

# Preparation system, salt scaling, absorption and internal damage in the CEN/TS 12390-9 concrete frost test

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# TITLE:

Preparation system, salt scaling, absorption and internal damage in the CEN/TS 12390-9 concrete frost test

Prepareringssystem, avskalling, absorpsjon og intern skade i CEN/TS 12390-9 betong fryse/tine-test



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# SUMMARY:

This thesis is a continuation of work with the CEN/TS 12390-9 frost test on fly ash concrete. The goal is to develop a preparation system and incorporate a dilatometry pilot project in order to test several characteristics of the concrete when it is exposed to freeze/thaw conditions, with the possibility of incorporating both tests into one.

The thesis will use two series; Series 1 will test and find the best preparation setup that Series 2 will use in a 56 cycle CEN/TS 12390-9 Frost test. UPV, absorption, and scaled material data was collected during both series. The dilatometry pilot project is monitored in the KylCity freeze/thaw-chamber with Series 2 to examine if it is possible to run the two experiments simultaneously. It also monitored the sample used for internal damages.

Series 1 showed that the best preparation system was a combination of butyl tape with the adhesives SikaFlex 11FC or Casco Marin og Teknikk. The data collected showed that samples not using a rubber material bottom had higher absorption. UPV remained almost the same, whilst the scaling material was close to 1 kg/m<sup>2</sup>.

Data from Series 2 shows that the samples with air-entrainment did considerably better than its counterpart when it comes to scaling, absorption as well as adhesion, water retention and sample deterioration.

Dilatometry did not clearly show any internal damages of the test sample used for 13 cycles, but the experiment showed that it is possible to run dilatometry and a CEN/TS 12390-9 test simultaneously and thus incorporate them into one test in the future.

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# Preface

This thesis is written for the Department of Structural Engineering at the Norwegian University of Science and Technology, NTNU. The thesis is written as a preliminary work, a pilot-project, into applying different materials in the CEN/TS 12390-9 Frost Salt Test using fly ash concrete. It is hoped that using this method will enable testing multiple characteristics of the concrete simultaneously. Characteristics such as absorption, scaling and internal damage through use of ultrasonic pulse velocity.

I would like to thank professor Stefan Jacobsen for his guidance and help alongside his confidence in my ability while working on this thesis. The help has been invaluable and the new insight and details has improved my work and thesis in several ways.

I would also like to thank PhD-candidate at NTNU, Andrei Shpak, whom has been the main discussion partner and laboratory collaborator. He has also aided me in testing the different material preparation setups and by collecting data during my time at the frost laboratory at the department of structural engineering.

Lastly I would thank the staff at the structural engineering laboratory, mainly Ove Loraas and Steinar Seehus. Their assistance has helped me master a lot of the laboratory equipment and given me practical advice on so many issues related to the laboratory work and materials used. This has given me better confidence that I can master many different tasks related to more practical work.

# Summary

This thesis is a continuation of the work previously done within CEN/TS 12390-9 frost testing of fly ash concrete. The work is related to the pursuit of learning more about the impact freezing/thawing has on fly ash concrete and finding more comprehensive, cost-and time efficient ways of testing different characteristics of fly ash concrete under freeze/thaw conditions simultaneously.

This thesis has three main objectives. The first is to develop an alternative preparation system to create CEN/TS 12390-9 qualified samples and test this preparation system on two concrete mixes using fly ash concrete with and without air-entraining admixture. The second objective is to create a dilatometry pilot project which could run simultaneously to a CEN/TS 12390-9 frost test. The third is to incorporate these two test methods into one comprehensive test.

The creation of the different preparation systems, called Series 1, are tested by freezing/thawingcycles calibrated in accordance with the CEN/TS 12390-9 standard. The Series is split into two parts, Material Test 1 and 2. The material tests shows that the preparation system that yields the best results are samples prepared with Sitko Elastic 605 butyl tape and the adhesive SikaFlex 11FC or Casco Marin og Teknikk.

Series 2 used the preparation system found in Series 1 to test the properties of 10 different fly ash concrete samples by subjecting the samples to a CEN/TS 12390-9 frost test. Of the 10 samples five of them are air-entrained and the other five are non-air-entrained.

The dilatometry pilot was split into three sub-experiments. Part 1 and Part 2 focused mainly on finding the best solution to have the invar frames inside the KylCity freeze/thaw-chamber, whilst Part 3 would put a CEN/TS 12390-9 qualified sample through a 13 cycle dilatometry test while simultaneously running Series 2 in the chamber.

Data collected from Material Test 2 in Series 1 shows that almost every sample scaled considerable amounts, and many of the samples went above the acceptable limit in regards to the scaling. The absorption of samples with no bottom attached to the concrete was substantially higher than the rest of the samples, whilst the UPV for all samples were fluctuating, but could not tell if any internal damages had occured.

Data collected from Series 2 shows that non-air-entrained concretes scale substantially more than their counterparts. The data also shows that samples that scale the most also have the largest absorption values. For all samples the mean UPV value was almost unchanged, and on average the UPV of the non-air entrained samples were larger.

The dilatometry pilot project shows that the readings fluctuated with the air temperature in the chamber. Part 1 and 2 show that it is possible to run dilatometry alongside a CEN/TS 12390-9 frost test. Part 3 also showed that the dilation doubles with the inclusion of water on the test surface, whilst the strain/temperature graph could not pick out any clear signs of internal damages in the concrete.

The experiments and the empirical evidence shows that it is possible to run two different experiments relying on the CEN/TS 12390-9 test cycle at the same time. This means that it is also possible to incorporate dilatometry into CEN/TS 12390-9 test testing for several characteristics of concrete under the freeze-thaw test conditions at the same time.

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# 1 Introduction

Testing the sustainability of concrete for scaling damages can be done in several ways. The European Standard, CEN/TS 12390-9, has three different methods. The main one being the *Slab* test, commonly known as a *Boråstest*, which is also the one used in this thesis. The two others are the CDF-test and the cube test [1]. The commonly used standard test method in North America is known as the ASTM C666 method, which emphasizes more on internal cracking, through rapid freezing and thawing.

In a slab test a prism of  $50mm \times 150mm \times 150mm$  is exposed to slow freezing and thawing cycles, one cycle lasting 24 hours, whilst having a 3mm layer of water with 3% sodium chloride on the  $150mm \times 150mm$  test surface. Figure 1 illustrates how the sample is supposed to be encapsuled before a test is performed.

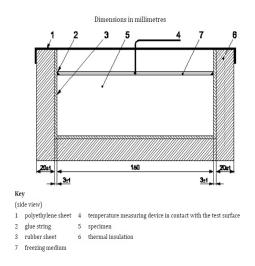


Figure 1: Encapsuled Slab Test Specimen

The main objective of a slab test is to measure the material that is broken off during freezing and thawing, known as scaled material. To do so, the samples are encapsulated as shown in Figure 1. It is hoped that using a combination of adhesives and butyl tape can replace the rubber sheet and the glue string, key points 2 and 3, respectively. The insulation boxes, key point 6, around each sample are made from any standard suited material as thermal insulation and is fastened around the fully prepared sample using moisture proof duct tape.

The thesis will include two main series of freeze/thaw-testing, using fly ash concrete. Series 1 will test adhesives and rubber materials in a freeze/thaw-chamber and in the end come

up with a preferred conifguration to be used in a CEN/TS 12390-9 slab test. The second series, Series 2, will use air-entrained and non-air-entrained fly ash concrete using the preferred setup found in Series 1. Both series will measure absorption, ultrasonic pulse velocity and surface scaling. Furthermore the thesis will include a pilot project monitoring the dilatometry of a sample while it is frozen/thawed. This test will run simultaneously to the other tests, to see if dilatometry can be incorporated into a CEN/TS 12390-9 test as well.

This work is a continuation of previous work done within this field, and its goal is to modify the setup from Figure 1 so several samples and its characteristics can be tested simultaneously. Characteristics such as: surface scaling, internal damages and absorption. Combine this with the incorporation of a dilatometry check of a sample as well and a new CEN/TS 1290-9 test will be extremely comprehensive and can save labour, time and money in the long run.

# 2 Background Theory

This section will include important topics regarding frost durability of concrete; the different damage types-and parameters as well as introduce the importance of absorption in regards to frost damage. The chapter will give a thorough and cohesive understanding of these topics and some theory from previous research done on these subjects in order to understand the mechanisms behind frost durability of fly ash concrete.

# 2.1 Different Frost Damage Types

When a concrete sample is exposed to freezing and thawing, two main damage types are prevalent; internal cracking and surface scaling. Jacobsen and Sellevold [6] found that all four possibilities of cracking and scaling during a freeze/thaw test occured in the samples they tested. When fly ash is included into a concrete mix, it will have a detrimental effect on a concrete's frost durability [2]. This is due to the irregular air void system and the slow property development, yet much research has proven that if air entrained admixtures (AEA) are used, fly ash concrete can be just as, and in som cases more, durable than regular concretes when exposed to freezing and thawing, [4].

# Internal Cracking

Internal cracking happens when permanent internal volume changes, caused by rapid freezing, develop in the concrete. The rapid freezing of the water in the capillary-and regular pores of the concrete will expand the water by  $\approx 9\%$  exerting pressure on the surrounding pore walls. While the internal pressure grows, the dilation of the concrete will eventually become higher than the concrete's tensile strength causing internal rupture of the pores, or cracking,[3].

Fagerlund has suggested that there are different mechanisms at play when it comes to internal cracking depending on whether or not there is moisture movement in the sample, [9]. Although the theories are slightly different he also suggests that the basic mechanisms behind them might be the same. When there is no, to little, moisture movement, there are three main internal cracking destruction mechanisms:

- 1. Hydraulic Pressure
- 2. Closed Container
- 3. Ice-Lens Growth

The closed container model is a special case of the hydraulic pressure model and all three models could be active at the same time. The effect of freezing is as such a result of more than one destruction type, [9].

The three models above is described using what Fagerlund refers to as a *Representative unit cell*, a unit that could represent the entire material as a bulk and they are briefly described below.

## **Closed Container**

The simplest material model, describing the cement paste as the *unit cells* consisting of holespheres with impermeable walls. The centrical hole contains all the water, which builds up pressure when it freezes. The magnitude of the pressure is dependent on the freezing temperature, and the pressure is transferred to the wall causing tensile stresses. The magnitude of these tensile stresses depends on the saturation of the container.

#### Completely Saturated Container

When a sample is completely saturated calculations show that *it is not possible for a concrete to survive*, if no consideration of the compressibility and ductility of the three phases, solid wall, ice, non-freezable water respectively, is made.

#### Unsaturated Container

This option takes into account that no tensile stresses will occur if the degree of saturation is below a critical saturation value. Once the level is reached tensile stresses will occur and induce damage on the pore walls.

### Hydraulic Pressure

Although the closed container is very similar to the hydraulic pressure model the differences are profound in the assumptions made in the destruction model. The hydraulic pressure model has a higher air requirement than the closed container, as the water has to travel from saturated areas to pores that are air-filled, if the moisture can find new air voids to move into. This flow will create a hydraulic pressure which in turn might exceed the tensile strength of the pore walls.

The theory illustrates the importance of air entrainment as shorter air void spacing will give the non-frozen water new pores to occupy without creating excessive pressure. The theory only explains the damages occuring and does not consider environmental factors. For instance it does not take into account that if the pores are small and the permeability of the concrete is increased, the pores will fill in the long term, allowing damages to still occur in spite of a low spacing factor.

#### **Ice-Lens Growth**

Ice-lens growth is a mechanism concerning the energy differences between non-frozen water and ice. The differences in energy will draw water to the bodies of ice in the voids creating larger ice-bodies in ice-filled capillaries. This exerts a greater pressure on the pore walls, which in turn increases the free energy of the ice.

The process will generally stop by itself, once the energy differences are equalized. This happens due to the ice drawing water from the gel-and capillary pores, drying them out and reducing the energy, ending in a final equilibrium between the residuel unfrozen water and the ice.

If there are numerous air pores the transport of water to an ice-body the reduced free energy from the amount of air voids will prevent the mechanism from taking place. This shows that the theory supports that there must be a critical distance between air-pores.

When there is moisture transfer over longer distances during freezing, Fagerlund has also suggested different theories which will not be gone through in detail. Such as: Moving ice front and frost heave.

#### Surface scaling

When the surface of concrete is exposed to water it will gradually deteriorate causing surface scaling. The effect of the scaling gets worse in the presence of chemical compounds that reduces the freezing point of water. Some such materials are: different alcohols, sea water, urea and deicing salts.

Through experiments of non-air-entrained and air-entrained concrete, Verbeck and Klieger found that these compounds would induce the most damage on the concrete if the concentration of the compounds is between 2 and 4%, [10]. They also showed that the scaling increased when the solution was refrozen instead of being replaced, which would more accurately represent the most severe forms of exposure. Furthermore, their results showed that surface scaling will only occur if the concrete surface is continously submerged/wet during freezing and thawing.

Verbeck and Klieger's results shows that surface scaling is not a chemical phenomena, but a physical one, [10], and Farstad and Sellevold implies that the dominant mechanism is based on osmosis, [8]. An osmotic mechanisms builds up pressure due to local concentration differences, moving material/mass from low concentration to high concentration. This means that building pressure requires time, but it can also lead to pressures way above the tensile strength of the concrete, damaging it in the process, [3]. This also means that the surface will experience more damage if the freezing of the specimen occurs slowly,[3].

Another theory proposed by Valenza and Scherer, namely the *glue-spall* theory has shown why increased damage occurs on test samples with "weak" solutions on the test surface during freezing and thawing of the sample. Meaning that although osmotic pressure probably occurs, it is not the main deterioration mechanism on the concrete surface. Glue-spall of the concrete surface occurs when the thermal contraction of ice is a lot higher than that of concrete, breaking apart the surface due to tensile stresses. The reason why the solution should contain a low salt content is because too high would not create stresses, while none at all would just cause the ice to creep. [2], page 15-5. This shows that there are still things to discover regarding the different mechanisms at work when evaluating the deterioration process of concrete during freezing and thawing.

# 2.2 Saturation

The saturation, S, indicates the amount of pores in the concrete that are filled with water. The definition of saturation can therefore be written as:

$$S = \frac{W}{\varepsilon_{tot}} \qquad \left[\frac{m^3}{m^3}\right]$$

Where the different parameters are:

•  $W = V_{water}$ 

•  $\varepsilon_{tot}$  = Total pore volume

It is important to note that the total pore volume also includes the entrained air pores in the concrete, meaning when we have complete saturation *all* pore space in the concrete is filled with water, [7]. Although complete saturation can happen, concrete will experience damage from frost deterioration long before complete saturation takes place. This value of saturation is known as the critical level of saturation,  $S_{cr}$ , and once saturation passes this threshold frost damage will appear.

This means that it is possible to define a freeze/thaw resistance, [7], namely:

$$F = S_{cr} - S_{act} \tag{1}$$

The equation shows that the resistance is dependent on the critical saturation and the actual saturation, with the critical degree being of great importance. The critical saturation value is independent of the amount of freeze-thaw cycles and is considered an *outer climatic condition* and is as such a material characteristic. Figure 2 illustrates this point.

As such,  $S_{cr}$  is a measurement of the concrete's frost durability as damage will only occur when  $S > S_{cr}$ , as shown in Equation 1.

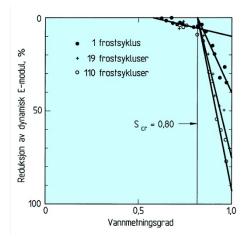


Figure 2: Frost damage as function of S

Frost immunity period is the time until damage occurs, or when  $S_{cr}$  is exceeded. Frost durability in concrete depends both on the Frost immunity period and the  $S_{cr}$ . A frost durable material can normally be in a moist environment for a long time without being damaged by freeze-thaw.

Normally  $S_{cr} \in \langle 0.75, 0.85 \rangle \Rightarrow PF \in \langle 0.15, 0.25 \rangle$ , [3] part 4.5, for most building materials. The PF or pore protection factor is a measurement of frost duarbility based on the amount of filled pores within a concrete.

# 2.3 Other important frost durability parameters

To create a frost durable concrete there are some other characteristics of the concrete used that is important. These are the *Pore protection factor*, PF, and the *Air void spacing factor*,  $\overline{L}$ .

#### The Pore protection factor, PF

PF is a measurement used to determine whether or not a concrete can be defined as frost durable. It is known as the amount of pores that are not filled with water. I.e, the pores that are not saturated, as mentioned above. PF can be defined as:

$$PF = \frac{A}{\varepsilon_{tot}} = \frac{A}{(A + \varepsilon_{suc})} = \frac{\varepsilon_{air}}{\varepsilon_{air} + \varepsilon_{suc}} \quad (2)$$

Equation 2 defines PF as a material parameter and indicates that a higher PF gives better frost durability, shown by figure 3. This is valid

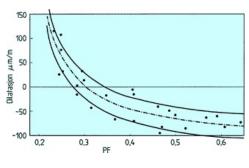


Figure 3: Influence of PF

as long as the suction porosity  $\varepsilon_{suc}$  is held accountable for all the water in the pores. Using the equation it is now possible to find values for the PF. Common values for good frost durability in regards to PF is:

$$PF = \begin{cases} \ge 0.20 & \text{Water without salt} \\ \ge 0.25 & \text{Water with salt} \end{cases}$$
[2]

# Air void spacing factor, $\overline{L}$

The air void spacing factor is an important parameter for frost durability. It is a measure of the half-distance between each air void and it measures how long the water front has to travel to enter a new pore, relieving pressure on the pore system. It can be expressed as:

$$\overline{L} = \frac{V_{material}}{\alpha \times A} \quad [m] \quad (3)$$

The goal is that  $\overline{L}$  should be as small as possible whilst the specific surface,  $\alpha \ [mm^{-1}]$ , should be as large as possible in order to secure good frost durability.

Combine this with a good air content and the result is a well proportioned pore system. A spacing factor of  $\overline{L} \leq 0,250mm$  is needed for good frost durability for fly ash concrete, [4]. The importance of the spacing factor is also illustrated by Figure 4.

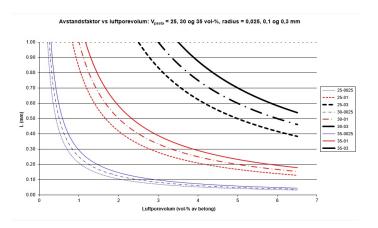


Figure 4: Influence of Spacing Factor

# 3 Methods and Materials

# 3.1 CEN/TS 12390-9 Salt Frost Scaling Test

The CEN/TS 12390-9 test method, also referred to as the *Slab Test* or *Borås Test*, is the common test procedure to determine the frost durability of concrete in Europe. A concrete sample, with salinated water on the test surface, is exposed to 56 cycles of freezing and thawing to determine its surface scaling, [1].

To produce samples of the quality required to perform a standardized test, they will have to follow a strict schedule for when they are cut, when external material is fastened and for when and how long it should be in different environments or receive another kind of preparation. The schedule, based on the CEN/TS 12390-9 standard,[1], shown in Table 1 below illustrates the entire process each sample must undergo before a test begins.

Day	Procedure	
First 24 hours	Samples are stored in their moulds and protected from drying	
	using a polyethylene sheet.	
1 day $\pm 2$ hours	Samples are removed from their mould and placed in bath of tap	
	water at $20 \pm 2^{\circ}C$ .	
7	Samples are placed in climate chamber <sup>1</sup> for storage	
	until test start.	
$21 \pm 1 \text{ day}$	Specimens are $\operatorname{cut}^2$ and returned to the climate chamber	
	after rinsing and measuring.	
$25 \pm 1 \text{ day}$	Attaching the different materials around the concrete sample	
	before returning it to the climate chamber.	
28	A 3mm deep water level is poured onto the test surface to saturate it.	
31	Replace the water with a 3mm layer of water with $3\%$ NaCl and start the test	

<sup>2</sup>The specimens are cut perpendicular to the top surface in  $50 \pm 2mm$  thick samples.

Table 1: CEN/TS 12390-9 Sample Preparation Procedure

#### **Evaluation of concrete durability**

The durability of the concrete is decided based on the amount of material scaling from the test surface of the sample. Scaling material is collected during the 56 cycle period after 7, 14, 28, 42 and 56 cycles/days, then dried in a dryig chamber at  $110^{\circ}C$  before it is measured using a scale with an accuracy of 0,05 grams. The scaled mass is then calculated using the following formula:

$$S_n = \frac{m_{s,n}}{A} \times 10^3 \qquad [kg/m^2] \tag{4}$$

The collected material from the different days are added together for a final accumulated scaling value for each sample. From these accumulated values the mean of the whole series is calculated which assigns the concrete into a frost durability rating system. These ratings range from *Not acceptable* to *Very good*.

Frost Durability Rating, [3]		
Rating	Description	
Very Good	No sample with $S_n \leq 0, 1kg/m^2$	
Good	Good Mean $< 0, 5kg/m^2$ ; less scaled material last 28 cycles than first 28	
Acceptable	Mean $< 1,0kg/m^2$ ; less scaled material last 28 cycles than first 28	
Not Acceptable	Concrete does not satisfy the <i>Acceptable</i> criteria	

Table 2: The Durability Rating of Concrete exposed to freeze/thaw

# 3.2 Calibration of the freeze/thaw-cycle

Before any materials can be tested prior to a proper CEN/TS 12390-9 test, a calibration of the freeze/thaw-cycle in the KylCity freeze/thaw chamber is performed. This is to ensure that the temperature of the samples are within the limits of a proper cycle during the entire test period, which is crucial for the credibility of a large scale test.

The chamber has 6 shelves and is fitted with five thermocouples to measure temperatures at different places inside the chamber. Three of them are in the air to accurately read the temperature at any given time. Both concrete samples receive a thermocouple to measure the temperature on the test surface which is submerged in salinated water. While the freeze/thaw-chamber runs the temperatures are logged using the program CatmanEASY, set to read a value at every 50th second. This will give enough datapoints to see if the temperatures are within the boundaries.

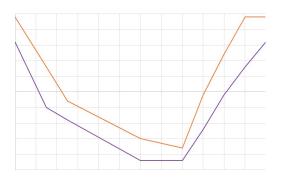


Figure 5: Background used in CatmanEASY

To simplify the evaluation of the graph appearing in CatmanEASY, a background picture of the boundary conditions, illustrated by Figure 5, of the CEN/TS 12390-9 test is in place. This will help identify eventual problem areas in the tested cycle.

During the calibration the chamber has 14 dummies and the two thermocoupled samples are given a 3mm sodium chloride water layer. The chamber itself has six shelves, where five have concrete on them. The samples were originally placed on the 2nd and 4th shelf, but this was changed to shelf one and five for cycle two,

three and four. This was to see how the temperature would look during the *extreme situation*. The bottom, 6th, shelf is used for the additional salinated water to imitate that there are 16 samples in the chamber. Total amount of water in the chamber is thus:

### $Water_{tot} = 16 \times 67ml = 1072ml$

The two samples in use have previously been CEN/TS 12390-9 tested for almost 3 cycles, but have been prepared anew, with a fresh layer of butyl tape, a *Sitko Elastic 605* produced by Tectis, as well as an adhesive. The samples were prepared by brushing the sides with a stiff haired steel brush to cleanse the surface. It was also cleaned using water on every side to remove any debris and other particles that might obstruct the glue. The adhesives used were *Sikaflex 11FC* and *Casco Marin og Teknik lim og fug.* 

The chamber will run for four individual cycles as well as one four day uninterrupted test. The first tests are to find the proper cycle that will be used, while the uninterrupted test is to see the progression of the samples in the long run. This is to ensure that the samples will always perform within the boundaires of the CEN/TS 12390-9 test.

# 3.3 Series 1 - Rubber material-and adhesive testing

To determine which application material preparation system that is most suitable for the full scale CEN/TS 12390-9 test, sample preparation tests in the freeze/thaw-chamber will be conducted. The use of different rubber materials-and adhesives have not been widely examined and it is therefore necessary to experiment in order to find good solutions. The cubes used to test the different materials and adhesives were cast on the 31st of October 2016, at Norbetong's factory in Fagervika by their factory workers. More information regarding the concrete used and its constituents can be found in Chapter 3.5 and in Appendix A.

# 3.3.1 Rubber Materials

The three rubber materials being used as a replacement for key point 3 in Figure 1 in the first round of testing are:

- Sitko Elastic 605 Butyl Tape
- 1mm thick Rubber Band
- 3mm thick EPDM

#### Sitko Elastic 605

Sitko Elastic 605 is a 10m long and 80mm wide butyl tape produced by Tectis. The butyl tape is self-vulcanizing and can be used on most surfaces, given that the surface is clean. To achieve this, the company recommends using a special butyl primer to prepare the material for the tape. The tape has an operational/flexibility temperature ranging from  $-30^{\circ}C$  to  $+80^{\circ}C$  and a thermal decomposition temperature of above  $+200^{\circ}C$ .

The tape itself is not very dangerous, but extremely sticky. It should be kept in a dry room free of dust and oil. There is no restrictions on touching the tape, but it should be kept away from oxidants and alkalies, as the tape is reactant to it, especially to oxidants. The tape will not rot and should be treated as industrial waste.

#### 1mm Rubber Band

The 1mm rubber band is a nitrile rubber bought from *Gummi-* Maskinteknikk. The band itself is 7mm high and 1 mm thick and was specially cut at the company's factory. The nitrile rubber has many different characteristics and can whithstand temperatures as low as  $-50^{\circ}C$ . The nitrile rubber used in this thesis has a temperature span of -50 to  $+120^{\circ}C$ .

## 3mm EPDM

The 3mm EPDM is an elastomer laminate sheet from Trelleborg. It is versatile and can sustain exposure to many different kinds of environments and temperatures. The range is typically from  $-45^{\circ}C$  to  $+80^{\circ}C$  and it remains flexible even during constant temperature swings. The material breaks down slowly and it is resistant against UV, ozone, saltpetre and varying differing weather conditions.

For more information regarding both the technical specifications and safety measures of the different equipment see Appendix E.

# 3.3.2 Adhesives

The three adhesives used as a connector, replacing key point 2 from Figure ?? between the different materials and the concrete surface for the material testing were:

- SikaFlex 11FC
- Casco Marin og Teknikk Lim og Fug
- Casco XtremFix

#### SikaFlex 11FC

This adhesive, produced by Sika, is an adhesive composed of polyurethane. It is quite viscous and decent to work with. It has a temperature range of  $-40^{\circ}C$  to  $+80^{\circ}C$  which is well below the working temperatures of the freezing/thawing chamber. It is considered an elastic joint sealant and multipurpose adhesive. It is flexible and elastic, has high mechanical resistance and it has good adhesiveness to most building materials.

# Casco Marin og Teknikk Lim og Fug

Marin and Teknikk Fugemasse is a sealant produced by Casco. It is *wetter* than the SikaFlex, and it is less viscous and easier to apply and work with. The adhesive has an operating temperature ranging from  $-40^{\circ}C$  to  $+ 90^{\circ}C$ . The adhesive is polyure than and silicone based, combining the best of the two materials to give a glue capable to work both in-and outdoors. The adhesive is very elastic and it dries fast, although it is recommended that there is at least 30% humidity in the air for the adhesive to work properly.



Casco XtremFix is a sealant adhesive produced to endure and tackle difficult challenges. The glue itself is extremely sticky and that which is glued together stays together immediatly. It is very strong with the possibility to carry up to  $200kg/m^2$ . The adhesive has many qualities, such as: frost durability, good applicational abilities, good adhesiveness towards most surfaces as well as its strength when used on both horizontal and vertical surfaces.

Further information regarding the technical details and the safety details of each product can be found in Appendix E. It is recommended to read through the material safety data sheets before using any of these adhesives to prevent any injuries and/-or allergic reactions.



Figure 6: Adhesives

# 3.3.3 Modified CEN/TS-12390-9 Test 1

The first modified test, from here on out, referred to as Material Test 1, is used as a pilot-project to see the impact different adhesives and rubber encapsulations, key point 2 and 3 from Figure 1, will have on a CEN/TS 12390-9 salt frost scaling slab test. This test will form the basis of what preparation system that could replace the current setup.

#### Initial preparation system setup

In order to find the best preparation system for a proper CEN/TS 12390-9 test it is important to have a varied pool of samples. The table below illustrates the different preparation setups that are possible with the eligble materials and adhesives obtained from Chapter 3.3.1. It also includes the last stages of the conditioning of the samples which are mentioned more in detail below.

Sample	Material	Adhesive	Conditioning
1	Sitko Elastic 605	None (reference)	4 day water saturation + prepared sides
2	Sitko Elastic 605	SikaFlex 11FC	4 day water saturation + prepared sides
3	Sitko Elastic 605	Casco Marin og Teknikk	4 day water saturation $+$ prepared sides
4	Sitko Elastic 605	Casco XtremFix	4 day water saturation $+$ prepared sides
5	1mm Rubber Band	SikaFlex 11FC	4 day water saturation $+$ prepared sides
6	1mm Rubber Band	Casco Marin og Teknikk	4 day water saturation $+$ prepared sides
7	1mm Rubber Band	Casco XtremFix	4 day water saturation $+$ prepared sides
8	3mm EPDM	SikaFlex 11FC	4 day water saturation + prepared sides
9	3mm EPDM	Casco Marin og Teknikk	4 day water saturation $+$ prepared sides
10	3mm EPDM	Casco XtremFix	4 day water saturation $+$ prepared sides

Table 3: Material Test 1 Initial System Setup Table

As seen from Table 3, there are nine test samples and one reference. The reference sample was cast in 2015 and will serve as a guideline and comparison between the different preparation systems used to assess the viability, if any, of the different samples and their preparation composition. Furthermore, the rubber materials and adhesives are spread out evenly, with three samples for each rubber material and one adhesive to each material. This will highlight the different strengths and weaknesses of the adhesives as the rubber materials are very different from each other.

# Preparation and conditioning of samples

To give every setup an equal chance of success, all  $9\ 100 \times 100 \times 100 \text{mm}^3$  cubes will be prepared in the same way, from drying to application of the materials, to ensure that the results are legitimate. The nine cubes available were dried for 5 days at  $120 \pm 2h$  in a heating chamber with an approximate temperature of  $55^{\circ}C$ . Normally a climate room would be used to dry the samples, since drying in a heating chamber would cause extra scaling in a test, but since the scaling is not of main interest, rather the suitability of the different materials and their setup, the results should still be credible. The samples were then put in a climate room for two days. The room has a relative humidity of 65% and an evaporation rate of  $40g/(m^2h)$ , which is well within the regulations of the standard. The 9  $100 \times 100 \times 100 mm^3$  samples were cut on in accordance with the CEN/TS 12390-9 standard, creating 18 samples. 9 would be used in Material Test 1 while the remaining 9 will be used in Material Test 2, see Chapter 3.3.4. The samples were cleaned afterwards to remove any debris and then returned to the climate room for four days. They were placed with the cut surface vertically with spacing between each sample.

Afterwards they were prepared on each side, except for the top-and bottom surface, using a brush with steel bristles to remove the laitence and form oil on the outer concrete layer. Each side was brushed for 30 seconds for equal consistency.

When all the samples had been prepared in similar fashion the adhesive was applied to one side of the concrete sample. The material was then fastened and the process was copied for each surface. A wooden spatula was used to spread the glue as evenly as possible in order to create the best possible surfaces to attach the different materials. After the different materials had been applied and fastened, the insulation capsules were created for the different samples. The entire sample preparation process lasted for 4 days. For a small tutorial on how to fasten the samples see Appendix B

The samples returned to the climate room were they were water saturated before the beginning of the test. Each test surface received  $\approx 30ml$  of water as this would create a 3mm water surface layer. The samples went through four days of saturation instead of the required three days to ensure that the samples would have saturated properly before test start.

Based on the initial material preparation system table, 3 and the different material constituents and properties from chapter 3.5, it is possible to establish a scientific name for each sample in the test. The sample number from Table 3 is the same as in the table below to help readability.

Scientific Names, Material Test 1			
Sample	Material	Adhesive	Scientific Name
1	Sitko Elastic 605	None	Reference Sample
2	Sitko Elastic 605	SikaFlex 11FC	0.40-FA35-N311016-10x10 ButSika
3	Sitko Elastic 605	Casco Marin og Teknikk	$0.40\ensuremath{\text{-FA35-N311016-10x10}}$ ButMarTek
4	Sitko Elastic 605	Casco XtremFix	$0.40\ensuremath{\text{-FA35-N311016-10x10}}$ ButXtrem
5	1mm Rubber Band	SikaFlex 11FC	$0.40\mbox{-}{\rm FA35\mbox{-}N311016\mbox{-}10x10}$ RubBandSika
6	1mm Rubber Band	Casco Marin og Teknikk	$0.40 \hbox{-} {\rm FA35} \hbox{-} {\rm N311016} \hbox{-} 10 {\rm x10} \ {\rm RubBandMarTek}$
7	1mm Rubber Band	Casco XtremFix	$0.40\mbox{-}FA35\mbox{-}N311016\mbox{-}10x10\mbox{-}RubBandXtrem$
8	3mm EPDM	SikaFlex 11FC	0.40-FA35-N311016-10x10 EPDMSika
9	3mm EPDM	Casco Marin og Teknikk	0.40-FA35-N311016-10x10 EPDMMarTek
10	3mm EPDM	Casco XtremFix	$0.40\text{-}\mathrm{FA35}\text{-}\mathrm{N311016}\text{-}\mathrm{10x10}\ \mathrm{EPDMXtrem}$

Table 4: Material Test 1 Scientific Name Table

From table 4 it is possible, due to consistency, to refer to each sample by using either its scientific name, or the sample number when examining the results of Material Test 1.

