3**
1/12/2009	1200	21	43.5	**52**	7.4	7.11	**4**
1/12/2009	1200	10.4	43.6	**76**	7.4	7.1	**4**
April 2010	1200	18.06	50	**64**	7.06	7.22	**2**
April 2010	1230	20.22	45.88	**56**	7.17	7.32	**2**
April 2010	1250	14.62	42.75	**66**	6.43	6.65	**3**
April 2010	1250	11.95	35.28	**66**	7.86	8.01	**2**
April 2010	1250	12.12	35.17	**66**	8.41	8.49	**1**
April 2010	1260	14.67	45.94	**68**	7.07	7.25	**2**
April 2010	1280	15.09	47.46	**68**	7.27	7.29	**0**
April 2010	1290	14.04	45.61	**69**	7.19	7.47	**4**
	Average	23	42	**45**	7	7	**3**
	Average_2009	23	37	**40**	8	7	**4**
	Average_2010	23	45	**49**	7	7	2
	SD	11	10	27	0	0	2
	Range	0-100			0-14		
	Accuracy	+- 0.5% of reading			+- 0.2		

date measurement	density (kg/m³)	ORP (mV)			T (°C)		
		sediment	water	**difference (%)**	sediment	water	**difference (%)**
1/12/2009	1050	-154	-122.7	**26**	19.6	20.3	**3**
1/12/2009	1050	-65	-115	**43**	24.3	25	**3**
7/12/2009	1050	-144	-65	**122**	19.66	20.31	**3**
1/12/2009	1100	36	-125	**129**	18.4	20.4	**10**
1/12/2009	1100	-98	-122	**20**	24.6	26.3	**6**
7/12/2009	1100	-170	-67	**154**	19.42	20	**3**
April 2010	1100	-104	-56.3	**85**	22.47	22.2	**1**
April 2010	1110	-125	69	**281**	21.71	22.62	**4**
April 2010	1120	-139	66.2	**310**	21.51	22.71	**5**
April 2010	1140	-120	35	**443**	21.53	22.6	**5**
1/12/2009	1150	-165	-119.9	**38**	19.7	*20.4*	**3**
1/12/2009	1150	-143	-91	**57**	25.6	26	**2**
7/12/2009	1150	-155	-72	**115**	19.47	20.3	**4**
April 2010	1160	-157	7.7	**2139**	21.4	22.58	**5**
April 2010	1190	-63	-4.6	**1270**	21.63	22.63	**4**
April 2010	1200	-159	-19	**737**	21.42	22.45	**5**
1/12/2009	1200	-142	-127	**12**	*22.3*	*20.45*	**9**
1/12/2009	1200	-172	-123	**40**	25.4	*25.5*	**0**
April 2010	1200	-124	-108.7	**14**	22.07	22.36	**1**
April 2010	1230	-159.9	-80	**100**	20.63	21.07	**2**
April 2010	1250	-86	-78	**10**	22.2	22.15	**0**
April 2010	1250	-147	85.6	**272**	21.91	21.94	**0**
April 2010	1250	-130	84.3	**254**	21.79	22.19	**2**
April 2010	1260	-158.9	-82	**94**	20.96	21.03	**0**
April 2010	1280	-158	-95.3	**66**	21.84	22.41	**3**
April 2010	1290	-159.7	78.7	**303**	21.36	21.07	**1**
	Average	-129	-48	**274**	22	22	**3**
	Average_2009	-125	-105	**69**	22	22	**4**
	Average_2010	-133	-6	**425**	22	22	**3**
	SD	45	75	468	2	2	2
	Range	-999 - +999			-5 - +70		
	Accuracy	+- 20			+- 2		

Annex 2: rheogram

Figure 53: shear stress as function of the height above the bottom for different measuring days at a shear rate of 0.1 1/s

Figure 54: shear stress as function of the height above the bottom for different measuring days at a shear rate of 0.2031 1/s

Figure 55: shear stress as function of the height above the bottom for different measuring days at a shear rate of 0.4125 1/s

Figure 56: shear stress as function of the height above the bottom for different measuring days at a shear rate of 0.8377 1/s
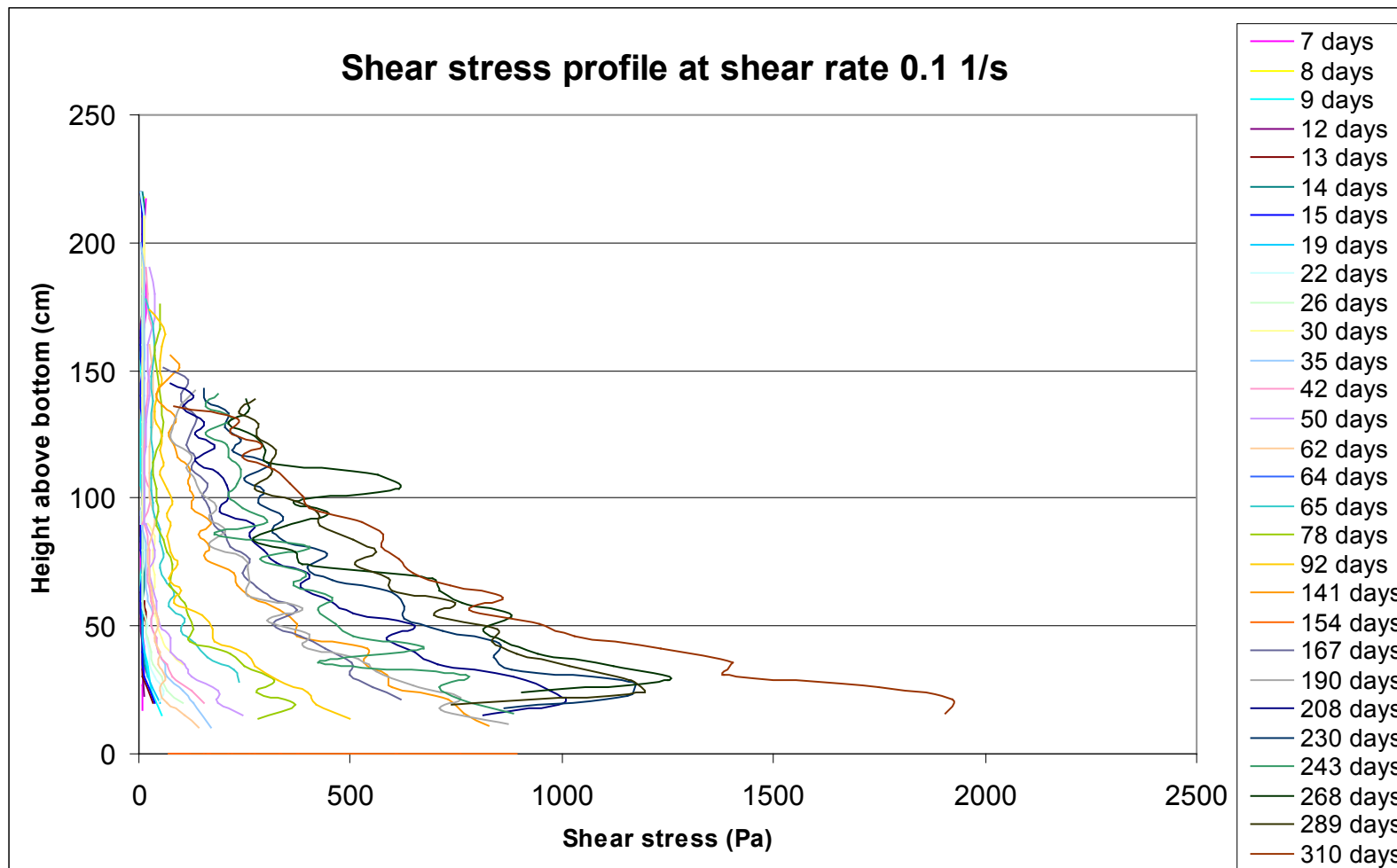
Figure 57: shear stress as function of the height above the bottom for different measuring days at a shear rate of 1.701 1/s

