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THE LUND INSTITUTE OF TECHNOLOGY

# DURABILITY AND AGEING OF SEALANTS

PER GUNNAR BURSTRÖM

REPORT TVBM-3003  
LUND SWEDEN 1977

# DURABILITY AND AGEING OF SEALANTS

PER GUNNAR BURSTRÖM

THIS REPORT REFERS TO PROJECT NUMBER 730221-4 FROM  
THE SWEDISH COUNCIL FOR BUILDING RESEARCH TO THE  
DIVISION OF BUILDING MATERIALS AT THE LUND INSTITUTE  
OF TECHNOLOGY

## PREFACE

This report is a translation of an earlier published report in the Swedish language, "Beständighet och åldring hos fogmassor", Lund 1976.

The research project that the report deals with has to a major part been financed by grants from the Swedish Council for Building Research to the Division of Building Materials at the Lund Institute of Technology.

The report deals with the basic factors affecting the ageing and their effect on eight common types of sealants. Changes of the deformation characteristics of the sealants, essentially the stress-strain curve determined at well defined conditions, have been used to determine the effect of the ageing causing factors on the different sealants. These results are connected to values obtained after outdoor exposure of sealants at both climatical and mechanical (varying joint movements) stress. Where a couple of sealants are concerned there are some connections outlined in the report between accelerated heat ageing and natural ageing.

Some dimensions used in the text and the figures will probably cause a lot of problems for especially the transatlantic reader. In order to minimize these problems, the most common dimensions are translated as follows:

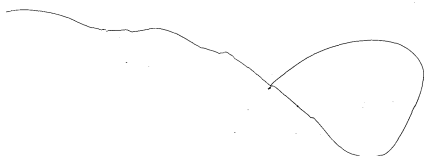
+ 2°C	=	+ 35.6°F
+20°C	=	+ 68 °F
+23°C	=	+ 73.4°F
+40°C	=	+104 °F
+70°C	=	+158 °F
1 mm	=	0.0394 am. inch
1 mm/min	=	0.0394 am. inch/min
1 Newton	=	0.22 pound
1 MPa	=	142.24 psi



It is my hope that some of the results will be useful when choosing a sealant, when working out appropriate accelerated ageing methods, and when making new products.

Lund, June 1977

Per Gunnar Burström



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## SUMMARY

*This report deals with different basic factors affecting the ageing and their effect chiefly on the deformation characteristics of the sealant. The effect of the factors has been specially studied on eight different, commonly used types of sealants. The ageing influencing factors which have been examined are temperature, moisture, alkaline water, UV-light, and ozone. Furthermore the effect of natural ageing, i.e. the effect of the natural climate combined with forced joint width variations, has been studied.*

*The temperature in the ageing influencing factor which has been found to have the greatest effect on the deformation characteristics of the sealants. But at the same time it can be established that heat storage affects different materials differently. It is therefore impossible to say, for example, whether or not there is a general connection between accelerated ageing in heat and natural ageing in different types of sealants. Where a couple of the sealants are concerned there are some connections outlined in the report between accelerated heat ageing and natural ageing.*

## Background

Most sealants are used in external walls, e.g. between concrete elements, between window frame and wall, in dilatation joints, when glazing, etc. This means that the sealants are exposed to great climatical stress. First and foremost the efficiency of the one-step joint, which is very common in Sweden, is completely dependent on the characteristics of the sealant, its ability to follow the joint movements without any permanent deformation arising and to be able to maintain this property also under the influence of the surrounding environment.

Because of the organic nature of the sealants, some changes in the material do occur with time - the material ages. The extent depends a great deal on the surrounding environment but the type of sealant is also important as some polymers are more resistant than others. But at present the knowledge regarding sealant ageing characteristics is very limited. This means that the material is often selected at random, and therefore, after a relatively short time, one can be faced with difficult and expensive re-sealings. When developing new materials problems also arise when the durability and life of the new material has to be estimated.

#### Aim

The aim of the project has been to try and clarify how certain isolated factors causing ageing affect the deformation characteristics of different types of sealants. After this the aim has been to combine some of these factors in order to find a possible combined effect. Apart from this the purpose has naturally been to correlate these results to the experiences obtained from practical experience.

The results from the investigation will hopefully contribute to making

- the selection of material easier
- estimates of the life
- good testing methods possible
- the formulation of the products easier.

#### Method

The effect of the factors causing ageing - temperature, moisture, alkaline, UV-light, and ozone - has been studied on eight different types of sealants. These consisted of two different polysulfide based sealants (soft and hard

types respectively), two different polyurethane based sealants (1 and 2 component respectively), two different acrylic sealants (solvent release), one water dispersed acrylic sealant, and one oleo resinous sealant.

In view of the changes in the deformation characteristics of the sealants, especially the stress-strain curves determined in well controlled conditions, the effects of the ageing factors have been quantified for every sealant. These results have been correlated to the values obtained after outdoor exposure of sealants to both climatic and mechanical (varying joint movements) forces.

In order to quantify changes in the materials (apart from determining the stress-strain curve) determinations have also been carried out regarding weight and volume losses in different climates. Photographing and to some extent Scanning Electron Microscopy have also been used. Furthermore changes in hardness have, as a matter of routine, been determined with an instrument measuring the Shore A hardness.

## Results

The temperature is the ageing influencing factor which has been found to have the greatest effect on the deformation characteristics of the sealants. Heat ageing at  $+40 - +70^{\circ}\text{C}$  usually increases the deformation resistance of the sealants to a high extent. But at the same time it is found that the heat storage affects the different materials differently. For instance the deformation resistance decreases in the soft polysulfide based sealant which has been heat aged at  $+70^{\circ}\text{C}$ . Experience so far has in most cases proved that heat ageing causes an increase in the hardness of polysulfide based sealants. But for most of the other types of sealants the heat ageing usually caused a definite increase in the hardness, which pretty well agrees with what happens in the case of ageing

in a natural climate. But the extent of the increase depends a great deal on the type of material. So, for example, it is absolutely impossible to be able to state a general connection between accelerated ageing in heat and natural ageing.

In materials where the effect of heat ageing clearly dominates the effects of other factors ageing influencing, some connections have been shown. In Fig 1 the stress at 50% tension as a function of time as shown for the polyurethane based sealant (1 component) after storing in different climates. By comparing the change of the stress-strain curve for the same material when exposed to outdoor environment (Fig 2) a connection between the time for heat and the time for out-door storage is obtained. Fig. 2 shows, for example, that  $\sigma_{50}$  for material exposed to out door environments for 7 months corresponds to  $\sigma_{50}$  for the material being heat aged for approximately 10 days at  $+70^{\circ}\text{C}$  (Fig. 1). This means that for this polyurethane based sealant the heat ageing at  $+70^{\circ}\text{C}$  produces an increase of hardness which is about 20 times as rapid as for outdoor environment.

For the oleo resinous based sealant, whose ability to fulfill its sealing function depends to a great extent on the characteristics of the skin (toughness and growth rate), the investigation has shown a good correlation between the growth rate of the skin in a climate of  $20^{\circ}\text{C}/50\%$  relative humidity and in an outdoor climate (Fig. 3). By comparing the skin growth rate at  $+70^{\circ}\text{C}$  with the curve in Fig. 3 it is found that the heat ageing increases the rate with a factor about 22.

In the same way moisture- and alkaline-storage affects the deformation characteristics but to a different extent. The variations are smaller than in the case of heat ageing. However for materials that normally cure because of the air humidity this ageing produces a marked increase in hardness. Most "plastic" sealants, which have been a



part of this investigation, usually absorb water which leads to a softening. If the water is allowed to evaporate this can also have led to an increase in hardness due to the leaching of some substances.

UV-light only affects the surface of the sealants. The effect that arises can e.g. be a crackling and an embrittlement of the surface, a folding, or a changing of the colour. In the case of outdoor exposed specimen joints a deeper cracking has been observed in some materials. This is due to the effect of the UV-light together with forced deformation changes. This effect can also be observed on materials in constructions used on buildings. It was mainly the soft polysulfide based sealant which showed this effect.

Ozone has only affected the two polysulfide based sealants. Furthermore the condition for ozone attack is that the materials are stored under stress. The attack that has arisen is a characteristic cracking located at right angles to the direction of the tensile stress.

The outdoor exposure of specimen joints and the results from measurements of the magnitude of the stress relaxation indicate that the two "elastic" polysulfide based sealants act in the same way as plastic materials for the seasonal joint movements and mainly like elastic materials for daily joint movements! Both the other "elastic" sealants, 1 and 2 komponent polyurethane based respectively, are essentially elastic both regarding seasonal and daily joint width variations.

$\sigma_{50}$ 

MPa

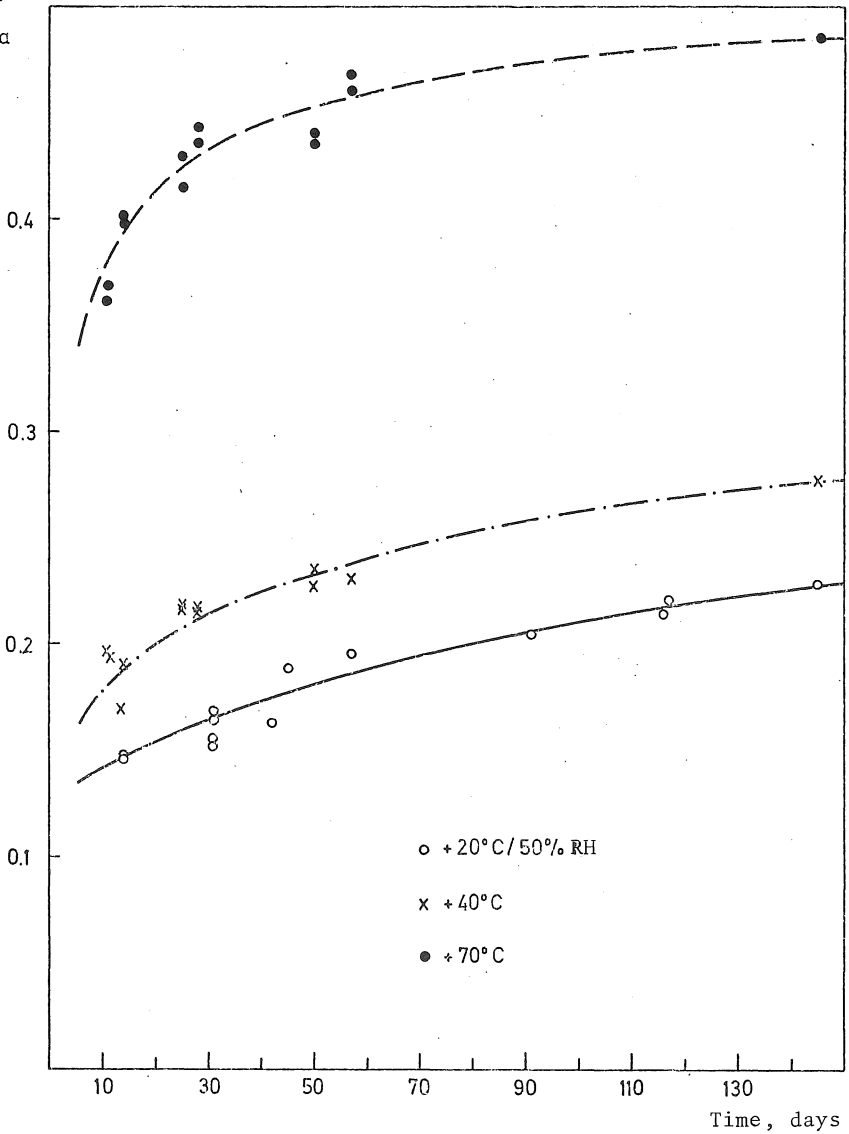


FIG 1 Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 3" as a function of storage time at different temperatures.

Initial storage: 7 days at +20°C/50% RH.

Tensile stress  
MPa

Outdoor exposure, months

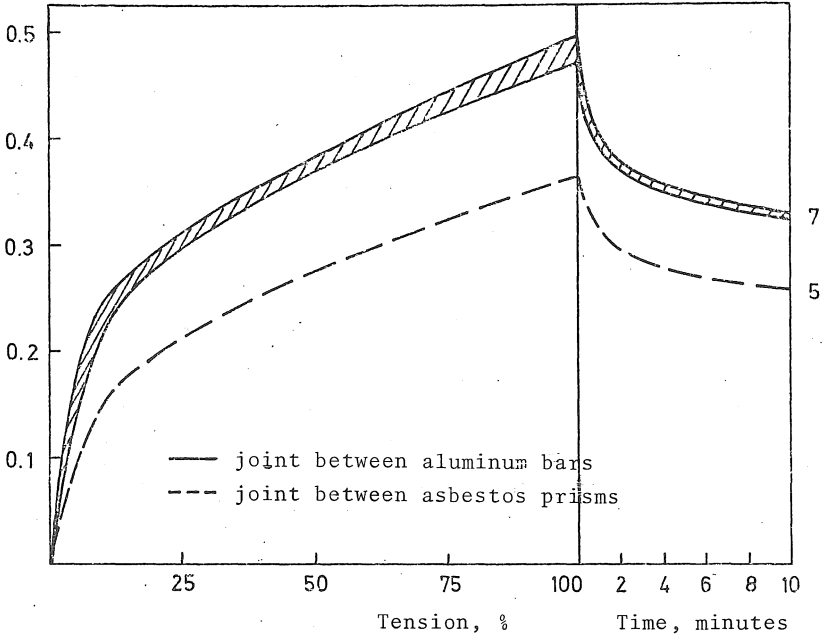


FIG 2 Stress-strain curves for "Material No 3" after varying time of outdoor exposure.  
Deformation speed: 1.5 mm/min  
Testing temperature: +2°C

Skin thickness (= S)

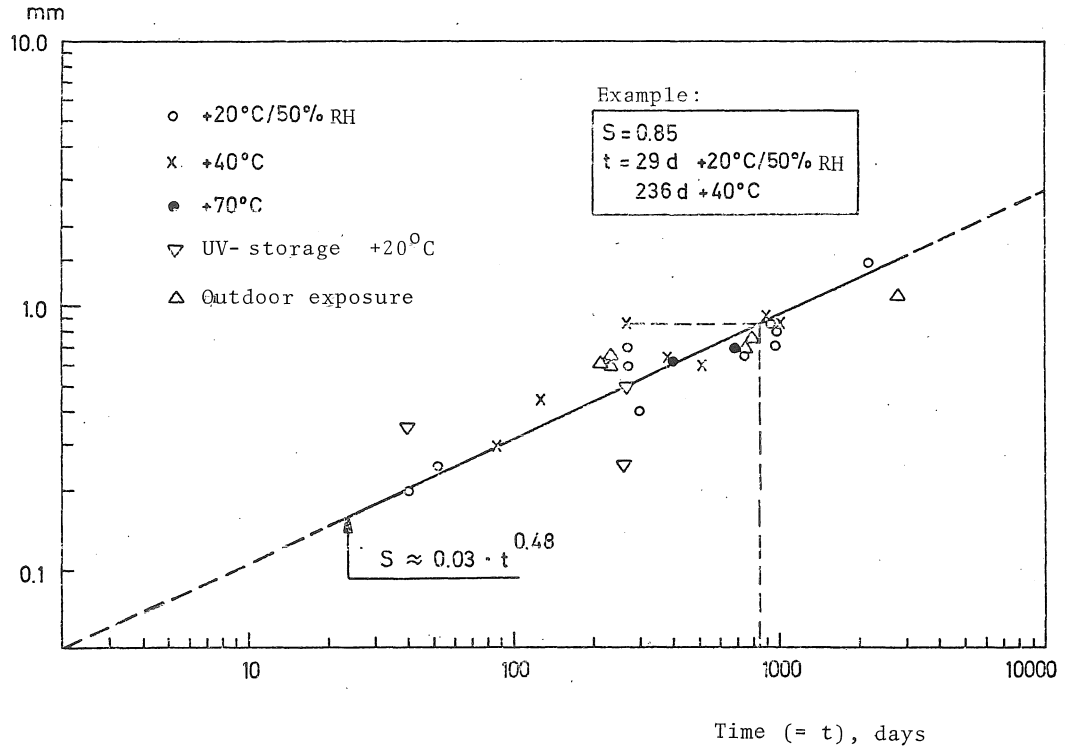


FIG 3 Skin thickness as a function of storage time at +20°C/50% RH for "Material No 8". Computing of equivalent storing time at +20°C/50% RH for material stored at +40°C and +70°C.

## 1. BACKGROUND

The durability problems of building materials in general are very complex and sealants are certainly no exception.

In spite of the fact that durability problems have existed as long as there have been sealants, this problem has been treated very sparsely in literature. Not until recently have some reports on this subject been published by Lerchenthal (1972), Künzel (1975), and Grunau (1976), for example.

The majority of sealants are used between external wall panels. For example, between prefabricated concrete panels, between window-frames and walls, in expansion joints, when glazing etc. This means that sealants are exposed to very hard climatic conditions. The efficiency of the single stage joint (which is very common in Sweden) is completely dependent on the properties of the sealant, its ability to follow the joint movement without any permanent deformation and its ability to maintain this property also under the influence of the ambient environment. Because of the organic design of the sealant some changes in the material can be observed after a time; the material ages. To what extent greatly depends on the ambient environment but the type of sealant also plays a certain role, as some polymers are more resistant than others. However, at present knowledge regarding the ageing factors of sealants is very limited: "In view of the limited knowledge regarding ageing factors of sealants, one ought to try to facilitate a future re-sealing already during the projecting stage" (Hus AMA 72).

## 2. AIM

The aim of this project has not been to answer all the questions that might arise concerning the durability and ageing factors of sealants. The problems are far too

extensive and complex for that.

The aim has been to try to produce a survey of how certain isolated ageing factors affect the deformation properties in different types of sealants. The aim has also been to combine some of these factors in order to find out whether or not there exists any possible combined effect. In publishing this survey the intention has naturally been to compare these results with practical experience.

Hopefully the results of the investigation will contribute towards

- making the choice of material safer
- calculating the durability
- creating reliable test methods
- facilitating formulation of the products

### 3. AGEING FACTORS

The climatic factors that first and foremost affect the sealants are temperature, humidity, UV light, and ozone.

Generally speaking one can talk about environmental factors. Sometimes, the alkali factor is added, for example when sealing a concrete panel or plaster. Mechanical ageing should also be mentioned here. That is the effect that repeated elongating and compressing joint movements have on the sealant.

In addition another environmental factor which could play a greater role in the future in this connection is the increasing atmospheric pollution.

#### 3.1 Temperature

Low temperatures usually only affect polymeric materials by making them brittle, to a greater or lesser extent. The material regains its properties when the temperature

becomes normal again. However, the increasing brittleness at low temperatures could be fatal if a material in this condition is exposed to great deformation. For example where sealants are concerned the demands on elasticity are at their greatest at low temperatures as that is when joint is at its widest.

In principle heat can influence sealants in two different ways:

1. When the temperature rises the rate of the chemical processes increases. Roughly, one can calculate with double the reaction rate when the temperature rises  $10^{\circ}\text{C}$ . Where sealants are concerned these processes can be, for example, the breaking of bindings and molecular chains, changes in the molecular chains and the creating of cross-links between the molecular chains.
2. Several sealants contain softeners, which are used to modify the hardness of the material. Certain types of softeners vaporize, at an increase in temperature. This results in the softener migrating from the sealant. Some sealants also contain solvents, which facilitate the application. Naturally, the ambient temperature determines how quickly the solvent disappears.

The most important effects of heat influence are primarily that the material becomes soft and secondly that it becomes harder and more brittle, the elongation at rupture decreases.

### 3.2 Humidity

Humidity or water induce certain polymer materials to hydrolysis, which could be defined as a chemical reaction, where a molecule or an ion is transformed into two or more molecules or ions by reacting to water.

A lengthy period of humidity can also lead to a leaching of certain components from the polymeric material. Water can also bring about a swelling of the material, which means that other forms of attack can take place.

Furthermore, if the water, as is often the case with concrete structures, contains  $\text{Ca(OH)}_2$ , serious attacks can take place in the form of saponification of the material, where some sealants are concerned.

### 3.3 Ultraviolet radiation

It has been known for a long time that the short-wave part of the solar spectrum (UV and X-ray radiation) creates photo-chemical reactions in organic materials. Fig 1 shows the energy per photon in sunlight as a function of the wavelength. The dissociation energy per binding for some of the most frequently used bindings in organic materials is also shown in the Fig. It can be seen that it is a chain of binding types which can be broken by the UV light.

In polymeric materials, the sunlight causes oxidation in the surface. A "skin" with very decreased elongation at rupture is then formed. After a while this "skin" cracks to form an irregular pattern, so called cracking.

### 3.4 Ozone

Normally, ozone occurs in very small concentrations in the atmosphere, but sufficiently high to cause attacks sometimes.

The formation of ozone takes place in the upper atmosphere by means of photochemical reactions with oxygen.

Oxygen ( $\text{O}_2$ ) is transmitted to the ozone ( $\text{O}_3$ ) because of the UV radiation from the sun. The ozone content is given in pphm = parts of ozone per hundred million parts of air. In Sweden the ozone content is normally 2-3 pphm. The average ozone concentration is usually at its lowest



in the winter, increases in the spring and reaches its maximum in the early summer only to decrease again.

Ozone is a very reactive gas and is therefore quickly decomposed to oxygen when it comes into contact with organic materials. When this happens the double bond in the unsaturated types of rubber is attacked and decomposed. These reactions happen on the surface of the rubber where a characteristic crack pattern occurs. This cracking only occurs if the rubber is exposed to elongation.

As a rule the greater the elongation the more serious the attack. The ozone attack takes place in daylight as well as in darkness.

### 3.5 Combined influence

Ageing is often a synergistic effect, which means that the effect of the ageing factors increases if the factors coincide. This naturally complicates the estimation of the life of different materials.

It is obvious that heat plays a great role together with other ageing factors. This is also the case where humidity is concerned.

Naturally the repeated deformation that a sealant is exposed to, together with other factors, also plays a considerable part in breaking down a sealant.

## 4. PLANNING OF THE EXPERIMENTS

### 4.1 Choice of sealant

When choosing between different types of sealants that were to be included in the investigation, the purpose was to get as many types as possible represented, of those that were used frequently when the project started. It was also intended that materials which were compara-

tively unknown on the market at the start of the project, but were expected to become increasingly used, should be included. For example, two different types of polyurethane based sealants and one water dispersed acrylic sealant have been included in the investigation.

Furthermore, the purpose has also been to make comparisons possible between sealants of the same type (approximately the same polymer) but of different formulations.

The final choice of materials has been made together with AG 3 in the Swedish Plastics Federation, where most of the largest Swedish manufacturers of sealants are represented.

The sealants used in the investigation are presented in Table 1. The classification of the sealants is based on the polymer.

Table 1. Different types of sealants in the investigation.

Material No	Kind of binder	Number of components	Note
1	Polysulfide	2	Hardness $\sim 13^{\circ}$ Shore A
2	Polysulfide	2	Hardness $\sim 40^{\circ}$ Shore A
3	Polyurethane	1	Hardness $\sim 25^{\circ}$ Shore A
4	$\sim$ Polyurethane	2	Hardness $\sim 18^{\circ}$ Shore A
5	Acrylic	1	Solvent release
6	Acrylic	1	Solvent release
7	Acrylic	1	Water dispersed Hardness $\sim 15^{\circ}$ Shore A
8	Oleo-resinous	1	

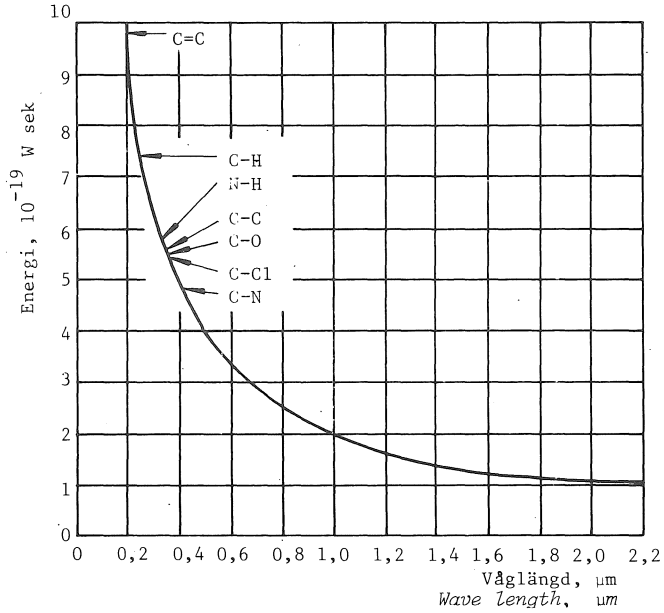


FIG 1 Energi per foton och dissociationsenergi per bindning Gjelsvik (1975)

*Energy per photon and dissociation energy per binding.*

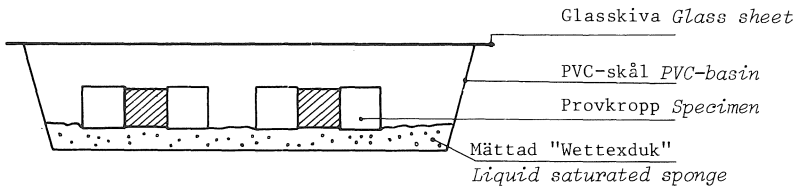


FIG 2 Fukt- respektive alkalilagring av provkroppar.

*Storeing of specimen in water and alkaline water*

Eight different materials of five different makes have been examined. The values of the Shore A hardness in the Table have been taken from information leaflets issued by the manufacturer.

#### 4.2 Choice of influencing factors

The ageing factors chosen for the investigation are temperature, humidity, alkali, UV radiation, and ozone.

The effect of the temperature on the sealants has been studied by storing specimens in the standard climate +20°C/50 % RH and afterwards at +40°C or +70°C.

The effect of humidity has been studied by storing the specimens on a sponge saturated with de-ionized water (pH~4.0) (see Fig 2). The storage temperature was +20°C. This procedure could, for example, be similar to when a back-up material for some reason or other becomes saturated by water.

The alkaline influence has been studied by storing the specimens on "sponges" saturated with a  $\text{Ca(OH)}_2$  solution, pH~11.0, at +20°C. This storage can create a similar situation to that which can arise when an absorbent back-up material between concrete elements becomes saturated with water.

When observing the effect of UV light the purpose was to examine only the effect of UV radiation on the sealants. A problem arose as it appeared that most of the available UV sources have a large part of their energy outside the UV range. This means that the lamps produce a great amount of heat. The type of lamp used was OSRAM HNS 15W, without ozone.

Six of these light sources were placed in a cylinder where specimens could be fixed on the inner wall. (See Fig 3).