# 3.3.4 Modified CEN/TS-12390-9 Test 2

The second material test, referred to as Material Test 2 from here on, is developed based on the results and empirical evidence collected from Material Test 1. The results of both material tests can be found in Chapter 4.4. This test will use 13 samples, with all 13 using the Sitko Elastic 605 butyl tape. All of these samples come from the same concrete batch cast on the 31st of October in 2016 by the workers at the Norbetong factory in Fagervika, see Chapter 3.5.

The samples will be of different sizes in this material test:

- 2 old 150 by 150 samples that have previously gone 11 cycles in the chamber
- 2 new 150 by 150 samples
- 9 new 100 by 100 samples

Material test 2 will continue for as many cycles as necessary to decide which of the preparation systems is the most appropriate one. This is to ensure that the system chosen will be durable during prolonged freezing/thawing, which the CEN/TS 12390-9 test requires.

# Initial preparation system setup

Below follows the initial material preparation system setup table and the scientific name table for for the second modified CEN/TS 12390-9 test:

Material Test 2 Test Setup				
#	Material	Adhesive	Conditioning	
1	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	3 day water saturation $+$ prepared sides	
2	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	3 day water saturation $+$ prepared sides	
3	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	3 day water saturation $+$ prepared sides	
4	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	3 day water saturation $+$ prepared sides	
5	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	3 day water saturation $+$ prepared sides	
6	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	3 day water saturation $+$ prepared sides	
7	Sitko Elastic 605 Butyl Tape	Casco XtremFix	3 day water saturation $+$ prepared sides	
8	Sitko Elastic 605 Butyl Tape	Casco XtremFix	3 day water saturation $+$ prepared sides	
9	Sitko Elastic $605$ Butyl Tape	Casco XtremFix	3 day water saturation $+$ prepared sides	
10	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	3 day water saturation $+$ prepared sides	
11	Sitko Elastic $605$ Butyl Tape	Casco Marin og Teknikk	3 day water saturation $+$ prepared sides	
12	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	3  day water saturation + prepared sides	
13	Sitko Elastic $605$ Butyl Tape	Casco Marin og Teknikk	3 day water saturation $+$ prepared sides	
	1 2 3 4 5 6 7 8 9 10 11 12	#Material1Sitko Elastic 605 Butyl Tape2Sitko Elastic 605 Butyl Tape3Sitko Elastic 605 Butyl Tape4Sitko Elastic 605 Butyl Tape5Sitko Elastic 605 Butyl Tape6Sitko Elastic 605 Butyl Tape7Sitko Elastic 605 Butyl Tape8Sitko Elastic 605 Butyl Tape9Sitko Elastic 605 Butyl Tape10Sitko Elastic 605 Butyl Tape11Sitko Elastic 605 Butyl Tape12Sitko Elastic 605 Butyl Tape	#     Material     Adhesive       1     Sitko Elastic 605 Butyl Tape     SikaFlex 11FC       2     Sitko Elastic 605 Butyl Tape     SikaFlex 11FC       3     Sitko Elastic 605 Butyl Tape     SikaFlex 11FC       4     Sitko Elastic 605 Butyl Tape     Casco Marin og Teknikk       5     Sitko Elastic 605 Butyl Tape     Casco Marin og Teknikk       6     Sitko Elastic 605 Butyl Tape     Casco Marin og Teknikk       7     Sitko Elastic 605 Butyl Tape     Casco XtremFix       8     Sitko Elastic 605 Butyl Tape     Casco XtremFix       9     Sitko Elastic 605 Butyl Tape     Casco XtremFix       10     Sitko Elastic 605 Butyl Tape     Casco Marin og Teknikk       11     Sitko Elastic 605 Butyl Tape     Casco Marin og Teknikk       12     Sitko Elastic 605 Butyl Tape     SikaFlex 11FC	

Table 5: Material Test 2 Initial System Setup Table

Size [mm]	#	Material	Adhesive	Scientific Name
	1	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	0.40-FA35-N311016-10x10 ButSika <sub>1</sub>
	2	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	0.40-FA35-N311016-10x10 $\operatorname{ButSika}_2$
	3	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	$0.40\mbox{-}{\rm FA35\mbox{-}N311016\mbox{-}10x10}$ ButSikaNoBot
	4	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	0.40-FA35-N311016-10x10 $\operatorname{ButMarTek}_1$
$100\ge 100$	5	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	0.40-FA35-N311016-10x10 $\operatorname{ButMarTek}_2$
	6	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	0.40-FA35-N311016-10x10 ButMarTekNoBot
	7	Sitko Elastic 605 Butyl Tape	Casco XtremFix	0.40-FA35-N311016-10x10 $\operatorname{ButXtrem}_1$
	8	Sitko Elastic 605 Butyl Tape	Casco XtremFix	0.40-FA35-N311016-10x10 $\operatorname{ButXtrem}_2$
	9	Sitko Elastic $605$ Butyl Tape	Casco XtremFix	$0.40\text{-}\mathrm{FA35}\text{-}\mathrm{N311016}\text{-}\mathrm{10x10}~\mathrm{ButXtremNoBot}$
150 x 150	10	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	0.40-FA35-N311016-15x15 ButSikaNew
	11	Sitko Elastic $605$ Butyl Tape	Casco Marin og Teknikk	$0.40\text{-}\mathrm{FA35}\text{-}\mathrm{N311016}\text{-}\mathrm{15x15}~\mathrm{ButMarTekNew}$
	12	Sitko Elastic 605 Butyl Tape	SikaFlex 11FC	0.40-FA35-N311016-15x15 ButSikaOld
150 x 150 Old samples	13	Sitko Elastic 605 Butyl Tape	Casco Marin og Teknikk	0.40-FA35-N311016-15x15 ButMarTekOld

Scientific Names Material Test 2 Setur

#### Scientific name setup table:

Table 6: Scientific Name Table

# Preparation and conditioning of samples

The nine  $100mm \times 100mm$  samples followed the same drying and cutting procedure mentioned in Chapter 3.3.3, before they were left in the climate room for 21 days. They were then brushed on all sides, except the test surface, to remove the form oil. This brush had stiffer steel bristles than the one used on the samples in material test 1.

The nine  $100mm \times 100mm$  samples were brushed alongside the two  $150mm \times 150mm$  samples. These two  $150mm \times 150mm$  samples had spent two months in laboratory air prior to the start of Material test 2. The two old samples were already prepared and were just kept in a dry room with 20 degrees celcius for 7 weeks before the test. The brushing was meticulous work to ensure that the surfaces on the sides and bottom had a rough exterior. The amount of work this demands varies depending on the result one wants. It is albeit necessary to brush so that the sand grains are exposed.

As a safety precaution, it is necessary to wear a facemask and goggles while brushin, since the concrete dusts quite a lot. Another measure is to brush under well ventilated conditions or under an air-suction duct that will suck in the dust particles. It can also be necessary to wet the surfaces before you start brushing to make the dust particles heavier so that more of them will fall to the ground. This practice was used when preparing the samples for this test.

After brushing, the eleven samples were returned to the climate chamber for six to seven days, until a batch of the samples were brought for their application of butyl tape and adhesives. The process lasted over two days. Finished samples were put back into the climate chamber whilst new ones were brought out for application continuously. When all samples were fully prepared, they were returned to the climate room.

The last step before a finished sample is to create their insulation cases. Once every sample was fitted with one, they were returned to the climate chamber and given a 3mm layer of water to

saturate them in preparation of the test. The saturation lasted for 68 and a half hours, which is slightly less than the  $72 \pm 2$  hours the standard requires. The last preparations were the final fitting of the insulation cases and the fixing of the hat of each sample. The test was started on Tuesday the 14th of March at 14:00 and if 56 cycles are run it will last until the 9th of May.

## Placement of samples in the freeze/thaw-chamber

Prior experience with 100 by 100 samples has shown that the amount of water on the test surface  $\approx 30ml$  could possibly dry out if a sample was on the top or 5th shelf (see Chapter -insert reference of result chapter here), only the 150 by 150 samples were placed on these shelves. The small samples were placed in thermically more *stable* environments on the 2nd, 3rd and 4th shelf, where the different types of samples were mixed. Primarily with samples using different adhesives.

Regarding the larger samples, one old and one new sample were placed on both the 1st and the 5th shelf, respectively. Four samples were also given a thermocouple while the last thermocouple monitored the air temperature of the chamber. This thermocouple was on the 3rd shelf, believing that since this is the most thermically stable shelf, its samples will keep within the limits of the CEN/TS 12390-9 scaling test.

The four other thermocouples were each placed in different samples on the different shelves, seen in Figure C35:

- 1st. shelf: Old 150 by 150 sample.
- 2nd. shelf: New 100 by 100 sample; middle sample out of 3.
- 4th. shelf: New 100 by 100 sample; middle sample out of 3.
- 5th. shelf: New 150 by 150 sample.



Figure 7: Freeze/Thaw Chamber

As previously mentioned, as many cycles as possible will be ran before the best preparation system is chosen. This means that samples in the chamber will become fewer and fewer. Rotation of the samples in the chamber is necessary, i.e., for each new cycle, the samples will be placed on a different shelf than it was previously at, to give it a slightly different load during the new scaling cycles.

There will be 16 samples in the chamber, and while 13 are proper samples, three of them are dummies. The dummies were placed on the 1st, 3rd and 5th shelf to spread them out evenly. The necessary amount of salinated water while using 16 samples has been calculated before, and stands at 1072 ml. The amount of water in the chamber:  $4 \times 67ml = 268ml$   $9 \times 30ml = 270ml$ Total water =  $1072ml \Rightarrow$  Excess water = (1072 - (268 + 270))ml = 538ml

The excess water will be placed in styrofoam containers, put on the 6th shelf in the chamber in order to simulate a proper test.

## Sample Evaluation

The samples are evaluated throughout the entire period the CEN/TS 12390-9 Frost Salt test lasts. The intervals of which an assessment is made is the same as the interval of material collection, i.e. 7th, 14th and 28th, 42nd and the 56th day. The characteristics the samples are evaluated on during the visual inspection during the experiment period are:

- Adherence, A, between concrete surface and adhesive/rubber material.
- Water level,  $W_l$ , after 7 days.

The samples will be graded using a grading system that ranges from 0 - 1, where 0 is the worst rating, and 1 is the best. It will be divided in an quarterly fashion, i.e.:

- 0 = Total loss of adhesion on one or more sides. SSD or Dry surface.
- $\frac{1}{4}$  = Weak adhesion on 2 to 4 sides.  $\leq 1mm$  water left.
- $\frac{1}{2}$  = Decent adhesion on all sides. Slight weaknesses. Keeps some water: 1-2mm left.
- $\frac{3}{4}$  = Good adhesion two sides, decent on two sides. Some water leakage: 2mm left.
- 1 = Good/Excellent adhesion on all sides. Negligible water leakage:  $\approx 3mm$  left

Samples with + signs have been refilled in between the 7 day interval with each + signifying a new refill. All samples were refilled every 7th day regardless of level of the solution, to ensure a 3mm sodium chlorided water level on the surface during the test period.

The results will be presented in a table showing the evaluation of each sample's respective score in the different characteristics of each interval.

#### Series 2 - Air-entrained vs. non-air-entrained concrete $\mathbf{3.4}$

A final examination of the preferred modified test preparation system chosen from Material Test 2 will be used in a 56 cycle long CEN/TS 12390-9 test. The test will comprise of ten fly ash concrete samples, five whom have been air-entrained (AEA) and five that have not been airentrained (non-AEA).

As the scope indicated, see Chapter 1, one of the main goals is to find a sample preparation system using new materials that can replace the ones currently used in the standard. And also providing a way to test several characteristics of one sample; saving time and cost. The preferred preparation system will be fully tested in this round of freeze/thaw testing.

The test will run for 56 cycles, but only 28 of them will be reported on in this thesis. The samples tested will be evaluated in two ways. The first evaluation is a visual inspection of the preparation method and the water retaining capabilites of the samples. This is a qualitative measure. The second evaluation is based on the data collected from every sample during the test. Both evaluations will occur at the same intervals declared by the standard; i.e on the 7th, 14th and 28th day of testing.

# Initial preparation system setup

Below follows the material preparation system setup table, as well as the scientific name table. Both tables use the same sample numbers and pointion in the table for easy readability. Making sure that each sample can be referred to either by their sample number, or by their scientific name.

Material		
	Adhesive	Conditioning
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation + 10 sec. s and $\rm blasting^3$
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. s and blasting
Sitko Elastic 605	SikaFlex 11FC	3 day water saturation $+$ 10 sec. sand blasting
	Sitko Elastic 605 Sitko Elastic 605 Sitko Elastic 605	Sitko Elastic 605     SikaFlex 11FC       Sitko Elastic 605     SikaFlex 11FC       Sitko Elastic 605     SikaFlex 11FC

 $^{1}$  This sample was only glued at the top edge, to spread adhesive over the edge. To see if butyl would stick well to sand blasted sides.  $^2$  1 side w/SikaFlex and 3 sides w/Marin og Teknikk due to running out of SikaFlex 11FC.

<sup>3</sup> All sides w/form oil was sand blasted for 10 seconds to achieve a coarse, sandy, surface.

Table 7: Series 2 Initial Preparation System Test Setup

Series 2 Scientific Name Setup Table					
Size [mm]	#	Material	Adhesive	Scientific Name	
	1	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA-041116-150x150-SikaGlueEdge	
	2	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA- $041116-150x150-SikaFlex2$	
150x150	3	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA- $041116-150x150-SikaFlex3$	
Non-air-entrained	4	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA-041116-150x150-SikaFlex4	
	5	Sitko Elastic 605	SikaFlex 11FC	$0.40\text{-}35 \text{FA-}041116\text{-}150 \mathrm{x} 150\text{-} \text{SikaFlex} 5$	
	6	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA-021116-150x150-SikaFlex6	
	7	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA- $021116-150x150-SikaFlex7$	
150x150	8	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA-021116-150x150-SikaMarinTel	
Air-Entrained	9	Sitko Elastic 605	SikaFlex 11FC	0.40-35FA- $021116-150x150$ -SikaFlex9	
	10	Sitko Elastic 605	SikaFlex 11FC	0.40-35 FA-021116-150 x150-SikaFlex10	

#### Scientific name setup table:

Table 8: Series 2 Scientific Name Setup

## Preparation and conditioning of samples

The samples followed the standard preparation procedure found in Table 1, with a few exceptions. They were cast on the 2nd and 4th of November. The air-entrained concrete was cast on the 2n, while the non-air-entrained was cast on the 4th. Meaning the samples had been in a water bath for 6 months before being used in the test. The application process with the butyl tape and adhesive took four days instead of the two day time window. This was because the preparation took slightly longer time as everything was fitted perfectly; from the insulation casing to the butyl tape and adhesive. Each sample also had their form oil removed with sand blasting 10 seconds on each side. This was to achive a coarse and sandy exterior surface which would hopefully make the adhesive stick better.

Since the process of fitting butyl tape, creating the insulation boxes and in general sample preparation takes slightly longer time than the standard allows, not all samples will be removed from the climate room at the same time. Batches of maximum four samples were removed from the climate room at the same time, allowing the samples not prepared to follow the schedule in Table 1 as closely as possible. When the first batch was prepared, they were returned to the climate room and a new batch was brought out. This continued until all the samples were properly fitted with butyl tape and an insulation box to ensure the preparation followed the CEN/TS 12390-9 standard as closely as possible.

#### Placement of samples in the freeze/thaw-chamber

The samples are placed in pairs inside the freeze/thaw-chamber. As there are ten samples, five shelves will be used to store the samples, while the bottom shelf will be used to store the excess water intended if there were 16 samples in the chamber. The setup in the chamber for the first cycle will be the following:

- 1st shelf: Sample 1 and 2
- 2nd shelf: Sample 3 and 4
- 3rd shelf: Sample 5 and 6
- 4th shelf: Sample 7 and 8
- 5th shelf: Sample 9 and 10

The position of the samples will change with every interval during the 56 day test, i.e on the 7th, 14th, 28th and 42nd day the samples will change position. No sample will have the same position from one interval to the next. It is also hoped that every sample will have a thermocouple installed on the test surface at one point during the test to see how it performs.



Figure 8: Series 2 samples

The samples are evaluated throughout the en-

Sample evaluation

tire period the CEN/TS 12390-9 Frost Salt test

lasts. The intervals of which an assessment is made is the same as the interval of material collection, i.e. 7th, 14th and 28th day. The characteristics the samples are evaluated on during the visual inspection during the experiment period are:

- Adherence, A, between concrete surface and adhesive/rubber material.
- Water level,  $W_l$ , after 7 days.

The samples will be graded using a grading system that ranges from 0 - 1, where 0 is the worst rating, and 1 is the best. It will be divided in an quarterly fashion, i.e.:

- 0 = Total loss of adhesion on one or more sides. SSD or Dry surface.
- $\frac{1}{4}$  = Weak adhesion on 2 to 4 sides.  $\leq 1mm$  water left.
- $\frac{1}{2}$  = Decent adhesion on all sides. Slight weaknesses. Keeps some water: 1-2mm left.
- $\frac{3}{4}$  = Good adhesion two sides, decent on two sides. Some water leakage: 2mm left.
- 1 = Good/Excellent adhesion on all sides. Negligible water leakage:  $\approx 3mm$  left

Samples with + signs have been refilled in between the 7 day interval; each + signifying a new refill. All samples were refilled every 7th day regardless of level of the solution, to ensure a 3mm sodium chlorided water level on the surface during the test period. The results will be presented in a table showing the evaluation of each sample's respective score in the different characteristics of each interval.

# 3.5 Concrete materials

This chapter will include the general specifications of the constituents used in the concrete used in Series 1 and Series 2.

All the concrete used in this thesis was mixed at the concrete batch plant at Norbetong in Fagervika. The concrete was mixed and made by the factory workers at the plant. The concrete used in Series 1 was mixed on the 31st of October 2016. The Air-Entrained concrete was cast on the 2nd of November 2016 and the non-Air-Entrained was cast on the 4th of November 2016. Every mix used the following constituents:

#### Cement

The cement used in the mix is a Norcem CEM II/A-V 42.5 N Anleggssement, which satisfies the requirements of the NS-EN 197-1:2011 standard.

### Fly Ash

The fly ash used in this mix is a *Norcem fly ash*, satisfying the NS-EN 450-1:2012, class A regulations.

### Admixtures

The two admixtures in the mix are a superplasticizer and an air entraining admixture, known respectively as *Dynamon SX-23* and *Mapeair 25 1:9*. Both admixtures are produced by Mapei. No air-entrainment admixture was used in the non-AEA fly ash concrete.

#### Aggregates

The aggregates in the concrete used test were Ramlo 0-8 and Nordfosen 8-16.

#### 3.5.1 Mixture proportions and AVA results

The mixture proportions and the properties, including air void analysis (AVA), of fresh concrete for Series 1 and Series 2 are listed in this Chapter.

## Properties

The properties for the concrete used in Series 1 and Series 2 are listed below in Table 9:

Mixture Proportions								
Mix	$(W/C)_{eff}$	Cement	Fly Ash	$\mathrm{SP}^4$	$AEA^5$	Slump	Density	Air
No.		$[kg/m^3]$	$[kg/m^3]$	$[L/m^3]$	$[L/m^3]$	[mm]	$[kg/m^3]$	[%]
$1^1$	0,443	299,3	84,6	2,308	4,994	210	$2323,\!13$	4,8
$2^{2}$	0,440	$299,\!6$	85,0	2,271	4,982	223	2441,88	$^{5,2}$
$3^3$	$0,\!430$	298,5	81,0	2,308	0	230	$2388,\!13$	1,9

 $^{1}$  Concrete used in Material Test 1 and 2

 $^{2}$  Air-Entrained Concrete

<sup>3</sup> Non-Air-Entrained Concrete

<sup>4</sup> Superplasticizer

<sup>5</sup> Air-Entraining-Admixture

Table 9: Concrete Properties, Series 1 and 2

#### Air Void Analysis results

The analysis of the air void system of the concrete used in Material Test 1 and 2, and Series 2 is located below.

Mix	Spacing Factor. $\overline{L}$	Specific Surface, $\alpha$	Air Content	Total Air Content
No.	[mm]	$[mm^{-1}]$	%	%
$1^1$	0,286	25,2	2,6	4,8
$2^{2}$	0,219	34,2	$^{2,4}$	5,2
$3^3$	1,903	$^{2,7}$	$^{5,4}$	1,9

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Alr	void	analysis.	iresn	concrete

 $^{1}$  Concrete used in Material Test 1 and 2  $\,$ 

<sup>2</sup> Air-Entrained Concrete

<sup>3</sup> Non-Air-Entrained Concrete

Note that Mix 3 has errouneous results. If air content is below 3% AVA does not give sensible results.

#### Table 10: Air Void Analysis Series 1 and 2

As the table indicates the data regarding the Air Entrained Concrete is very good. The air content is between the recommened value for good frost durability, between 4 to 7% and the spacing factor is also below the recommened value of 0.250mm, [4]. The results found for Mix No. 3, the non-air-entrained concretes are not viable as the results from an AVA-test is not sensible on a concrete that has an air content below 3%, as is the case with this mix. The data regarding the aggregates, fly ash, cement and admixtures were provided by Andrei Shpak, [12], and further information about the concrete and its constituents can be found in Appendix B.

### 3.5.2 Curing and Preconditioning

All samples in this test were made at Norbetong's factory in Fagervika. The samples were brought back to the laboratory where they were laid to rest in a water bath for 6 months prior to testing. All samples that have been used have been prepared after the CEN/TS 12390-9 standard and each section specifically states when this was not followed, and for what reason. The samples that have not been used are kept in the climate room which has an evaporation rate of  $40 \ g/m^2h$ .

For specific information regarding the preparation cycle of the samples used in each individual test see Chapters 3.3.3, 3.3.4, 3.4 and 3.8. Further information regarding the climate room can be found in Appendix B

# 3.6 Ultrasonic Pulse Velocity, UPV

Ultrasonic Pulse Velocity measures the transit time through layers of material, from a transmitter to a receiver using a pair of transducers using a PUNDIT device. The transducers are placed on opposite sides of the concrete sample after an ultrasound gel is applied to each. The test is non-destructive and it has a low coefficient of variation of 2% if measuring on the same spot, making it highly repeatable.

The transducers in the lab run at  $\approx 50kHz$ , which is well within the recommended value when using UPV on concrete which is  $\epsilon \langle 24kHz, 150kHz \rangle$ . UPV is used as an indicator of internal damages of the concrete as it can show an increase in transit time over time. This might indicate that there has been an increase of cracks in the sample.

For the tests made in this thesis the UPV is measured before, and after, saturation. To provide accurate readings, the equipment is calibrated using the calibration rod provided before every measurement session. The UPV will be measured continuously during the entire test period, given that the sample survives from one interval to the next. According to the CEN/TS 12390-9 standard, measurements of the scaling should be performed every 7th, 14th, 28th 42nd and 56th day if the specimen gets that far. The same interval will therefore be used to check the UPV of the samples as well.

# 3.7 Absorption

It is also of interest to check the absorption of the samples tested during the CEN/TS 12390-9 test. This is done by weighing a dry sample while adding the scaled mass to compensate for the evaporable water loss of the sample at given intervals. The formula below shows how:

$$Absorption = \left(\Delta W \times \left(\frac{0,001}{A}\right)\right) + \left(\left(S_n + S_{n-1}\right) \times \frac{w_{suc}}{w_{dry}}\right) \quad [kg/m^2] \tag{5}$$

Where the different parameters are known as:

- $\Delta W$  = Weight difference of sample,  $W_n W_0$
- A = Area of test surface
- $S_n$  = Scaling at particular time,  $[kg/m^2]$
- $S_{n-1} =$  Scaling value from previous interval
- $\frac{w_{suc}}{w_{dry}}$  = Weight, fully saturated sample, divided by Weight, dry sample.

Jacobsen and Sellevold proved the importance of calculating the absorption of the samples during freeze/thaw testing and their research shows that there is a good correlation between a samples absorption and its scaling values. The paper concludes that freezing and thawing with a NaCl-solution on the test surface increases the absorption and in turn explains part of the scaling damages the sample receives, [5].

## 3.8 Dilation Measurements

A pilot-project to run and monitor samples using dilatometry in the freeze/thaw-chamber is also of interest. To do so, the two invar frames used will have to be tested inside the chamber. To give a proper evaluation as to whether or not it is possible to run dilatometry inside the freeze/thaw chamber while a CEN/TS 12390-9 some testing is required.

#### Preparation of concrete samples

The concrete used was cast on 31st of October 2016, see Chapter 3.5 for further details, and the two fly ash concrete samples used in Part 2 and Part 3 were previously used in Material Test 1 in Series 1. The samples have **not** been exposed to freezing/thawing cycles previously. They were prepared anew after being in laboratory air,  $\approx 20^{\circ}C$  for approximately 1 and a half months. The samples were stripped of their former layers, sandblasted on each side and then placed in the climate chamber for five days. Afterwards they were prepared with a new layer of adhesive and butyl tape before they were returned to the climate room to rest for two days in compliance with Table 1. The sample used in Part 3, *CEN/TS 12390-9 Test* was then saturated for 3 days as the CEN/TS 12390-9 standard requires before being brought out for testing.

#### Pilot project setup

The pilot project will be split into three parts to see what problems are encountered while running dilatometry simultaneously to freeze/thaw test. This will make sure that eventual errors/faults can be fixed before a proper sample is tried and tested in the chamber.

The equipment used for the experiment consists of two invar frames, two dilation measuring devices and two rods. These rods are made from regular stainless steel and invar steel respectively.

- 1. Blank test Two frames in the middle shelf of the freeze/thaw chamber. The test will use one invar rod and one steel rod. This will give the benchmark dilation for future testing. It will also dsplay if the rigs made will experience any problems. These might include uneven measurements due to shaking of the chamber. Other factors tested for are: instability of the rig and gravity influence on the samples.
- 2. Concrete Sample One frame in the middle shelf of the freeze/thaw chamber. The goal is to see how the concrete will differ in dilation compared to the blank run and check dilation occuring when exposed to external air. It will also test the horizontal rig system, which was also tested in Part 1.
- 3. CEN/TS 12390-9 Test One frame in the middle shelf of the freeze/thaw chamber. Preparation following the CEN/TS 12390-9 standard using the preparation system found in Series 1. The sample will have a thermocouple on the test surface, while being exposed to 3% salt solution, to correlate temperature on the surface with the internal dilation whilst being exposed to freeze/thaw-cycles.

After the tests are run comparisons will be made between the different setups and check the influence the different stages are having on the concrete. For Part 3 there will also be a strain/temperature analysis to evaluate whether or not the concrete has experienced any internal damages. The results, as well as the setup, will be evaluated and discussed in Chapter 5.

Details about the rig systems and system setup for Part 1 and Part 2 is located in Appendix D.

# 4 Results

# 4.1 Calibration of Freeze/Thaw cycle

The freeze/thaw cycle was calibrated using four individual cycles and one uninterrupted 4 day cycle. The samples used were the two old  $150 \times 150$  samples previously used in an assignment in the Autumn of 2016.

The preferred cycle was achieved in test number 3 and the cycle achieved then looked like this:

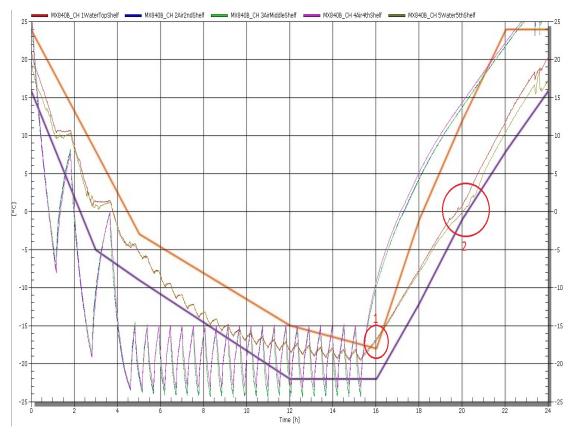


Figure 9: Cycle Test 3

It can be seen from Figure 9 that the cycle is well within the allowed boundaries of the CEN/TS 12390-9 test during more than 90% of the test time. The cycle is probably a little fast to freeze the water in the two samples during the first 10 hours, but the two test samples are still within the margin. There is a slight area, right where the temperature of the samples are rising, that the two samples are outside of the boundaries.

This slight period outside is acceptable as it is for a very short amount of time compared to the entire 24 hour cycle. Furthermore, because a longer run of testing, say 7 days, 14 days, or even longer, will influence the starting temperature of the samples from one cycle to the next, most likely will create some differences compared to this controlled cycle.

When looking at the figure, especially when the temperature is rising and around  $-4^{\circ}Cto + 4^{\circ}C$  that there is a slight dip in the incrasing temperatures. It is thought that this happens because of

the salt in the frozen water, which, when thawing, will induce some sort of freezing mechanism, halting the temperature from rising.

Regarding the four day testing of the prferred cycle, number three, it showed that the freeze/thawchamber would be able to keep itself within and even stabilize itself during a testing period. This is good news as this indicates that the samples AND chamber do not need to be looked at every day. Figure 10 below illustrates the results of the four day test.

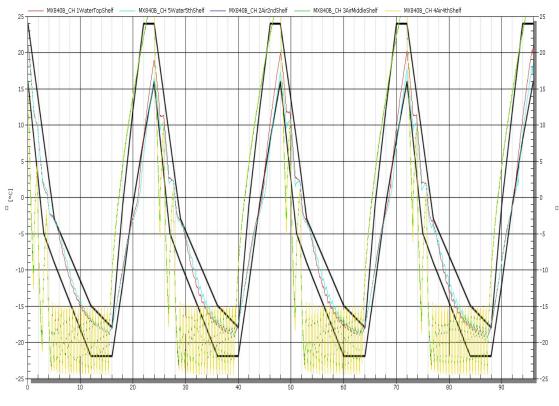


Figure 10: The 4 Day Cycle Test

From the figure it is possible to see that there are some troublesome areas. Especially, as mentioned earlier in this chapter, around zero degrees during thawing. Furthermore, the material lines, the red and light blue one, are very close to the upper boundary during freezing after  $\approx 4$ hours, but it does not exceed it.

What should be especially noticeable, is that the little dent that is very large around zero degrees during thawing in the first cycle seems to even out over the course of the test. At the same interval in the last cycle it is barely even outside of the limit, which is really promising.

The three other test cycles developed to find the preferred test cycle can be found in Appendix C.

## 4.2 Series 1 - Sample Evaluation - Leakage and Adhesion

The results of the material tests performed to develop and improve the current CEN/TS 12390-9 standard Borås test are given in this chapter. The results are based on experimental data and empirical evidence accumulated during the entire testing period, which lasted from early February to early May regarding Matrial Test 1 and Material Test 2. Further information regarding the different materials, the different adhesives as well as the preconditioning and preparation of the samples can be found both in Chapters 3.3.3 and 3.3.4 and in Appendix B and F.

## 4.2.1 Modified CEN/TS 12390-9 Preparation Test 1 - Sample Evaluation

The first material test performed started on the 20th of February and finished on the 27th. The precursor to this test was accumulation of materials to be used, the adhesives to attach the materials to the concrete, as well as the preconditioning and preparation of the samples.

This chapter will only include results concerning the visual inspection of the samples; their adhesion and performance and every observation noted during freezing and thawing. Numerical data of collected material can be found in Chapter 4.4.

## **Application-and Sample Evaluation**

The material test consisted of nine samples and one reference sample from a concrete cast in 2015. The material properties of the samples used are listed in Chapter 3.5, so this chapter will focus its attention to the different processes the samples went through, how they performed and practical considerations regarding each sample.

The following table summarizes the application processes, mentioned in 3.3.3, that each sample has gone through; whether they have failed during a step in the process or considered impractical for further use.

Sample	Sample Name	Application	UPV	Saturation	7 day test
1	Reference Sample	Success	Success	Success	Success
2	ButylSikaFlex	Success	Success	Success	Success
3	ButylMarTek	Success	Success	Success	Success
4	ButylXtremFix	Success	Success	Success	Success
5	$1 \mathrm{mmRubBandSikaFlex}$	Success	Success	Failed (Leakage)	-
6	$1 \mathrm{mmRubBandMarTek}$	Failed (Not properly adhered)	Success	-	-
7	1 mm Rub Band X trem Fix	Success	Success	Success	Impractical
8	3mmEPDM SikaFlex	Failed (Impossible to adhere)	-	-	-
9	3mmEPDM MarTek	Failed (Impossible to adhere)	-	-	-
10	3mmEPDM XtremFix	Success (After much toil)	Success	Success	Impractical

Sample Progress, Material Test 1, Series 1

Table 11: Application Success Table, Material Test 1

As Table 11 indicates, there were some samples that did not make the cut to a full seven day long CEN/TS 12390-9 test. The reasons are varied, from impossible application to impractical to use for many samples, or even leakage. Further details regarding the samples and why they succeeded/failed are found in Appendix E and discussed in Chapter ??.

The Table also shows that all samples using the butyl tape qualified for the full seven day test, whilst the rubber band and the 3mmm EPDM rubber materials could only be applied using the strong XtremFix adhesive.

