



Influence of temperature, moisture, salt, cleaner compounds and rainwater on the expansion of intumescent fire seals

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Abstract

The expected lifetime of many parts of a building can be very long. All elements are subjected to different types of degradation and ageing mechanisms. Thus, the fire resistance of the elements can decline by e.g. degradation of intumescent fire seals that are typically used in passive fire protection. The purpose of this study was to investigate the influence of different environmental parameters on the thermal behaviour of fire seals.

Eight different fire seals were conditioned in eight different environments such as drying, water storage, salt solution, rain and cleaner compound, before exposing them to heat. Thermal behaviour, thermal expansion and reaction temperature, of the fire seals were examined. The results of the measurements indicated that the thermal behaviour of the selected fire seals was sensitive, to varying degrees, to different environmental parameters.

Key words: Intumescent, seal, passive fire protection, water, time-temperature, salt, cleaner compound, acid precipitation.

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Preface

This work has received financial support from the Swedish Fire Research Board (Brandforsk). The work has performed by SP Technical Research Institute of Sweden, Department of Fire technology. A reference group was assigned to the project. The group consisted of

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Summary

Determination and classification of the fire resistance of various elements in building structures is obtained through testing of newly produced elements. All elements are subjected to different types of degradation mechanisms. Thus, the fire resistance of the elements can deteriorate over time. The degree of the deterioration of each material in these elements varies due to the material properties and surrounding conditions like relative humidity, temperature, UV radiation and acidic environment.

Intumescent seals and coatings are typically used in passive fire protection of building elements. The most important function of these intumescent fire seals is to swell and fill the gaps between elements to restrict the spread of smoke, e.g. sealing of fire doors and sealing between glass and glazing beads.

Degradation of a fire seal can result in a lower expansion than the desired expansion and/or affect the reaction temperature. These changes in a fire seal can reduce the fire resistance of the building element, i.e. smoke and combustion gases can pass through the gaps between two elements and cause integrity failure.

The purpose of this study was to investigate the influence of different environmental parameters on the behaviour of intumescent fire seals with respect to fire resistance. Two types of intumescent fire seals, graphite based and sodium silicate based, were investigated

In order to be able to determine the expandability and reaction temperature of fire seals, a measurement setup was developed. The measurement setup proved to give a uniform temperature around the samples and make it possible to measure expansion of the fire seals by a video measurement gauge.

The general conclusions from the study are that:

- The reaction temperature of the fire seals was less sensitive to environmental parameters than their expandability.
- The expandability of fire seals based on graphite, depends on the rate of temperature increase. The expandability of sodium silicate fire seal did not vary with rate of temperature increase.
- Fire seals are hygroscopic material. A low moisture content in the fire seals causes lower expandability and lower reaction temperature.

In this study the fire behaviour of the fire seal is studied generically i.e. not coupled to a specific end use application. It would be useful to also study the function of the fire seals in the actual building elements after environmental exposure. However, the approach taken here by studying them generically is useful in evaluating how different compositions are affected by different environmental conditions. A more robust approach would be to apply the same type of durability requirements that are put on many building materials on fire seals. The seals can then be exposed in different climate chambers developed specifically for the purpose and then evaluated in a small scale furnace like the one used in this project.

1 Introduction

Determination and classification of the fire resistance of various elements in building structures is obtained through testing of newly produced elements. The expected lifetime of many parts of a building can be long. All elements are subjected to different types of degradation mechanisms. Thus, the fire resistance of the elements can be declined. The degree of the declination of each material in these elements varies due to the material properties and surrounding conditions like relative humidity, temperature, UV radiation and acidic environment.

Most materials expand slightly when they are exposed to heat. An intumescent is a substance which expands dramatically and create a char as a result of heat exposure, thus increasing in volume, and decreasing in density. The expansion could be 4-10 times its original thickness. The intumescent produce a soft or hard char, which has a low thermal conductivity, thus retarding heat transfer.

Intumescent seals and coatings are typically used in passive fire protection of building elements. The most important function of these intumescent fire seals is to swell and fill the gaps between elements to restrict the spread of smoke, e.g. sealing of fire doors and sealing between glass and glazing beads.

Generally intumescent materials consist of three basic components: charring agent or carbon source, acid source (solvent), gas source (blowing agent) and binder. Intumescent material can be divided in different types based on their chemical compounds, chemistry of fire residency, or physical behaviour e.g. hard char or soft char. Information sheet No. 1 from the Intumescent Fire Seals Association (ifsa) [1] describes three types of fire seals used in sealing of fire resisting door-sets namely: Ammonium phosphate, Hydrate Sodium Silicate and Intercalated Graphite.

The conclusions of a previous study conducted at SP Technical Research Institute of Sweden related to the ageing effects on the fire resistance of building structures indicated that intumescent fire seals used in various elements could be sensitive to different degradation mechanisms [2]. The intumescent fire seals association (ifsa), in their information sheet No.2 [3] inform that ‘Other intumescent, such as hydrate sodium silicate and mono-ammonium phosphate, are hygroscopic and need to be properly protected to prevent deterioration through the absorption of atmospheric moisture’. The sensitivity and improvement of intumescent seals made of sodium silicate to water is investigated by Han, Ku and We [4]. The results of their investigation revealed that sodium silicate coating was vulnerable to water. In another investigation it was found that a low oxygen content in the atmosphere significantly affects the rate of degradation of the char material at temperatures greater than 540 °C [5].

Degradation of the chemical composition of a fire seal can result in a lower expansion than the desired expansion and/or affect the reaction temperature i.e. the fire seal swells at a lower or higher temperature level than the desired activation temperature. These changes in a fire seal can reduce the fire resistance of a building element i.e. smoke and combustion gases pass through the gaps between two elements and cause integrity failure.

There are two alternatives for the investigation of degradation effects on fire behaviour of a intumescent seal in building elements. The first alternative is to investigate a full scale building element e.g. the element should be conditioned in a predefined relative humidity before fire testing of the element. This type of investigation gives results that is close to the actual behaviour of the element in a building. However, full scale fire tests are

expensive and time demanding. The second alternative is to perform small scale tests on fire seals exposed to degradation. The investigations in this report are based on the second alternative.

The purpose of this study was to investigate the influence of different environmental parameters on the behaviour of intumescent fire seals with respect to fire resistance. In addition, two different rates of temperature increase were used and the swelling studied. Furthermore, a small scale measurement setup was developed.

2 Specimens, time-temperature curve and conditionings

The measurement setup was designed to investigate the thermal behaviour of the fire seals exposed to heat, i.e. reaction time, reaction temperature and expandability. The samples were conditioned in eight different environments. In addition, reference samples of the fire seals were conditioned to normal laboratory conditions i.e. 20°C and 60% relative humidity. The thermal behaviour of the reference samples was determined for two linear time-temperature curves. One of these time temperature-curves was then chosen for measurements concerning the fire seals that had been exposed to a more harsh environment. The thermal behaviour of the conditioned samples was determined and compared with the thermal behaviour of the reference samples. The results of the comparison showed the influence of each conditioning case on the fire resistance of the fire seals.

2.1 Specimens

Eight fire seals of two types, Sodium Silicate and Graphite based, were purchased. These fire seals were denoted fire seal 'A' to 'H'. The dimensions of the samples were 60x20x t (length, width and thickness). The thickness and type of the fire seals is presented in **Table 1**. The comments in this table were provided by the retailer.

Table 1. The thickness and type of the fire seals.

Fire seal	Type	Thickness [mm]	Comments
A	Graphite	2.5	Contains EPM rubber
B	Graphite	2.6	As 'A' but no EPM rubber
C	Graphite	2.5	As 'B' with a casing of TPE* + PVC**
D	Graphite	2.8	Contains organic binder
E	Graphite	2.2	As 'D' strengthened by fibre glass
F	Sodium Silicate	1.9	
G	Sodium Silicate	2.0	As 'F' with a casing of TPE* + PVC**
H	Graphite	2.5	

* Thermoplastic elastomer

** Polyvinyl chloride

2.2 Temperature curve

The Time-temperature curve used for testing the fire-resistance rating of passive fire protection according to EN 1363-1 [6] is rather severe. However, fire seals are rarely exposed to instant fire temperatures. Thus, the measurements in this study were performed by exposing the fire seals to a 10°C/min temperature increase.

In addition, to find out how the rate of temperature increase affects the thermal behaviour of the fire seals, they were exposed to a 5°C/min increasing temperature exposure in some cases.

2.3 Conditions

The fire seals were conditioned in eight different types of environment namely: water storage for 3 or 30 days; drying; freezing; immersion in salt solution of 5 or 100% concentration; immersion in cleaner compound and finally immersion in acid precipitation.

The reasons for each condition are:

- Water storage (fire seals exposed to high humidity)

Fire seals are hygroscopic materials. A hygroscopic material takes/gives moisture from/to the atmosphere. The moisture content of a hygroscopic material varies by variation of the relative humidity of the surrounding environment. A fire seal that is exposed to 100% relative humidity at ambient temperature (23-25 °C) for a infinite period will reach an equilibrium moisture content of 100% relative humidity. This is an extreme condition concerning the moisture content of the fire seal at ambient temperature.