Figure 58: shear stress as function of the height above the bottom for different measuring days at a shear rate of 3.456 1/s
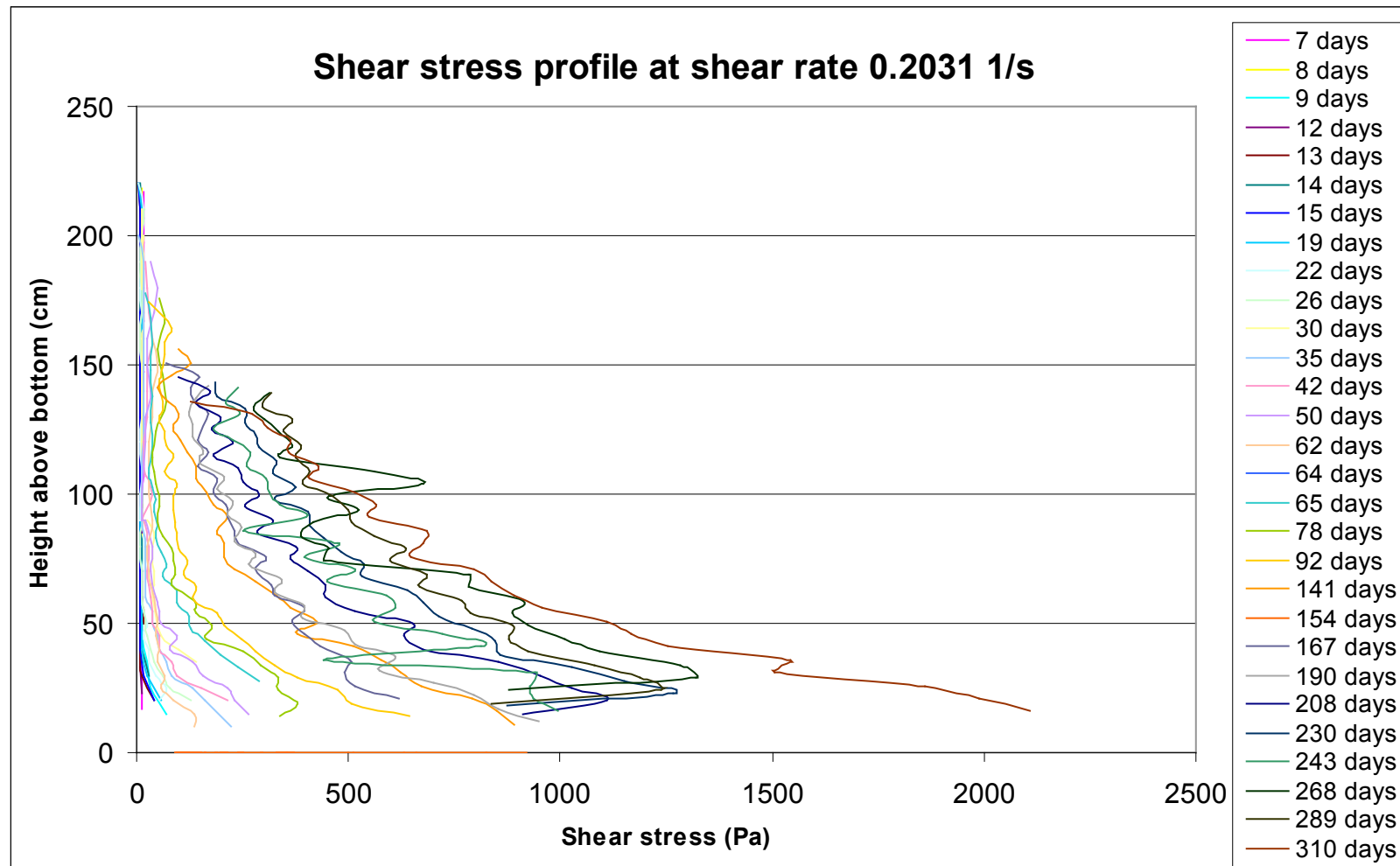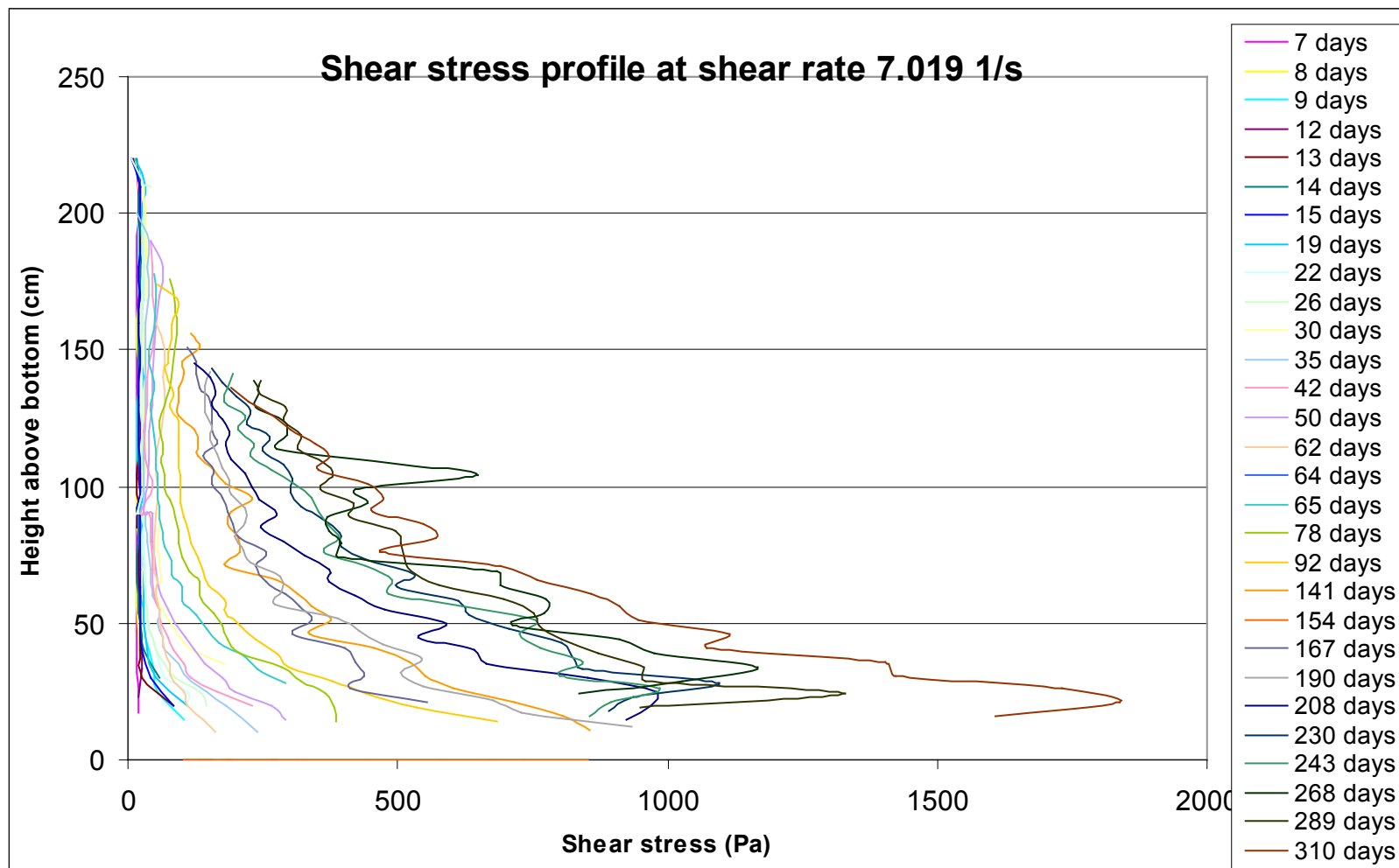
Figure 59: shear stress as function of the height above the bottom for different measuring days at a shear rate of 7.019 1/s
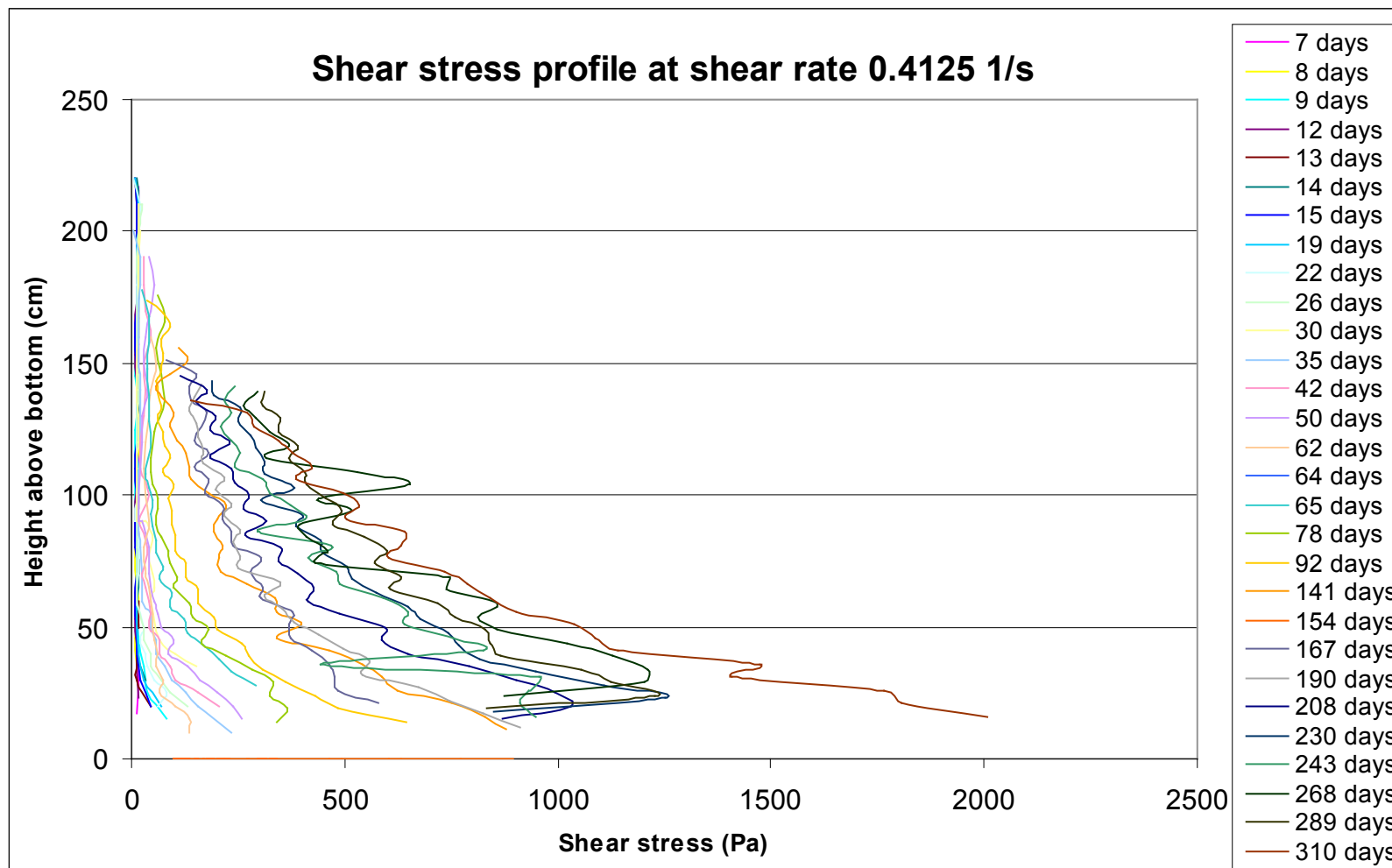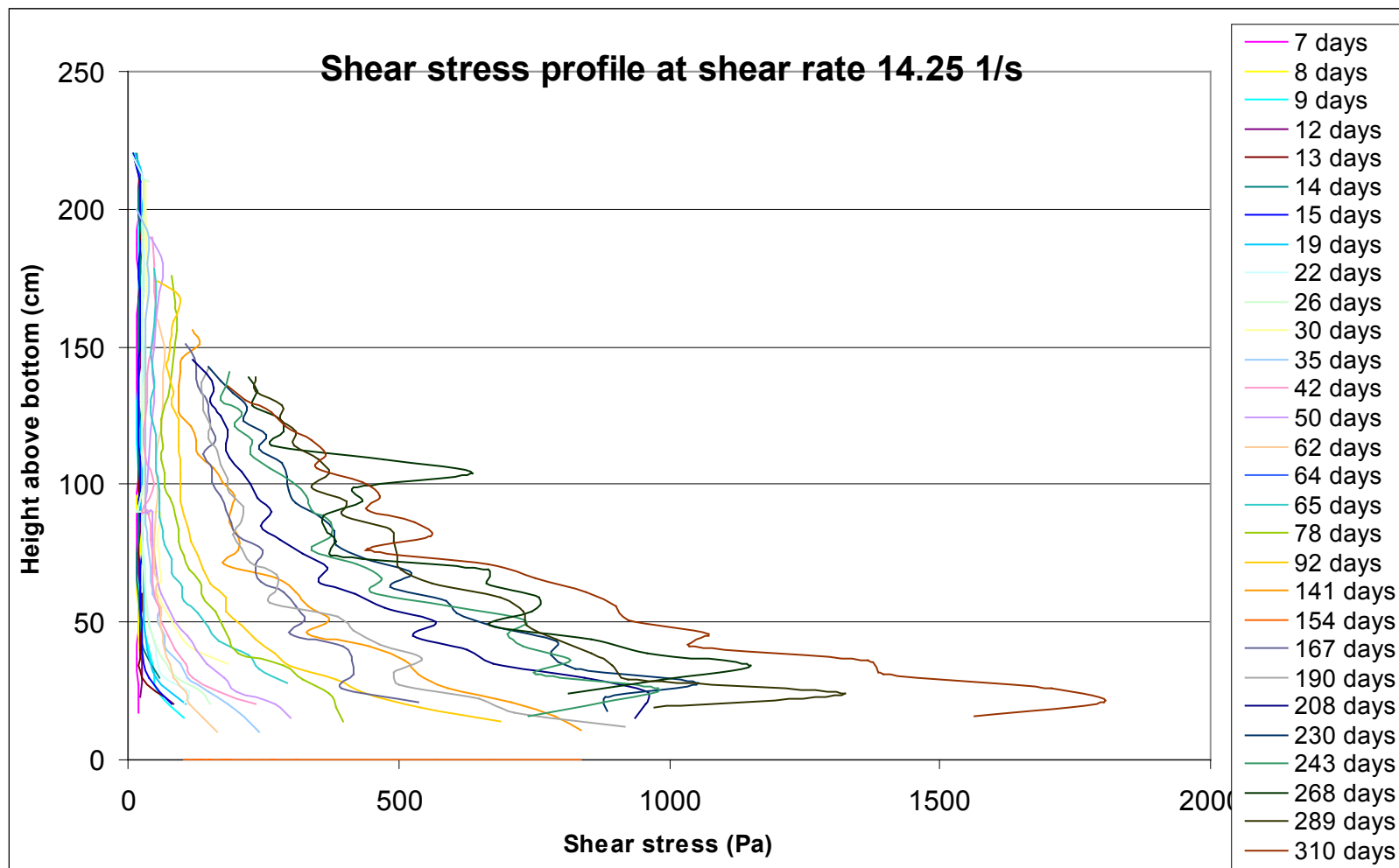