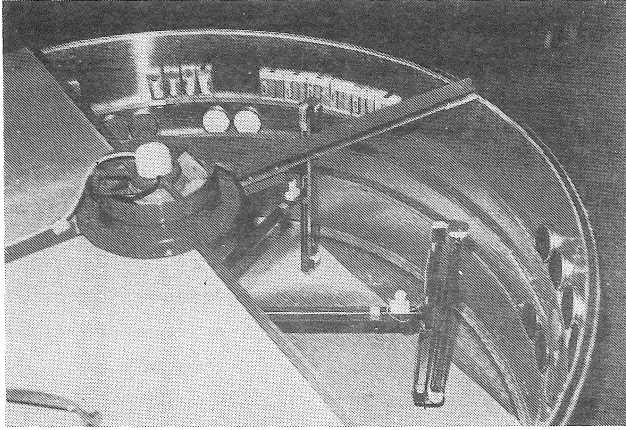


FIG 3 UV-klimatkammare  
*UV climate chamber*

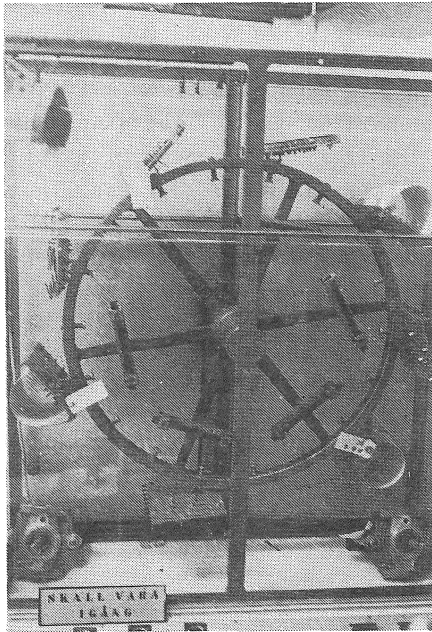


FIG 4 Ozonskåp, modell Trelleborg  
*Ozon climate chamber, made in Trelleborg*

The temperature in the cylinder was maintained constantly at about  $+20^{\circ}\text{C}$ . The purpose was also to combine the UV radiation with heat ( $+40^{\circ}\text{C}$ ), but due to lack of time this has not yet taken place.

As different types of sealants can be expected to have a different absorption spectrum in the UV range, the aim has also been to examine the effect of another type of UV source. There has nor been time for that either.

By the courtesy of the Trelleborg Rubber Factory Limited (engineers Allan Rydsbo and Arthur Jonasson) it has been possible to study the effect of ozone on sealants. Specimens have been placed in ozone chambers. The chambers were developed at the Trelleborg Rubber Factory. These chambers have now been standardized. The design of the ozone chamber is shown in Fig 4. The concentration of ozone can be varied from a few pphm to 400 pphm. The chambers are normally operated at 50 and 200 pphm.

The effect of ozone has been studied on all types of sealants. It has been pointed out in literature that ozone cracking only occurs if a rubber material is elongated, that is, it has an internal tensile stress. Therefore, only the sealants with a predominant elastic portion have been elongated when exposed to ozone. The temperature was  $+30^{\circ}\text{C}$ .

#### 4.3 Choosing methods of quantification

It is difficult to choose a realistic method for quantifying changes in the sealants, especially as demands on these methods are known to be hard. For example a good quantifying method should be

- easy to carry out
- sensitive
- preferably non-destructive
- adaptable to all types of materials.

As yet the method which can fulfill all these demands does not exist. After studying literature certain methods have been chosen.

The determination of the shape of the stress-strain curve according to different forms of ageing is used in many connections. In this case the method used was where the specimens were elongated at a rate of 1.5 mm/min and at a temperature of  $+2^{\circ}\text{C}$ . As the eight different materials included in this investigation have very different properties concerning deformation resistance the temperature  $+2^{\circ}\text{C}$  was chosen. At this temperature the elongation resistance of the plastic materials increases, especially after certain forms of ageing.

When quantified, all the specimens were extended 100%, which is less than elongation at rupture for the majority of the materials. There are several reasons why deformation only up to 100% was chosen:

- the length of the specimen has a greater effect on the test result the greater the elongation (see chapter 4.4)
- generally, the adhesive stresses increase with increased elongation. As the sealant is primarily to be examined this can mean that adhesive failure may occur and interfere with the comparisons
- 100% elongation is very much on the safe side compared to the deformation the materials are usually dimensioned for.

When 100% elongation was reached the stress relaxation was registered for about 10 minutes. One can suppose that a material mainly plastic can have stress-strain curves up to 100% which are similar to those of an "elastic" material. The determination of the time dependence of the stress relaxation shows the difference between an "elastic" and a "plastic" sealant.

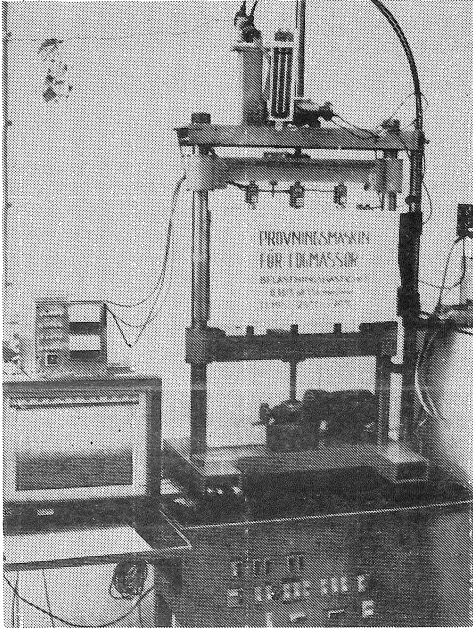


FIG 5a Provningsapparat för fogmassor. Klimatkammaren stängd. Deformationshastighet 0.001 - 1.5 mm/min, drag/tryck. Provnings temperatur  $-25^{\circ}\text{C}$  -  $+70^{\circ}\text{C}$ .

*Testing equipment for sealants. Climate chamber closed. Deformation speed 0.001 - 1.5 mm/min, tension/compression. Testing temperature  $-25^{\circ}\text{C}$  -  $+70^{\circ}\text{C}$ .*

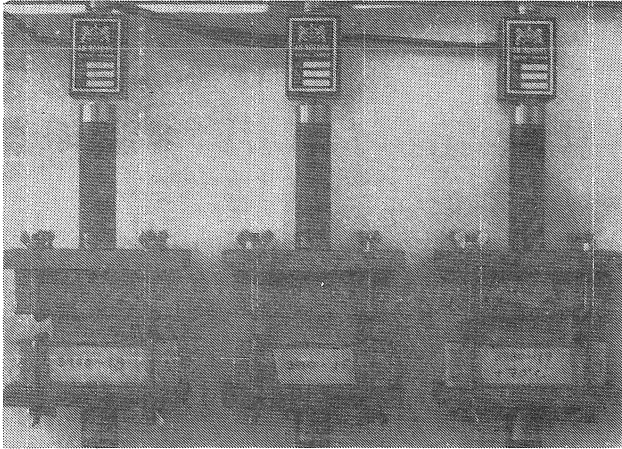


FIG 5b Provningsapparat för fogmassor. Inspänning av provkroppar och celler för kraftmätning.

*Testing equipment for sealants. Fixing of specimens and force gauges.*



The appearance and shape of the stress-strain curve supplies information regarding softening or hardening, brittleness, increased temperature dependence etc. The test equipment used for this purpose is shown in Fig 5.

The determination of the loss in volume and weight supplies information regarding the amount of volatile components, the thermo-stability of the softener, and the changes in density. These determinations have been made approximately according to the method NM Bygg F10 "Determination of free shrinkage" (Report No 1:71 from the ER-committee). The methodology is shown in Fig. 6.

One can only obtain a rough approximation of the changes by photographing the specimens before and after ageing, but even so this method has been used to some extent.

SEM photography has been used for a few experiments. These investigations were carried out at the Zoological institution at Lund University.

When choosing appropriate UV sources for studying the effect of UV radiation on sealants the question arose regarding the absorption of different materials within the UV range. As different types of material absorb radiation on different wavelengths it is possible that an incorrectly chosen UV source hits some type of material unnecessarily hard or, on the other hand, favours some UV-sensitive materials. This led us to examine the UV absorption spectrum of the sealants. This spectrum will probably change when the material ages. This should mean that the method could also be used as a method of quantification. Certain introductory research in this direction has therefore been carried out. One method which was also tried and showed very promising results at first was DTA analysis. These investigations, which were carried out together with the Rheology Group at The Centre of Chemistry, Lund Institute of Technology,

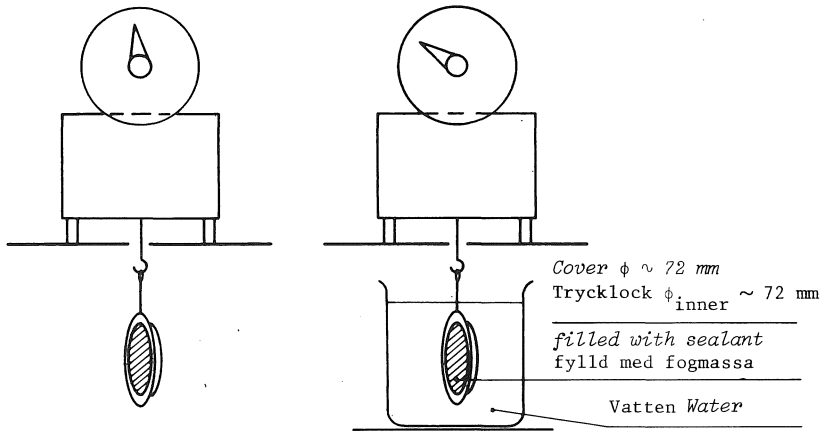


FIG 6 Metodik för bestämning av viktninskning och krympning.  
*Measuring of weight loss and shrinkage.*

turned out to be impossible to reproduce.

In spite of this, Künzel et al (1975) report successful results when using DTA analysis for the determination of the glass transition temperature of sealants. They even go as far as stating that "the displacement of the glass temperature should be measured in all ageing experiments, for example by means of DTA"!

#### 4.4 Choosing the shape of specimens

For example, when examining the elongation resistance of sealants, discussions regarding the appropriate shape of the specimens often occur. Where the shape of the cross section is concerned the width/depth relationship must be as large as possible, in order to minimize the tensile stresses that occur and thereby also the adhesion stresses. Because of other reasons this relationship cannot be unlimited.

Where the length of the joint specimen is concerned, great strains on a short joint will result in stresses, which are too small compared with what they are exposed to in buildings.

An estimation of the extent to which the specimen size affects occurring tensions in different materials has been presented in a more concise pilot study. Three types of material were included in this study:

1. One-component silicone sealant, 25-30<sup>0</sup> Shore A.
2. Two-component polysulphide sealant, approx 15<sup>0</sup> Shore A.
3. One-component acrylic sealant, solvent release.

The cross section was nominally 12 x 8 and 12 x 12 mm (width x depth). The length was 50, 80 or 150 mm.

Before starting the experiments, the real dimensions were measured. The specimens were elongated at +20°C with a deformation rate of 1.5 mm/min to approx 50 % elongation. Figs 7-9 show the stress-strain curves obtained. For the silicone sealant, which can be characterized as elastic, the relative positions of the different specimens correspond well to the positions expected, see Fig 7. For example, in a case of 25 % deformation a specimen with a length of 50 mm has about 7 % less tension than one with a length of 150 mm. The difference will be somewhat larger at 50 % elongation.

Where the more plastic polysulphide sealant (Fig 8) is concerned the distribution of the results has produced a somewhat unexpected order in the curves. However the total dispersion is very small. In this case the variations in the specimen shape can be considered to have a negligible effect.

The same relation is valid for the acrylic material according to Fig 9. The distribution of the results dominates over the effect of the specimen shape. Consequently the curve which was expected to be the highest was the lowest.

Summing up one could say that the variation in specimen shape is of no significance for the measured stresses at deformations below approx 50 % and for materials of a certain plasticity.

For these reasons, the specimen shape 12 x 12 x 50 mm has been chosen for the continued experiments. These dimensions correspond well to the American, English and German standards.

Asbestos cement, alternatively aluminium (grade SIS 41 04-6), have been used as jointing materials. The specimen shape is shown in Fig 10.

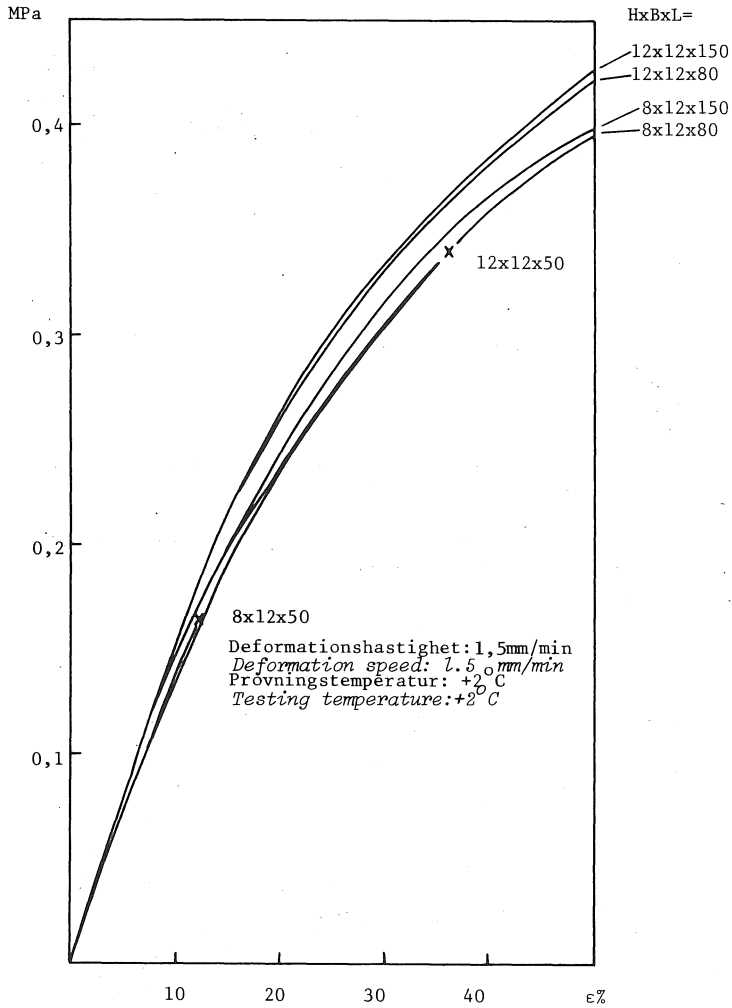
Dragspänning *Tensile stress*

FIG 7 Arbetskurvas beroende av provkroppsdimensionen för en silikonbaserad fogmassa.

*The influence of the specimen dimension on the stress-strain curve for a silicon based sealant.*

Dragspänning Tensile stress  
MPa

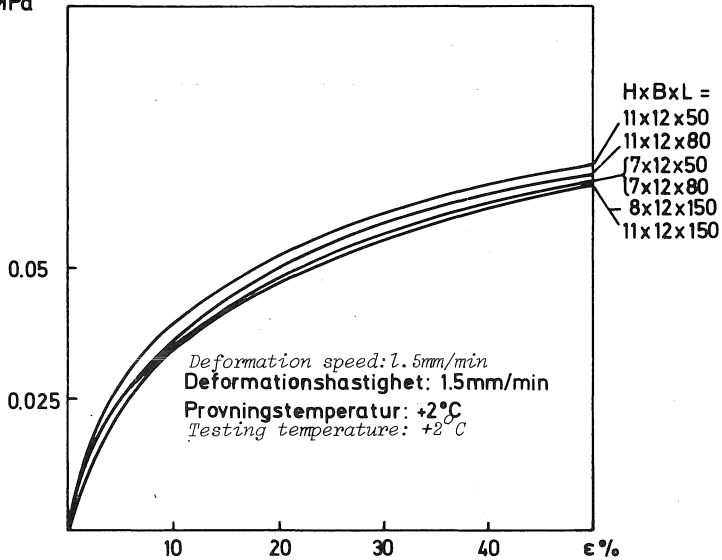


FIG 8 Arbetskurvas beroende av provkroppsdimensionen för en polysulfidbaserad fogmassa.

*The influence of the specimen dimension on the stress-strain curve for a polysulphide based sealant.*

Dragspänning Tensile stress

MPa

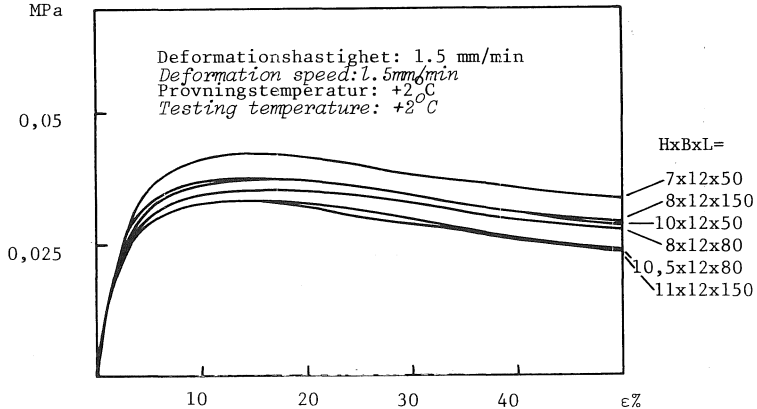


FIG 9 Arbetskurvas beroende av provkroppsdimensionen för en akrylatbaserad fogmassa.

*The influence of the specimen dimension on the stress-strain curve for an acrylic based sealant.*

#### 4.5 Outdoor experiments

The purpose was to compare the results obtained from laboratory experiments with ageing results closer to reality. For example, this can be done by comparing laboratory tests with tests on materials used in building constructions. However problems arise, for example, in the quantifying of changes and the formulation of materials might also have changed during the time of the tests. This comparison is both interesting and important and must not be neglected, however.

We have also built a construction where the specimen joints were exposed to varying joint movements and natural climatic influence.

##### 4.5.1 Description of equipment for outdoor exposure of joint specimens

A survey is presented in Fig 11. The equipment consists of an aerated concrete element (250 x 900 x 6000 mm) placed on one fixed and one rolling bearing. Two aluminium tubes, connected with "rungs" of aluminium, are placed on this element. This "ladder" is fixed at one end to the aerated concrete and at the other to a moving rolling bearing.

An L-profile is fixed to the aerated concrete and to each rung. A number of holes are drilled in the L-profiles. Specimens can be fixed between these coupled L-profiles with the asbestos cement (alternatively aluminium) prism fixed to the aerated concrete (via the L-profile) and the other prism is fixed to the rung. The attachment part is shown in Fig 12.

This means that the width of each fixed specimen will change according to the changes in the humidity and temperature of the aerated concrete and the temperature of the aluminium ladder.

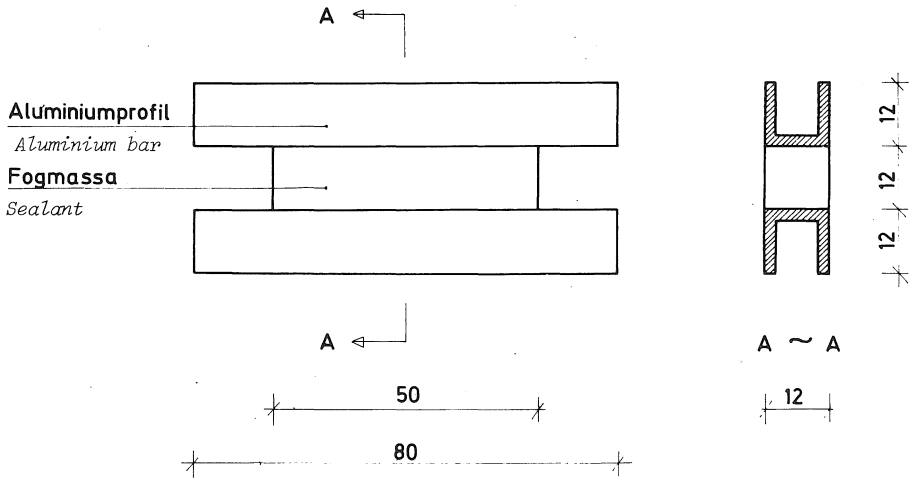


FIG 10 Provkropparnas utformning.  
*The specimens dimensions.*

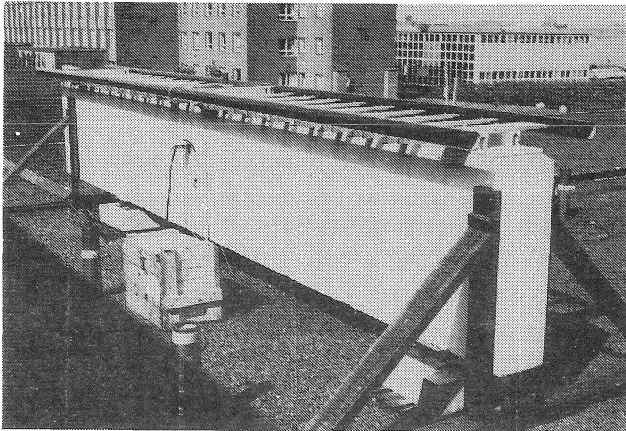


FIG 11 Utrustning och konstruktion för utomhusexponering  
 av provkroppar  
*Testing equipment for outdoor exposition of specimens.*



To emphasize the differences in the dimension changes in aerated concrete and in aluminium still further, the aerated concrete was painted white while the aluminium tubes were painted black.

Assuming that

$\alpha$  aerated concrete is approx  $8 \cdot 10^{-6}$  m/m<sup>0</sup>C,

$\alpha$  aluminium is approx  $24 \cdot 10^{-6}$  m/m<sup>0</sup>C and

the variation in temperature is within the range -20 - +50<sup>0</sup>C, the maximum difference in length would be around 7.2 mm. If the effect of the humidity variation is considered, the difference would theoretically be about 7.35 mm. This change in dimension will decrease linearly towards zero (where the aluminium ladder is fixed to the aerated concrete).

Thermo couples were placed on the construction, which is located in a north-south direction. Inside the aerated concrete there are also three gauges for registering the relative humidity. An inductive deformation gauge was also placed on the aerated concrete in order to register the maximum difference in movement. The number of hours of sunshine were registered by a so called photoelement. All measured values were registered on a 12-channel point recorder.

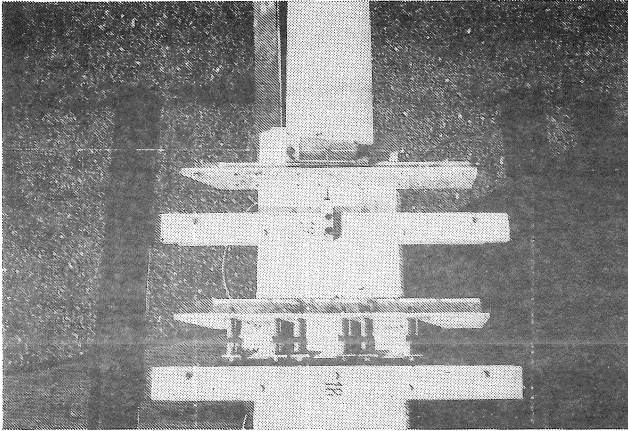


FIG 12 Utomhusexponering av provkroppar. Detalj av provkroppsfästning och lägesgivare.

*Outdoor exposition of specimens. Detail of specimen fixation and deformation gauge.*

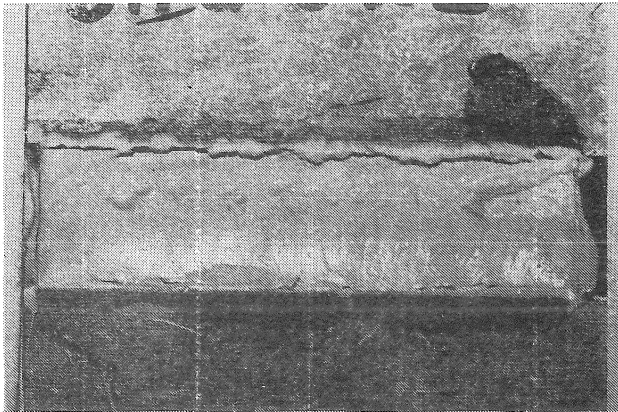


FIG 13 Yta av värmeåldrad lösningsmedelsbaserad akrylatfogmassa. Sprick- och blåsbildning p g a snabb bortgång av lösningsmedel.

*Specimen surface of a heataged acrylic based (solvent release) sealant. Forming of cracks and bubbles due to a rapid release of solvents.*

## 5. TEST PLAN

### 5.1 Temperature influence

+20°C/50% RH was used as a standard climate for the so called zero specimens. Otherwise the normal standard climate for sealants is +23°C/50% RH but, as there was such a large number of specimens, the space available at +23°C/50% RH was insufficient.

Storage at an increased temperature, +40°C and +70°C, started after the specimens had been stored at +20°C/50% RH for 7 or 21 days. Some undesirable effects can occur if the hot storage is started too early. This is shown in Figs 13-14, which show a solvent based acrylic sealant. In this case hot storage at +70°C was started after 7 days. The remaining solvent has evaporated from the material with blisters as a result. Consequently the rapid shrinkage also caused adhesion cracking on large surfaces. These after effects need not necessarily occur in sealants during natural ageing.

The testing plan for temperature influence on the whole is shown in Table 2.

For some materials the hot storage time has been longer than the times stated in the Table.

Table 2. Temperature influence, experimental plan

Climate	Time (days)		
	+20°C/50% RH	21 35	35 49
+40°C	7 + 14	7 + 28	7 + 56
	21 + 14	21 + 28	21 + 56
+70°C	7 + 14	7 + 28	7 + 56
	21 + 14	21 + 28	21 + 56

Note: The time 7 + 14 means that the specimen has been stored 7 days in +20°C/50% RH and after that 14 days in +40°C (+70°C) and so on.

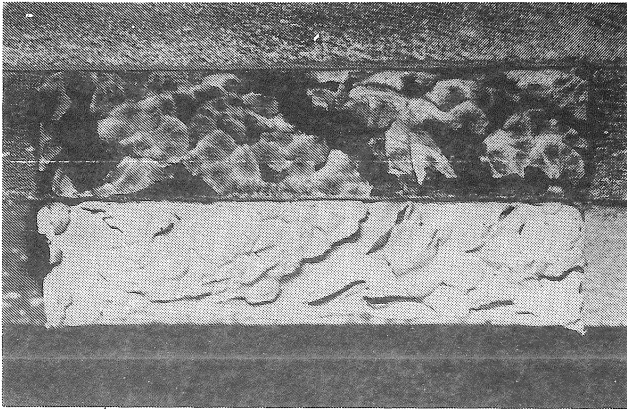


FIG 14 Brottyta av värmeåldrad lösningsmedelsbaserad akrylatfogmassa enligt fig 13.

