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## Alkali-silica reactions in Scania, Sweden : a moisture problem causing pop-outs in concrete floors

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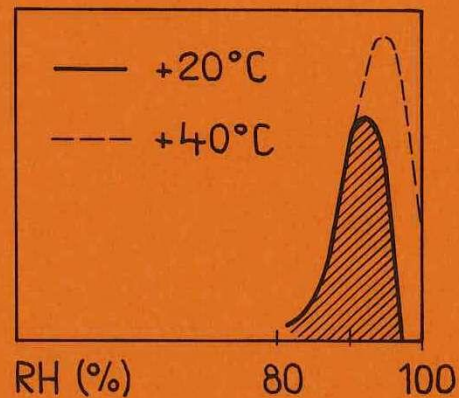
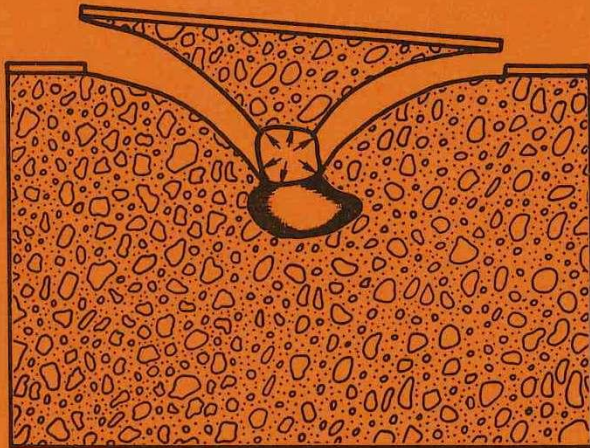
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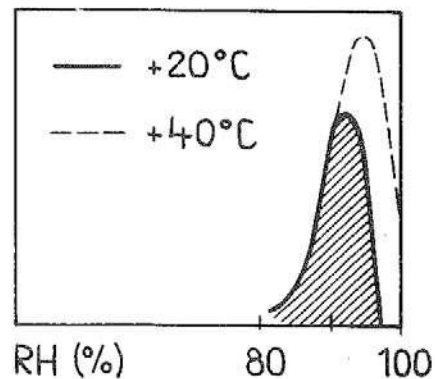
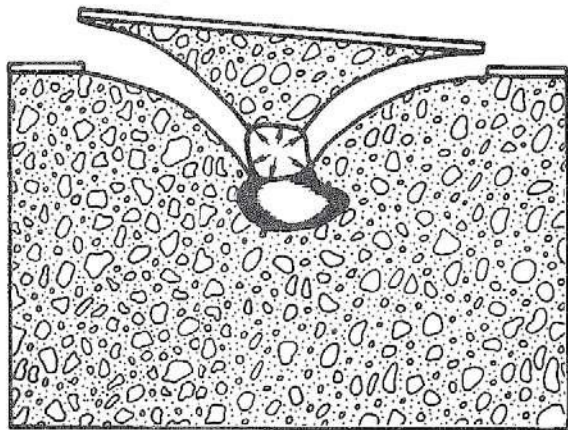


# ALKALI-SILICA REACTIONS IN SCANIA, SWEDEN

- A MOISTURE PROBLEM CAUSING POP-OUTS  
IN CONCRETE FLOORS

LARS-OLOF NILSSON

OLOF PETERSON



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## PREFACE

The Division of Building Materials at the Lund Institute of Technology has carried out research and investigations regarding moisture problems since the division started in 1964. When alkali-silica reactions suddenly began to occur in 1975 it was in the form of pop-outs mainly on concrete floors on the ground. That particular structure had been studied for many years by the division and when a working group was formed by CEMENTA AB in 1978 to study alkali-silica reactions in Scania (the southernmost part of Sweden), representatives from the division acted as chairman and secretary of the group.

After the working group had completed the work, and outlined some preventive actions based on the knowledge at that time, a research project was started in 1980 with the following objectives:

To explain the cause and mechanism of the damages, i.e. the pop-outs.

To investigate the participation of moisture in alkali-silica reactions.

To develop test methods for concrete

To test some potential inhibitors.

This report is the Final Report of this research project and briefly summarizes the present knowledge, presents the "Scanian problems", describes the performance of the experiments and their results, gives some conclusions regarding the objectives.

#### ACKNOWLEDGEMENT

I greatly acknowledge the assistance of the staff at the Division of Building Materials, Lund Institute of Technology, in carrying out the experimental work and preparing the report.

Olof Peterson has performed the experiments described in part 3.5 and also written that part. He has successfully contributed to the continued work in the other parts of the project and thoroughly examined the report, particularly from a cement-chemical point of view.

The research was financed by a grant from CEMENTA AB, Sweden and the National Swedish Board for Technical Development.

LUND, August 1983

Lars-Olof Nilsson

Project leader

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#### SUMMARY

Problems due to alkali-silica reactions in Sweden are new and coincide with the introduction of a new cement in the south of Scania where an ordinary concrete aggregate contains reactive grains of opaline flint and sandstone. The earlier used cement, however, was a low-alkali cement and no alkali-silica reactions were known before.

The problems are known only as pop-outs on certain structures and appear to be a definite moisture problem as pop-outs have never been found on outdoor structures even when they are wet. Problems have only been reported on "moist indoor structures" that have had a certain history of moisture changes. The most frequently affected structure is concrete slabs on the ground. More than 9 out of 10 problems with pop-outs due to ASR are to be found on such a structure.

The moisture effects on the alkali-silica reaction have been studied by measurements of moisture distributions on some damaged structures and by several laboratory experiments with varying climatic conditions. Relative humidities of the concrete close to 90 % RH, at different temperatures, have been shown to be "pessimal", and cause the most pop-outs.

A test method for pop-outs has been developed using the "moisture history" as an aid to reproduce the damages in the laboratory.

The effect of some potential inhibitors has been studied in the test method, and has produced promising results.



## SAMMANFATTNING

Problemen med alkali-kiselsyrareaktioner i Sverige ("AKRIS") är nya och började uppträda då ett cement med högre alkalihalt ersatte det tidigare lågalkaliecementet som använts i södra Skåne, där betonggrus normalt innehåller alkalireaktiva korn av opalflinta och opalsandsten. Problemen är kända bara som "pop-outs" på vissa konstruktioner och visar sig vara ett speciellt fuktproblem eftersom pop-outs aldrig har rapporterats förekomma på utomhuskonstruktioner även om de är blöta. Bara på "fuktiga inomhuskonstruktioner", som har haft en speciell fukthistoria, har pop-outs uppkommit. Den konstruktion som oftast drabbats är betonggolvet på mark med täta golvbeläggningar. Storleksordningen 90 % av problemen har uppträtt på sådana golv.

Inverkan av fukt på alkali-kiselsyrareaktioner har studerats i en del skadefall genom mätning av fuktfördelningar och genom åtskilliga laboratorieförsök med varierande klimatförhållanden. En relativ fuktighet nära 90 %, vid olika temperaturer, har påvisats vara "pessimal", dvs orsakar flest pop-outs.

En provningsmetod för pop-outs har utvecklats som innebär att man använder fukthistorien som ett verktyg att reproducera skadorna vid laboratorieförsök.

Inverkan av några potentiellt förebyggande medel (inhibitorer) har testats med provningsmetoden och ett par visar lovande resultat.

## 1 A SHORT INTRODUCTION TO ALKALI-SILICA REACTIONS

Damages to concrete structures due to alkali-silica reactions are the result of expansion of the reaction product, the alkali-silicate gel, when it absorbs water. The gel was first identified in the USA by Stanton (1940) to be the product of chemical reactions between the alkalis from the cement and the silica in some concrete aggregates.

The swelling of the gel may cause cracks in a concrete structure. In unrestrained concrete with a high content of evenly distributed reactive grains the cracks form a special pattern often referred to as "mapcracking", FIG 1.1, where the interior of the structure expands and mapcracks are visible on the surface. The situation of a big reactive grain is often found where three cracks meet in one point.

If the reactive grains are few, swelling of the gel from a grain close to the surface may produce a "pop-out", FIG 1.2. A piece of the concrete surface is pushed out by the gel and the grain or the gel is visible at the bottom of the crater.

For the alkali-silica reactions to occur in a damaging way, four conditions must be complied with:

- o The pore solution must have a high content of alkaline metal hydroxide, originating from high-alkali cement or from an external source
- o The aggregate must contain alkali-soluble silica
- o A sufficient amount of moisture must be suitably distributed in space and time
- o The concrete composition, the composition of the ingredients and the internal and external climatic conditions must be close to "pessimum conditions"

These factors are discussed briefly below.

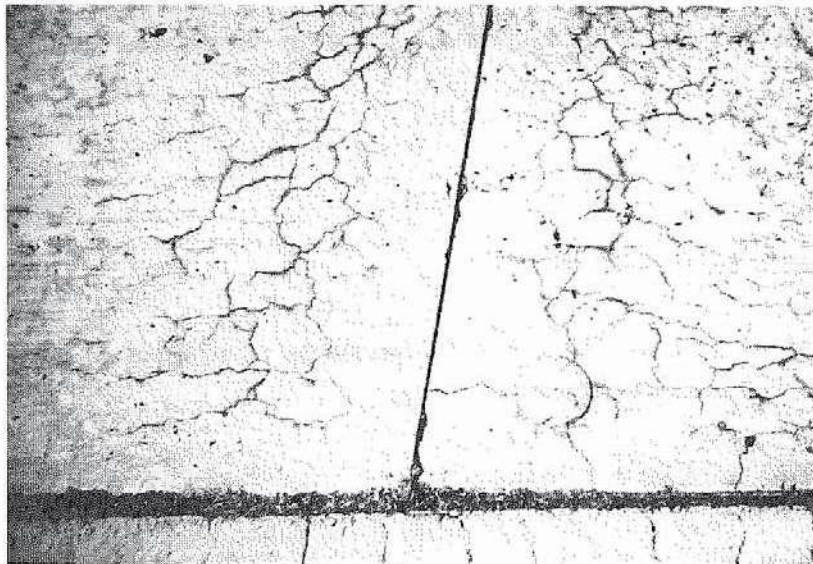
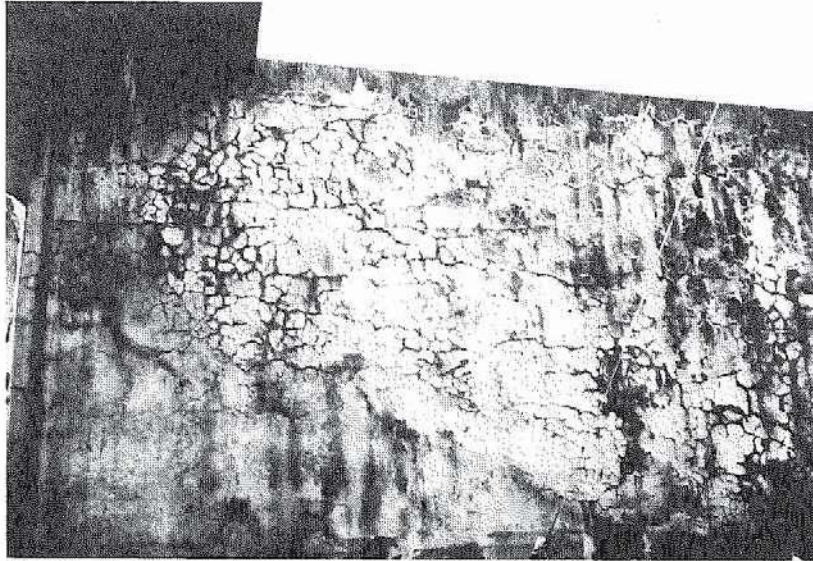


FIG 1.1 Mapcracking on the Landsdowne road bridge (above) and the concrete road N2/1 (below), outside Cape Town in South Africa.

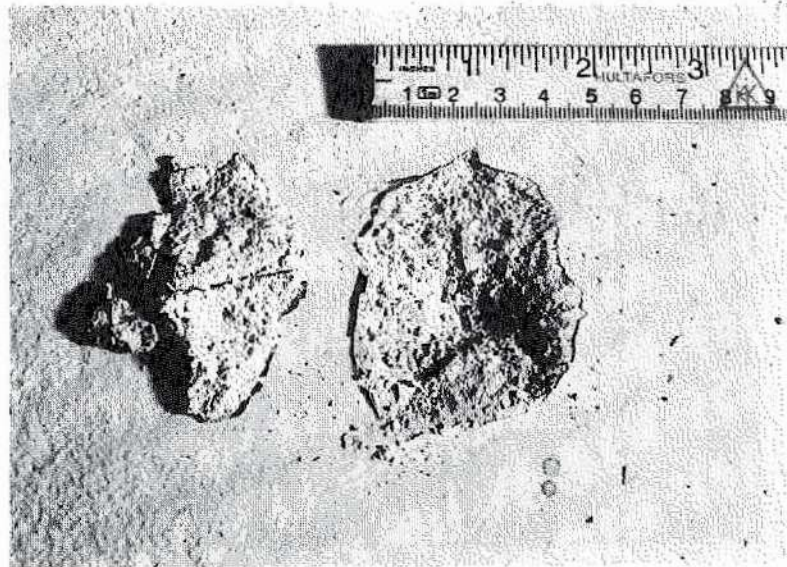


FIG 1.2 A pop-out on a basement floor in Malmö, Scania

### 1.1 Alkalis in the pore solution

The amount of alkalis ( $\text{Na}^+$ ,  $\text{K}^+$ ) in cement is usually expressed in per cent by weight as equivalent sodium oxide:

$$\% \text{Na}_2\text{O}_{2 \text{ eq}} = \% \text{Na}_2\text{O} + 0.658 \times \% \text{K}_2\text{O}$$

(In a cement, the alkalis are never to be found as oxides.)

The alkalis in a cement are always derived from the raw materials, most commonly from the minerals, which carry the aluminium. Previously, when fuels were cheap, the wet process for burning the clinker was common, and some of the alkalis were allowed to escape with the kiln dust.

Today, the attempts to save energy have led to the development of new types of cement kilns where most of the dust is fed back to the burning process together with most of the alkalis. So, modern Swedish cements have an average equivalent alkali content of approximately 1.0 %, which is to be

classified as "medium alkali cements". "Low-alkali cements" have an alkali content of below 0.6 %, this limit being regarded as a threshold for expansive alkali-silica reactions.

Attempts to find preventive measures against the alkali-silica reaction can consequently, in two ways, be regarded as attempts to save energy:

1. It would take more fuel to manufacture a low-alkali cement
2. Some cement will be saved, if ASR-inhibitors can be used.

Most of the water-soluble alkalis in cement are present as sulphates and dissolve rapidly. TABLE I shows an example of the ionic concentration of the pore solution.

TABLE I. Analysis of pore solutions from Portland cement pastes,  
 $\text{Na}_2\text{O} = 1.2$ , Page & Vennessland (1983)

Curing time (days)	Ionic concentration (moles per litre)					pH
	Na <sup>+</sup>	K <sup>+</sup>	Ca <sup>2+</sup>	OH <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	
7	0.26	0.61	0.001	0.79	0.02	13.9
28	0.27	0.63	0.001	0.83	0.03	13.9
56	0.33	0.70	0.003	0.84	0.04	13.9
84	0.32	0.64	0.002	0.74	0.03	13.9

In a few days the concentration of Ca<sup>2+</sup> has been reduced to almost zero and the hydroxide concentration is governed by the amount of dissolved alkalis. After a few days the concentration of alkalis is almost constant and approximately equal to the concentration of hydroxide. The small increase during the first two months may be explained by the continuing dissolution of alkalis in the cement clinker liberated by the hydration. The sealed curing should also cause some increase in concentration as the evaporable water content in the pores decreases by self-dessication and consequently the amount of solvent is decreased.

It is not the alkalis themselves, or alkaline metal ions, that attack the soluble silica in the aggregate grains, but the hydroxyl ions ( $\text{OH}^-$ ). All forms of water soluble alkalis in cement will change to alkaline metal hydroxide during the first few days hydration (TABLE I).

In the absence of alkalis the pore solution would be a saturated solution of  $\text{Ca}(\text{OH})_2$  (approx. 0.04 moles of  $\text{OH}^-$  and 0.02 moles of  $\text{Ca}^{2+}$  per liter). If some of the silica is sufficiently reactive to be attacked by such a dilute  $\text{OH}^-$  solution, the product can be nothing else than a calcium silicate hydrate, which is the same type of material that gives hardened concrete its strength.

TABLE I, Page & Vennessland (1983), gives a concentration of hydroxide ions of about 0.8 moles per litre, corresponding to a pH of 13.9. The solubility product of calcium hydroxide ought to limit the concentration of calcium ions to  $5 \times 10^{-5}$  moles per litre. Factors, such as the high ionic strength of the pore liquid, admit the somewhat higher level in the Table. The low availability of calcium ions is probably of more importance for the formation of alkaline metal silicate gel than the high concentration of hydroxide ions.

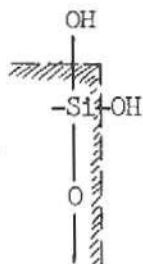
The addition of pozzolans, such as silica-fume, fly-ash etc, causes a reduction in alkalinity of the pore solution simply because there will be a rapid "alkali-silica-fume reaction" that consumes some of the alkalis. An example by Page & Vennessland (1983) shows a decrease in pH from 13.2 to 12.0, after two to three months of sealed curing, when 30 % of the cement is replaced by silica-fume.

A concrete structure may receive additional alkalis from sea water and sodium chloride as de-icing salt. Using a low-alkali cement to avoid alkali-silica reactions is not always safe in such environments. Special attention must be paid to structures where NaCl is a natural ingredient in the environment. The exact way for sodium or potassium chloride to promote ASR is under discussion.

## 1.2 Silica in the aggregate

A reactive siliceous aggregate contains alkali-soluble silica. The ability of silica in an aggregate to be attacked by an alkaline pore solution in concrete or mortar is, according to Krogh (1975), dependent on the amount of broken bonds in the silica lattice.