Figure 60: shear stress as function of the height above the bottom for different measuring days at a shear rate of 14. 25 1/s
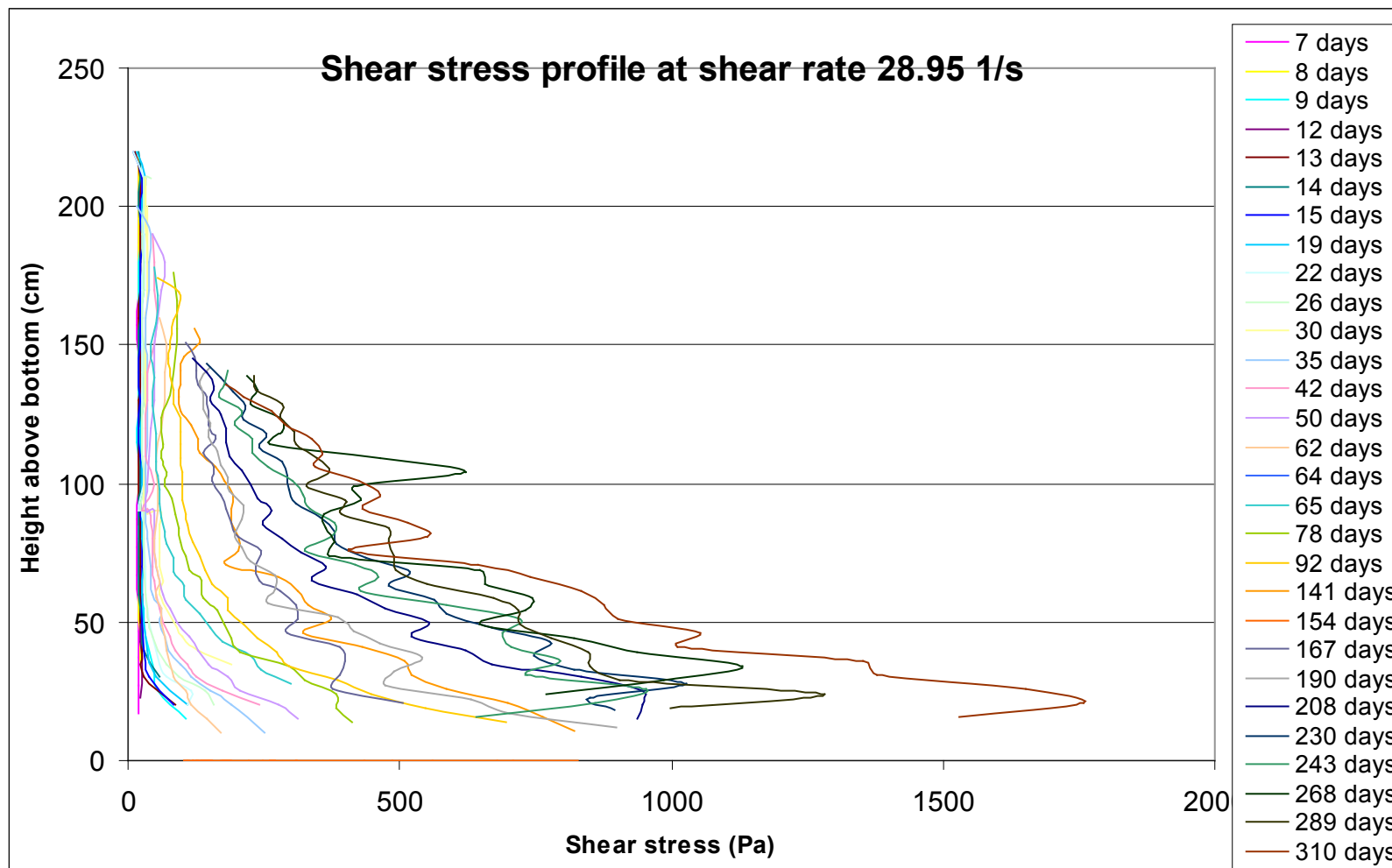
Figure 61: shear stress as function of the height above the bottom for different measuring days at a shear rate of 28.95 1/s
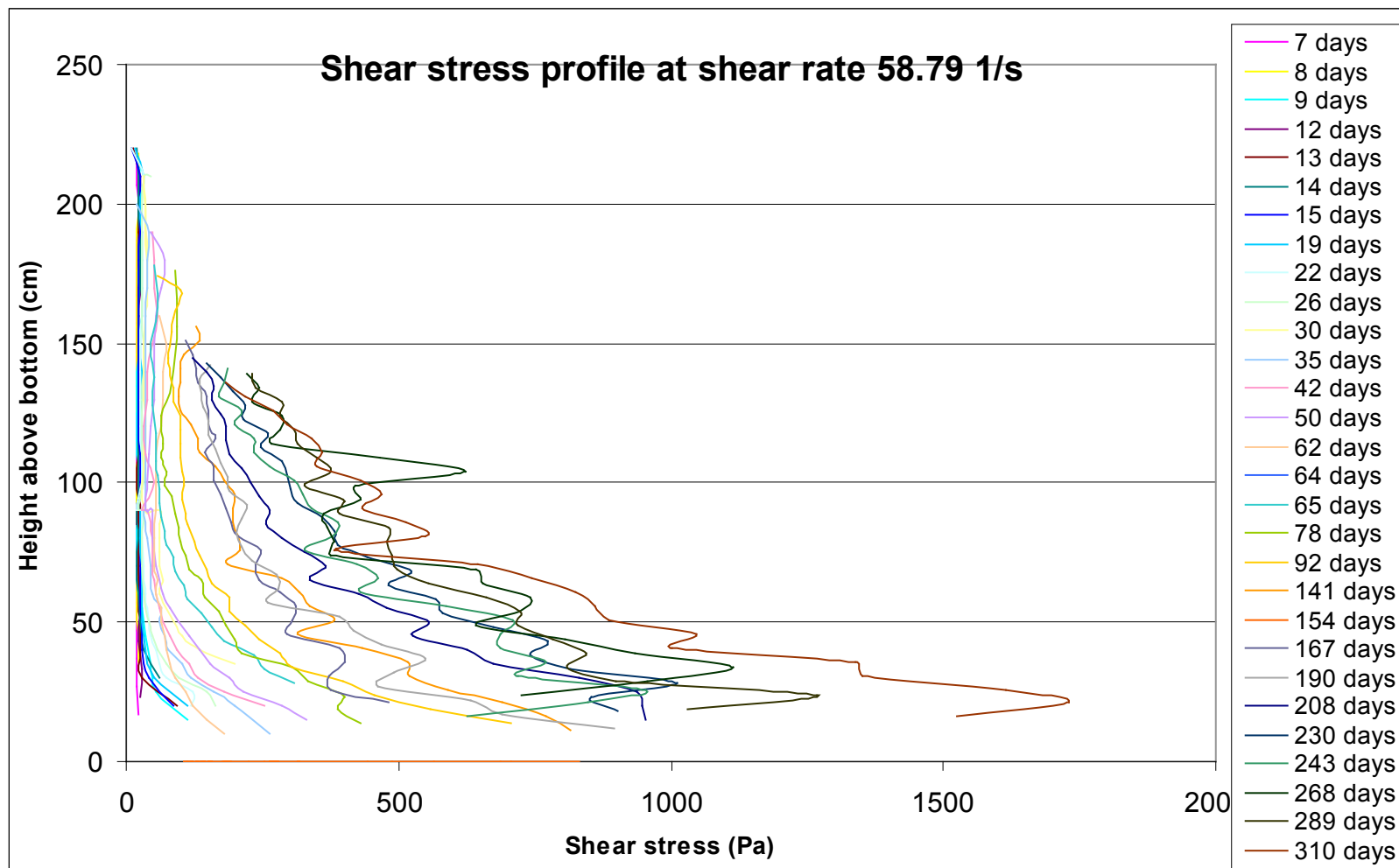
Figure 62: shear stress as function of the height above the bottom for different measuring days at a shear rate of 58.79 1/s