To reach the equilibrium condition is time demanding. In order to achieve a high level of moisture content in the fire seals in a short time, samples of the fire seals were immersed in a water bath for three days. Furthermore, in order to find out the influence of a long term water storage samples of the fire seals, they were stored in a water bath for 30 days.

- Drying (fire seals exposed to hot and dry climate)

Drying of the fire seals corresponds to 0% relative humidity which is in contrast to water storage of the fire seals. Samples of the fire seals were dried in a oven at 105 °C for three days.

- Freezing (fire seals exposed to high humidity and cold climate)

The samples were immersed in a room temperature water bath for one hour. The water bath was then placed in a chamber (temperature of chamber was -5 °C) for 24 hours. Finally the temperature of the chamber was increased to 0 °C and the samples were taken out of the chamber when the temperature of the water bath was ± 0 °C.

- NaCl solution (fire seals in building elements close to sea)

Samples of the fire seals were stored in salt solution for 3 days. Two different concentrations of NaCl in water were chosen, i.e. 5% and 100% .

- Cleaner compound solution (windows cleaning/polishing)

Samples of the fire seals were immersed in a bath of cleaner compound for 3 days. The content of the cleaner compound was anionic tenside < 5%, glycol ether, isopropanol and water.

- Sulphuric acid solution (fire seals exposed to acid precipitation-rain)

The term " acid precipitation" is used to mean the deposition of acidic components in rain, snow, fog, dew, or dry particles. "Clean" or unpolluted rain has a slightly acidic pH of 5.6. The extra acidity in rain comes from the reaction of air pollutants, primarily sulfur oxides and nitrogen oxides, with water in the air to form strong acids (like sulphuric and

nitric acid). Measurements carried out by SP showed that a PH of 4 could be a good average for rain water. Thus, samples of the fire seals were immersed in a bath of sulphuric acid (pH=4) for 3 days.

Two samples of each fire seal were prepared for conditioning in each environment according to **Table 2**, e.g. we had 20 samples of fire seal 'E'. Four reference samples were stored in the laboratory at 20 °C and 60% relative humidity. The reference samples were used for the determination of the influence of a temperature increase in time.

All fire seals were not conditioned in all environment e.g. the fire seals C and G, which both had a casing, were of the same type as the fire seals A and F respectively. These fire seals (C and G) were used to determine the influence of the casing on the behaviour of the fire seals. Furthermore, fire seals 'E', 'F' and 'H' were used to determine the thermal behaviour at 5°C/min. Information concerning the number of fire seals in each environments is presented in **Table 2**.

Table 2. Samples of fire seals in different condition

Condition/Fire seal	A	B	C	D	E	F	G	H
Reference	X	X	X	X	X	X	X	X
T(t)=10°C/min	X	X	X	X	X	X	X	X
T(t)=5°C/min	-	-	-	-	X	X	-	X
Water storage-3 days	X	X	-	X	X	X	-	X
Water storage-30 days	X	X	-	X	X	X	-	X
Drying-3days	X	X	-	X	X	X	-	X
Freezing	X	X	-	X	X	-	-	X
NaCl- solution 5%	X	X	-	X	X	-	-	X
NaCl- solution saturation	X	X	-	X	X	-	-	X
Cleaner compound	X	X	-	X	X	-	-	X
Acid precipitation water	X	X	-	X	X	-	-	X

3 Experimental set-up

A chamber furnace Nabertherm model L40/11 with a regulator of type B170 and a controller of type 2132i was used in the measurements. The maximum available temperature in the furnace is 1200°C. The door to the furnace was open during the measurement process to facilitate video recording and measuring of the thermal expansion but a fireproof pane of glass covered the opening as seen in **Figure 1**.



Figure 1. Furnace and the pane of fireproof glass

Two frames made of stainless steel wires were mounted on a stainless steel plate. The height and distance between the frames was 70 mm. In addition to these frames a ceramic plate, 120 mm height, was also mounted on the stainless steel plate. A picture of the frames is show in **Figure 2**.

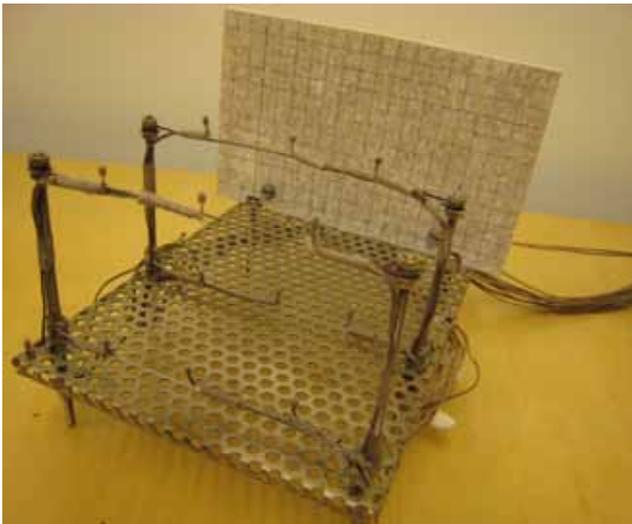


Figure 2. The frames were set up by stainless steel plate and wires.

In order to make observations easier a grid was drawn on the ceramic plate. Sixteen thermocouples were located on the steel wires, eight on each frame. Five of these eight thermocouples were located almost at level of steel plate and the remainder located at 70 mm above the surface of steel plate. The distance between thermocouples on the same level was 40 mm.

The use of glass instead of the door can cause a higher heat loss through heat radiation through glass than the standard door does and thus lead to a non uniform temperature distribution in the furnace. Thus temperatures in different positions at the frame were measured by placing the frame without any samples in the furnace and increasing the temperature of the furnace. The measured temperature showed the degree of temperature uniformity around the samples.

A video measuring gauge, FOR-A-IV 560, was used for measuring the thermal expansion of the fire seals. FOR-A IV-560 is a video measuring gauge that superimposes circles, squares and/or crosslines around any object in a TV picture and gives horizontal, vertical and diagonal values in XY- coordinates. The ceramic plate, height=120mm, could be covered by 197 steps of the Video measuring gauge i.e. 0.6 mm per step. A rough estimation of the possible observation errors, concerning measuring thermal expansion of the fire seals, was approximated to ± 0.6 mm or one step.



Figure 3. FOR-A IV-560 and TV screen

The two samples of same fire seal that had been exposed to the same environmental conditions were placed in the measuring frame, see **Figure 4**.



Figure 4. Position of the samples in measuring frame.

The samples were photographed before heating. The measuring frame and samples were placed in the oven. The pane of glass was placed in front of the furnace opening. Heating of the samples and video recording was started. Temperatures were measured by the thermocouples on the frames and the results were logged. The experiment continued until a maximum thermal expansion was achieved, this was determined by looking at the sample. The samples were taken out of the oven and photographed. Reaction time and thermal expansion of the samples were determined using the FOR-A IV-560 and the reaction temperature was determined using the reaction time and the measured temperatures.



Figure 5. Video recording and photographing of samples after heating

4 Results and analysis

The results from the measurements of the thermal expansion and reaction temperature are discussed in the following sections separately for each of the different pre-test environmental conditions. The analysis of the experiments relies however on the temperature and possible gradients in the furnace and therefore this is discussed first.

4.1 Temperature in the furnace

Temperatures in different positions on the frame were measured. A top view of the furnace and location of the frame with the position of the thermocouples is shown in **Figure 6**, see also Figure 2.

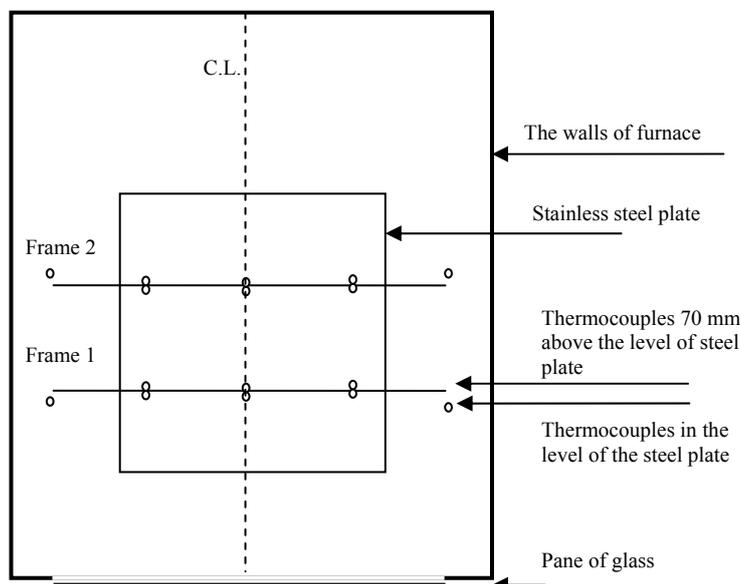


Figure 6. Top view of the furnace, the frame and the position of thermocouples

The results of measurements show that there was a temperature gradient in the furnace chamber. The temperature gradient was about 20 °C between the thermocouples located in the level of the steel plate and the thermocouples that were located 70 mm above the level of the steel plate. This temperature gradient was developed when the temperature in the chamber furnace was about 200 °C, see **Figure 7**. The temperature gradient was of the same range (20 °C) for both frames.