*Fracture surface of a heataged acrylic based (solvent release) sealant according to fig 13.*

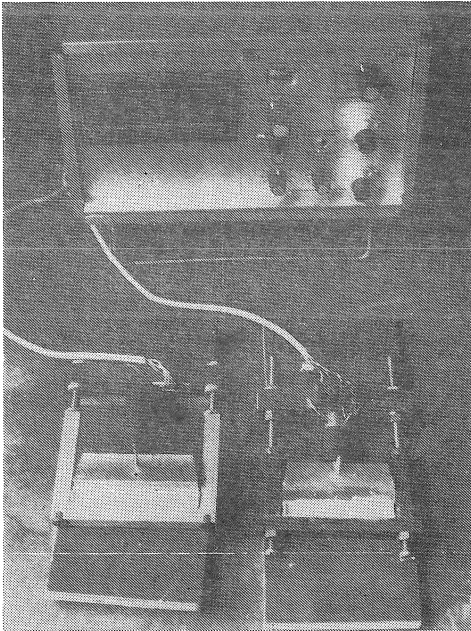


FIG 15 Utrustning för mätning av krympspänningar i fogmassor.

*Device for measuring of shrinkage stresses for sealants.*

## 5.2 Humidity and alkali influence

The influence of humidity and alkali was studied by storing the specimens on a sponge saturated with different media (see Fig 2). In this case "humidity" means de-ionized water with pH approx 4. The alkali solution used was a saturated  $\text{Ca(OH)}_2$  solution diluted to pH approx 11.0. This level can be considered to be the same as the alkalinity when water is poured onto a concrete surface.

Storage took place at  $+20^\circ\text{C}$ . Before storage the materials were initially hardened for 7 or 21 days at  $+20^\circ\text{C}/50\% \text{RH}$ . Testing was carried out after 14, 28 and 56 days' exposure.

## 5.3 Influence of UV radiation

Storage in UV light was started after 7 days initial hardening at  $+20^\circ\text{C}/50\% \text{RH}$ . Testing was then carried out after 14, 28 and 56 days in UV light. Some materials were also stored for a longer period of time.

## 5.4 Ozone influence

In order that rubber materials are to be affected by ozone, tensile stresses must exist in the material. This means that the specimens must be stored elongated. Some of the chosen sealants were regarded as being much too plastic for the elongation to be of any value. For this reason, sealants were divided into two groups, according to Table 1.

Group I, mainly comprising elastic materials, consists of materials Nos. 1-4 and material No 7.

Group II, mainly comprising plastic materials, consequently comprise materials Nos. 5-6 and material No 8.

In spite of group II comprising materials without tensile stresses it was considered to be of interest to study if any other effects of ozone storage might occur.

Other testing conditions had to be adapted to the standards valid for this range. These included e.g. ozone content, time and storage temperature (+30°C).

The ozone tests started on a Monday morning. For practical reasons, the specimens were transported to Trelleborg on Friday afternoon. The specimens that were to be kept under tension had previously been exposed to 25% or 50% elongation. Where these specimens were concerned it meant that the stress built up in the material had time to relax to a certain extent before the ozone storage began. In order to estimate the amount of relaxation, this was measured on the specimens exposed to elongation to 25% and 50%.

The test plan for the effect of ozone is shown in Table 3.

Table 3. Ozone resistance, experimental plan

Variables	Levels	Note
Ozon content	50 pphm 200 pphm	
Age	35 days	Half the number of specimens were heataged 14 days in +70°C
Time	1 x 96 h	
Deformation	0% 25% 50%	Sealants in group No. I and II Sealants in group No. I Sealants in group No. I

### 5.5 Outdoor experiments

At least 8 specimens of each material were exposed to outdoor tests and simultaneous deformation. Each material (regardless of the "hardening mechanism") was kept for 7 days at +20°C/50% RH before being installed. This meant that the hardening of the two-component materials was quite advanced. This was also the case for material No. 3, a one-component polyurethane sealant, and material No. 7, a one-component dispersed acrylic sealant, even if these were far from being fully cured.

Materials Nos. 5-6 and 8 were extremely plastic when placed outdoors.

Half the amount of the specimens had asbestos cement as a substrate and the other half aluminium. This was done in order to create both a certain alkali influence and a climatic influence. The water poured over the asbestos cement had a pH value of about 9-10.

When choosing the position on the aluminium ladder, the basis has been the movement absorbing capability stated by the manufacturers. This result in material No. 4 being exposed to the largest variation in joint width. The materials, from the largest to the smallest joint movement, were: Materials Nos. 6, 3, 8, 1, 2, 7, and 5.

Specimens of each material were also installed where they were exposed to about half the maximum joint movement for the respective material.

Specimens were mainly installed at +5 - +10°C. This meant, that with a supposed maximum variation in temperature of -20°C - +50°C the specimens were to somewhat greater extent more compressed than elongated.

### 5.6 Measuring shrinkage stresses

The shrinkage which occurs to a varying extent in sealants when they age and sometimes when they harden/dry, could be expected to result in certain consequences.

If shrinkage occurs because of, for example, the softener disappearing, this will naturally affect the hardness in a negative manner. But, if shrinkage occurs because solvents (used in the material in order to facilitate the application) disappear, this could be expected to have a positive effect on the deformation absorbing properties. The joint profile then receives an increase in the bi-concave profile which is favourable from a deformation point of view.

If this shrinkage should be followed by large shrinkage stresses, these would, in addition to the tensile stresses occurring when the joint width is increasing, lead to adhesive or cohesive failure. Some tests have been carried out in order to obtain an idea of how large these shrinkage stresses are. The sealants most prone to shrinkage, and at the same time also the mainly "elastic" materials, were chosen to be included in a test regarding the measuring of shrinkage stresses. The choice was based on the determination of the decrease in weight and volume.

The materials tested on these grounds were materials Nos. 1, 3 and 7. Material No. 8 was also examined, in spite of it not usually being regarded as elastic.

Two different methods were used. To begin with, the equipment shown in Fig. 5 was used. With this equipment it is possible to measure forces down to approx.  $6 \cdot 10^{-2}$  N. The specimens were fixed, completely newly applied to the equipment. The ends of the specimens were supplied with millimeter thick cellular plastic of polyethylene and at the bottom there was silicone treated paper. The temperature during the test period



varied between  $+21^{\circ}$ -  $+23^{\circ}$ C. In order not to occupy the equipment for too long a time two more test instruments were made where the sealants could be fixed directly. See Fig. 15. The active measuring device consisted of two strain gauges.

## 6. TEST RESULTS

### 6.1 General

To begin with some observations are made regarding the studied materials during the test period. This may explain certain phenomena which occur later on.

Sometimes, it has been difficult for certain materials to attain adequate adhesion to the surfaces of the substrate. This has been the case in spite of the instructions from the manufacturers having been followed carefully. Adhesive failure was frequent, e.g. when examining the ozone influence on the elongated material. Some specimens were kept under 25 or 50% elongation for one week. Where materials Nos. 1, 2 and 7 were concerned, adhesive failure occurred in several specimens already after a short time. See Fig. 16. This is the reason why some of these materials sometimes are not included in the comparisons. The specimens kept elongated during the ozone storage, and which were heat aged in advance, usually had better adhesion.

The two solvent based sealants, materials Nos. 5 and 6, are very sensitive to too early a heat ageing. See Chapter 5.1. Even if heat ageing is started after three weeks' storage at  $+20^{\circ}$ C/50% RH, the rapid disappearance of the remaining solvent resulted in bubbles and adhesive failure in the joint profile. This has also upset the comparisons to some extent.

Material No. 7, the water dispersed acrylic sealant, has caused some problems when preparing the specimens. This material requires a different jointing technique,

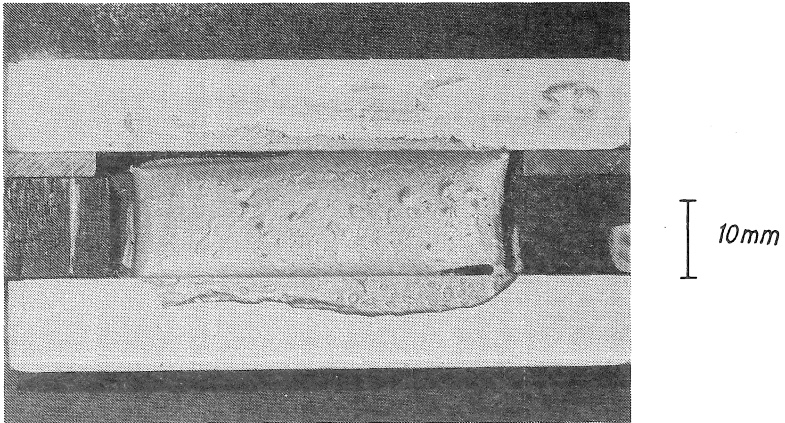


FIG 16 Adhensionsbrott vid ozonprovning av "Material 2", 25% töjning.

*Failure of adhesion at testing of ozone resistance for "Material No 2", extended to 25%.*

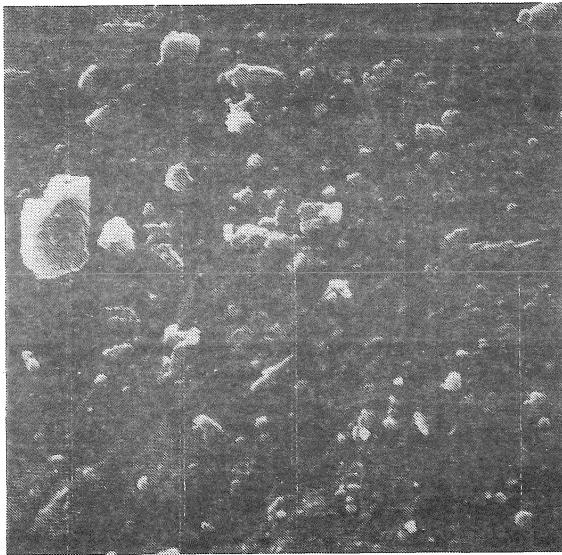


FIG 17 SEM-bild av ytan av en polysulfidbaserad fogmassa lagrad i +20°C/50% RF. Förstoring: 600x. Foto: Zoologiska institutionen, Lunds universitet.

*SEM-picture of the surface of a polysulphide based sealant stored at +20°C/50% RH. Enlargement: 600x. Photo: The Zoological institution of Lund University.*

compared to that used for other sealants. In spite of building up the joint cross section in different layers it was sometimes difficult to fill the entire joint space. The same problem seems to arise on site.

There are great differences between the eight different sealants included in this investigation, for example, hardening time, hardening mechanism, functioning, deformation characteristics etc. These have caused certain problems: For example, will the same accelerating ageing methods be valid for all types of materials? Can one characteristic for one type of sealant justify a "tailor made" method for just this material?

## 6.2 Temperature influence

For several reasons a relatively thorough study has been carried out concerning temperature influence on the ageing and deformation properties of the sealants: storing in heat, frequently at  $+70^{\circ}\text{C}$ , is used quite often in laboratories in order to accelerate the ageing of the sealants. This method is also very simple to carry out. Literature provides information regarding the length of time accelerating ageing takes compared with natural ageing.

Hockenberger and Soyka (1969) state that, for example "heat ageing for 7 days at  $+100^{\circ}\text{C}$  corresponds to outdoor ageing for about 1 1/2 years". According to other information "14 days storage in air at  $+70^{\circ}\text{C}$  normally causes effects similar to one year of natural ageing".

Some SEM photographs have been taken of a polysulphide sealant after different heat ageing. Fig. 17 shows the sealant surface after storage at  $+20^{\circ}\text{C}/50\% \text{RH}$ . Fig. 18 shows the same material heat aged at  $+70^{\circ}\text{C}$  for 21 days. The effect is obvious.

Three of the materials have been studied more thoroughly than others. These three materials are Nos. 1, 3 and 6. From a hardening point of view these materials represent

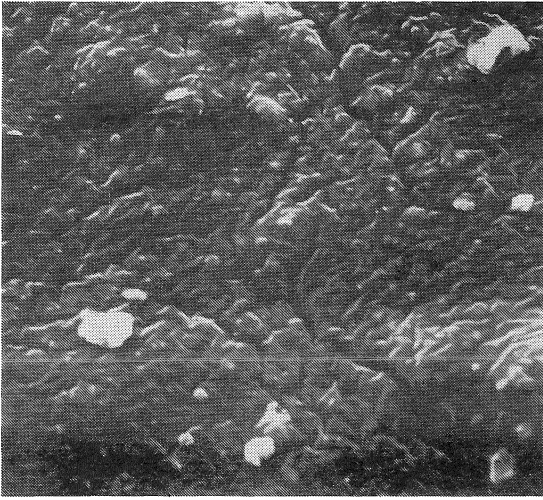


FIG 18 SEM-bild av ytan av en värmeåldrad (21 dygn i +70°C) polysulfidbaserad fogmassa. Förstoring: 600x. Foto: Zoologiska institutionen, Lunds universitet.

*SEM-picture of the surface of a heataged (21 days at +70°C) polysulphide based sealant. Enlargement: 600x. Photo: The Zoological institution of Lund University.*

Dragspänning, Tensile stress  
MPa

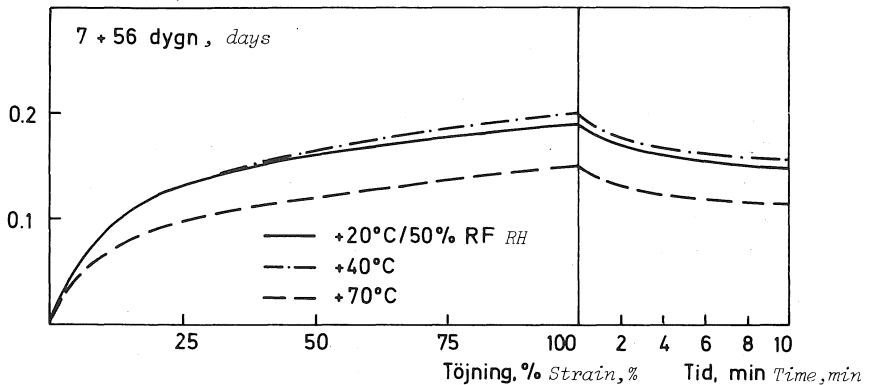


FIG 19 Arbetskurvor för "Material 1" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provnings temperatur: +2°C.

*Stress-strain curves for "Material No 1" heataged in different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

three different types of sealants: 2-component chemically curing, 1-component chemically curing and 1-component chemically curing /drying sealant respectively.

#### 6.2.1 Material No. 1

Figs. 19-22 summarize the results concerning the temperature influence on material No. 1, a 2-component polysulphide sealant. According to the manufacturer the hardness is approx 13° Shore A.

Fig. 19 shows the stress-strain curve for the material that has been cured/aged at different temperatures. All the sealant specimens were first cured for 7 days at +20°C/50% RH. Afterwards certain specimens were heat aged at +40°C or at +70°C. The ageing at +40°C hardly affected the material at all in comparison to the standard climate 20/50. On the other hand, heat ageing at +70°C made the sealant softer! This is very surprising. So far, from experience we know that polysulphide sealants generally get harder when heat aged. By means of heat ageing at 70°C it has also, as was previously mentioned, been possible to imitate this increased hardening quite successfully.

The possibilities of varying the composition of polysulphide sealants are many, and where this material is concerned the manufacturer has apparently totally succeeded in eliminating this increase in hardening.

Corresponding tests, shown in Fig. 19, have been carried out after varying times of heat ageing. Fig. 20 summarizes the results from these tests.  $\sigma_{50}$  is the stress that occurs in the specimen at 50% elongation. Tendencies from the former Fig are also valid here.

For the chiefly elastic materials the determination of the hardness was calculated by using a Durometer

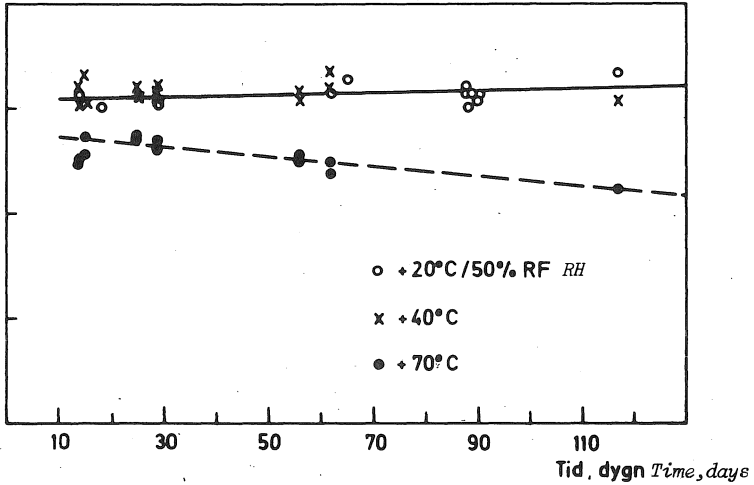
$\sigma_{50}$   
MPa


FIG 20 Dragspänning vid 50% töjning ( $=\sigma_{50}$ ) för "Material 1" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RH.

*Stress at 50% tension ( $=\sigma_{50}$ ) for "Material No 1" as a function of storing time at different temperatures. Initial storing: 7 days at +20°C/50% RH.*

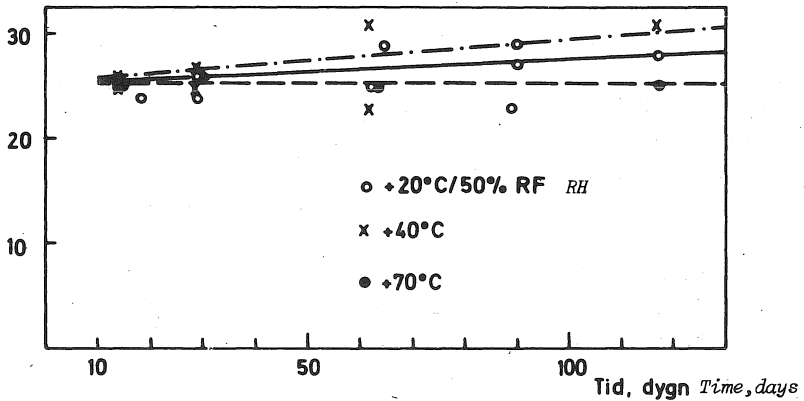
 $^{\circ}$ Shore A


FIG 21 Härdhet i  $^{\circ}$ Shore A för "Material 1" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RH.

*Shore A-hardness for "Material No 1" as a function of storing time at different temperatures. Initial storing: 7 days at +20°C/50% RH.*

graduated in Shore A. These determinations were made in connection with other tests. However, the method is marred by a relatively great uncertainty in the values obtained. For example, the magnitude of the values depends on how quickly the durometer is applied to the material, the time of reading (instantaneous, after 5 seconds etc.); the condition of the surface of the material.

The method has the advantage of being quick and easy to carry out. One can also measure the hardness in the finished joint immediately.

The instantaneous hardness values in  $^{\circ}$ Shore A as a function of the time of curing/ageing are shown in Fig. 21. In spite of a fairly large scatter among the readings, the tendency from Fig. 20 for those stored in normal climate and those stored at  $+40^{\circ}\text{C}$  is similar. But the effect from heat ageing as shown in Fig. 21 has disappeared. The dispersion of the Shore A values hides this effect.

Note also that the value of  $13^{\circ}$  Shore A as stated by the manufacturer corresponds very badly with the measured values. A more accurate value is approx  $25^{\circ}$  Shore A.

The stress-strain curve has been used to quantify changes in the sealants (see Chapter 4.3). The deformation rate was 1.5 mm/min and the temperature  $+2^{\circ}\text{C}$ . This rate is much too fast compared with the rates of the joint movements which occur in real expansion joints. Hasselblad and Andersson (1972) have measured the magnitude  $10^{-3}$  -  $10^{-2}$  mm/min for the rate of the joint movement.

As most so called elastic sealants contain a varying plastic content this means that the shape of the stress-strain curve and the measured stresses are greatly dependent on the deformation rate.

Because of this the stress-strain curve has also been fixed at the deformation rate 0.0013 mm/min. The results are shown in Fig. 22. The decrease in rate has caused more than a halving of the developed tensile stress in the material.

For example with the help of Fig. 22 it is possible to estimate the forces transmitted by the jointing material to concrete elements, the adhesion stresses and the tensile stresses of adjoining materials. The Fig. also shows the dependence of the deformation properties on the temperature.

#### 6.2.2 Material No. 2

Fig 23 shows the effect of heat ageing on material No. 2, a two-component polysulphide sealant. Hardness approx 40<sup>0</sup> Shore A, according to the manufacturer. The comparison is upset by the fact that after heat ageing at 70<sup>0</sup>C the specimens showed the beginnings of adhesive and cohesive failure. When slightly elongated, the specimens can be considered to be unaffected, but a tendency to a decrease in the elongation resistance after heat ageing could to some extent be observed.

When measured in <sup>0</sup> Shore A, the material is unaffected by the heat ageing. The hardness value to some extent exceeds the value stated by the manufacturer.

#### 6.2.3 Material No. 3

This material, a one-component polyurethane sealant, shows a great increase in hardness when exposed to heat ageing. The stress-strain curves can be seen in Fig. 24. The increase in hardness is to some extent dependent on the heat ageing having accelerated the rather slow curing mechanism at the normal climate of 20/50. This can be seen in Fig. 25 where the stress at 50% elongation is noted as a function of the storage time in different climates.



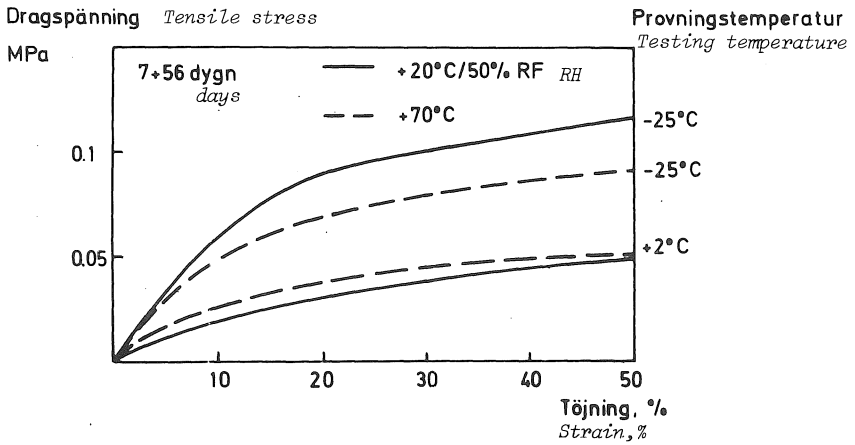


FIG 22 Arbetskurvor för "Material 1" efter värmelagring i olika klimat. Deformationshastighet: 0.001 mm/min.

*Stress-strain curves for "Material No 1" heat-aged in different types of climate. Deformation speed: 0.001 mm/min.*

Dragspänning *Tensile stress*

°Shore A

MPa

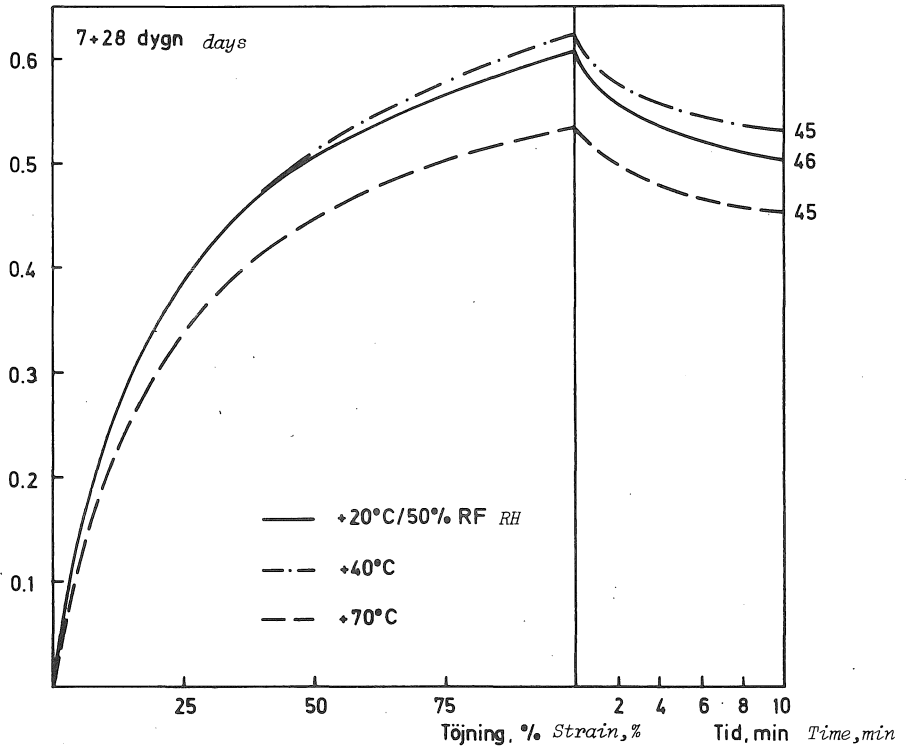


FIG 23 Arbetskurvor för "Material 2" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +20°C.