Figure 63: shear stress as function of the height above the bottom for different measuring days at a shear rate of 119.4 1/s
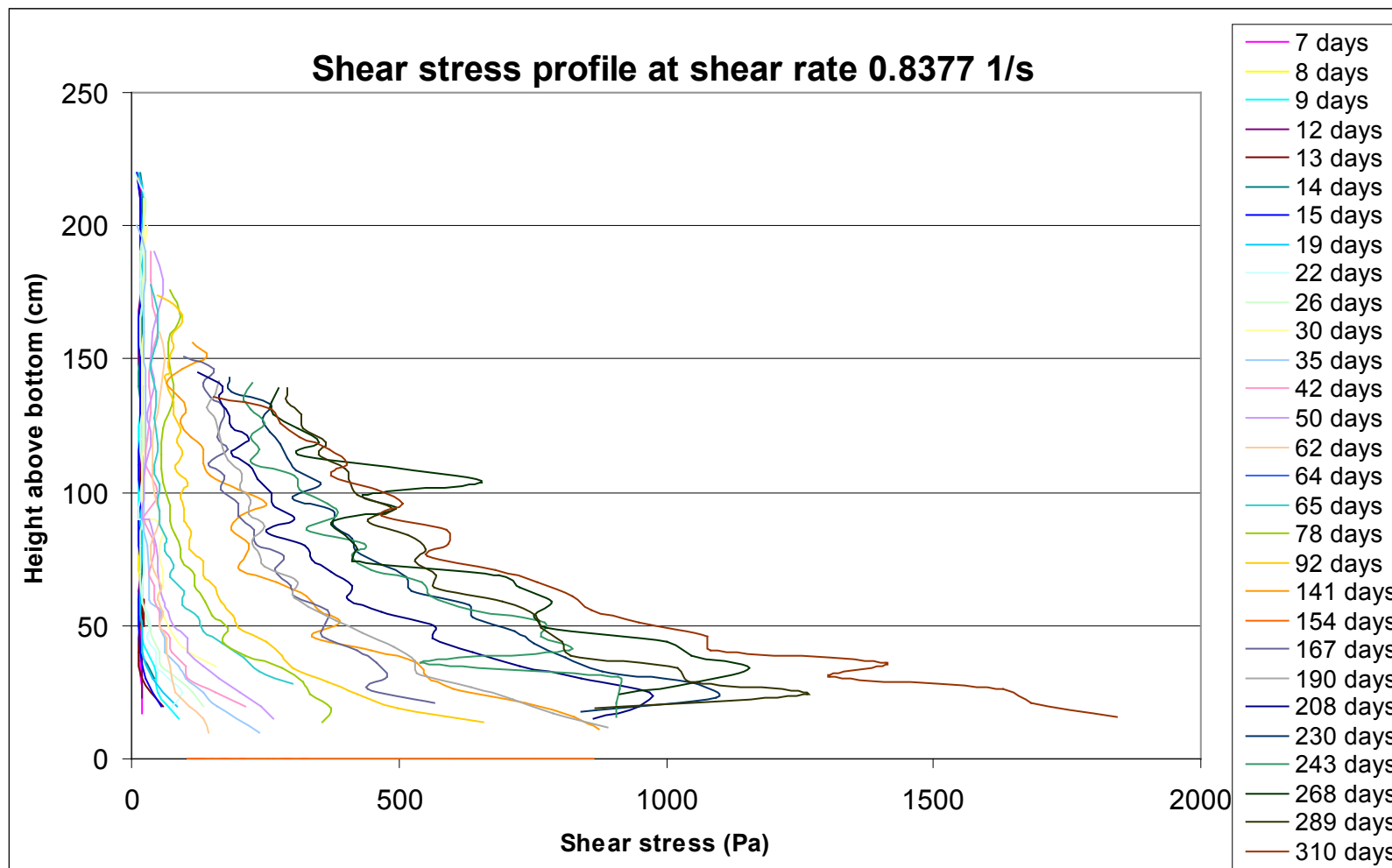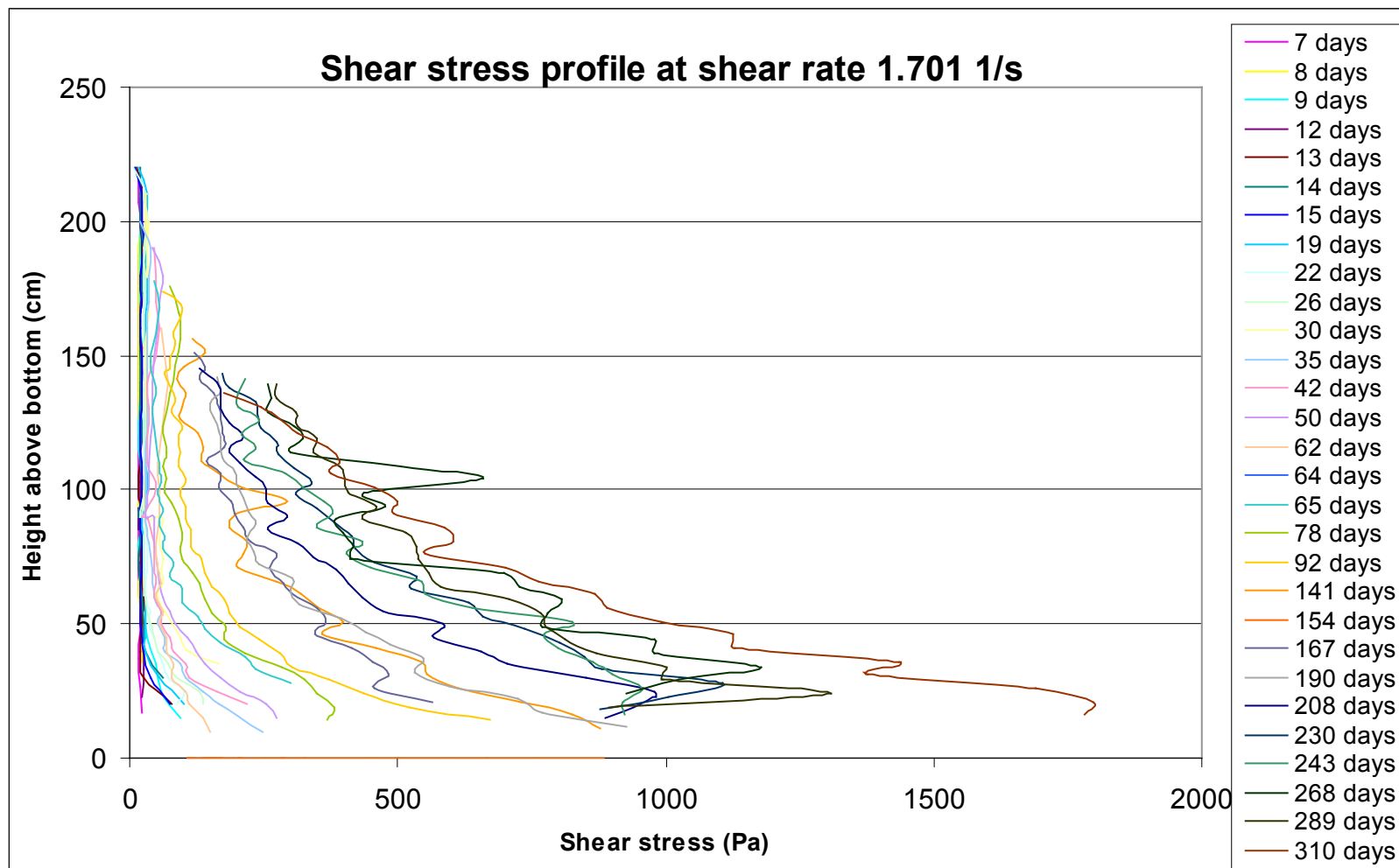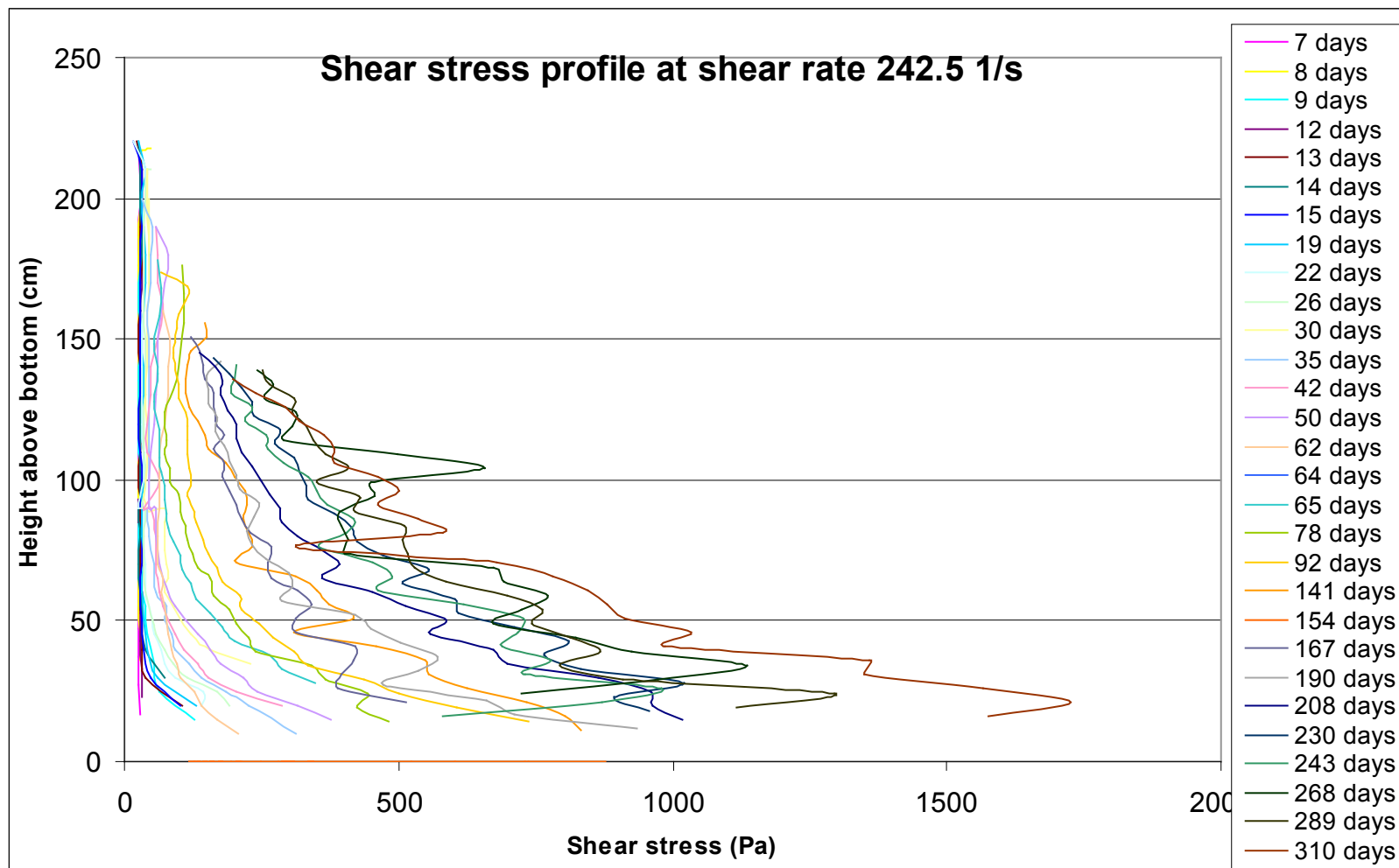
Figure 64: shear stress as function of the height above the bottom for different measuring days at a shear rate of 242.5 1/s
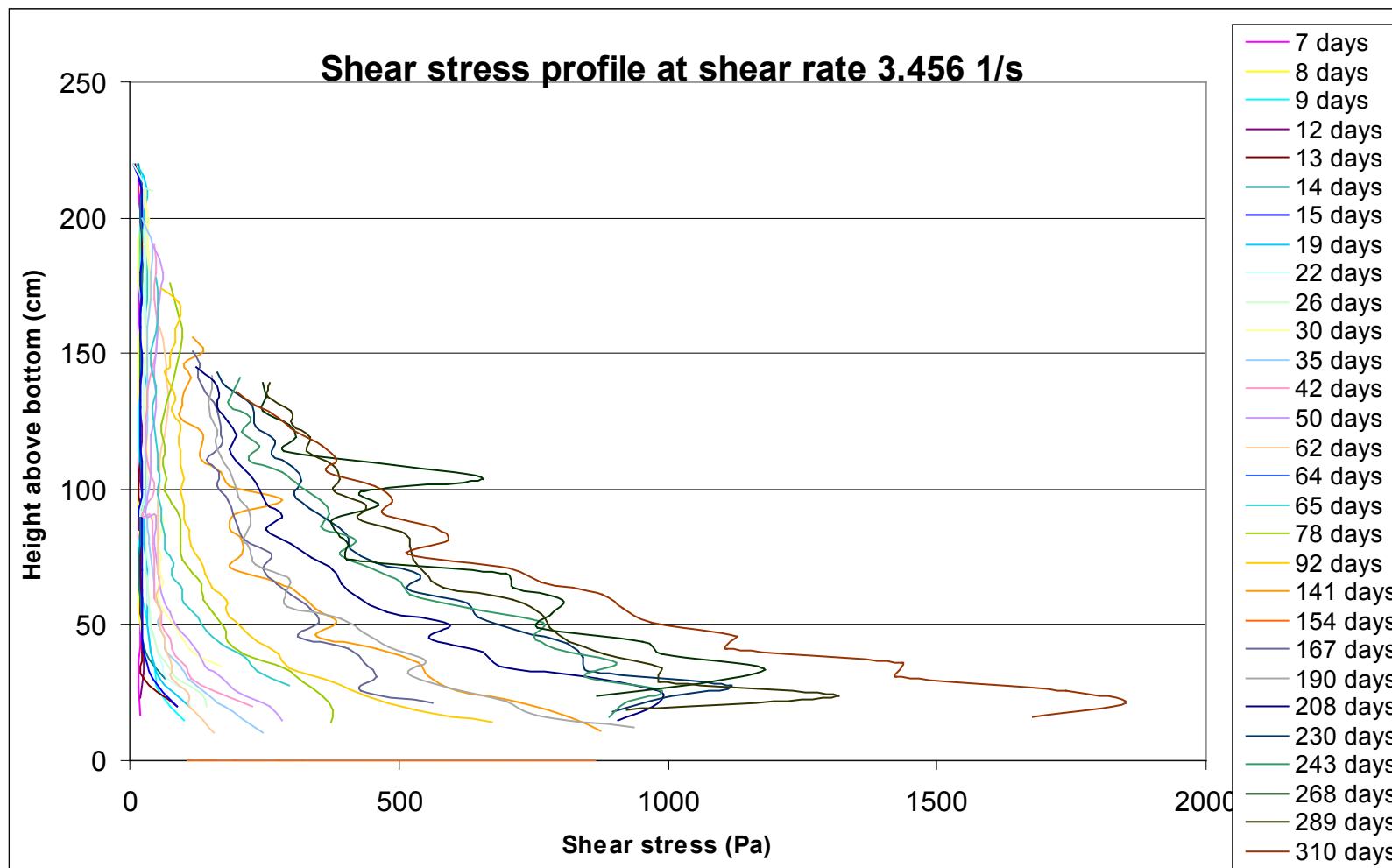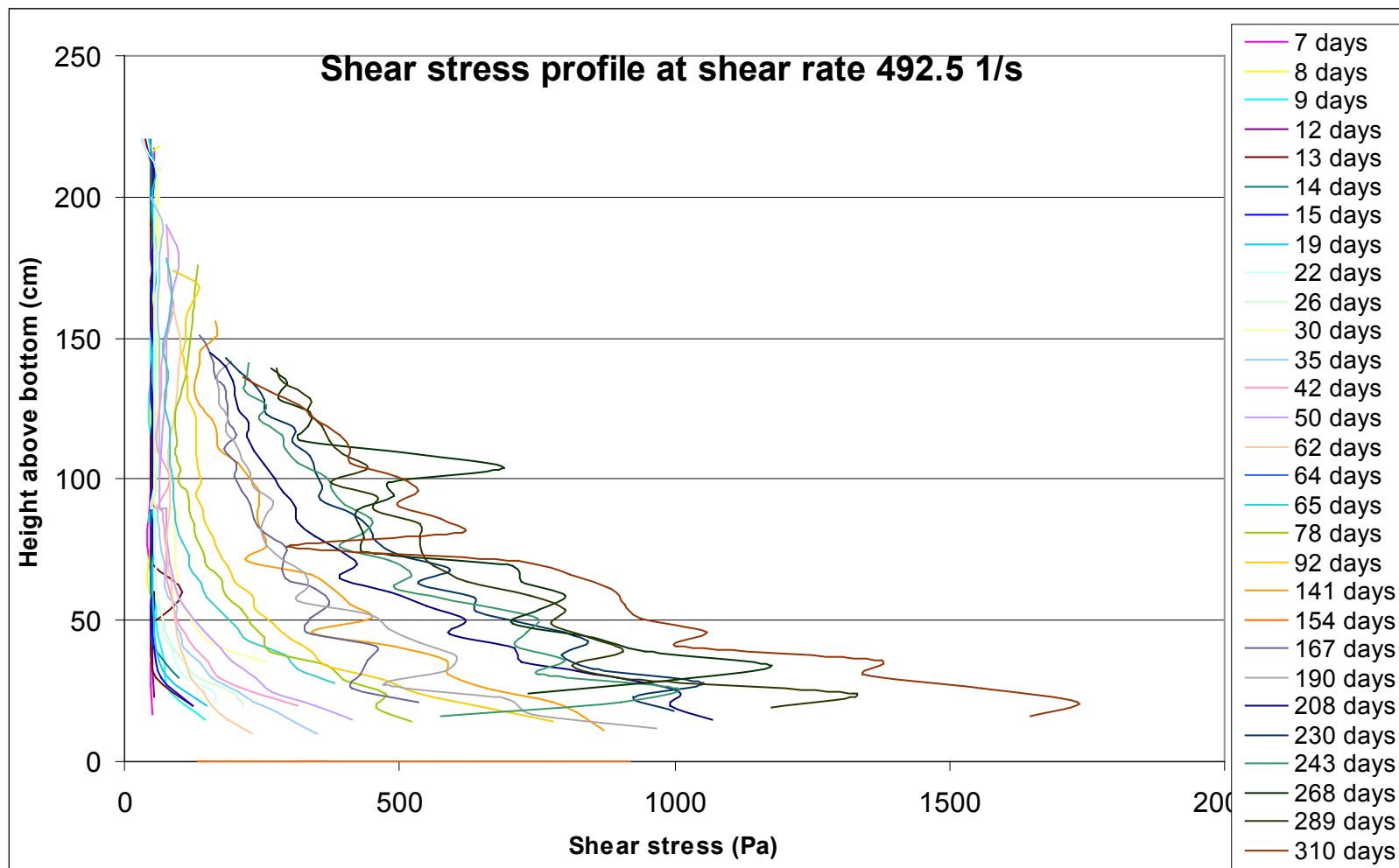