By assuming a linear gradient, a temperature difference of 20°C over a 70 mm height gives a temperature increase of 1°C for each 3.5mm. The thickness of samples was about 2-3 mm thus, the temperature gradient over the cross section of the samples could be about 1°C.

The mean temperature of the thermocouples on the frame 1 was 3 °C lower than the thermocouples on the frame 2. The mean temperature of the thermocouples located on the symmetry line was about 2 °C higher than the thermocouples on the right- and left hand side of the symmetry line, see figure 6.

The temperature measured by the thermocouples located at the level of the stainless steel plate differed by ± 2 °C. This level of temperature difference was accepted to be reasonable for further measurements.

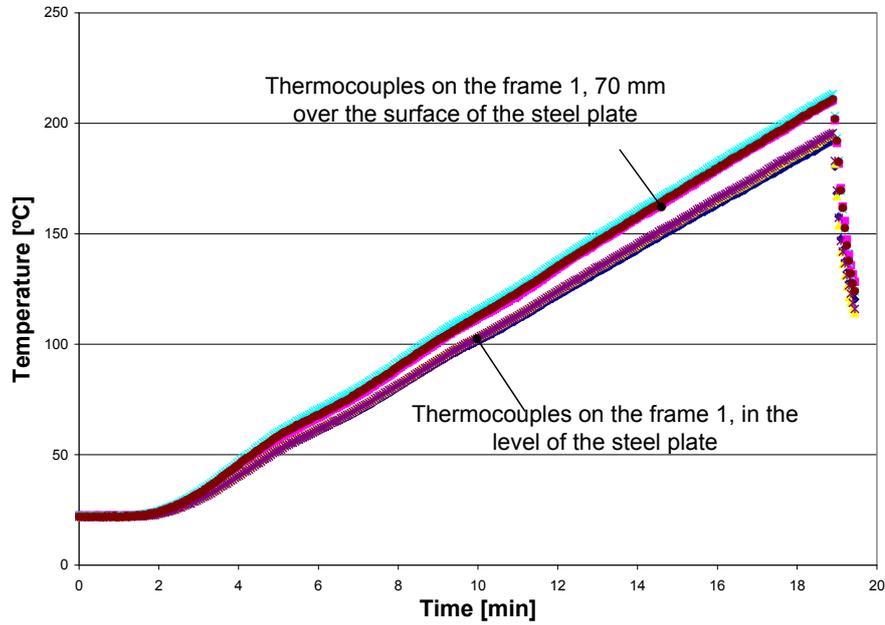


Figure 7. Measured temperature by thermocouples located on the frames 1.

The temperature difference between the frames was 3°C and the samples were placed on the stainless steel plate with a mean temperature difference of 2°C. This means that the temperatures at the level of the stainless steel could be accepted as a uniform temperature distribution. Thus, the temperatures measured by thermocouples placed on the level of the stainless steel plate were used as the temperature which the samples of fire seals were exposed to in further measurements.

4.2 Measuring of reference samples

Two reference samples of fire seals A-H, see **Table 1**, were prepared and exposed to a temperature increase of 10 °C/min. Furthermore, samples of fire seals E, F and H were exposed to a temperature increase of 5 °C/min. Reference samples were samples that had not been exposed to a harsh environment but to normal lab conditions, i.e. 20°C and 60% relative humidity.

The results of these measurements are presented in **Table 3**. The measured parameters are reaction time (Rtim), reaction temperature (Rtemp), initial thickness (Ini-thic), thickness after expansion (Exp-thic) and expansion ratio (Exp-ratio) which is Exp-thic divided by Ini-thic.

Table 3. Reaction time, reaction temperature and expansion of the reference samples

Seal	Rtim [min]	Rtemp [°C]	Ini-thic [mm]	Exp-thic [mm]	ExpRatio
A	27.0	281	4.2**	21.3	5.0
B	17.0	171	2.6	7.9	3.0
C	18.5	188	2.5	6.1	2.5
D	22.0	217	2.5	21.9	9.0
E	17.0	169	2.2	25.0	11.5
E*	31.0	161	2.2	18.9	8.6
F	13.5	133	1.9	14.0	7.5
F*	21.0	117	1.9	14.0	7.4
G	14.0	140	3.3	15.8	5.0
H	17.0	169	2.5	26.2	10.5
H*	29.0	151	2.5	19.5	7.8

* Results related to the 5 °C/min temperature increase

** Fire seal 'A' had an irregular (U-shape) geometry. The presented value in the table is the thickness of the edge of the fire seal.

- Influence of time-temperature

The results presented in **Table 3**, comparing fire seals 'E' and 'E*', 'F' and 'F*', 'H' and 'H*', indicate that the thermal behaviour of the fire seals varies depending on the rate of temperature increase. The Reaction temperature of the samples 'E', 'F' and 'G' when exposed to 5 °C/min were lower than the reaction temperature for 10 °C/min temperature increase. Expansion ratios, the ratio of the initial thickness to the expanded thickness, of the samples 'E' and 'H', graphite based fire seals, exposed to 5 °C/min were also lower than 10 °C/min exposure, see **Figure 8** and **9**. The expansion ratio of sample 'F', Sodium Silicate based fire seal, was the same in both exposure rates, see **Figure 10**.



Figure 8. Fire seal E, Left: exposed to 10 C/min Right: exposed to 5 C/min

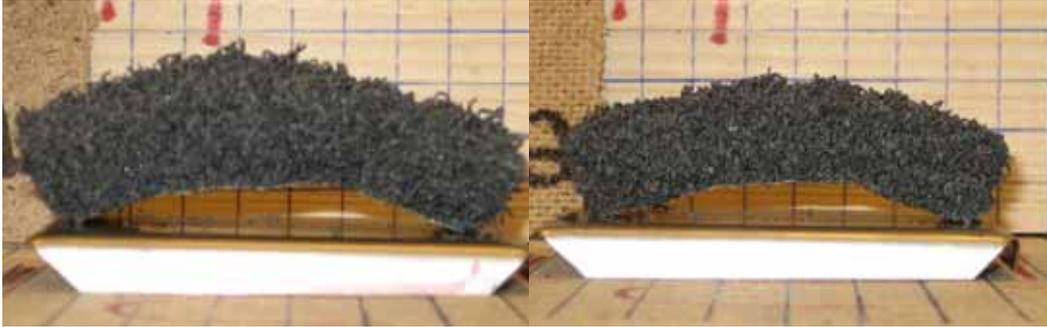


Figure 9. Fire seal H, Left: exposed to 10C/min Right: exposed to 5C/min

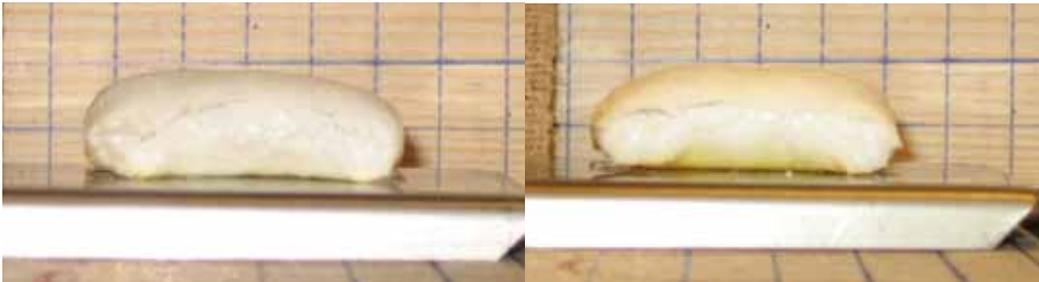


Figure 10. Fire seal F, left: exposed to 10 C/min right: exposed to 5 C/min

- Influence of casing

Samples of fire seal 'C' had the same chemical composition as samples of fire seal 'B' with an integrated casing of TPE+PVC. Using a casing for fire seal 'B' caused a 10 % higher reaction temperature (17 °C), and about 17 % lower expansion ratio as shown in **Figure 11**.