*Stress-strain curves for "Material No 2" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +20°C.*

Dragspänning *Tensile stress*  
MPa

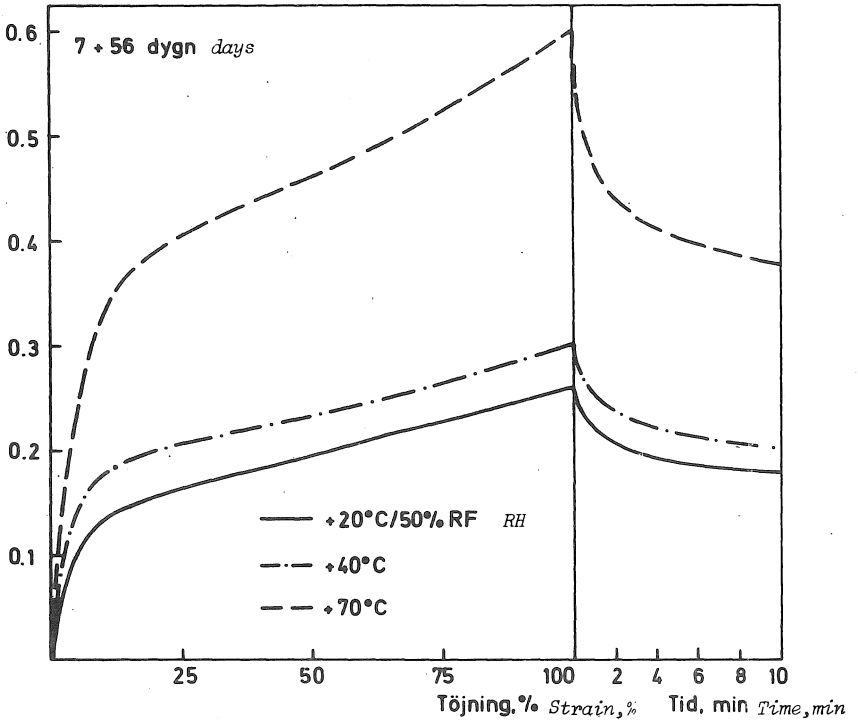


FIG 24 Arbetskurvor för "Material 3" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 3" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

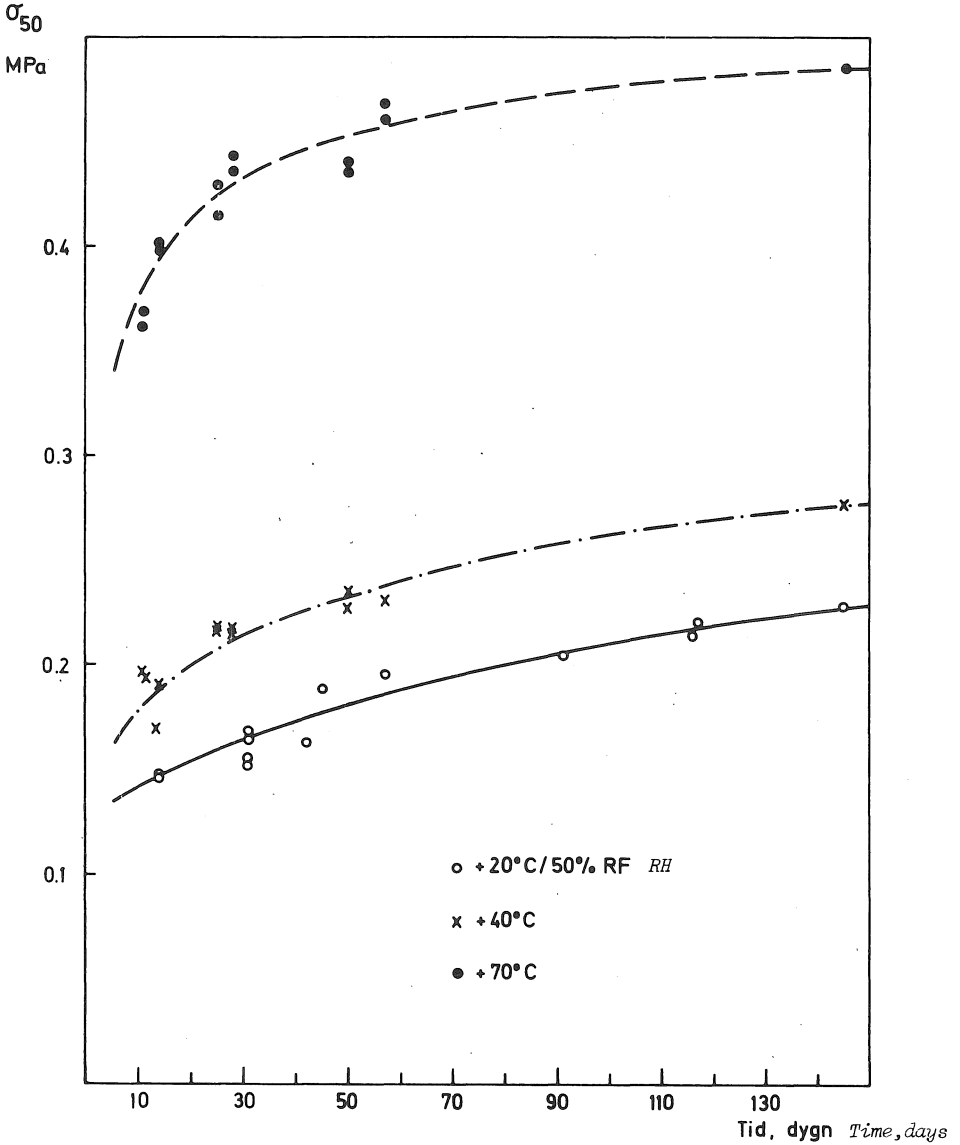


FIG 25 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 3" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RF.

*Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 3" as a function of storing time in different temperatures. Initial storing: 7 days at +20°C/50% RH.*

One can see that the normal hardening still continues after more than 150 days' storage at 20/50.

Roughly the same tendency can be observed in Fig. 26. The hardness in °Shore A is considerably greater than the value stated by the manufacturer ( $25 \pm 5$ ).

The great differences in deformation resistance between heat aged materials and normally stored materials (as shown in Fig. 24) is to some extent equalized at the slower deformation rate (Fig. 27).

#### 6.2.4 Material No. 4

Material No. 4 is a two-component sealant based on a polymer with a certain relationship to polyurethane. The hardness is stated by the manufacturer as being approx. 18°Shore A.

The stress-strain curves after heat ageing can be seen in Fig. 28 where the measured hardness values in °Shore A are also presented. The material is hardly affected by the heat ageing. The Shore A values are considerably above those stated by the manufacturer.

The effect of the test temperature and deformation rate on the stress-strain curve can be seen in Fig. 29. The heat ageing has hardly affected the material. The deformation resistance increases when testing at  $-25^{\circ}\text{C}$ .

#### 6.2.5 Material No. 5

This material is an acrylic sealant, solvent release. The material is plastic where deformation is concerned and therefore it is impossible and meaningless to try and give a hardness value in °Shore A.

The stress-strain curves for normally stored and heat aged materials can be seen in Fig. 30. The heat aged material became considerably harder and more brittle

° Shore A

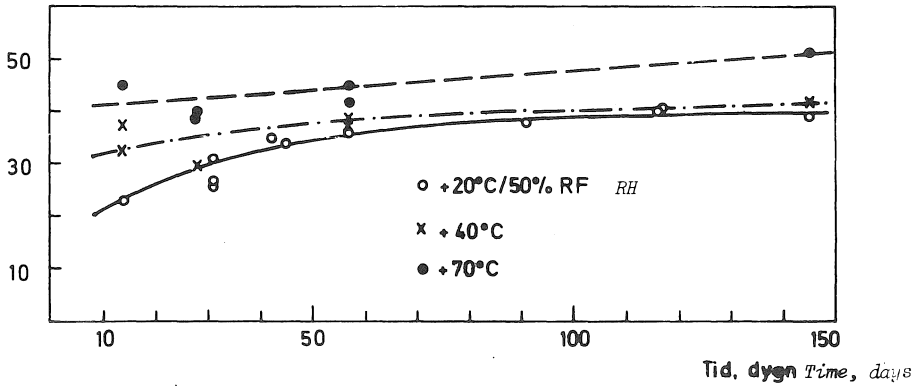


FIG 26 Hårdhet i °Shore A för "Material 3" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RF.

*Shore A-hardness for "Material No 3" as a function of storing time at different temperatures. Initial storing: 7 days at +20°C/50% RH.*

Dragspänning Tensile stress

MPa

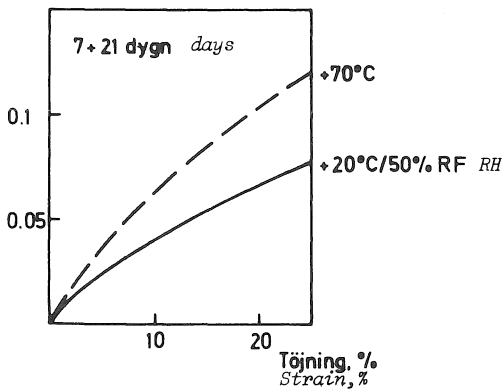


FIG 27 Arbetskurvor för "Material 3" efter värmelagring i olika klimat. Deformationshastighet: 0.001 mm/min. Provningsstemperatur: +20°C.

*Stress-strain curves for "Material No 3" after heat-ageing in different types of climate: Deformation speed: 0.001 mm/min. Testing temperature: +20°C.*

Dragspänning *Tensile stress*

° Shore A

MPa

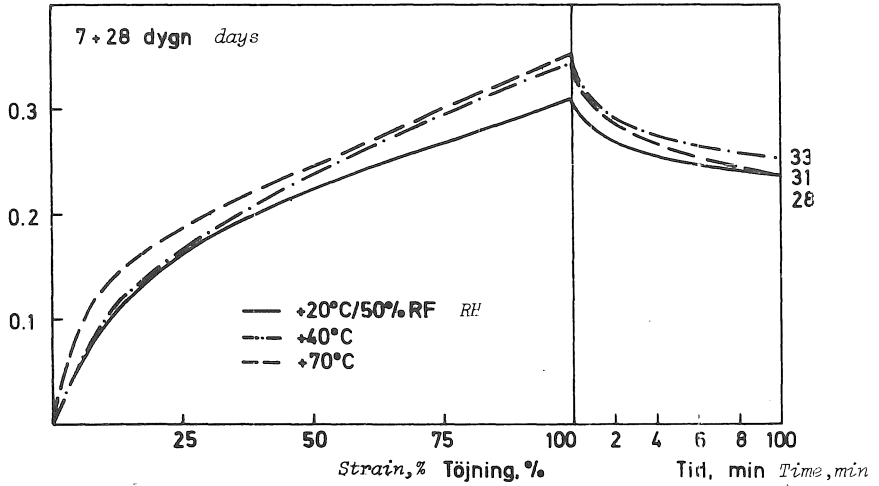


FIG 28 Arbetskurvor för "Material 4" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 4" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning *Tensile stress*

Provningsstemp.

MPa

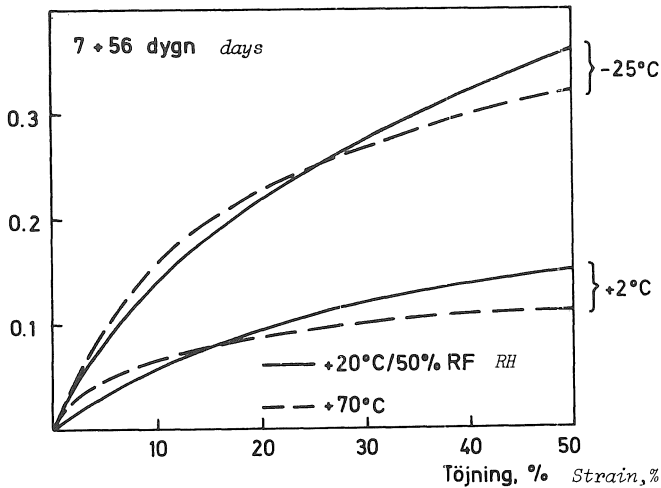
*Testing temperature*

FIG 29 Arbetskurvor för "Material 4" efter värmeåldring i olika klimat. Deformationshastighet: 0.001 mm/min.

*Stress-strain curves for "Material No 4" after heat ageing in different types of climate. Deformation speed: 0.001 mm/min.*

Dragspänning *Tensile stress*  
MPa

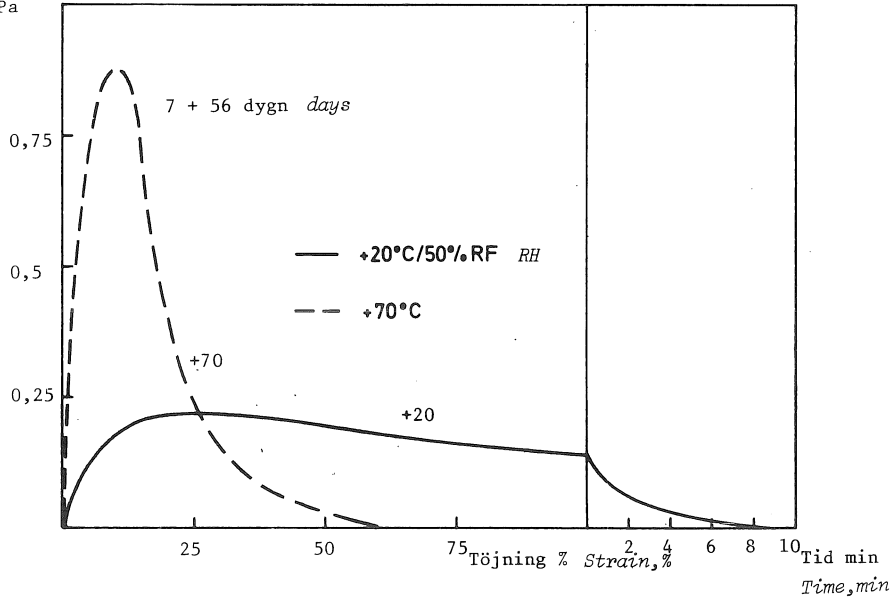


FIG 30 Arbetskurvor för "Material 5" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 5" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*



and the break elongation decreased. The pronounced tensile stress peak of the heat aged material is typical of so called plastic sealants. The deformation is distributed unequally over the joint width and the deformed part absorbs less and less stress, calculated according to the original joint cross section.

In this case, the heat ageing started after 7 days at +20°C/50% RH. At this time the material still contained a large quantity of solvent release (See Chapter 5.1). This has meant that the solvent had evaporated from the sealant leaving large pores as a result, see Figs. 13-14. This probably means that the stress-strain curve for heat aged materials in reality is higher than that in Fig. 30.

#### 6.2.6 Material No. 6

This material is also an acrylic sealant, solvent release. The polymer and the composition are very different to the previous material (No. 5).

The heat ageing at +70°C increases the deformation resistance considerably, see Figs. 31-33. The deformation process of the heat aged material is of a ductile nature, which to some extent is rather like that of certain elastic sealants. The large relaxation at 100% elongation shows the plasticity of the material.

No stress-strain curves are shown after 56 days heat ageing at +70°C. This is because partial adhesive failure usually occurred after such a lengthy heat ageing, irrespective of whether the heat ageing started after 7 days or after 21 days in a normal climate.

Figs. 31-32 show that the time when the heat ageing is started is of importance. Specimens where heat ageing started after 7 days in a normal climate had a stress-strain curve which was higher than for specimens heat aged after 21 days at 20/50. This is explained by the fact that different quantities and different components

Dragspänning *Tensile stress*  
MPa

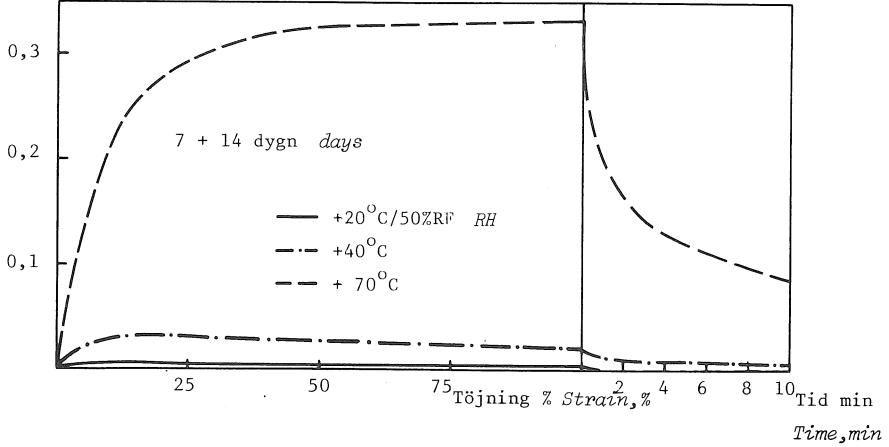


FIG 31 Arbetskurvor för "Material 6" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 6" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning *Tensile stress*  
MPa

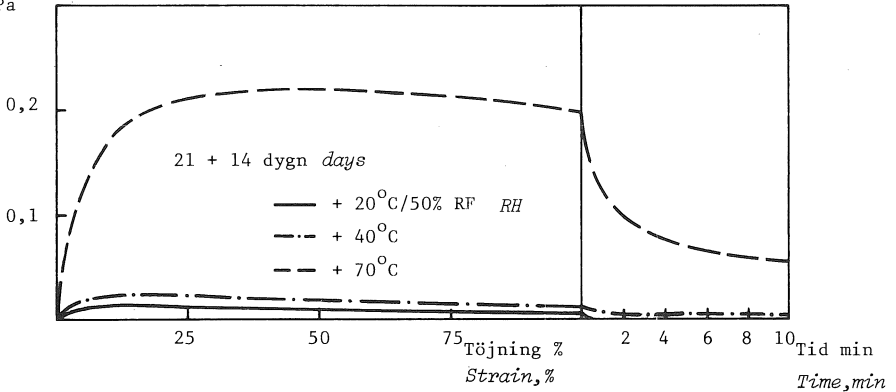


FIG 32 Arbetskurvor för "Material 6" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 6" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

included were removed in both cases (see Chapter 6.7).

The obtained tensile stress values at 50% elongation for material No. 6 are summarized in Fig. 34. The increase in the  $\sigma_{50}$  values for materials stored in a normal climate at +40°C follows a regular pattern. After heat ageing at +70°C there was very large dispersion of the values. This depended on the adhesion cracks which occurred after long period of storage at +70°C. The material became considerably harder when the heat ageing temperature was increased from +40°C to +70°C.

If the material is exposed to elongation at a more reasonable deformation rate one obtains stress-strain curves according to Fig. 35. The values for the heat aged specimens are rather uncertain due to the adhesive failures and cracks occurring during heat ageing. The Fig. shows that materials stored normally only became slightly harder with a decrease in the testing temperature to -25°C. The heat aged specimen is definitely thermoplastic.

#### 6.2.7 Material No. 7

Material No. 7, a water dispersed acrylic sealant, differs considerably from both of the solvent based acrylic sealants which were previously discussed. The material has a pronounced elastic part. This can be seen in Figs. 36-37. The elongation resistance is almost doubled after heat ageing. But the plastic part causes the stress relaxation at 100% elongation to become rather large (Fig. 36).

At a decrease in deformation rate, Fig. 37, the elongation resistance decreases considerably for the heat aged material as well as for the material stored normally.

Dragspänning *Tensile stress*  
MPa

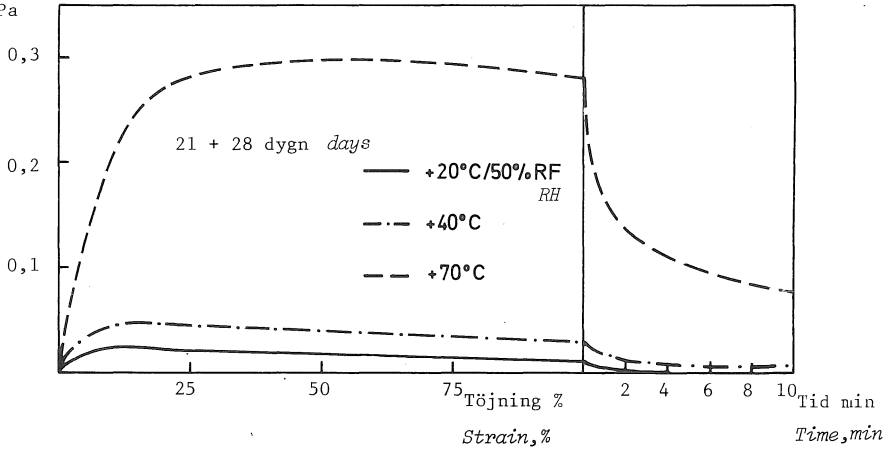


FIG 33 Arbetskurvor för "Material 6" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provnings temperatur: +2°C.

*Stress-strain curves for "Material No 6" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

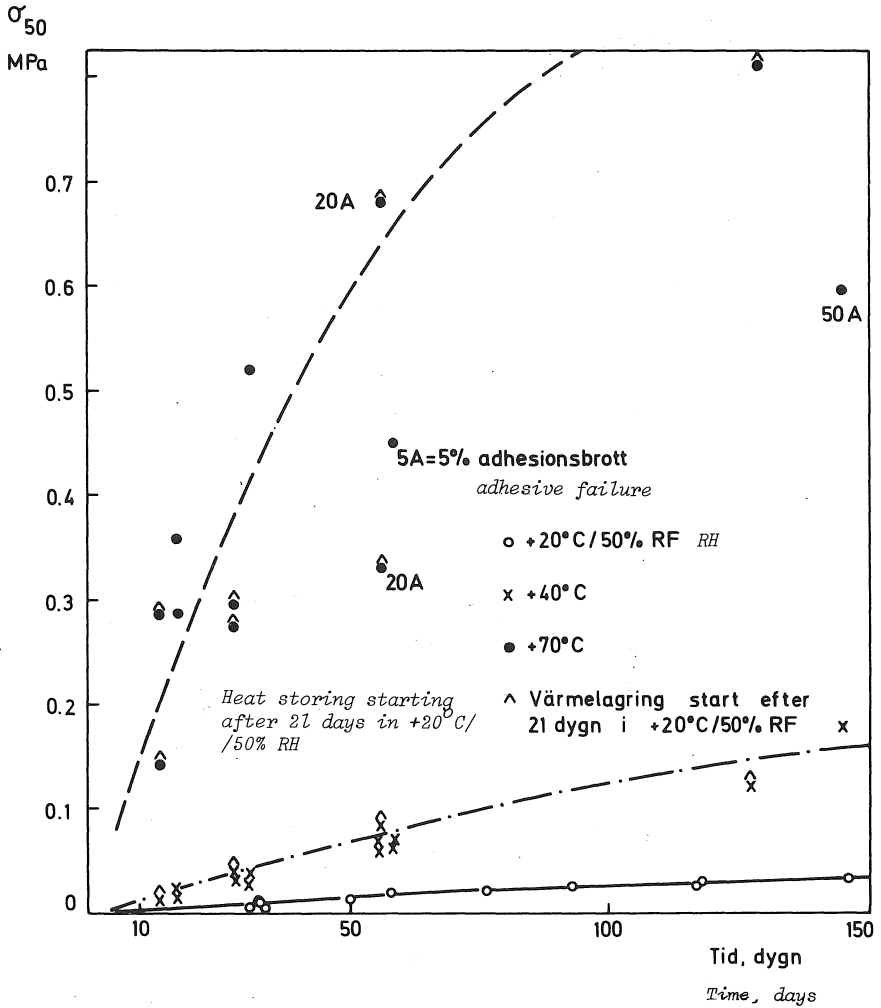


FIG 34 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 6" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RF.

Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 6" as a function of storing time in different temperatures. Initial storing: 7 days at +20°C/50% RH.

Dragspänning *Tensile stress*

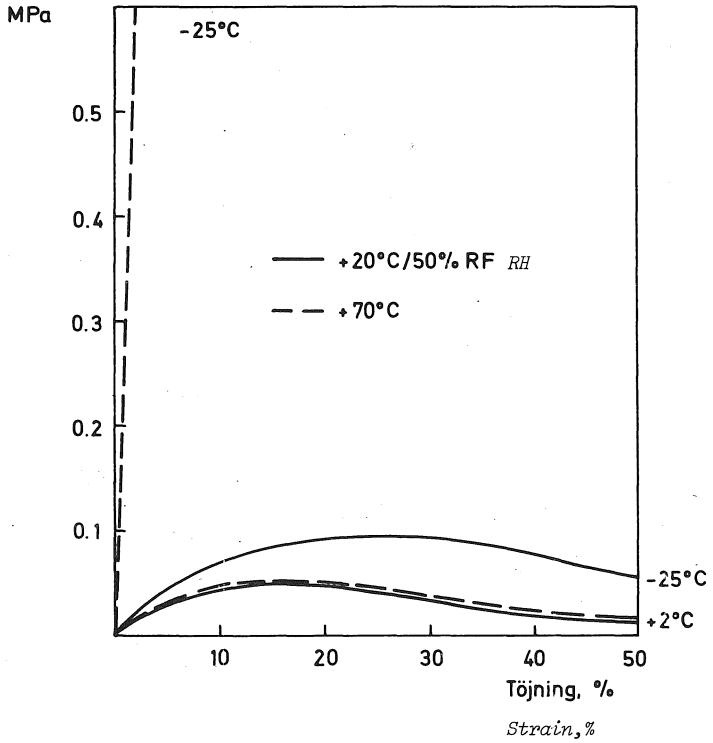


FIG 35 Arbetskurvor för "Material 6" efter värmeåldring i olika klimat. Deformationshastighet: 0.001 mm/min.

*Stress-strain curves for "Material No 6" after heat ageing in different types of climate. Deformation speed: 0.001 mm/min.*

Dragspänning *Tensile stress*  
MPa

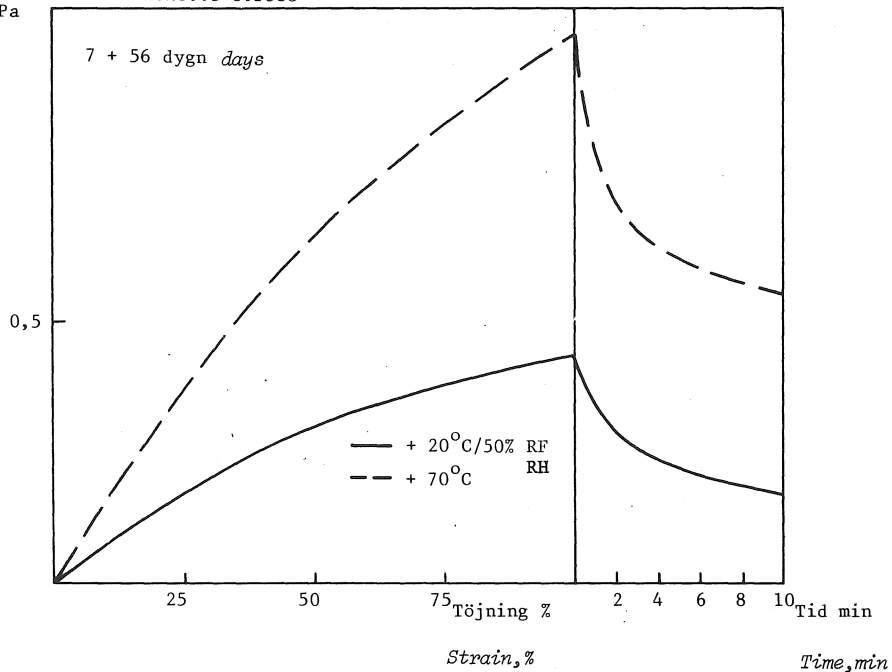


FIG 36 Arbetskurvor för "Material 7" åldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 7" aged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

The material is sufficiently elastic for the hardness values in °Shore A to be of interest. Fig. 38 shows these values after varying times in different climates. The hardness of the material when stored at +20°C/50% RH slowly continues to increase until, after about 50 days, most of the water has evaporated. See Chapter 6.7. The hardness values obtained can be compared to those stated by the manufacturer, i.e. approx 15°Shore A. The values measured here differ considerably from this value.