Figure 65: shear stress as function of the height above the bottom for different measuring days at a shear rate of 492. 5 1/s
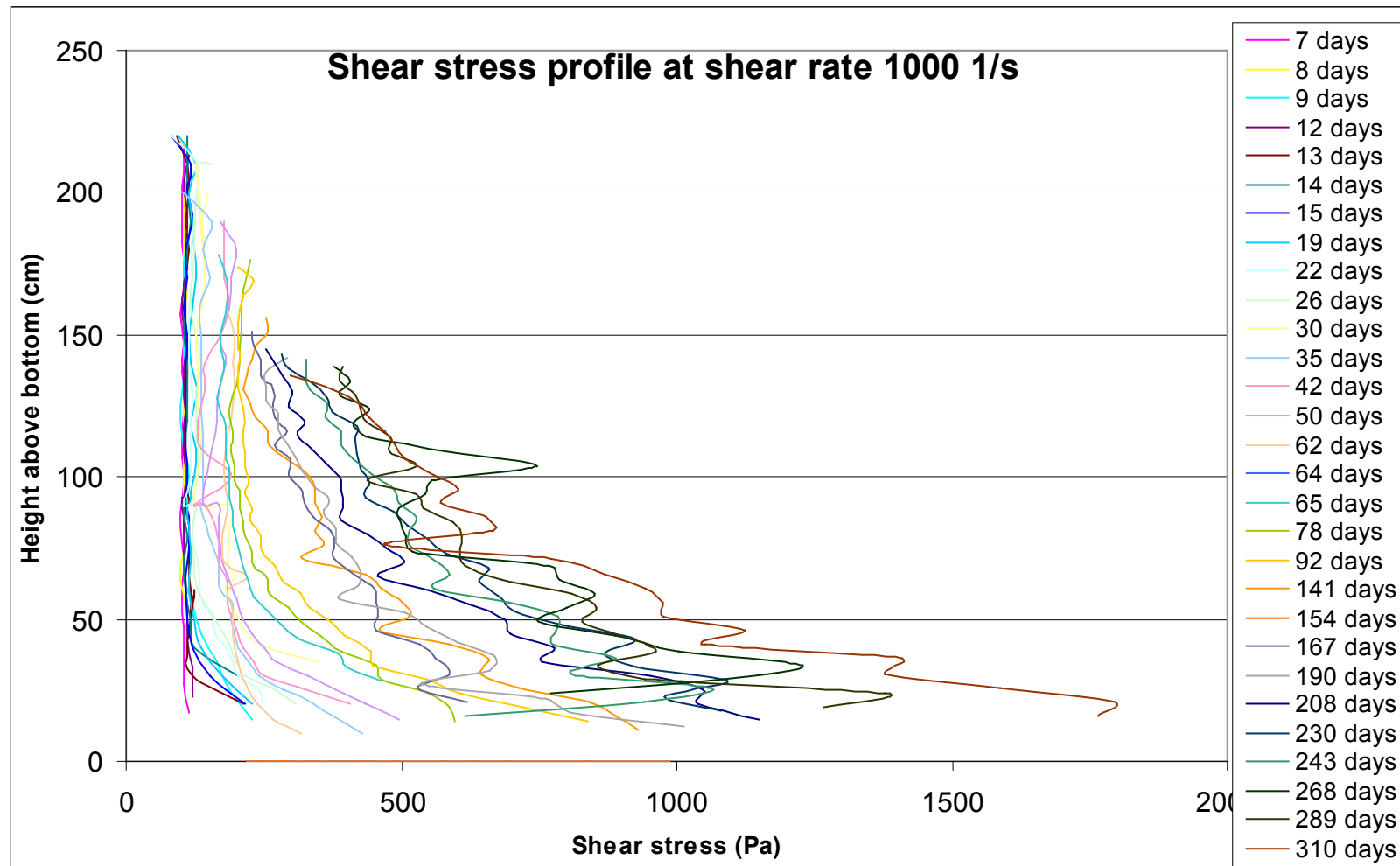
Figure 66: shear stress as function of the height above the bottom for different measuring days at a shear rate of 1000 1/s
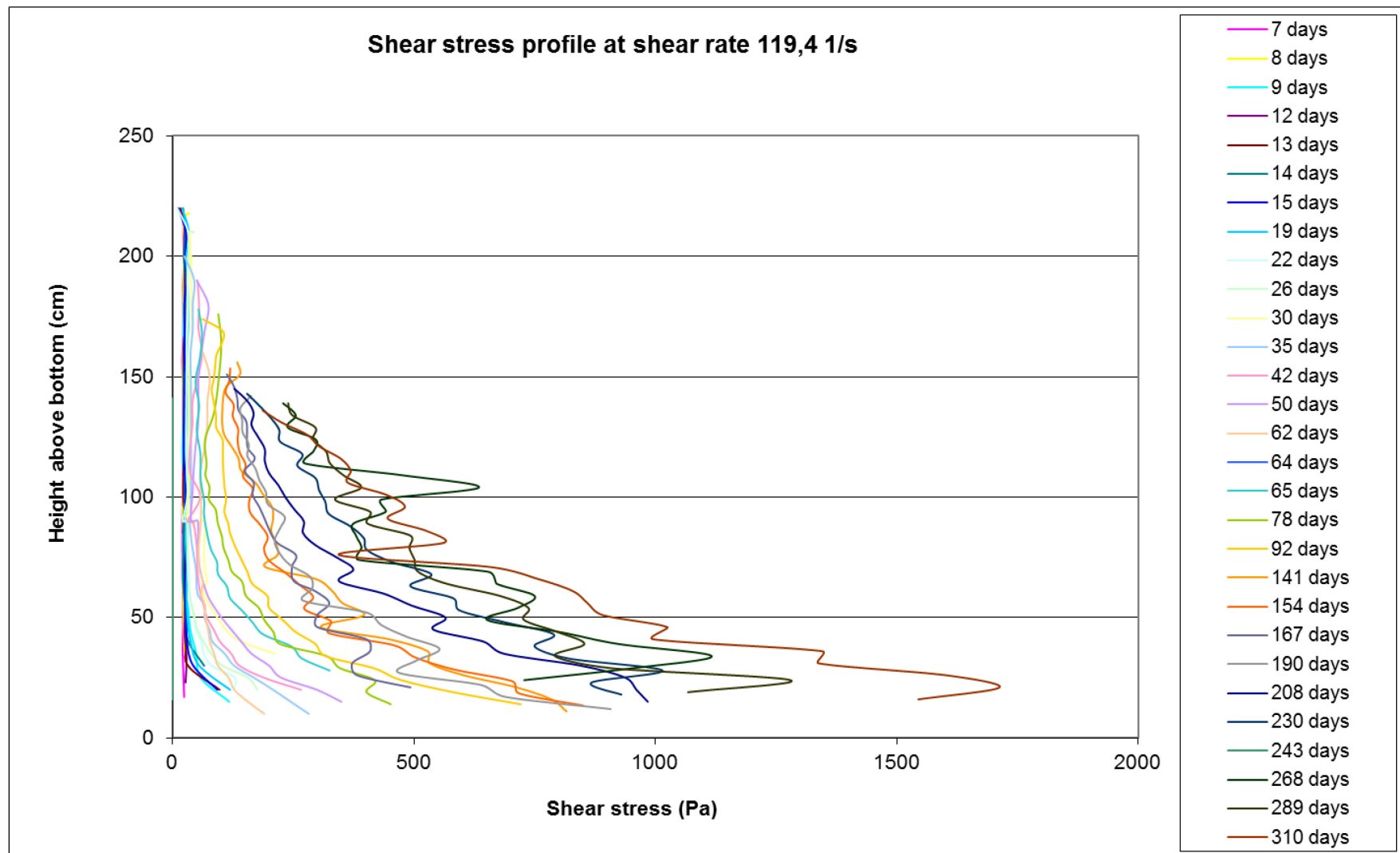
Annex 3: technical sheet of the Particle Size Analyser 'Mastersizer 2000'