The samples which did not adhere to the concrete surface did so because of their lack of self adhesion around the concrete side edges, as Table 11 shows. This meant that the material flexed around the corners, creating gaps between the rubber sheeting material and the concrete side.

#### **Cycle Evaluation**

When looking at the Material Test 1 cycle, Figure C34, it seems that the size of the samples influences the temperature, notably on both shelves that are extremes. Meaning that the differences are especially noticeable on the samples on the first and fifth shelf as the samples seems to be both freezing and warming at accelerated rates in this position. The samples on the other shelves do a lot better, staying inside the boundaries most of the time during both freezing and thawing during every cycle.

There is a notable difference from the first cycle compared to the six others. A closer inspection reveals that during the first cycle, the samples on both shelf one and five are outside the lower limit. They also display larger disparities in temperature in the following cycles.

Furthermore the dent in the performance during thawing, point 2 in Figure 9, is still present in this test, although it seems to set in at a later time especially for the samples on shelf one and five. The curvature on the samples on shelf two and four is almost similar to those found in the calibration cycle.

Figure C34 shows that the two samples on shelf one and five are the butyl tape w/Sikaflex glue and the rubber band w/XtremFix. Referencing this with Table 16 shows that these two samples also did not scale.

## 4.2.2 Modified CEN/TS 12390-9 Preparation Test 2 - Sample Evaluation

After the first material test, it was decided that the most practical material to use was the butyl tape. The next question that needs answering is: *Which adhesive is the best to use*? Since the first Material Test did not give any clear answer, new samples will be prepared and more care and consideration will be taken when making the samples. The results found in this chapter are evaluations of the material preparation system, see Table 5, the application phase and if the samples were following the calibrated cycle shown in Figure 9.

#### Sample evaluation

After every finished cycle interval in agreement with the CEN/TS 12390-9 standard a visual inspection of each of the 13 samples is performed. This is used as a tool to evaluate each sample's material composition and its viability to be used in a full 56 cycle CEN/TS 12390-9 Borås test in the future.

A summary of the performance of the 13 samples can be found in the two tables below, Table 12 and 13. The intent is to show how the samples performed during the two stages that is being considered during the tests; preparation/application and the full CENTS 12390/9 test.

The table regarding preparation and application, Table 12, will include a very general grading system, where each sample is given a written grade, where each grade given is explained at the bottom of the table. It is important to note that proper application is of extreme importance for samples to succeed in a full scale test, and inspecting the samples before test start is highly recommended.

Size [mm]	#	Sample Name	Application	Saturation	Pre-test inspection
	1	0.40-FA35-N311016-10x10 $\operatorname{ButSika}_1$	$Success^1$	Success	No Faults Found <sup>2</sup>
	2	0.40-FA35-N311016-10x10 $\operatorname{ButSika}_2$	Success	Success	No Faults Found
	3	$0.40\mathchar`-FA35\mathchar`-N311016\mathchar`-10x10$ ButSikaNoBot	Success	Success	No Faults Found
	4	0.40-FA35-N311016-10x10 $\operatorname{ButMarTek}_1$	Success	Success	No Faults Found
100x100	5	0.40-FA35-N311016-10x10 ButMarTek_2	Success	Success	No Faults Found
	6	$0.40\mbox{-}{\rm FA35\mbox{-}N311016\mbox{-}10x10}$ ButMarTekNoBot	Success	Success	No Faults Found
	7	0.40-FA35-N311016-10x10 ButXtrem <sub>1</sub>	Success	Success	No Faults Found
	8	0.40-FA35-N311016-10x10 $\operatorname{ButXtrem}_2$	Success	Success	No Faults Found
	9	$0.40\mbox{-}FA35\mbox{-}N311016\mbox{-}10x10~ButXtremNoBot$	Success	Success	No Faults Found
150x150	10	0.40-FA35-N311016-10x10 ButSikaNew	Success	Success	No Faults Found
1002100	11	$0.40\mbox{-}{\rm FA35\mbox{-}N311016\mbox{-}10x10}$ ButMarTekNew	Success	Success	No Faults Found
150150	12	0.40-FA35-N311016-10x10 ButSikaOld	-	-	No Major Flaws
150x150 Old samples	13	0.40-FA35-N311016-10x10 ButMarTekOld	-	-	No Major Flaws

#### Material Test 2 Preparation Table

<sup>1</sup> Success is easy appliance, good adherence and no leakage found after saturation period.

 $^2$  No faults found - Sample has no damages and shows no indication of problems.

 $^3$  No major flaws - Sample has some damages, but not detrimental to its performance.

Table 12: Application Success Table, Material Test 2

As Table 12 indicates all the samples were qualified to be included into the second material testing. The only samples that require special notice are the 12th and 13th sample, considering that they are older samples that have not been prepared for this test in accordance with the

CEN/TS 12390-9 standard. They have also been through 11 previous cycles, which lowers their *grading*, from a very good *No Faults Found* to *No Major Flaws*. This indicates that the samples have some previous damages that are not considered detrimental to their inclusion in this test.

The table below shows the visual grading based on the grading system presented in Chapter 3.3.4.

	7 D	7 Days 14 Days		Days	28 I	Days	42	Days	56 I	Days
#	A	W	А	W	А	W	A	W	A	W
1	1	3/4	1	1 +	3/4	0	1/2	1/2 +	1/2	1/4
2	1	3/4	1	3/4	1/2	0	1/2	1/4 +	1/4	0
3	1	3/4	1	1	1	1/4	3/4	1/4	1/2	0
4	3/4	1	3/4	1 +	3/4	1/2	3/4	3/4	1/2	0
5	1	1	1	1 +	3/4	0	1/2	3/4	1/4	0
6	1	1	1	1/2	1	0	1	3/4 +	1/2	0
7	1/2	1	0	3/4	-*	-	-	-	-	-
8	1/2	3/4	0	1 +	-	-	-	-	-	-
9	1/2	1/4	0	1 +	-	-	-	-	-	-
10	1	1	3/4	1 +	1/2	0	1/2	1 +	1/4	0
11	1	1	3/4	1/4	3/4	1/4	1/4	1/2 +	0	0
12	1/2	1/2	1/2	0	1/4	0	0	0	x**	x
13	3/4	1/4	1/2	1/2 +	0	0	0	1/4++	x	х

Material Test 2 Sample Evaluation

\* Sample has been removed from the test due to adhesion loss.

\*\* Sample removed after 48 cycles and has no virtues left.

## Table 13: Evaluation Summary Table, Material Test 2

The table shows that after 14 freeze/thaw cycles all three samples using the *XtremeFix* adhesive failed. The reason for the failure was the same in all three samples; lack of adhesion between the adhesive and the concrete specimen. Sample 12 and 13 were removed after 48 cycles of Material Test 2, i.e they had then been freeze/thaw tested for 59 cycles in total. They were removed because they had no virtues left and they were frequently drying out as well.

As can be seen from Table 13 every sample that made it to the end of the test was either in decent to poor condition or in poor condition overall. The samples using SikaFlex seem to be doing just as well as the samples using Marin og Teknikk Lim og Fug and there is not a lot separating them in terms of water retention or adhesion. Of the large samples the SikaFlex did slightly better, yet both samples have very poor ratings in the end, both for water retention and adhesion to the concrete surface. For in-depth detail description of each sample during the intervals, see Appendix E.

The rating of each sample is qualitative and is based on first hand experience with the samples as well as photographs of them and the written sample details in Appendix E.

#### **Cycle Evaluation**

The 56 cycles logged during Material Test 2 shows that the samples are well within the boundaries most of the time during testing, especially after a short maintenance period. The cycles also show that the samples are gradually becoming more and more difficult to keep within the boundary conditions of the CEN/TS 12390-9 Slab test as the test goes on.

The cycles also show that they will stabilize themselves well within the limits set by the CEN/TS 12390-9 standard when the samples are being freeze/thaw tested for long periods.

The cycles also show after 28 days that more maintenance of the samples is required as the oscillations become greater and appear more frequently in all samples after this point. To see all of the cycles in their respective order, see Appendix C

## 4.3 Series 2 - Sample Evaluation - Leakage and Adhesion

In Series 2 the preparation setup from Material Test 2 proven to yield the best results will be used. This section will contain the results based on the visual inspection of the samples and the performance of the samples in the CEN/TS 12390-9 test cycle, Figure 9.

#### Sample Evaluation

The samples used in Series 2 have been continuously evaluated throughout the 28 day test period used in this thesis. The individual rating of each sample is displayed in Table 14 showing the progress of deterioration of the samples based on the grading system found in Chapter 3.4.

	7 D	Days	14 I	Days	28 Days		
#	А	W	А	W	А	W	
1	3/4	0	1/2	0	1/4	0	
2	1	0	1/2	1	1/4	0	
3	1	0+	1	0	3/4	0	
4	1	0+	3/4	0	1/4	1/4	
5	1	0	3/4	0	1/2	0	
6	1	1+	1	3/4	1	1/4	
7	1	0+	1	1	1	3/4	
8	1	3/4	3/4	0	3/4	1/4	
9	1	0	1	1	1	1	
10	1	0 +	3/4	0	3/4	3/4	

Series 2 Sample Evaluation

Table 14: Evaluation Summary Table, Series 2

Adhesive was added over the duct tape on the test samples throughout the testing period to improve their leakage resistance. Sample 2 and 6 received this treatment after 7 cycles. Sample 7 and 9 received the treatment after 14 cycles. The rest of the samples received the treatment after 21 cycles.

Table 14 shows that the samples that are air-entrained are better at retaining water and have better adhesion than their non-air-entrained counterparts. The only sample close to the level of the air-entrained ones is sample 3, which has been been performing better than the other non-air-entrained samples when it comes to adhesion, but not in water retention.

The evaluation shows that all samples struggled to keep water in the beginning, but that the water retaining capabilities of all samples improved during the course of the test.

Table 14 shows that sample 9 has been the best sample of the entire group. Scoring the highest grade on adhesion throughout the entire test. It has also been the sample with the most consistent water retaining abilities, having never been refilled between intervals.

The table also shows that the worst sample was sample 1. This was the sample according to Table 8 that had an adhesive layer on the edge of the sample. It performed poorly both in adhesion and water retention throughout the test period-

All evaluations are qualitative; based on experience and material and information collected during the test period. Detailed information regarding the samples, with pictures and interval description, can be found inn Appendix E.

#### Cycle Evaluation

The 28 cycles show that the samples are keeping well within the boundaries during most of the cycle period. The interesting thing of notice that is correlated to Table 14 is that when samples struggle to retain water, they perform poorly in the cycles. The solution temperature in the samples which have low gradings, indicating a low to no solution level, will have greater oscillations, displaying a greater need for maintenance and water refill.

Another interesting take is that as the performance and the water retaining capabilities have improved over the test, this is noticeable in the cycles as well. This is seen in Figure C44 where all samples are performing well inside the required CEN/TS 12390-9 boundary conditions during the entire cycle.

## 4.4 UPV, Absorption and Scaling results

This chapter will revolve around the collected data whilst performing a standard CEN/TS 12390-9 slab test. The information collected in this chapter will come from three separate runs; Material Test 1, Material Test 2 and the Air-entrained concrete versus the non-air-entrained concrete. These runs lasted for 7 days, 56 days and 28 days for each respective test done.

The data collected from each run was: UPV, scaled material and sample weight from the three different runs. Material Test 2 and Series 2 - AEA vs. Non-AEA fly ash concrete also checked the absorption of the samples.

#### Material Test 1

This section will concern the information collected during the one week long Material Test 1 to give an indication of the quality of the concrete that has been used in this Material Test.

#### UPV reults

The table below illustrates the UPV gathered from all samples at the beginning and at the end of the test, with the last column indicating their difference.

Sample	Material	Adhesive	$\mathrm{UPV}_{0}$	$\mathrm{UPV}_7$	Difference
1	Sitko Elastic 605	None	4255	4049	-206
2	Sitko Elastic 605	SikaFlex 11FC	3717	3623	-94
3	Sitko Elastic 605	Marin og Teknikk	3745	3731	-14
4	Sitko Elastic 605	XtremFix	3663	3460	-203
5	1mm Rubber Band	SikaFlex	4000	-	-
6	1mm Rubber Band	Marin og Teknikk	3876	-	-
7	1mm Rubber Band	XtremFix	3731	3636	-95
8	3mm EPDM	SikaFlex 11FC	-	-	-
9	$3 \mathrm{mm} \mathrm{EPDM}$	Marin og Teknikk	-	-	-
10	$3 \mathrm{mm} \mathrm{EPDM}$	XtremFix	3378	3356	-22

Material Test 1, UPV - Transit velocity, [m/s], Mix 1, CEN/TS 12390-9 Test

## Table 15: UPV, Material Test 1

As Table 15 shows there is decreased velocity through every sample, with the biggest change being -206 m/s across the reference sample. The table also shows that the adhesive layers add some extra material for the ultrasound to pass through, giving lower transit velocity for every sample with adhesive compared to the reference sample. The Marin og Teknikk sample had the lowest velocity drop, with the SikaFlex sample coming in second, while the XtremeFix samples had the largest mean velocity drop.

#### Scaled material measurements

Table 16 below shows the amount of scaling occuring on these samples after 7 days of exposure to freeze/thaw cycles while the test surface submerged in a 3mm layer of water with 3% sodium chloride solution.

	Sample	$m_7$	$\Rightarrow$	$S_7$
1	Reference Sample	2,72	$\Rightarrow$	0,272
2	0.40-FA35-N311016-10x10 ButylSikaFlex	-	$\Rightarrow$	-
3	0.40-FA35-N311016-10x10 ButylMarFug	$3,\!98$	$\Rightarrow$	0,398
4	0.40-FA35-N311016-10x10 ButylXtremFix	$3,\!91$	$\Rightarrow$	$0,\!391$
7	$0.40\ensuremath{\text{-}FA35}\ensuremath{\text{-}N311016}\ensuremath{\text{-}10x10}\ensuremath{1\text{mmRubBandXtremFix}}$	-	$\Rightarrow$	-
10	0.40-FA35-N311016-10x10 3mmEPDMXtremFix	$3,\!49$	$\Rightarrow$	0,349

Material Test 1 Scaled material, S,  $[kq/m^2]$ , Mix 1, CEN/TS 12390-9 Test

tial after 7 days.  $[kg/m^2]$ 

Table 16: Scaled Material, S, Material Test 1

Table 16 shows that the samples are scaling rapidly, with a possible end result way above the accetable limit, see Table 2. Neither preparation method separates itself with significantly lower scaling. The reference sample scaled the least. The samples that did not scale, 2 and 7, were quite dry on their surface indicating possible leakage of the test surface.

## Weight of samples, Material Test 1

The weight of the samples is used to determine water uptake in the sample. Table 17 below shows the weight development of the samples for the duration of the test.

Sample	Material	Adhesive	$\mathrm{W}_{\mathrm{0}}$	$W_7$	Difference
1	Sitko Elastic 605	None	1275,1	1288,9	+13,8
2	Sitko Elastic 605	SikaFlex 11FC	1220,8	1238,5	+17,7
3	Sitko Elastic 605	Marin og Teknikk	1197,3	1216,4	+19,1
4	Sitko Elastic 605	XtremFix	$1243,\!9$	1271,8	+27,9
5	1mm Rubber Band	SikaFlex	1201,7	-	-
6	1mm Rubber Band	Marin og Teknikk	-	-	-
7	1mm Rubber Band	XtremFix	1241,1	1262,2	+21,1
8	$3 \mathrm{mm} \mathrm{EPDM}$	SikaFlex 11FC	-	-	-
9	$3 \mathrm{mm} \mathrm{EPDM}$	Marin og Teknikk	-	-	-
10	3mm EPDM	XtremFix	$1272,\! 6$	1299,1	+26,5

Material Test 1, Weight, [g], Mix 1, CEN/TS 12390-9 Test

Samples with an "-" have been removed from the test prior to test start. Sample 5 was found to be leaking after weighing and was removed from the test.

Table 17: Weight, Material Test 1

Table 17 shows that all samples have significantly increased their weight during the 7 day test period. The table also shows that the samples that have gained the most weight are the samples using the XtremFix adhesive, with a weight gain of more than 20 grams on all samples.

## Material Test 2

This section will focus on the results gathered from the modified CEN/TS 12390-9 Slab Test 2, Material Test 2, over the course of 56 cycles. The tables illustrate the progress of deterioration of the samples; from pre-testing to a final CEN/TS 12390-9 test. To increase readability of the larger tables, the scientific sample name will not be listed, only the sample number.

## $UPV\ measurements$

Table 18 below, shows the ultrasonic pulse velocity transit time of each sample used in the test. The transit times were measured during the entire test period for all samples at appropriate intervals of 7, 14, 28, 42 and 56 days.

Size [mm]	#	$\mathrm{UPV}_{0}$	$\mathrm{UPV}_7$	$UPV_{14}$	$UPV_{28}$	$UPV_{42}$	$UPV_{56}$	Difference
	1	3597	3663	3636	3774	3676	3650	+53
	2	3690	3745	3731	3774	3788	3774	+84
	3	3636	3650	3636	3774	3774	3802	+166
	4	3676	3690	3676	3802	3717	3759	+83
100x100	5	3676	3745	3731	3788	3774	3731	+55
	6	3717	3704	3676	3831	3861	3906	+189
	7	3546	3571	3597				+51
	8	3584	3610	3623				+39
	9	3559	3559	3597				+38
150x150	10	3817	3827	3866	3979	3927	3846	+29
190X190	11	3788	3797	3916	3856	3906	3876	+88
150150	12	3807	3906	3846	3968	3886		+79
150x150 Old Samples	13	3846	3797	3846	3989	3916		+70

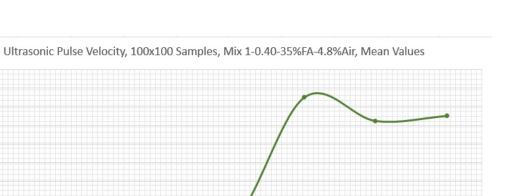
Material Test 2, UPV - Transit velocity, [m/s], Mix 1, CEN/TS 12390-9 Test

## Table 18: UPV, Material Test 2

Table 18 shows that of the 6  $100 \times 100 mm^2$  samples that made a full 56 cycle test the samples without any bottom has increased their velocity the most. The other small  $100 \times 100$  samples have also increased their transit velocity and as such decreased their time through the samples. All samples have increased their velocity during the test period, but the values for the  $150 \times 150 mm^2$  samples does not show any major differences in the material preparation setup.

Comparing these values in Table 18 with the values in the absorption table, Table 22, it is seen that the samples that have increased their velocity the most have also absorbed the most water during the test.

The Table also shows that the highest readings on average are achieved on the 28th day of testing. This can also be seen on the progression of the mean UPV value in Figure 11 and 12 below: -3





No. of Cycles

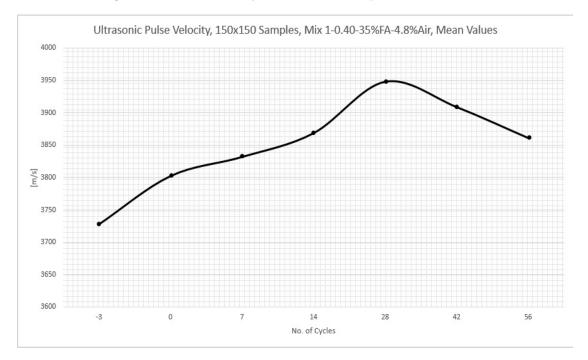


Figure 12: Transit Velocity,  $150 \times 150 \times m^2$  samples, Material Test 1

## Scaled material measurements

The tables found in this segment give information regarding the scaled material from each sample during the 56 cycle slab test. The mass gathered from each sample shows how the quality of the concrete affects the deterioration result of the different samples. Each value is expressed as single values except for values found in the last column in each table, which displays the cumulative scaled mass for the respective sample.

Size [mm]	#	$m_7$	$m_{14}$	$m_{28}$	$m_{42}$	$m_{56}$	$m_{Cumulative,56}$
	1	1,30	1,21	2,9	$0,\!51$	0,4	6,32
	2	2,01	$3,\!28$	4,8	1,25	$^{2,6}$	$13,\!94$
	3	0,97	0,88	$^{2,5}$	2,86	$1,\!8$	9,01
	4	$1,\!69$	1,26	$^{3,8}$	$1,\!91$	$^{2,4}$	11,06
100x100	5	2,31	1,33	$^{3,1}$	$0,\!42$	$1,\!6$	8,76
	6	$1,\!12$	1,26	$^{3,8}$	0,96	$^{1,1}$	7,88
	7	2,06	1,73				3,79
	8	$2,\!13$	$2,\!52$				4,65
	9	$1,\!94$	$2,\!08$				4,02
150x150	10	17,61	6,85	5,7	0,37	1,3	31,83
190X190	11	18,22	11,78	5,1	$1,\!51$	1,4	38,01
150x150	12	8,78	9,80	7,0	2,16		$27,74^{1}$
Old Samples	13	1,56	$7,\!30$	8,4	2,33		$19,59^{2}$

Material Test 2, Scaled mass,  $m_n$ , [g], Mix 1, CEN/TS 12390-9 Test

 $^1$ Ran for 59 cycles. Removed before test finished, heavy damage - scaled mass larger than displayed.  $^2$ Ran for 59 cycles. Removed before test finished, heavy damage - scaled mass larger than displayed.

Table 19: Scaled Mass, m, Material Test 2

Table 20 below	shows t	he	corresponding	scaled	material,	$\mathbf{S},$	$\operatorname{to}$	$_{\mathrm{the}}$	$\operatorname{collected}$	$\operatorname{dried}$	${\rm mass}$	from
Table 19.												

Size $[mm]$	#	$S_7$	$S_{14}$	$S_{28}$	$S_{42}$	$S_{56}$	$\mathbf{S}_{Cumulative,56}$
	1	$0,\!130$	0,121	0,290	0,051	0,040	$0,\!632$
	2	0,201	0,328	$0,\!480$	$0,\!125$	0,260	$1,\!394$
	3	0,097	0,088	$0,\!250$	0,286	$0,\!180$	0,901
	4	0,169	$0,\!126$	$0,\!380$	$0,\!191$	0,240	$1,\!106$
100x100	5	0,231	$0,\!133$	$0,\!310$	0,042	0,160	0,876
	6	$0,\!112$	$0,\!126$	$0,\!380$	0,096	$0,\!110$	0,788
	7	0,206	$0,\!173$				0,379
	8	0,213	0,252				0,465
	9	0,194	0,208				0,402
Mean $S_{56}$							0,950
150x150	10	0,783	0,304	0,253	0,016	0,058	1,414
150X150	11	0,810	0,524	$0,\!227$	0,067	0,062	1,690
150x150	12	0,390	0,436	0,311	0,096		$1,233^{1}$
Old Samples	13	0,069	0,324	0,373	0,104		$0,870^{2}$
Mean $S_{56}$							1,301

Material Test 2, Scaled material,  $S_n$ ,  $[kg/m^2]$ , Mix 1, CEN/TS 12390-9 Test

 $^1$  Ran for 59 cycles. Removed before test finished, heavy damage - scaled mass larger than displayed.  $^2$  Ran for 59 cycles. Removed before test finished, heavy damage - scaled mass larger than displayed. Sample 7, 8 and 9 not included in mean value as they did not survive 56 cycles.

Table 20: Scaled Material, S, Material Test 2

Table 20 shows that all four large concrete samples are over the accepted scaling limit according to Table 2. As for the small samples, sample 2 and 4 were damaged the most, while all samples except for sample 1 are close to the *Not Acceptable* limit.

The mean value of the sample shows that the small  $100 \times 100 mm^2$  samples are just below the *Not Acceptable* threshold as a group, whilst the large  $150 \times 150 mm^2$  samples are considerably over.

The samples also show that they scale less after 28 days than they do before, which is also in accordance with the CEN/TS 12390-9 standard. This is helped by the scaling values after 42 cycles which are lower than in the other intervals for many of the samples, particularly the  $150 \times 150 mm^2$  samples.

#### Weight and absorption

The results regarding the progression of the weight and absorption of the samples are found here. Weight is an important characteristic to measure as it measures the water uptake of the samples. Table 21 and Table 22 below illustrates the weight progression and absorption of the samples in Material Test 2.

Size $[mm]$	#	$W_{-3}$ <sup>1</sup>	$W_0$	$W_7$	$W_{14}$	$W_{28}$	$W_{42}$	$W_{56}$	Difference
	1	1223,2	1235,6	1239,0	1240,0	1241,4	1244,7	1245,1	+9.5
	2	1240,0	1249,4	1252,3	1250,2	1251,7	1255,3	1252,1	$^{+2,7}$
	3	1221,8	1231,5	$1236,\! 6$	1241,3	1249,0	$1247,\!8$	1246,8	+15,3
	4	$1247,\!8$	1259,5	$1262,\! 6$	1264,0	1265,3	1266,7	1265,3	+5,8
100 x 100	5	$1239,\!8$	1249,3	1251,1	1251,5	1252,9	1256,1	1256,1	+6,8
	6	1208,0	1219,9	1226,1	1230,5	1234,1	1236,1	1234,8	+14,9
	7	$1250,\!0$	1262,2	1266,0	1268, 8				$+6,\!6$
	8	$1245,\!5$	1256,3	1259,1	$1259,\! 6$				+3,3
	9	1218,2	1229,3	1236,3	$1242,\!3$				+13,0
150x150	10	2599,7	2624,2	2614,3	2612,7	2611,2	2617,4	2616,4	-7,8
190X190	11	$2561,\! 6$	2586,2	2576,0	2570,3	$2572,\!3$	$2576,\!8$	2575,5	-10,7
150150	$12^{2}$			2649,8	2644,6	2640,8	2645,5		-4,3
150x150 Old Samples	$13^{3}$			$2821,\! 6$	2821,5	2815,9	2818,2		-3,4

Material Test 2, Weight, [g], Mix 1, CEN/TS 12390-9 Test

 $^{1}$  Weight of samples before saturation.

 $^2$  Sample was not saturated before test start as it was an old sample used in previous tests.

 $^3$  Sample was not saturated before test start as it was an old sample used in previous tests.

## Table 21: Weight, Material Test 2

Table 21 shows a gradual increase of the weight of all the small samples and a weight loss in the large ones. The weight gain has been gradual and it is difficult to see if there are any intervals that are more prone to gaining mass than others. For the large samples, especially in the new samples, it is clear that the first and second scaling are imperative in the overall weight loss of the samples. While both samples scale more material than they gain from absorption during these two intervals in particular.

The table also shows that the samples that have no bottom are the samples that have gained the most weight during the test period. Even sample 9, which only lasted for 14 cycles, show this trend very clearly.

Table 22 below gives the entire 56 day test with the accumulated absorption for each sample at every interval when:

$$\frac{w_{suc}}{w_{dry}} = 1,06$$

Size $[mm]$	#	$A_{-3}{}^{1}$	$A_0$	$A_7$	$A_{14}$	$A_{28}$	$A_{42}$	$A_{56}$
	1	-1,24	0,00	0,478	0,706	1,153	1,538	1,620
	2	-0,94	0,00	0,503	0,641	1,300	1,592	1,748
	3	-0,97	0,00	$0,\!613$	$1,\!176$	2,211	$2,\!394$	$2,\!485$
	4	-1,23	0,00	$0,\!489$	0,763	$1,\!296$	$1,\!638$	1,752
100x100	5	-0,95	0,00	$0,\!425$	0,606	$1,\!074$	$1,\!439$	1,503
	6	-1,19	0,00	0,739	1,312	2,075	2,377	2,363
	7	-1,22	0,00	$0,\!598$	1,032			
	8	-1,08	0,00	0,506	0,823			
	9	-1,11	0,00	0,906	1,726			
150x150	10	-1,09	0,00	0,390	1,082	1,283	1,576	1,593
190X190	11	-1,09	0,00	0,405	1,160	$1,\!489$	1,761	1,769
150x150	$12^{2}$		0,00	0,982	0,688	0,849	1,160	
Old Samples	$13^{3}$		0,00	1,166	0,413	0,559	0,771	

Material Test 2, Absorption, A, accumulated,  $[kg/m^2]$ , Mix 1, CEN/TS 12390-9 Test

<sup>1</sup> Absorption of samples before saturation.

 $^{2}$  Sample was not saturated before test start as it was an old sample used in previous tests.

<sup>3</sup> Sample was not saturated before test start as it was an old sample used in previous tests.

Table 22: Absorption, Material Test 2

Table 22 shows that samples 3, 6 and 9 of the  $100 \times 100 mm^2$  are absorbing considerably more than the other small samples. When comparing the absorption of the small samples to the scaling of the series, Table 20, it shows that the largest absorption values are not related to the largest scaling.

Comparing the absorption to the UPV values, Table 18, shows a relation between high absorption and increased velocity over time. This is visible in samples 3 and 6, which has the most dramatic increase in UPV and the largest absorption values.

The table also shows that from the large  $150 \times 150 mm^2$  samples the newer have a more gradual increase of absorption. The old ones fluctuate more, starting at a high absorption before it drops before gradually absorbing more water again.

To really see the impact the different material preparations have on the differences in absorption Figure 15 below will illustrate the importance of choosing the right kind of material preparation setup. It is important to note that the numbers for each preparation are based on the average value of the samples that have the same preparation. All samples that have no bottom are considered to be one preparation38,1 3, as the samples share the same qualities.



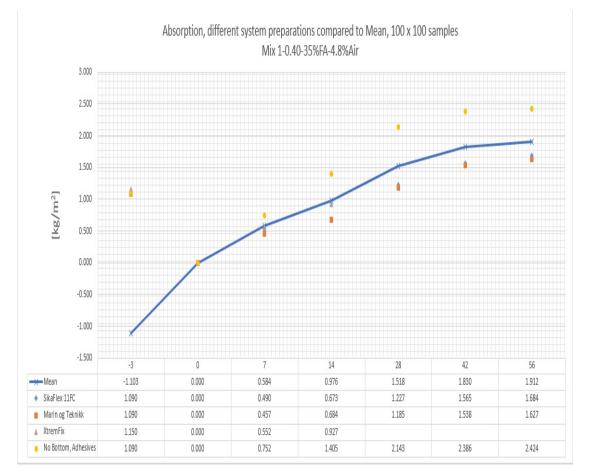


Figure 13: Absorption when  $\frac{W_{suc}}{W_{dry}} = 1.06$ 

Figure 13 illustrates a clear difference in absorption values between the samples that have a bottom and those that do not. The no-bottom samples have the largest weight gain as well, see Table 21, which is related to the samples' water uptake during testing.

Figure 13 also shows that all samples with a bottom do well compared to the series' mean value. With both the SikaFlex 11FC and Casco Marin og Teknikk Fug og Lim performing well below the average value. The table also indicates that these two adhesives do better than the XtremFix samples, which are closer to the mean value of the entire series.

### Series 2 - AEA vs. non-AEA fly ash concrete

The results of the UPV, scaling and absorption regarding Series 2 are found here.

#### UPV measurements

As with the previous tests, checking the UPV of the samples could show internal damages as well as alterations in the air void systems. The table below gives the UPV values of the non-air-entrained and the air-entrained fly ash concretes used in Series 2.

				,		
Size [mm]	#	$\mathrm{UPV}_{0}$	$\mathrm{UPV}_7$	$\mathrm{UPV}_{14}$	$\mathrm{UPV}_{28}$	Difference
	1	4249	4225	4225	4274	+25
	2	4065	4021	3979	4054	-11
150x150	3	4132	4098	4032	4098	-34
Non-air-entrained	4	4155	4167	4190	4213	+58
Mix 3	5	4098	4121	4132	4155	+57
Mean $UPV_{non-AEA}$		4140	4127	4112	4159	+19
	6	3906	3896	3866	3876	-30
	7	3937	3896	3866	3846	-91
150x150	8	3906	3979	3836	3856	-50
Air-Entrained	9	3916	3846	3846	3846	-70
Mix 2	10	3876	3797	3788	3797	-79
Mean $UPV_{AEA}$		3908	3883	3840	3844	-64

Series 2, UPV - Transit velocity, [m/s], Mix 2 and 3 CEN/TS 12390-9 Test

Table 23	UPV,	Series 2
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Table 23 shows that the concretes with air-entrainment have decreased their transit velocity across every sample, while the non-air-entrained have, for the most part, increased theirs. Sample 2 and 3 are the ones who have decreased values.

The comparison between the mean values of the sample shows that the transit velocity is between 2 to 300 [m/s] less for the air-entrained concretes. Figure 14 below better illustrates the development of the UPV for the duration of the test.



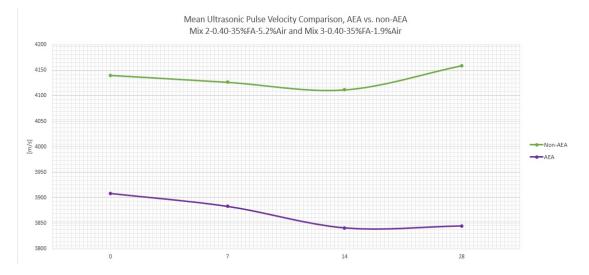


Figure 14: UPV, AEA vs. Non-AEA Fly Ash concrete

Figure 14 shows that all samples decreased their transit velocity on average during the interval between 0 to 14 cycles, before stabilizing and ascending a little in the end. The rise from the 14th cycle to the 28th is a lot higher in the non-AEA concretes than in the AEA-samples.