In the following, a typical silicon atom in a reactive mineral will be symbolized according to the formula:



With two of the bonds  
the atom is kept on its  
place in the lattice.

The remaining two bonds  
are hydrated.

Macrocrystalline silica, such as quartz, is usually not reactive at all, but amorphous silica (opal) is highly reactive. Microcrystalline silica, e.g. chalcedony, has a reactivity somewhere between these two extreme cases.

Opal and chalcedony are the main constituents of flint and are combined in different ways and proportions in different grains of flint. The dense part of flint usually contains chalcedony. The porous part at the surface of the dense flint and in inclusions usually contains opal. In the sand fractions, single flint grains often consist mainly of one of these components.

Opal can also be found in sandstones. Opal is to some extent a cementitious mineral in opaline sandstone, which is a highly reactive porous aggregate with opal in the pore system.

Grains containing reactive minerals vary in reactivity depending on the microstructure of the minerals and the pore system of the grain. The particle size is also a decisive parameter for a potentially reactive grain.

Diamond (1976) reviews the properties of aggregates susceptible to being alkali-silica reactive.

### 1.3 The reaction in principle

When the pore liquid attacks the silica in the aggregate grains the rate of dissolution, and the properties of the reaction products, will be affected by the concentration of  $\text{OH}^-$ -ions, i.e. the pH-level. This takes place in three steps:

1. Directly. Increased concentration of hydroxide ions increases the speed of dissolution.
2. During the attack, the quotient between fresh hydroxide ions and already formed alkaline metal silicate ions governs the fluidity of the solution of the reaction products.
3. In a mortar or a concrete with cement or lime, the hydroxide ion concentration greatly affects the calcium ion concentration in the pore liquid, and in this way governs the composition of the gel and its capacity to absorb water and expand.

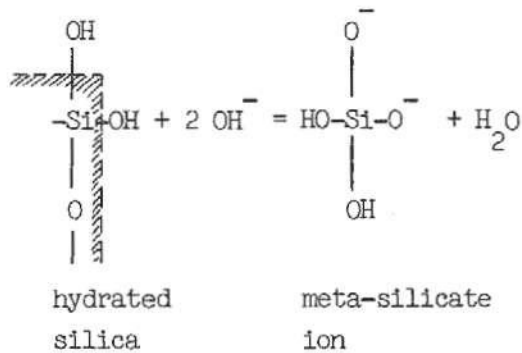
At pH-levels of 12.5 or less, calcium hydroxide has full solubility. If the silica is reactive enough to dissolve, this will take place slowly, and only calcium silicate hydrates will be produced, similar to those which are formed during normal cement hydration.

At high pH-levels the amorphous silica will be dissolved more rapidly. The pore liquid will be very poor in calcium ions, and the reaction product will be an alkaline metal silicate (or in short alkali silicate) gel.

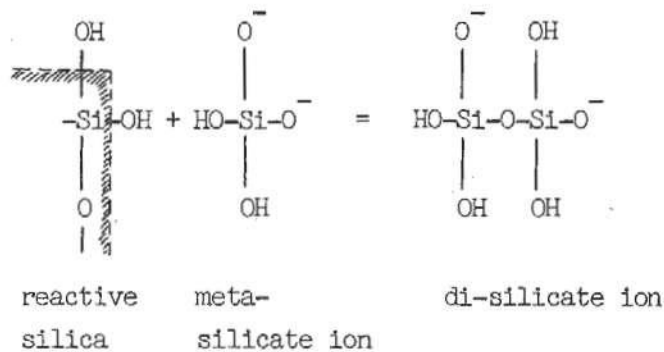
The mechanism of reaction and expansion is not yet fully understood but can be regarded as a two-stage process. In the first stage the alkalis and the easily soluble silica react rapidly as long as more of this type of silica is available and the alkalinity of the pore solution is high in the vicinity of the reactive grain. The product of this first reaction will have a low silica/alkali-ratio, and its solution in the pore liquid is very fluid.



First reaction



Second reaction:



The second stage of the process, to some extent takes place simultaneously with the first stage. The fluid reaction product of the first reaction reacts with further silica. Consequently the silica/alkali ratio increases. The liquid turns to a gel with, for a given concentration, decreased fluidity, or increased rigidity, when the ratio increases. At the same time, the pH-level of the gel, and its surroundings, will decrease.

The mechanism is very much a transport problem. The availability of alkalis close to a reactive grain is limited as they move by diffusion. The amount of fluid reaction product from the first reaction is also limited and its mobility depends on its fluidity and the pore system in the reactive grain. The sizes of the sodium ion and the potassium ion are different, and this may affect the physical properties of the gel.

All alkali silicate gels are hygroscopic. They expand when they take up water. The absorption of water in a gel is capable of damaging the surrounding concrete, or mortar, only if the gel exerts a pressure when expanding. The most reasonable way of shaping this pressure is that each molecule of silica, which is dissolved, absorbs some water from the pore liquid. The concentration of ions, dissolved in the gel, will in this way be somewhat greater than the concentration of ions in the pore liquid outside the gel. So the water molecules outside the gel have a tendency to enter the gel. The pressure from the gel will be of the same magnitude as the osmotic pressure between solutions with the same difference in concentration. Experience shows that the gel will sometimes exert a greater force by expansion than the mortar can take, and cracks will be formed.

If the cement paste contains capillary pores, and the gel is of the fluid type, with a low ratio silica/alkali, some gel may escape. If this happens, the pressure will be reduced. No cracks form, but sometimes the surface may be stained, in patches, by the escaping gel liquid.

Struble & Diamond (1981) have tried to describe the correlation between free expansion and the pressure exerted during hindered expansion but so far no straight-forward connection between the expansion and the composition has been found. Gels that swell greatly when free expansion is possible do not necessarily generate a high pressure when the possible expansion is hindered by the surrounding mortar.

Because of the complexity of the mechanism of the reaction and the expansion it is not yet possible to calculate, or even estimate, the expansion of a concrete even if the composition, the properties of the constituents and the environmental conditions are known. Some attempts on mortars containing a standard reactive aggregate have, however, been successful, cf. Hobbs (1981). Expansion measurements are usually used to estimate the behaviour when alkali-silica reactions occur.

#### 1.4 The effect of some factors; pessimum effects

A high alkali content, a large amount of reactive silica and water saturated conditions do not necessarily cause deleterious alkali-silica reactions in a concrete structure. These three factors, alkali, silica and moisture, are necessary but not sufficient conditions for deleterious reactions to occur. The factors must be present in certain proportions to generate maximum expansion and damage. Such a combination is called the pessimum combination, cf. FIG 1.4.1.

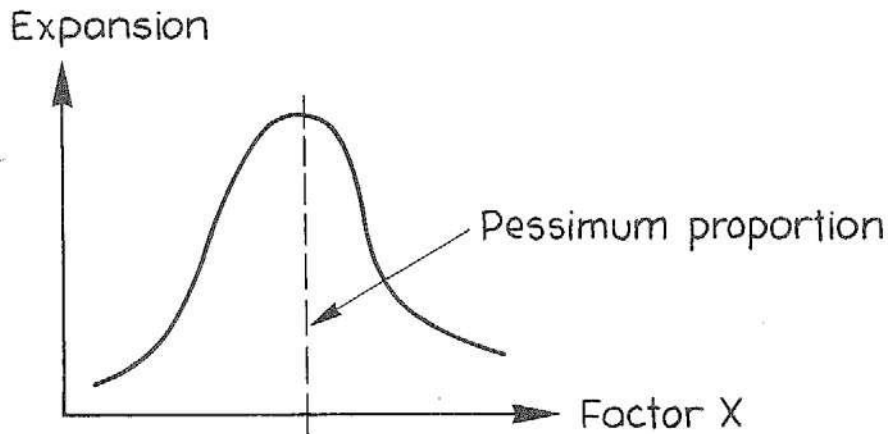


FIG 1.4.1 The pessimum effect in principle

With other factors remaining constant, if that is possible, pessimum effects are found in several parameters affecting the degree of damage due to alkali-silica reactions. Examples of such parameters are:

- o Alkali content of the cement
- o Total amount of reactive material in the aggregate
- o Size-distribution of reactive grains
- o Water-cement ratio
- o Cement content
- o Moisture content and moisture history
- o Temperature

As these parameters often influence each other, pessimum effects make it very difficult to study the effect of a single parameter. For instance the water-cement ratio influences in various ways.

A low  $w/C$  means a high concentration of alkalis in the pore solution.

If the degree of hydration is high, the cement paste will be practically free from capillary pores, and this will decrease the availability of the alkalis.

However, the high concentration of alkalis in the pore liquid may limit the degree of hydration.

Consequently the effect of a low water cement ratio upon the availability of the alkalis is very difficult to predict.

At a higher  $w/C$  the concentration of alkalis in the pore liquid is lower. At the same time, the mobility of the alkalis is greater and the concrete strength is lower.

Obviously there are one or more pessimum effects of the water-cement ratio.

The total amount of reactive minerals in the aggregate also has a pessimum effect, cf. FIG 1.4.2.

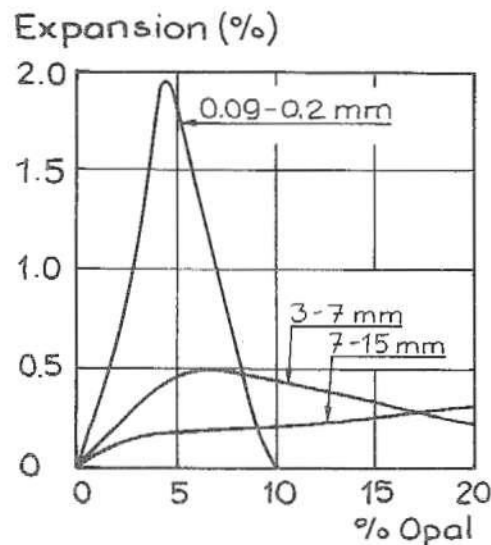


FIG 1.4.2 The pessimum effect of the total amount of reactive mineral, Locher & Sprung (1973)

With a low content of reactive silica, in this case opal, the expansion increases with an increasing silica content. With contents of silica above the pessimum proportion there is a decreasing effect of increasing silica content, due to the insufficient supply of alkalis to the reactive grains. An excess of soluble silica will reduce the pH-value, which will increase the availability of calcium ions, promoting a sound hydration of the cement.

The pessimal silica content varies with the size of the reactive grains, cf. FIG 1.4.2. For very small grains most of the alkalis are already consumed before the concrete hardens, if the amount of silica in small grains is high. These grains act as a pozzolan.

Other parameters act in a similar manner. The effect of various parameters was investigated early by Vivian et al (1947, 1950-52, 1955). Their reports, see the Reference List, are still applicable for understanding the different factors affecting the deleterious alkali-silica reactions.

### 1.5 The effect of moisture

Moisture participates in many ways in the mechanism of the alkali-silica reaction and the swelling pressure generated causing over-all expansion and pop-outs. Water is a solvent for the dissolved ions taking part in the reaction. The ability of adsorbed water to act as a solvent is questionable as it has a thickness of only a few molecule layers. The capillary condensed water, usually with a negative pressure, can, however, act as a solvent. This means that not all of the water in concrete acts as a solvent and that a diminishing moisture content results in an increasing alkali concentration. If the concentration in capillary saturated concrete is called  $(N)_{e 100}$ , where 100 means 100 % RH, and all of the moisture in the concrete is supposed to be a solvent, the concentration can vary with the pore humidity as in the example shown in FIG 1.5.1, based on data from Nilsson (1980). If, which is more likely, only the capillary condensed water acts as a solvent, the concentration increase will be even greater than indicated in FIG 1.5.1.

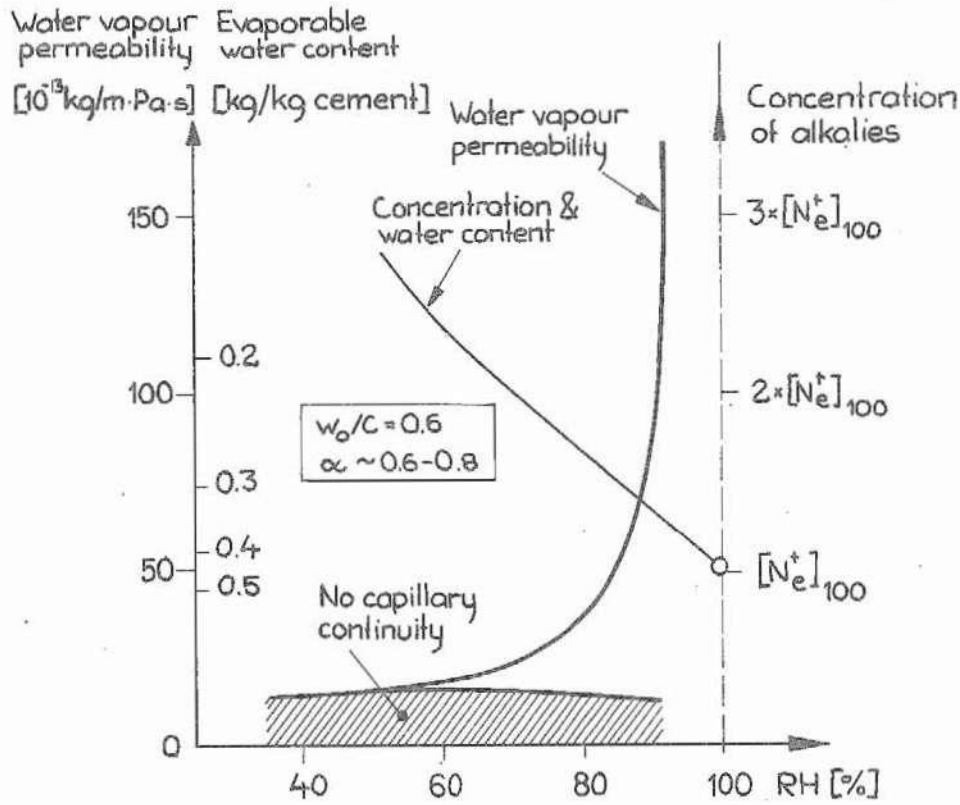


FIG 1.5.1 Examples of the possible effect of moisture on some factors participating in the alkali-silica reaction. Data from Nilsson (1980).

(Note: The water vapour permeability is a coefficient expressing the total moisture flow in terms of a gradient in vapour pressure.)

Water is also a transport medium for the dissolved ions. The flow of water is affected by the pore humidity as shown in the example in FIG 1.5.1, indicating a very slow moisture transport by vapour diffusion at lower humidities and an increasing flow rate with increasing humidity due to an increasing capillary continuity of the liquid phase. The flow of water naturally causes an increasing transport of dissolved ions, but the moisture flow as vapour diffusion at lower humidities is a "capillary break", i.e. an obstacle to such a transport. This is also what happens when no moisture flow takes place and the flow of ions by diffusion in water is prevented at lower humidities when no continuous liquid phase is present. Increasing humidity means an increasing number of paths for such a flow.

The flow of water containing dissolved ions also results in an increasing alkali concentration in the evaporation zone where the ions in the liquid flow are deposited when the moisture flow continues as diffusion. A very high concentration increase is possible due to this effect, cf. Nixon et al (1979).

The swelling of the gel is affected by the surrounding pore humidity. Krogh (1976) measured the absorption of water by synthetic gels and indicated a critical moisture condition in this respect of about 90 % RH. The gels act similarly to a hygroscopic salt, cf. FIG 1.5.2.

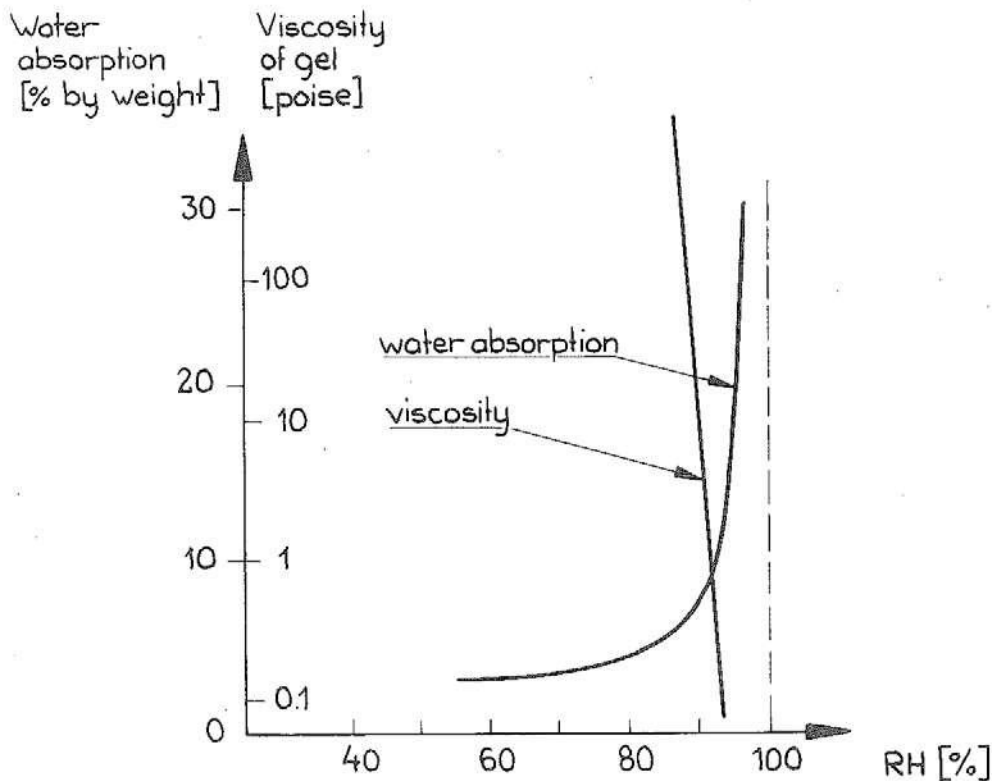


FIG 1.5.2 Examples of the effect of the moisture conditions on the behaviour of an alkali-silicate gel; absorption data from Krogh (1976) and approximate viscosity data from Moore (1978).