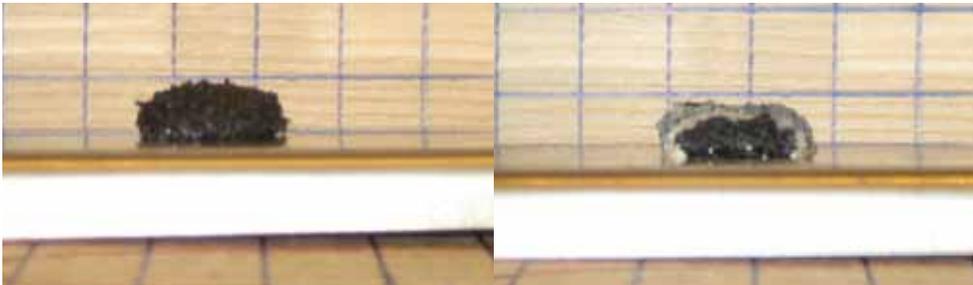


Figure 11. Left: Expansion of fire seal 'B' Right: Expansion of fire seal 'C'

Samples of fire seal 'G' has the same chemical composition as samples of fire seal 'F' with a separate protection casing of TPE+PVC. The thickness of the protection casing is 1.4 mm. The thickness of the samples 'G' after expansion was 15.8 mm. Subtracting the thickness of the protection casing from the thickness of sample 'G' after expansion, assuming that the casing did not expand; indicate that the casing has almost no influence on the expansion ratio. However, the reaction temperature increased slightly, see **Figure 12**.

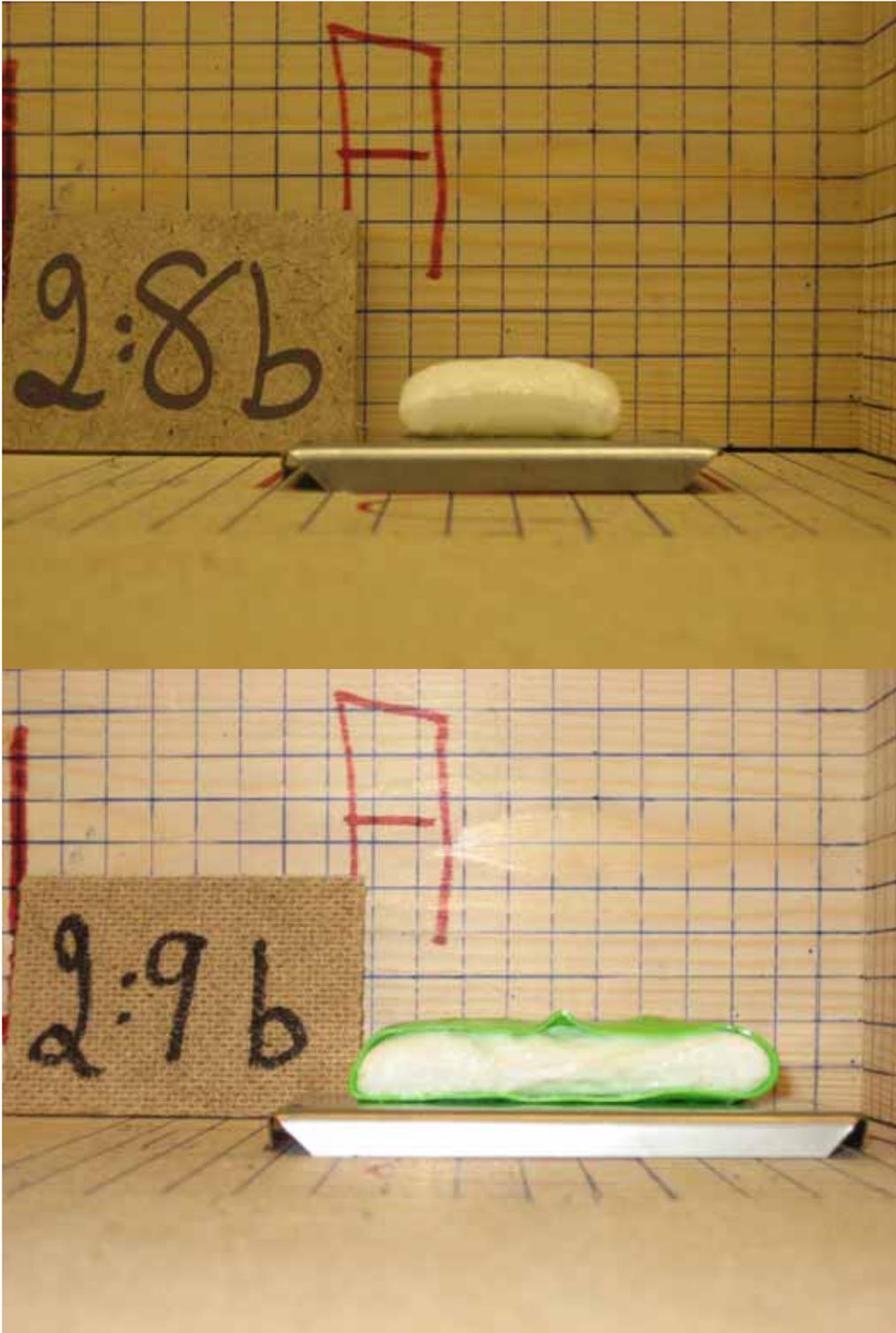


Figure 12. Upper: Expansion of fire seal 'F' Lower: Expansion of fire seal 'G'

4.3 Water storage

Four samples of each fire seal ‘A’, ‘B’, ‘D’, ‘E’, ‘F’ and ‘H’ were stored in a water bath. Two samples were stored for three days and two samples were stored for thirty days before exposing them to the heat in the furnace. Fire seal ‘F’ resolved partly after three days and the F samples were totally dissolved after thirty days. The samples were weighed before and after water storage, see **Fel! Hittar inte referenskälla.**

Table 4. Water absorption of the fire seals after 3 and 30 days of water storage

Seal	Water absorption [%] of initial mass	
	Three days	Thirty days
A	1.48	3.05
B	0.00	3.25
D	5.71	9.81
E	8.26	29.75
H	26.13	38.60

It is known that the longer the time of water storage, the more water absorbed by the fire seals until they are water saturated. Water absorption ratio of the fire seals varied largely between seals as seen in **Fel! Hittar inte referenskälla.** Fire seal ‘B’ absorbed no water after three days and absorbs about 3% of its mass after 30 days i.e. a very slow water absorption ratio. Fire seal ‘H’ absorbed 26% of its mass after three days and about 39% after 30 days i.e. a very fast water absorption ratio.

These samples were then tested according to the same procedure as the reference samples and the reaction temperature and expansion ratio compared, as presented in **Figure 13**, **Figure 14**, **Table 5** and **Table 6**.

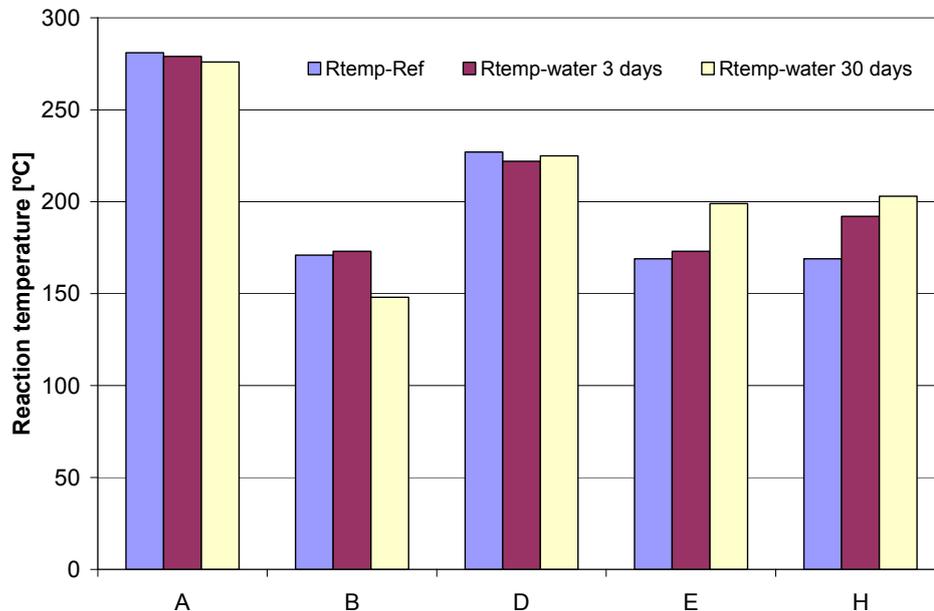


Figure 13. Reaction temperature of water stored samples and reference sample.

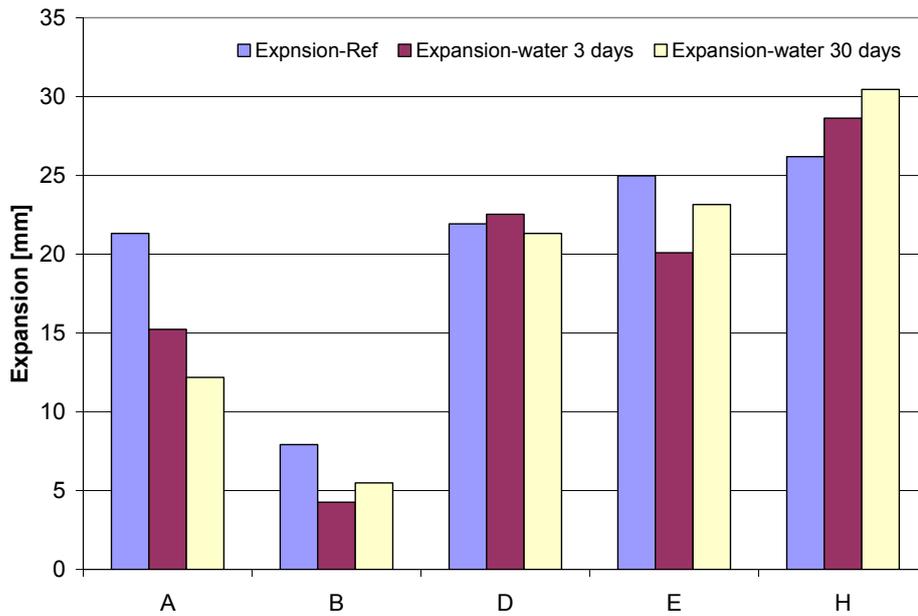


Figure 14. Expansion due to heat exposure of water saturated samples and reference sample.