## Scaled material measurements

The following tables give the result in scaled material, both in dried mass, m, and in scaled material, S. The last column lists the cumulative values, whilst the other columns use single values.

Size [mm]	#	$m_7$	$m_{14}$	$m_{28}$	${ m m}_{Cumulative,28}$
	1	$11,\!9$	7.71	23,42	43,03
	2	16,5	$21,\!12$	$32,\!15$	69,77
150x150	3	$^{3,1}$	$3,\!90$	15,72	22,72
Non-air-entrained	4	$12,\!5$	9,94	32,43	54,87
Mix 3	5	10,0	$10,\!24$	26,24	46,48
	6	$^{0,4}$	1,04	$3,\!88$	$5,\!32$
	7	$^{0,2}$	$0,\!29$	$2,\!28$	2,77
150x150, Mix 2	8	$0,\!3$	$0,\!39$	0,78	$1,\!47$
Air-Entrained	9	0,2	$0,\!23$	1,24	$1,\!67$
Mix 2	10	$^{0,1}$	$0,\!12$	0,16	$0,\!38$

Series 2, Scaled mass,  $m_n$ , [g], Mix 2 and 3, CEN/TS 12390-9 Test

Table 24: Scaled Mass, m, Series 2

				,	
Size [mm]	#	$S_7$	$S_{14}$	$S_{28}$	$\mathbf{S}_{cumulative,28}$
	1	$0,\!53$	0,34	1,04	1,91
	2	0,73	$0,\!98$	$1,\!43$	$3,\!14$
$150 \times 150$	3	$0,\!14$	$0,\!17$	0,70	1,01
Non-air-entrained	4	$0,\!56$	$0,\!44$	1,44	$2,\!44$
Mix 3	5	$0,\!44$	$0,\!46$	$1,\!17$	2,07
	6	$0,\!02$	0,05	$0,\!17$	$0,\!24$
	7	$0,\!01$	0,01	$0,\!10$	$0,\!12$
$150 \times 150$	8	$0,\!01$	0,02	$0,\!03$	0,06
Air-Entrained	9	$0,\!01$	$0,\!01$	$0,\!06$	0,08
Mix 2	10	0,004	0,005	0,007	0,016

Series 2, Scaled material,  $S_n$ ,  $[kg/m^2]$ , Mix 2 and 3 CEN/TS 12390-9 Test

Table 25: Scaled Material, S, Series 2

As the scaling shown in both Tabel 24 and 25 shows, there is a clear difference between the air entrained concretes and the non-air entrained concretes. The values for the non-air entrained ones are way above the accepted limit illustrated in Table 2.

The air-entrained fly ash concretes show that a concrete, even with as high amount of Fly Ash as 35 %, can still perform at a *Very good* level according to the standard.

The reason for this are the good frost durability qualities of Mix 2. Both the air content measured at 5,2% using the pressure method, and an air void spacing factor as low as 0,219, measured by AVA are well within the margin believed to give very good frost durability, see Chapter 2.3.

#### Weight and absorption

Below follows the tables regarding the weight of the samples as well as the calculated absorption for the duration of the freeze/thaw test. The information shown in the absorption table will give clear indications as to whether or not there might be more internal damages in the concrete, as increased absorption values tend to be related to internal damages.

Size [mm]	#	$W_0$	W <sub>7</sub>	$W_{14}$	${{W}_{28}}^3$	Difference
	1	2755,9	2747,4	2743,4	2729,3	-26,6
	2	$2827,\!8$	$2816, 1^1$	2799,5	2774,5	-53,3
150 x 150	3	$2766,\! 6$	2767,8	2766, 8	2756,5	-10,1
Non-air-entrained	4	2726,4	2714,5	$2713,\!8$	2687,7	-38,7
Mix 3	5	2721,8	$2721,\!1$	2714,0	2693,3	-28,5
	6	2669,7	$2673,\!6^2$	2675,3	$2674,\!4$	+4,7
	7	2647,5	2652,3	2655,5	2664,5	+17,0
150 x 150	8	2733,5	2738,8	2741,0	$2738,\!8$	+5,3
Air-Entrained	9	2642,7	2646,3	2646,4	2648,4	+5,7
Mix 2	10	2645,4	2649,9	2649,7	2651,2	+5,8

Series 2, Weight, [g], Mix 2 and 3 CEN/TS 12390-9 Test

<sup>1</sup> Adhesive up duct tape sides to see if it helped against leakage.  $Weight_{glue} = 2826, 6$ 

 $^2$  Adhesive up duct tape sides to see if it helped against leakage.  $Weight_{glue}=2686,3$ 

Adhesives addded to sample 7 and 9 between 7 and 14 days  $\Rightarrow$  Large weight increase on 14th day. <sup>3</sup> Sample 1,3,4,5,8 and 10 received an adhesive layer over duct tape after 21 days, explaining the weight increase/lack of weight loss in respective samples on the 28th day.

#### Table 26: Weight, Series 2

All samples received a layer of adhesive during testing to cover up the duct tape to see if it would stop eventual leaks. This is because leakage has been the dominant cause of drying og the samples, see Table 14 and Table 13. It is likely that the leakage is caused by a capillary suction process between the water front and the duct tape.

Adhesives were first applied to sample 2 and 6, which were weighed before and after adhesive was applied. The result is seen in point 1 and 2 in Table 26. The extra weight added to the samples is calculated below:

$$Mean = \frac{((2826, 6 - 2816, 1) + (2686, 3 - 2673, 6))}{2} = 11, 6 \quad [g]$$

The adhesive applied to sample 6 and 2 gave a positive result, and helped each sample keep a solution layer on the test surface without refill for 7 days. Because of the positive results, all samples were subjected to the same treatment. Since the rest of the samples were not weighed, the average weight gain will be set to 11,6 grams as the equation above states. The weight in

Table 26 has been adjusted for the added weight, meaning 11,6 grams have been subtracted from the samples weight in every interval since the adhesive was added. -Discussion or methods?

The absorption is calculated using equation 5. The result of this calculation is shown below in Table 27.

		-	, ,		
Size [mm]	#	$A_0$	$A_7$	$A_{14}$	$A_{28}$
	1	0,00	0,18	$0,\!37$	0,85
	2	0,00	0,26	$0,\!52$	0,92
150x150	3	0,00	$0,\!20$	0,34	$0,\!62$
Non-air-entrained	4	0,00	$0,\!06$	$0,\!50$	0,87
Mix 3	5	0,00	0,44	$0,\!61$	0,92
	6	0,00	0,19	0,32	$0,\!46$
	7	0,00	$0,\!22$	$0,\!38$	$0,\!89$
$150 \times 150$	8	0,00	$0,\!25$	$0,\!37$	$0,\!31$
Air-Entrained	9	0,00	$0,\!17$	$0,\!19$	0,33
Mix 2	10	0,00	$0,\!21$	0,20	$0,\!28$

Series 2 Absorption, A, accumulated  $[kg/m^2]$ , Mix 2 and 3, CEN/TS 12390-9 Test

Table 27: Absorption, Series 2

Table 27 show that absorption levels are higher in the non-air-entrained concrete, compared to the air-entrained samples. . Comparing Table 27 with the scaled material table, Table 25 it shows that the highest scaling also produces the highest absorption. This supports Jacobsen and Sellevolds conclusion that there is a good correlation between scaling and absorption.

Table 27 shows that sample 3 is displaying a significantly lower absorption value compared to the other non-air-entrained fly ash concrete samples. It also shows that sample 7 displays a considerably higher absorption than any of the other air-entrained fly ash concrete samples. Table 16 also shows that of all the samples, only sample 8 has a lower absorption from the 14th cycle to the 28th cycle.

Figure 15 and 16 on the next page shows the absorption of the non-air-entrained and the airentrained concretes compared to the mean value of the entire series.



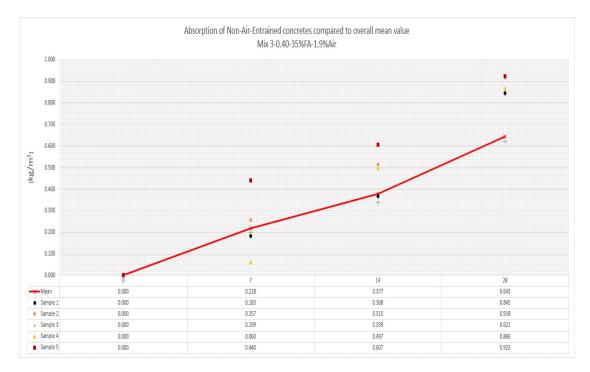


Figure 15: Absorption, 35% FA concrete, non-AEA



Figure 16: Absorption, 35% FA concrete, AEA

Figure 15 shows that all samples which was not air-entrained, except for sample 3, is above the mean value for the whole set. Figure 16 shows that all the air-entrained concrete samples in this set is below the mean value of the entire set. The exception being sample 7, which has been previously mentioned.

A comparison of the absorption of the samples with the scaling has been performed and this is shown in Figure 17 below.

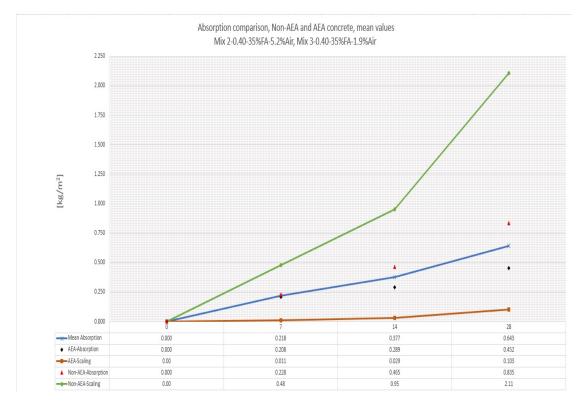


Figure 17: Correlation of Absorption and Scaling

Figure 17 shows that the increased scaling in the non-air-entrained concretes creates a larger absorption in the same samples. This correlation is also evident in the air-entrained ones where the slightly increased level of scaling increases absorption values as well.

Figure 17 shows that the scaling is substantially larger for the non-air-entrained fly ash concretes compared to the air-entrained. Almost 20 times larger scaling compared to twice as large absorption.

## 4.5 Dilatometry

This section will go into detail about the results and experiences collected during the dilatometry pilot experiment. The goal was to be able to develop a setup that would allow dilatometry samples to run alongside regular CEN/TS 12390-9 Salt Frost Scaling test. The experiment was divided into three parts, which can be found in Chapter 3.8, and are briefly summarized below:

- 1. Part 1 Blank test. Testing the invar frames and the freeze/thaw chamber for eventual problems related to shaking and other distortions, upsetting results.
- 2. Part 2 Prepare and test the system rig with a concrete sample. The sample test surface will be exposed to freezing/thawing cycles whilst checking dilatometry simultaneously.
- Part 3 Full scale CEN/TS 12390-9 prepared sample, with with a 3mm solution layer on the test surface, running alongside a freeze/thaw test. A full dilatometry test inside a CEN/TS 12390-9 Slab test.

There will be a four day dilation comparison between part 2: "concrete test surface exposed to chamber freeze/thaw cycle" and part 3: "CEN/TS 12390-9 prepared sample with 3% NaCl solution on the test surface exposed to freezing/thawing".

#### Part 1 - Blank Test and Rig Testing

The main goal of Part 1 was to diagnose eventual difficulties with running sensitive equipment in the freeze/thaw chamber. The two samples, one invar rod and one steel rod, remained untouched for nigh on 24 hours in order to see if the chamber would provide any problems for future testing. The result of the measurements can be seen below in Figure 18.

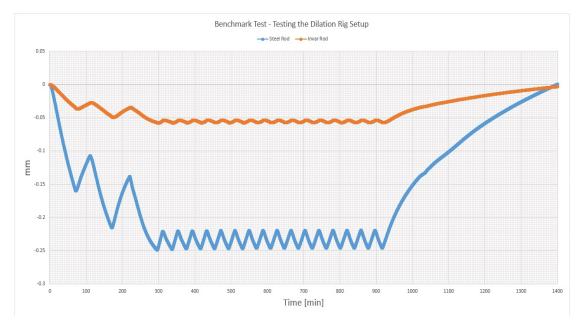


Figure 18: Part 1 Dilation Results

Figure 18 shows that the invar rod does not contract nor expand as much as the steel rod. Furthermore there are smaller oscillations from the invar rod compared to the steel rod. Another important aspect shown by Figure 18 is that the dilation follows the air temperature of the chamber in a strict manner; expanding when the chamber is thawing and contracting when it is freezing. This makes sense considering it is the temperature that influences the contraction/expansion of the materials used.

The clear readings also show that the rig is not exposed to shaking or disruption during testing. The sensitivity of the equipment would pick up if the system was being distorted in any way during testing. This shows that the rig is stable while testing and it can be tried on a proper concrete sample.

#### Part 2 - Concrete Sample Exposed to Air Test

The second part was a close to four day test of the dilatometry of a concrete sample compared to the iron rod sample. The main goal was to check the influence the temperature would have on a concrete sample, if the rig would be destabilized/disrupted during testing and test for uncertaintites in with the chamber. In short it was a continuation of **Part 1**, replacing one of the rods with a concrete sample.

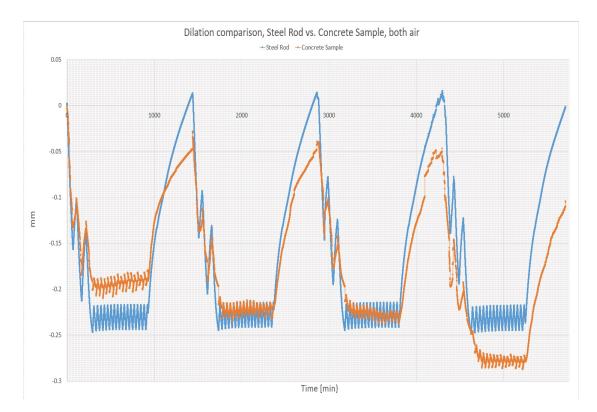


Figure 19: Part 2 Dilation Results

Figure 19 shows that the oscillation pattern of the stainless steel and concrete are similar and highly correlated to the warming/freezing of the chamber. As was the case in Part 1, Figure 18. The other striking piece of information is how the concrete is gradually contracting more and more during the freeze/thaw testing, eventually having a contraction close to -0, 30mm. Figure 19 also shows that the oscillations of the concrete sample are smaller than the ones for the steel rod.

Concerning the system setup there seems to be some distortions in the final stages of the third cycle and the beginning of the fourth, seen as jumps in the readings. This has probably been influenced by entering and leaving the chamber while checking on the CEN/TS 12390-9 tested samples that are in the freeze/thaw chamber at the same time.

The setup seems to be reliable, and the concrete sample delivers clear readings and good data regarding itself. While the rig itself is stable and not influenced at all by its surroundings. The strain/temperature relation for Part 2 can be found in Appendix D.

### Part 3 - CEN/TS 12390-9 Test

The last test is a fully prepared sample, preparation cycle is found in Chapter 3.8, with an insulation box and a 3mm solution layer while checking the dilatometry of the sample. It became evident that when producing the sample, the invar studs were not long enough not create sufficient hold between the socket and the dilation measurer. This meant that the sample would need to be slightly altered and a support device was placed underneath the sample to hold it in place. The rig used is shown in the picture below:



Figure 20: Rig used in Part 3

From Figure 20 it is seen that the insulation has been carved out around the invar stud in order to create a proper attachment between the dilation device and the invar stud. The goal of the experiment was to see if it was possible to run a proper sample through a CEN/TS 12390-9 test while also monitoring the dilation of the specimen.

Figure 21 below shows the dilation measurement for the first 4 days of the Part 3 test. It also displays the correlation of the dilation to the air and water and if solution on the test surface is well within the CEN/TS 12390-9 limits:

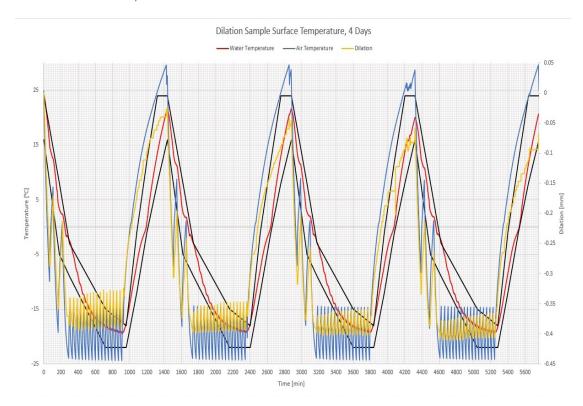


Figure 21: 4 Day Dilation. Correlation graph

Figure 21 shows that the sample follows the freezing/warming of the chamber whilst also slowly contracting more and more during the test period. It is also shows that the amplitude of the oscillations are gradually decreasing during the four day period displayed.

Comparing the water temperature in Figure 21 with the one in Figure 9 shows that the dilatometry sample is doing extremely well, even having lower oscillations than the calibration samples. It also shows that the water solution temperature is well within the limits set by the CEN/TS 12390-9 standard.

Figure 21 shows that when the solution temperature is ascending between -5 to  $+5^{\circ}C$  the dilation becomes more irregular.

Figure 21 readings also show that there are no issues with the dilation device equipment when the chamber is not being disturbed in any way. The drop in the third cycle would indicate that some disruption has occured at this point. It also seems to have lowered the peak point, which before that time was gradually decreasing.

Both correlation graphs for the 13 day test series can be found in Appendix D.

#### Part 3 sample compared to Part 2 sample

One of the main goals is to compare the results of Part 2 with Part 3. Figure 22 below shows the dilation of Part 2 compared to Part 3:

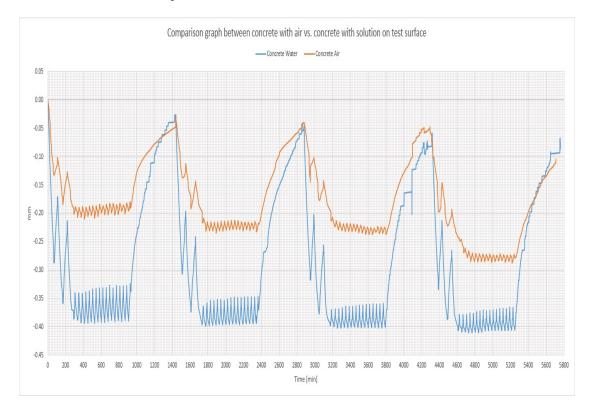


Figure 22: Dilation comparison; Part 2 vs. Part 3

Figure 22 shows that the sample with salt solution on the test surface dilates considerably more, close to twice as much, than the sample only exposed to air. The oscillations of the sample exposed to both air and salt solution is also of greater magnitude than the one for the Part 2 sample. Figure 22 shows that the fluctuation of the dilation depends on the external air, whilst the amplitude of the oscillations depends on the solution temperature.

Figure 22 shows that, over time, the difference in dilation is decreasing between the samples and that the Part 2 sample is more prone to inreased contraction compared to the sample in Part 3. The CEN/TS 12390-9 prepared sample also has a gradual increase in contraction, but the contraction increases more gradually for this sample.

An important aspect to investigate regarding dilatometry of a sample is the strain/temperature curves the freeze/thaw cycles exhibits. This gives the clearest indications as to whether or not the internal strains in the concrete surpasses the strain capacity of the concrete. Normally a blank run would be run beforehand to correct the influence of the invar stude used in the samples, but this has not been done in this test. Figure 23 below illustrates this strain in comparison to the temperature for all 13 dilatometry cycles performed.

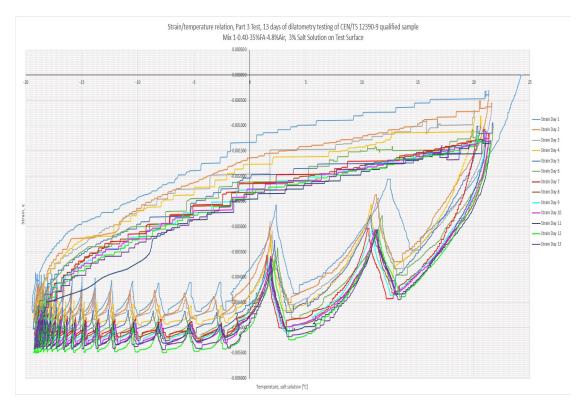


Figure 23: Strain/Temperature relation, Part 3

Figure 23 shows large oscillations during the prefreezing period and many small oscillations after the sample has passed the freezing point,  $\approx -4^{\circ}C$ , for salt solution. Comparing the strain values with Figure 21 shows that the strain oscillation during the freezing period from +20 to -20°C coincide very well with the air fluctuation.

Figure 23 shows that the cycles are gradually becoming more similar as the duration of the test increase. The value of the strain at the end of one cycle is approaching the strain value at the start of a new cycle.

Figure 23 also shows that the sample gradually increases its negative dilation. It contracts, which coincides well with Figure 21, where the contraction is gradually increasing as well.

Other points of interest is to check the thermal expansion coefficient of the cycles from Figure 23. This is done by creating a regression line when sample is thawing between 0 to  $+20^{\circ}C$  and extrapolate it to cover the tested temperatures. The *Prefreeze* phase, +20 to  $0^{\circ}C$ , should be used to create the regression lines, but because of the nature of the strain, see Figure 23, it is not possible to create usable regression lines in this part.

It is also necessary to check strain losses during testing. The two important parameters regarding strain loss are: the residual strain,  $\Delta r_i$  in Figure 24 and the strain deviation from where the thawing cycle begins,  $\Delta \varepsilon_{thawing,i}$ , and the regression line. Figure 24 below illustrates these parameters for cycle 1, 6 and 13.

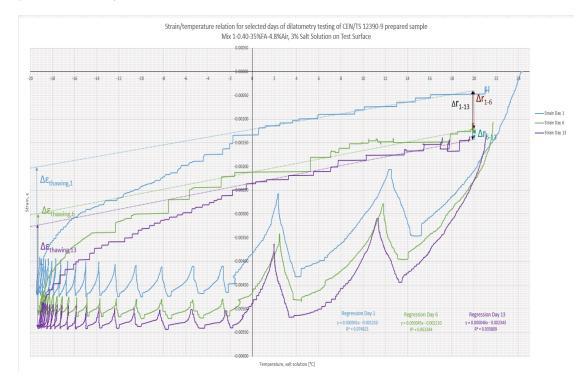


Figure 24: Strain/Temperature Regression for 3 selected cycles

From Figure 24 Table 28 can be produced:

Cycle	Υ	CTE	${\rm Y}(\approx -19)^1$	Y(20)	$\Delta \varepsilon_{thawing,s}$
1	0,000041x - 0,001210	$4,1 \times 10^{-5}$	-0,002010	-0,000390	-0,002627
6	0,000045x - 0,002110	$4,5 \times 10^{-5}$	-0,002968	-0,001210	-0,002050
13	0,000046x - 0,002343	$4,6 \times 10^{-5}$	-0,003227	-0,001423	-0,002143

Strain/Temperature Characteristics Table, Mix 1-0.40-35%FA-4.8%Air

<sup>1</sup> The lowest Temperature for the sample before thawing begins.

Table 28: Dilatometry Strain/Temperature Characteristics

The values for the residual strains are:  $\Delta r_{1-6} = -0,000820$   $\Delta r_{1-13} = -0,001033$  $\Delta r_{6-13} = -0,000213$ 

Table 28 shows that the thermal expansion coefficient is between 4,1 to  $4,6 \times 10^{-5}$  which is above the estimated value concrete has, which is around 0,8 -  $1 \times 10^{-5}$ .

The comparison between the regression line and the values before thawing starts to occur in the sample shows that we have losses above 20‰in the start of thawing phase of the concrete; a very high value. Figure 24 confirms what Figure 23 that the strain cycle seems to contract more over time.

The residual values also show high strain losses from cycle 1. From cycle 1 to cycle 6 the loss is  $-8.2 \times 10^{-4}$  [mm/mm]. Which is a considerable loss of strain. The residual value between cycle 6 and cycle 13 shows that the loss is  $-2.1 \times 10^{-4}$ , confirming that the residual losses are decreasing between the cycles the longer the testing progresses.

From Figure 24 and Table 28 there are no clear signs that internal damages have occured during the test period.

# 5 Discussion and Conclusions

This Chapter will consist of a discussion and conclusion regarding the results found when experimenting with the different preparation systems and the dilatometry pilot project.

## 5.1 Discussion

Figure 9 point 2 shows an indentation in the ascension of the temperature, appearing at approximately -5 to  $+5^{\circ}C$ . This might have something to do with the chemistry when salt solution is thawed and approaching zero degrees, slowing the temperature growth in the solution.

The XtremFix adhesive seems to create very strong bond forces between the concrete surface and the adhesive, eventually leading to the preparation systems destruction.

The samples struggles with leakage over time, which is related to the capillary suction of the duct tape. The problem has been prevalent in all the material tests, as duct tape is used over the butyl tape edge to ensure that the scaled material does not stick to the butyl surface.

The small  $100 \times 100 mm^2$  samples have a greater tendencey to scale along the transition zone between the adhesive and the concrete side. The larger  $150 \times 150 mm^2$  samples have scaling more evenly distributed across the test surface.

The sample cycles, see Appendix C shows that the samples at times are outside of the limits set by the CEN/TS 12390-9 standard. This is probably due to many different reasons such as:

- polyester hat on the sample is loose, letting air into the sample evaporating the salt solution.
- There is a lack of alt solution in the sample; the sample is close to SSD or dry.
- The thermocouple has corroded and will not measure until it is fixed.
- The thermocouple is not touching the concrete test surface anylonger.

The cycles therefore also show when maintenance should be performed on the samples and might also indicate that it is necessary to refill the samples.

The samples in Material Test 2 scaled a decent amount in the interval between 48 to 56 cycles although the samples were most likely SSD for most of that period. The reason for this is unknown, but it might be caused by osmosis within the top layers of the concrete. That the gradual build up of pressure over time increased the amount of scaling material in this interval.

The absorption of sample 3 and 6, Material Test 2 - Series 1, in Table 18 seems to have influenced the transit time across the sample. Both of these samples exhibit significantly higher transit velocity across the sample than at the beginning of the test. This makes sense as water travels faster through water than air. The higher absorption might also have made these samples more prone to internal damages as there are more water in the pores that can freeze.

The UPV values collected after the 28th cycle in Table 18 is in general a lot higher than the others collected for almost all samples. This might have been caused by self heal of the concrete samples from cycle 14 to cycle 28. Correlating this with Table 13 shows that almost every sample in the test was SSD, which might have started the self-healing process.

The absorption of sample 7 from Series 2, see Table 27, has most likely received some faulty readings. This is because the absorption values does not match the UPV, nor the scaling, see Table 23 and Table 25. Research regarding absorption from Jacobsen and Sellevold, [5], shows that high absorption is related to increased scaling values, yet this is not the case for this sample. The transit velocity has not increased, although water travels faster through liquid than through air.

The invar frames used in the dilatometry pilot project is not made for horizontal dilatometry testing, so the system rigs used in this pilot project are not specifically designed for the job. This could have influenced the results. Furthermore, there is uncertainty of the calibration regarding the dilation measuring equipment as this was not done before the test started.

Figure 21 shows that the sample seems to create abruptive patterns, especially when getting closer to the -5 to  $+5^{\circ}$  threshold. What this indicates is uncertain, but the dilatometry readings have not been obstructed or tampered with during testing.

Figure 23 shows that there are large differences between the different cycles, possibly caused by hysteresis in the dilatometry registering equipment.

Strain day 11 from Figure 23 shows a pattern that is very non-linear in the beginning of the thawing phase (-20 to  $0 \,^{\circ}C$ ). According to [11] this might be cause by internal damage, although the other evidence collected does not support this.

Whilst all the dilatometry tests show that the dilatometry follows the external air temperature of the KylCity freeze/thaw-chamber, it seems to be the solution on the test surface that creates the largest amplitudes in the oscillations, which is odd seeing as the fluctuation in the water temperature is so small during the freezing part (0 - 20  $^{\circ}$ C). Maybe it is related to the constant strain the solution is exerting on the sample test surface.

## 5.2 Conclusions

Overall the experiments show that it is possible to run absorption, UPV and scaled material testing for one sample when using the preferred preparation system, essentially creating three tests in one. The preferred preparation systems, that yields the best results, replacing key points 2 and 3 in Figure 1, is a combination of Sitko Elastic 605 butyl tape and either SikaFlex 11FC and Casco Marin og Teknikk Fug og Lim. The preferred preparation system is also very repeatable, time saving and cost-effective.

The samples perform very well in the preferred calibration cycle, with samples well within the limits set by the CEN/TS 12390-9 standard for most of the test period during for the duration of every 7 day cycle period.

Scaled material values from Material test 2 shows that all the large  $150 \times 150mm^2$  samples have scaled above the 1  $kg/m^2$  threshold set by Table 2. Of the  $100 \times 100mm^2$  samples two out of six samples that made it to the 56th cycle are also above the limit. The four other samples are also very close to failing. The mean value of the scaling gives the small  $100 \times 100mm^2$  samples a scaled material value of  $S_{56} = 0.950 \ kg/m^2$ , right below the threshold.

Scaling measurements in Series 2 show that the air-entrained fly ash concretes scale significantly less than their non-air-entrained fly ash concrete counterparts. All samples with air-entrainment displayed a *Good* scaling rating, or better, based on Table 2, whilst all non-air-entrained concretes are above the *Acceptable* limit.

The leakage problem in the samples was mainly caused by capillary suction of the duct tape, which is fixed by spreading adhesive over the duct tape, stopping the water from entering the small air voids.

Absorption measurements from Series 1 show that samples that do not use a rubber sheeting on the bottom will absorb substantially larger amounts of water than samples that use rubber sheeting material to cover the concrete bottom. Absorption measurements from Series 2 show a clear correlation between the scaled amount of material and the absorbed level of water. Increased damage on the concrete test surface leads to increased absorption values.

UPV shows no clear signs of internal damages occuring. Neither does the dilatometry.

The dilatometry in Part 3, see Figure 21, shows strong correlation to the external air imposed by the KylCity freeze/thaw-chamber. Figure 22 shows that the salt solution imposes greater oscillations in the measurements than external air alone, doubling the dilation and increasing the amplitude of the strain oscillations.

The rigs used for dilatometry testing all proved adequate for one time testing, and the KylCity freeze/thaw-chamber does not influence the readings in regards to shaking or other forms of distortion.

## 5.3 Future Research

Research the influence the strength the adhesives have on the concrete sides and the impact of adhesives on absorption, scaling and UPV results.

Run dilatometry and CEN/TS 12390-9 freeze/thaw test on fly ash concrete samples that have been subjected to water penetration using different pressures.

Running blank runs in a more controlled environment and improve horizontal testing of concrete samples. Follow up with CEN/TS 12390-9 qualified samples in the controlled and tested environment.

A digital image correlation (DIC) pilot project to monitor the test surface of a concrete sample when running a CEN/TS 12390-9 freeze/thaw test.

Start a pilot project regarding the "warping box" for concrete samples with-and without airentrainment.

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## A Concrete materials and specifications

This appendix will contain further information regarding the concrete used in Material Test 1, Material Test 2 and the AEA vs. Non-AEA concrete. Information regarding compression strength, air void analysis, as well as the product data sheets of the different constituents- and admixtures used in the concretes can be found in this appendix.

## Material Test 1 and 2

The concrete used in both of these Material Tests were of similar make, cast by Andrei Shpak on the 31st of October 2016 and had been lying submerged since casting before being used in the experiments in this thesis. Below, tables regarding the aggregates and the compression strength are found. These tables are followed by a table of general information regarding some important values for both the cementitious materials as well as the aggregates.

## Data for coarse and fine aggregates

Distrib	Distribution of coarse and fine aggregate							
Vol [%]	41,479	58,521	100,000					
Sieve, [mm]	NF8-16	Ramlo 0-8	Total					
32	100,000	100,000	100,000					
22,4	100,000	100,000	100,000					
16	$92,\!597$	100,000	96,929					
11,2	33,713	100,000	72,505					
8	5,087	99,500	60,338					
4	1,405	89,700	53,076					
2	1,329	70,400	41,750					
1	$1,\!139$	$51,\!400$	$30,\!552$					
$0,\!5$	0,873	35,300	21,020					
$0,\!25$	$0,\!645$	21,300	12,733					
0,125	$0,\!456$	9,000	5,456					
0,063	0,000	$2,\!400$	1,405					
Bottom	0,000	0,000	0,000					

Distribution of coarse and fine aggregate

Table A29: Aggregate distribution

### **Compression Capacity**

The data showing the compression strength of the concretse used in Material Test 1 and 2 and the AEA vs. Non-AEA test are given in the tables before

	Con	pression Stre	ength, Mater	ial Tests, $f_{ck}$ ,	$_k, [MPa]$				
Time	$1 \mathrm{~day}$	2 days	$7 \mathrm{~days}$	28 days	56  days				
Typical values	15	24	37	55	-				
TC-2016-0072	13,0	21,4	37,4	50,3	57.3				
TC-2016-0076	$15,\!4$	$25,\!5$	38,5	$53,\!9$	-				

Table A30: Compression Capacity of Concrete, Material Test 1 and 2  $\,$ 

The compression strength of the concrete used in the AEA vs. Non-AEA test was tested after 6 months. The samples were cubes of  $100 \times 100 \times 100 \text{ mm}^3$  and the mean value was calculated from three samples tested.