The damage caused by the swelling of the gel is also affected by moisture as it affects the viscosity of the gel, cf. Moore (1978) and FIG 1.5.2. A very small increase in the humidity of the gel will cause a decrease in viscosity and a possibility of the gel penetrating the surrounding cement matrix without causing any expansion.

The resulting moisture dependence of the expansion due to ASR is determined in one case by Lenzner & Ludwig (1978). Their results are shown in FIG 1.5.3, and indicate a critical moisture condition of about 85 % RH for the composition used.

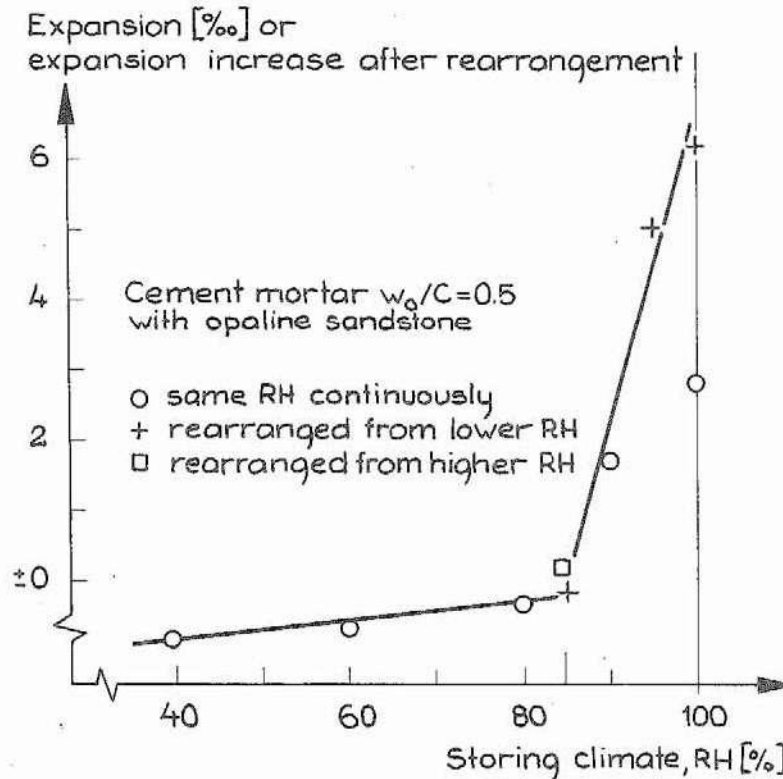


FIG 1.5.3 Effect of atmospheric humidity on expansion due to alkali-silica reaction; data from Lenzner & Ludwig (1978).

The results shown in FIG 1.5.3 also indicate amplified effect of moisture changes. Drying followed by a humidification to a humidity over the critical level, causes greater expansion than storing continuously at higher humidity. Similar results have been reported by Hobbs (1980).

The moisture condition affects the reaction and the swelling pressure, generated by the gel, in different ways. The critical moisture condition, above which the reaction continues, seems to be lower than the critical moisture condition for obtaining the maximum swelling pressure. A suitable humidity close to a reactive grain can therefore result in the production of a rather dry gel with a high viscosity. A subsequent humidification can then create a very high swelling pressure. The aim has been to make use of this mechanism in order to try to reproduce pop-outs experimentally, as described



in Chapter 3.

### 1.6 Current test methods

Different test methods are used to determine the reactivity of concrete aggregates, cements and combinations of these. The types of methods generally used are three:

- o Chemical measurements
- o Expansion measurements
- o Petrographic examination

Some of the methods are presented briefly below. The Scanian aggregate used in the experiments described in Chapter 3 has been included in a comparison of current test methods, Olafsson & Thaulow (1981). The results of these tests are shown when the different test methods are described.

#### 1.6.1 ASTM C 227 Mortar Bar Method

Mortar bars, 1" x 1" x 10", are cast with a specified flow and aggregate-cement ratio. The bars, after demoulding and a first length measurement are continuously stored at a temperature of + 38° C and a relative humidity of 100 %. Length changes after 1, 3, 6 and 12 months are determined as a measure of the reactivity of the cement/sand combination tested.

In FIG 1.6.1 the results of the tests with different cements mixed with the Scanian sand "HB" are shown. The ASTM reactivity criteria of more than 0.1% expansion after 6 months is marked in the Figure.

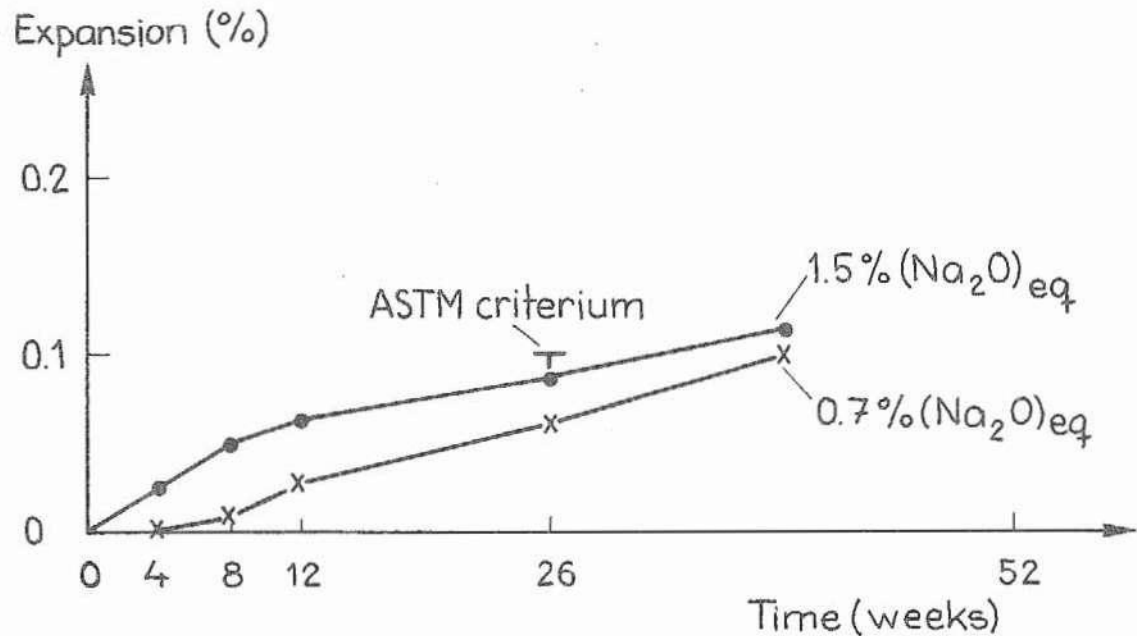


FIG 1.6.1 Results from the mortar bar method on a Scanian sand, Olafsson & Thaulow (1981)

According to ASTM C 227 the sand should not be considered as being capable of harmful reactivity. In practice no cases with over-all expansions are known involving this sand. Only pop-outs have occurred.

The Scanian sand produces expansions almost independent of the alkali content of the cement and the addition of 7.5% silica-fume had no beneficial effect on the expansion. However, most Danish and Icelandic sands tested simultaneously showed an increasing expansion with increasing alkali content of the cement used. Only some highly reactive Danish and Icelandic sands expanded more than the Scanian sand when mixed with the cement with an equivalent alkali content of 0.7 %.

#### 1.6.2 T.I. Mortar Bar Method (NaCl)

According to this method, developed at the Technological Institute in Tåstrup, Denmark, mortar bars 4 x 4 x 16 cm are used. They are stored in a saturated NaCl solution at +50<sup>o</sup> C. Average expansions of three mortar bars are determined. Results for the Scanian sand HB are shown in FIG 1.6.2.

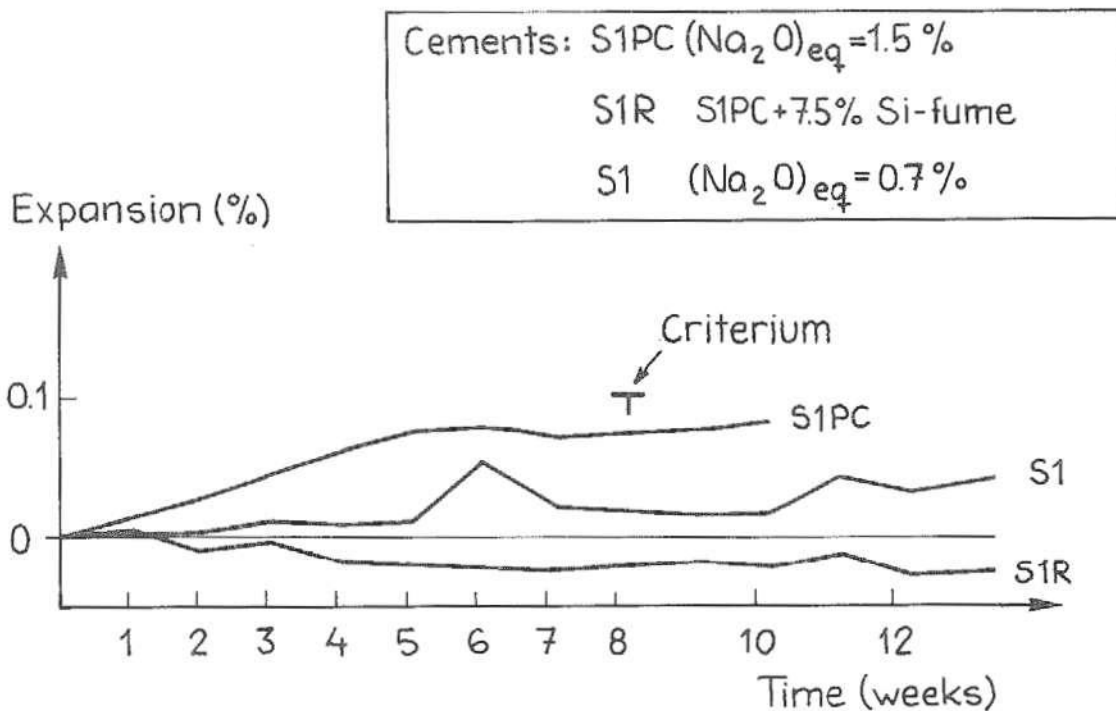


FIG 1.6.2 Expansions in saturated NaCl solution at +50°C of sand HB

The original reactivity criteria was >0.1 % expansion after 8 weeks but this was later modified to >0.1 % expansion after 20 weeks. The Scanian sand HB is "not reactive" according to both criteria.

### 1.6.3 ASTM C 289 Quick Chemical Test (QCT)

An aggregate sample is crushed in a specified way and treated with a NaOH solution (1 mole/litre) at +80°C for 24 hours. The amount of dissolved silica ( $S_c$ ) and the reduction in alkalinity ( $R_c$ ) of the solution are measured. A potentially deleterious aggregate is indicated if the point ( $R_c, S_c$ ) lies on the deleterious side of the curve in FIG 1.6.3.

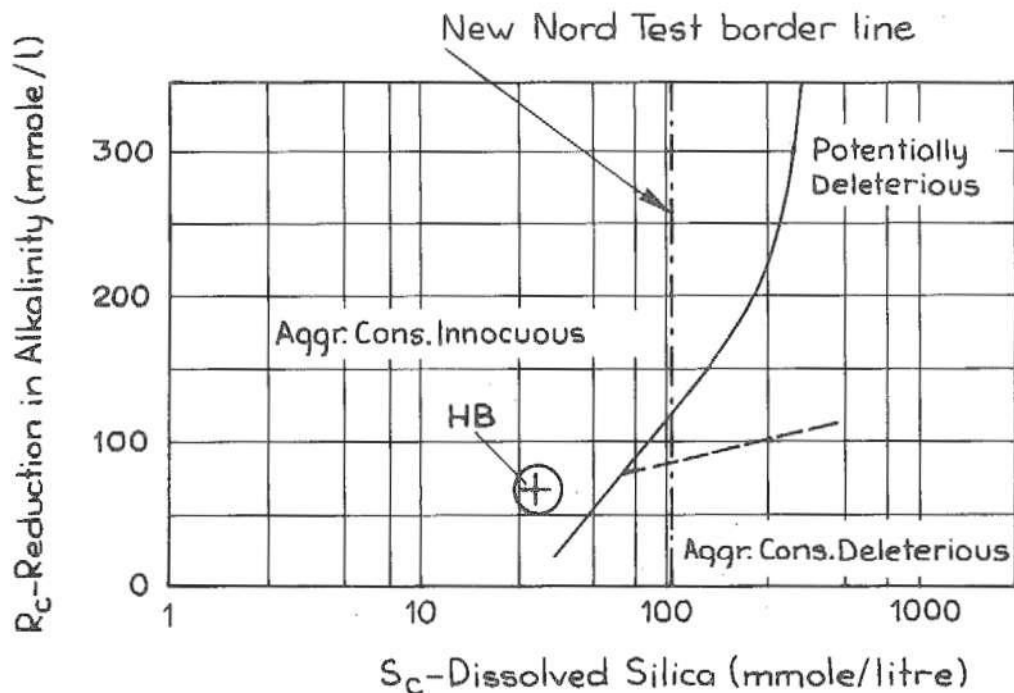


FIG 1.6.3 The division between innocuous and deleterious aggregates on the basis of the QCT.

For the sand HB a reduction in alkalinity of  $R_c = 60.2 - 63.7$  mmole/litre and an amount of dissolved silica of  $S_c = 29.2 - 30.4$  mmole/litre were obtained. This Scanian sand is consequently "considered innocuous" even with the new border line,  $S_c = 100$  mmole/litre, now recommended as a Nordtest method for accepting sands, Olafsson & Thaulow (1981). This Modified QCT is obviously not valid where pop-outs are concerned.

#### 1.6.4 "Vorbeugende Massnahmen"

In this German test method the sand is treated with a NaOH-solution at  $+90^\circ\text{C}$  for 4 hours and the weight losses in the different sand fractions are determined. If the weight loss is  $<0.5\%$  the aggregate is regarded as "non-reactive".

For the sand HB Olafsson & Thaulow (1981) determined a weight loss of  $0.3\%$  for the fraction 1-2 mm and  $0.8\%$  for the fraction 2-4 mm. The latter fraction is only "possibly useful" according to this test method. They conclude, however, that the method is not reliable for sands.

#### 1.6.5 Petrographic examination

Thin sections made from specimens of laboratory or field concrete are examined microscopically and the types of aggregate present are determined. A qualitative examination is carried out based on the known behaviour of various types of aggregates. Reaction products, cracks etc can also be discovered in the thin sections.

The Scanian sand HB was classified as "accepted by petrography".

#### 1.6.6 Other methods

A number of other methods are used to test the potential reactivity of an aggregate. The gel-pat test is a very simple one where selected grains are embedded in a cement paste disc. One face of the disc is ground to expose the grains and the disc is then immersed in an alkali solution and the development of any reaction product from any of the grains is studied for several weeks, cf. Sims (1981).

In other methods a scanning electron microscope before and after immersing the specimens in an alkali solution, density measurements etc. are used.

## 2 THE SCANIAN PROBLEMS

Problems due to alkali-silica reactions in Sweden are new and coincide with the introduction of a high-alkali cement in Scania, in the south of Sweden, where an ordinary concrete aggregate contains reactive grains of opaline flint and sandstone. The problems occur only as pop-outs on certain structures and turn out to be a definite moisture problem as pop-outs have never been found on outdoor structures even when they are wet. Problems arise only on "moist indoor structures", that have had a certain history of moisture changes. The most frequently affected structure is concrete slabs on the ground. More than 9 out of 10 problems with pop-outs due to ASR are found on such a structure. Also other concrete structures of great thickness with a more or less vapour tight surface cover have been affected.

In this chapter the moisture conditions of these typical structures are briefly described. The result of a simple inventory of damages is shown and some damage investigations are summarized.

### 2.1 The typical structures concerned

Concrete structures close to the ground, such as a concrete floor, acquire a moisture contribution from the ground. The structure and its "moisture history" are shown in principle in FIG 2.1.1.

The surface will dry out until a vapour tight surface cover is applied. Then the structure will continue to dry out slowly, if possible, towards the ground through the heat insulation, if any. If not, a moisture flow from the ground will take place continuously.

As long as no surface cover has been applied, drying continues and the surface is dry. When an impermeable surface cover is applied the remaining building moisture is "trapped" in the concrete for a long time. The drying time needed to reach an average moisture content corresponding to a RH of 90 % depends mainly on the slab thickness as shown in FIG 2.1.2, Nilsson (1980).

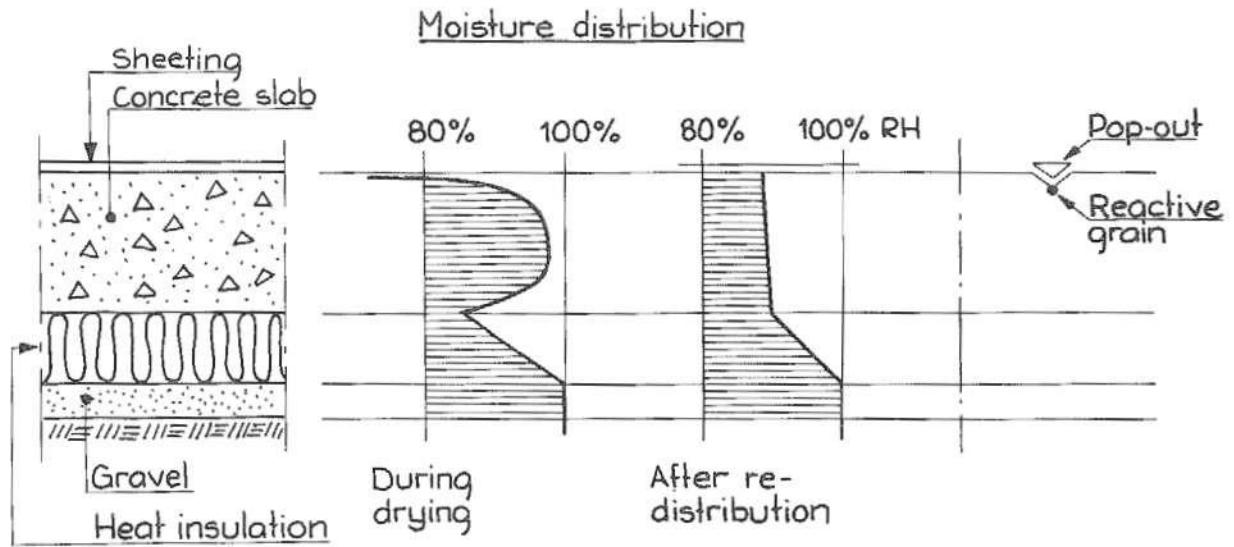


FIG 2.1.1 Typical floor structure and moisture distribution in a concrete slab on the ground.