Table 5. Relation between reaction temperature of water stored samples and reference samples

Seal	(Rtemp) / (Rtemp-Ref)	(Rtemp) / (Rtemp-Ref)
	Three days	Thirty days
A	0.99	0.98
B	1.01	0.87
D	0.98	0.99
E	1.02	1.18
H	1.14	1.20

Table 6. Relation between expansion of water stored samples and reference samples

Seal	(Exp)/(Exp-Ref)	(Exp)/(Exp-Ref)
	Three days	Thirty days
A	0.71	0.57
B	0.54	0.69
D	1.03	0.97
E	0.80	0.93
H	1.09	1.16

Results presented in **Figure 13** and **Figure 14** indicate that the expandability and reaction temperature of the seals varies with water content. The magnitude and behaviour of these variations was different for each fire seal, thus the behaviour of each fire seal was examined individually.

The expandability of water exposed samples of fire seal 'A' in comparison with the reference sample was reduced by 30% and 40% after three and thirty days of water storage, respectively. However, the reaction temperatures were in the same range as the reaction temperature of the reference sample.

The expandability of water exposed samples of fire seals 'B' was reduced by 50% and 40% after three and thirty days of water storage, respectively. The reaction temperature of

fire seal 'B' after three days of water storage was of the same order of magnitude as the reaction temperature of the reference sample but the reaction temperature after thirty days was lower than reaction temperature of the reference sample. Here it should be mentioned that the mass of fire seal 'B' was not changed after three days thus, it is more interesting to investigate the reasons for the reduction of the expandability after 3 days of water storage.

The expansion of water exposed fire seals 'D' changed slightly due to the water storage while the reaction temperatures of fire seal 'D' after three and thirty days of water storage respectively were of same order of magnitude as the reaction temperature of the reference sample.

The expansion of water exposed fire seals 'E' was reduced by 20% and 8 % after three and thirty days of water storage, respectively. The reaction temperature of fire seal 'E' after three days water storage was of the same order of magnitude as the reaction temperature of the reference sample but the reaction temperature after thirty days was higher than the reaction temperature of the reference sample.

Finally, the expansion of saturated fire seals 'H' were increased by 10% and 15 % after three and thirty days of water storage, respectively and the reaction temperature of fire seal 'H' increased by 12% and 20 % after three days and thirty days of water storage, respectively.

This altered behaviour of the fire seals indicated that different chemical compositions of the fire seals had been affected by water storage depending on the duration of the water storage.

4.4 Drying

Two samples of each fire seal ‘A’, ‘B’, ‘D’, ‘E’, ‘F’ and ‘H’ were stored in an oven at 105 °C for three days before exposing them to heat. The moisture content of the samples was determined by weighing the fire seals before and after drying, see **Table 7**. The samples were then exposed to heat according to the same procedure as the reference samples and the results compared as presented in **Fel! Hittar inte referenskölla.** and **Fel! Hittar inte referenskölla.**

Table 7. Moisture content of the samples

Seal	Moisture content mass by mass [%]
A	1.49
B	1.60
D	0.75
E	0.41
F	3.96
H	1.55

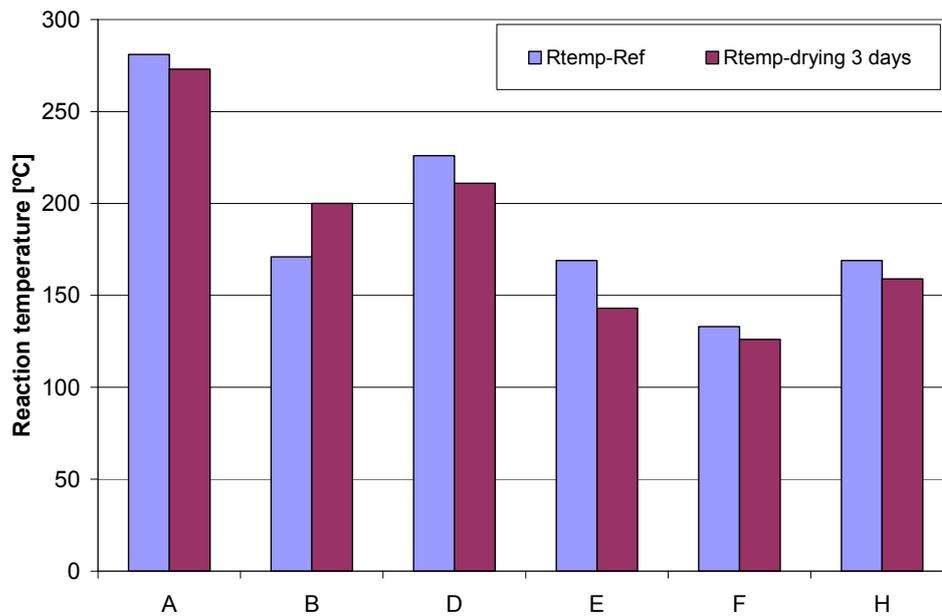


Figure 15. Reaction temperature of dried samples and reference sample.

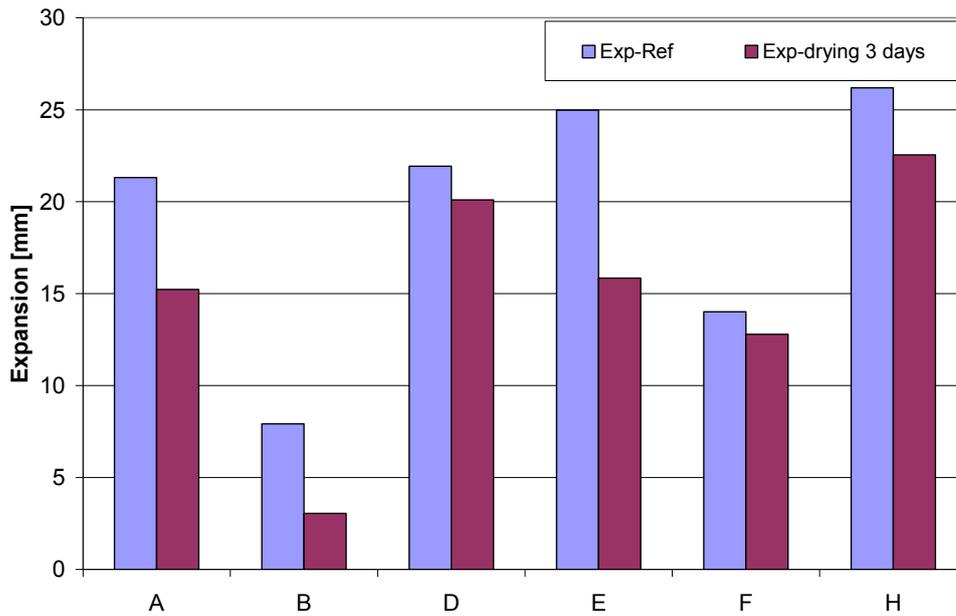


Figure 16. Expansion of dried samples and reference sample.

Generally, the thermal expansion of the dried samples were lower than the thermal expansion of the reference samples of the fire seals. The relation between expansion of the dried samples and the expansion of the reference samples is presented in **Table 8**.

Table 8. Relation between reaction temperature and expansion of dried samples and reference samples

Seal	(Rtemp-dried) / (Rtemp-Ref)	(Exp-dried) / (Exp-Ref)
A	0.97	0.71
B	1.17	0.38
D	0.93	0.92
E	0.85	0.63
F	0.95	0.91
H	0.94	0.86

With the exception of fire seal 'B' the reaction temperature of all fire seals after drying was lower than for the reference samples.

The expansion of fire seal 'B' after drying was 60% lower than the expansion of the reference sample i.e. this fire seal was very sensitive to drying. The expansion of the other fire seals dropped about 10-40% after drying.

4.5 Freezing

Two samples of each fire seal ‘A’, ‘B’, ‘D’, ‘E’ and ‘H’ were immersed in a water bath for one hour, the water bath was then placed in a chamber at $-5\text{ }^{\circ}\text{C}$ for 24 hours. Finally the temperature of the chamber was increased to $0\text{ }^{\circ}\text{C}$ and the samples were taken out of the chamber when the temperature of the water bath was $\pm 0\text{ }^{\circ}\text{C}$ before exposing them to heat. The results of the measurements on the samples that had been frozen were compared with the results of reference samples, see **Figure 17** and **Figure 18**.