Compression	Compression Strength, AEA vs. Non-AEA, $f_{ck}$ , $[MPa]$							
Sample 1	Sample 2	Sample 3	Average					
$61,\!66$	$75,\!85$	75,74	71,1					

Table A31: Compression Capacity Series 2

# Constituents used

Below follows a general table for the constituents used in the concretes used in this thesis.

Constituents, general table							
Aggregate	Density, $[kg/m^3]$	Absorption, $\%$					
Nordfosen 8 - 16	2680	0,6					
Ramlo 0 - 8	2700	0,8					
Cementitious		K-factor					
Norcem Anlegg FA	3020	1					
Fly Ash	2300	0,7					
Admixtures		Dry content [%]					
Rescon Dynamon SX 23	1050	23					
Mapeair 25	1000	$0,\!4$					

Constituents.	general	table
Constituents.	general	Lanc

Table A32: General information table

0

The following data sheets are further information regarding the constituents used in this concrete.

ANLEGGS	EMI	ENTFA	
CEM II/A-V AST REVISION JUNE 2015 he cement satisfies the requir ement CEM II/A-V 42.5 N.	ements acco	rding to NS-EN 197-	1:2011 to Portland-fly ash
Properties		Declared values	Requirements according t NS-EN 197-1:2011
Fineness (Blaine m/kg)		390	
Specific weight (kg/dm <sup>3</sup> )	Sec. Sec.	3.02	
Soundness (mm)	1	1	≤ 10
Initial setting time (min)		165	≥ 60
	1 day	15	
Compressive strength (MPa)	2 days	24	≥ 10
	7 days	37	
	28 days	55	≥ 42.5 ≤ 62.5
Sulfate (% SO <sub>3</sub> )	1000	≤ 3.5	≤ 3.5
Chloride (% Cl⁻)		≤ 0.085	≤ 0.10
Water soluble chromium (ppm Cr <sup>6</sup> +)		≤2	≤ 2 <sup>1</sup>
Alkalies (% Na <sub>2</sub> O <sub>ekv</sub> ) <sup>2</sup>		0.6	
Clinker (%)		83	80-94
Fly ash (%)		17	6-20



#### FLYGEASKE

www.norcem.no

Norcem AS ivaretar salg av flygeaske til sement- og betongproduksjon. Flygeasken er sertifisert i overensstemmelse med kravene i NS-EN 450-1, klasse A.

Flygeaske er et bearbeidet restprodukt fra kull brukt i kullkraftverk. Flygeaske er silikatholdig og er et pozzolan som sammen med sement og vann gir en tettere betong. Kombinert med sement har flygeaske vært brukt i Norge siden 80-tallet. Norcem FA-sementer inneholder flygeaske.

C	DEKLARERTE VEI	RDIER
Flygeasken er sertifisert i overen	sstemmelse me	d kravene i NS-EN 450-1:2012, klasse A.
Egenskap	Deklarerte verdier	Krav i henhold til NS-EN 450-1
Glødetap (%)	≤ 5,0	Tilfredsstiller kravene gitt NS-EN 450-1
Klorid (% Cl <sup>-</sup> )	≤ 0,10	Tilfredsstiller kravene gitt NS-EN 450-1
Sulfat (% SO <sub>3</sub> )	≤ 3,0	Tilfredsstiller kravene gitt NS-EN 450-1
Fritt kalsiumoksid (% fri CaO)	≤ 1,5	Tilfredsstiller kravene gitt NS-EN 450-1
Reaktivt kalsiumoksid (% reaktiv CaO)	<u>≤</u> 10	Tilfredsstiller kravene gitt NS-EN 450-1
Partikkeldensitet (kg/m³)	2300	Dekl.verdi +/- 200 kg/m³
Øvrige kjemiske og fysiske parametere		Tilfredsstiller kravene gitt NS-EN 450-1



#### A CONCRETE MATERIALS AND SPECIFICATIONS



#### **PRODUCT DESCRIPTION**

Dynamon SX-23 is a very effective superplasticizing admixture based on modified acrylic polymers. The product is part of the Dynamon System. This system is based on DPP technology (Designed Performance Polymers) developed by Mapei, where the properties of the admixtures are custom built for different kinds of concrete. The Dynamon System is developed from Mapei's own composition and production of monomers.

#### APPLICATION AREAS

Dynamon SX-23 is specially designed for ready-mix concrete production and can be used to increase the workability for all types of concrete and/or reduce water consumption.

Some special areas of application are:

- Watertight concrete that requires high or very high strength and strict requirement to concrete durability in aggressive environments.
- · Concrete with high workability requirements.
- Self-compacting concrete with somewhat longer open times. If necessary, this type of concrete can be stabilised with a viscosity enhancing admixture, such as Viscofluid or Viscostar.

- Frost resistant concrete in combination with an air entraining agent like Mapeair. The type of air entraining is chosen based on the properties of the other ingredients.
- Concrete for flooring to create a more pliable concrete with greater workability. High dosages of the product and low temperatures can lead to a certain retardation of the concrete.

Dynamon SX-23 differs greatly from superplasticizing admixtures based on sulphonated melamines and naphthalenes, as well as from first-generation acrylicbased polymers – by having a higher water-reducing effect and a longer open time. The necessary dosage to achieve the desired workability/water reduction will be significantly lower when using Dynamon SX-23, than with older types of superplasticizing admixtures.

The time of closing **Dynamon SX-23** is not so important, but the shortest mixing time is generally obtained when adding **Dynamon SX-23** after at least 80 % of the mixing water is added. It is advisable to do some preliminary testing to obtain optimal utilization of the mixing equipment.





#### PROPERTIES

**Dynamon SX-23** is a water soluble product of active acrylic polymers that effectively disperses cement within the mixture.

This effect can be utilised in three ways:

- To reduce the amount of mixing water, but at the same time maintain the concrete workability. Lower w/c ratio gives increased strength, reduced permeability and improved durability.
- To increase workability compared to concrete with the same w/c ratio. The strength remains the same but ease of placement is improved.
- 3. To reduce both the water and the cement without altering the mechanical strength. Through this method it is possible to reduce costs (less cement), shrinkage (less water) and also the risk of temperature gradients due to the lower heat of hydration. This last effect is particularly important for concrete containing a high percentage of cement.

#### COMPATIBILITY WITH OTHER PRODUCTS

Dynamon SX-23 can be combined with Mapei's other concrete additives, for example accelerators like Mapefast and setting retardants like Mapetard.

The product can also be combined with air entraining agents for production of frostresistant concrete, **Mapeair** products. Selecting the type needed is done based on the properites of the other ingredients.

When combining products, it is recommended that testing is done to obtain the desired effect in the mixture. You may also contact our technical department.

#### DOSAGE

**Dynamon SX-23** is added to achieve the desired effect, strength, durability, workability and cement reduction by varying its dosage between 0.3 and 2.0 % of the amount of cement + fly ash + microsilica.

Higher dosages increase the concrete's open time, which means the time where the concrete can be worked. A higher dosage and a lower concrete temperature will cause some retardation.

Test mixtures using the selected parameters are always recommended.

#### PACKAGING

**Dynamon SX-23** is available in 25 liter cans, 200 liter drums, 1000 liter IBC tanks and in tank.

#### STORAGE

The product must be stored at temperatures between +8 and +35°C, and will retain its properties for at least one year when stored unopened in original packaging. If the product is exposed to direct sunlight, colour variation may occur, but this will not affect the technical properties of the product.

#### SAFETY INSTRUCTIONS FOR PREPARATION AND USE

Dynamon SX-23 is not considered dangerous according to the European regulation regarding classification of mixtures. It is recommended to wear gloves and goggles and to take the usual precautions for handling of chemicals.

For further and complete information about safe use of our product please refer to our latest version of the safety data sheet.

PRODUCT FOR PROFESSIONAL USE

#### WARNING

Although the technical details and recommendations contained in this product data sheet correspond to the best of our knowledge and experience, all the above information must, in every case, be taken as merely indicative and subject to confirmation after long-term practical application: for this reason, anyone who intends to use the product must ensure beforehand that it is suitable for the envisaged application: in every case, the user alone is fully responsible for any consequences deriving from the use of the product.

Please refer to the current version of the technical data sheet, available from our web site www.mapei.com

All relevant references for the product are available upon request and from www.mapei.com

#### A CONCRETE MATERIALS AND SPECIFICATIONS



#### AREA OF USE

Mapeair® 25 1:9 is a surface active agent which promotes the formation of small air bubbles and is used to improve the frost resistance of concrete and mortar. Mapeair® 25 1:9 also gives improved workability and reduces the risk or segregation. The product is usually used in combination with Mapei's plasticising or superplasticising admixtures.

Mapeair® 25 1:9 is based on synthetic tensides and tall oil derivatives.

#### **TECHNICAL CHARACTERISTICS**

Concrete always contains a certain amount of air (1 - 3 %). In order to meet the usual requirements of 4 - 6 % air in fresh concrete, **Mapeair® 25 1:9** is added, which produces smaller and more evenly distributed air bubbles, which leads to improved freeze-thaw resistance.

Air introduced during mixing is transformed into small evenly distributed pores in the presence of **Mapeair® 25 1:9**. These entrained air bubbles also improve the workability and reduce the amount of water required. Increased air content leads generally to a decrease in compressive strength. A general guide is that 1 % of air reduces the compressive strength by 5 %. This is partly compensated for by the reduced need for water and by adding plastic ising and/or superplasticizing admixtures.

Mapeair® 25 will also improve stability during transportation by reducing the risk of segregation for

concrete containing a low volume of fine particles and actively preventing bleeding (transportation of water to the surface of fresh concrete).

#### WORKING INSTRUCTIONS

Mapeair® 25 1:9 is delivered ready for use and can be added directly into the mixer. To obtain an even distribution of air from batch to batch, it is important that Mapeair® 25 1:9 is added at the same stage of the mixing procedure each time.

The dosage required to give the desired air content varies with aggregates, cement type and quantity present. Other additives may also have an influence. It is important that the addition of **Mapeair® 25 1:9** is determined by trial mixing and that the air content in the fresh concrete is checked regularly.

#### DOSAGE

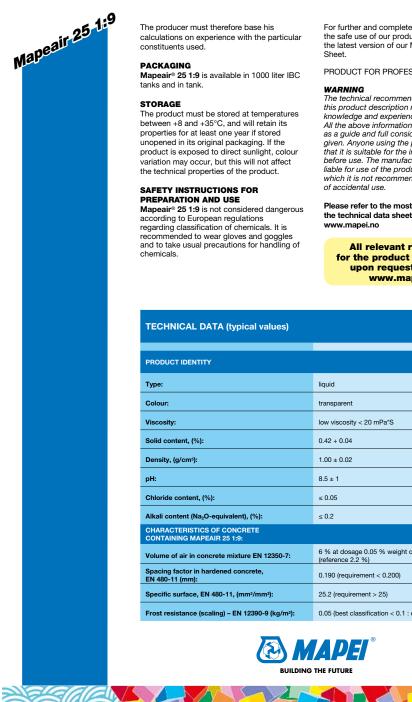
0.5 - 5.0 kg of Mapeair® 25 1:9 pr. m3 of concrete.

#### ATTENTION

Variations in other components in the concrete can greatly influence the formation of air bubbles in concrete. In some cases duration of transport and transportation equipment used can produce variations in air content.

If the mixing time has been too short the total measured air content may increase from production to delivery, whereas in most cases a reduction of air content is observed. Normally this reduction is the result of the release of larger, undesirable air bubbles.





The producer must therefore base his calculations on experience with the particular constituents used.

#### PACKAGING

Mapeair® 25 1:9 is available in 1000 liter IBC tanks and in tank.

#### STORAGE

The product must be stored at temperatures between +8 and +35°C, and will retain its properties for at least one year if stored unopened in its original packaging. If the product is exposed to direct sunlight, colour variation may occur, but this will not affect the technical properties of the product.

#### SAFETY INSTRUCTIONS FOR PREPARATION AND USE

Mapeair® 25 1:9 is not considered dangerous according to European regulations regarding classification of chemicals. It is recommended to wear gloves and goggles and to take usual precautions for handling of chemicals.

For further and complete information about the safe use of our product please refer to the latest version of our Material Safety Data Sheet.

PRODUCT FOR PROFESSIONAL USE

WARNING The technical recommendations and details in this product description represent our current knowledge and experience of the products. All the above information should be treated All the above information should be treated as a guide and full consideration should be given. Anyone using the product must ensure that it is suitable for the intended purpose before use. The manufacturer cannot be held liable for use of the product for purposes for which it is not recommended or in the event of accidental use.

Please refer to the most recent version of the technical data sheet on our website at www.mapei.no

All relevant references for the product are available upon request and from www.mapei.no

PRODUCT IDENTITY	
Гуре:	liquid
Colour:	transparent
/iscosity:	low viscosity < 20 mPa*S
Solid content, (%):	0.42 + 0.04
Density, (g/cm³):	1.00 ± 0.02
oH:	8.5 ± 1
Chloride content, (%):	≤ 0.05
Alkali content (Na <sub>2</sub> O-equivalent), (%):	≤ 0.2
CHARACTERISTICS OF CONCRETE CONTAINING MAPEAIR 25 1:9:	
/olume of air in concrete mixture EN 12350-7:	6 % at dosage 0.05 % weight of cement (reference 2.2 %)
Spacing factor in hardened concrete, EN 480-11 (mm):	0.190 (requirement < 0.200)
Specific surface, EN 480-11, (mm²/mm³):	25.2 (requirement > 25)
Frost resistance (scaling) – EN 12390-9 (kg/m²):	0.05 (best classification < 0.1 : excellent)

# **B** Equipment used and other necessary information

This Appendix contains information regarding some of the laboratory equipment used in this thesis. Information about the KylCity Freeze/thaw chamber, UPV-equipment, the butyl tape application method as well as information regarding the climate room that has been used in this thesis is found in this appendix.

# Freeze/Thaw chamber

The freeze/thaw chamber used for the experiment is a Kylcity freeze-thaw chamber shown in figure B25b:



(a) Close up of the control center



(b) KylCity freeze-thaw chamber

A close up of the control center, the white display, and the two temperature screens are shown in figure B25a. The setup to complete a proper Borås test was calibrated by Ole Petter Vimo in the summer of 2016 as well as a guide on how to complete a test. A user manual on how to use the chamber is not found, so one can be found below.

# User Manual for freeze-thaw chamber

Primarily, it is important to know what the different devices on the chamber do and how they are controlled. There are two displays and one control center. The two displays are on the left and right, respectively, while the control device, produced by Theben, is the white box in the center.

From this point on, Display 1 will be the display on the left when looking at the chamber, see Figure B25a, while Display 2 is the one on the right. The control device, in the middle, will henceforth be referred to as the control center.

Display 1 controls the freezing of the chamber. Display 2 controls the thawing/heating of the chamber. The control center controls when Display 1 and Display 2 are operating and decides therefore when you are freezing and thawing your samples. As such it is only necessary to learn to operate the control center in order to perform a CEN/TS 12390-9 slab test.

#### How to operate the control center:

The control center has four buttons:

- 1. Menu The menu button is pressed to enter the menu, or exit from the current action.
- 2.  $\Leftarrow$  Indicates moving to the left or selecting a value.
- 3.  $\Rightarrow$  Indicates moving to the right or selecting a value.
- 4. Ok The decisions made are satisfactory and you wish to continue.

The most important action to do is to create a new program for when the chamber is supposed to start freezing or thawing. In the control center you follow these steps to create a new program:

1. Press  $Menu \rightarrow 2$ . New Program  $\rightarrow 3$ . Relay On/Off  $\rightarrow 4$ . Numbers of programs available, press Ok  $\rightarrow 5$ . Set time  $\rightarrow 6$ . Date, Mon – Sun, choose start date, Ok  $\rightarrow 7$ . Copy/Save

These are the steps to create a new program, but it does not tell you directly if you are freezing or thawing or when the freezing/thawing will begin or is finished. It is not very intuitive.

The third step, "Relay On/Off", says something about which mode is about to be activated:

- Relay (relè in Swedish) On = Freezing (Display 1 will be turned on)
- Relay (relè in Swedish) Off = Thawing (Display 2 will be turned on)

When "Relé On" is chosen, the chamber will begin to freeze your samples, the opposite happens if "Relé Off" is chosen.

The fifth step indicates which time to start the program during the day. The display will show 0:00 as midnight as the initial setting and it is considered the start of the day, not the end of one. This means that there is no AM or PM system, it uses the 00:00 to 23:59 system.

### Example 1

0:00 Sun, relé On = When Saturday becomes Sunday, not when Sunday becomes Monday. The chamber will start to freeze the samples (Display 1) at 00:00 Sunday morning.

### Example 2

7:30 Sun, relé Off = 7:30 in the morning, Sunday, as you can choose between all the hours of the day. There is no AM or PM system. The chamber, which was freezing the samples, will now turn on heating (Display 2) and warm the chamber again.

# A short tutorial:

- 1. Turn the refrigerator on using the green button
- 2. Check that the red button is on
- 3. Enter the control center and enter the program of your choice, following the guide above. a. Remember to be precise when entering a new program
  - b. Remember that if you want the chamber to run for a long period you can create weekly

cycles by copying the same dates to days after each other using the *Copy* function when creating a new program.

4. Remember have a logging device running during the freezing/thawing to see if the samples corresponds within the limits of the CEN/TS 12390-9 Standard. NTNU frost laboratory uses CatmanEASY.

5. Turn off the chamber using the green button when the test is done.

### A short maintenance guide:

To ensure that the freeze/thaw-chamber performs its best for a prolonged period of time, it will most likely be necessary to perform some general maintenance of the chamber.

Before a test occurs, inspect the floor of the chamber and the shelves thoroughly. The shelves should be removed from the chamber before they are inspected (if possible). It might also be necessary to check the walls as well as the tracks the shelves are placed in. Check for salt deposits or rust spots in all areas before-mentioned.

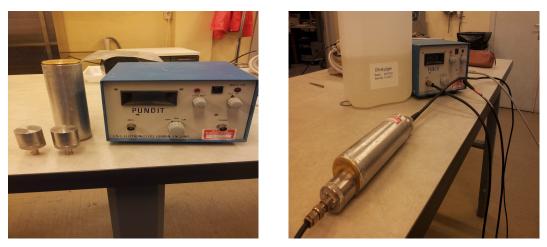
If any salt deposits are found, which is a common problem when he test is run the way it is in this thesis, they are easily removed by using a wet cloth and then drying over with a dry cloth. Clean the surface area until the salt is gone and will pose no further problems.

If rust spots are found it indicates that the maintenance has probably been below par lately and needs to be improved upon. Rust is most commonly found on the shelves and can be prevented with proper maintenance over time and removal of water and salt deposits in the chamber.

It is important to check the chamber frequently, especially during a test period, if the method used is one which causes a lot of condensation. This will increase the risk of problems and will in turn increase the need for maintenance in the chamber.

# Pundit UPV measurer

To measure the ultrasonic pulse velocity a PUNDIT device, from C.N.S Electronics ltd. London, is used. The reason for this is because it runs at a frequency of approximately 50kHz, which is optimal for the test to find ultrasonic pulse velocity through a concrete sample. As indicated by figure B26a, the equipment is dated, but it still does a proper job of finding the velocity through a material when calibrated correctly.



(a) UPV Equipment

(b) Calibration setup

The calibration rod is the large metal cylinder in figure B26a and has a calibration speed of  $26\mu s$ . Calibrating the equipment properly is important to make sure that the results we get through the concrete are consistent. Another important aspect is the gel used on the transmitter and receiver in order to create an optimal surface for sound to travel through transmitter, calibration rod and receiver.

## Application of the Adhesive and Butyl Tape

To ensure proper samples the application of the butyl tape and the adhesive must be done properly. The samples should follow a step by step procedure that goes like this:

- 1. Brush or sandblast every side for a grainy and coarse exterior.
- 2. Wash every sample side to ensure clean surfaces.
- 3. Apply adhesive to one side, and spread it out evenly. Use proper equipment.
- 4. Remove the protective film from butyl tape and place the sample with adhesive side down. Adjust to 2cm gap.
- 5. Apply adhesive to all sides and rotate sample to fasten each side to the butyl tape. For best connection along the edges, tighten lightly along edges by pulling sample towards corner.
- 6. Rotate until every side is covered. Cut of tape using a scissor.
- 7. Lightly press butyl tape in place on every side; push glue over edge of concrete sample. Apply duct tape over butyl dam edge and drag the glue over duct tape to prevent capillary suction.
- 8. Remove excess tape from bottom and apply butyl tape to bottom.

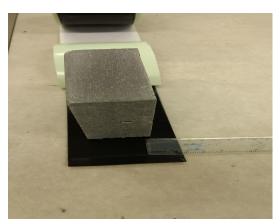
To see this more clearly, some of the application steps are illustrated with pictures below. It is worth noting that this step by step procedure requires practice and experience, and application methods comes down to personal experience and approach.



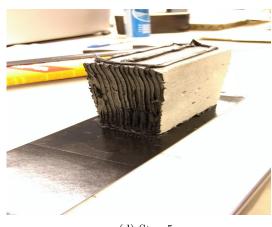


(a) Step 1





(c) Step 4



(d) Step 5



(e) Step 6



(f) Step 6

# Climate Room details

Here follows the details following the climate room used to store samples used in this thesis.

Climate roo										
Climate room in	n the laboratory a	t Konstruk	sionstekk. 3	setups tri	ed to find	a good valu	ue for the evan	oration rate for futu	ire concrete	e specimens.
		e nonser un.	Jensteinut	, secups cri	cutonnu	a 8000 tan				- specificity
Wanted qualiti	es from the clima	te room:								
Evaporation rat	45 ± 15	[g/m <sup>2</sup> h)								
RH:	65	%								
Temperature:	20	degrees C								
Water bowl:										
Area:	0.0324	m <sup>2</sup>								
Mass:	50.8									
	0010	0								
								1.		
Date	Place	Time	dt	dt [h]	mass [g]	A [m <sup>2</sup> ]	d <sub>mass</sub> [g]	Evap. Rate [g/(m <sup>2</sup> h	)]	
	In box w/concret	te								
	samples									
22.09.2016		11:10	0	0	1562.3	0.0324				
23.09.2016		9:12	22h + 2m	22.03	1500.9	0.0324	61.4	86.02		
26.09.2016		11:38	74h + 26m	74.43	1324.9	0.0324	176	72.98		
27.09.2016		10:54	23h + 16m	23.27	1270.8	0.0324	54.1	71.76		
								Way above target!		
	Second shelf, 60	cm								
	above floor									
30.09.2016		9:19	0	0	1677.95	0.0324				
03.10.2016			75h + 31m	75.52	1505.55	0.0324	172.4	70.46		
04.10.2016		14:42	25h + 52m	25.87	1456.75	0.0324	48.8	58.22		
07.10.2016		9:28	66h + 46m	66.77	1336.26	0.0324	120.49			
	0 1 10 1							Inside target, but j	ust barely -	not sufficient
		Same shelf w/crate								
	on side and paper blockade in front									
08.11.2016	blockage in Iron	10:58	0	0	1581.28	0.0324				
11.11.2016			73h + 46m	73.77				40.96		
14.11.2016			73h + 40m 72h + 39m	72.65					8	5
17.11.2016			72n + 39m 71 + 13m	72.05						
17.11.2010		12.30	11 1 1011	/1.22	1300.43	0.0324	51.57	This is inside the ta	arget	
								And close to the go	-	
								of 45.	/ul	8

Figure B28: Climate room specifications

# C Cycle Testing and cycles from Series 1 and 2

In this Appendix, the different calibrations of the cycles of the freeze/thaw-chamber is located. This is to give an indication as to how the different problems in the previous test cycles were dealt with with every incrementation of the test. The different backgrounds used for the four-and seven day test will also be located here.

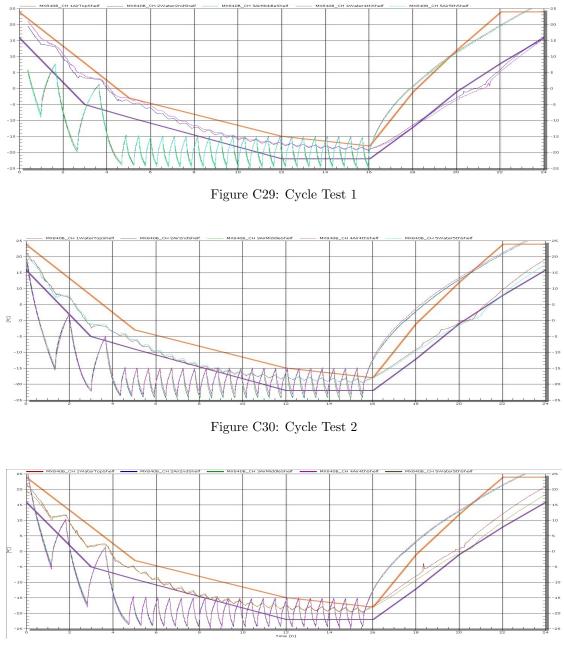


Figure C31: Cycle Test 4

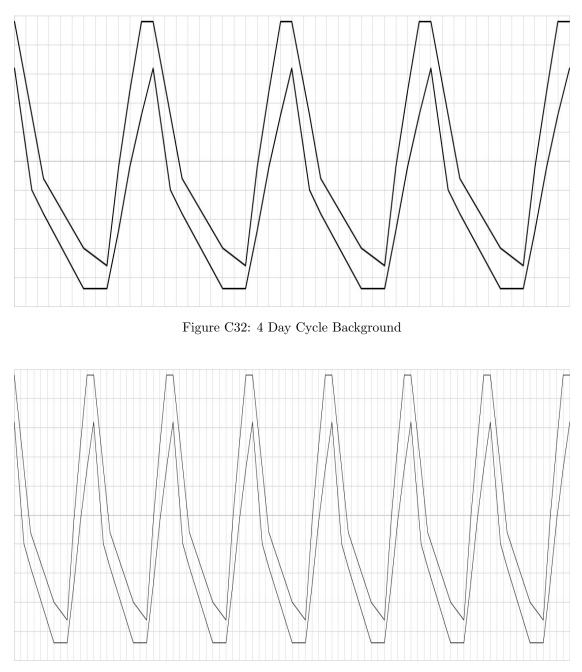
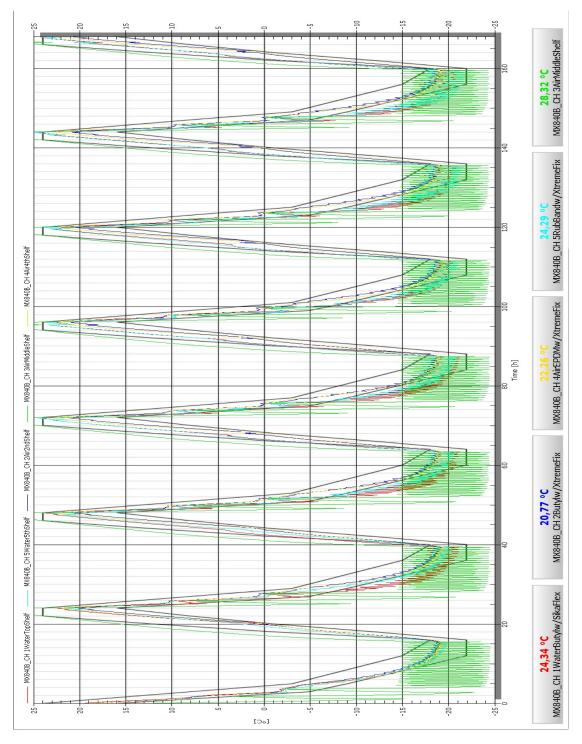


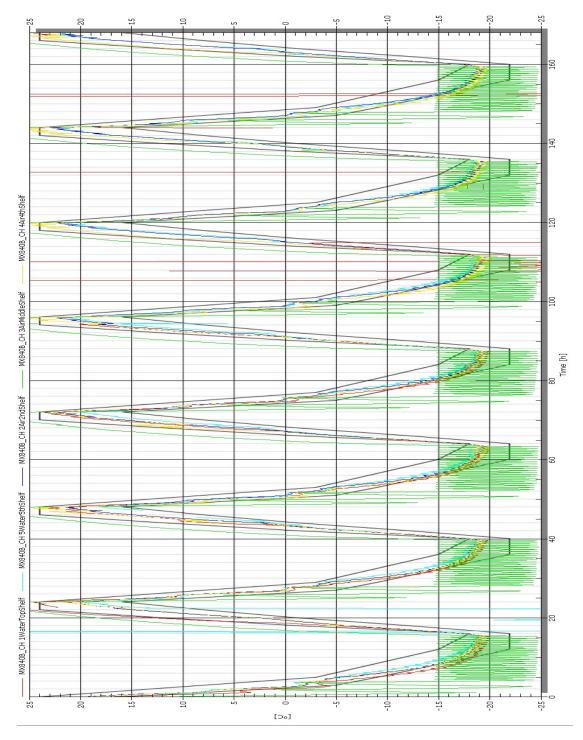
Figure C33: 7 Day Cycle Background

On the next pages follows the cycles from Material Test 2 and Series 2 - AEA vs. Non-AEA. This is to show the development of the samples during the test and also what great help it is to monitor the samples during CEN/TS 12390-9 testing in order to pick up any flaws that might occur during the test.