#### 6.2.8 Material No. 8

This material is an oil based sealant. Most of the properties differ considerably from those shown in the previous materials, for example both for the curing time, curing mechanism and the way in which the material fulfills its sealing function. This also means that it is rather complicated to test the material in the laboratory.

After the application, an oxidation starts on the surface and a somewhat elastic skin is formed. The material functions well as long as this skin is intact as it prevents continued oxidation of the inner part of the joint section.

The effect of heat ageing is shown in Fig. 39. The position of the stress peak moved towards a lesser elongation after heat ageing. This is due to the fact that the rise in temperature increased the thickness of the skin at the same time as a certain oxidation took place in the centre of the material. The skin cracks at a certain elongation. This means that the continued elongation of the joint width will take place in the part of the centre where the surface crack entered. Because of the oxidation that has partly taken place around the centre due to the high temperature, the ability to distribute the elongation has decreased to some extent.

If the elongation rate is lowered to approx 0.001 mm/min,



Dragspänning *Tensile stress*  
MPa

Provningstemp.  
*Testing temperature*

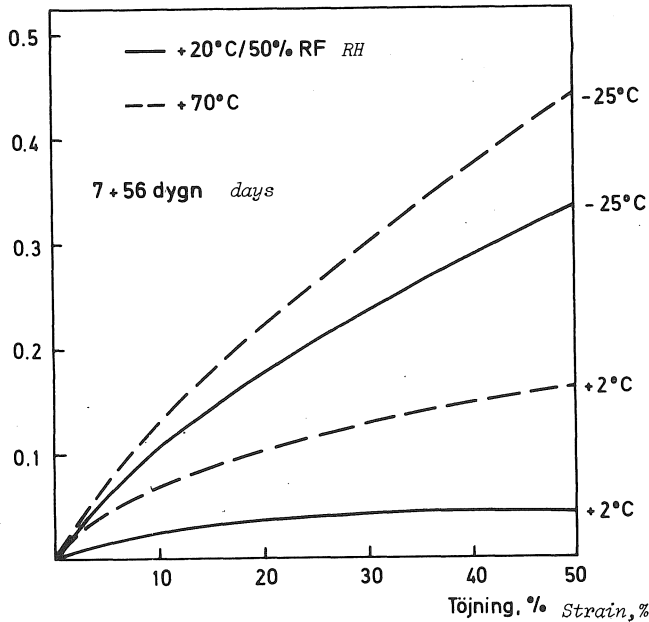


FIG 37 Arbetskurvor för "Material 7" åldrat i olika temperaturer. Deformationshastighet: 0.001 mm/min.

*Stress-strain curves for "Material No. 7" aged at different temperatures. Deformation speed: 0.001 mm/min.*

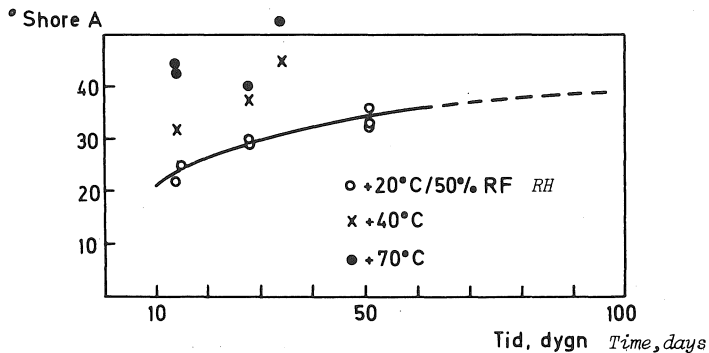


FIG 38 Hårdhet i °Shore A för "Material 7" som funktion av lagringstid i olika temperaturer. Initiallagring: 7 dygn i +20°C/50% RF.

*Shore A-hardness for "Material No. 7" as a function of storing time at different temperatures. Initial storing: 7 days at +20°C/50% RH.*

Dragspänning *Tensile stress*

MPa

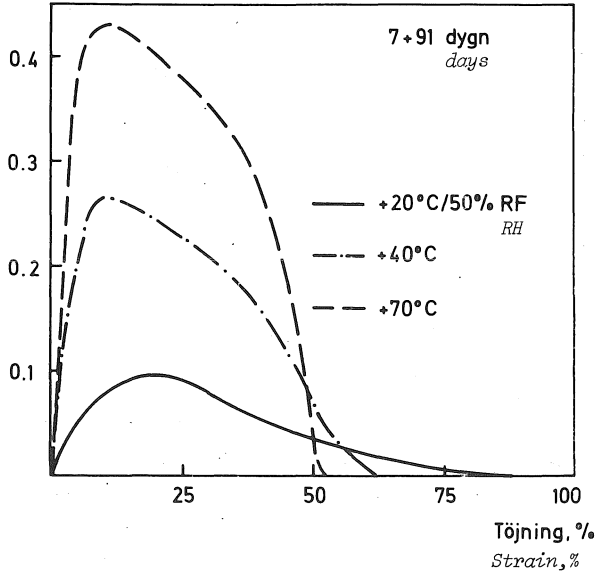


FIG 39 Arbetskurvor för "Material 8" värmeåldrat i olika temperaturer. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 8" heataged at different temperatures. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

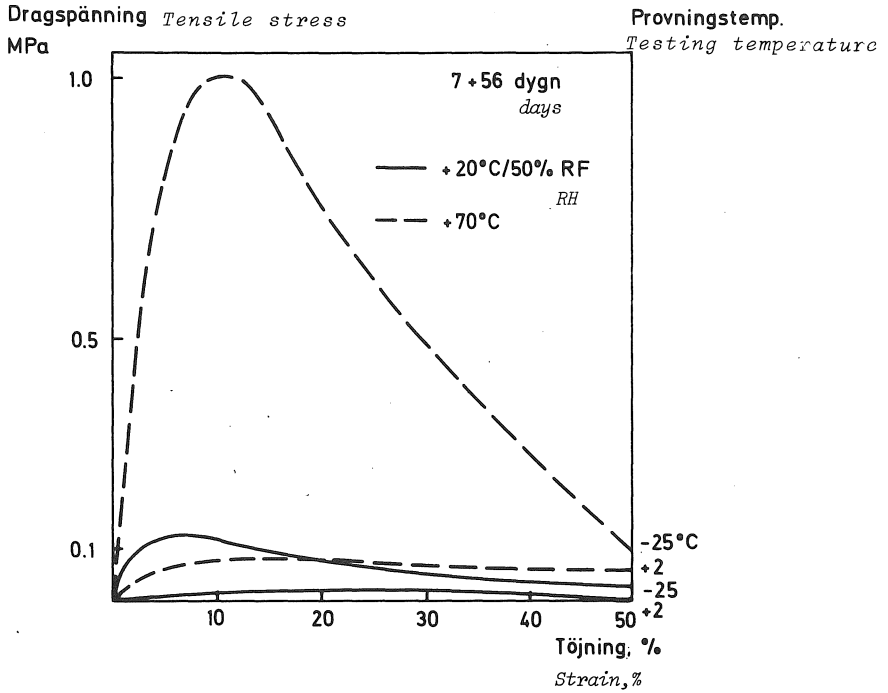


FIG 40 Arbetskurvor för "Material 8" efter värme-  
åldring. Deformationshastighet: 0.001 mm/min.

*Stress-strain curves for "Material No 8"  
after heat ageing. Deformation speed: 0.001  
mm/min.*

Fig. 40, the tensile stress decreases drastically. The Fig. also shows the effect of the test temperature on the test result.

The ability of an oil based sealant to function is greatly dependent on the fact that the thickness of the surface skin does not increase too quickly and that the skin has a certain ductility at the same time.

The thickness of the skin has therefore been determined at different times. This has been carried out after storage in different climates. A cut was made in the sealant with a scalpel. The cut was studied under a magnifier. By using the scale built into, the skin thickness has been calculated with an accuracy of about 0.05 mm. As the line between the skin and the centre is well defined, there were no problems in measuring the thickness.

Fig. 41 shows the connection between the thickness of the skin as a function of time. The relation is approximately a straight line in the log-log diagram. The curve is valid for material stored at  $+20^{\circ}\text{C}/50\% \text{ RH}$  and for material stored at a normal laboratory climate, about  $+22^{\circ}\text{C}/40\% \text{ RH}$ . Values for specimens stored in the UV chamber at  $+20^{\circ}\text{C}$  are also included in the Fig. Values for specimens stored outdoors are also included. One item relates to a specimen from a roughly eight year old joint (2920 days) between concrete elements, 2.7 m long. The joint faces southwest. Most of these values (except two UV stored specimens) correspond well to the other values in the Fig.

When storing specimens at an increased temperature the thickness growth of the skin of course took place quicker than the Fig. shows.

With the aid of skin thickness, measured after storage at  $+40^{\circ}\text{C}$  and  $+70^{\circ}\text{C}$ , the real storage times at the respective temperatures have been recalculated to the equivalent times at  $+20^{\circ}\text{C}/50\% \text{ RH}$ . The calculations are partly

Skinntjocklek (=S) *Skin thickness*

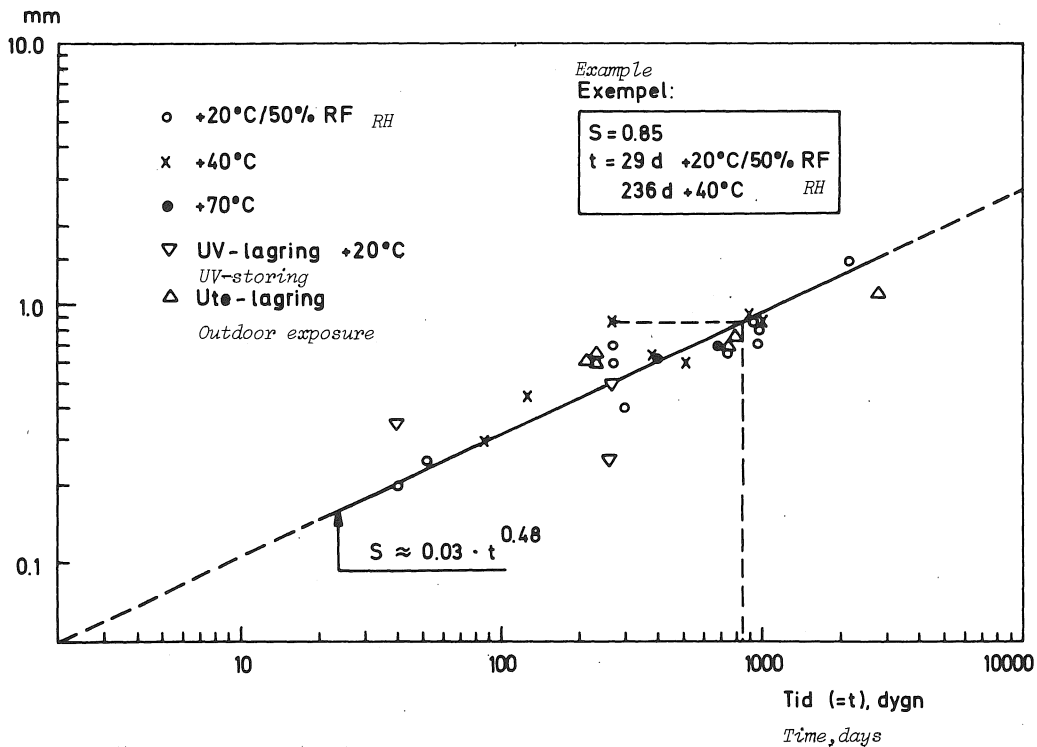


FIG 41 Skinntjockleken som funktion av lagringstiden i +20°C/50% RF för "Material 8". Beräkning av ekvivalent lagringstid i +20°C/50% RF för material lagrat i +40°C eller +70°C.

*Skin thickness as a function of storing time at +20°C/50% RH for "Material No 8". Computing of equivalent storing time at +20°C/50% RH for material stored at +40°C and +70°C.*

shown in the example in the Fig.

Point S =  $0.85/t = 265$  (29 days at +20/50 and 236 days at +40°C) has, according to the Fig., an equivalent hardening time of about 850 days at 20/50. As an example, suppose that the skin growth is  $x$  times faster at +40°C than at +20°C/50% RH.

$$29 + x \cdot 236 = 850$$

$$x = \text{approx. } 3.5$$

The average value for the corresponding calculations for a number of other points was about 4.0. After having recalculated the real times in the respective climates to equivalent times, the values of the heat aged specimens have been marked in the Fig. As can be seen the values are relatively well gathered.

Corresponding calculations for material stored at +70°C resulted in a growth factor of about 22.

One can consequently say that heat storage at +40°C results in ageing which is 4 times faster than for storage at +20°C/50% RH. The factor 22 is valid for heat storage at +70°C.

However the time at +70°C must be limited (approx < 100 days) as, in the long run, such a high temperature results in oxidation and a considerable increase of the viscosity in the centre, regardless of the growth of the thickness of the skin. This does not occur in a normal climate.

### 6.3 Humidity and alkali influence

Problems arose in keeping the intended pH-values in the humidity and alkaline baths. The intended level of the pH-value in humidity storage was approx. 4.0. However, this value later changed towards 6.0. By changing the baths relatively often, they could be kept at a pH of 4-6.

The pH-value changed rather quickly, also with alkaline influence. The intended level was 11.0 but this value decreased after a week to approx 6.5. The bath had to be changed often in this case as well. On the average the pH-value for the alkaline baths was 9-10.

Humidity and alkaline influence are presented parallely below, as the test methods for the different tests are very similar. It also appeared that where most materials are concerned there were only slight differences in humidity and alkaline influence.

#### 6.3.1 Material No. 1

By comparing stress-strain curves for specimens stored in the water and alkaline baths, Fig. 42, with the stress-strain curve for material stored at the normal climate 20/50 one can draw the conclusion that there was no significant difference.

This is also the case when comparing  $\sigma_{50}$  at other times, Figs. 43 and 20.

#### 6.3.2 Material No. 2

Where this material is concerned, the same conditions are valid as for the previously mentioned material. Several adhesive and cohesive failures have also upset the comparisons.

#### 6.3.3 Material No. 3

Figs. 44 and 45 show that no significant differences exist between the water and the alkaline stored specimens.

On the other hand, if one compares these specimens with those stored at 20/50 (Figs. 44 and 24) one finds marked differences. When comparing the curves in Fig. 44 with those in Fig. 24 the water storage brings about a considerably larger increase in hardness than for example

Dragspänning *Tensile stress*  
MPa

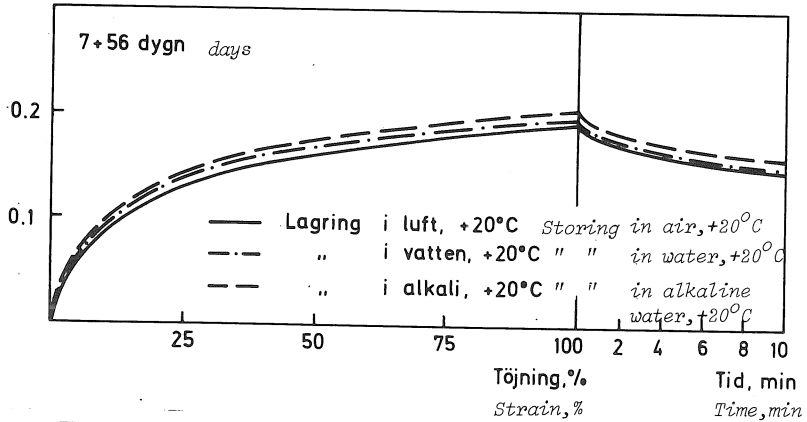


FIG 42 Arbetskurvor för "Material 1" efter lagring i luft, vatten och alkali. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +20°C.

*Stress-strain curves for "Material No 1" after storing in air, water and alkaline water. Deformation speed: 1.5 mm/min. Testing temperature: +20°C.*

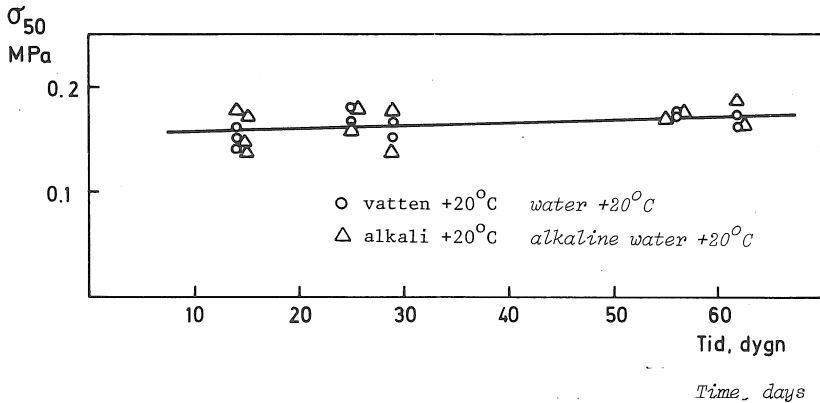


FIG 43 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 1" som funktion av lagringstid i vatten och alkali. Initiallagring: 7 dygn i +20°C/50% RF.

*Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 1" as a function of storing time in water and alkaline water. Initial storing: 7 days at +20°C/50% RH.*



Dragspänning *Tensile stress*  
MPa

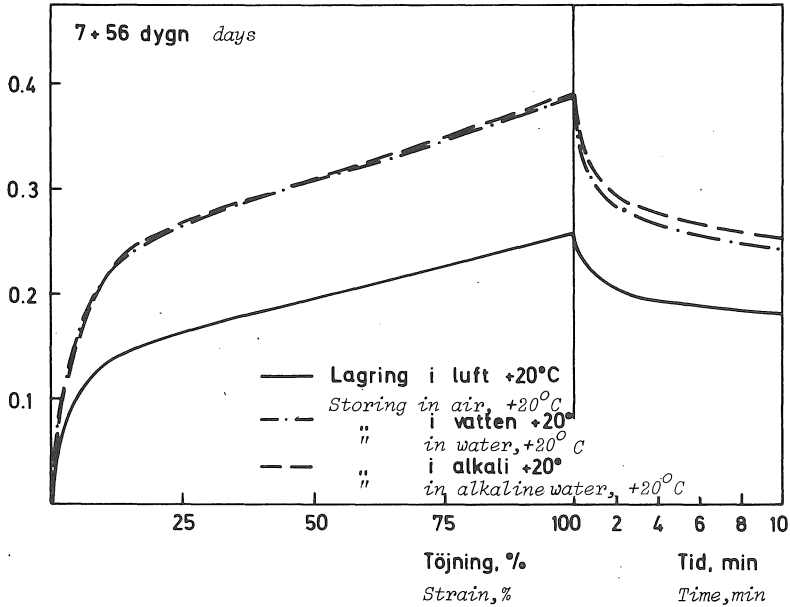


FIG 44 Arbetskurvor för "Material 3" efter lagring i luft, vatten och alkali. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 3" after storing in air, water and alkaline water. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

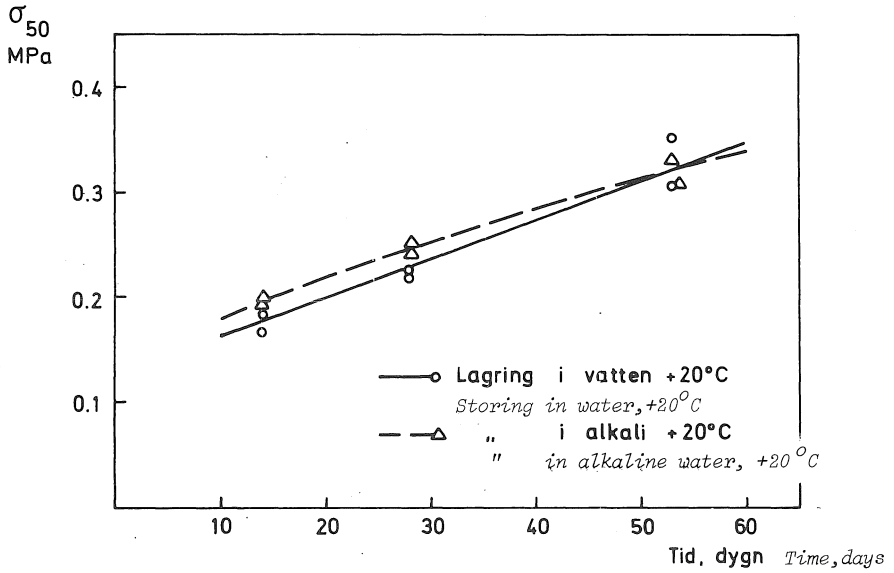


FIG 45 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 3" som funktion av lagringstid i vatten och alkali. Initiallagring: 7 dygn i +20°C/50% RF.  
 Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 3" as a function of storing time in water and alkaline water. Initial storing: 7 days at +20°C/50% RH.

+40°C. The same thing can be observed if Fig 45 is compared with Fig. 25. Fig. 45 also shows that the increase in hardness still continues after more than 50 days of water storage.

As material No. 3 cures by means of the air humidity, it is natural that water storage decreases the curing time. After more than 50 days this "normal" curing should be complete. The increase in hardness at this time is probably connected with other mechanisms.

#### 6.3.4 Material No. 4

For material No. 4 storage in water and alkali baths results in a certain softening. This is shown in Fig. 46. It probably depends on the fact that the material absorbs a certain amount of water during storage in water. See Chapter 6.7.

#### 6.3.5 Material No. 5

There are no stress-strain curves to be shown for material No. 5. This is due to the plasticity of the material and the fact that the material produces such a small deformation resistance that possible differences are difficult to quantify in this way. However, shrinkage and weight loss have been determined after storage in water and alkali. See Chapter 6.7.

#### 6.3.6 Material No. 6

Material No. 6 is also mainly plastic, but in spite of this Fig. 47 shows stress-strain curves after storage in water and alkaline. If the stress-strain curve is determined immediately after storage in water or alkaline, one can see that the material has softened. This depends on the fact that the material has absorbed water. See Chapter 6.7. An ocular inspection also shows that the joint profile has expanded and the surface begins to break up. The latter refers particularly to alkali storage.

Dragspänning *Tensile stress*  
MPa

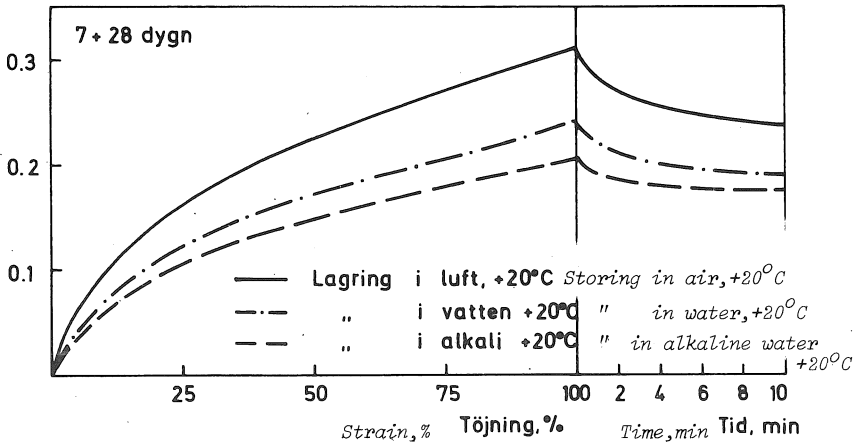


FIG 46 Arbetskurvor för "Material 4" efter lagring i luft, vatten och alkali. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 4" after storing in air, water and alkaline water. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning *Tensile stress*  
MPa

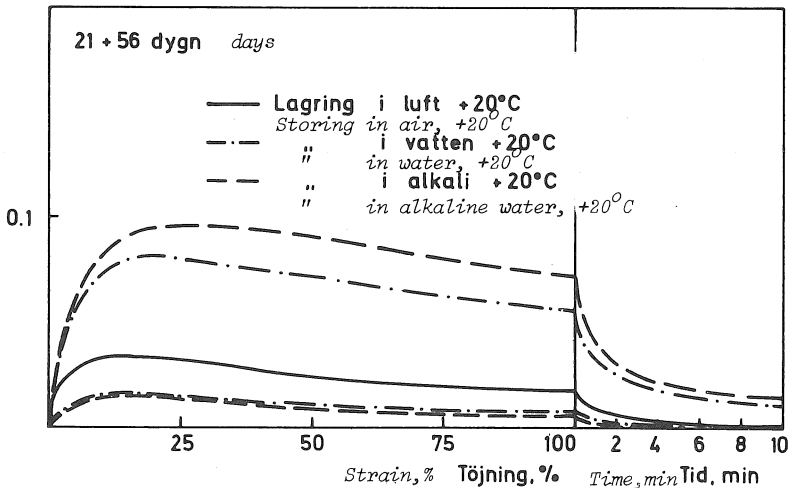


FIG 47 Arbetskurvor för "Material 6" efter lagring i luft, vatten och alkali. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 6" after storing in air, water and alkaline water. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

If the specimens had been dried out after storage in moisture and alkaline respectively, the effect would have been an increase in hardness of the material instead. Fig. 47.

#### 6.3.7 Material No 7

No stress-strain curves after moisture and alkaline storage are available for material No. 7. But shrinkage and weight loss have been measured during moisture and alkaline storage. Chapter 6.7.

#### 6.3.8 Material No 8

The same conditions are valid for material No.8. This material is also much too plastic for it to be worthwhile to present stress-strain curves after this type of ageing.

### 6.4 Influence of UV radiation

Generally it can be said that the UV-radiation only slightly affect the materials. For example, this has been in the form of change in colour, a dull surface, wrinkling and in certain cases cracking.

As an example of the latter, Fig 48 shaws a SEM photo of a polysulphide sealant exposed to UV radiation for about 21 days. The cracking appears to go deep into the material. A cut through the specimen, Fig. 49, shows that the effect of the UV radiation is of a very superficial nature. However, it is both possible and probable that this effect in the long run contributes to an increased cracking when the UV light is combined with varying joint movements, which is the case outdoors.



FIG 48 Yta av polysulfidbaserad fogmassa UV-bestrålad 21 dygn. Förstoring: 600x. Foto: Zoologiska institutionen, Lunds universitet.

*Surface of a polysulphide based sealant UV-irradiated for 21 days. Enlargement: 600x. Photo: The Zoological institution of Lund Univeristy.*

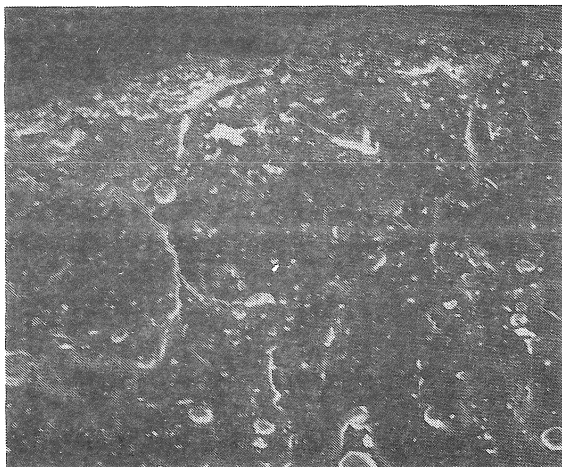


FIG 49 Snitt genom UV-bestrålad polysulfidbaserad fogmassa enligt fig 48. Förstoring: 120x. Foto: Zoologiska institutionen, Lunds universitet.