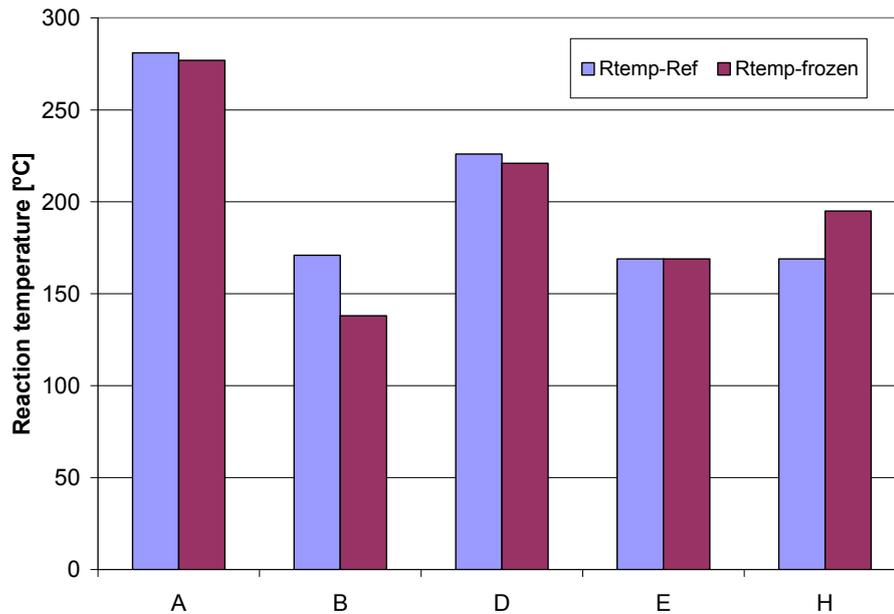


Figure 17. Reaction temperature of frozen samples and reference sample.

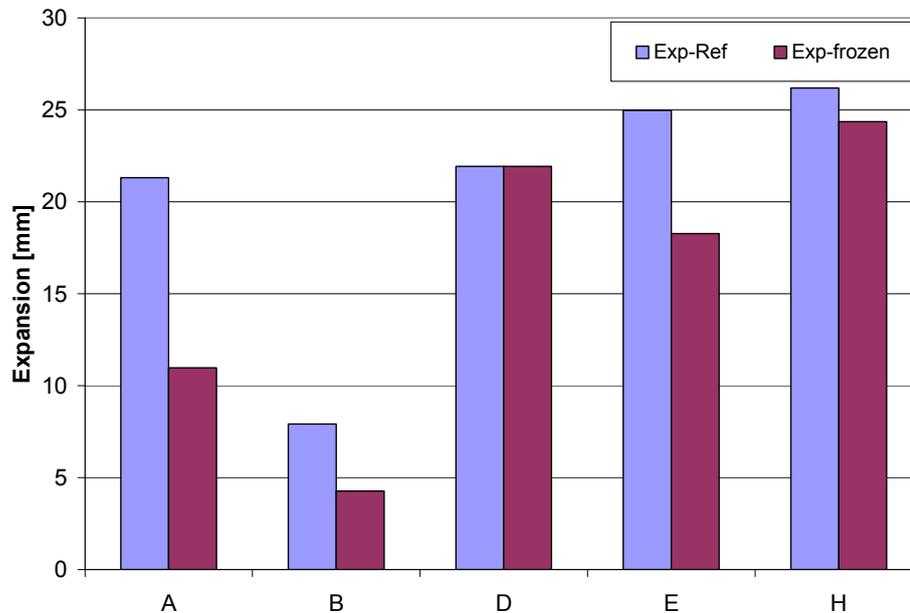


Figure 18. Expansion of dried samples and reference sample.

The reaction temperatures of fire seals ‘A’, ‘D’ and ‘E’ were in the same order of magnitude as the reference samples of the fire seals, i.e. there were no influence of freezing on reaction temperature. The reaction temperature of fire seal ‘B’ decreased by

about 20% and the reaction temperature of fire seal 'H' increased by 15% compared to the reference samples.

Table 9. Relation between reaction temperature and expansion of frozen samples and reference samples

Seal	(Rtemp-frozen) / (Rtemp-Ref)	(Exp-frozen) / (Exp-Ref)
A	0.99	0.51
B	0.81	0.54
D	0.98	1.00
E	1.00	0.73
H	1.15	0.93

With the exception of fire seal 'D' expandability of the fire seals was affected by freezing. Fire seals 'A' and 'B' were most sensitive to freezing and lost about 50% of expandability.

4.6 NaCl -solution

Two samples of each fire seal ‘A’, ‘B’, ‘D’, ‘E’ and ‘H’ were stored in salt solutions for 3 days before exposing them to heat. Two different concentrations of salt in water were chosen namely 5% and 100% (saturated). The results of the reaction temperature and expansion measurements were compared with results of the reference samples and the results of three days of water storage, see **Figure 19** and **Figure 20**.

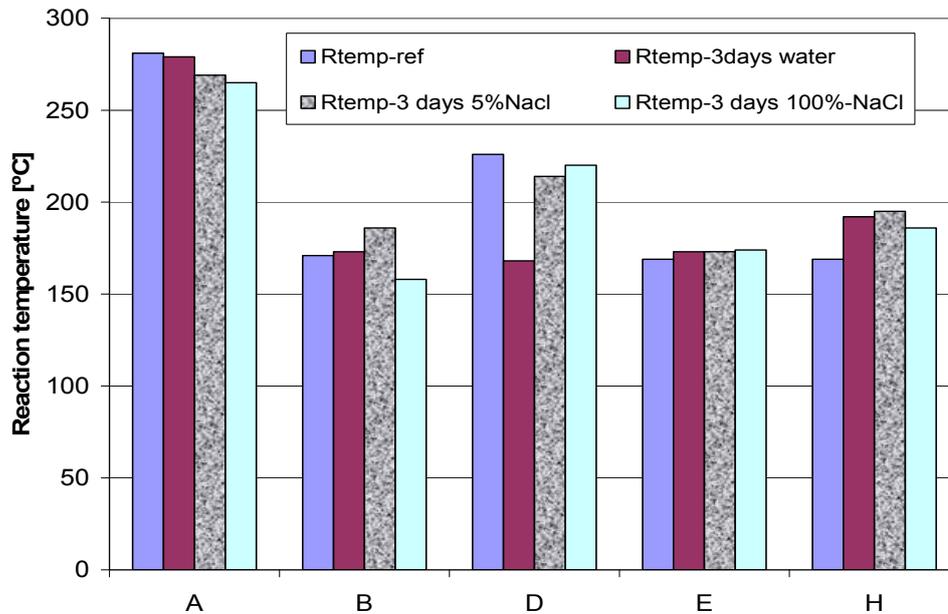


Figure 19. Reaction temperature of samples stored in salted water, reference sample and samples stored three days of water storage.

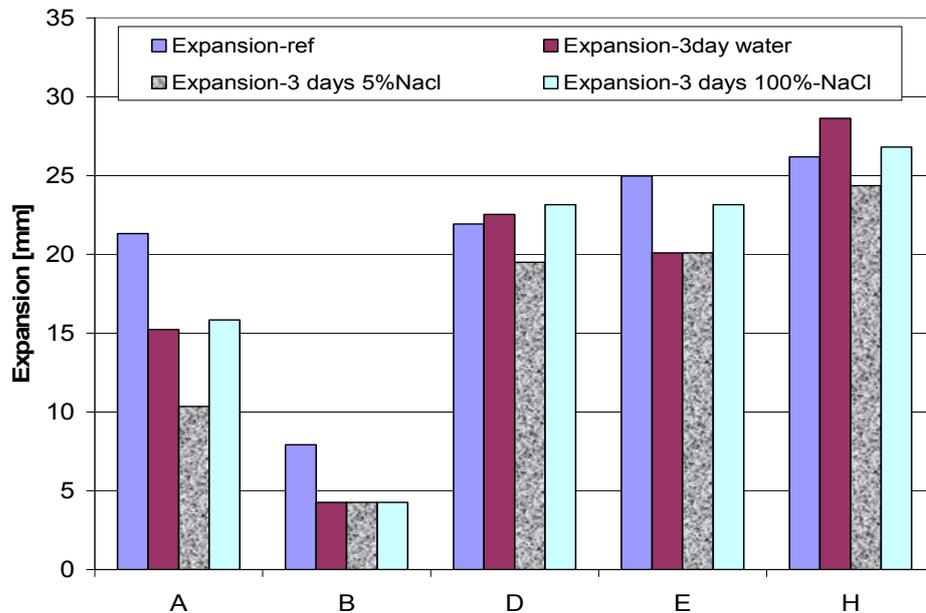


Figure 20. Expansion of samples stored in salted water, reference sample and samples stored in water for three days .