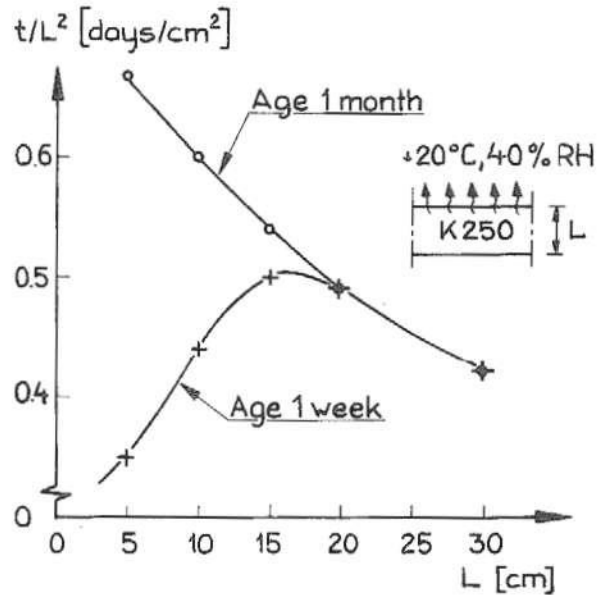


FIG 2.1.2 Effect of the slab thickness on the required drying time to reach an average moisture content corresponding to  $<90\%RH$ , Nilsson (1980)

Depending on the vapour resistance of the surface coverings, the moisture distribution in the slab will be distributed to different degrees throughout the depth and consequently the surface will slowly become wetter.

Eventually the concrete structure reaches equilibrium moisture conditions. The relative humidity at a certain depth can then be estimated approximately from the following expression

$$RH(x=d) \cong \frac{v(T_{gr})/v(T_{in}) + RH(in) \cdot x}{1 + x} \quad (2:1)$$

where  $RH(x=d)$  = relative humidity at depth  $d$

$RH(in)$  = relative humidity inside

$v(T_{gr})$  = saturation vapour content of air at ground temperature

$v(T_{in})$  = saturation vapour content of air inside

$x$  =  $Z_a/Z_b$

$Z_a$  = vapour resistance of slab thickness  $L-d$  and underlying materials

$Z_b$  = vapour resistance of slab thickness  $d$  and floor covering

The moisture conditions close to the surface, where pop-outs may occur, are consequently determined by several variables. At equilibrium relative humidities below 85 % RH close to the surface will be present in the following conditions:

- o an underlying heat insulation that acts as a capillary break and creates a temperature gradient of at least  $3^{\circ}\text{C}$ , i.e. in Scania for slabs not larger than 15-20 m

or

- o a surface cover with a resistance to vapour flow,  $Z$ , not over  $10 - 50 \times 10^3$  s/m for a slab thickness of 0.1 m.

If neither of these conditions are fulfilled moisture conditions corresponding to 85 - 95 % RH may be present even if the structure includes a capillary breaking material.



## 2.2 Simple inventory of damages

An attempt was made to inventory the experiences from floor structures built as a concrete slab on the ground during the period when a high-alkali cement was used in the south of Scania. The limits of this simple inventory were:

- o Objects: groups of more than five small houses (information from "Länsbostadsnämnden")
- o Time: concrete delivered during the period 1975 - 1977
- o Geography: south of a line between Lund and Kivik (where all the problems had occurred)
- o Structure: concrete floor directly on the ground with floor coverings applied to the concrete surface (built-up floors and floor elements excluded)

Information about the objects within these limits was not always easy to obtain. In spite of repeated requests information about 40 % of the houses was missing. It is, however, reasonable to believe that there was a lack of interest due to the fact that no problems had occurred. The remaining 60 % is probably over-representative where problems with pop-outs are concerned.

Some of the information requested was difficult to obtain. Details of the structures, type of floor coverings, date when pop-outs occurred and drying times before applying the floor coverings have not been available for all of the 60 % that answers were received from.

The inventory can be summarized as follows:

- o The inventory includes a total of 1400 small houses
- o Pop-outs had been developed in 190 of these; a "frequency" of 14 %

o The dependence of type of underlying heat insulation was

- mineral wool	"frequency": 19 %
- expanded polystyrene	14 %
- expanded clay	14 %

o Time of the year when the concrete was casted: no significant differences

Half of the frequency for underlying mineral wool arose from one single building site. Further information from each object is consequently needed to gain more knowledge from the damages incurred in practice. Additional information has been collected from some damaged structures, including a few of those above, and this is presented as case studies in 2.3.

### 2.3 Case studies

The moisture effects have been studied by measuring the moisture distribution on some damaged structures. Observations and results from a number of cases are summarized below.

#### 2.3.1 Case 1: An epoxy painted basement floor

Several large pop-outs (up to 60mm in diameter!) have occurred in an epoxy painted concrete floor in a basement. In different parts of the basement, the moisture distribution through the floor slab has been determined by measuring the relative humidity (RH) on samples taken at different depths, cf. Nilsson (1980). Some results are shown in Figure 2.3.1.

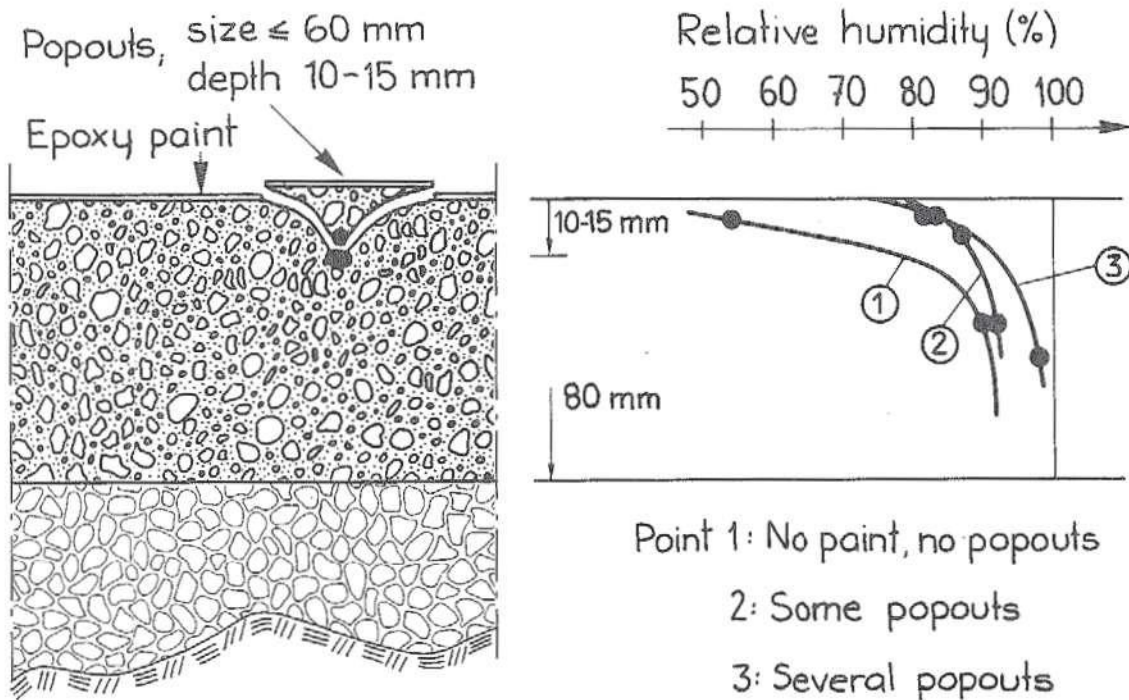


FIG 2.3.1 Moisture distributions in "case 1": An epoxy painted basement floor

The concrete floor is wet close to the ground and there is a moisture flow upwards. Where the floor is not painted, however, the upper part of the floor slab is dry and no pop-outs have been created here. The epoxy paint has a certain resistance to moisture flow, depending on its thickness and quality. This results in higher moisture conditions in the upper part of the floor; around 90 % RH in the upper 20 mm, a little lower on the concrete surface.

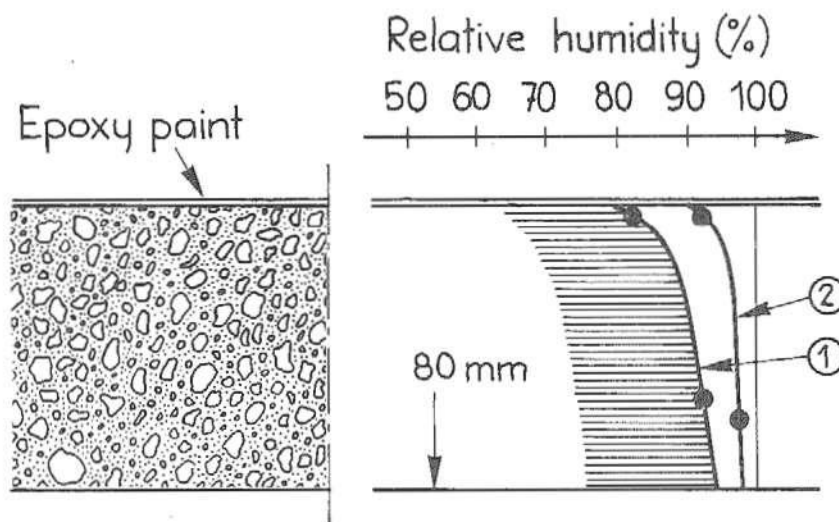
Several very large pop-outs occurred originating from reactive grains at a depth of 10 to 15 mm where the relative humidity is over 85 % RH but lower than 95 % RH. Closer to the surface the humidity is even lower and consequently no small pop-outs from this depth have been created.

### 2.3.2 Case 2: An epoxy painted floor in a heated hangar

At an epoxy painted hangar floor pop-outs, gel extrusions and deterioration of the paint film have occurred. The moisture distributions were measured at different parts of the floor, see Figure 2.3.2.

The hangar was built on a slope. Consequently the layer of sand beneath the concrete floor was only 0.15m at one end of the hangar but 0.5 to 1.0m at the other. This meant that there was a capillary suction of water to the slab at one end but only a much more limited moisture flow at the other. The epoxy paint had a high resistance to moisture flow resulting in a rather small moisture gradient over the slab.

Where the sand beneath the slab acted as a capillary break, the humidity in the upper part of the floor was around 90 % RH, resulting in several pop-outs. At the other end of the hangar the humidity was higher than 95 % RH and no pop-outs were created here, but a lot of gel extrusions through holes in the paint film was found. The film had also become deteriorated by moisture. Too wet conditions obviously counteract the creation of pop-outs.



Point 1: Popouts, diameter 5 - 25 mm

2: No popouts; fluid gel and

"ordinary moisture damages"

FIG 2.3.2 Moisture distributions in an epoxy painted hangar floor

### 2.3.3 Case 3: PVC-covered floors on a heat insulation of expanded polystyrene

In bathrooms, toilets and kitchens with concrete floors on the ground with an underlying heat insulation of expanded polystyrene, a lot of pop-outs occurred under the floor-coverings of PVC. Some PVC-coverings were replaced but new pop-outs were created within a few days.

The moisture conditions in one concrete slab were measured. The relative humidity was evenly distributed through the slab and a RH of 85 % was obtained.

Obviously 85 % RH is sufficient for the alkali-silica reaction to proceed, but no expansion or pop-out will occur unless more water is added. In this case the small amount of water present in the floor adhesive was more than sufficient.

### 2.3.4 Case 4: PVC-covered floors on a heat insulation of mineral wool

A concrete slab on the ground with an underlying heat insulation of mineral wool founded on concrete poles and is consequently rather thick, 0.25 m. Several small pop-outs were created under the PVC-sheets on the floors.

The moisture conditions were not measured until five years after the first pop-outs were found. The measured moisture distribution at two points, one close to a pole head, are shown in FIG 2.3.3.

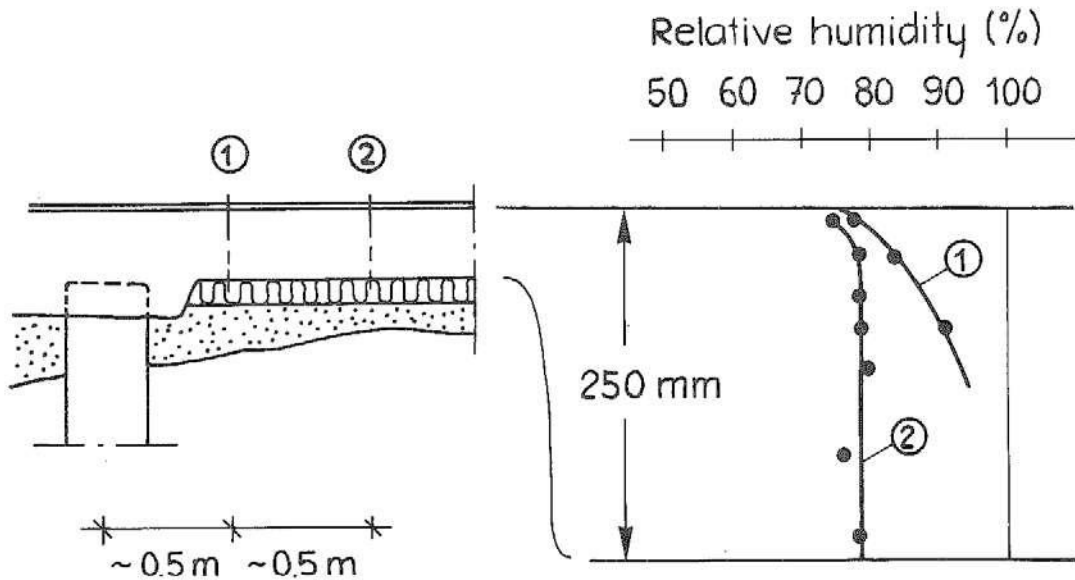


FIG 2.3.3 Measured moisture distributions in a poled concrete slab

The main part of the concrete slab is now dry; close to 80 % RH evenly distributed. No further pop-outs are likely and the previous ones were obviously caused by the building moisture in the concrete, that has now dried out downwards through the permeable mineral wool due to the temperature gradient. Because of the large slab thickness the drying-out time has been very long and when the PVC-sheets were applied large amounts of building moisture were still present. Afterwards the concrete surface was slowly humidified to a RH over 85 % and pop-outs occurred.

A contribution of moisture to the slab close to the pole heads still causes elevated moisture conditions. At a distance of 0.5 m from the poles, where the moisture samples were taken, the surface is too dry to cause any problems. However, in the future pop-outs may occur just above the poles.

#### 2.3.5 Case 5: PVC-painted kitchen ceilings

In some three-storey apartment houses a few very large pop-outs occurred in the kitchen ceilings. The concrete aggregate used did not, before or after, cause any pop-outs at all, but in this case some large reactive grains did. One of the grains studied had even produced two pop-outs, one small one before the ceiling was painted, as shown in FIG 2.3.4.

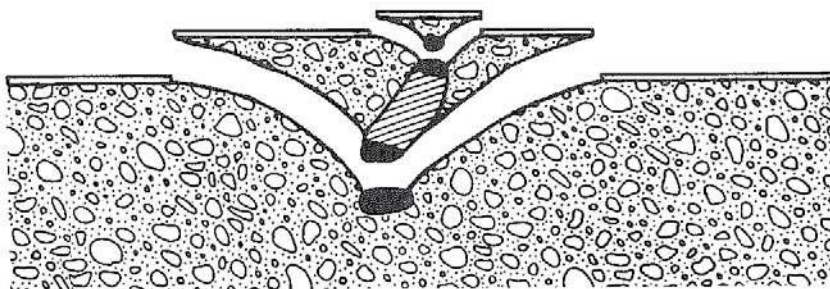


FIG 2.3.4 A reactive grain causing two pop-outs

The exact "moisture history" of the ceilings has not been investigated. A high water-cement ratio ( $>0.9$ ) and rain during the building period meant that the concrete structures had been very wet for a long time. Where the concrete surfaces had been covered on both sides, with a PVC-sheet and PVC-paint respectively, the moisture conditions made it possible for a reaction to occur. The use of sand-filling in the ceilings added more water.

### 3 EXPERIMENTAL

Attempts to reproduce the damages obtained in practice, pop-outs, have been made experimentally by using a concrete composition similar to the ones used in practice and expose the surface to a "history of climatic conditions" similar to those that an ordinary concrete slab is exposed to. This has been done stepwise starting with attempts only to create the pop-outs, as this turned out to be rather difficult in a few pre-experiments that had been carried out previously.

The next step was aimed at creating pop-outs under laboratory conditions in a reproducible way. In these experiments a preliminary study of the surface strength against pop-outs was included.

To be able to quantify the effect of various factors a special study has been made regarding the possibilities of correlating expansion measurements to the occurrence of pop-outs. In an experimental series separate reactive grains have been selected and their reaction studied when embedded in cement mortar.

The effect of some essential parameters on the occurrence of pop-outs was then studied. Finally some preliminary test methods have been examined during a relatively long period in order to obtain as much experience as possible. These tests also include attempts to develop an inhibitor.

The procedures used and the results obtained from these experiments are presented below.

#### 3.1 Attempts to create pop-outs at all

To be able to create pop-outs, one full-scale experimental series (I) was carried out under conditions as similar to practice as possible and one series (II) included a larger number of small specimens covering a wider range of variables.