*Cut through a UV-irradiated polysulphide based sealant according to picture 48. Enlargement: 120x. Photo: The Zoological institution of Lund University.*

#### 6.4.1. Material No 1

Using the stress-strain curve as a quantifying method, material No 1 remained unaffected by the UV radiation, Fig 50. Fig 51 should be compared with Fig 20.

Ocular inspection of the UV irradiated surface shows a fine cracking and dullness of the surface. When the material is exposed to elongation this cracking breaks but the cracks are too shallow to be seen on the stress-strain curve. See also Fig 49.

#### 6.4.2. Material No 2

No important effects of the UV radiation could be seen on material No 2. This is also the case where surface cracking, was concerned. However a certain dullness could be observed on the surface.

#### 6.4.3. Material No 3

No difference in the form or level of the stress-strain curve was observed when comparing normally stored material with UV irradiated material (see Figs 52 and 25).

On the other hand, the surface of the specimens became yellow, which was probably due to the fact that the sealant was originally white. This phenomenon was also mentioned by the manufacturer. A certain embrittlement of the surface could also be observed. In some cases, about one millimeter deep cracks occurred along the edges of the joint profile.

#### 6.4.4. Material No 4

The only noticeable effect on material No 4 was a slight change in colour. No surface cracking or change of the stress-strain curve were observed.

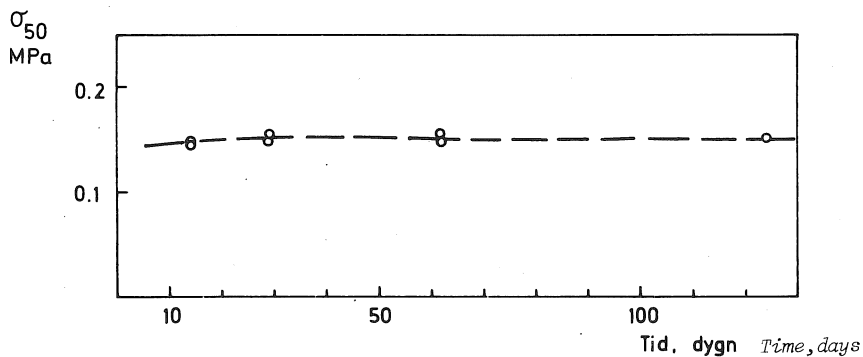


FIG 50 Arbetskurvor för "Material 1" efter lagring i luft och UV-ljus. Deformationshastighet: 1.5 mm/min. Provnings temperatur: +2°C.

*Stress-strain curves for "Material No 1" after storing in air and UV-light. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning Tensile stress  
MPa

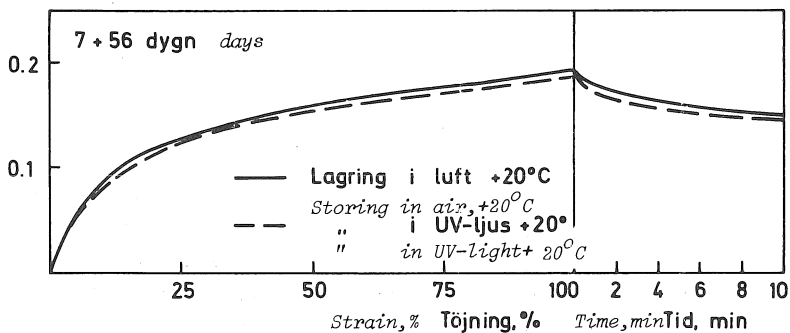


FIG 51 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 1" som funktion av lagringstid i UV-ljus. Initiallagring: 7 dygn i +20°C/50% RF.

*Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 1" as a function of storing time in UV-light. Initial storing: 7 days at +20°C/50% RH.*



#### 6.4.5 Material No 5

The material was not exposed to UV radiation.

#### 6.4.6 Material No 6

No difference was noticed between normally stored and UV irradiated specimens where the level or the shape of the stress-strain curve were concerned. On the other hand, the original white surface turned slightly yellow during the UV radiation, and a certain wrinkling occurred at the same time. A thin skin with limited ductility was formed. See Fig 53.

The absorption spectrum within the UV range was determined for material No 6 after normal storing as well as after UV radiation. The material had an absorption peak at about 296 nm, regardless of the ageing climate.

#### 6.4.7 Material No 7

The material was not exposed to UV radiation.

#### 6.4.8 Material No 8

No difference was measured between normally stored and UV irradiated specimens where the level or the shape of the stress-strain curve were concerned. The white surface of the sealant turned yellow during the UV radiation. At the same time, the surface wrinkled slightly. An elongated specimen is shown in Fig 54.

UV radiation did not increase the skin formation noticeably. See Fig 41.

### 6.5 Ozone influence

All the 8 materials were exposed to ozone. No effects of the ozone on materials that were not elongated have been observed, neither after ocular inspection nor when the stress-strain curve was determined. This is also the case

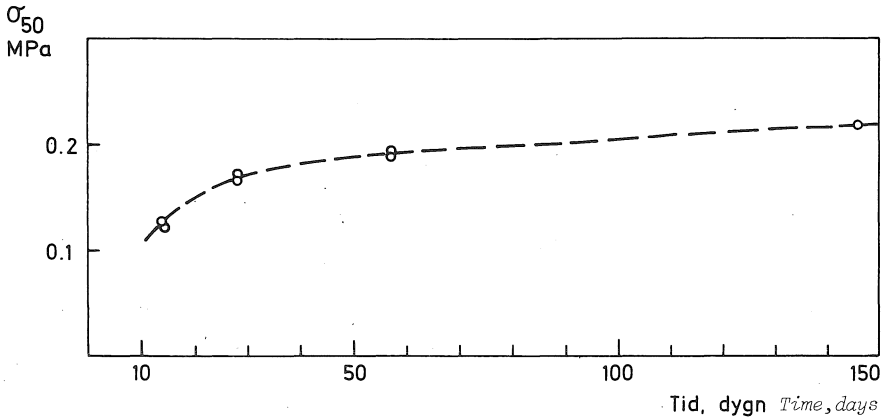


FIG 52 Dragspänning vid 50% töjning ( $= \sigma_{50}$ ) för "Material 3" som funktion av lagringstid i UV-ljus. Initiallagring: 7 dygn i  $+20^{\circ}\text{C}/50\% \text{ RF}$ .

*Stress at 50% tension ( $= \sigma_{50}$ ) for "Material No 3" as a function of storing time in UV-light. Initial storing: 7 days at  $+20^{\circ}\text{C}/50\% \text{ RH}$ .*

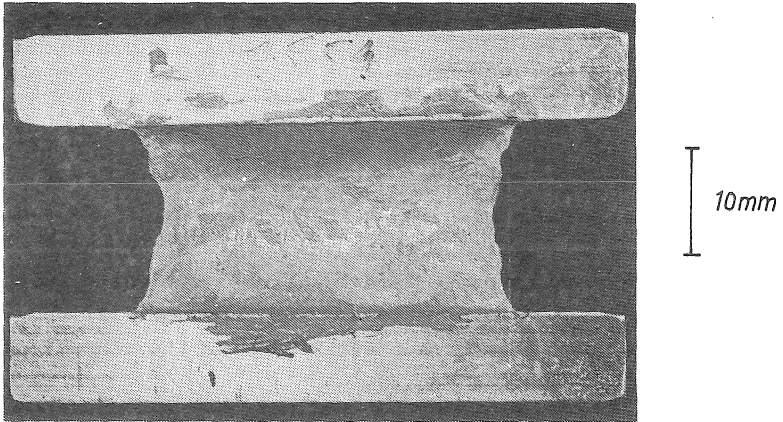


FIG 53 Sprickbildning i ythud som bildats vid UV-lagring av "Material 6".

*Crack formation in surface skin formed at the UV-storing of "Material No 6".*

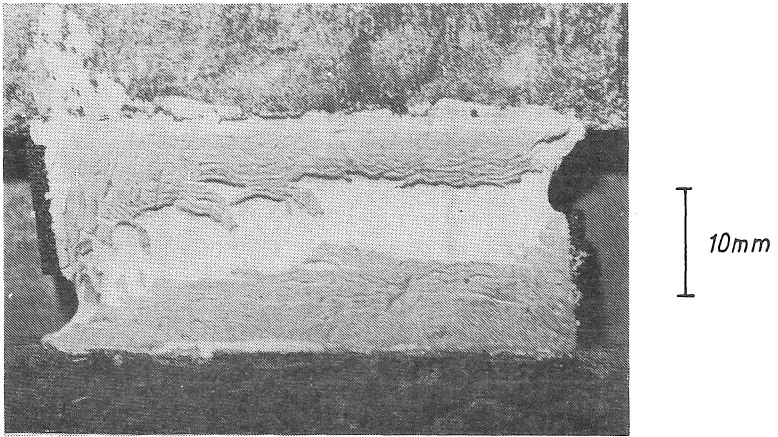


FIG 54 Sprickbildning i ythud som bildats vid UV-lagring av "Material 8".

*Crack formation in surface skin formed at the UV-storing of "Material No 8".*

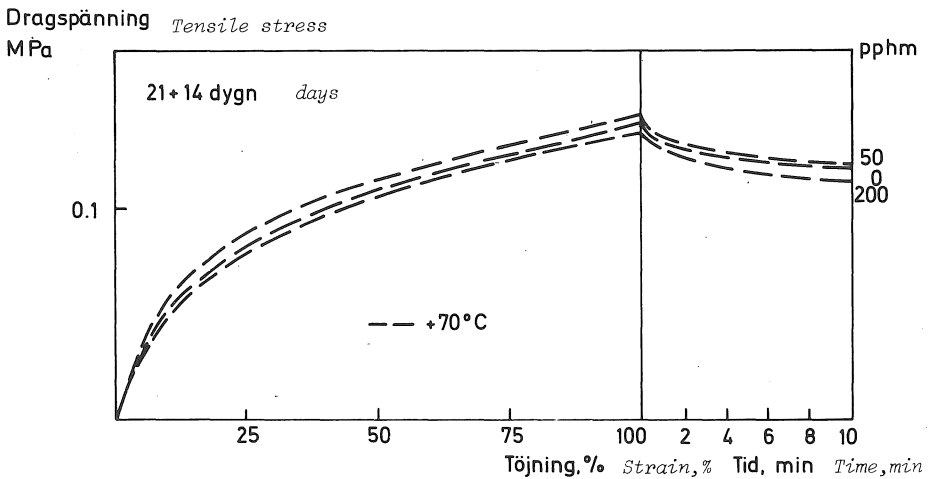


FIG 55 Arbetskurvor för "Material 1" efter lagring vid 25% töjning och varierande ozonhalt. Deformationshastighet: 1.5 mm/min. Provningstemperatur: +2°C.

*Stress-strain curves for "Material No 1" after storing at 25% tension and different ozone level. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

for materials Nos 5, 6 and 8, and for specimens from the other materials, which were not elongated.

For practical reasons, a long period of time passed between when the specimens were elongated and when they were exposed to ozone (see chapter 5.4). During this time the extent of relaxation was measured. The level for the different materials is shown in Table 4. The Table shows that for certain materials, a large part of the original stress had time to relax before being exposed to ozone after about 65 hours.

#### 6.5.1 Material No 1

Several adhesive failures have sometimes made comparisons difficult. The adhesion cracks occurred both when fixing the stress-strain curves and when the specimens were kept, elongated, in the ozone chamber.

Fig 55, specimens stored at 25 % elongation, shows that there was no significant influential difference between the different ozone contents.

Fig 56, valid for specimens elongated 50 %, shows a certain decrease in the deformation resistance for the highest ozone content. The reason for this is shown in Fig 57. The specimen shows typical ozone cracks oriented at right angles to the direction the elongation. Figs 57 and 58 show both the effect of the ozone content and the extent of the elongation. 50 % elongation in 50 pphm ozone content produces no cracks, but 25 % elongation at 200 pphm produces certain tendencies to cracking.

#### 6.5.2 Material No 2

Due to both adhesive and cohesive failures, both elongated and when kept in the ozone chamber, there are no definite comparisons deriving from the stress-strain curves.

However, Fig 59 shows, that ozone cracks occurred both at 25 and 50 % elongation at 200 pphm.

Table 4. Stress relaxation at +22°C and different levels of deformation and curing

Material No.	$\sigma_{25}^{\text{MPa}}$		$\sigma_{50}^{\text{MPa}}$		% Remaining stress after t hours			
	$\frac{+20^{\circ}\text{C}}{50\% \text{ RH}}$	Heataged in +70°C	$\frac{+20^{\circ}\text{C}}{50\% \text{ RH}}$	Heataged in +70°C	$\sigma_{25t}^{\text{MPa}}$	$\sigma_{25}^{\text{MPa}}$	$\sigma_{25}^{\text{MPa}}$	$\sigma_{25}^{\text{MPa}}$
1	adh.	0.07	adh.	0.10	-	46	-	52
	fail.		fail.			(t=25)		(t=65)
2	adh.	0.34	adh.	0.46	-	58	-	coh.
	fail.		fail.			(t=46)		fail.
3	0.10	0.27	0.15	0.29	52	33	49(t=53)	41
					(t=61)	(t=17)	46(t=143)	(t=42)
4	0.09	0.14	0.15	0.16	62	44	54(t=50)	52
					(t=60)	(t=20)	50(t=143)	(t=43)
7	0.05	0.22	0.12	0.34	19	15	17(t=53)	16
					(t=61)	(t=17)	14(t=143)	(t=42)

Dragspänning , *Tensile stress*

pphm

MPa

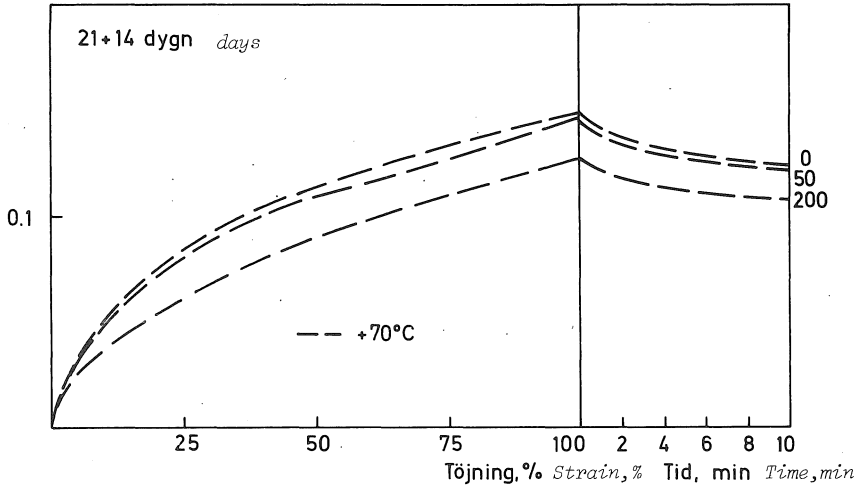


FIG 56 Arbetskurvor för "Material 1" efter lagring vid 50% töjning och varierande ozonhalt. Deformationshastighet: 1.5 mm/min. Provningstemperatur: +2°C.

*Stress-strain curves for "Material No 1" after storing at 50% tension and different ozon level. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

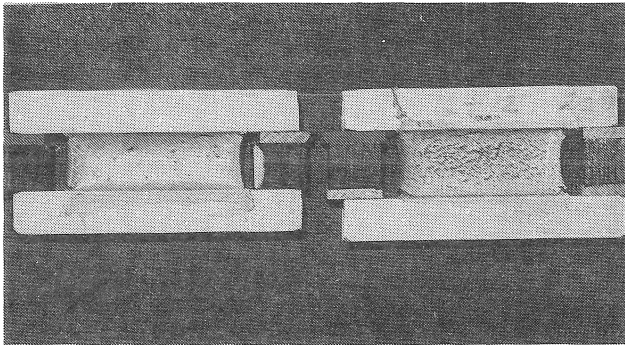


FIG 57 Värmeåldrade provkroppar av "Material 1" efter ozonlagring i 200 pphm. Vänster töjning 25% och höger provkropp töjning 50% vid ozonlagring.

*Heat aged specimens of "Material No 1" after storing in ozon of 200 pphm. The specimen to the left extended to 25% and the specimen to the right extended to 50% during the ozon exposition.*

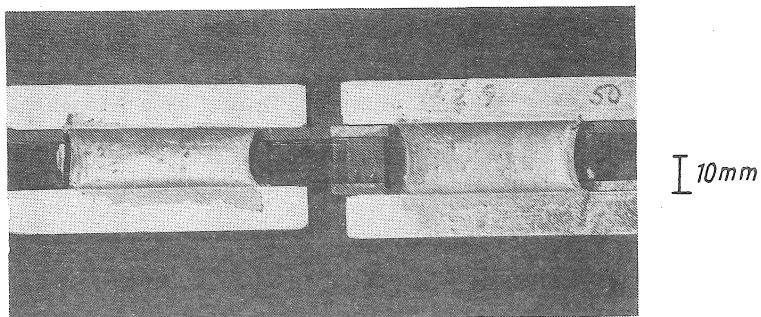


FIG 58 Värmeåldrade provkroppar av "Material 1" efter ozonlagring i 50 pphm. Väns- ter töjning 25% och höger töjning 50% vid ozonlagringen.

*Heat aged specimens of "Material No 1" after storing in ozon of 50 pphm. The specimen to the left extended to 25% and the specimen to the right extended to 50% during the ozon ex- position.*

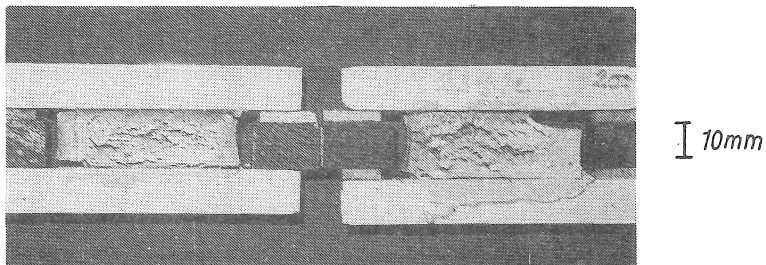


FIG 59 Värmeåldrade provkroppar av "Material 2" efter ozonlagring i 200 pphm. Väns- ter töjning 25% och höger töjning 50% vid ozonlagringen.

*Heat aged specimens of "Material No 2" after storing in ozon of 200 pphm. The specimen to the left extended to 25% and the specimen to the right extended to 50% during the ozon ex- position.*

Material No 2 is slightly more sensitive to ozone than the closely related material No 1. This is probably explained by the fact that material No 2 is harder, i.e. exerts a higher deformation resistance when elongated. This means that the stress, when exposed to ozone (Table 4), is higher in material No 2 than in material No 1.

#### 6.5.3 Material No 3

No significant effect of the ozone exposure has been proved, in spite of relatively great stresses in the material during exposure (Table 4).

#### 6.5.4 Material No 4

The same conditions are valid here as for material No 3.

#### 6.5.5 Material No 7

No significant effect of the ozone exposure has been proved. The remaining stress during exposure has probably been modest. (Table 4).

### 6.6 Outdoor exposure of joint specimens

Results from the determination of stress-strain curves for specimens installed in the equipment for outdoor exposure of joint specimens during various periods of time are presented below (See Chapter 4.5). There are still 3 specimens of each material installed. They will be tested after further outdoor exposure.

Some results obtained when measuring joint width variations and temperatures are also presented.



### 6.6.1 Measuring of joint width variations

Fig 60 shows examples of measured joint width variations on a sunny day with great temperature variations. The joint movement noted is the maximum joint width variation in the construction, i.e. at the end where the aluminium ladder is placed on movable bearings.

On this day the joint width changed by 3.35 mm. From the Figure it can be observed that a change in the surface temperature of the aluminium ladder immediately changes the joint width. The speed of the joint movement according to the Figure is about  $10^{-3}$ - $10^{-2}$  mm/min. During a given time the joint width remained almost unchanged.

The change in temperature in the centre of the 250 mm thick aerated concrete element was naturally slower. The maximum atmosphere temperature in this case was around mid day while the highest temperature in the aerated concrete occurred about 6 hours later. The gradually increasing temperature inside the aerated concrete resulted in a subsequent change in joint width in spite of the ladder temperature remaining at about the same high level.

Fig 61 shows a corresponding cycle for a day with less temperature variation. The maximum joint width variation for 24 hours was 1.55 mm.

The rapid changes in temperature measured in the aluminium construction and the slow changes measured in the aerated concrete are very important in practice. This means that sealants between aluminium constructions will be exposed to a large number of joint width variations at a comparative high deformation rate. For example, the joint width will change when the sun is temporarily hidden behind clouds. When sealants are used between concrete or aerated concrete elements they will mainly be exposed to a fairly regular, approximately sinusoidal joint movement, varying over a period of 24 hours.

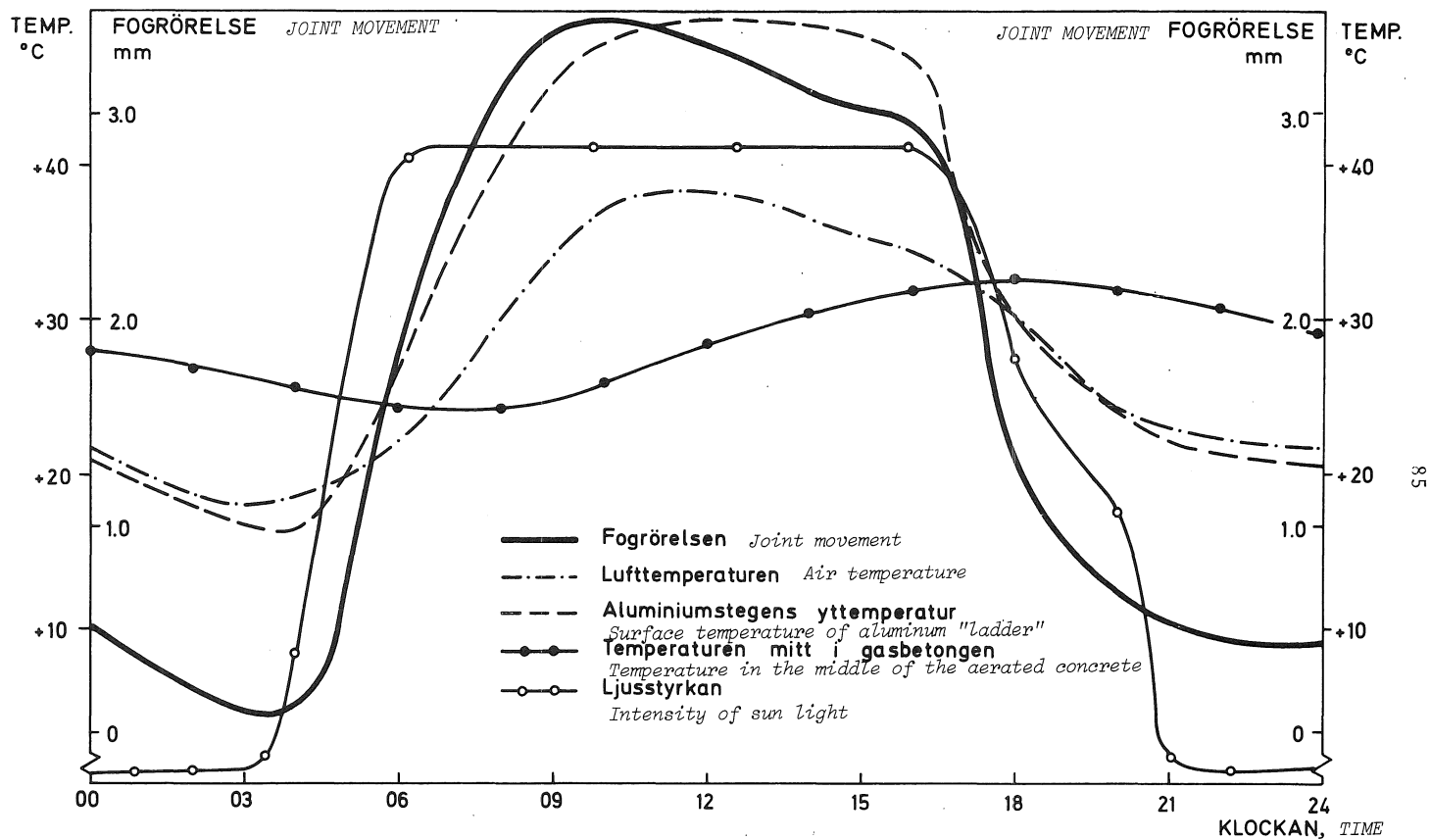


FIG 60 Exempel på ett dygn (750809) med stora uppmätta temperaturvariationer och därmed sammanhängande fogrörelser i utomhusutrustning för exponering av provkroppar.

An example of a day (750809) with large measured temperature variations and in connection to that joint movements in the outdoor equipment for exposition of specimens.