Mastersizer 2000

2000

Integrated systems for particle sizing

detailed specification sheets from www.malvern.co.uk/ms2000

MALVERN
INSTRUMENTS

Mastersizer 2000

Comprehensive particle sizing

The Mastersizer 2000 is a practical, reliable solution to the everyday particle sizing needs of industry.

Driven by Standard Operating Procedures (SOPs), the system has set exacting standards in laser diffraction particle sizing.

The Mastersizer 2000 embodies a great deal of expertise and experience. It also comes with the assurance of Malvern's long history in particle sizing and the company's strong culture of customer-focused innovation.

> **The Malvern Mastersizer 2000 is the major workhorse for a range of applications involving suspensions and emulsions systems in our group.**
>
> **Given these diverse requirements it represents a robust solution to particle sizing over a wide dynamic range.** *Richard Williams, Leeds University*

It is a flexible and modular, but fully integrated, particle sizing system with assured measurement performance from submicron to millimetre, wet or dry, from milligram quantities of precious pharmaceuticals to the measurement of bulk chemicals and minerals.

Now proven in diverse applications throughout the world, the Mastersizer 2000 meets even the most stringent measurement and regulatory requirements. Yet it offers such simple, straightforward operation with clear result interpretation that anyone can use it.

2 Mastersizer 2000

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Mastersizer 2000

Advanced technology made simple

An integrated approach

Malvern's systems approach to sample dispersion, instrument control and measurement has resulted in full automation and SOP-driven operation for the Mastersizer 2000, dramatically reducing the need for user intervention.
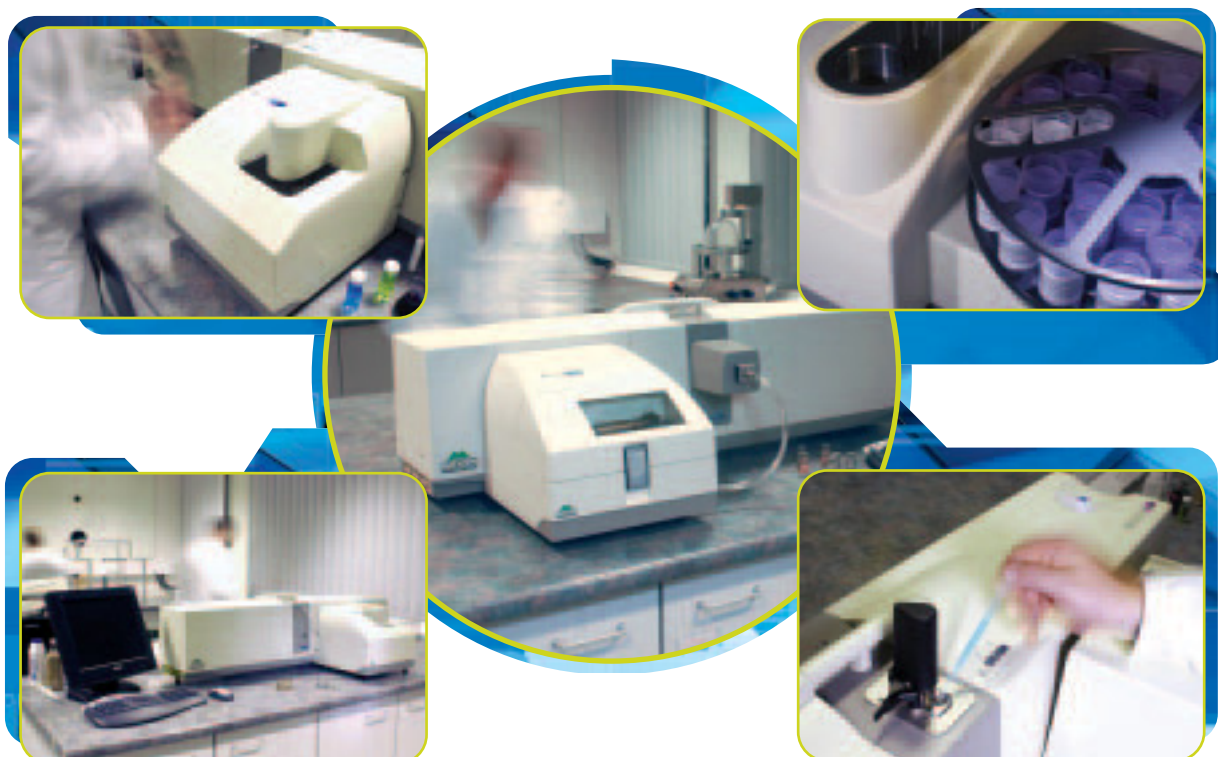
Not only does this remove a major source of measurement variability, it also makes the system especially easy to use – often requiring no more than single button operation.

Software-controlled plug-and-play dispersion units deliver the optimal sample presentation necessary for the production of reliable, comparable, high-quality results time after time.

With such a high degree of standardization, global method transfer and method sharing become both viable and entirely straightforward.

- **SOP-driven for simple, consistent operation and reliable method transfer**
- **Full automation with software-driven control and data management**
- **'Plug-and-play' dispersion units for all sample types**
- **Automated control of dispersion for optimal sample presentation**
- **Wet or dry measurements, with simple changeover between sample types**
- **Wide dynamic range: 0.02μm - 2000μm**
- **Single button operation**
- **Wide application**

3   **Mastersizer** 2000

Mastersizer 2000

Measurements made simple

The Mastersizer 2000 has a friendly user interface – a simple gateway that allows access to all of the system functions and extensive analytical capabilities. The Mastersizer software provides control and management of all size measurement and data management tasks, guiding you at every stage with on-line help and advice.
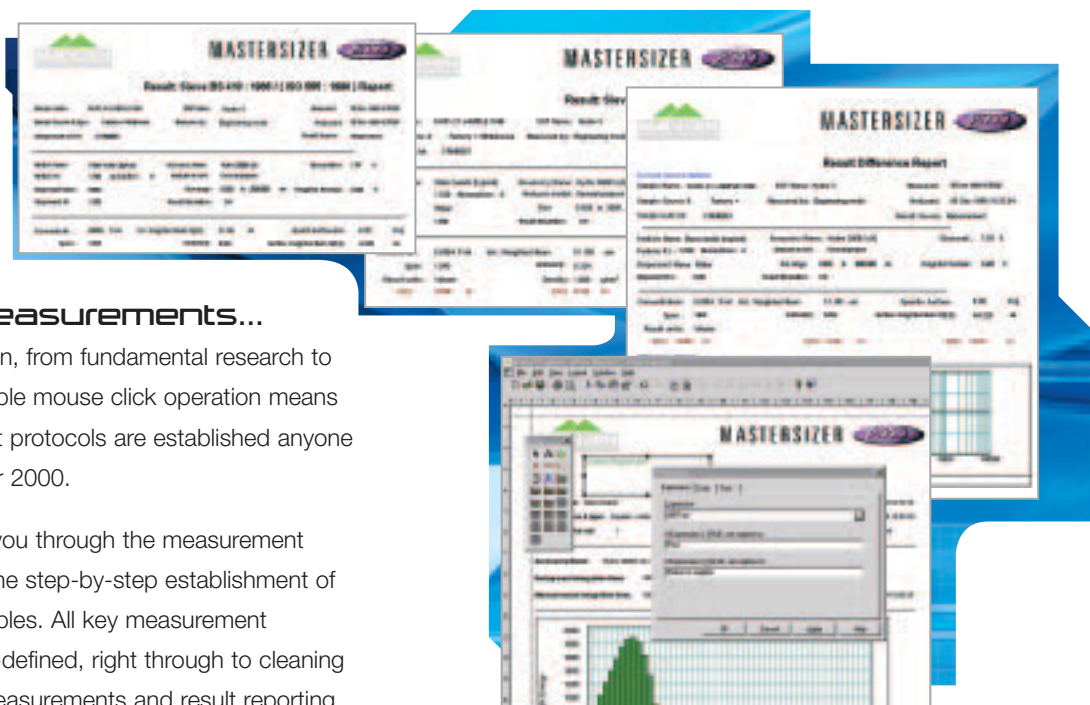
Controlled measurements...