In both series the concrete composition was the same, i e a high-alkali Portland cement, "Slite Std-79", with an equivalent alkali content of 1.04 %. the aggregate 0-8 mm was from "Hassle-Bösarp" and contains 5-10 % opaline flint and sandstone in the fractions >1 mm. The coarser aggregate was crushed 8-12 mm from "Hardeberga". The proportions were:

cement	213 kg/m <sup>3</sup>	
water	189 kg/m <sup>3</sup>	w <sub>o</sub> /C=0.76
aggregate 0-8 mm	970 kg/m <sup>3</sup>	aggregate 0-8/cement ratio 4.55
aggregate 8-12 mm	975 kg/m <sup>3</sup>	

Series I contained six specimens each divided into four parts as shown in FIG 3.1.1. The specimens were cured for six weeks, then dried for ten days before filling the surface. Then the drying continued until a relative humidity of 85 % was reached at a depth of approximately 10 mm beneath the surface. The method of measuring RH is shown in FIG 3.1.1. One half of each specimen was 2/3 covered with PVC-sheeting and the remaining third with an epoxy coating. The other halves of the specimens were covered in a similar way when the humidity had dropped to 75 % RH.

With the exception of specimen A the sand beneath the mineral wool was kept wet as shown in FIG 3.1.1. The difference in treatment of the six specimens was:

Specimen	Special treatment:
A	No additional water from below
B	Sealed curing at +20°C
C	Cured in 93 % RH at +20°C
D	Sealed curing at +4°C and +20°C, changed every day
E	As D but was wet once a week
F	Cured outdoors

The experiments continued for six months. During this period only specimen C showed any pop-outs and these were few and small, 5-10 mm, and occurred during curing in 93 % RH. When the PVC-sheetings were removed after six months, the specimens C-F had some cracks showing initiated pop-outs. The size and extent of these pop-outs, however, are not at all of the same magnitude as those that occurred in practice.

Series II contains 52 small specimens, 150 x 100 mm, with a thickness of 80 mm and sealed on all surfaces but one. The parameters dealt with were surface treatment during casting, curing climate and curing time, drying climate and drying time and the way of rewetting the specimen surface. Only one specimen from each combination of variables was used and in some of the excluded combinations the "paths" for the 52 specimens became as shown in FIG 3.1.2. The results are also shown in FIG 3.1.2 for gel extrusion in nine of the specimens and small pop-outs in five.

The pop-outs that were obtained were very small, most probably due to too short curing and drying times, 8 to 12 weeks. Those obtained, however, are the result of sealed curing at +20°C for eight weeks, drying in 65-85 % RH for four weeks and then rewetted by sealing the surface and adding water from the bottom. With longer curing and drying times the sizes might have been larger. The results, however, indicate the ways in which the attempts to create pop-outs on purpose and in a reproducible way should continue.

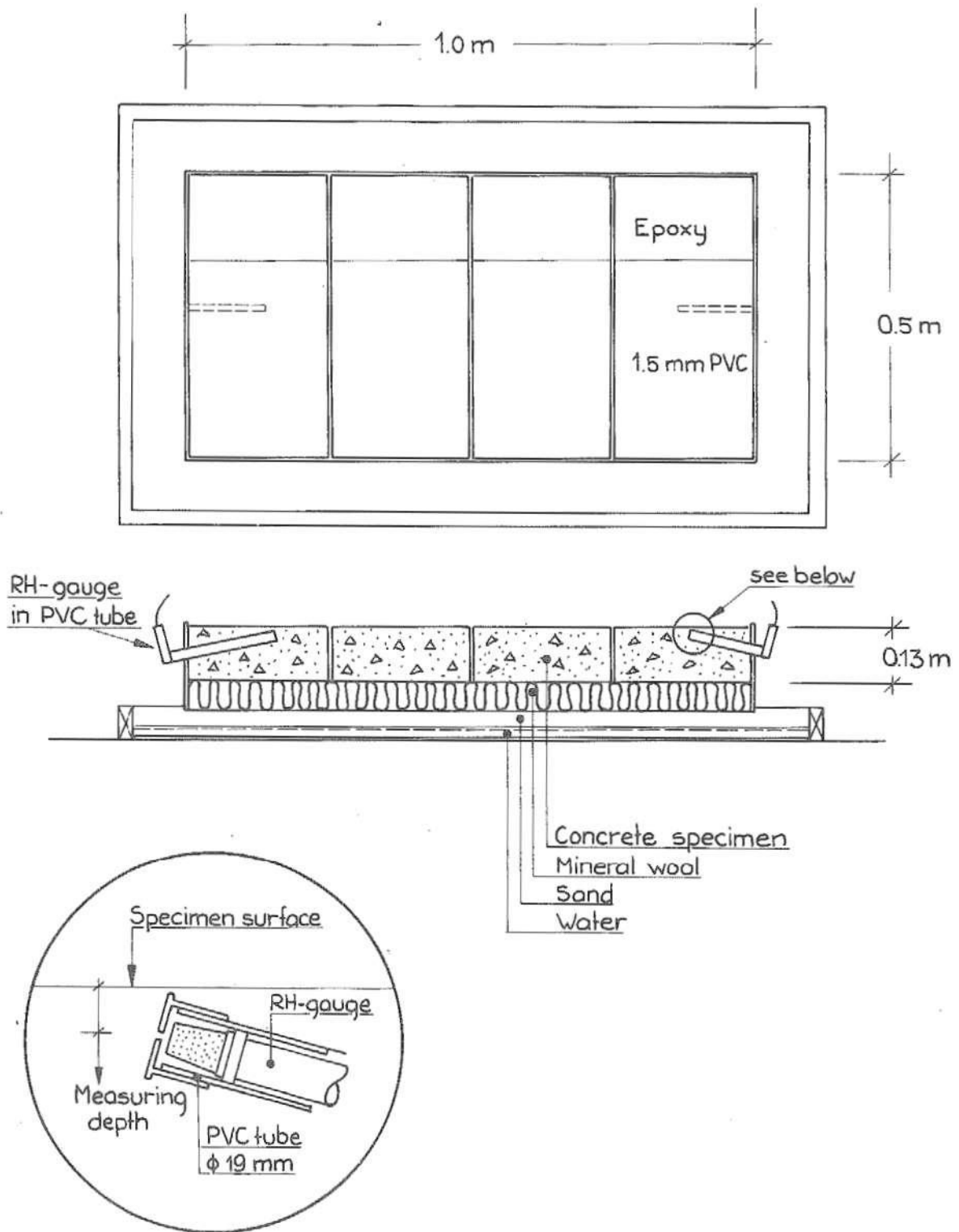


FIG 3.1.1 Full-scale specimens used in the attempts to create pop-outs.

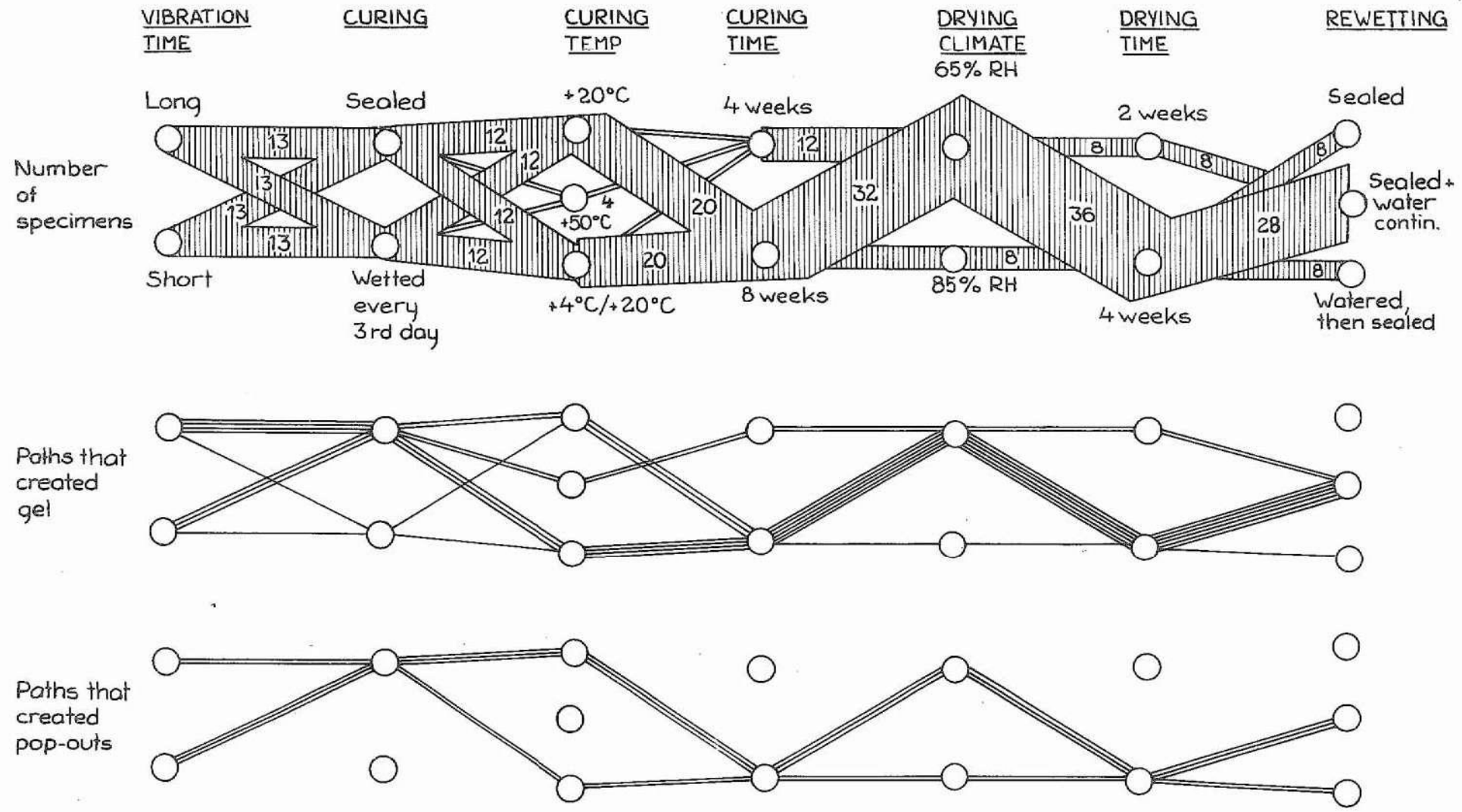


FIG 3.1.2 Summarized results of the experiments on small specimens with the objective of creating pop-outs.

3.2 Attempts to reproduce pop-outs under laboratory conditions

Attempts to reproduce the damages incurred in practice have been made experimentally by using a concrete composition similar to the ones used in practice and exposing the surface to a "history of climatic conditions" in a reproducible way.

Mortar and concrete specimens, as shown in FIG 3.2.2 have been made using the same constituents as in 3.1 but in slightly different proportions  $w/C=0.7$  and aggregate 0-8/cement ratio of 3.8. The surface was floated after a couple of hours and then steel trowelled. After a certain curing time at  $+20^{\circ}C$ , the specimens were dried in various climates in climate rooms at  $+20^{\circ}C$  and climate boxes as shown in FIG 3.2.2. Finally the specimens were rewetted, usually in one of two different ways, cf. FIG 3.2.2. The "moisture history" used, is shown in principle in FIG 3.2.1.

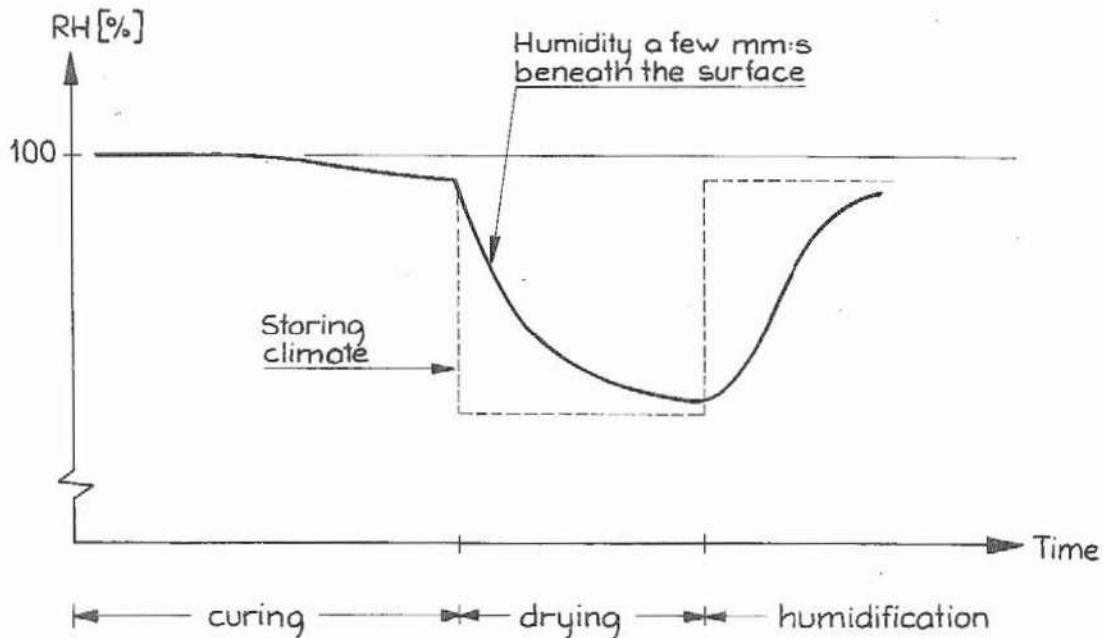


FIG 3.2.1 Approximate "moisture history" for reactive grains close to the surface and the storing climate of the specimens.

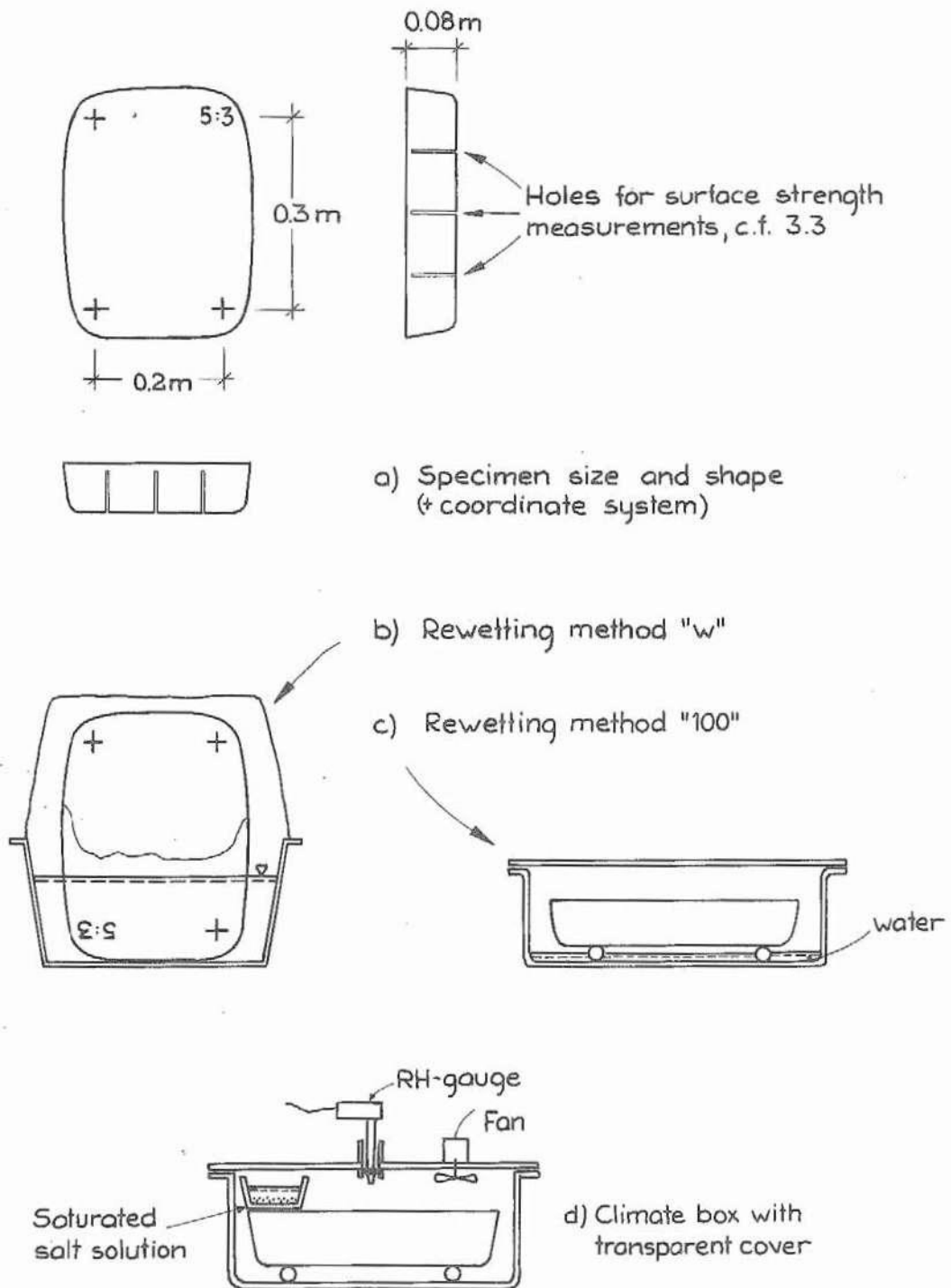


FIG 3.2.2 Specimens and arrangements used in the attempts to reproduce pop-outs by using controlled climatic conditions.

The moisture history of each specimen is shown in TABLES II-VI together with the results expressed as number and sizes of pop-outs obtained. In FIG 3.2.3 a photo of one specimen, sealed cured one week and then stored over water, is shown. The number of reactive grains close to the surface is indicated by the gel extrusions.

The results can be summarized as follows. Only storing under perfect sealed or wet conditions produces a lot of gel extrusions but no pop-outs. A large amount of gel is produced as a result of the ASR, but the gel has too low a viscosity to affect the surrounding mortar. Instead the gel penetrates the cement mortar as seen in FIG 3.2.3. In some cases the conditions during curing were not perfectly sealed and occasionally small pop-outs were created during the curing period.