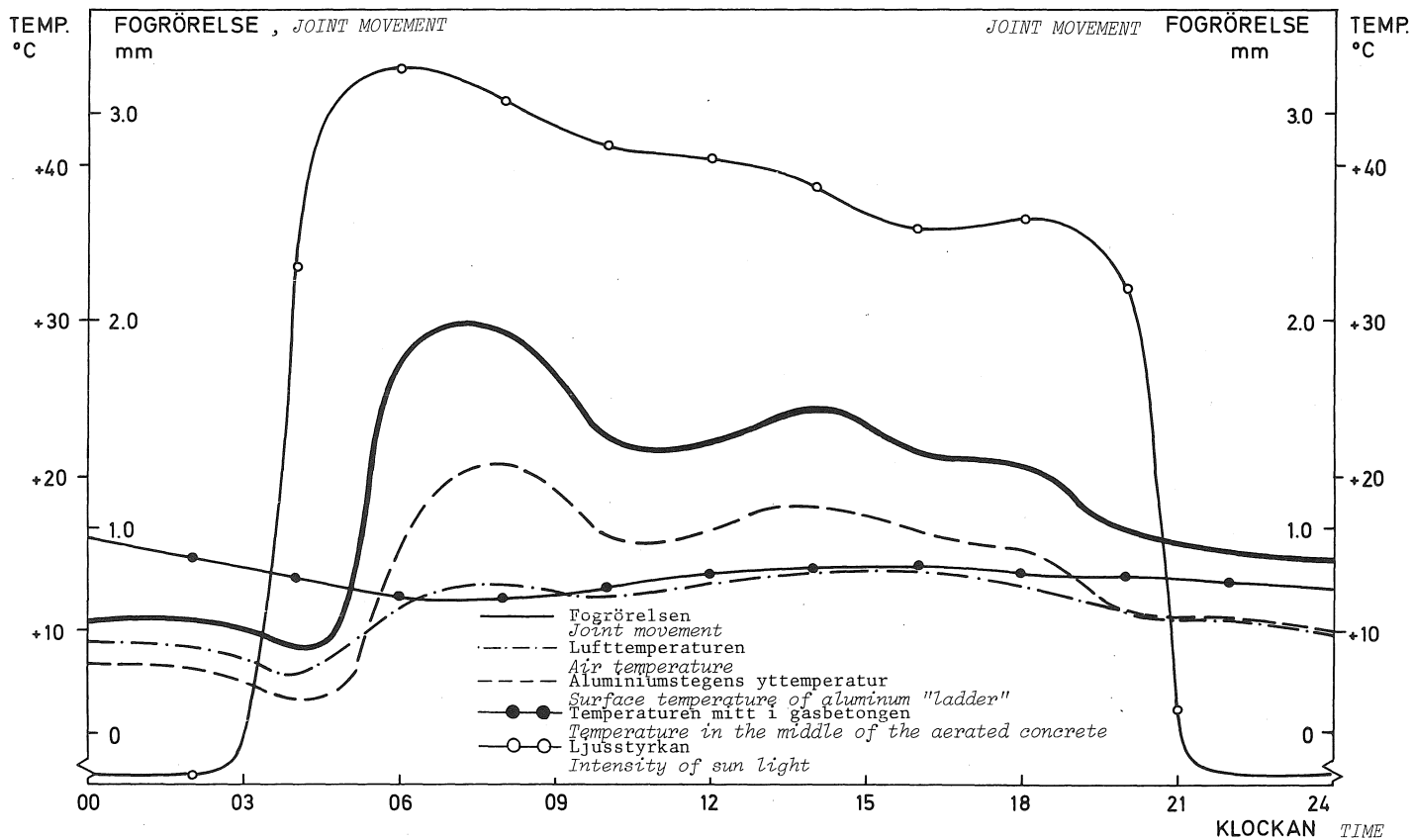


FIG 61 Exempel på ett dygn (760605) med små uppmätta temperaturvariationer i utomhusutrustning för exponering av provkroppar.

An example of a day (760605) with small measured temperature variations in the outdoor equipment for exposition of specimens.

Table 5 shows a summary of the maximum joint width variations of each specimen during the test period. During this period, the maximum elongating movement was 1.57 mm (calculated from the position when the specimens were fastened). At that time the ladder temperature was  $-10.3^{\circ}\text{C}$ . The maximum compressing movement was 3.63 mm. The ladder temperature was then  $+45.7^{\circ}\text{C}$ . Starting from the position of the specimen concerned along the construction, the movement of each specimen was calculated, under the assumption that the deformation decreased linearly to zero where the aluminium ladder was attached to the aerated concrete.

The measured distribution of movement indicated that the specimens mainly become compressed during the measuring period. This was also the purpose. There are indications which indicate that compressive movements cause greater stress on a sealant than a corresponding elongating movement, Hasselblad and Andersson (1972), Burström (1973), Hockman (1975). This both depends on the fact that large local deformation occurs during compression and that stress relaxation is greater at higher temperatures (the temperature is often higher when a joint is compressed).

Table 5 shows the values, stated by the manufacturer, for the maximum movement permitted in the joint. These values are not completely. Sometimes a value for the distribution of the movement, for example  $\pm 20\%$  is given, but mostly only a total maximum movement for the distribution is stated.

Table 5 also includes notes made during ocular inspection of the specimens.

All the specimens of material No. 1 clearly show surface cracking. This type of cracking is rather common in polysulphide sealants. Fig. 62 shows a specimen which was also exposed for about 7 months, but with mainly elongating joint movements. The specimen was also exposed for a long period of time during the hot season. This,

Table 5. Measured maximum deformations after 7 months of outdoor exposure

Material No.	Specimen No.	Max. tension %	Max. compression %	$\Sigma$ deform. %	Max. deform. according to the producer	Note
1	1261-62	8.9	20.6	29.5	33	Surface cracks Start adh. failure (1262)
	1263-64	3.5	8.0	11.5		"
	1266	8.9	20.6	29.5		"
	1267-68	3.5	8.0	11.5		"
2	2361-62	8.3	19.2	27.5	30	~20% adh. failure (2361) 100% adh. failure (2362)
	2363-64	2.8	6.6	9.4		
	2366	8.3	19.2	27.5		100% adh. failure
	2367-68	2.8	6.6	9.4		
3	3261-62	10.1	23.4	33.5	±20	Surface cracks Visible with magnifier
	3263-64	5.3	12.3	17.6		"
	3266	10.1	23.4	33.5		"
	3267-68	5.3	12.3	17.6		"
4	5561-62	11.9	27.7	39.6	50	
	5563-64	6.5	15.1	21.6		
	5566	11.9	27.7	39.6		
	5567-68	6.5	15.1	21.6		
5	6261-62	7.2	16.6	23.8	No inf.	Folded, dirty surface,
	6263-64	1.7	3.9	5.6		"
	6266	7.2	16.6	23.8		"
	6267-68	1.7	3.9	5.6		"
6	7561-62	10.8	25.0	35.8	50	Folded, dirty surf. Fig. 63
	7563-64	5.9	13.8	19.7		Small folds in the middle, dirty surf.
	7566	10.8	25.0	35.8		Folded, dirty surf.
	7567-68	5.9	13.8	19.7		Small folds in the middle, dirty surface

Table 5. (Continued)

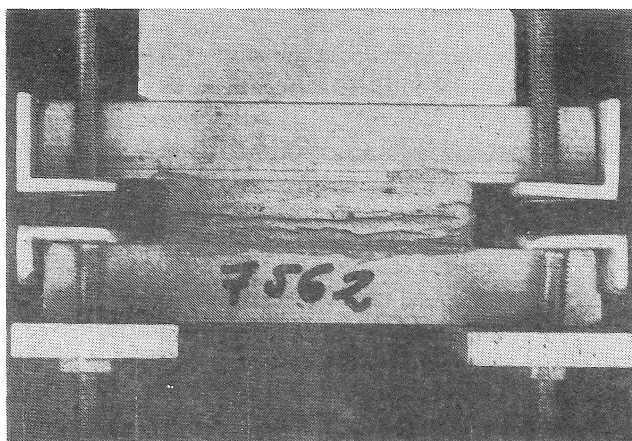
Material No.	Specimen No.	Max. tension %	Max. comp-ression %	$\Sigma$ deform. %	Max.deform. according to the producer	Note
7	8261-62	7.7	17.8	25.5	15	Dirty surface
	8263-64	2.3	5.2	7.5		"
	8266	7.7	17.8	25.5		Dirty surface, folded, Fig. 64
	8267-68	2.3	5.2	7.5		Dirty surface
8	9561-62	9.4	21.7	31.1	35	Folded
	9563-64	4.1	9.4	13.5		Slight folded
	9566	9.2	21.3	30.5		~5% adh.failure at the edge.Folded
	9567-68	4.1	9.4	13.5		Slight folded



10mm

FIG 62 Provkropp av "Material 1" som utomhus-exponerats ca 7 månader.

*Specimen of "Material No 1" outdoor exposed for about 7 months.*



10mm

FIG 63 Provkropp av "Material 6" som utomhus-exponerats ca 7 månader. Max drag 10.8%, max tryck 25.0%.

*Specimen of "Material No 6" outdoor exposed for about 7 months. Maximum tension 10.8%, maximum compression 25.0%.*

together with the repeated movements, resulted in the cracking penetrating rather deep into the specimen.

The sometimes defective adhesion, which has been previously mentioned regarding material No. 2 with its pertaining primer, is again confirmed by the outdoor exposed joints. One of the reasons for the many adhesive failures is naturally the high cohesion (large elongation resistance) of this material.

Material No. 3 also shows a tendency to surface cracking. This is difficult to distinguish with the naked eye, but it can be seen clearly if the specimen surface is studied through a magnifier.

Above all, materials Nos. 5, 6 and 7 and, to some extent, material No. 8 are so thermo-plastic in character that they became rather dirty.

The characteristic surface wrinkling of a mainly plastic sealant can be seen in Fig. 63, material No. 6.

Material No. 7 is more elastic but, in spite of this, there was a certain surface wrinkling. See Fig. 64.

#### 6.6.2 Temperatures in coloured sealants, exposed outdoors

In order to determine the temperatures in sealants of different colours, the temperature was measured in four different types of sealants:

<u>Material</u>	<u>Colour</u>
Silicone based sealant	Glossy black
Silicone based sealant	Glossy white
Material No. 7	Dull black. The surface of the specimen was very dirty after about 7 months of outdoor exposure.
Material No. 6	Dull white. The material was originally white. The surface somewhat dirty after 7 months of outdoor exposure.






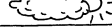


It is essential to know the temperature when heat ageing a sealant (so that the heat ageing is not pursued too far), when to determine deformation resistance, stress relaxation, sagging tendency, etc. All these properties are definitely temperature dependent for most sealants.

A thermocouple was inserted in each sealant so that the point was just below the surface. Fig. 65. Then the specimens were fastened to a vertical stand. The joint surfaces faced South.

Table 6 shows examples of measured temperatures. ( $^{\circ}\text{C}$ )

Table 6. Example of measured temperatures ( $^{\circ}\text{C}$ ) in sealants of different colours.

C o l o u r				Air temp (shade)	Cloudiness
Glossy black	Glossy white	Dull black	Dull grey/white		
42.3	40.0	44.3	41.3	30.0	
37.1	34.6	38.0	35.5	27.1	
34.8	32.3	36.6	33.7	24.3	
40.0	37.0	41.0	38.1	28.3	
28.0	25.7	29.0	26.3	17.4	
36.7	33.8	38.0	35.3	24.3	

As expected, the dull, black material reached the highest temperatures. The temperature difference between glossy and dull surfaces is about  $1.5^{\circ}\text{C}$ . The greatest difference in temperature was naturally between the dull, black surface and the glossy, white surface. The average difference was approx.  $4^{\circ}\text{C}$ .

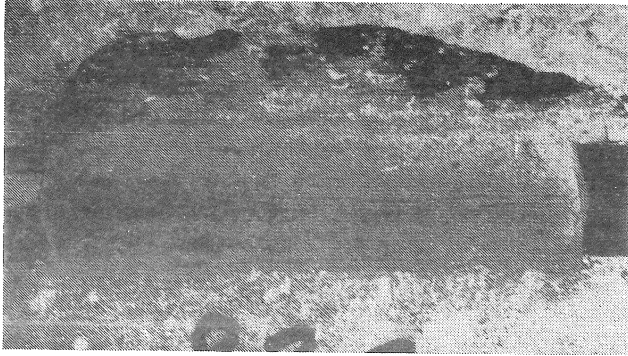


FIG 64 Provkropp av "Material 7" som utomhus-exponerats ca 7 månader. Max drag 7.7%, max tryck 17.8%.

*Specimen of "Material No 7" outdoor exposed for about 7 months. Maximum tension 7.7%, maximum compression 17.8%.*

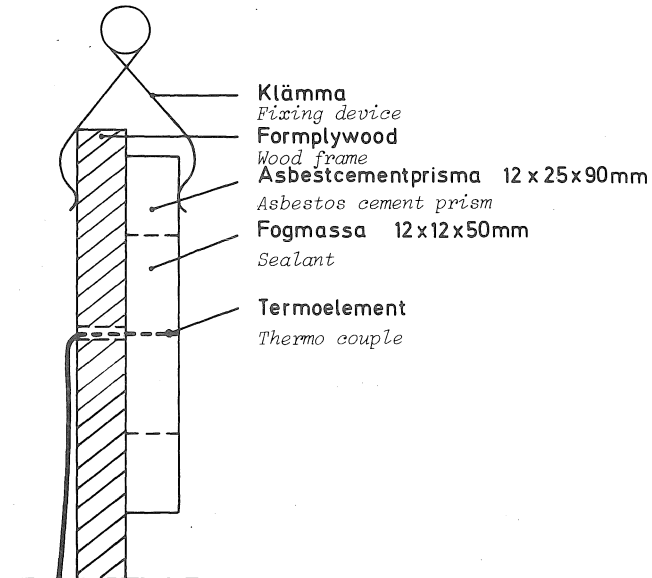


FIG 65 Mätning av temperaturer i olikfärgade fogmassor vid utomhusexponering.

*Measuring the temperatures in sealants of different colours when outdoor exposed.*

### 6.6.3 Stress-strain curves for material No. 1

Fig. 66 shows the stress-strain curves for material No. 1 after a varying degree of outdoor exposure. The hatched surface indicates the curves with the exposure times from 7 months to about 1 year. No significant difference could be seen between specimens exposed to varying degrees of deformation during the exposure, or between the type of element material (asbestos cement or aluminium). The curve applies to materials exposed for only 4 months, mainly during the cold season. The shape of the stress-strain curve deviates considerably from the other curves. This is probably due to the fact that the hardening is not yet completed.

In comparison to Fig. 19 the curve for storage in a normal climate corresponds extremely well to the curve in Fig. 66.

Also note that when disconnecting the specimens, they were mainly free from stress, i.e. the sealant had undergone plastic deformations and had become adapted to the new, forced joint width.

The variation in joint width can be understood as being a seasonally dependent part and a superimposed daily movement. The results obtained from the outdoor exposure of test joints and from investigations regarding the extent of the stress relaxation, Chapter 6.5, and Nimmermark & Olsson (1976) show that: Material No. 1 probably functions as a plastic material where the seasonal movements are concerned and as a mainly elastic material where the daily movements are concerned!

### 6.6.4 Stress-strain curves for Material No. 2

Fig. 67 shows the stress-strain curves for material No. 2 after 4 and 7 months of outdoor exposure (no more curves are available due to adhesive failure). An increase in hardness took place, which probably depended on continued curing. According to Fig. 23, the stress-strain curve

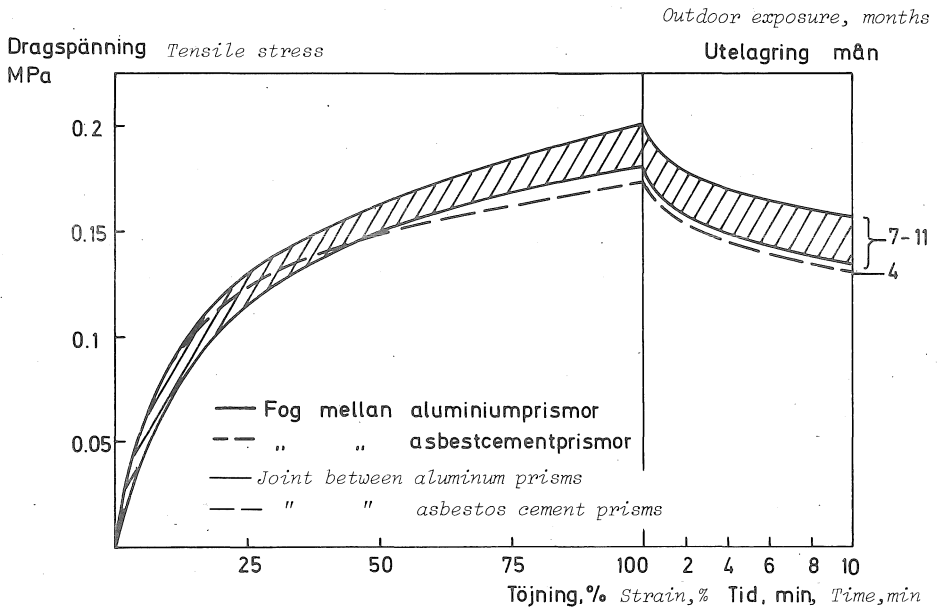


FIG 66 Arbetskurvor för "Material 1" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 1" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning *Tensile stress*  
MPa

Utelagring mån

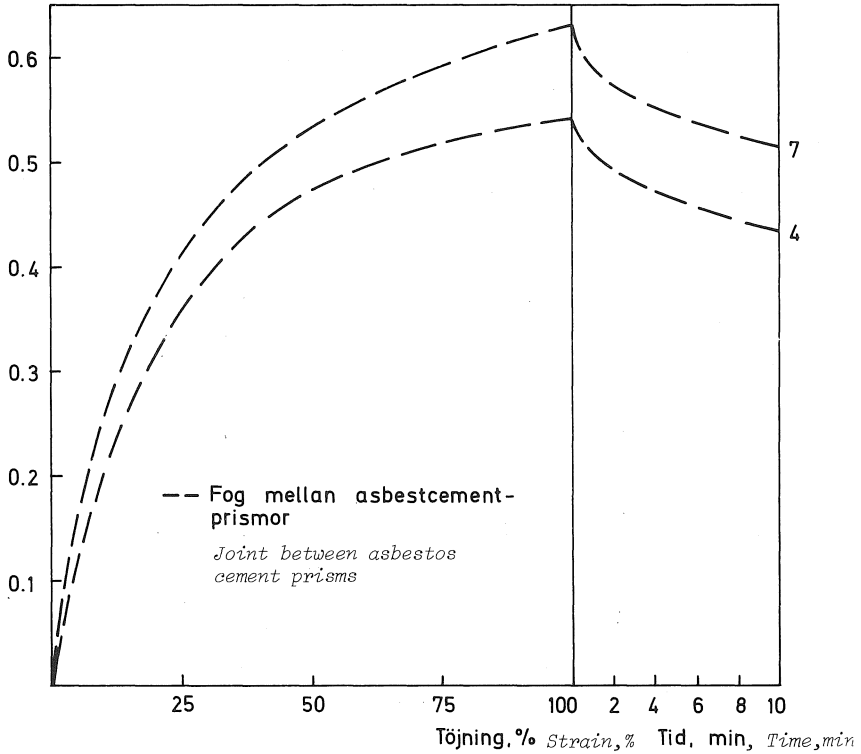


FIG 67 Arbetskurvor för "Material 2" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provnings temperatur: +2°C.

*Stress-strain curves for "Material No 2" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

for 7 months corresponds well to the stress-strain curve for a normally stored specimen.

The plastic deformation corresponding to the yearly joint movements is also valid for material No. 2!

#### 6.6.5 Stress-strain curves for material No. 3

Fig. 68 shows the stress-strain curves for material No. 3. The hatched surface indicates the stress-strain curves for materials with an exposure time of about 7 months with different degrees of deformation and different element materials. One can see that a continuing increase in hardening has taken place during 5 to 7 months of exposure.

However, Fig. 25 showed, that this increase also occurs when storing at  $+20^{\circ}\text{C}/50\% \text{ RH}$ . The increase in hardness has, however, been considerably quicker when storing outdoors. Even the curves for the 7 months material approach the curve for heat ageing at  $+70^{\circ}\text{C}$  in Fig. 25. The value of the tensile stress at 50% elongation ( $= \sigma_{50}$  MPa) in Fig. 68 is about 0.375 MPa. This value corresponds in Fig. 25 to about 10 days heat ageing at  $+70^{\circ}\text{C}$ . Using the same method of calculation as in Chapter 6.2.8 one finds that the heat ageing at  $+70^{\circ}\text{C}$  for  $1/20$  of the time results in about the same effect on the deformation resistance as outdoor exposure. However observe, that the curve in Fig. 25 has a very steep line at the beginning. This makes it necessary to compare the magnifying factor with the stress-strain curves which will later be determined after still further outdoor exposure. However, it is reasonable to suppose that the increase in hardness of the specimens which are stored outdoors is also at its greatest at the beginning. This means that, for the time being, the magnifying factor 20 will be valid as a good standard value.

This value is not acceptable when the specimens are new. In this case heat ageing at  $+40^{\circ}\text{C}$  has good resemblance.

Dragspänning *Tensile stress*  
MPa

Utlagring mån  
*Outdoor exposure, months*

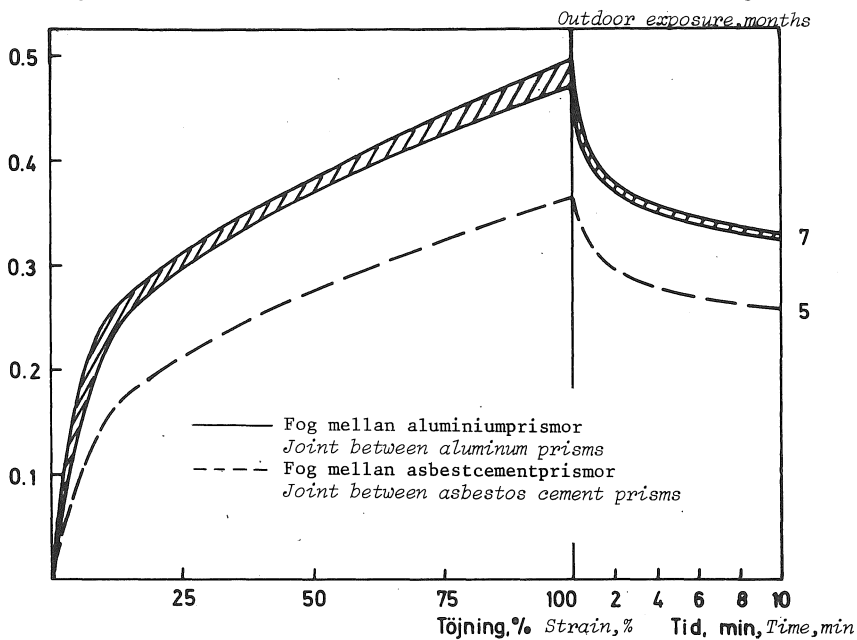


FIG 68 Arbetskurvor för "Material 3" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 3" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Dragspänning *Tensile stress*  
MPa

Utlagring mån.  
*Outdoor exposure, months*

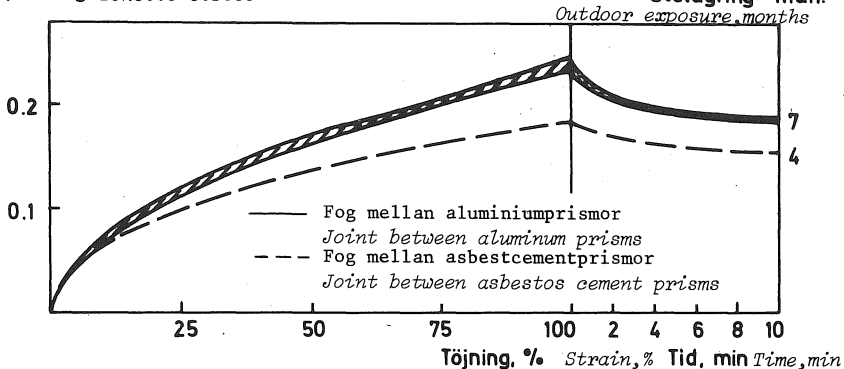


FIG 69 Arbetskurvor för "Material 4" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 4" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

Results from the outdoor exposure and the determinations of the degree of stress relaxation have so far shown that material No. 3 functions mainly as an elastic material, when the movement cycle of one year is studied.

#### 6.6.6 Stress-strain curves for material No. 4

The stress-strain curves can be seen in Fig. 69. A certain increase in hardness has taken place between 4 and 7 months' exposure. The difference between the varying degree of deformation or between different element materials cannot be proved (the dispersion after 7 months' exposure is very small).

When comparing normal and heat stored curves in Fig. 28 one can see that the outdoor exposed specimens are considerably softer! One of the reasons for this could be a slower curing process. In spite of this, material No. 4 functions, from a deformation point of view, mainly as an elastic material both where "short" and "long" movements are concerned.

#### 6.6.7 Stress-strain curves for material No. 5

Material No. 5 is a completely plastic material. The stress-strain curves for the outdoor exposed specimens are shown in Fig. 70. Because of the material's plastic properties the joint profile showed rather deep wrinkles, which makes accurate dimensions difficult to measure. This caused rather a great dispersion of the measured results.

The comparison with Fig. 30 shows that heat ageing at  $+70^{\circ}\text{C}$  caused completely different effects than those obtained during outdoor exposure. 56 days at  $+70^{\circ}\text{C}$  seems to result in much too hard an ageing.

#### 6.6.8 Stress-strain curves for material No. 6

Material No. 6 is a mainly plastic material. However, the elastic properties increase slowly with time. This plastic characteristic has caused wrinkling in the joint profile



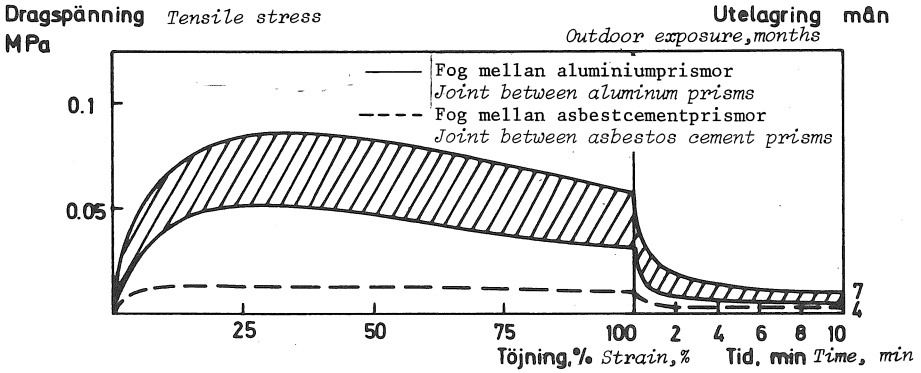


FIG 70 Arbetskurvor för "Material 5" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provingstemperatur: +2°C.

Stress-strain curves for "Material No 5" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.

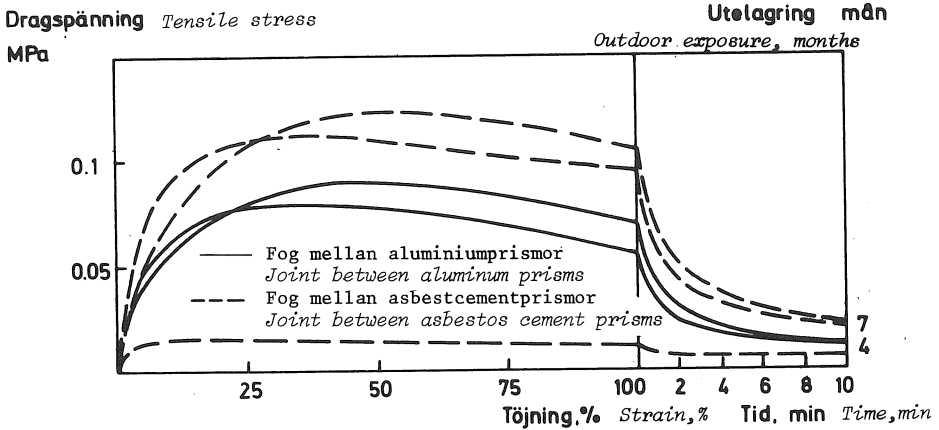


FIG 71 Arbetskurvor för "Material 6" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provingstemperatur: +2°C.