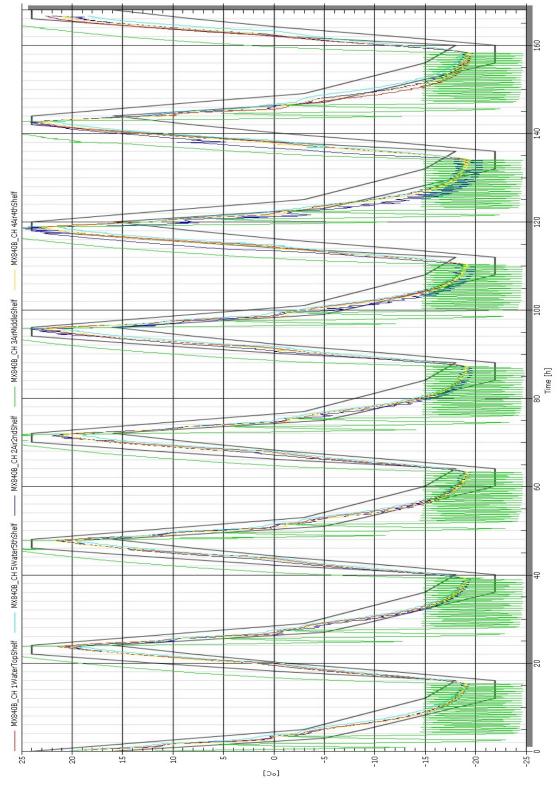
# Material Test 1 Cycle

Figure C34: Material Test 1 Cycle 1, 7 Days



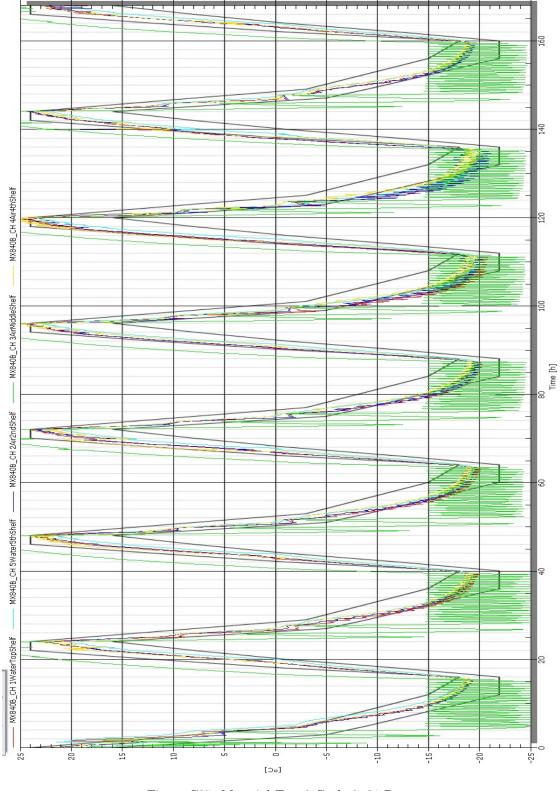
# Material Test 2 Cycles

Figure C35: Material Test 2 Cycle 1, 7 Days



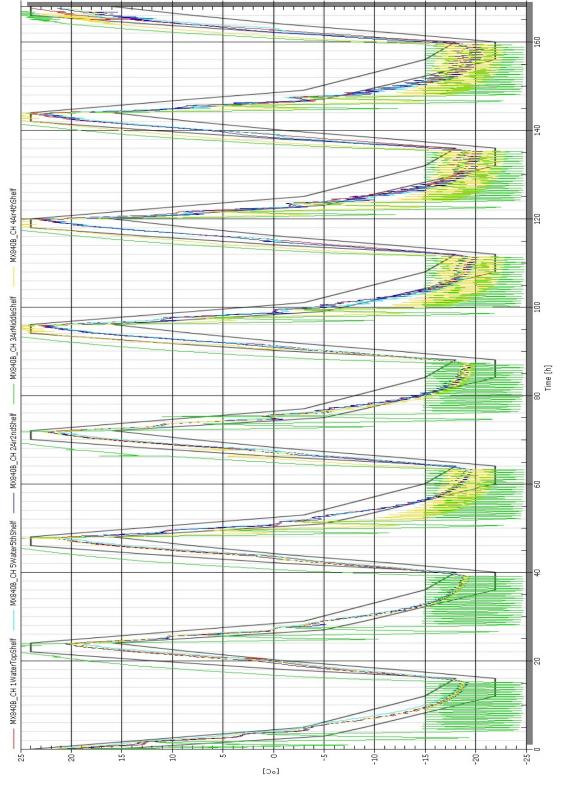


80



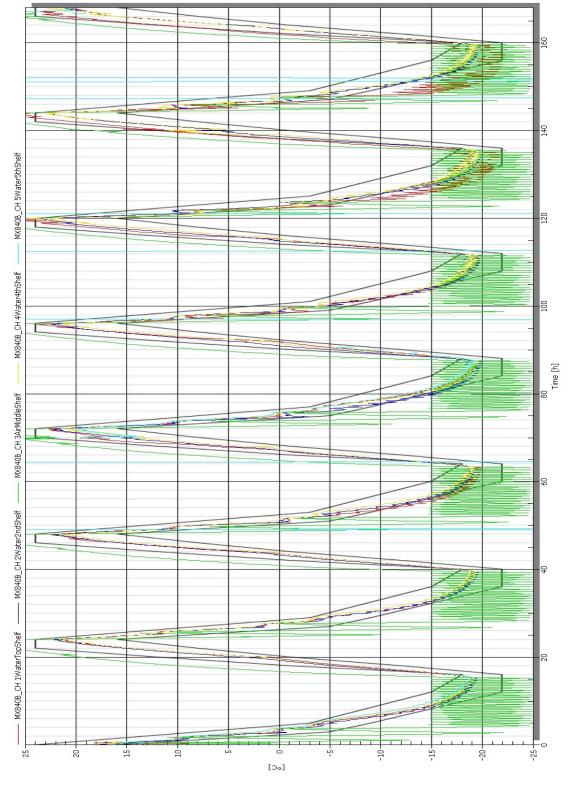


81



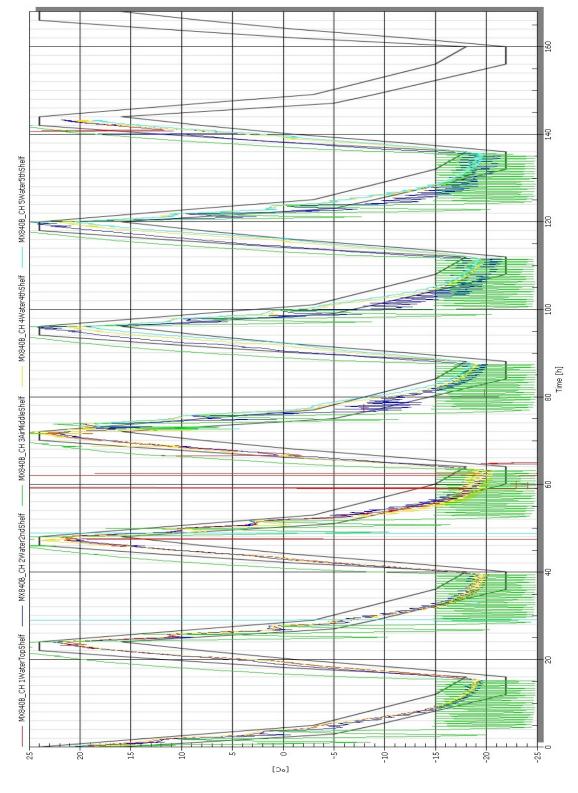


82

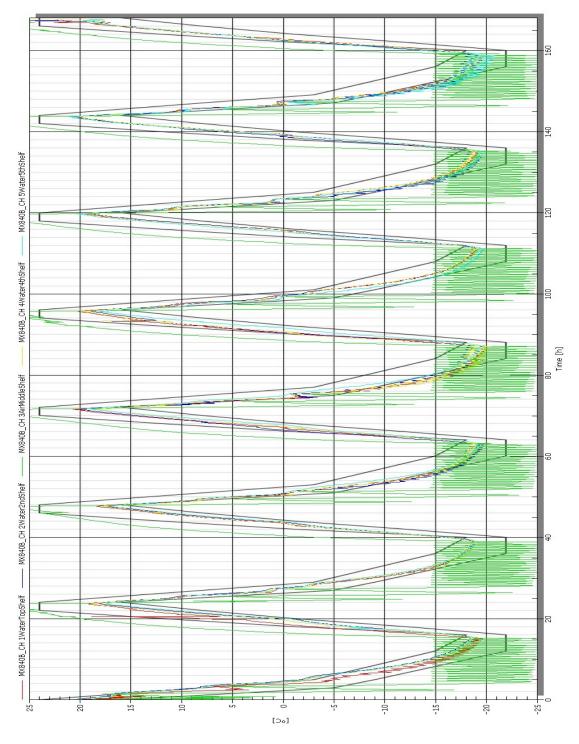




83

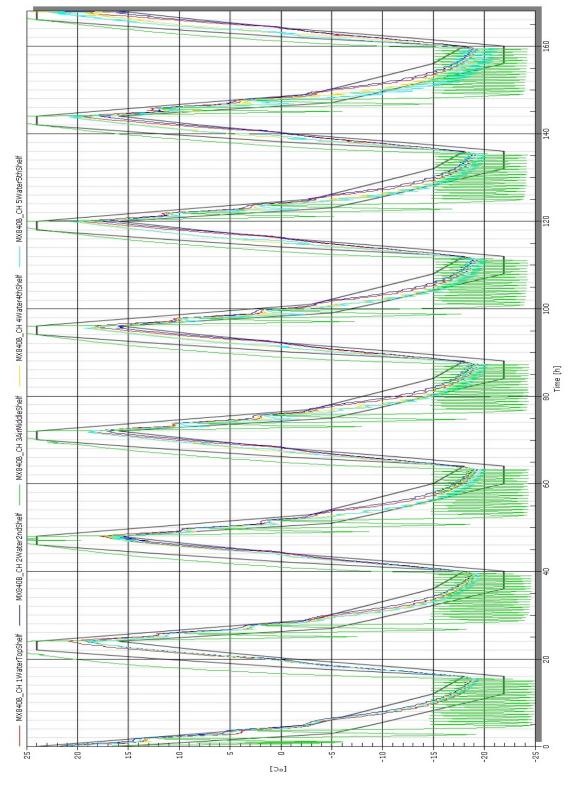




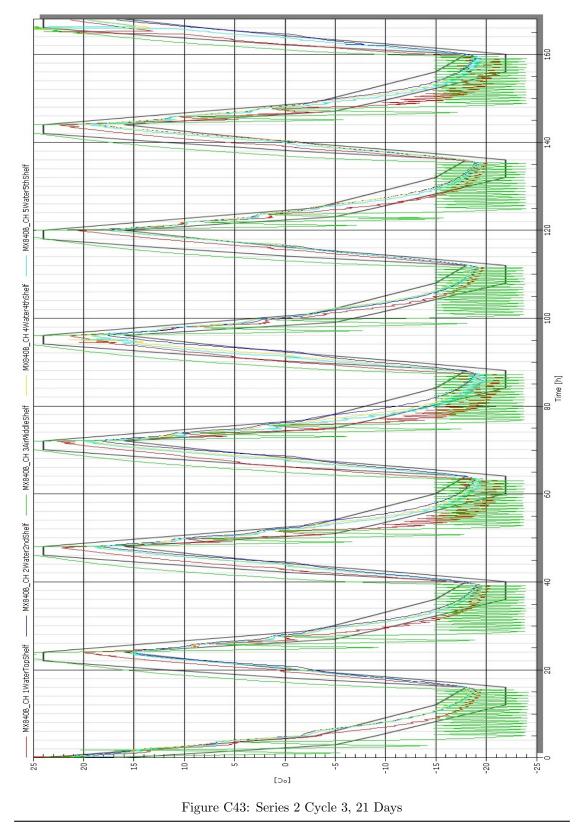


Series 2 - AEA vs. Non-AEA Cycles

Figure C41: Series 2 Cycle 1, 7 Days









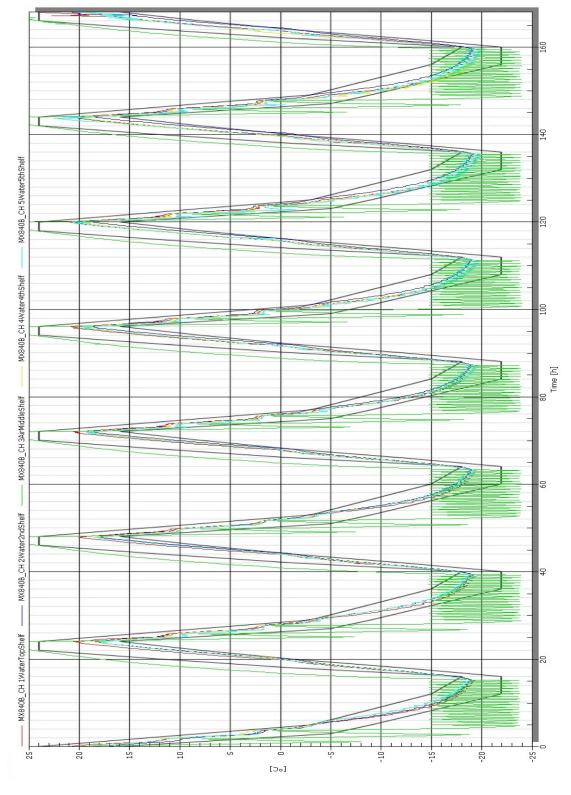


Figure C44: Series 2 Cycle 4, 28 Days

# D Dilation Cycles and dilation setup

This Appendix will include the entire 13 day correlation graph, split into two graphs, to get a clear picture of the development of a dilation test run in the freeze/thaw chamber and its effect on a concrete sample. It will also include the preparation setup used in Part 1 and Part 2 of the dilatometry testing.

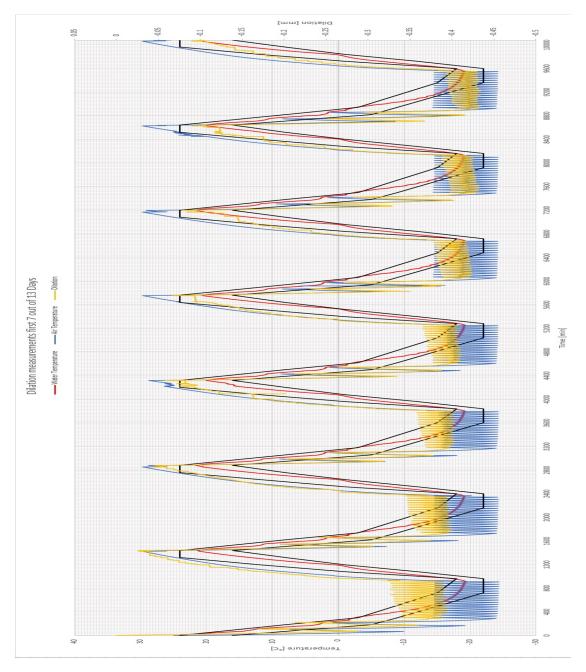


Figure D45: First 7 Days Correlation Graph

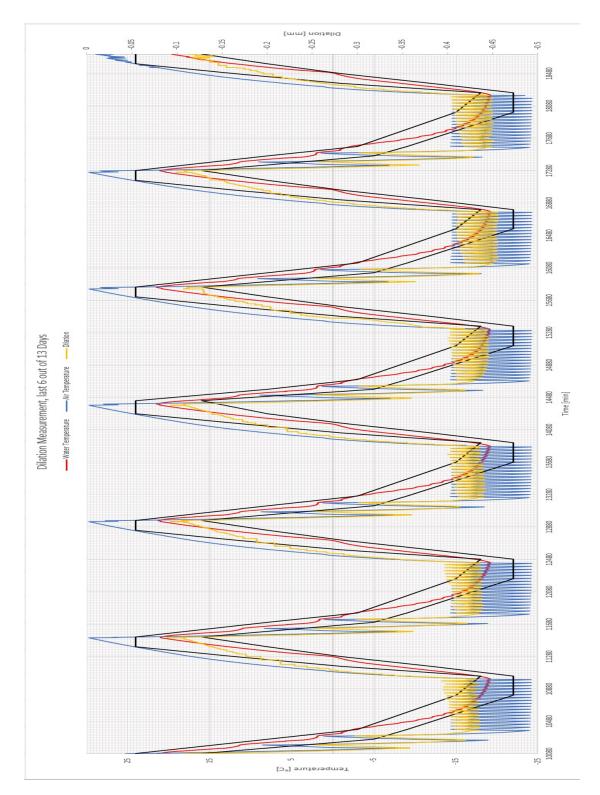
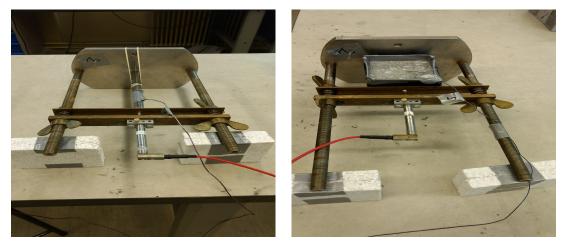


Figure D46: Last 6 Days Correlation Graph

#### Rigging system in Part 1 and Part 2

The following figures show the setup of Part 1 and Part 2 used in the dilatometry test.



(a) Part 1, Dilatometry Setup

(b) Part 2, Dilatometry Setup

Figure D47: Dilatometry setup, Part 1 and 2

From Figure D47a it is possible to see that in Part 1 of the test, rubber bands are used to hold the rods in place. This is a necessary measurement to keep the rods in their sockets because vibrations when entering and leaving the chamber could potentially knock them over. The rubber band did not have any influence on the dilation as the readings were continuous throughout the entire 2 day process they were in the freeze/thaw chamber.

In Figure D47b the setup is pretty much the same, but the only difference is that invar frame 2 now has a concrete sample attached to it, instead of the invar steel rod. The sample does not have any water on the test surface, nor is it encapsulated in insulation like a proper CEN/TS 12390-test. This was because the intent was to see how the dilation would look on a concrete sample that was not CEN/TS 12390-9 prepared and compare it to a sample that was CEN/TS 12390-9 prepared.

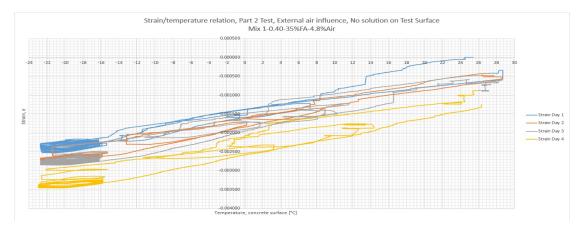


Figure D48: Strain/Temperature relation Part 2 Test

# **E** Sample Details

In this appendix further information regarding the different samples are found. More detailed descriptions regarding each and every sample during the visual inspection from every compelete cycle ran for both Material Test, Material Test 2 and the AEA vs. non-AEA test. This appendix will also include pictures of the samples from the respective tests to show the impact the CEN/TS 12390-9 test will have on samples during the test. It is also worth noting that the assessments for Material Test 1 and 2 are made before the discovery regarding the capillary suction of the duct tape and its effect on the water level of the samples, see Chapter 5. More pictures of all samples can be provided if requested by sending a mail to olechrb@gmail.com

## Material Test 1

Below follows an in-detail description of the nine samples, and one reference sample, ran for one seven day cycle called Material Test 1. All samples followed the preparation procedure from Chapter 3.3.3. If samples were prepared in a different fashion it will be described in the respective section for that sample. The goal is to give in depth information of all samples and shed further light on the decisions made in regards to the material setup and preparation in the future Material Test 2.

# Sample 1, Reference Sample

The reference sample used was already in the climate room, having been there since October 2016, whilst also being from an older batch, cast in 2015. The content of the concrete is unknown. The collection of UPV showed no anomalies and it had the lowest transit times of all the samples 15, both before and after the test. But it increased slightly.



Figure E49: Sample 1, Material Test 1

During the water saturation of the samples, the sample showed no signs of leakage and was given the green light to be used in the material testing. This was one of two samples placed on the third shelf in the freeze/thaw-chamber. The sample was not fitted with a thermocouple during testing. No refill was made during the seven day test period and it had a good water level,  $\approx 3mm$  throughout the test, see Figure E49.

After the test was finished the sample had experienced slight evaporation inside the polyester hat. Water was also found on the top surface of the hat, possibly from a leakage in a different specimen. Traces of water was found on the lower parts of the insulation box, as well as on the bottom plate. The adhesion between the tape and the concrete was decent and the scaling from the top surface was also minimal, see Table 16. The sample's weight showed

a big increase from test start to test finish, see Table 17.

These findings indicate that *Reference Sample* does not contain Fly Ash.

#### Sample 2 0.40-FA35-N311016 10x10 ButylSikaFlex, 3mm EPDM Bottom

The adhesive capabilities of the butyl tape significantly helped the application process. The adhesive itself, SikaFlex 11FC was easy to spread on the concrete surfaces as well. The EPDM bottom was fastened using more SikaFlex on the bottom surface. After the materials had been applied, the specimen was weighed and its UPV measured, showing no anomalies.



Figure E50: Sample 2, Material Test 1

During the water saturation the sample showed no signs of leakage during the four days it was in the climate room and as such it was deemed fit for the 7 day freeze/thaw material test. The samples was placed on the top shelf in the chamber, flanked by two dummies, one on each side. It had a thermocouple on the water surface to read its temperature measurements since the top shelf is the shelf which often has the largest temperature swings. See Chapter 4.1

During the material testing it became evident that the sample was either leaking water or had extremely large evaporation rate. This was discovered during a checkup four days after test start. The most likely cause is a combination of the different mechanisms mentioned.

The sample was examined thoroughly after the test finished to find out what had happened to it. The sides did not seem to have leaked as the adhesion was good, indicating that the sample might have lost all its water through the bottom, which is unlikely, but not impossible.

Evidence to support this was the fact that the sides were dry on the outside, so either the condensation had already evaporated (the test surface was almost dry just 3 days after applying new water to it), or the water had escaped through the bottom.

Further evidence was the fact that the bottom surface, the 3mm EPDM sheet attached by the SikaFlex 11FC adhesive, was barely attached to the bottom at all. It was easily removed without effort.

The sample did not scale at all, indicating that there was a rapid loss of water from the sample, as scaling will only occur if the sample has an continuous water layer on top of it. The weight of the sample had also increased significantly, as well as the value for the Ultrasonic Pulse Velocity. The reasons for this are unclear, because if the water was absorbed, the sample should have lower UPV values, whilst that is not the case. Furthermore, the sample has gained weight, indicating that the sample should have absorbed it, but ti does not show. The data just does not make sense.

# Sample 3 0.40-FA35-N311016 10x10 ButylMarFug, no bottom

The application on this sample was just as straight forward as it was for the previous SikaFlex sample. The adhesive used is slightly more *wet* than the SikaFlex one and as such it is easy to spread and distribute along the concrete surface. Nothing was attached to the bottom, as the CEN/TS 12390-9 Standard indicates that it is not necessary to have any material adhered to the bottom surface. It was also in order to see if there would be any differences between different setups.



Figure E51: Sample 3, Material Test 1



Figure E52: Sample 3, Material Test 1

The sample was considered fit for the seven day material test as it successfully passed the water saturation. During the test itself, the sample was placed on the third shelf in the freeze/thaw-chamber alongside the reference sample. This sample did not have a thermocouple to register its different water temperatures, since the middle shelves usually have lower variations between each cycle.

After the testing was done slight condensation was noticed both on the outside and the inside of the polyester sheet. The sample was not refilled during the testing period and it was discovered that there was still water on the test surface, albeit not the 3mm it had at the test start. The sample had also scaled a decent amount, especially along the edges of the sample, in the interface zone between the adhesive and the concrete.

During further inspection it was discovered that the bottom insulation was wet and that there were wet marks on the bottom surface. Furthermore the butyl tape on the sides were found to be slightly loose. After removing it from the insulation, it showed that the sample had increased its weight greatly, but its UPV was relatively unchanged. Which is a fair result, but still not very reasonable. It would be expected that the UPV would be lower than the initial value.

When looking at Figure E51 the damages are quite severe along the edges, but not so much in the middle of the test surface. The adhesion loss is quite clear as well as the damages the forces between the concrete and the adhesion creates during freezing and thawing of the sample.

# Sample 4 0.40-FA35-N311016 10x10 ButylXtremFix, Butyl bottom

The application of the XtremFix adhesive is slightly more cumbersome as it is thicker. It took more effort to distribute it around the sides and bottom of the concrete. On all the surfaces a butyl tape layer was attached to envelope the entire sample in an XtremFix and butyl tape casing except for the test surface.



Figure E53: Sample 4, Material Test 1

After water saturation no leaks were found and the sample was then placed on the second shelf in the freeze/thaw-chamber for the seven day material testing. The sample had a thermocouple attached to its test surface to see its temperature development during the entire test period.

After the test the sample was inspected and condensation was found on the inside of the polyester sheet. The condensation can explain why water was found on the inside of the insulation box as well as on the bottom of the sample. Leakage was also a possibility. It is also worth noting that there was a decent portion of water left on the test surface and the sample was not refilled.

The amount of scaling, especially alongside the edges are detrimental to the sample. Even though it has managed to keep a good layer of water during the entire test, the scaling are of such a bad degree that once the top layer of concrete is removed, the sides easily detach from the concrete surface. See Figure E54. This might be because the adhesive is creating large tensile forces in the interface zone during freezing and thawing.



Figure E54: Sample 4, Material Test 1

The UPV of the sample had increased slightly, as well as the weight. Whats worth noting is that the UPV had to be taken from the two other sides than before the material test begun. When trying to get a reading from the preferred sides nothing happened, most likely because of the increased void between the tape and concrete which occured during the extensive process of getting out some of the scaled material from This might have influenced the the sides. result as the weight gain was a whopping 27,9 grams, showing that the sample absorbed a lot of water, which shoould have lowered the transit time through the sample

Sample 5 0.40-FA35-N311016 10x10 1mmRubBandSikaFlex, 3mm EPDM bottom The rubber band material used to create a dam in this sample was fairly easy to apply, although sepecial care had to be used on the edges between each side, as the rubber band material is not self-adhesive. This meant the process of applying it to the concrete sample was slightly more difficult than the butyl tape. Even so, the process was quite smooth and the SikaFlex glue seemed to attach nicely to both the rubber band and the concrete surface.



Figure E55: Sample 5, Material test 1

It became evident after the water saturating phase of the samples that this sample was in fact leaking quite heavily. Tt was decided that the sample would not be used for the seven day material testing. Since the sample did not qualify to undergo the seven day material testing, its UPV value and weight was not measured afterwards. It is expected that the UPV should remain the same since no changes has happened to the sample. Its weight might have increased slightly due to the water added during the saturation phase, which might have filled up its pores slightly.

# Sample 6 0.40-FA35-N311016 10x10 1mm-RubBandMarFug, 3mm EPDM bottom

This sample used the Marin og Teknikk adhesive, which is slightly more wet than SikaFlex. This made the application process slightly challenging as the adhesive used a long time to cure to both the concrete surface and the 1mm thick rubber band. Fastening the edges proved a challenge. To keep the rubber band in place while the sample dried weights had to be placed on the sample in order for it to fasten in an as proper way as possible.



Figure E56: Sample 6, Material Test 1

Even with measures like: weights, caution along the edges and long procedure time were used, the sample did not want to adhere properly to every surface layer of the concrete. As evident by figure E56. There are many flaws, visibly close to every corner/edge, due to the rubber bands lack of flexibility, unless tightened and stretched in an obscene manner, around these sharp edges. The sample was therefore not deemed fit to be water saturated, and it did not pass the material testing, both due to its impracticality while applying and because it did not properly seal the sample to create a proper dam.

Sample 7 0.40-FA35-N311016 10x10 1mmRubBandXtremFix, 3mm EPDM bottom This sample was the only sample, out of three rubber band samples, that made it to the seven day material testing. The application, as with the previous XtremFix samples, was cumbersome. The inherent strength of the adhesive made the process of applying the rubber band to the concrete easier.

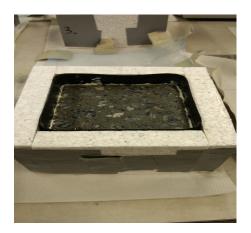


Figure E57: Sample 7, Material test 1

The sample showed no signs of leakage after four days of saturation. The sample was cleared for the seven day material test and was placed on the fifth shelf during the testing period. It was placed between two dummies and had a thermocouple installed to monitor its temperature.

During the material test, the sample seemed to dry out rather quickly and the sample was refilled four days after test start. The cycle for the material test backs up the claim that the sample lost water rapidly, which might have been absorbed, and this might explain the large weight increase. Yet the UPV value did not decrease, but rather increase. Which does not make a lot of sense for reasons previously mentioned.

#### Sample 8 0.40-FA35-N311016 10x10 3mm EPDMSikaFlex, 3mm EPDM bottom

This sample was very challenging due to the thickness of the EPDM material, the small size of the cubes,  $100mm \times 100mm$ , the lack of bond in the adhesive between the concrete and the material before proper setting. All of these factors made sure that this sample was virtually impossible to make.



Figure E58: Sample 8, Material Test 1

The issues became very evident along the edges of the concrete, as the 3mm thickness of the EPDM sheet created an arch on both sides, not adhering to the concrete surface at all. Fixing this would require help to keep the material attached to the concrete until the adhesive actually adhered and dried.

As can be seen from Figure E58, there is so much tension around the edges that it virtually pushes the EPDM layer away. It should also be mentioned that the end result was *not* as bad as the referred figure indicates, but it gives a good indication of the problems occuring.

The setup is considered impractical, at least for this size of samples, to even consider using 3mm thick EPDM with this "*weak*" adhesive.

#### Sample 9 0.40-FA35-N311016 10x10 3mmEPDMMarFug, 3mm EPDM bottom

This sample suffers from the same problems as sample 8 and did not perform well. There were too many problems related to the adherence between the EPDM sheet and the concrete. Further problems were related to the inflexible rubber material being used. This was especially noticeable around the concrete edges. Different measures were taken in order to make a proper sample: clamps, weights, setting over night, but it would not comply.



Figure E59: Sample 9, Material Test 1

system.

sive Marin og Teknikk being very free-flowing and runny, especially when in the early phases of hardening.

This can possibly be explained with the adhe-

From Figure E59 it is possible to identify that it is not impossible to attach the concrete to the material, and that it is possible to bring it around the edges as well. The problem arises when one side has to be taken at a time, waiting for each side to dry and so forth, making the entire process meticulous.

This sample was considered to be impractical and no further testing was done with this preparation

# Sample 10 0.40-FA35-N311016 10x10 3mmEPDMXtremFix, 3mm EPDM bottom



Figure E60: Sample 10, Material Test 1

The only sample using the 3mm thick EPDM sheeting that made it to the material test and it has the same issues, but the adhesive used was sticky enough to create a proper sample. The thickness of the EPDM sheet meant that this sample had the highest transit times of all tested samples. No leakage was found after the saturation stage and it qualified for the seven day material test.

The sample was placed on the 4th shelf in the freeze/thaw-chamber with a thermocouple monitoring its temperature. The cycle showed that the sample was within the CEN/TS 12390-9 test limits during most of the test. Afterwards condensation was found on the inside of the hat. Lots of water was also found on the bottom plate and the inside of the insulation. As Figure E60 shiws the sample had scaled decently, especially around the edges.

The sides were completely loose on three out of four sides. This might explain the large weight increase and the slightly increased UPV value.

#### Material Test 2

Below follows an in-detail description of the thirteen samples in Material Test 2. This visual inspection is a supplement to Table 13. The samples went through the entire 56 day cycle period. Sample 12 and 13 ran for a total of 59 cycles with all previous cycles included. The preparation of the samples is described in detail in Chapter 3.3.4. The goal is to give further details regarding each and every sample and shed further light on the results found, the conclusions made and the effect of these conclusions after the decisive Material Test 2.

The samples will be described briefly for every week they were inspected and will give information regarding if they were refilled or not during the interval. The check up intervals were on the 7th, 14th, 21st, 28th, 35th, 42nd, 48th and 56th day of the CEN/TS 12390-9 test. Furthermore the samples were inspected in-between these intervals to see if the samples needed to be refilled. This has also been registered and will be included.

Additionally every sample description will follow with pictures showing the progress of the sample from the first seven cycles to the 56th. This is to see the impact of the test.

#### Sample 1, 0.40-FA35-N311016-10x10 ButSika<sub>1</sub>

#### 7th Day

The sample has suffered from condensation on the inside and water is found on the inside of the insulation box. The sample has not been refilled in the seven day period and has a decent amount of scaling. The adhesion seems good on all sides. Most of the scaling is located in the interface zone between adhesive and concrete edge/side. The sample has developed some air pockets.

#### 14th Day

Sample had condensation on the sides, which tasted like regular water mixed with salt. Bottom plate was wet, indicates leakage/evaporation. Sample was refilled after 1 day before examination. Decent amount of scaling, and adhesion on all sides seems good. Scaling seems to be located across sample, slightly more in interface zone.

#### 28th Day

The sample suffers from evaporation and there is SSD conditions on the test surface. The insulation box is starting to deteriorate, especially the hat, which has come loose on two sides. All sides still seems to be decently adhered.

#### 42nd Day

Sample still has a decent amount of water left, refilled from SSD conditions 4 days prior. Condensation on sides in small water droplets, tasting both of regular and salinated water. Very low scaling. Problems with insulation box(hat  $\Rightarrow$  maintenance more frequently required. Sample has 1 well adhered side and 3 that are decently/poorly adhered.

#### 56th Day

Slightly wet surface, almost SSD. Sample was refilled 8 days prior. Little to no condensation on the sides of the sample. Bottom plate is wet. Low amount of scaling, meaning most damages occured in the interface zone for this sample. 3 Sides still seem decently adhered to the sample, while there is one side that has become weakly attached.



(a) Sample 1, 7 Cycles



(b) Sample 1, 56 Cycles

Figure E61: Damages, Sample 1, Material Test 2

-1

#### Sample 2, 0.40-FA35-N311016-10x10 ButSika<sub>2</sub>, thin adhesive layer

#### 7th Day

Sample has a decent amount of water; not refilled during the period. It has some condensation on the sides as well as air pockets. The water tastes like a mix of normal-and salinated water. The bottom plate is wet. The lower parts inside the insulation box is wet too. All sides seems well attached and the amount of scaling is decent.

#### 14th Day

The sample has a decent amount of water and was not refilled. The inside insulation is still wet as well as the bottom plate. Condensation on sides too, which the sample suffers from. The scaling is high and is trending along the transition zone between the adhesive and concrete edge. The adhesion on all sides is still good.

#### 28th Day

Sample had SSD conditions and was refilled 8 days prior to examination. The sample struggles with evaporation. The sample has scaled a lot, especially along the edge. The adhesion is still good on two sides, decent on a third, but at least one other sides look weak.

#### 42nd Day

The sample had close to SSD conditions; refilled 4 days prior to examination. The scaling amount is low. The insulation case is still wet on the insde and the bottom plate is wet as well. The maintenance of the insulation boxes is necessary as the tape displays weakness to be exposed to lots of water. The sample has 3 decent sides and one quite poor.

#### 56th Day

The sample was SSD when examinated; refilled 8 days prior. The sample had developed some air pockets throughout and more condensation was discovered on the sides. The water is still a mix between regular and salinated. The scaling amount is large. Adherence on two sides are good; the other two are poor.



(a) Sample 2, 7 Cycles

(b) Sample 2, 56 Cycles

Figure E62: Damages, Sample 2, Material Test 2

#### Sample 3, 0.40-FA35-N311016-10x10 ButSikaNoBot

#### 7th Day

The sample had decent amount of water and was not refilled. Mixed water on the sides, some leakage and some condensation. Air pockets had formed in the sample. Sample bottom is wet as well as the bottom plate. It has small concrete particles. Little scaling in the interface zone. Good adherence on all sides. Inside of insulation was also wet.

#### 14th Day

Sample had good amount of water and was not refilled. Large amount of water on sides. Salinated and regular. Inside of insulation and bottom plate is wet. Hat is loose. The scaling has spread out, still small amounts. The adhesion is good on all sides.

#### 28th Day

Sample was refilled 7 days prior, and had now a 1mm layer left on the test surface. The scaling was low and well distributed. The insulation box is starting to require more maintenance. The adhesion is still good on all sides.

#### 42nd Day

Low amount of water, almost SSD; sample was not refilled prior. The scaling was decent, and still distributed well. 3/4 sides are good. Water droplets on sides, mixed. One side had regular water only. Bottom of sample, as well as bottom plate and inside of insulation is also wet.

#### 56th Day

SSD; refilled 8 days prior. The sample scaled decently, both on test surface and on bottom, which can be seen on the bottome plate (Small concrete pieces). Bottom is wet; bottom plate as well. Regular water on the sides. Formation of small air pockets. Decent adherence on 3/4 sides.



(a) Sample 3, 7 Cycles

(b) Sample 3, 56 Cycles

Figure E63: Damages, Sample 3, Material test 2

#### Sample 4, 0.40-FA35-N311016-10x10 ButMarTek<sub>1</sub>

7th Day

Good amount of water; not refilled. Scaling in interface zone on one side. Most likely weakest side. Other sides are well adhered. Developed air pockets. Water found on insulation and bottom plate.