Most of the pop-outs occurred in specimens that were dried at very high humidities, over 80 % RH. Pop-outs in specimens dried in a somewhat lower humidity occurred only after rather a long period of slow humidification when probably more gel is produced.

The used histories of moisture changes, however, have not given reproducible results. The mechanical properties of the surface and the depths where the reactive grains were located, are probably not the same in all of the series due to some differences in surface treatment. This means that different levels of swelling pressure are needed in order to create pop-outs and consequently the reaction time before rewetting was always sufficient.

From series 8-11 it can be seen that something in the humidification process seems to be decisive. It is not necessarily the rate, but the level to which the surface is rewetted, that matters. Rewetting by storing over water means a slow passing of each humidity condition between the drying climate and 100 % RH. The effect of this has been studied in the later series by rewetting to humidities lower than 100 % RH or storing continuously in such climates, and sometimes at a higher temperature.

As seen from series 15 at a temperature of +30 to +40<sup>o</sup> C pop-outs were created already during drying, if the humidity was in the vicinity of 90 % RH. At lower humidities pop-outs occurred after rewetting. The drying times were only four weeks. A few large pop-outs occurred one year after the

rewetting started.

When stored continuously in different climates for a year and a half, as in series 16, only a few pop-outs occurred at  $+20^{\circ}\text{C}$  if RH was less than 80 % or greater than 95 %. For humidities between 85 % and 95 % RH more pop-outs were definitely obtained. At  $+40^{\circ}\text{C}$  a lot of pop-outs were obtained at 88 % RH but some even occurred at 96 % RH.



TABLE II Specimens, series 2-4, with different "moisture histories"

Curing	Drying	Rewetting climate	Specimen	Number of pop-outs	Size (mm)
Sealed 1 week 95-100 % RH 1	85 % RH for 8 weeks	100 % RH	2:1 mortar	20 <sup>x</sup> ) +3	4-15
			2:2 concrete	20 <sup>x</sup> ) +1	4-15
		93	2:3 mortar	7 <sup>x</sup> ) +2	4-10
			2:4 concrete	10 <sup>x</sup> ) +6	5-10
Sealed for 12 weeks (not perfect)	65 % RH for 4 weeks	W+100 % RH	3:1 mortar	3 <sup>*</sup>	7-15
			3:2 concrete	6 <sup>*</sup> +2	5-10
	93 % RH for 4 weeks	W+100 % RH	3:3 mortar	4 <sup>*</sup>	4-10
			3:4 concrete	4 <sup>*</sup>	4-15
95-100 % RH for 3 weeks	condensation, then sealed		4:1 concrete	7	4-20
x) some smaller pop-outs occurred during drying *) " " " " " " curing +3 additional pop-outs during 1.5 year W=rewetting by putting one third of the specimen in water for one day					

TABLE III Specimens, series 5-7, with different curing conditions and climates. Rewetting by method "W+100", cf. FIG 3.2.2.

Curing	Dried 4 weeks in	Mortar specimens			Concrete specimens		
		No	Number of pop-outs	Size (mm)	No	Number of pop-outs	Size (mm)
Sealed 8 weeks	93 % RH	5:1	1 <sup>x</sup> )	15	5:13	0	
	80	5:2	1 <sup>x</sup> ) +1	12	5:14	0	
	65	5:3	0+1		5:15	1 <sup>x</sup> )	8
Sealed 16 weeks	93	5:4	2 <sup>*</sup> )	5-10	5:16		
	80	7:5	0+1	13	7:17	0	
	65	7:6	0		7:18	0+2	50
100 % RH 8 weeks	93	7:7	0		7:19	0	
	80	7:8	0		7:20	0	
	65	6:9	0		6:21	0	
100 % RH 16 weeks	93	6:10	0		6:22	0	
	80	6:11	0		6:23	0	
	65	6:12	0+1	8	6:24	0	
*) one small pop-out during curing; the other directly after rewetting x) pop-outs occurred after >6 weeks storing in 100 % RH + additional pop-outs during 1.5 years							

TABLE IV Specimens, series 8-11, cured 1 week (series 8 in 95-100%RH; series 9-11 sealed). Then dried in different climates and humidified in two different ways.

Drying climate		Rewetting	Mortar specimens			Concrete specimens		
First week	2nd-5th week		No	Number of pop-outs	Size (mm)	No	Number of pop-outs	Size (mm)
93%RH	80%RH	W+100%RH	8:1 9:7 11:1	0+2 10*)+1 0+2	11-20 5-10, 45 25	8:13 9:19 11:13	0+1 2*) 0	13 5-8
80	65	W+100	8:2 9:8 11:2	0+2 0 0	13-40	8:14 9:20 11:14	0 0 0	
65	65	W+100	8:3 10:9 11:3	0 0+1 0+1	20 50	8:15 10:21 11:15	0 0 0	
93	80	100	8:4 10:10 11:4	1 9+3 2	7 3-10-15 4-7	8:16 10:22 11:16	0+3 6+2 0	3-12 5-7-10
80	65	100	9:5 10:11	0 9	3-10	9:17 10:23	0 3+5	4-7, 8-18
65	65	100	9:6 10:12	3+2+3 5x)+4	4-7, 15-30 4-10-15	9:18 10:24	0 1x)+3	5-15
W = rewetting by putting one third of the specimen in water for one day 100 = rewetting by storing in 100 % RH *)within a week after rewetting x)pop-outs occurred after >4 weeks in 100 % RH								

TABLE V Specimens, series 12, sealed cured for 12 weeks, then dried 8 weeks in different climates.

Drying climate for 8 weeks		Rewetting	Specimen No	Number of pop-outs	Size (mm)
+20°C	93 % RH	W+100	:1	0	
		97	:4, :8	0	
		100	:5	0+1	10
	80	W+100	:2	0+2	8-9
		100	:6	0+1+1	8
	65	W+100	:3	3x)	9-12
		100	:7	2x)	3-8
x)pop-outs occurred before start of drying					

TABLE VI Specimens, series 13-16, sealed cured 1 week (series 15 additional curing at +40°C), then dried in different climates.

Additional curing	Drying climate		Rewetting	Specimen No	Number of pop-outs	Size (mm)
	93 % RH 1st week	93 % RH 2nd-9th	W+100 100	13:4 :8	0+2 0+1	5-25 25
	93	80	W+100 100	:1 :5	3+1 1+2	6-13 4
	80	65	W+100 100	:2 :6	1 2+1+2	6 3-6-10
	65	65	W+100 100	:3 :7	1 0	6
	93 % RH 4 weeks	1 year: 93%RH <97		14:1 :4	1+2 1	6 5
	80		86 93 <97	:7 :2 :5	5-10+3 2 0+1+1	2-5,8-10 7-13 15,25
	65		86 93 <97	:8 :3 :6	0+4 0 6	5-10
+40°C 1 week	93 80 65		100 <100 <100	15:1 :2 :3	3+1 0+2 2+3	7-11-20 5-40 2-4
+40°C 2 weeks	93 80 65		<100 <<100 100	:4 :5 :6	1+1 2 0+1	15+35 8 40
+40°C 2 weeks	88 75 75	+40°C 4 weeks +40°C +30°C	100 100 100	:7 :8 :9	7* 3+1 8	4-15 3-10,25 4-16
	79 % RH cont. 86 91 93 <97		- - - - -	16:1 :2 :5 :7 :8	0+few +1+1 0+8+4+2 0+5+3 0+3+1+1 0+2	<5,8,10 <10,15 5-15 10,50,25 15-20
	+40°C 88%RH 3 weeks	+40°C 96%RH		:3	4+2+2	5-13
	+40°C 88%RH 5 months	+20°C 93%RH		:4	5+15-20+1	5-16,20, 40
	+40°C 96 % RH continuously		-	:6	3+3+1	5-10, 18-20
*) already during curing						



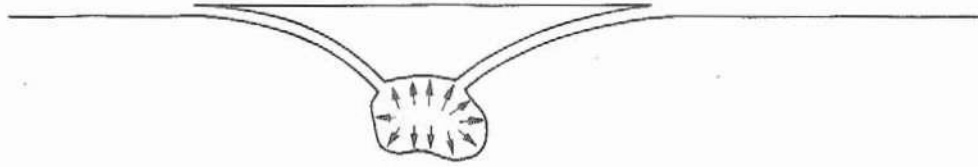
FIG 3.2.3 Surface of specimen No 6:24, sealed cured for one week and then stored in 100 % RH at +20°C, showing no pop-outs whatsoever but many gel extrusions.

### 3.3 Surface strength against pop-outs

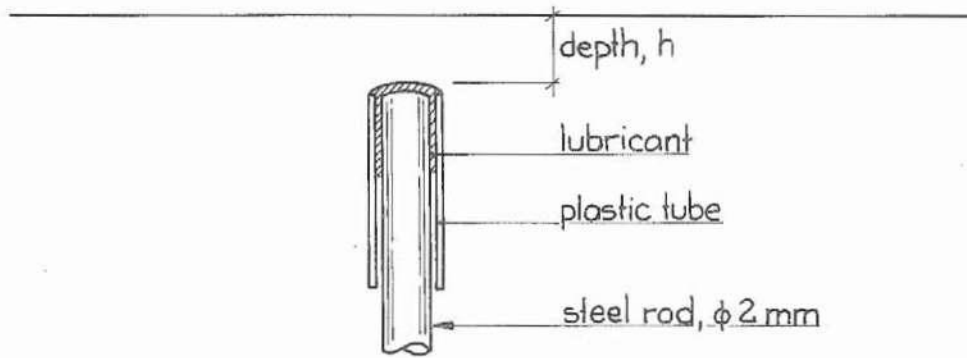
The swelling pressure needed to create a pop-out depends on the size and depth of the reactive grain and the concrete properties of the surroundings of the grain. Bache & Isen (1968) studied the resistance to pop-out formation of a mortar and expressed the effect of the size and depth of the grain. The grain size they used, however, was rather large and not valid in the least for the reactive grains in the aggregate 0-8 mm which are of interest in this study. With a grain size smaller than most of the particles in the concrete aggregate, the variations of the surface strength must be very great at a given grain size and depth.

To estimate the pressure required and observe any deviation from specimen to specimen, a simplified method of measuring the surface strength against pop-outs has been developed by using a steel rod to apply a force, see FIG 3.3. 1. These steel rods were placed in every specimen mould, nine in each mould as shown in FIG 3.2.2, with the ends close to the prospective specimen surface. A thin plastic tube was put on each rod and a lubricant at the top was used to prevent cement paste from entering between the rod and the tube. The specimens from each series were tested, after one week of sealed curing, by measuring the force-deformation relationship when a pop-out was created, cf. FIG 3.3.1. Additional specimens were tested after a year and a half, when the specimens had been dried and rewetted in different ways. The results obtained are shown in FIG 3.3.2, the full curves being the result of a regression analysis.

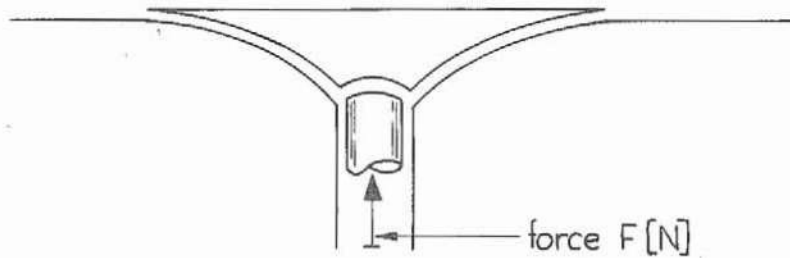
Obviously the surface strength varies very much when pressure is applied very close to the surface and in a small area. This is exactly what happens when pop-outs are created. The same pressure generated from a gel in different spots may very well create a pop-out in one spot but not in another, where perhaps twice the pressure is required.



a) The creation of a pop-out in principle



b) Steel rod at casting a specimen



c) Application of a force at testing

FIG 3.3.1 Method of testing the surface strength against pop-outs.

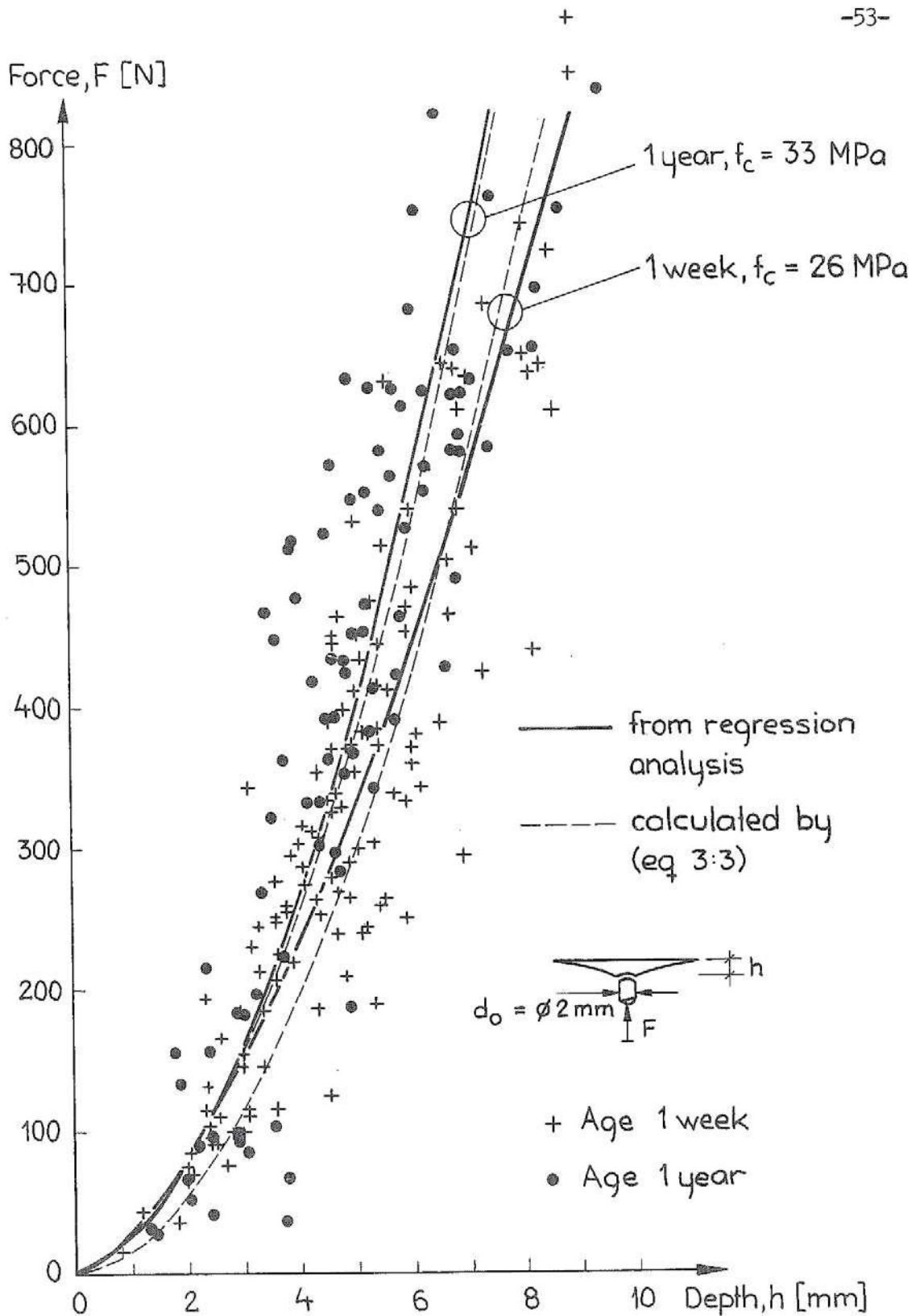


FIG 3.3.2 Results of testing the surface strength against pop-outs, series 5-16.

The failure mechanism when a pop-out is created depends on the fluidity of the gel. A fluid gel also exerts a pressure inside the cracks forming the pop-out. A rigid gel, however, creates a pop-out in a way that very much coincides with the application of a concentrated force as in the described experiments.

Consequently the failure mechanism is very similar to what happens in pull-out tests and punching of slabs. The mechanism of punching has been theoretically examined by Braestrup (1979) by plastic analysis of punching shear failure. His results can be used to estimate the resistance to pop-out formation of a concrete surface.

Braestrup (1979) expresses his solutions in terms of a non-dimensional "load parameter"

$$\frac{F}{f_c \pi (d_o + 2h)h} \tag{3:1}$$

where  $F$  is the failure load,  $f_c$  the compressive strength,  $d_o$  the punching diameter and  $h$  the slab thickness.

This load parameter is found to be almost independent of the punch diameter, with the exception of small diameters. For  $d_o$  smaller than the slab thickness the results of Braestrups calculations can approximately be expressed as

$$\text{"Load parameter"} = 0.06(1+0.02d_o/h) \tag{3:2}$$

The absolute value is additionally somewhat depending on the tensile strength of the concrete.

From (3:1) and (3:2) the punching force can be expressed as

$$F = 0.06 \pi f_c (h+0.02d_o)(d_o + 2h)$$



or approximately

$$F = 0.12 \pi f_c (h + 0.26d_o)^2 \quad (3:3)$$

The forces needed to create pop-outs at the surfaces of the mortar and concrete specimens, as a function of the depth of the swelling pressure, have been calculated from equation (3:3), and compared to the experimental results. Then  $h$  is the thickness of mortar between the steel rod and the surface. The punching diameter,  $d_o$ , has been chosen to be the diameter of the free surface at the bottom of the hole where the rod is inserted, cf Fig 3.3.1, since in the experiments the cracks forming the pop-outs were found to originate from the edge of this surface.

With a compressive strength of 26 MPa at one week and 33MPa at a year and a half, the agreement between calculations and experiments is very good, as shown in Fig 3.3.2. The lines of short dashes show the calculation results.

A compressive strength of 26 MPa for a cement mortar and concrete with a  $w/C$  of 0.7 and a week of age is a reasonable choice. The development of the compressive strength in more than a year after the first week, however, normally is greater than from 26 to 33 MPa. The agreement between calculations and experiments, in spite of this, indicates that the growth in compressive strength (and tensile strength) close to the surface is very much detained if the surface is early dried and then rewetted.