Stress-strain curves for "Material No 6" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.

(Fig. 63). This makes it difficult to measure cross sections. To some extent this explains the dispersion in Fig. 71, where the stress-strain curves are presented. Varying degrees of deformation have so far been of no significance where the deformation properties of the material are concerned. On the other hand, the alkalinity or/and the greater water absorption by the asbestos cement prisms have to some extent increased the hardness of the material (dashed curves). This corresponds favourably to Fig. 47, where this tendency also appeared for the specimens which were dried after alkaline/water storage.

Fig. 33 shows that when compared to Fig. 71 the heat ageing at  $+70^{\circ}\text{C}$  increase the deformation resistance of the material very considerably. This ageing also causes some undesirable effects such as blisters and cracks. This makes it more suitable, where material No. 6 is concerned with heat ageing at a lower temperature than  $+70^{\circ}\text{C}$ .

#### 6.6.9 Stress-strain curves for material No. 7

The stress-strain curves for material No. 7, after outdoor exposure, are shown in Fig. 72. The curves only concern joints between asbestos cement prisms. When using aluminium, all the specimens failed showed adhesively failure during the test. On the other hand, when asbestos cement was used, the adhesion was excellent. This water dispersed sealant obviously requires a fairly absorbant base in order to obtain a good adhesive capacity or alternatively, primer treatment.

However, by measuring the hardness in  $^{\circ}\text{Shore A}$  it could be found that the sealant between the asbestos cement prisms had a considerably higher value,  $50^{\circ}\text{Shore A}$  compared to about 28 for the other specimens. The reason for this is difficult to figure out. It is probable that the alkalinity affected the sealant in some way.

Fig. 72 shows an increase in deformation resistance with time. With more comparison material it should be possible to accelerate the increase in hardness by means of heat ageing (Fig. 36).

Dragspänning *Tensile stress*

Utelagring mån

MPa

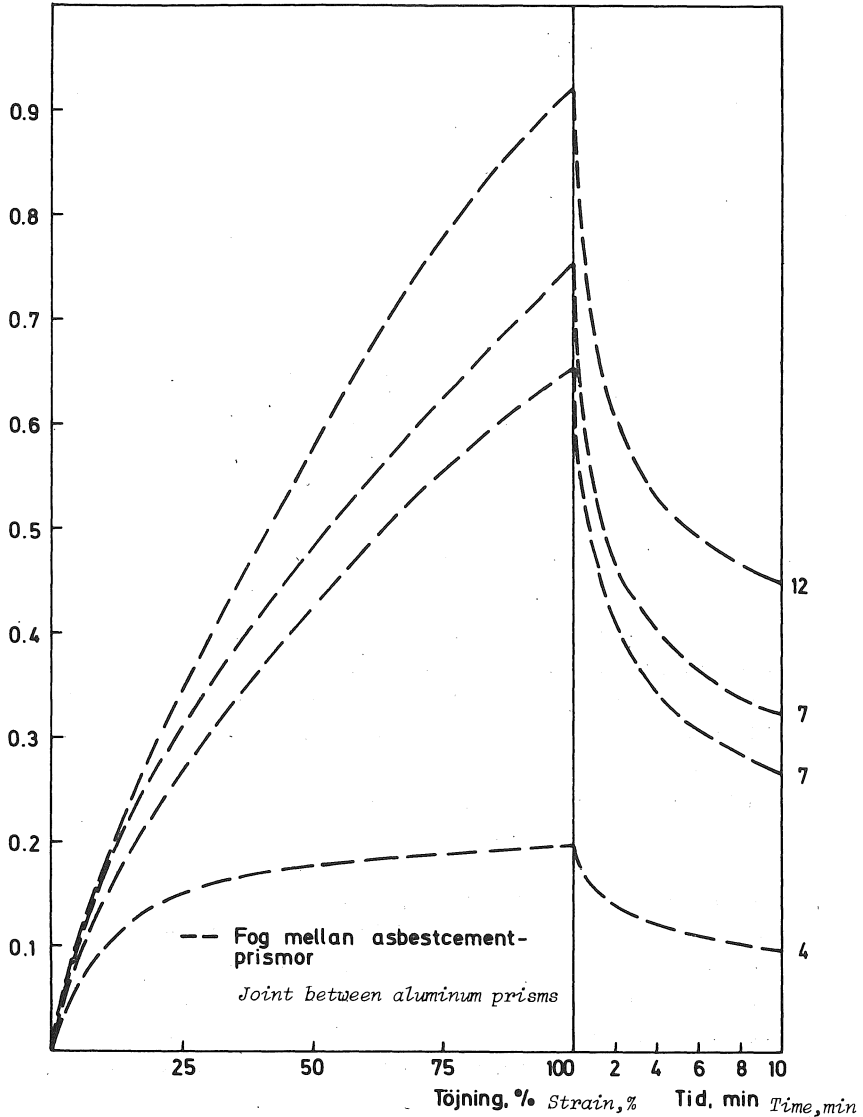
*Outdoor exposure, months*

FIG 72 Arbetskurvor för "Material 7" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provningsstemperatur: +2°C.

*Stress-strain curves for "Material No 7" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.*

The magnitude of the forced deformation during outdoor exposure has, to a certain extent, affected the curves in Fig. 72. The lower of the two curves, for 7 months' outdoor storage, concerns materials exposed to the largest deformation, i.e. elongation 7.7% and compression 17.8% (this also exceeded the value of a total 15%, stated by the manufacturer).

Overloading caused wrinkling in the joint and when the stress-strain curve was determined there was a partial cohesion cracking in this section.

Material No. 7 acted as a plastic material where movements varying with the season were concerned and partly as an elastic material where daily movements were concerned.

#### 6.6.10 Stress-strain curves for material No. 8

Fig. 73 shows the stress-strain curves for material No.8. As the material is plastic regarding the ability to absorb deformation and also maintained a very soft consistency for a long time, it was difficult to measure the dimensions. This made the dispersion of the results wide. No definite conclusions could be drawn from the curves in Fig. 73, for example where the movement absorbance or the alkaline sensitivity were concerned.

According to Fig. 39 the comparison between the heat aged specimens show that it should be possible to accelerate the ageing of the material, for example by means of some type of heat ageing. Furthermore, if one connects the ageing of the material with the skin changes due to time, and there are good reasons for this, one can see that Fig. 41 and the discussions regarding this Fig have already shown the connection between the accelerating heat ageing and the natural ageing.

The practical use of material No. 8 shows that extremely good experience exists. It has been used in a great amount

Outdoor exposure, months

Dragspänning  
MPa

Tensile stress

Utelagring mån.

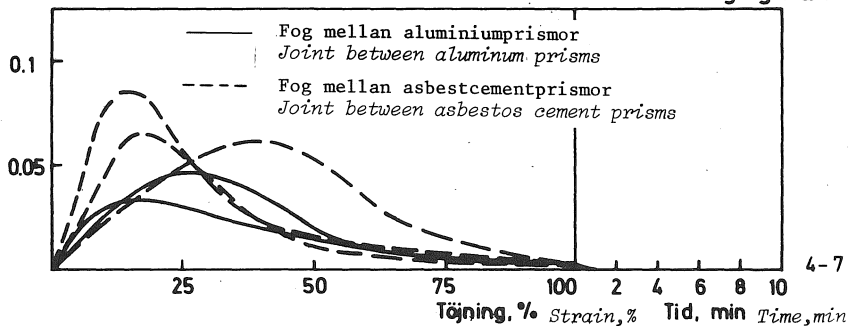


FIG 73 Arbetskurvor för "Material 8" efter olika lång tids utomhusexponering. Deformationshastighet: 1.5 mm/min. Provnings temperatur: +2°C.

Stress-strain curves for "Material No 8" after varying time of outdoor exposition. Deformation speed: 1.5 mm/min. Testing temperature: +2°C.

Volymminskning  
%

Volume loss

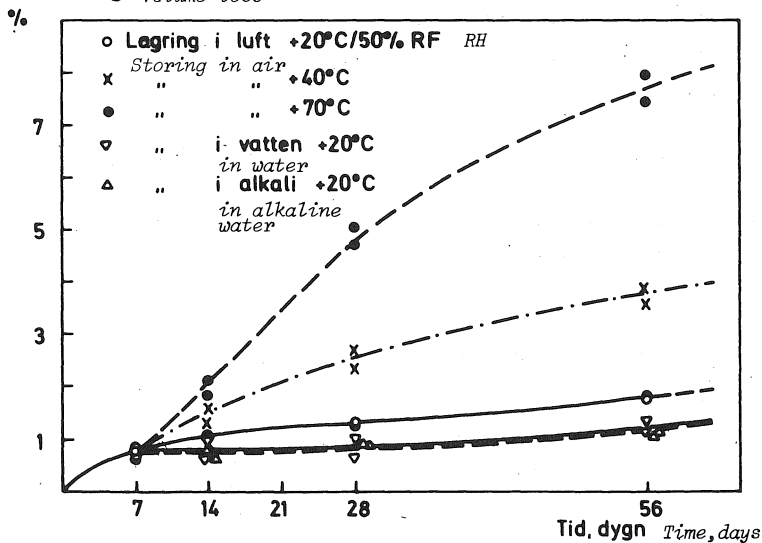


FIG 74 Volymminskning (krympning) som funktion av lagringstid i olika klimat för "Material 1".

Shrinkage as a function of storing time in different types of climate for "Material No 1".

of constructions for more than 10 years, and most of the joints are still in an excellent condition. This is surprising! Experience of the so called oil based sealants has otherwise shown that they dry out in quite a short time, shrink, and result in a leaking joint.

The reason why material No. 8 is so good could possibly be as follows: the sealant shows a shrinkage of about 12-15% by volume (Chapter 6.7). This makes the front surface of the joint concave. As the ability of the joint to absorb movements is dependent on the ductility of the skin, this new (due to shrinkage) extended surface lead to the fact that the movement absorbing part of the sealant extends. During following elongation and compression the inner, still soft, part of the sealant only serves as a support for the skin. Because of the mainly plastic characteristic of the skin, a certain wrinkling occurs in connection with the movements.

A condition is that the skin formation is not too fast (can be checked according to Fig. 41), and that the skin is tough.

The disadvantages with the way the oil based sealant worked were the occurring wrinkling (was sometimes aesthetically disturbing) and the limited ability to resist mechanical attacks (poking holes etc.).

#### 6.7 Determining weight and volume-losses when storing in different climates

Nimmermakr & Olsson (1976) have determined the magnitude of the shrinkage and the decrease in weight in materials Nos. 1-8 when stored under different conditions (See Chapter 4.3). The different types of storage were:

- +20°C/50% RH
- +40°C
- +70°C
- water +20°C
- alkali +20°C pH approx 11
- UV chamber +20°C

Storage in different "climates" was usually started after storage for 7 days at +20°C/50% RH. However, the heat storage at +40°C and +70°C was also started after 21 days in a normal climate, 20/50.

Some examples of the results will be presented here. Fig. 74 shows the shrinkage as a function of time for material No. 1. The heat storage considerably increases the shrinkage compared to storage at 20/50. Reasons for the shrinkage could, for example, be loss of solvent, softener and certain non-reacted groups of polymer. An increased degree of curing due to the heat storage could also, to some extent, cause a contraction.

Storage in water and alkali led to an absorption of the water and an expansion that in some way counteracted the shrinkage that took place at 20/50.

There are great differences between different makes of the same type of sealant (the same polymer), for example where shrinkage and thermal stability are concerned. Fig. 75 shows heat aged shrink specimens of polysulphide sealants. The specimen on the left is material No. 1 and the one on the right is a material of another make.

Fig. 76 shows the effect of heat storage on the shrinkage of material No. 3. The effect of the time for the start of the heat storage is also shown. The shrinkage after 56 days will consequently be greater when stored at +70°C after first 21 days at 20/50! However, if the decrease in weight is considered as being a function of time, Fig. 77, one can see that it is dependent on the time when the storage starts. This means that closed pores are formed when the

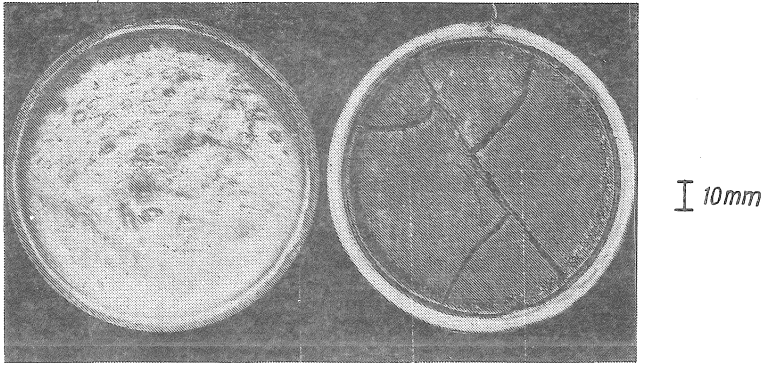


FIG 75 Bestämning av vikt- och volymminskning för två olika polysulfidbaserade fogmassor vid lagring i +70°C. Vänster provkropp är "Material 1", den högra av annat fabrikat.

*Measuring of weight loss and shrinkage of two different polysulphide based sealants when stored at +70°C. The specimen to the left is "Material No 1", the one to the right of another make.*

Volymminskning, Volume loss

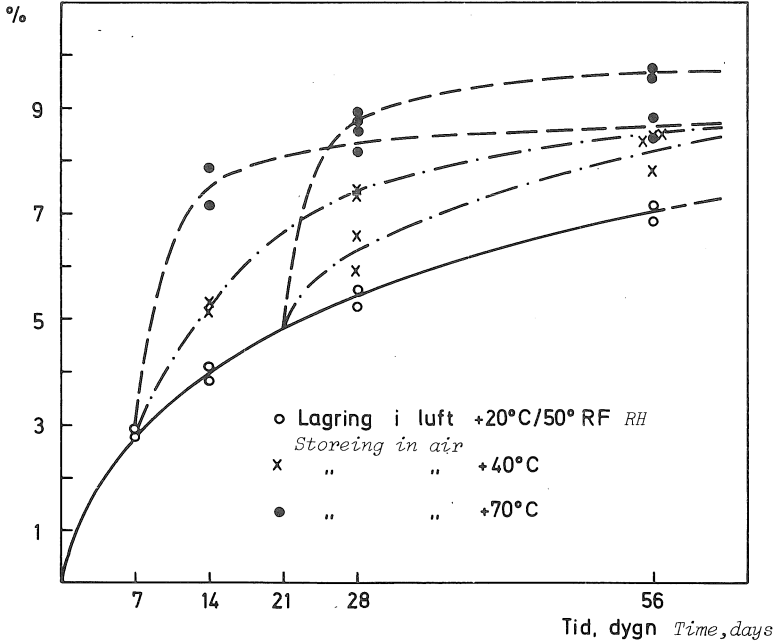


FIG 76 Volymminskning (krympning) som funktion av lagringstid i olika klimat för "Material 3".

*Shrinkage as a function of storing time in different types of climate for "Material No 3".*



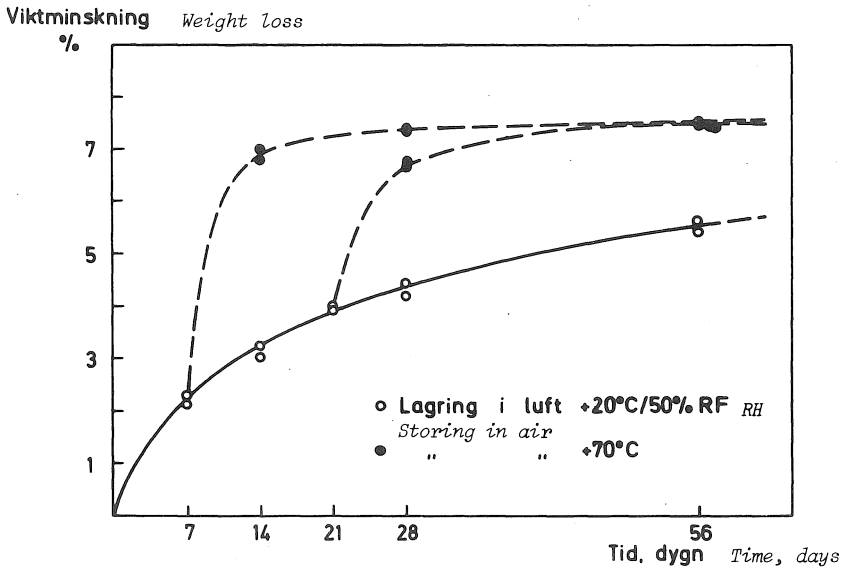


FIG 77 Viktminskning som funktion av lagringstid i olika klimat för "Material 3".

*Weight loss as a function of storing time in different types of climate for "Material No 3".*

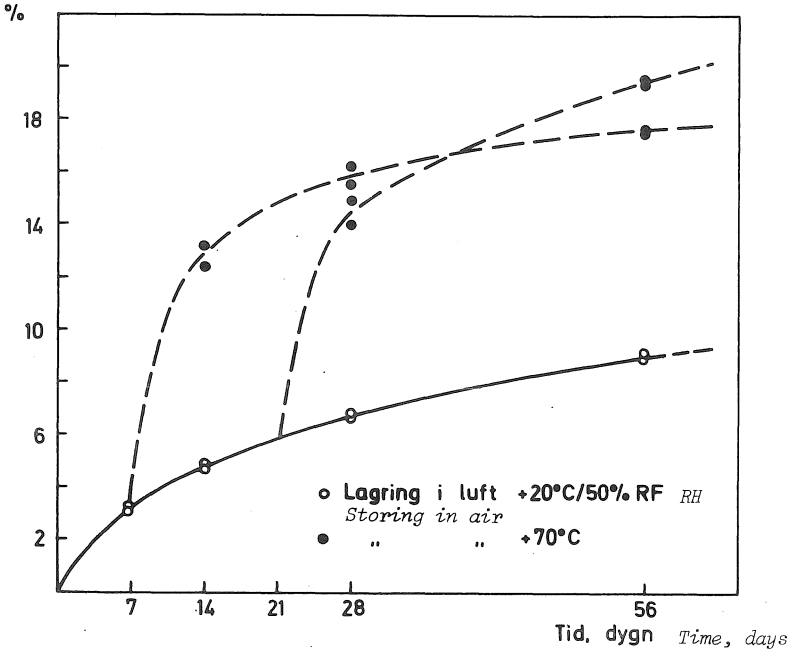


FIG 78 Volymminskning (krympning) som funktion av lagringstid i olika klimat för "Material 6".

*Shrinkage as a function of storing time in different types of climate for "Material No 6".*

Viktninskning *Weight loss*

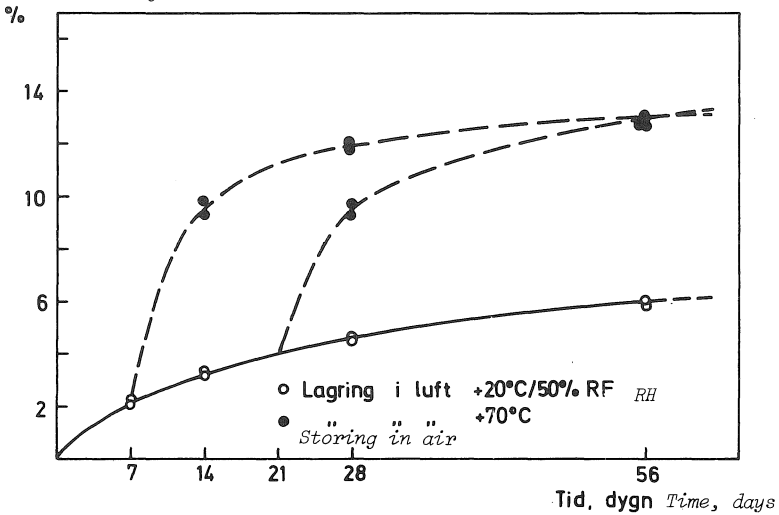


FIG 79 Viktninskning som funktion av lagringstid i olika klimat för "Material 6".

*Weight loss as a function of storing time in different types of climate for "Material No 6".*

heat ageing starts early, which results in an, apparent, smaller shrinkage. The pore formation is mainly connected with a loss of solvents.

In Figs. 78-79 the same condition is shown for material No. 6, i.e. the magnitude of the shrinkage is dependent on how soon the heat ageing is started.

Tables 7 and 8 summarize the results from the determinations of the decrease in volume and weight of all the materials. Table 7 gives information concerning the shrinkage which is stated by the respective manufacturer. As is apparent, the information sometimes differs considerably from the values found by Nimmermark & Olsson (1976).

By using the values from the determinations of the decrease in weight and volume one can calculate the average densities for the substance(s) which disappeared in different cases. Table 9 shows a comparison of these values.

Table 9. Calculated densities ( $\text{g/cm}^3$ ) of lost substances in different climate

Material No.	Climate			
	56 days $+20^{\circ}\text{C}/50\%RH$	7 days $20/50$ 49 days $+40^{\circ}\text{C}$	7 days $20/50$ 49 days $+70^{\circ}\text{C}$	21 days $20/50$ 35 days $+70^{\circ}\text{C}$
1	0.22	0.23	0.24	0.24
2	0.31	0.27	0.64	0.63
3	0.92	0.93	1.02	0.91
4	0.73	0.75	0.59	0.65
5	0.87	0.88	1.13	1.09
6	0.93	0.91	1.04	0.93
7	1.00	0.99	1.07	1.02
8	0.55	0.63	0.82	0.79

Table 7. Shrinkage (vol-%) after 56 days in different climate

Material No.	Climate								Shrinkage according to the producer
	+20°C/ 50% RH	+40°C		+70°C		Water	Alkali	UV	
		7 days in 20/50	21 days in 20/50	7 days in 20/50	21 days in 20/50	+20°C	+20°C	+20°C	
1	1.8	3.8	3.4	7.7	7.0	1.2	1.1	1.8	"negligible"
2	2.0	4.0	3.4	4.0	3.7	0.9	0.8	1.8	"no shrinkage"
3	7.0	8.6	8.2	8.6	9.7	1.8	3.4	7.7	no information
4	4.3	5.9	5.6	8.4	7.8	0	0.2	4.2	"7%"
5	8.2	13.3	10.1	15.1	15.4	8.6	5.8	8.1	"moderate"
6	9.1	13.2	13.5	17.6	19.6	2.8	2.8	6.9	"10%"
7	22.0	23.2	23.4	22.8	24.6	-	-	22.0	no information
8	10.4	14.8	14.0	17.5*	17.9	-21.3	-14.2	9.1	"15%"

\* only one specimen

Table 8. Weight loss (weight-%) after 56 days in different climate

Material No.	Climate							
	+20°C/ 50% RH	+40°C		+70°C		Water +20°C	Alkali +20°C	UV +20°C
		7 days in 20/50	21 days in 20/50	7 days in 20/50	21 days in 20/50			
1	0.3	0.6	0.5	1.2	1.1	-0.3	-0.3	0.3
2	0.4	0.7	0.7	1.7	1.6	-0.4	-0.3	0.4
3	5.5	6.8	6.7	7.5	7.5	1.1	2.4	5.8
4	2.6	3.7	3.6	4.1	4.2	-1.8	-0.9	2.6
5	5.3	8.6	6.6	12.6	12.4	5.2	3.5	4.7
6	6.0	8.5	8.7	13.0	12.8	1.5	2.0	4.2
7	14.7	15.3	15.7	16.3	16.7	-	-	14.2
8	3.8	6.2	5.9	9.5*	9.4	-17.7	-12.0	2.8

\* only one specimen

For materials Nos. 1 and 2 very low density values were obtained. This could probably be partly explained by the fact that chemical hardening is followed by a contraction.

For material No. 3 the calculated density corresponds relatively well to the density of the solvent release. However, the early started heat ageing at +70°C caused a pore formation which made the density too high. This was also the case for materials Nos. 5, 6, 7 and to some extent, material No. 8.

### 6.8 Determination of shrinkage stresses

Chapter 6.7 shows that the shrinkage is quite considerable for certain materials. A series of tests was completed where the aim was to measure the shrinkage stresses that might occur when different sealants cure/dry. Materials Nos. 1, 3, 7 and 8 were tested. The temperature during the test periods was +21 to +23°C.

By using the equipment according to Fig. 15 the shrinkage stresses were studied for max 31 days. The results are shown in Table 10. As it was a question of measuring very small stresses there was some uncertainty regarding the measurements. The equipment is sensitive to changes in temperature etc. Therefore an approximation of the measuring accuracy was made. This resulted in a general addition to the measured values according to Table 10. The values of the shrinkage stated in the Table were taken from Chapter 6.7 or from Nimmermark & Olsson (1976).

Table 10. Results from measurements of shrinkage stresses

Material No.	Time days	Shrinkage vol-%	Measured stress MPa	Maximum error in measurements MPa	Max. shrinkage stress MPa
1	12	1	0	$2 \cdot 10^{-3}$	$2 \cdot 10^{-3}$
3	27	5.5	$3 \cdot 10^{-3}$	"	$5 \cdot 10^{-3}$
7	31	21	$2 \cdot 10^{-3}$	"	$4 \cdot 10^{-3}$

With the help of the equipment according to Fig. 5 the same measurements were made for materials Nos. 3, 7 and 8. The shrinkage was measured for max. 46 days.

For material No. 3 the shrinkage stress after 46 days was about  $12 \cdot 10^{-3}$  MPa. The shrinkage was then approx. 6.5% by volume. After 27 days the corresponding values were about  $6 \cdot 10^{-3}$  MPa and 5.5% by volume respectively. The latter value corresponded well with the value in Table 10.

For material No. 7 the shrinkage stress after 46 days was  $< 5 \cdot 10^{-3}$  MPa. The shrinkage was then about 22% by volume. This value also corresponded well to the one in Table 10.

Material No. 8 showed no noticeable shrinkage stress after 46 days. With the measuring accuracy as a limiting factor, this meant that the stress was less than  $10^{-4}$  MPa.

The results obtained from the measurements consequently show that the shrinkage stresses which occur because of shrinkage are very small. For material No. 3 the measured shrinkage stress could be compared with the tensile stresses occurring in the material at a very low elongation (Fig. 27). At approx. 1.5% deformation the tensile stress was  $12 \cdot 10^{-3}$  MPa for the heat aged material in Fig. 27.

Corresponding comparisons for materials Nos. 1 and 7, Figs. 22 and 37 respectively, resulted in still smaller deformation.

The reason for such small shrinkage stresses is that the materials were still too plastic when the major part of the shrinkage took place.

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