Whatever the application, from fundamental research to production control, simple mouse click operation means that once measurement protocols are established anyone can use the Mastersizer 2000.

Software wizards lead you through the measurement process, starting with the step-by-step establishment of SOP's for different samples. All key measurement parameters can be pre-defined, right through to cleaning procedures, multiple measurements and result reporting. Automated operation ensures everyone can be confident as they deal with a variety of sample types, interchange dispersion units and switch from one type of measurement to another.

For complete security, the software grants different privileges, enabling you to protect measurement procedures or allow specific individuals or groups more comprehensive access to the system.

...with meaningful results

Live displays allow the tracking of sample measurements in real time, allowing you to monitor all aspects of the measurement process. Result reporting is completely configurable, enabling you to define exactly what information is displayed by customizing screen views and printed reports. A wide variety of graphing functions includes trend graphs where outlying values are automatically highlighted. Customizable record pages permit at-a-glance comparison of key particle size parameters, ensuring consistent data management and reporting.

A simple, automated data export function enables full integration of particle sizing data with other analytical information.

4 Mastersizer 2000

Mastersizer 2000

Robust measurements

The versatility and wide dynamic range of the Mastersizer 2000 make it suitable for a diverse range of applications. Configuring the system for individual use is entirely straightforward, as is changing between different measurement types.

SOPs bring definition and consistency to measurements. A simple software wizard guides the user through method definition, allowing the establishment of detailed SOPs for global adoption in minutes. Once established, SOPs can be saved and transferred by email to other Malvern systems.

SOP's can be used to determine:

- **Dispersion unit type**
- **Dispersion settings**
- **Material to be measured, with optical properties**
- **Dispersant, with optical properties**
- **Measurement time**
- **Number of measurements per sample**
- **Calculation type**
- **Operator on-screen instructions**
- **Report format**
- **Labelling protocols**
- **Automation cycles**

Method development made easy

The Mastersizer 2000's SOP function makes method development and transfer straightforward. Multiple SOPs can be developed on the same system and running the samples is reduced to single button operation. Method transferability assists not only large companies operating globally, but also in areas such as the standardization of materials specifications between supplier and customer.

Data representation in your hands

An integrated report designer enables you to customize what you see or print, allowing you the option to focus on critical parameters. Saving and exporting data then allows further analysis as required. A graph zooming facility for on-screen data allows the magnification of specific areas to give precise values and graphs can be cut and pasted into other applications. Special data calculations include Tromp curves, Rosin Rammler fits, Phi plots and tables. The definition of custom control parameters is also possible.

5 Mastersizer 2000

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Mastersizer 2000

Advanced technology made simple

Qualified performance

A total quality approach is central to all Malvern Instruments' operations. The company has ISO9001:2000 with TickIT accreditation and is able to provide full traceability of changes in software and design. Naturally the Mastersizer 2000 also meets the rigorous quality and validation requirements demanded throughout modern industry.

Standardizing measurements

ISO13320-1 is the first formal international standard for particle size analysis by laser diffraction, providing a methodology for proper quality control. The standard offers advice on the expected capabilities and modelling requirements for diffraction systems as well as guidance on how reproducible measurements can be achieved. Within the Mastersizer 2000 system, consistent and controlled measurements are achieved through the clear specification of sample dispersion units and critical operating parameters within SOPs. With the automatic application of full Mie theory, the appropriate optical model is used whatever the size range being measured. This ensures method development and definition to ISO13320-1 guidelines.

Quality assurance standards

Monitoring the performance of any analytical instrument is key to ensuring the production of reproducible results.

The performance of the Mastersizer 2000 optical bench can be verified using standard latices following Malvern's well-established IQ/OQ procedures. This ensures that the optical performance is the same as it was when the system was manufactured. However, successful measurements will also depend on good performance of the sample dispersion units. Verification of the performance of dispersion units requires the use of a well controlled, polydisperse, spherical particle sizing standard. This requirement is fulfilled by the Malvern single-shot Quality Assurance Standards. These are glass bead standards in the size range 10µm to 120µm, providing a sufficiently broad distribution to challenge the capabilities of both wet and dry dispersion units. A sample-to-sample variability of only 0.3% delivers assured measurement reproducibility, enabling the performance of the dispersion units to be verified in a meaningful way.

Mastersizer 2000

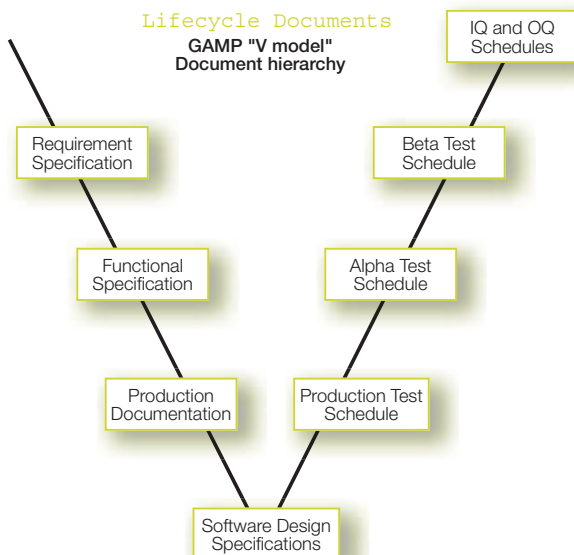## Advanced technology made simple

Validated performance

As a leading supplier to the pharmaceutical industry, Malvern Instruments supports the validation process with its comprehensive QSpec Validation Elements package. This provides users with a set of tools to enable the development of individual system validation plans. Full lifecycle documentation, following GAMP guidelines, provides complete traceability for the design, production and functionality of the Mastersizer 2000 hardware and software. Users can exercise a right-to-view this documentation or otherwise obtain generic questionnaire answers which cover many of the key issues addressed during a GAMP audit. Other elements include IQ/OQ documentation – the basic building block of any validation plan. Support for software updates is also available to ensure comparability of results. Finally, peace of mind is achieved through the introduction of Escrow agreements, ensuring continued support for the Mastersizer 2000 software system, even in the unlikely event of any failure of Malvern Instruments.

QSpec Elements Validation tool kit includes the following elements:

- **IQ/OQ documentation**
- **Software Update Verifications**
- **Escrow contracts**
- **Generic audit questions and answers relating to Malvern Instrument's quality control system**
- **Ability to exercise a right-to-view Malvern's development documentation**
- **User Requirements Specification Matrix**
- **Method development guides**
- **Latex and Glass Bead Standards for Performance Verification**
- **21CFR Part 11 feature key to enable ER/ES support**
- **Validation FAQs**
- **QSpec logbook and validation workbook as a primary record for presentation at a regulatory audit**

Lifecycle Documents
GAMP "V model" Document hierarchy

- Requirement Specification
- Functional Specification
- Production Documentation
- Software Design Specifications
- Production Test Schedule
- Alpha Test Schedule
- Beta Test Schedule
- IQ and OQ Schedules

21 CFR Part 11

21 CFR Part 11 applies safeguards to electronic records and the use of electronic signatures. Key features offered within the Mastersizer 2000 software to aid technical compliance include more rigorous authority checks, safeguards against unauthorized system access and the provision of an audit trail which logs all key instrument and data modification processes. Unauthorized deletion and modification of records can also be prevented, with automatic backups being retained where deletion or modification has been allowed. Electronic signatures are also supported by the use of Adobe Acrobat® PDF Writer.
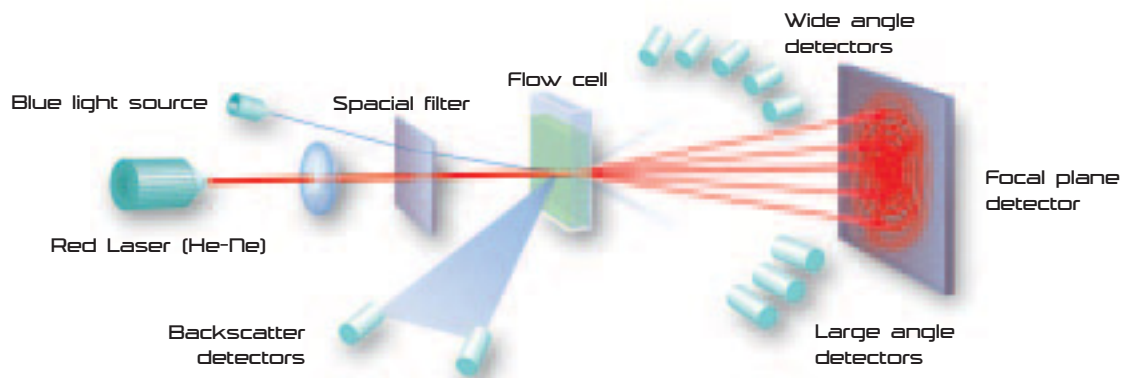
Mastersizer 2000

Advanced technology made simple

Fundamentals of the technology

The Mastersizer 2000's wide dynamic range and flexible operation are achieved through Malvern's capacity to precisely engineer and optimize the system according to the physics of light scattering.

During the laser diffraction measurement, particles are passed through a focused laser beam. These particles scatter light at an angle that is inversely proportional to their size. The angular intensity of the scattered light is then measured by a series of photosensitive detectors. The number and positioning of these detectors in the Mastersizer 2000 has been optimized to achieve maximum resolution across a broad range of sizes.

The map of scattering intensity versus angle is the primary source of information used to calculate the particle size. The scattering of particles is accurately predicted by the Mie scattering model. This model is rigorously applied within the Mastersizer 2000 software, allowing accurate sizing across the widest possible dynamic range.

Dual wavelength measurement
Increased sub-micron resolution is delivered via the Mastersizer 2000 patented dual-wavelength detection system. A short wavelength blue light source is used in conjunction with forward and backscatter detection for enhanced sizing performance. This, combined with red-light measurements, provides superior sensitivity across a wide size range.

Single lens detection
The entire 0.02-2000µm measurement range is accessed using a single-lens system. This rugged configuration ensures that changing between different dispersion units and sample types is immediate and uncomplicated. Any reconfiguration is automatic and software-controlled.