#### 14th Day

Good amount of water; refilled. Low amount of scaling, located mostly to one side. Weaker adherence here. Three other sides are good. Water found on sides, with mixed flavour. The inside of insulation is wet as well as the bottom plate.

#### 28th Day

The sample has good amount of solution, 2mm, on test surface; refilled 1 week prior. Large quantity of scaled material. The insulation box was wet, same as bottom plate. Water found on sides. Insulation box is becoming poorer, requires more maintenance.

#### 42nd Day

Sample has good amount of water; not refilled. Low/decent amount of scaling. Most of it located to one side. 3 good sides, one weak side. Water on the side, caused both by some form of leakage and evaporation. Tasted like a mix. More air pockets on butyl surface.

#### 56th Day

Sample is SSD; refilled 8 days earlier. Visual scaling and a decent amount. Regular water on the sides, from condensation only. Three sides are decently adhered while the last is poorly adhered. Insulation box wet on the inside, bottom plate wet as well. Hat and insulation box is weak even after lots of maintenance.





(a) Sample 4, 7 Cycles

(b) Sample 4, 56 Cycles

Figure E64: Damages, Sample 4, Material Test $\mathbf{2}$ 

#### Sample 5, 0.40-FA35-N311016-10x10 ButMarTek<sub>2</sub>

7th Day

The sample has a good amount of solution left; not refilled. Decent amount of scaling, in interface zone. Water is found on the side of the concrete as well as the bottom plate. Mixed origin, leakage and condensation. lots of condensation on inside of hat. Small air pockets have been developed. Good adherence on all sides.

#### 14th Day

Sample has good amount of water; refilled from SSD. Water droplets found on the sample side, with salty taste, most likely a mix. Low amount of scaling, but evenly spread, with a slight concentration in one corner. Insulation and bottom plate is wet. The adherence seems good on all sides.

#### 28th Day

The sample was SSD when examinated; refilled one week prior. Scaling was decent and still evenly distributed. The sample suffered from evaporation, but the bottom plate was still wet.

#### 42nd Day

The sample had a good amount of water; refilled. Condensation levels in the hat were high and water was found inside insulation box and on bottom plate. The amount of scaling was very low, almost none at all. Problems with keeping a good insulation box showing on this sample as well. Two out of four sides are still good/decently adhered.

#### 56th Day

When examined the sample was SSD; refilled 8 days prior. Low/decent amount of scaling which is evenly spread. One side is very poorly adhered, the three others are ok. They seem prone to water intrusion by now. Debris at edges. The sides were almost completely dry, still some air pockets. Wet bottom plate and inside of insulation.



5

(a) Sample 5, 7 Cycles

(b) Sample 5, 56 Cycles

Figure E65: Damages, Sample 5, material test 2

#### Sample 6, 0.40-FA35-N311016-10x10 ButMarTekNoBot

#### 7th Day

Sample has retained a good amount of solution; not refilled. Low amount of scaling, but it is evenly distributed along the test surface. All sides seem to be well adhered. Some air pockets created. Liquid found on the sides; water droplets varying in size and flavour. The bottom of the sample is wet. The bottom plate is wet and has some very fine concrete grains attached.

#### 14th Day

Decent amount of solution left in the sample; not refilled. Condensation on underside of the hat, while bottom plate has liquid on it, still small specks of concrete. Scaled amount is low and spread out. All sides seem to be well adhered. Droplets found on sides, of varying size and of regular water and salinated.

#### 28th Day

Sample was SSD when examined; refilled one week earlier. High amount of scaling, evenly distributed. Sides are well adhered. Seems to suffer from evaporation.

#### 42nd Day

The sample had retained a good amount of water; refilled during interval. The amount of scaling is low. The adherence seems very good. Bottom plate is wet w/concrete particles on it. Sample needs regular maintenance now to perform adequately.

#### 56th Day

Sample had almost retained water, slightly wet, but close to SSD; refilled 8 days prior. The amount of scaling is low. All sides are still decently adhered to the concrete and no side seems particularly weaker than the other. Water droplets on the side of varying size and of mixed flavour. Concrete particles on wet bottom plate.





(a) Sample 6, 7 Cycles

(b) Sample 6, 56 Cycles

Figure E66: Damages, Sample 6, Material Test 2

#### Sample 7, 0.40-FA35-N311016-10x10 ButXtrem<sub>1</sub>

#### 7th Day

Sample has retained a good amount of water; not refilled. There is a decent amount of scaling and most of it is found in the interface zone. The sample experienced some water droplets of varying taste. The bottom plate was wet, and so was the inside of the insulation. Many air pockets were also found when examining. The adherence between the the concrete and adhesive seems too strong, as if a sort of pushing and pulling is going on during testing.

#### 14th Day

Sample has retained a good amount of water; not refilled. Condensation on underside of the hat. The insulation is wet on the inside and on the bottom plate. The scaling seems more evenly spread out. The adherence is very poor, with one side completely detached from the concrete, see Figure E67b. Not recommended to keep running with the sample.



(a) Sample 7, 7 Cycles

(b) Sample 1, 14 Cycles

Figure E67: Damages, Sample 7, Material Test 2

#### Sample 8, 0.40-FA35-N311016-10x10 $ButXtrem_2$

#### 7th Day

The sample had retained a decent amount of water; not refilled during interval. Lots of scaling was collected, most of it from the interface zone. The sample seems to struggle with the same pushing and pulling motion as sample 7 and 9 during freeze/thaw. One of the sides are good, but the three others seem below adequate/poor. Small droplets are found on the butyl tape, with a salty taste. There is a lot of liquid on the bottom plate and the insulation is quite wet on the inside.

#### 14th Day

The sample had retained a good amount of water; refilled some days before examination. The scaling is located at the edge, in the interface zone and is contributing to the poor adhesion on all sides. From Figure E68 it is seen that two of four sides are barely attached at all, and that continuing with the sample is pointless.





(a) Sample 8, 7 Cycles

(b) Sample 1, 14 Cycles

Figure E68: Damages, Sample 8, Material Test 2

#### Sample 9, 0.40-FA35-N311016-10x10 ButXtremNoBot

#### 7th Day

The sample retained water poorly during the first seven days, and it was not refilled. Decent amount of scaling, mostly in the interface/transition zone. There were are few droplets of water with mixed flavour. The bottom plate had a lot of liquid on it and the bottom of the concrete was very wet. The adherence seems decent, but there are issues related to the strength of the adhesive when pushed and pulled during freeze/thaw. Similar issues as samples 7 and 8 display.

#### 14th Day

The sample had retained a good amount of water due to it being refilled some days prior to examination. Water was found on the bottom plate where small particles of concrete were found as well. The insulation was wet and the insulation hat had decent amount of condensation on it. The sample had scaled a decent amount, mostly in the interface zone. Further inspection showed that the sample had completely lost adherence on one side, making this sample unfit to continue testing.



(a) Sample 9, 7 Cycles

(b) Sample 9, 14 Cycles

Figure E69: Damages, Sample 9, Material Test 2

#### Sample 10, 0.40-FA35-N311016-15x15 ButSikaNew

#### 7th Day

The sample has retained a good amount of water on the test surface; not refilled. The scaling amount was large and evenly spread on the test surface. There were large amounts of water droplets on the sides and the bottom plate was wet. Air pockets on the butyl tape sides. The insulation was wet on the inside and the hat experienced a lot of condensation. The adherence seemed good on all sides.

#### 14th Day

The sample had retained water well during the next interval, refilled. few water droplets and low condensation found on sides, but the bottom plate was still very wet; mix of regular and salinated water. The amount of scaling was decent, spread evenly. The sample had some condensation on the inside of the hat. the insulation box was wet on the inside. Good adherence on three out of four sides.

#### 28th Day

The sample was examined in SSD conditions; showed signs of leakage and evaporation. The sample scaled a decent amount. Insulation issues with the hat. A pocket of liquid was found under the butyl at the bottom of the specimen. All sides decently adhered.

#### 42nd Day

The sample had retained a good amount of water; refilled from less than 1mm layer. The condensation on the sample was high and there were droplets on the side of the sample. The bottom plate had a lot of water on it. The scaling was insignificant. Sample had good adherence on three sides.

#### 56th Day

The sample was examined in SSD conditions; refilled 8 days prior. The amount of scaling was very low; three sides were decently adhered. One side was poorly adhered. Regular water was found on the sides and the sample has developed some air pockets.





(a) Sample 10, 7 Cycles

(b) Sample 10, 56 Cycles

Figure E70: Damages, Sample 10, Material Test 2

#### Sample 11, 0.40-FA35-N311016-15x15 ButMarTekNew

#### 7th Day

The sample has retained a good amount of water; no refill. Large amount of scaling over entire test surface. Liquid is found on bottom plate, varying flavour. Sides have mixed water. Adherence seems good on all sides.

#### 14th Day

Little amount of water left, approximately 1mm; not refilled. The sample has scaled a high amount across the entire test surface. Water on sides and on bottom plate. Large air pockets starting to form along every side.

#### 28th Day

The sample has less than 1mm free liquid on the surface. Decent amount of scaling. Possible leakage and evaporation problem. Water on sides and on bottom plate as well.

#### 42nd Day

Good amount of water, almost 2mm; refilled. The amount of scaling was insignificant, so no surface changes. Condensation spotted on the inside of hat. Wet bottom plate and wet sides of mixed water. The adherence seems weak on three sides, but one side is still very good.

#### 56th Day

The sample is SSD; refilled 8 days prior. The scaling is close to insignificant. Water found on sides is a mix of regular and salinated water. One side is completely loose, two are poorly attached and one side is well attached in middle. Probably because the adhesive is well applied at this location.



(a) Sample 11, 7 Cycles

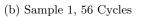


Figure E71: Damages, Sample 11, Material Test 2

#### Sample 12, 0.40-FA35-N311016-15x15 ButSikaOld

#### 7th Day

The sample has retained some water on test surface; not refilled. The scaling was decent and evenly spread. The adherence is impacted by previous runs, but is still holding, but looks pretty damaged. Lots of water on bottom plate is found. More water is found on the sides with mixed flavour.

#### 14th Day

The sample has not retained much water, SSD; not refilled. The level of scaling is decent and evenly spread. The sides are not very wet, but the bottom plate is. There are formations of small air bubbles. The adherence seems slightly weaker, but it is not ruined yet.

#### 28th Day

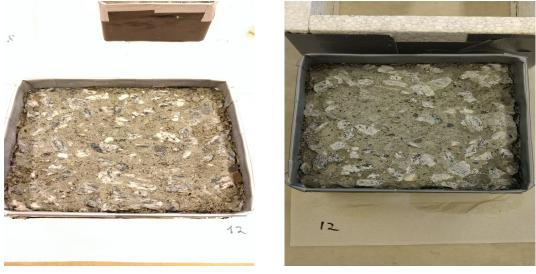
The sample is in SSD conditions. The sample scaled a decent amount. Water is found on the bottom plate and a little on the sides. Suffers from evaporation and leakage. The sides are slowly giving way, and adherence is becoming poor.

#### 42nd Day

SSD; refilled four days prior. The scaling is low, but that is mostly related to the sample leaking, loosing water too fast. The sample has no virtues left and was removed 6 days later, on the 48th day of Material Test 2. After 59 cycles for this sample.

#### 59th Day

The sample is removed 8 days before test finish after its 59 cycles. All sides are poorly adhered, all of them are loose. The scaling is insignificant and the sample will not hold water.



(a) Sample 12, 7 Cycles

(b) Sample 12, 56 Cycles

Figure E72: Damages, Sample 12, Material Test 2

#### Sample 13, 0.40-FA35-N311016-15x15 ButMarTekOld

#### 7th Day

The sample was close to SSD when examined; not refilled. Insignificant amount of scaling, associated to the lack of decent water level on surface. The bottom plate had lots of water on it. Not a lot of water on the sides. Adherence is still decent on all sides.

#### 14th Day

The sample had decent water left; refilled some days prior. Decent amount of scaling evenly spread. Not a lot of liquid on the sides, but more on bottom plate. Adherence looks good on three out of four sides.

#### 28th Day

Signs of liquid on the surface, close to SSD. There was a decent amount of scaling which was spread out evenly. The adherence is very poor on one side and it has a 10mm deep gap on one side. Two sides were poor and one was good. Water on bottom plate and not much water on the sides. Hat is struggling.

#### 42nd Day

Liquid on test surface; refilled twice in a week. The scaling is low but still evenly spread. There is a lot of water droplets on the sides and the bottom plate is very wet. Mixed flavour. The adherence is very poor on three sides, but quite decent on the last side.

#### $59 th \ Day$

Sample was removed after its 59th cycle (48th in Material Test 2) because it had no virtues left. See Figure E73b. Three of the sides are barely attached and the sample will not hold water. Yet one side is still very good and shows no adherence issues.





(a) Sample 13, 7 Cycles

(b) Sample 13, 56 Cycles

Figure E73: Damages, Sample 13, Material Test 2

#### Series 2 - in-depth sample analysis

In this part of the appendix follows an in-depth description of the 10 samples that ran for 28 days in a CEN/TS 12390-9 test. The samples have been evaluated on the 7th, 14th and 28th of the testing and the notes below are the observations noted about each sample on the respective day.

#### Sample 1 0.40-FA35-Non-AEA

#### 7th Day

Examined in SSD conditions; refilled. Good amount of scaling. Sample had some air bubbles on the side and wet bottom plate discovered. The water on the side is a mix of regular water and then some salty water. The sides are well adhered on most sides, but it seems weak in general.

#### 14th Day

Examined in SSD conditions; not refilled. There seems to be scaling alongisde the edges, which now decent/weak adherence on three out of four sides. Many air bubbles on the sides; not filled with water. The bottom plate is wet once more. The water on these areas does not taste salty.

#### 28th Day

Sample was examined in SSD conditions; not refilled. High amount of scaling, which is evenly distributed along the test surface. The sample has condensated, as expected, and there is water droplets on the sides and underneath on the bottom plate. All of wet spots are of regular water. Air bubbles still prevalent. The adhesion on the sides are now weak on three sides whilst one side is ok.

The damages and scaling were very high on this sample as can be seen from the pictures below, Figures E74a and E74b. There are not many "clean" surfaces left and most of the sample looks ravished, and the sides are in very speculative conditions.



(a) Sample 1, Damages after 28 Cycles



(b) Sample 1, Scaling after 28 Cycles

Figure E74: Damages, Sample 1, Series 2

#### Sample 2 0.40-FA35-Non-AEA

#### 7th Day

The sample was found in SSD condition; not refilled. The scaling is good and evenly spread. The water droplets on the side taste like a mix of regular water and salt. Same with the bottom plate. The adhesion is still good on all sides. They do not appear much weakened after the first 7 days.

### 14th Day

The sample has a good amount of water left; not refilled. Because of the continuous water level, the sample has scaled a lot, and the damage is evenly spread across the test surface. The water found on the sides is a mix of regular water and salt solution, whilst the water on the bottom plate is from condensation. The adhesion is decent to weak on most sides, related to the amount of damages the sample has received.

#### 28th Day

The sample was in SSD conditions before examination started; not refilled. The scaling was very high, but also evenly spread on the sample, which can be seen in Figure E75b. The adhesion is also becoming very weak on at least two of the sides, illustrated in picture E75a. The water that was found on the sides of the sample and the bottom plate was not salty, meaning it is condensation. The sample also has some large air bubbles on the side.



(a) Sample 2, Damages after 28 Cycles

(b) Sample 2, Scaling after 28 Cycles



#### Sample 3 0.40-FA35-Non-AEA

#### 7th Day

The sample was found in SSD conditions, in spite of being refilled. The sample lost water rapidly and it did not scale a great amount compared to the other non-AEA samples, but it was spread evenly on the test surface. The water on the sides was a mix of slt solution and regular water, with a distinct salty taste. The same with the bottom plate. Air pockets found on the sides. The adhesion is still good on every side, corresponding to low damage.

#### 14th Day

Sample was almost dry, so SSD; not refilled during this period. The scaling is still evenly spread, and comparatively low for a non-AEA, sample. The adhesion is also still good on all sides, further indicating that the issues with retaining water is influencing the damages on the sample. The water found on the sides is a mixture of salt solution and regular water. The same is found on the bottom plate. There seems to be slightly more air pockets now.

#### 28th Day

The sample is found in SSD conditions; not refilled. Larger amount of scaling than previously, seen in Figure E75b, evenly spread across the test surface. The damages on the sample found after 28 days can be seen in Figure E76a. In spite of the damages, the adhesion is still decent to good on most sides. Two sides are good, whilst the two others are slightly weaker/decent. Water and air pockets were found on the sides of the sample. The water was pure, no salty taste. Regular water was found on the bottom plate as well.



(a) Sample 3, Damages after 28 Cycles

(b) Sample 3, Scaling after 28 Cycles

Figure E76: Damages, Sample 3, Series 2

#### Sample 4 0.40-FA35-Non-AEA

#### 7th Day

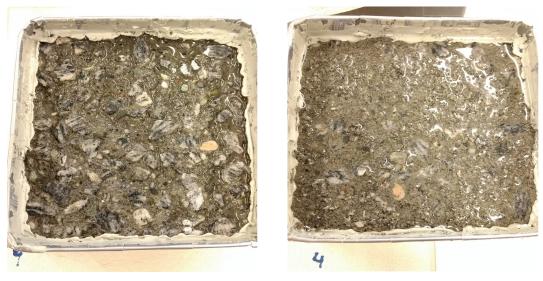
Sample did not retain any water, examined in SSD conditions; refilled earlier. The sample has scaled a lot, evenly spread out across the entire test surface. The water found on the sides of the sample is a mixture of condensation and salt solution. The same problem has arrived on the bottom plate. The sample has good adherence on all sides.

#### 14th Day

The sample had not retained any water, it was SSD; not refilled. The scaled amount was high and evenly spread out on the test surface, not located to any interface between the adhesive and concrete. The adhesion in general seems fairly good on two sides, but only decent on two others. The sample had regular water located on the sides as well as on the bottom plate.

#### 28th Day

Sample had retained approximately 1mm of water; not refilled. The scaled amount was very high, as indicated by Figure E77b. The water found on two sides was a mix between salt solution and regular water, whilst the two others had regular water on them. Bottom plate was also found wet with normal water. The adhesion is starting to become quite weak on the sample, as can be seen by Figure E77a. There are serious damages on one of the sides.



(a) Sample 4, Damages after 28 Cycles

(b) Sample 4, Scaling after 28 Cycles



#### Sample 5 0.40-FA35-Non-AEA

#### 7th Day

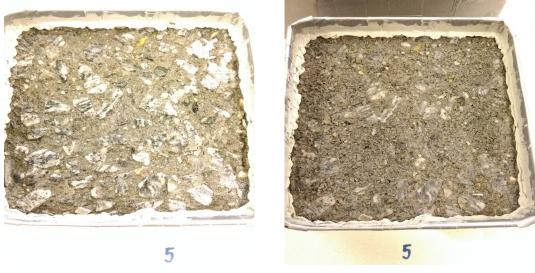
The sample has not retained any water, SSD; not refilled during this period. There is a good amount of scaling, evenly spread out across the test surface. Even though substantial damage the adherence is still good on all sides. Water was found on the sides and it was a mixture of regular water and salt solution. The bottom plate was also wet with the same characteristics as the sides. Sample has developed some small air pockets.

#### 14th Day

The sample has not retained any water, SSD; not refilled during the interval. There is a good amount of scaling, evenly spread. The adhesion is becoming slightly weaker on the sides, but it is still strong. Not excellent, but decent. The water found on the sides is mixed. The same with the bottom plate. Air pockets are still prevalent.

#### 28th Day

The sample has not been refilled during this period and was SSD when examination began. The sample has scaled a lot, as shown in Figure E78b. It is evenly spread across the test surface and does not reside in any particular zone. There was water found on the side which was condensation, whilst the bottom plate had some indications of salty flavour in its water. The adhesion is becoming decent at best, with all sides a lot weaker than at the start. The damages done to sample 5 can be seen in Figure E78a.



(a) Sample 5, Damages after 28 Cycles

(b) Sample 5, Scaling after 28 Cycles

Figure E78: Damages, Sample 5, Series 2

#### Sample 6 0.40-FA35-AEA

#### 7th Day

The sample has close to 3mm water left on the test surface, but it was refilled earlier. The scaling is almost negligible. Condensation on the sides as well as on the bottom plate. One of the butyl sides is of mixed flavour. The adhesion is excellent.

#### 14th Day

The sample has retained a good amount of water, close to 3mm, without being refilled in between. Condensation is found on three sides of the butyl tape whilst the last side has a mixed solution. The bottom plate also has some water on it with a mixed, salty, flavour. The scaling is low across the entire test surface and the adhesion is very good.

#### 28th Day

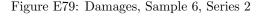
The sample had retained some water, close to 1mm, without having been refilled prior. The sample has also scaled a decent amount, considering it is an air-entrained sample. This can be seen in Figure E79b and it shows that it is evenly spread across the surface. Condensation was found on the sample sides and bottom plate. The sample also shows very good adhesion on all sides, as can be seen in Figure E79a as there is little damage along the interfce zone between the adhesion and concrete.



(a) Sample 6, Damages after 28 Cycles



(b) Sample 6, Scaling after 28 Cycles



#### Sample 7 0.40-FA35-AEA

#### 7th Day

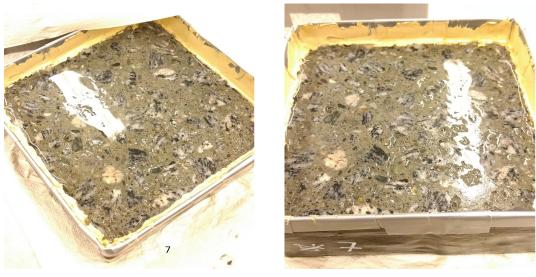
Sample did not retain any water and is in SSD conditions, in spite of being refilled during the interval. There seems to be close to no scaling on the surface. Condensation is found on all sides and the bottom plate, which is mixed with salt solution. The bottom plate also has the same issue. The sample has very good adhesion.

#### 14th Day

The sample has retained 3mm of water on the test surface throughout the interval without being refilled. The scaled amount is very low, barely visible. The sides have condensation on them. The bottom plate has a mixture of salt solution and normal water. The adhesion is still excellent and there is barely any visible damage on the test surface.

#### 28th Day

The sample has retained a decent amount of water; a little under 2,5cm. There is some visible scaling on the test surface which is evenly spread out, which can be seen on Figure E80b. The water found on the sides are condensation mostly, but it carries the distinct taste of salt. The bottom plate has pure water on it. Some air pockets are found on the sides. The adhesion is still excellent and from Figure E80a it is difficult to see any disjointed parts between the adhesion and the concrete.



(a) Sample 7, Damages after 28 Cycles

(b) Sample 7, Scaling after 28 Cycles



#### Sample 8 0.40-FA35-AEA

#### 7th Day

The sample has retained approximately 2cm of water during the interval without being refilled. The amount of scaling is close to negligible. There is condensation on the sides and the sample has small air pockets as well. The bottom plate is also wet and the water is a mixture between regular water and salt solution. The adherence seems good on all sides. 14th Day

#### The sample did not retain much water and was in SSD conditions; not refilled. The sample has not scaled a lot and there are in general little damages in the interface zone. The sample has one slightly weaker side though, where the adhesion is a little above decent. The sample has liquid on the sides which is a mix of regular water and salt solution. The bottom plate is wet as well and the water has a salty taste.

#### 28th Day

The sample has retained a slight liquid layer on the top of the test surface, not SSD, but not very deep water layer. There is some mixed water on the sides of the sample. The bottom plate has regular water on it. The scaling is very low as well as the damages on the sample. The adhesion is very good, but one side is slightly weaker, see Figure ??, whilst the damages are almost negligible, Figure E81a.







(b) Sample 8, Adhesion after 28 Cycles

Figure E81: Damages, Sample 8, Series 2

#### Sample 9 0.40-FA35-AEA

#### 7th Day

The sample did not retain any water and was in SSD condition. The sample was not refilled during the interval and there is very little scaling on the test surface. The liquid on the side is a mixture of regular water and salt solution, so some condensation and some leakage issues. The adhesion is still excellent.

#### 14th Day

The sample has retained a good amount of water, close to 3 mm, without being refilled. The sample has liquid on the sides which is regular water. The bottom plate also had regular water on it. The scaling was insignificant and the adhesion is still excellent.

#### 28th Day

The sample has retained a very good amount of water without being refilled. Close to 3mm. As with the previous interval both the sides and the bottom plate has regular water on them. No salt taste detected. The scaling was low, as indicated by Figure E82b, and it also shows that the damages have been evenly spread out throughout the 28 days. The damages, Figure E82a, show that there are no problems with the bond between the adhesive and the concrete and the sample is in excellent condition.



(a) Sample 9, Damages after 28 Cycles

(b) Sample 9, Scaling after 28 Cycles



#### Sample 10 0.40-FA35-AEA

#### 7th Day

The sample had not retained much water and was in SSD conditions; refilled earlier during the interval. There is a negligible amount of scaling on the test surface. The liquid found on the sides taste like salt, while the bottom plate also has the same issue. Which indicates leakage. The adhesion is very good, corresponding to the lack of damage.

#### 14th Day

The sample did not manage to retain any water and was in SSD condition, but it was not refilled. The scaling is negligible, but the little scaling that is is not located to any distinct part of the sample. The sample has liquid on the sides which is a mix of regular water and salt solution. The bottom plate has a mixed solution on it as well. The adhesion is good to decent on all sides.

#### 28th Day

The sample has retained a good amount of water, close to 3mm, without being refilled. The scaling is negligible and the damages are also negligible. The sample is in very good condition, except on one side where the adhesion is slightly weak. The liquid on the bottom plate and on the sides are regular water and air pockets are also located on the sides of the sample.



Figure E83: Damages, Sample 10, Series 2

# F Technical data

This section includes the full material data sheet (MDS), or the most important extract from the MDS, of the equipment used, such as: adhesives, tapes and materials. The appendix will also include a link to each respective material safety data sheet (MSDS), as well as extracts from the most important part of each MSDS respectively

#### Sitko Elastic 605 Butyl Tape



# Sitko Elastic 605 Butyl Tape Safety Data Sheet

	ety Regulations (EC 1907/2006)	Tectis Oy
issued: (	07/09/2005 revised: 05/05/2011	page: 1/4
1. Inforr	mation on Product, Production and Compa	ny
	Product details: Product name:	Sitko 605
	Manufacturer/Supplier:	Tectis Oy Mänkimiehentie 19 FI-02780 ESPOO Tel.: +358 (0)9 4393 460 Fax: +358 (0)9 4393 4610
	Enquiry department: Emergency telephone:	Tectis Oy: Tel.: +358 (0)400 421 125
2. Haza	ards Identification	
	Description of dangers:	
	Special hazards to health and environr None	nent:
3. Com		nent:
3. Com	None	Polyethylene adhesive tape with a butyl rubber adhesive.
	None position / Information on Ingredients Chemical characterization:	Polyethylene adhesive tape with a butyl
	None position / Information on Ingredients Chemical characterization: Description:	Polyethylene adhesive tape with a butyl
	None position / Information on Ingredients Chemical characterization: Description: Aid Measures	Polyethylene adhesive tape with a butyl
	None position / Information on Ingredients Chemical characterization: Description: Aid Measures General comments:	Polyethylene adhesive tape with a butyl rubber adhesive.
	None position / Information on Ingredients Chemical characterization: Description: Aid Measures General comments: After inhalation:	Polyethylene adhesive tape with a butyl rubber adhesive.

EU Safety Regulations (EC 1907/2006) Tectis Oy

issued: 07/09/2005 revised: 05/05/2011 page: 2/4

5. Fire-Fighting Measures

Suitable fire extinguishing media: CO<sub>2</sub> extinguisher or halogen extinguisher Special equipment for fire-fighters: None

6. Accidental Release Measures

Personal and environmental precautionary measures: The adhesive tape is extremely rot proof and not dangerous to ground water.

Special cleaning procedure: Adhesive residues may be removed with surgical spirit.

7. Handling and Storage

Handling: None, except for possible carbon monoxide formation resulting from incomplete combustion.

**Storage:** Keep in dry rooms, free of dust and oil. Do not store together with strong oxidants.

8. Exposure Control / Personal Protection

Exposure limit information: None

Personal protective clothing: General safety and hygiene measures: Usual measures (cleanliness, order).

Inhalation protection: Hand protection: Eye protection:

Not necessary Not necessary Not necessary

#### 1mm Nitrile Rubber Band Materialegenskaper

For valg av riktig gummimateriale er det viktig at man kjenner hvilket medium det skal tette mot.

Arbeidstemperatur og trykk influerer også på valg av materiale og hardhet. Det er ingen fordel å velge et gummimateriale som dekker et større temperaturområde enn nødvendig.

Ved å gjøre dette kan andre egenskaper bli borte og medføre ekstra kostnader. Tabellen er kun retningsgivende ved valg av gummimateriale. Temperaturgrensene er omtrentlige verdier, som i spesielle tilfeller kan være høyere eller lavere. De oppgitte hardheter er nominelle.

ASTM koder Materialer	ISO 1629	Strekkfasthet	Bruddfortengelse	Rivestyrke	Maks. temperatur *C	Min. temperatur °C	Slitasjeegenskaper	Vær/aldringsegenskaper	Ozonbestandighet	Elektrisk motstand	Bestandighet olje	Bestandighet syrer	Bestandighet alkalier	Bestandighet varmt vann
Butylgummi	IIR	4	2	3	+130	-40	3	2	2	2	6	2	3	2
Epiklorhydringummi	ECO	4	3	3	+145	-40	3	2	2	5	1	4	4	4
Etylen/propylengummi	EPDM	4	2	3	+130	-40	3	1	1	2	5	2	2	1
Etyleneakrylatgummi	ACM	5	3	4	+160	-20	4	2	2	4	2	5	5	5
Fluorgummi (Viton®)	FPM	5	3	4	+200	-20	5	1	1	3	1	1	1	2
Fluorsilikongummi	FMVQ	6	4	6	+200	-80	5	1	1	3	2	4	4	4
Kloroprengummi (Neopren®)	CR	3	2	2	+120	-40	3	2	2	3	3	3	3	3
Klorsulfonisert polyetylengummi (Hypalon®)	CSM	4	3	4	+120	-20	3	2	2	4	2	2	2	2
Naturgummi	NR	1	1	1	+90	-40	1	3	4	2	6	3	3	3
Nitrilgummi	NBR	5	2	3	+120	-50	3	4	4	4	1	4	3	3
Perfluorgummi (Kalrez®)	FFKM	5	3	3	+316	-20	4	1	1	3	1	1	1	1
Polytetrafiuoretylen (Teflon®)	PTFE	1	3	-	+200	-190	3	1	1	1	1	1	1	1
Polyurethanegummi	PUR	1	2	1	+100	-25	1	1	1	3	2	6	5	6
Silikongummi	MVQ	6	4	6	+200	-70	5	1	1	1	2	4	4	4
Styrol/butadienegummi	SBR	4	2	3	+100	-50	1	3	4	2	6	4	4	3

1 Særdeles god 2 Meget god 3 God 4 Nokså god 5 Dådia

5 Dárlig 6 Anbefales ikke

Ved tvil om hvilket materiale som skal/bør brukes, ta kontakt.

Viton®, Hypalon® og Kalrez® er registrert varemerke for DuPont Performance Elastomers Teflon® registrert varemerke for DuPont

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#### **EPDM Sheet**

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# EPDM sheeting

# Description

EPDM sheeting is one of the most versatile types of rubber sheeting and is the most suitable for outdoor applications. It is highly resistant to ageing, even when exposed to the most aggressive external conditions: steam, UV rays, ozone, saltpetre or extreme weather conditions.

EPDM sheeting maintains its properties even when subjected to a wide range of temperature differences. As a result it is consistently used in general industry and in construction, marine and outdoor applications.

## Properties

- Excellent inherent high and low temperature ranges. Typically -45° C to +120° C for standard compounds.
- Special compounding can increase some grades to function continually at +140 °C.
- EPDM is inherently resistant to attack by oxygen, U.V., Ozone and extreme weather environments, and will give long service in these conditions.
- EPDM does not have good adhesion properties.
- Resistance to Chemicals; resistant to many chemicals and solvents. Good resistance to many corrosive chemicals. The performance of EPDM in hot water and high pressure steam is better than in dry heat.

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#### Casco Marin og Teknikk Product Sheet



#### TEKNISK DATA

Typ: SMP-polymer. Färg: White (4061), Grey (4062), Light oak/Light wood (4063), Exotic wood (4064), Black (4066), Dark oak/dark wood (4068), White (2994), Black (2993)Konsistens: Tixotrop (lättsprutad). Densitet: Ca 1350 kg/m<sup>3</sup>. Hårdhet: 45 Shore A. Temperaturbeständighet: - 40°C - +90°C.

APPLIKATIONSDATA Arbetstemperatur: +10°C - +25°C. Skinntid: Ca 45 minuter. Härdtid: 3 mm p/dygn. Foarörelse: 20 %. Glättningsväska: Rent vatten blandad med diskmedel. Jämna ytan med fuktad fogpinne/finger. Övermålningsbar. Ja. Lagringstid: 12 månader (ej öppnad förpackning). Förvaras svalt och frostfritt. Verktyg: Casco fogpistol.