The influence of the salt solution on the fire seals was carried out by comparing the results obtained for the samples stored in water for three days and the samples stored in

salt solution for three days. This comparison illustrates the influence of salt in water on the reaction temperature and the expandability of the seals. Furthermore, the influence of 5% and 100% concentration of salt in water on the behaviour of the fire seals was investigated, see **Table 10** and **Table 11**.

Table 10. Relation between reaction temperature of samples stored in 5% and 100% salt solution with samples stored in water for three days.

Seal	(Rtemp-5% salt)/(Rtemp water 3 days)	Rtemp-5% salt/Rtemp-100%salt
A	0.96	1.02
B	1.08	1.18
D	0.96	0.97
E	1.00	0.99
H	1.02	1.05

The reaction temperature of fire seals 'B' was sensitive to the salt solution. The changes in the reaction temperature of the other fire seals were almost of the same order of magnitude as the reaction temperature of the samples stored in water.

Table 11. Relation between expansion of samples stored in 5% and 100% salt solution with samples stored in water for three days.

Seal	(Exp-5% salt) / (Exp water 3 days)	(Exp-5% salt) / (Exp-100%salt)
A	0.68	0.65
B	1.00	1.00
D	0.86	0.84
E	1.00	0.87
H	0.85	0.91

At the 5% salt concentration in solution, the expandability of fire seals 'A', 'D' and 'H' was reduced by 35% ,15% and 15% respectively, relative to the samples stored in water for three days. At the 5% salt concentration in solution, the expandability of these fire seal increased almost to the same level as the samples stored in water for three days .

The expandability of fire seal 'B' was not affected by the 5% or 100% salt concentration in the solution.

Fire seals 'E' was not affected by 5% salt concentration in solution however, its expandability was reduced by 13% in the saturated salt solution.

4.7 Cleaner compound

Two samples of fire seal ‘A’, ‘B’, ‘D’, ‘E’ and ‘H’ were immersed in a bath of cleaner compound for 3 days before exposing them to heat. The results of the reaction temperature and expansion measurements were compared with results of the reference samples and the results of three days of water storage, see **Figure 21** and **Figure 22**.

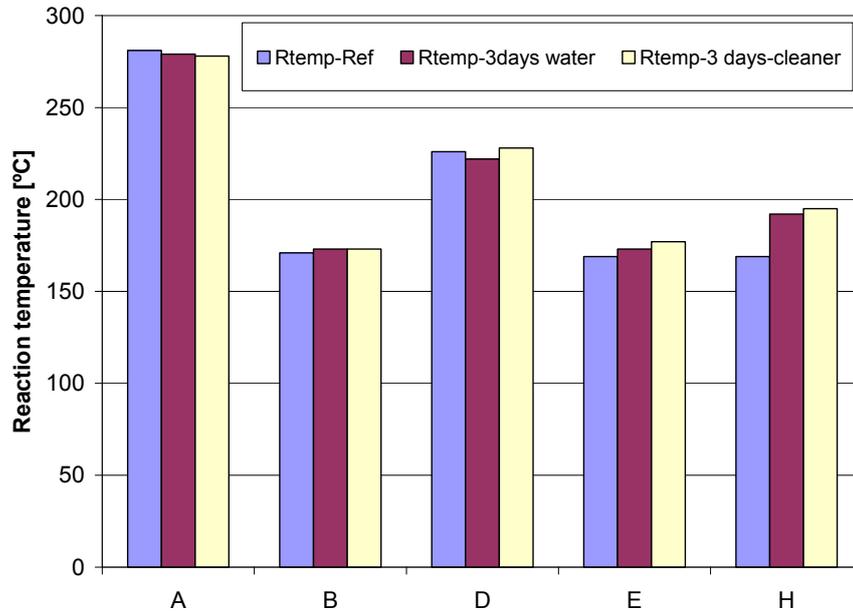


Figure 21. Reaction temperature of samples stored in cleaner compound, reference sample and samples stored for three days in water.

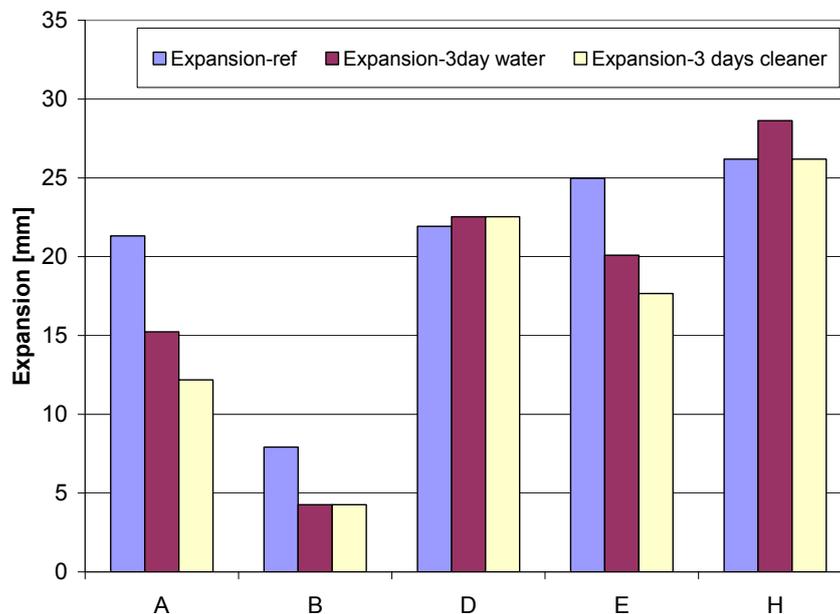


Figure 22. Expansion of samples stored in cleaner compound, reference sample and samples stored in water for three days.

Table 12. Relation between reaction temperature of samples stored in cleaner compound with reference samples and samples stored in water for three days.

Seal	(Rtemp-cleaner) / (Rtemp-Ref)	(Rtemp-cleaner) / (Rtemp-water 3 days)
A	0.99	1.00
B	1.01	1.00
D	1.01	1.03
E	1.05	1.02
H	1.15	1.02

The changes in the reaction temperature of the fire seals conditioned in cleaner compound were almost in the same order of magnitude as the reaction temperature of the samples stored in water and they were also very close to the reaction temperature of the reference samples.

Table 13. Relation between expansion of samples stored in cleaner compound with reference samples and samples stored in water for three days.

Seal	(Exp-cleaner) / (Exp-Ref)	(Exp-cleaner) / (Exp-water 3 days)
A	0.57	0.80
B	0.54	1.00
D	1.03	1.00
E	0.71	0.88
H	1.00	0.91

The expandability of fire seals 'B' and 'D' was not affected by cleaner compound, see the third column in **Table 13**. The expandability of fire seals 'E' and 'H' was reduced by about 10% and the expandability of fire seal 'A' reduced by 20% in comparison with the expansion of the samples conditioned in water for three days..

4.8 Acid precipitation (rain)

Two samples of the each of the fire seals were immersed in a bath of sulphuric acid for 3 days before exposing them to heat. The results of the measurements during heat exposure were compared with the results of reference samples and three days of water storage, see **Figure 23**. **Reaction temperature of samples stored in a bath of sulphuric acid, reference sample and samples stored three days of water storage.**Figure 23 and **Figure 24**.

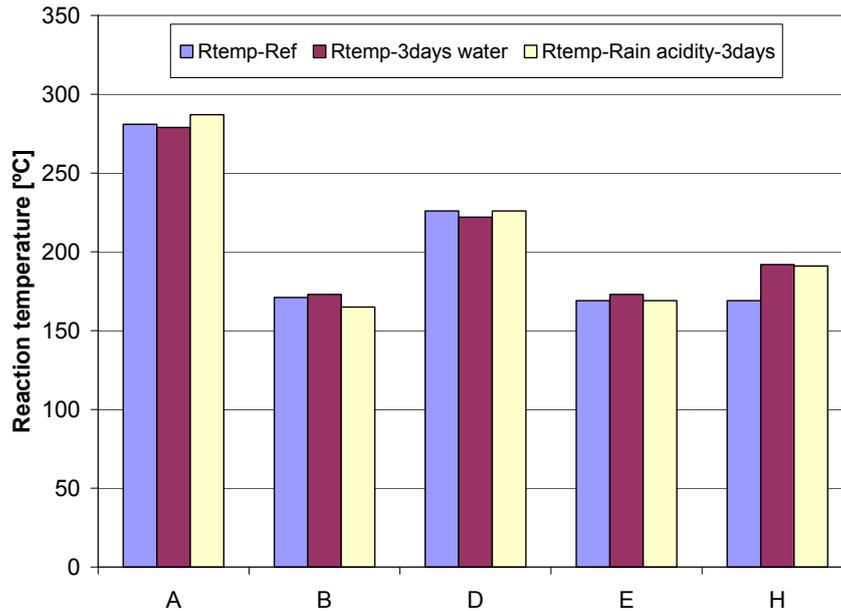


Figure 23. Reaction temperature of samples stored in a bath of sulphuric acid, reference sample and samples stored three days of water storage.