For the specimens in question, the surface strength probably has decreased during drying due to surface cracks and the slow-down of the cement hydration close to the surface. During the additional curing during rewetting in moist air for more than a year, the surface strength has increased again, but only to an extent just above the strength at one week of age.

Moisture is needed close to the surface to generate swelling pressures, creating pop-outs from the grains near the surface. Moisture will also make it possible for the surface strength to grow. The generation of a swelling pressure, however, is much more rapid than the strength development. Consequently the resistance to pop-out formation may still be the smallest when the concrete surface is rather dry, but the gel in reactive grains

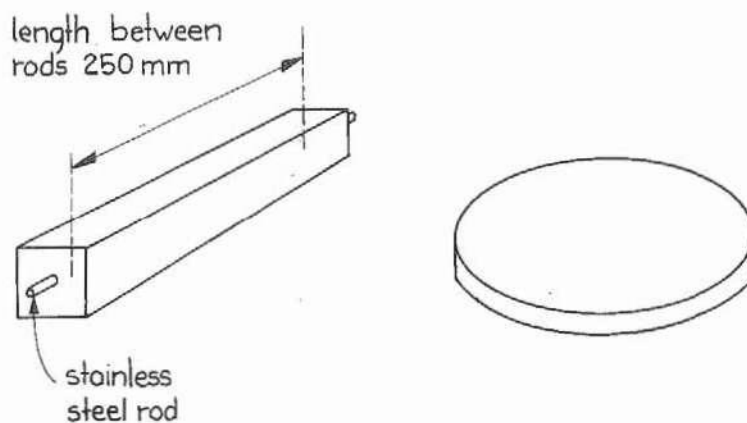
close to the surface has been rewetted to some extent and a swelling pressure has been generated.

The forces measured have to be translated into pressures to be able to compare the resistance to pop-out formation and the working capacity of the swelling gel. Some experiments using an oil pressure instead of a steel rod have been carried out with results of the same magnitude as Bache & Isen (1968).

### 3.4 Correlation of expansion and the occurrence of pop-outs

Most studies regarding alkali-aggregate reactions are carried out with the aid of expansion measurements, usually with the mortar bar test according to ASTM C 227. Such tests were carried out in Sweden before introducing a high alkali cement but the expansions obtained were small. In spite of this, damages in the form of pop-outs occurred. Obviously the mortar bar test is not suitable where pop-outs are concerned. Of course the amount of reactive grains affects an over-all expansion in a different way than the creation of pop-outs. However, the occurrence or not of some pop-outs is not a good measure of the reaction and is difficult to use when quantifying the effect of a variable.

In order to find a way of carrying out this "translation", attempts have been made to correlate expansion measurements to the occurrence of pop-outs by using moisture changes as a variable. As the occurrence of pop-outs is a surface phenomenon and expansion is due to over-all reactions, the specimens have to be thin in order to obtain comparable moisture histories for the grains concerned. The specimen sizes and shapes used are shown in FIG 3.4.1.



Prisms 20×20×280 mm

Discs φ 170×10 mm

$$\text{"Characteristic thickness" } t = \frac{\text{volume of specimen}}{\text{surface of evaporation}} = 5 \text{ mm}$$

FIG 3.4.1 Specimens used in the attempts to correlate expansion with the occurrence of pop-outs. The shapes of the specimens have the same "characteristic thickness" during drying.

The prisms and the discs have the same characteristic thickness and change average moisture contents at the same rate during drying. They respond to a humidity change very quickly and a state close to equilibrium is obtained within a few weeks. The mortar composition is the same as in 3.2 but the aggregate fraction 4-8 mm was excluded because of the small dimension of the specimens. After demoulding the specimens were sealed cured at +20<sup>o</sup>C for one week after which the initial measurements were made. The specimens were then exposed to various climatic conditions in the same way as shown in FIG 3.2.2.d.

Each series contains one disc and three prisms exposed to the history of moisture changes. The curing, drying and rewetting conditions of each series are shown in TABLES VII-VIII together with the expansions obtained and observations of pop-outs. Besides length changes, changes in weight and natural frequency were measured.

The series presented in TABLE VII were dried in different ways and then continuously rewetted over water, i e in 100 % RH. Not very many pop-outs were obtained and they were usually small. However, it was only during drying in 93 % RH the first month that resulted in any-pop-outs at all; there were none in all the other climates. Curing in different ways did not show any deviation regarding this matter. Hardly no expansion at all was obtained, cf. also FIG 3.4.2. Rewetting in 100 % RH after drying indicated a very small expansion that decrease later on, probably when 100 % RH has been reached all over the specimens.

In the series described in TABLE VIII, and to some extent in FIG 3.4.3, the specimens were continuously stored in different conditions. However small, pop-outs seem to be obtained if the reaction was amplified either by additional alkalis, by storing in a saturated NaCl-solution, or by using a higher temperature during drying. This coincides very well with the results obtained from 80 mm slabs, c f 3.2 series 15, see TABLE VI.

Some specimens start to expand if stored over water or in a saturated NaCl-solution. However, this expansion is not always accompanied by the occurrence of pop-outs. Storing over water gives some expansion but no pop-outs; sealed curing gives the opposite result. Storing over water is obviously too "wet", as has also been shown in 3.2. The created gel has too

low a viscosity to affect the mortar in any harmful way.

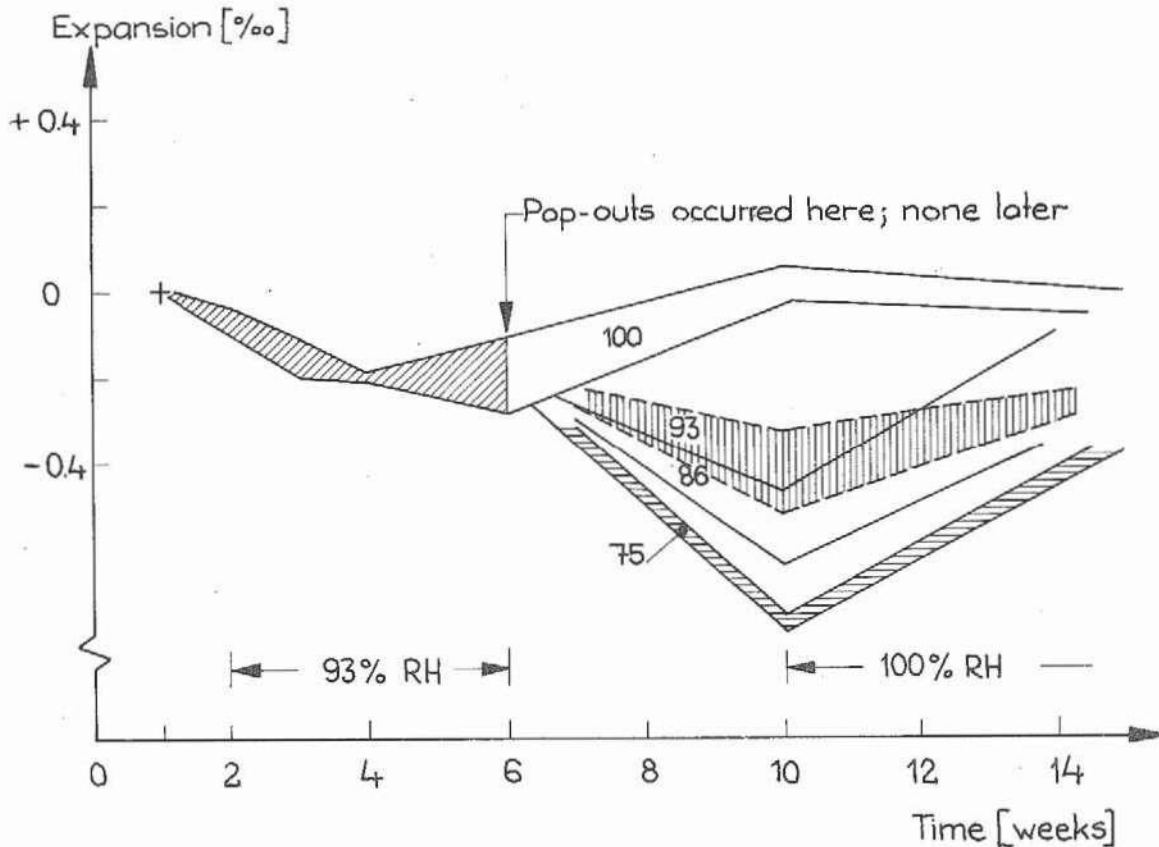


FIG 3.4.2 Results from expansion measurements on prisms dried 4 weeks in 93 % RH and then restored in various climates.

Consequently no correlation of expansion to the occurrence of pop-outs has been found. Examination of the few prisms that did expand, and sometimes cracked after rewetting, showed that if the reactive grains in the center of the specimens had reacted to a sufficient extent and the gel produced had not too low a viscosity, expansion occurred due to a single grain!

Those grains had probably caused pop-outs if they were situated closer to one of the four surfaces but, if this was the case, no expansion would be obtained.

With this kind of aggregate, only a few reactive grains, expansion seems to occur if one suitable grain happens to be included in a mortar bar. If not, no expansion at all will happen.

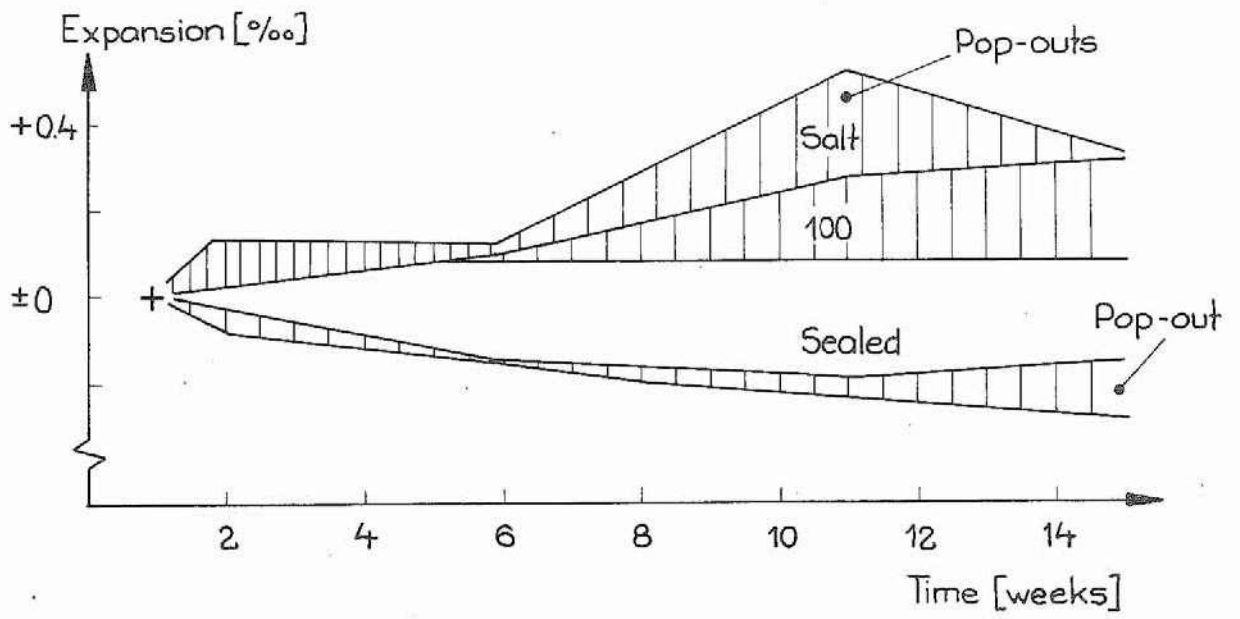


FIG 3.4.3 Results from expansion measurements on prisms continuously stored in NaCl-solution, over water or sealed.

TABLE VII Expansion measurements and observations of pop-outs on prisms and discs dried in different ways. Sealed cured 1 week.  
 Rewetted at 100 % RH over water continuously after drying.

Curing 2nd week	Drying climate		Expansion <sup>1)</sup> o/oo	Disc No	Pop-outs (*)	Size (mm)	
	First 4 weeks	Second 4weeks					
Sealed at +20°C +30°C +40°C	93 % RH	-	+0(+0.8)	1A	1(+1)	4+big	
				+0	3A	2	6-10
				+0.1	3B	1	4
	93 % RH	86 % RH	-0.2	1B	1	3	
75 % RH		-0.4	1C	2	3-5		
93 % RH		-0.2	1D	1	5		
Sealed at +20°C	86 % RH	-	-0	2A			
		86 % RH	-0.3	2B			
	75 % RH	-	-0.2	2C			
		75 % RH	-0.5	2D			
Over water	93 % RH	-	+0	3C	3 <sup>x)</sup>	4-5	
	86 % RH	-	-0.1	3D	1 <sup>x)</sup>	4	
	75 % RH	-	-0.2	4A			
None	93 % RH	-	+0	4B	2	3-4	
	86 % RH	-	-0.1	4C			
	75 % RH	-	-0.3	4D			
*) all pop-outs but two (x) were created during drying 1) mean value of 3 prisms (+) additional expansion and pop-out during 6 months of repeated drying in 79 % RH and rewetting in 100 % RH for one series of prisms & disc							

TABLE VIII Expansion measurements and observations of pop-outs on prisms and discs treated in various ways. Sealed cured 1 week.

Treatment	Expansion (o/oo)	Discs No	Pop-outs	Size (mm)	
Continuously: sealed cured cured over water	-0.2 +0.1+0.4	5A 5B	1+	6	
In saturated continuously NaCl-solution: 9 weeks then dried 4 weeks in 75 % RH and rewetted	+0.3+0.8+1.2 +0.4+0.8	5C 5D	>10+ >10+	2-4 5-15	
Continuously: +20°C	93 % RH	-0.2+0.7	6A		
	97 % RH	-0.1	6D		
	+40°C	88 % RH	-0.3	7C	>10+
		96 % RH	-0.1	6B	4+1 2-5
Continuously: +20°C	93 % RH	-0	7A	+1 small	
	97 % RH	+0+0.4	7D	+	
	+40°C	88 % RH	-0.4+0.2	6C	>10 2-5
		96 % RH	-0+0.2	7B	



### 3.5 Separate, reactive grains

A pop-out due to ASR is the result of one separate, reactive grain close to the surface. Independent of the number of reactive grains in a specific concrete aggregate, one grain is enough to create a pop-out. For examining the conditions for developing pop-outs from an aggregate it ought to be a good way to pick out some reactive grains and then study them separately, embedded in mortar specimens.

Some preliminary tests have been carried out in this way using the same cement and aggregate 0-8 mm as before. The fraction 4-5.6 mm was separated by high density liquids, and some of the "suspected grains" were embedded in small mortar specimens with  $w/C=0.7$  and non-reactive aggregate. Then, the surface was covered, wet in wet, with a 2 mm layer of a mortar with  $w/C=1.2$ . The intention was to simulate a concrete surface, where stones and the coarse sand grains have sedimentated because of too great fluidity of the fresh concrete.

The specimens were cured for one week over water, dried in 85 % RH for five weeks, and then rewetted with water. No pop-outs occurred, but when cutting the specimens into halves it was apparent that the surface layer of the first series was too thick, 8 mm. All of the grains selected, however, had reacted, and at least some gel was present.

The method of studying a single reactive stone in a mortar specimen seems to be useful. This chapter deals with some further series, mainly in accordance with the same principles. In two series, the following five variables were studied, the effect of one inhibitor was one of them:

- A. One reactive stone in mortar with  
non-reactive or reactive fine aggregate.
- B. Cementing agent Slite Std Cement,  
pure, or replaced with 1, 2, 5, or 10 per cent of silica-fume.

- C. Specimen cured  
at 100, or at 93 per cent relative humidity.
- D. Cured specimen dried for 28 days  
at 20, or at 40<sup>o</sup> C.
- E. Cured and dried specimen re-humidified  
in moist air, or partially immersed in water.

### 3.5.1 Aggregate

A pop-out in a concrete floor is most frequently caused by one single stone, about 3 - 4 mm in size. A common source of concrete sand is Hassle-Bösarp, about 24 kilometers east of Trelleborg. A sample of sand and fine gravel from this place was chosen for the present work.

The active test stones were selected from the grain size fraction 4.0 - 5.6 mm of the test sand from Hassle-Bösarp. The selected stones were rounded in form, and white or grey in colour. They could contain soft components (chalk), as well as hard components, such as opal and quartz.

The separation with heavy liquids was omitted. Previous work indicated that the correlation is bad between the density of a grain and its reactivity in mortar, the cement of which has the actual contents of alkalis.

All test stones were dried, in order to avoid dilution of the alkaline pore liquid of the mortar with pure water in the test stone. This means that a vacuum treatment is necessary for helping the pore water to enter the pore system of the test stone.

The fine aggregates of all main mortars always consisted of sand in the range 0.25 - 2.0 mm.

The non-reactive aggregate for Series 1 was chosen from a sample of Åstorp-sand (a quartz sand). The following fractions were extracted by sieving, and then mixed together in equal parts, by weight:

0.25 - 0.50 mm  
0.5 - 1.0 mm  
1.0 - 2.0 mm

In Series 2, the test stone was placed in a mortar with part of the fine aggregate reactive. The aggregate was taken from the concrete sand sample from Hassle-Bösarp. The same three fractions were taken as from the Astorp sand. They were mixed in accordance with the natural grain size distribution of the test sand:

0.25 - 0.5 mm 31.3 per cent by weight  
0.5 - 1.0 mm 53.3 "-  
1.0 - 2.0 mm 15.4 "-

A visual examination indicated that grains, suspected for containing reactive silica, were most frequently occurring in the coarse fraction, and least frequently in the fine fraction.

Among the non-reactive minerals, quartz was the most frequent one.

For the top layer mortar, the water cement ratio ought to be 1.2. In order to get a mortar with such a high value, a specially fine aggregate was chosen. A sample of practically pure quartz dust was taken from a quarry at Hardeberga, 5 kilometers east of Lund, in Scania. The dust was dried and sieved through a 0.5 mm sieve.