Intelligent Auto Align
Perfect optical alignment is maintained by a unique software-controlled Auto Align system. Alignment is carried out in seconds either as part of an automated measurement or with a single mouse click on screen. This ensures robust measurements, time and again.
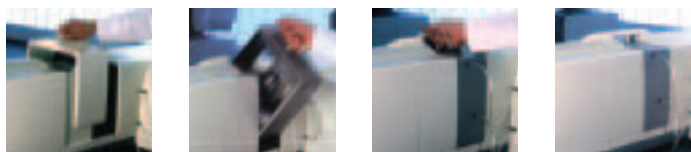
8 **Mastersizer 2000**

Mastersizer 2000

Presenting the sample

Inadequate sample dispersion is a major source of measurement error and one that is shared across all particle sizing techniques.

By making sample dispersion an integral part of the measurement process, the Mastersizer 2000 directly addresses this issue. A wide range of software-controlled sample dispersion units allows the dispersion conditions to be matched to individual applications and samples. Whether you are measuring inert, hazardous or abrasive materials, wet or dry, solvent-based or aqueous dispersions, fragile or robust samples – there is a Mastersizer 2000 dispersion unit designed for the job.

Two or more dispersion units can be connected to the system at any one time. Simple plug-and-play recognition makes changeover almost instantaneous and leaves no room for error. Setting the operating parameters of all units for particular sample types is a straightforward and automatic operation. Using SOPs ensures that any operator bias is minimized.

Sample units can be simply interchanged and set up is automatic

9   Mastersizer 2000

Mastersizer 2000

Advanced technology made simple

Automated operation....

The Mastersizer 2000 Autosampler delivers the ultimate in laboratory productivity and efficiency. This intelligent sample preparation system completely automates laborious sample preparation tasks, enabling true round the clock, unattended operation. This not only increases sample throughput but also frees the user to concentrate on data analysis rather than carrying out routine sample measurements.

Scheduling of autosampler measurements is handled by an integrated database system. This allows the user to view the status of the current batch of samples and change sample measurement priorities to allow urgent sample measurements. The user can search, sort and recall measurement data after measurements are completed. Measurement pass-fail criteria can also be viewed, allowing problem samples to be instantly recognised and selected for further analysis.

With a capacity of up to thirty-six sample pots, the autosampler unit is equipped with an automatic barcode reader that allows it to recognise the contents of each pot. The Mastersizer 2000's powerful SOP system is then used to automatically apply the appropriate measurement protocol, including the addition of any additives required to achieve a stable dispersion. Measurement reproducibility is achieved through the use of a highly effective isokinetic sampling system. This enables the correct sampling of even the broadest particle size distributions without loss of either the coarse or fine fraction. The system also features an auto-concentration facility, ensuring that the sample concentration is always optimal for a perfect size measurement. Dry powders, suspensions and emulsions are all handled equally well.

Autosampler specifications:

- **36 sample capacity**
- **Bar code sample recognition**
- **Robust, iso-kinetic sampling mechanism removes measurement bias**
- **Auto-concentration facility optimizes sample measurement**
- **Precise, automated dosing of up to 2 additives ensure reproducible sample dispersion**
- **Disperses and prepares emulsions and dry powders**
- **Off-line sample scheduling allows pre-preparation of sample trays**
- **Automatic operation via SOPs**
- **Compatible with the Hydro 2000S and Hydro 2000G wet dispersion accessories**

10 **Mastersizer** 2000

Mastersizer 2000

Advanced technology made simple

Mastersizer 2000 technical specifications

Optical Unit	Specification
Size range	Materials in the range 0.02µm to 2000µm
Measurement principle	Mie scattering
Detection systems	Red light: forward scattering, side scattering, back scattering Blue light: wide angle forward and back scattering
Light sources	Red light: helium-neon laser Blue light: solid-state light source
Optical alignment system	Automatic rapid align system with dark field optical reticle
Sample dispersion unit interchange	Sample dispersion units automatically recognized, configured and enabled on insertion of measurement cell cassettes into sizer
Laser system	Mastersizer 2000: Class 1 laser product Autosampler 2000: Class 2 laser product LASER RADIATION DO NOT STARE INTO BEAM CLASS 2 LASER PRODUCT
Software and data processing	
Minimum Computer Specification	IBM compatible PC Pentium 166MHz, 32MByte RAM (64MByte recommended) and CD-ROM. SVGA screen with 800 x 600 resolution, 256 colour. At least 100MByte of free hard disk space is required to operate the software. This specification does not take into account the operating system requirements. Please note:The MS2000 Autosampler requires 128MByte of free hard disk space and a 1024 x 768 screen resolution.
Operating Systems	Windows NT v 4.0 (Service Pack 6A or Higher), Windows 2000 Professional (Service Pack 2 or Higher) or Windows XP Professional. Windows 2000 Professional is the recommended operating system.
Database utility	Searching, sorting and filtering by search criteria of data records on all parameters of interest.
Custom report facility	Custom report designer using drag-and-drop selection, positioning and sizing of key report elements.
Creation of SOPs and automation	Set up by means of SOP Wizard with extensive advice at all stages of SOP creation. A library of SOPs for common materials is built into the software as standard.
Operating modes	Automated using SOPs created in the software. Manual, using on-screen controls and hot keys.

Weights and dimensions

Model	Unpacked weight (kg)	Dimensions (length; depth; height in mm)
Mastersizer 2000 optical bench	31.0	1293 x 255 x 375
Hydro 2000G	13.7	344 x 352 x 330
Hydro 2000S	11.0	352 x 355 x 332
Hydro 2000MU	15.4	320 x 375 x 335/490
Hydro 2000 Micro Precision	12.2	287 x 253 x 338
Scirocco 2000	11.7	352 x 355 x 332
Autosampler 2000	32.0	550 x 365 x 560

11 Mastersizer 2000

Malvern Instruments Limited

Enigma Business Park • Grovewood Road • Malvern • Worcestershire • UK • WR14 1XZ
Tel: +44 (0)1684 892456 • **Fax:** +44 (0)1684 892789

Malvern Instruments Worldwide

Sales and service centers in over 50 countries for details visit www.malvern.co.uk/contact

Mastersizer
2000

Advanced technology made simple

distributor details

Malvern Instruments is part of Spectris plc, the Precision Instrumentation and Controls Company.

spectris

detailed specifications at www.malvern.co.uk

MALVERN
INSTRUMENTS

Annex 4: technical sheet of the Anton Paar DMA 38

DMA 38 Density Meter

▸ Proven Anton Paar oscillating U-tube method
▸ Extremely user-friendly
▸ Rugged design for reliable use

DMA 38 Density Meter

More than 25 years of experience in designing density meters for fluids led to this extremely user-friendly instrument. It is easier to use than any conventional hydrometer and is accurate to 0.001 g/cm³.

Features

Easy to use
▸ Just two keys for all routine operations
▸ No influence of sample viscosity on measuring results
▸ Can be directly connected to automatic filling and sampling systems
▸ Automatic air and water adjustment

Advanced software simplifies your measurements
▸ Operations are display-guided and easy to follow
▸ Density, specific gravity or concentration values, e.g. API gravity, °Brix, % battery acid, etc. can be displayed and printed
▸ Customer specific density/concentration tables can be programmed
▸ °C or °F can be selected

Rugged design for reliable use
▸ All wetted parts are made of borosilicate glass and PTFE
▸ Built-in solid state thermostat
▸ Separate computer and printer interfaces as standard feature
▸ Minimum lab space required
▸ Virtually maintenance-free

Applications
▸ Measurement of true density and density-related values
▸ Concentration determination, e.g. in the food, beverage, photography, pharmaceutical, cosmetic, polymer, chemical and petrochemical industries
▸ General quality control and rapid product identification

Technical specifications

Measuring range	0 to 3 g/cm³
Accuracy	
Density	±0.001 g/cm³
Temperature	±0.3 °C (±0.5 °F)
Repeatability	
Density	±0.0002 g/cm³
Temperature	±0.1 °C (±0.2 °F)
Measuring temperature	+15 to +40 °C (+59 to +104 °F)
Temperature equilibrium time	0.5 to 3.5 minutes
Pressure range	Up to 10 bar (150 psi)
Minimum amount of sample	Approx. 0.7 ml
Measuring time per sample (incl. filling and cleaning)	1 to 8 minutes
Dimensions (L x W x H)	280 x 210 x 270 mm (11.0 x 8.3 x 10.6 inches)
Weight	Approx. 10 kg (22 lbs)
Power supply	AC 85 to 260 V, 48 to 65 Hz
Power consumption	20 VA
Interfaces	Two RS 232 interfaces for external printer and/or computer

Specifications subject to change without notice.

Your distributor:

01/08 A87IP01-D

Annex 5: lab results

LABORATORIUM ECCA NV
chemische en biologische analyses - leefmilieuproblemen

Vlaamse Overheid
Departement Mobiliteit en Openbare Werke
Berchemlei 115
2140 Antwerpen

Definitief rapport

Merelbeke , 09/06/2009

Rapport	: 09-006906

Onze referentie	: 09-006906/01
Datum ontvangst	: 22/04/2009
Matrix	: slib
Bemonstering door	: Klant, afhaling ECCA

Omschrijving	Externe referentie	: Slib
	Aantal recipiënten	: 1
	Staat van het staal	: OK
	Verpakking	: Plastic emmer(s)

09-006906/01

Parameter	Resultaat	Eenheid	Startdatum
Tributyltin	0.024	mg/kg DS	06/05/2009
Sulfaten	11000	mg/kg DS	29/04/2009
Droge stof	21	%	22/04/2009
Sulfide	<0.10	mg/kg	29/04/2009
Nitraat-stikstof	15	mg/kg DS	22/04/2009
Totaal opgeloste stikstof (TON)	110	mg/kg DS	22/04/2009
Kjeldahl-stikstof	1470	mg/kg DS	30/04/2009
Totale stikstof	1590	mg/kg DS	04/05/2009
Arseen (As)	23	mg/kg DS	27/04/2009
Cadmium (Cd)	<0.50	mg/kg DS	27/04/2009
Chroom (Cr)	79	mg/kg DS	27/04/2009
Koper (Cu)	20	mg/kg DS	27/04/2009
Kwik (Hg)	0.22	mg/kg DS	27/04/2009
Lood (Pb)	47	mg/kg DS	27/04/2009
Nikkel (Ni)	27	mg/kg DS	27/04/2009
Zilver (Ag)	<5.0	mg/kg DS	27/04/2009
Zink (Zn)	130	mg/kg DS	27/04/2009
Totaal fosfor	1200	mg/kg DS	27/04/2009
Zwavel (S)	6500	mg/kg DS	27/04/2009

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Ambachtsweg 3 - 9820 Merelbeke | tel. 09 252 64 44 | secretariaat@labecca.be | KBC 442-7013501-43 | BTW BE 0420.054.045
fax 09 252 64 24 | www.labecca.be | bank van Breda 645-5271324-88