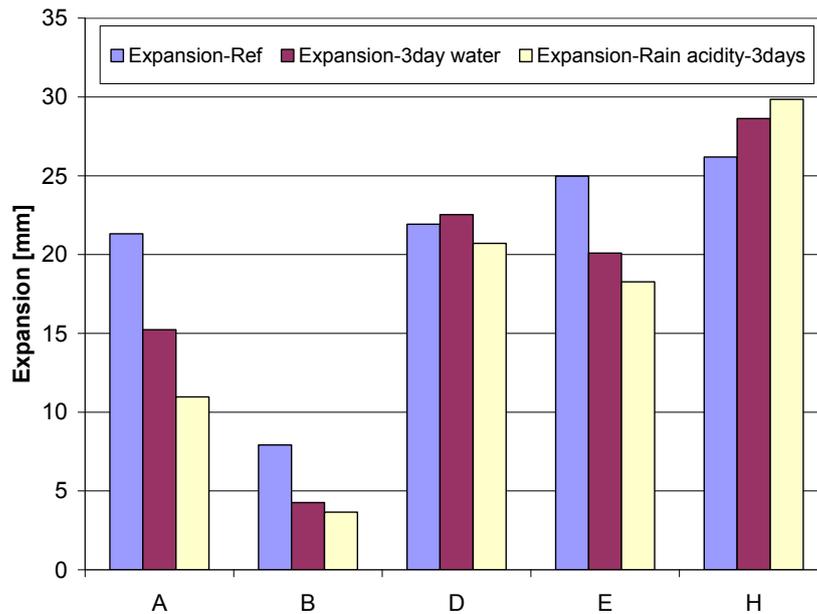


Figure 24. Expansion of samples stored in a bath of sulphuric acid, reference sample and samples stored in water for three days

Table 14. Relation between reaction temperature of samples stored in acid precipitation with reference samples and samples stored in water for three days

Seal	(Rtemp-acid precipitation) / (Rtemp-Ref)	(Rtemp-acid precipitation) / (Rtemp-water 3 days)
A	1.02	1.03
B	0.96	0.95
D	1.00	1.02
E	1.00	0.98
H	1.13	0.99

The changes in the reaction temperature of the fire seals conditioned in a bath of sulphuric acid were almost in the same order of magnitude as the reaction temperature of the samples stored in water and they were also very close to the reaction temperature of the reference samples, see **Table 14**.

Table 15. Relation between expansion of samples stored in acid precipitation with reference samples and samples stored in water for three days.

Seal	(Exp-acid precipitation) / (Exp-Ref)	(Exp-acid precipitation) / (Exp-water 3 days)
A	0.51	0.72
B	0.46	0.86
D	0.94	0.92
E	0.73	0.91
H	1.14	1.04

Comparing results of these measurements with the results of the samples stored in water for three days indicated that the expandability of fire seal 'H' was not affected by the sulphuric acid solution. The expandability of fire seal 'E' and 'D' was reduced by about 10% and the expansion of fire seal 'A' and 'B' was reduced by 28% and 14% in comparison to the expansion of the samples stored in water for three days, see **Table 15**.

4.9 Summary of the results

A summary of the results of measurements is presented in **Table 16**. In order to identify the influence of salt, cleaner compound and sulphuric acid, the results of these conditions were compared to the results of three days water storage.

In order to get a better overall view of the measured results, the measured changes in expandability and reaction temperature were classified (see in **Table 6**). Changes in the range of 0-5% , 5-10%, 10-20%, 20-30%, 30-40%, and 40-50% were classified as Class '0' to Class '5' respectively.

Table 16. Influence of different conditioning cases on expansion and reaction temperature in comparison with the reference samples and three days water stored samples.

Expansion								
Seal	Water-3days	Water-30days	drying	Freezing	Nacl-5%	Nacl-100%	Cleaner	Acid precipitation
A	3	4	3	5	4	0	2	3
B	5	4	6	5	0	0	0	2
D	0	0	1	0	2	0	0	1
E	2	1	4	3	0	2	2	1
F	Resolved	Resolved	1	-	-	-	-	-
H	1*	2*	2	1*	2	1	1	0
Reaction temperature								
A	0	0	0	0	0	0	0	0
B	0	2	2*	2	1*	1	0	0
D	0	0	1	0	0	0	0	0
E	0	2*	2	0	0	0	0	0
F	Resolved	-	1	-	-	-	-	-
H	2*	2*	1	2*	0	0	0	0

0-5% reduction is equal to 0, 1= 5-10%, 2= 10-20%, 3= 20-30%, 4= 30-40%

* Expansion or reaction temperature increase

Comparison with three days of water storage are shadowed.

5 Conclusions

Fire seal's sensitivity to different harsh environments such as acid rain, humidity, dry conditions, salty humid air has been investigated by exposing fire seals to extremes of these conditions and then exposing the seals to a linearly increasing temperature in a small scale furnace. Parameters of thermal behaviour that were studied includes reaction time, reaction temperature and expansion. The term reaction temperature refers in this case to the temperature reading in the oven when the seal start to expand. The thermal behaviour parameters for the fire seals exposed to different harsh conditions were compared with the same values for fires seals that has not been exposed to harsh conditions

The fire seals tested were of two different types i.e. sodium silicate and graphite based seals with varying composition, in total eight different fire seals were tested.

The measurement setup developed, using a fireproof pane of glass on a chamber furnace, proved to give a uniform temperature around the samples and make it possible to measure expansion of the fire seals by a video measurement gauge.

The following general conclusions can be drawn for all fire seals involved in this investigation:

- the reaction temperature of the fire seals was less sensitive to environmental parameters than their expandability.
- the expandability of fire seals based on graphite (sample E and H) depends on the rate of temperature increase. The expandability of these seals was reduced by 25% at a temperature increase of 5°C/min compared to a 10°C/min temperature increase. The expandability of sodium silicate fire seal did not vary with rate of temperature increase.
- the expandability and the reaction temperature of the dried samples were lower than the reference samples of the fire seals. Fire seals are hygroscopic material. A low moisture content in the fire seals causes lower expandability and lower reaction temperature.

The sodium silicate fire seal was dissolve partly after three days in a water bath and was totally resolved after thirty days in a water bath. Thus, the freezing, salt solution, cleaner compound and sulphuric acid solution tests were not performed on the sodium silicate based fire seals.

The expandability of the graphite based fire seals stored in water was 0- 40% lower than the expandability of the reference samples.

The expandability of the graphite based fire seals stored at freezing condition, salt solution and cleaner compound was 0-20% lower than the expandability of samples stored in water for three days.

The expandability of the fire seals conditioned in sulphuric acid solution was 0-30% lower than the samples stored in water for three days.

Finally, The results of these measurements indicated that different chemical compositions of the graphite based fire seals can be affected to different degree under different conditions.

In this study the fire behaviour of the fire seal is studied generically i.e. not coupled to a specific end use application. Normally, fire seals are applied to a building element i.e. they are enclosed between one or two other materials e.g. wood or steel. It would be useful to also study the function of the fire seals in the actual building elements after environmental exposure. However, the approach taken here by studying them generically is useful in evaluating how different compositions are affected by different environmental conditions. A more robust approach would be to apply the same type of durability requirements that are put on many building materials on fire seals. The seals can then be exposed in different climate chambers developed specifically for the purpose and then evaluated in a small scale furnace like the one used in this project.

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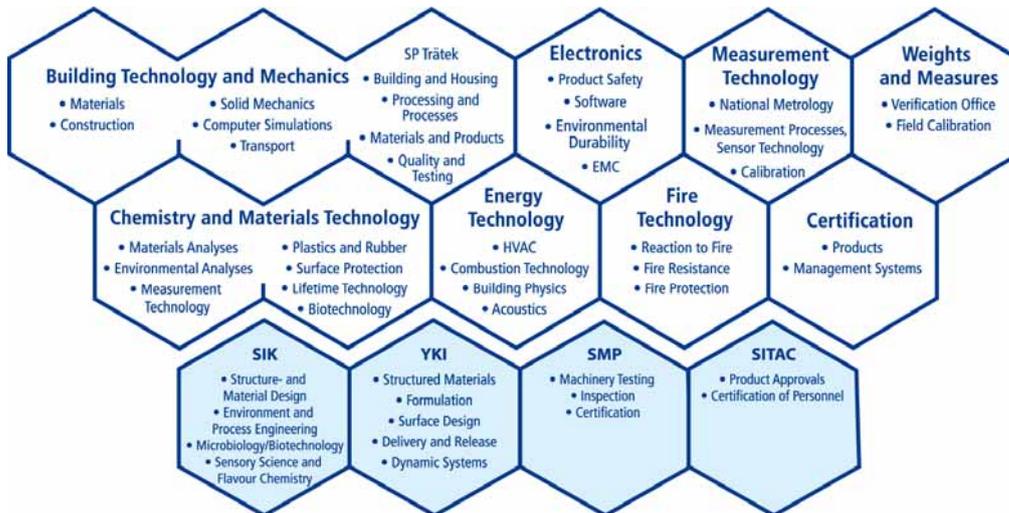
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