HÄLSA OCH MILJÖ Märkning

Hantering och Rengöringsföreskrifter Rengör hud med tvål och vatten. Verktyg rengörs enligt följande. Ohärdad produkt – med vatten. Härdad produkt – mekaniskt.

Om massan kommer i ögonen, spola genast med mycket vatten och sök läkare. Vid hudkontakt, tvätta med tvål och vatten. Undvik hudkontakt. Förvaras oåtkomligt frö barn. Rengör verktyg med lacknafta. Härdad massa tas bort mekaniskt.

Ytterligare information finns i Säkerhetsdatabladet.

#### BRUKSANVISNING

1. Rengör/torka av ytorna 2. Öppna patronen. Skär av spetsen i 45<sup>0</sup> vinkel till Oppna patronen. Skar av spetsen 145° vinket till något bredare än den önskade fogbredden. Använd en fogpistol vid applicering av Marin & Teknik i patron.
 Fogning/tätning: Eftersläta ytan på fogen med en fogpinne fuktat i en blandning av vatten och lite diskmedel. Limning: Stryk im på ena ytan. Lägg

olskmedel. Limining: Stryk im på ena ytan. Lagg samman och fixera ytorna. 4. Patron: Behöver patronen sparas, tryck ut fogmassa och låt det härda. Det täpper till och gör att inte luft på samma sätt kommer åt fogmassan så härdningen uppstår. Sätt tillbaka hatten om tuben ska sparas. Uppstar. Satt ulibaka hatten om tuben ska sparas. 5. *Tub*: Beröver tuben sparas, sätt en klick vaselin i korken innan förslutning. Det hjälper mot härdning. Glöm inte att torka bort översta lagret vid nästa användning, annars påverkas vidhäftningen. 6. Förvara patronen/tuben i frysen eller svalt. Tag fram i rumstemperatur i god tid innan användning.

Underhåll

övermålning av en elastisk fogmassa kan minska fogrörligheten. Marin & Teknik är dock kompatibel med

#### Casco Marin og Teknikk Safety Data Sheet

SAFETY DATA SHEET according to Regulation (EC) No. 1907/2006 Marin & Teknik Revision Date 02.01.2017 Version 1.0 Print Date 02.01.2017 SECTION 1: Identification of the substance/mixture and of the company/undertaking 1.1 Product identifier Trade name : Marin & Teknik 1.2 Relevant identified uses of the substance or mixture and uses advised against Product use : Sealant/adhesive 1.3 Details of the supplier of the safety data sheet : Sika Norge AS Company Sanitetsveien 1 2013 Skjetten Telephone +4767067900 E-mail address kundeservice@no.sika.com 1.4 Emergency telephone number Emergency telephone num- : Giftinformasjonen: 22 59 13 00 ber **SECTION 2: Hazards identification** 2.1 Classification of the substance or mixture Type of product : Mixture Classification (REGULATION (EC) No 1272/2008) Not a hazardous substance or mixture. 2.2 Label elements Labelling (REGULATION (EC) No 1272/2008) Not a hazardous substance or mixture. Additional Labelling: EUH210 Safety data sheet available on request.

#### 2.3 Other hazards

This substance/mixture contains no components considered to be either persistent, bioaccumulative and toxic (PBT), or very persistent and very bioaccumulative (vPvB) at levels of 0.1% or higher.

#### SECTION 3: Composition/information on ingredients

#### 3.2 Mixtures

#### Hazardous components

Chemical name	Classification	Concentration	l
CAS-No.	(REGULATION (EC)	[%]	l
 NO 1000000055			

Country NO 10000006355

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#### SAFETY DATA SHEET according to Regulation (EC) No. 1907/2006 Marin & Teknik

Revision Date 02.01.2017

Version 1.0



#### **SECTION 8: Exposure controls/personal protection**

#### 8.1 Control parameters

Contains no substances with occupational exposure limit values.

#### Occupational exposure limits of decomposition products

Components	CAS-No.	Value	Control parame-	Basis *
			ters	
methanol	67-56-1	TWA	100 ppm	FOR-2011-12-
			130 mg/m3	06-1358

\*The above mentioned values are in accordance with the legislation in effect at the date of the release of this safety data sheet.

#### 8.2 Exposure controls

#### Personal protective equipment

Eye protection	: Safety glasses
Hand protection	: Chemical-resistant, impervious gloves complying with an ap- proved standard must be worn at all times when handling chemical products. Reference number EN 374. Follow manu- facturer specifications.
	Butyl rubber/nitrile rubber gloves (0,4 mm), Recommended: Butyl rubber/nitrile rubber gloves.
Skin and body protection	<ul> <li>Protective clothing (e.g. Safety shoes acc. to EN ISO 20345, long-sleeved working clothing, long trousers). Rubber aprons and protective boots are additionally recommended for mixing and stirring work.</li> </ul>
Respiratory protection	: No special measures required.
Environmental exposure of	controls
General advice	: No special environmental precautions required.
SECTION 9: Physical and cl	nemical properties

# 9.1 Information on basic physical and chemical properties Appearance : paste Colour : various Country NO 10000006355 4 / 10

SAFETY DATA SHEET according to Regulation (EC) No. 1 Marin & Teknik	907/2006	<b>Sika</b> ®
Revision Date 02.01.2017	Version 1.0	Print Date 02.01.2017
10.2 Chemical stability		
The product is chemically stal	ble.	
10.3 Possibility of hazardous rea	actions	
Hazardous reactions	: No hazards to be specially mentioned.	
10.4 Conditions to avoid		
Conditions to avoid	: No data available	
10.5 Incompatible materials		
Materials to avoid	: No data available	
10.6 Hazardous decomposition	products	
Hazardous decomposition products	: methanol	
	No decomposition if stored and applied as di	rected.

#### **SECTION 11: Toxicological information**

#### 11.1 Information on toxicological effects

Acute toxicity Not classified based on available information.

<u>Components:</u> trimethoxyvinylsilane:	
Acute oral toxicity	: LD50 Oral (Rat): ca. 7.120 mg/kg
Acute inhalation toxicity	: LC50: ca. 16,8 mg/l Exposure time: 4 h Test atmosphere: dust/mist
Acute dermal toxicity	: LD50: 3.540 mg/kg
Skin corrosion/irritation	
Not classified based on avail	able information.
Serious eye damage/eye ir	ritation
Not classified based on avail	able information.
Respiratory or skin sensiti	sation
	fied based on available information. t classified based on available information.
Germ cell mutagenicity	
Not classified based on avail	able information.
Carcinogenicity	

## Carcinogenicity

Not classified based on available information.

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onents considered nd toxic (PBT), or (vPvB) at levels of

# wherever possible. Empty containers or liners may retain some product residues. This material and its container must be disposed of in a safe way. Dispose of surplus and non-recyclable products via a licensed waste disposal contractor.

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#### **Casco Xtremfix Product Sheet**



Erstatter: 2011-11-08

# XtremFix 3895

Monteringslim som fester umiddelbart inntil 200 kg/m<sup>2</sup>. Brukes til liming av f eks speil, trematerialer, metall, naturstein, bygningsplater, takplater og lister. Egner seg også til små limflater som kroker og beslag. Kan brukes på tette og sugende flater, ute og inne, også på fuktige flater.

#### • Svært gode monteringsegenskaper

- God vedheft mot de fleste materialer
- Tåler frost
- Gir en sterk og noe elastisk, tettende limfuge
- Fester umiddelbart inntil 200 kg/m<sup>2</sup> vertikalt
- Fester umiddelbart inntil 1000 kg/m<sup>2</sup> horisonatlt
- Holdfasthet etter herding 5000 kg/m<sup>2</sup>
- Inneholder ikke isocyanat, ftalater eller silikon.

#### TEKNISK DATA

Produkttype: Farge: Emballasje: Løsemiddel:

Tørrstoff: Viskositet: Densitet: Skinndannelse:

Hardhet: Herdetid:

Arbeidstemp.: Temp.bestandighet: Forlengelse: Strekkstyrke: 100 % modul: Oppbevaring: SMP teknologi Hvit 300 ml Ingen, herder ved hjelp av luftens fuktighet. 100 % Smidig pasta 1,57 g/cm<sup>3</sup> Ca. 10 min. ved normal Arbeidstemperatur Shore A 55 3 mm pr. døgn ved + 20°C. Herdetiden vil for -lenges ved lavere temperatur og lav luftfuktighet. +5 °C - +40° C -40°C - +10°C 250 % (DIN53504) 2,2 MPa (DIN53505) 1,39 MPa (DIN 53504) Minst 12 mndr. i uåpnet emballasje ved romtemperatur

#### FORBEHANDLING

Flatene skal være rene og fri for fett olje og støv. Benytt evt Casco Limtvätt til rengjøring.

#### PÅFØRING

Påføres med fugepistol av god kvalitet som f eks Casco Pro Gun P 160 fugepistol med tilhørende V-formet patronspiss. Hold pistolen i 90° vinkel i forhold flaten som limet skal påføres. Limfugen skal være formet som en stående trekant. Påfør limet punktvis eller limstrenger vertikalt med maks 5 cm mellomrom. Kontroller at lim filmen har en tykkelse på ca 3 mm ved liming av tyngre materialer. Sørg for tilgang til fuktighet ved liming av to tette materialer.



#### Casco XtremFix Safety Data Sheet

SAFETY DATA SHEET according to Regulation (EC) No. 1907/2006 Casco<sup>®</sup> XtremFix Revision Date 05.12.2016 Version 2.0 Print Date 05.12. SECTION 1: Identification of the substance/mixture and of the company/undertaking 1.1 Product identifier Trade name : Casco® XtremFix 1.2 Relevant identified uses of the substance or mixture and uses advised against Product use : Sealing system 1.3 Details of the supplier of the safety data sheet : Sika Sverige AB Company Domnarvsgatan 15 163 53 Spånga Telephone +4686218900E-mail address : miljo@se.sika.com 1.4 Emergency telephone number Emergency telephone num- : 112 Ask for Poison Information ber **SECTION 2: Hazards identification** 2.1 Classification of the substance or mixture Type of product : Mixture Classification (REGULATION (EC) No 1272/2008) Not a hazardous substance or mixture according to Regulation (EC) No. 1272/2008. 2.2 Label elements Labelling (REGULATION (EC) No 1272/2008) Not a hazardous substance or mixture according to Regulation (EC) No. 1272/2008. Additional Labelling: EUH210 Safety data sheet available on request. EUH208 Contains N-(3-(trimethoxysilyl)propyl)ethylenediamine, dioctyltin acetylacetonate, N-[3-(dimethoxymethylsilyl)propyl]ethylenediamine, bis(1,2,2,6,6-pentamethyl-4-piperidyl) sebacate, methyl 1,2,2,6,6-pentamethyl-4-piperidyl sebacate. May produce an allergic reaction.

#### 2.3 Other hazards

This substance/mixture contains no components considered to be either persistent, bioaccumulative and toxic (PBT), or very persistent and very bioaccumulative (vPvB) at levels of 0.1% or higher.

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#### SAFETY DATA SHEET according to Regulation (EC) No. 1907/2006 Casco® XtremFix

Revision Date 05.12.2016

Version 2.0



#### **SECTION 3: Composition/information on ingredients**

3.2 Mixtures

Hazardous components

Chemical name CAS-No. EC-No. Registration number	Classification (REGULATION (EC) No 1272/2008)	Concentration [%]
trimethoxyvinylsilane 2768-02-7 220-449-8 01-2119513215-52-XXXX Contains: tetramethyl orthosilicate <= 0,2 %	Flam. Liq.3; H226 Acute Tox.4; H332	>= 1 - < 2,5
N-(3-(trimethoxysilyl)propyl)ethylenediamine 1760-24-3 217-164-6 01-2119970215-39-XXXX	Eye Dam.1; H318 Skin Sens.1B; H317	< 1
dioctyltin acetylacetonate 54068-28-9 483-270-6 01-0000020199-67-XXXX	Skin Sens.1; H317 STOT SE2; H371	< 1
N-[3-(dimethoxymethylsilyl)propyl]ethylenediamine 3069-29-2 221-336-6 01-2119963926-21-XXXX Contains: methanol <= 0.5 %	Eye Dam.1; H318 Skin Sens.1; H317	< 1
bis(1,2,2,6,6-pentamethyl-4-piperidyl) sebacate 41556-26-7 255-437-1 01-2119491304-40-XXXX	Skin Sens.1A; H317 Aquatic Chronic1; H410 Aquatic Acute1; H400	>= 0,025 - < 0,25
methyl 1,2,2,6,6-pentamethyl-4-piperidyl sebacate 82919-37-7 280-060-4	Skin Sens.1A; H317 Aquatic Acute1; H400 Aquatic Chronic1; H410	>= 0,025 - < 0,25

For the full text of the H-Statements mentioned in this Section, see Section 16.

#### SECTION 4: First aid measures

#### 4.1 Description of first aid measures

General advice	: No hazards which require special first aid measures.
If inhaled	: Move to fresh air.

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Revision Date 05.12.2016	Version 2.0	Print Date 05.12.201
In case of skin contact	: Take off contaminated clothing and shoes Wash off with soap and plenty of water.	
In case of eye contact	: Remove contact lenses. Keep eye wide open while rinsing.	
If swallowed	: Do not induce vomiting without medical ac Rinse mouth with water. Do not give milk or alcoholic beverages. Never give anything by mouth to an uncor	
4.2 Most important symptoms an	nd effects, both acute and delayed	
Symptoms	: See Section 11 for more detailed informat and symptoms.	tion on health effects
Risks	: No known significant effects or hazards.	
4.3 Indication of any immediate r	nedical attention and special treatment nee	ded
Treatment	: Treat symptomatically.	
	sures	
SECTION 5: Firefighting meas 5.1 Extinguishing media Suitable extinguishing media	: Use extinguishing measures that are appr cumstances and the surrounding environn	
5.1 Extinguishing media Suitable extinguishing media	: Use extinguishing measures that are appr cumstances and the surrounding environn	
<ul> <li>5.1 Extinguishing media</li> <li>Suitable extinguishing media</li> <li>5.2 Special hazards arising from</li> </ul>	: Use extinguishing measures that are appr cumstances and the surrounding environn	nent.
<ul> <li>5.1 Extinguishing media</li> <li>Suitable extinguishing media</li> <li>5.2 Special hazards arising from Hazardous combustion prod-</li> </ul>	: Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture	nent.
<ul> <li>5.1 Extinguishing media         Suitable extinguishing media     </li> <li>5.2 Special hazards arising from         Hazardous combustion products     </li> <li>5.3 Advice for firefighters</li> </ul>	: Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture	nent. nown
<ul> <li>5.1 Extinguishing media         <ul> <li>Suitable extinguishing media</li> </ul> </li> <li>5.2 Special hazards arising from             Hazardous combustion products</li> <li>5.3 Advice for firefighters             Special protective equipment</li> </ul>	<ul> <li>Use extinguishing measures that are appr cumstances and the surrounding environn</li> <li>the substance or mixture</li> <li>No hazardous combustion products are kr</li> </ul>	nent. nown
<ul> <li>5.1 Extinguishing media Suitable extinguishing media 5.2 Special hazards arising from Hazardous combustion products </li> <li>5.3 Advice for firefighters Special protective equipment for firefighters </li> </ul>	Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture     No hazardous combustion products are kr     In the event of fire, wear self-contained br     Standard procedure for chemical fires.	nent. nown
<ul> <li>5.1 Extinguishing media         <ul> <li>Suitable extinguishing media</li> </ul> </li> <li>5.2 Special hazards arising from             Hazardous combustion products</li> <li>5.3 Advice for firefighters             Special protective equipment             for firefighters             Further information</li> </ul> <li>SECTION 6: Accidental release</li>	Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture     No hazardous combustion products are kr     In the event of fire, wear self-contained br     Standard procedure for chemical fires.	nent. nown eathing apparatus.
<ul> <li>5.1 Extinguishing media         <ul> <li>Suitable extinguishing media</li> </ul> </li> <li>5.2 Special hazards arising from             Hazardous combustion products</li> <li>5.3 Advice for firefighters             Special protective equipment             for firefighters             Further information</li> </ul> <li>SECTION 6: Accidental release</li>	Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture     No hazardous combustion products are kr     In the event of fire, wear self-contained br     Standard procedure for chemical fires.	nent. nown eathing apparatus.
<ul> <li>5.1 Extinguishing media         <ul> <li>Suitable extinguishing media</li> </ul> </li> <li>5.2 Special hazards arising from             Hazardous combustion products</li> <li>5.3 Advice for firefighters             Special protective equipment             for firefighters             Further information</li> </ul> <li>SECTION 6: Accidental release     <ul> <li>6.1 Personal precautions, protection</li> </ul></li>	Use extinguishing measures that are appr cumstances and the surrounding environn the substance or mixture     No hazardous combustion products are kr     In the event of fire, wear self-contained br     Standard procedure for chemical fires.     Standard procedure for chemical fires.     Eremeasures tive equipment and emergency procedures	nent. nown eathing apparatus.

Country SE 10000007908

sco <sup>®</sup> XtremFix		Jika
evision Date 05.12.2016	Version 2.0	Print Date 05.12.20
Personal protective equipr	ent	
Eye protection	: Safety glasses	
Hand protection	proved standard must be wo	ous gloves complying with an ap- orn at all times when handling ce number EN 374. Follow manu-
	Butyl rubber/nitrile rubber glo Recommended: Butyl rubber	
Skin and body protection	long-sleeved working clothin	ety shoes acc. to EN ISO 20345, rg, long trousers). Rubber aprons ditionaly recommended for mixing
Respiratory protection	: No special measures require	ed.
Environmental exposure c	ntrols	
General advice	: No special environmental pre	recautions required.

#### **SECTION 9: Physical and chemical properties**

9.1 Information on basic physica Appearance	l a :	nd chemical properties paste	
Colour	:	white	
Odour	:	mild	
Odour Threshold	:	No data available	
Flash point	:	> 100 °C	
Autoignition temperature	:	No data available	
Decomposition temperature	:	No data available	
Lower explosion limit (Vol-%)	:	No data available	
Upper explosion limit (Vol-%)	:	No data available	
Flammability	:	No data available	
Explosive properties	:	No data available	
Oxidizing properties	:	No data available	
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asco <sup>®</sup> XtremFix		Jika
Revision Date 05.12.2016	Version 2.0	Print Date 05.12.201
SECTION 11: Toxicological i	nformation	
1.1 Information on toxicologic	al effects	
Acute toxicity Not classified based on avail	able information.	
Components:		
trimethoxyvinyIsilane: Acute oral toxicity	: LD50 Oral (Rat): ca. 7.120 mg/kg	
Acute inhalation toxicity	: LC50: ca. 16,8 mg/l Exposure time: 4 h Test atmosphere: dust/mist	
Acute dermal toxicity	: LD50: 3.540 mg/kg	
N-(3-(trimethoxysilyl)propy Acute oral toxicity	I)ethylenediamine: : LD50 Oral (Rat): 2.995 mg/kg	
dioctyltin acetylacetonate: Acute oral toxicity	: LD50 Oral (Rat): 2.500 mg/kg	
Skin corrosion/irritation Not classified based on avail	able information.	
Serious eye damage/eye in Not classified based on avail		
	<b>sation</b> fied based on available information. t classified based on available information.	
Germ cell mutagenicity Not classified based on avail	able information.	
Carcinogenicity Not classified based on avail	able information.	
Reproductive toxicity Not classified based on avail	able information	
STOT - single exposure	and mornation.	
Not classified based on avail	able information.	
STOT - repeated exposure Not classified based on avail	able information.	
Aspiration toxicity		

Country SE 10000007908

according to Regulation (EC) No. 190 Casco <sup>®</sup> XtremFix		Jika
Revision Date 05.12.2016	Version 2.0	Print Date 05.12.201
SECTION 12: Ecological inform	nation	
12.1 Toxicity		
Components:		
dioctyltin acetylacetonate :		
Toxicity to daphnia and other	: EC50: > 22 mg/l, 48 h, Daphnia magna (Wat	er flea)
aquatic invertebrates Toxicity to algae	: EC50: > 31,55 mg/l, 72 h, Scenedesmus cap water algae)	ricornutum (fresh
12.2 Persistence and degradabilit	v	
No data available		
12.3 Bioaccumulative potential		
No data available		
12.4 Mobility in soil		
No data available		
12.5 Results of PBT and vPvB ass	sessment	
Product:		
Assessment	<ul> <li>This substance/mixture contains no compone to be either persistent, bioaccumulative and t very persistent and very bioaccumulative (vP 0.1% or higher</li> </ul>	oxic (PBT), or
12.6 Other adverse effects		
Product:		
Additional ecological infor- mation	: There is no data available for this product.	
SECTION 13: Disposal conside	erations	
13.1 Waste treatment methods		
Product	<ul> <li>The generation of waste should be avoided o wherever possible.</li> <li>Empty containers or liners may retain some p This material and its container must be dispo</li> </ul>	product residues.
	way. Dispose of surplus and non-recyclable produ waste disposal contractor.	cts via a licensed
	Disposal of this product, solutions and any by at all times comply with the requirements of e	

Country SE 10000007908

## SikaFlex 11 FC Safety Data Sheet

SAFETY DATA SHEET Sikaflex 11FC		
Version 1.0	MSDS Number: 000000601550	Revision Date: 22.06.2015
SECTION 1. PRODUCT AND CO	MPANY IDENTIFICATION	
Product name	<sup>:</sup> Sikaflex 11FC	
Product code	: 000000601550	
Type of product	: liquid	
Manufacturer or supplier's o	details	
Company	: Sika Australia Pty. Ltd.	
Address	: Elizabeth Street 55 Wetherill Park NSW 2164	
Telephone	: +61297251145	
Emergency telephone num- ber	: + 61 1800 033 111	
Telefax	: +61297253330	
E-mail address	: EHS@au.sika.com	

SECTION 2. HAZARDS IDENTIFICATION

GHS Classification Flammable liquids	: Category 4
GHS Label element Hazard pictograms	: None
Signal word	: Warning
Hazard statements	: H227 Combustible liquid.
Precautionary statements	<ul> <li>Prevention:</li> <li>P210 Keep away from heat/sparks/open flames/hot surfaces No smoking.</li> <li>P280 Wear protective gloves/ eye protection/ face protection.</li> <li>Response:</li> <li>P370 + P378 In case of fire: Use dry sand, dry chemical or alcohol-resistant foam for extinction.</li> <li>Storage:</li> <li>P403 + P235 Store in a well-ventilated place. Keep cool.</li> <li>Disposal:</li> <li>P501 Dispose of contents/ container to an approved waste disposal plant.</li> </ul>
Other hazards which do no None known.	t result in classification

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

### Sikaflex 11FC



Version 1.0 MSDS Number: 000000601550

Substance / Mixture

## Aixture : Mixture

Chemical Name	CAS-No.	Concentration (%
xylene	1330-20-7	0 - < 10
Hydrocarbons, C9-C12, n-alkanes, isoalkanes, cyclics, aromatics (2-25%)	64742-82-1	0 - < 10
4-isocyanatosulphonyltoluene	4083-64-1	0 - < 10
4,4'-methylenediphenyl diisocyanate	101-68-8	0 - < 10

#### SECTION 4. FIRST AID MEASURES

General advice	: No hazards which require special first aid measures.
If inhaled	: Move to fresh air. Consult a physician after significant exposure.
In case of skin contact	<ul> <li>Take off contaminated clothing and shoes immediately.</li> <li>Wash off with soap and plenty of water.</li> <li>If symptoms persist, call a physician.</li> </ul>
In case of eye contact	<ul> <li>Flush eyes with water as a precaution.</li> <li>Remove contact lenses.</li> <li>Keep eye wide open while rinsing.</li> </ul>
If swallowed	<ul> <li>Clean mouth with water and drink afterwards plenty of water.</li> <li>Do not give milk or alcoholic beverages.</li> <li>Never give anything by mouth to an unconscious person.</li> </ul>
Most important symptoms and effects, both acute and delayed	<ul> <li>No known significant effects or hazards.</li> <li>See Section 11 for more detailed information on health effects and symptoms.</li> </ul>
Notes to physician	: Treat symptomatically.

#### SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media	: Carbon dioxide (CO2)
Unsuitable extinguishing media	: Water
Hazardous combustion prod- ucts	: No hazardous combustion products are known
Specific extinguishing meth- ods	: Standard procedure for chemical fires.
Special protective equipment for firefighters	: In the event of fire, wear self-contained breathing apparatus.

#### SECTION 6. ACCIDENTAL RELEASE MEASURES

#### Sikaflex 11FC



Version 1.0	MSDS Number: 000000601550	Revision Date: 22.06.201
Methods and materials for containment and cleaning up	: Wipe up with absorbent material (e.g. clot Keep in suitable, closed containers for dis	
SECTION 7. HANDLING AND STO	DRAGE	
Advice on protection against fire and explosion	: Normal measures for preventive fire prote	ction.
Advice on safe handling	: Do not get in eyes, on skin, or on clothing. For personal protection see section 8. Follow standard hygiene measures when products	
Hygiene measures	<ul> <li>Handle in accordance with good industrial practice.</li> <li>When using do not eat or drink.</li> <li>When using do not smoke.</li> <li>Wash hands before breaks and at the end</li> </ul>	
Conditions for safe storage	: Store in original container. Keep in a well-ventilated place. Observe label precautions. Store in accordance with local regulations	

#### SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Components with workplace control parameters					
Components	CAS-No.	Value type (Form of exposure)	Control parame- ters / Permissible concentration	Basis	
xylene	1330-20-7	STEL	150 ppm 655 mg/m3	AU OEL	
		TWA	80 ppm 350 mg/m3	AU OEL	
		TWA	100 ppm	ACGIH	
		STEL	150 ppm	ACGIH	
4,4'-methylenediphenyl diiso- cyanate	101-68-8	TWA	0.02 mg/m3 (As -NCO)	AU OEL	
	Further information: Category 2 (Carc. 2) Suspected human car- cinogen, Sensitiser				
		STEL	0.07 mg/m3 (As -NCO)	AU OEL	
	Further information: Category 2 (Carc. 2) Suspected human car- cinogen, Sensitiser			d human car-	
		TWA	0.005 ppm	ACGIH	

#### Personal protective equipment

Respiratory protection : Use respiratory protection unless adequate local exhaust ventilation is provided or exposure assessment demonstrates that exposures are within recommended exposure guidelines. The filter class for the respirator must be suitable for the maximum expected contaminant concentration (gas/vapour/aerosol/particulates) that may arise when handling the product. If this concentration is exceeded, self-

#### Sikaflex 11FC



Version 1.0	MSDS Number: 000000601550	Revision Date: 22.06.2
	contained breathing apparatus must b	be used.
Hand protection	<ul> <li>Chemical-resistant, impervious gloves proved standard should be worn at all chemical products if a risk assessmer essary.</li> </ul>	times when handling
Eye protection	: Safety eyewear complying with an app be used when a risk assessment indic	
Skin and body protection	: Choose body protection in relation to tration and amount of dangerous subs cific work-place.	

#### SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance	: paste
Colour	: grey
Odour	: aromatic
Odour Threshold	: No data available
pН	: Not applicable
Melting point/range / Freezing point	: No data available
Boiling point/boiling range	: No data available
Flash point	: ca. 64.6 °C (148.3 °F) Method: closed cup
Evaporation rate	: No data available
Flammability (solid, gas)	: No data available
Upper explosion limit	: No data available
Lower explosion limit	: No data available
Vapour pressure	: No data available
Relative vapour density	: No data available
Density	: ca. 1.26 g/cm3 (23 °C (73 °F) ())
Solubility(ies) Water solubility	: insoluble
Partition coefficient: n- octanol/water	: No data available

#### Sikaflex 11FC



	and the second sec
MSDS Number: 000000601550	Revision Date: 22.06.20
: No data available	
: No data available	
: No data available	
: Not applicable	
: No data available	
: No data available	
	<ul> <li>No data available</li> <li>No data available</li> <li>No data available</li> <li>Not applicable</li> <li>No data available</li> </ul>

#### SECTION 10. STABILITY AND REACTIVITY

Reactivity	: No dangerous reaction known under conditions of normal use.
Chemical stability	: The product is chemically stable.
Possibility of hazardous reac- tions	: Stable under recommended storage conditions.
Conditions to avoid	: No data available
Incompatible materials	: No data available

#### SECTION 11. TOXICOLOGICAL INFORMATION

# Acute toxicity Not classified based on available information. Components: 4,4'-methylenediphenyl diisocyanate: Acute inhalation toxicity : Acute toxicity estimate: 1.5 mg/l Test atmosphere: dust/mist Method: Expert judgement Skin corrosion/irritation Not classified based on available information. Serious eye damage/eye irritation Not classified based on available information. Respiratory or skin sensitisation Skin sensitisation: Not classified based on available information. Respiratory sensitisation: Not classified based on available information. Chronic toxicity

#### Germ cell mutagenicity

Not classified based on available information.

Carcinogenicity

Not classified based on available information.

3M 6969 Duct Tape Product Data Sheet

## **3M** Extra Heavy Duty Duct Tape 6969

Product Description	composed of	an abrasio backing wit	n resistant		strength duct tape over a dense cloth scrin e used for demanding
Product Construction	Backing	Adhesive	Colors	Standard Roll Length	Standard Roll Width
	Polyethylene film over cloth scrim	Rubber	Black, olive, silver	60 yds. (54.8 m)	Black and olive: 1.89 in. (48 mm) Silver: 1.89, 2.83 in. (48, 72 mm)
Typical Physical Properties	Note: The following technical information and data should be considered representative or typical only and should not be used for specification purposes.				
	A. II		<b>54</b> /	(1) (50 N/(400 mm))	ASTM Test Method
	Adhesion to Steel: Tensile Strength:		51 oz./in. width (56 N/100 mm) 34 lbs./in. width (595 N/100 mm)		D-3330 D-3759
	Elongation at Break:		16%		D-3759
	Thickness:		10.0 mils (0.25 mm)		D-3652
	Temperature Use Range:		Up to 200°F (93°C)		
	Burn Test:*	Ū	Flame Sp	oread Index 0 Developed Index 5	E-84
	*ASTM E-84, UL-723, NFPA 255, and UBC 8-1 are publications of the same test method.				
Features	Offers industrial strength backing, easy tear, and abrasion resistance.				
	• Olive color is useful for military applications.				
	• Low tack roll edges (48 mm) so edges stay cleaner for less surface contamination, and less waste.				
	• Individual roll wrap (48 mm) that preserves integrity of the roll for less waste				
	• Polyethylene coated cloth scrim that resists moisture to prolong tape bond.				
	• Rubber adhesive that adheres aggressively to most surfaces for good holding power.				
Application Ideas	<ul> <li>A good choice of tape for HVAC, industrial, construction, abatement, trucking and automotive industries.</li> </ul>				
	<ul> <li>Reinforcing, bundling, moisture proofing, sealing, splicing, temporary repair</li> </ul>				
	<ul> <li>Hanging poly, repairing and splicing tarps, seaming.</li> </ul>				

# 3M<sup>™</sup> Extra Heavy Duty Duct Tape 6969

Storage	Store under normal conditions of 60° to 80°F (16° to 27°C) and 40 to 60% R.H. in the original carton.
Shelf Life	To obtain best performance, use this product within 18 months from date of manufacture.
Technical Information	The technical information, recommendations and other statements contained in this document are based upon tests or experience that 3M believes are reliable, but the accuracy or completeness of such information is not guaranteed.
Product Use	Many factors beyond 3M's control and uniquely within user's knowledge and control can affect the use and performance of a 3M product in a particular application. Given the variety of factors that can affect the use and performance of a 3M product, user is solely responsible for evaluating the 3M product and determining whether it is fit for a particular purpose and suitable for user's method of application.
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Tesa 4688 Duct Tape Product Data Sheet

# productinformation

#### tesa<sup>®</sup> Professional 4688 Standard polyethylene coated cloth tape

tesa® 4688 is a standard cloth tape with a PE-extruded substrate and a natural-rubber adhesive mass. This combination forms the basis for easy handling: the tape is easy to unroll and simple to tear by hand. The repair tape is strong but also sufficiently flexible to be a reliable aid to craftsmen. It can be used for a variety of applications and offers very good adhesion even on rough surfaces. The waterproof and ageing-resistant duct tape is available in seven colours: black, white, blue, yellow, red, silver/matt and green.

#### Main Application

- Indoor and outdoor applications
- Bonding of construction films
- Bundling of cables
- Repairing, packaging, protection, marking

#### Technical Data

<ul> <li>Backing material</li> <li>Total thickness</li> <li>Type of adhesive</li> <li>Adhesion to steel</li> </ul>	PE extruded cloth 260 μm natural rubber 3.4 N/cm	<ul> <li>Elongation at break</li> <li>Tensile strength</li> <li>Temperature resistance (30 min)</li> <li>Mesh</li> </ul>	9 % 52 N/cm 110 °C 55 count per square inch
Properties			
<ul><li>Hand tearability</li><li>Straight tear edges</li><li>Certified according to</li></ul>	••• TLV 9027/01/06 for use in	<ul><li>Abrasion resistance</li><li>Water resistance</li></ul>	•••

Evaluation across relevant tesa® assortment: •••• very good ••• good •• medium • low

nuclear power plant

For latest information on this product please visit <u>http://l.tesa.com/?ip=04688</u>



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As of 16/12/2016

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