### 3.5.2 The cement mixture

A sample of Slite Standard portland cement was used, containing 1.25 per cent potassium oxide ( $K_2O$ ), and 0.22 per cent sodium oxide ( $Na_2O$ ). The total content of alkaline metal oxides, calculated as sodium oxide, was 1.04 per cent. It should be well understood that there never exist any alkaline metal oxides in free state in any cement. They always are combined, in the actual sample mainly as sulphates.

Norwegian silica-fume is claimed to be a suitable suppressor of alkali silica reactions. Its effectiveness was tested by replacing a part of the cement with the same weight of silica-fume.

The following mixtures of cement and silica-fume were used for the test specimens (cement mix):

Cement mix, parts of weight		Designation of specimen		Note
Cement	Silica-fume	Type of aggregate		
		Non-reactive	Reactive	
100	0	10	20	First figure in designation gives the type of aggregate
99	1	11	-	
98	2	12	22	Second (and third) figures give the percentage of cement, replaced with silica-fume
95	5	15	25	
90	10	110	210	

### 3.5.3 The test mortar

For each designation, at first one batch of main mortar, and then one batch of top mortar was mixed. The size of each batch was calculated to allow for the preparation of twelve test specimens.

The main mortar was mixed, in parts of weight, from:

Cement, or cement mix	1.0
Water	0.7
Sand mixture	3.6

130 milliliters, or 290 grammes, of this mortar mix was used for each specimen.

The non-reactive and the reactive sand mixtures had grains with different shapes. The grain size distributions also were different. Even so, the same weight mix could be used.

Silica-fume had a tendency to make the test mortar more cohesive. Even so, all the mixtures could be handled in the same manner as those with pure cement, when mixing and moulding the mortar.

The top layer mortar was mixed, in units of weight, from:

Cement, or cement mix	1.0
Water	1.2
Quartz dust	3.36

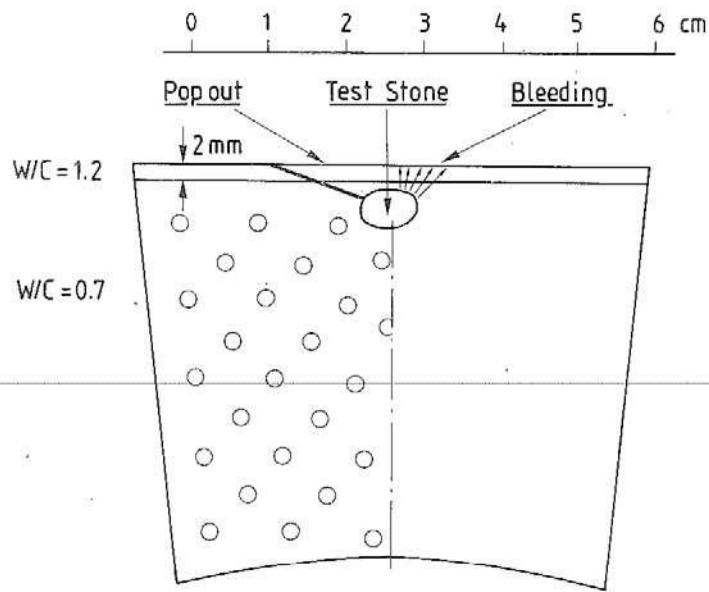
6.3 milliliters, or 15 grammes, of mortar was used for each specimen.

It was not necessary to adjust the weight composition of the mortar when a part of the cement was replaced with silica-fume.

#### 3.5.4 The test specimens

The main mortar was moulded in, at least, four consecutive layers in polystyrene beakers. Each layer was compacted on a vibrating table.

In order to improve the compaction of the ready test mortar by removing entrapped air, the freshly moulded beakers were placed in a vacuum desiccator and the air pressure was reduced to about 20 torr. The air bubbles expanded, and the enclosed air could escape. The fresh mortar was, however, stiff enough for permitting many of the air voids to remain open. Thus, a large quantity of air re-entered the mortar after the vacuum treatment, and it became necessary to re-compact the test mortar by vibration.



Silica fume: 0, 1, 2, 5, 10 p.c.

Curing: 100 or 93 p.c. RH

Drying 85 p.c. RH: 20 or 40 degr. C

FIG 3.5.1 The test specimen used in the study of separate reactive grains

It is highly questionable, whether this vacuum treatment really contributed to the compaction of the test specimen or not. Observations in cut test specimens showed that they generally contained some air bubbles, in spite of very careful treatment during the moulding and compaction.

In the central point of the top surface of each one of the ready compacted mouldings, one test stone was placed.

After which, the top layer mortar was prepared. The beaker was given a short vibration period, and the test stone was pressed down into the main mortar with a pair of tweezers, so that the stone was just covered.

Then the top layer mortar was applied with an average thickness of 2 millimeters (6.3 milliliters or 15 grammes of mortar), and the beaker was given a short period of vibration for making the top surface smooth.

In order to get the pore system of the test stone as far as possible filled with mortar water, the beaker was given a very mild vacuum treatment. The vacuum was limited to 55 per cent.

The test specimen is shown in FIG 3.5.1

### 3.5.5 The curing

The beaker was placed in a desiccator with pure water in the bottom space. After one day the beakers were stripped off from the specimens.

The stripped test specimens were marked with their designation number on the mantle surface (the ink must be lime proof, e. g. Indian ink).

Six specimens of each designation were given the letter "-A" after the designation number, and the residual six specimens the letter "-B".

Finally, the six specimens with the same letter were numbered "-1" to "-6".

The specimens were given one day more of curing in a desiccator with pure water in the bottom space. Then, the top surface of each specimen was ground to smoothness with wet grinding paper nr 120. Water was used for rinsing.

The marked and top-smoothed specimens were to be cured at about 20<sup>o</sup> C for 26 days more. They were separated into two groups.

All specimens marked "-A-" were stored in a desiccator with pure water in the bottom space (100 per cent RH).

The "-B-specimens" were stored in a box, where the relative humidity was kept at 93 per cent by wet potassium nitrate ( $\text{KNO}_3$ ) in a bowl. The air was kept moving by a fan, and the motor was placed above the lid, in order to avoid undesired heating of the test specimens. It was essential that the solution and the solid salt were in contact with the air in the box all the time, or the control of the humidity would fail.

After final curing, each specimen with the last figure "-1" was cut with a diamond saw, flushed with kerosine. The cut followed the axis of the test specimen, and it was essential that the test stone became visible.

### 3.5.6 Drying at 85 per cent relative humidity

The drying was performed in one box at  $20^{\circ}$  C and in another at  $40^{\circ}$  C. The same salt was used for both temperatures: potassium chloride (KCl). The boxes were equipped with fans, similar to the box with 93 per cent RH. All the time, the air in the box must contact the solid salt and also the saturated solution.

The specimens marked "-2" and "-4" from each designation and curing group were carried over to the box for drying at  $20^{\circ}$  C. After 28 days, the storing was interrupted. The specimens marked "-2" were cut along the axis of each specimen, so that the test stone became visible. The specimens marked "-4" were carried over for re-humidification.

The specimens marked "-3", "-5", and "-6", from each designation and curing group, were carried over to the box in the oven for drying at  $40^{\circ}$  C. After 28 days drying, the specimens marked "-3" were cut through the test stone, and the specimens marked "-5" and "-6" were carried over for re-humidification.

The 40 degrees box happened to crack during the last period, so that the potassium chloride solution dried out. Whiskers developed from the crystal bed and happened to carry over some potassium chloride lumps to the specimens 210-A-3, 210-B-3, 210-A-5, 210-B-5, and to parts of the edge of the top surface of specimen 25-A-3.

### 3.5.7 Re-humidification of the test specimens

These processes started at an age of 56 days.



For re-humidification in moist air all specimens marked "-4" and "-5" were carried over to a desiccator with pure water in the bottom space. The air in this desiccator was kept as near to 100 per cent RH as possible, but no stirring was arranged. The specimens needed a lot of time to adapt their humidity to the new type of storing. If observations did not reveal any visible changes, the specimens had to stay in moist storing for several months.

The re-humidification in a flat dish with a thin layer of water was intended to simulate the case when, e.g., a floor has become treated with magnesium fluosilicate solution. All specimens marked "-6" were selected for this type of storing. The duration was, principally, the same as for the moist air storing, but the access of water to all parts of the specimens was essentially better.

### 3.5.8 Observations

Totally 108 single test stones were involved.

In one case, designation 20-B-3, the reactive test stone caused a pop-out.

The fine aggregate was of the reactive type, but no silica-fume was used.

The curing was performed mainly at 93 per cent RH.

The drying at 85 per cent RH took place at 40° C.

After cutting, the half specimens were stored in moist air at room temperature. Within five weeks, the stone grew up 1.5 millimeters above the cut surface. In spite of this "volume escape", the top layer of the specimen cracked loose in the form of a rather large pop-out.

In one other case, designation 10-B-4, the reactive stone had caused a wet spot on the top surface during the drying period.

The fine aggregate was non-reactive, and no silica-fume was used.

The curing was performed mainly at 93 per cent RH.

The drying at 85 per cent RH took place at 20<sup>o</sup> C.

After cutting, the specimen was stored for five weeks in moist air. Liquid gel flowed out over the surrounding mortar, and the stone changed into a wet fine sand.

In seven cases the cut test stone showed milder signs of gel formation, without any mechanical harm to the test mortar, and without any visible wet spots on the top surface of the specimen. In the following Table, these specimens are put together in order of decreasing reactivity.

The line "Gel form" shows how far the gel liquid had spread over the surroundings, or whether only a brown border had formed around the surface of the cut stone.

The line "Stone char." tells about the hardness of the cut stone surface.

"Hard" refers to a content of, e. g., quartz or opal,

"soft" to a content of, e. g., limestone.

Design.	12-B-3	10-B-3	20-A-5	11-B-5	11-A-5	10-A-1	25-B-6
Gel form.	5 mm	3 mm	3 mm	1.5 mm	0.75 mm	border	border
Stone char.	hard	hard	rel h	soft	rel h	hard	soft

A test stone may very well feel hard, and still be able to generate a rather large volume of liquid gel. This seems reasonable, since opal is a hard mineral.

A summation of the Table may be the following:

In three cases: no silica-fume was used.

In two cases: one per cent of the cement  
was replaced with silica-fume.

In one case: two per cent, and  
in one case: five per cent of the cement  
was replaced with silica-fume.

In five cases: the mortar contained  
only non-reactive aggregate.

In two cases: the mortar also contained  
reactive fine aggregate.

In the series 2 small reactive grains, at most 2 millimeters in size, were part of the main mortar, in addition to the large test stone.

After the cutting of the test specimen, the halves were stored in a desiccator with pure water in the bottom space. Grains, which had suffered an alkali silica reaction, very soon absorbed water, and a drop of gel became visible.

The number of wet reaction spots, observed on the saw cut surfaces, have been put together in the following Table, as average values from all specimens with common last number.

Nr	History	Spots on 25 cm <sup>2</sup>	Pop outs on 25 cm <sup>2</sup>
-1	Just cured	7	1
-2	Dried 20 degr.	7	4
-3	Dried 40 degr.	6	2
-4	20 degr, air hum.	7	2
-5	40 degr, air hum.	6.5	2
-6	40 degr, wet stored	12	2

In the cut surfaces, most grains which are able to react with alkalis, seem to do so already during the 28 days curing period.

The relative humidity at curing

the temperature when drying in 85 per cent RH, and the content of silica-fume do not seem to influence the number of grains in the reaction state significantly, but re-humidification in a thin layer of water seems to increase the number strongly.

Reaction spots on the top or mantle surfaces of the specimens mainly develop during the re-humidification.

The reaction spots may be a grain, which generates a gel liquid, spreading to the surroundings. Sometimes, part of a grain may shoot up and form a "micro pop-out".

Cracks may originate from a reaction spot on the top or mantle surface, and mainly when five percent of the cement was replaced with silica-fume. No cracks occurred on specimens, stored in a thin layer of water.

Contamination of four test specimens with potassium chloride (look Chapter 3.5.6) resulted in "map cracking", though as much as ten per cent of the cement was replaced with silica-fume.

In the contaminated specimens an increased number of reaction sites was observed, and the attacked grains could be placed in the top or mantle surface, or in surfaces shaped by cutting. No re-humidification was necessary.

In the saw cut surfaces, an accumulation of a special type of translucent gel could be observed between the main mortar and the top layer. This layer of gel could sometimes be observed also on top surfaces of specimens, or on the upmost part of the mantle surfaces. The test stone could also be surrounded with gel of this type.

The formation of this type of gel was not dependent on, whether the fine aggregate of the mortar was reactive or non-reactive. Most of the gel seemed to be formed in mortars with cement mixes containing 2 or 5 per cent of silica-fume.

Such gel never transformed into a liquid when exposed to moist air. Probably, this gel mainly consisted of a hydrated mixture of cement mix and water, and it became visible when coarse aggregate sedimentated. The amount of visible gel was dependent on how easy the coarse aggregate grains could settle in the cement paste.

As the amount of this inter-layer gel is sometimes rather large, even when the specimen contained only one reactive stone, it should absolutely not be mistaken for alkali silicate gel. There is no doubt that, sometimes, mistakes happened in the preliminary tests with "micro-concrete" and one enclosed reactive stone.

### 3.5.9 Discussion

It is reasonable to presume that the chemical and physical laws for alkali silica reactions are the same, whether the particles are small or big.

The fact that only one single pop-out has developed in all the 108 test stones in this work may be explained in different ways:

1. It was too difficult to choose test stones with equivalent reactivity.
2. A sufficient content of silica-fume in the cement mixture may protect the test stone against the alkalinity of the cement, even if the stone is strongly reactive. The small reactive grains in the mortar are not protected even if so much as ten per cent of the cement is replaced with silica-fume. Probably their speed of reaction is too high, compared with that of the silica-fume.

On the other hand, most experience indicates that the fine reactive grains from Hassle-Bösarp (at most 2 mm) are not sufficiently common to be able to damage the concrete seriously. This point corresponds to the variable B. in Chapter 3.5.

3. The pop-out in the specimen 20-B-3 may have been promoted by the rapidly available silica in the fine aggregate of the mortar. This may possibly increase the quotient silica/alkaline metal oxides in the gel, thus making the gel more rigid at a given water content.

4. In a practical concrete, the combination 20-B-3 seems to be much more probable than 10-B-4, with all reactive silica concentrated in the test stone. This combination will probably promote the formation of a gel with low viscosity, which easily escapes through the pore system of the mortar. The points 3. and 4. correspond to the variable A. in Chapter 3.5.

5. A high calcium content of the gel is claimed to promote a great rigidity, thus increasing the capacity to shape cracks in the surrounding mortar, or concrete, by absorbing water.

The observation seems to be reasonable, but there is probably no direct causal connection. Probably, the primary factor is the access of reactive silica in the stone, in relation to the alkaline metal hydroxide in the immediate surroundings. If the quotient silica/alkaline metal oxides in the gel can grow to a high level, the hydroxide ion concentration will be low enough to make the calcium hydroxide in the mortar soluble. The gel formed from the more calcium rich solution will contain more calcium than the gel formed from the strong hydroxide solution with almost no calcium ions.

It may be true that a gel with calcium ions is rigid, but this does not prove that the calcium ions make the gel rigid.

Tests were made with a scanning electron microscope with EDAX to find out whether the gel composition in the two specimens 10-B-4 and 20-B-3 were different with respect to silicon, potassium and calcium. However, the tests were not successful. The microscope could neither analyse the gel substance separated from the non-reactive

grains from the attacked test stone in 20-B-3, nor the thin layer of gel liquid separated from the mortar under the gel in 10-B-4.

6. The two most powerfully reacting stones, in the specimens 20-B-3 and 10-B-4, were cured at 93 per cent RH. This point corresponds to variable C. in Chapter 3.5.

7. The test stone in specimen 20-B-3, which had shaped a pop-out, had been dried at 85 per cent RH at 40° C. This point corresponds to variable D. in Chapter 3.5.

8. Re-humidification in water essentially leads to more of the small reactive grains reacting, than storing in moist air, but does not increase the number of "micro pop-outs". This point corresponds to variable E. in Chapter 3.5.

### 3.6 The influence of essential parameters

The effect of some essential parameters, such as concrete composition and the addition of a potential inhibitor, was dealt with by using the "moisture histories" that seemed to be most suitable in 3.4.

For the series 8-11, 14, 15 and 21-25 the following climates were chosen on the basis of previous results:

- o +20°C 93 % RH (over saturated  $\text{KNO}_3$ -solution)
- o +40°C 88 % RH ( " " " )
- o +20°C 100 % RH (over pure water)

the number of specimens being duplicated in the first climate. Series 8-11 were rewetted after one year and the others after six months. The results are summarized in TABLES IX-XI.

Sand R, produced no pop-outs in practice, showed only gel extrusions and only at +40°C 88%RH. In the other climates no sign of reaction was observed on the other discs. However, one of the prisms cracked, and on examination the crack showed that a single reactive grain had caused the crack. This sand obviously contains a small number of reactive grains and with a certain moisture history a few pop-outs may occur, cf. the additional test in 3.7. The petrographic examination performed previous to this research project indicated this result, Larsen et al (1979).

Two types of sand with unknown behaviour in practice, HG and B, were tested in series 14 and 15. Only two and one pop-out, respectively, were obtained, but they were rather large expansions and one of the prisms cracked. These two types of sand are obviously reactive and should be examined further. This is shown in 3.7.

In series 9-11 some of the cement was replaced with a Norwegian silica-fume (N) as a pozzolan. The percentage of cement replaced by the same amount of silica-fume will from now on be written as "% Si-replacement".



Some minor expansion was measured on single bars. Pop-outs were observed mainly at the elevated temperature in 88 % RH. With 10% silica-fume only very small pop-outs occurred and in series 11, with 15 % Si-replacement, only where alkali was added. One of the discs was submerged by mistake in the alkaline solution. The part of the disc that was submerged in the alkali showed some small pop-outs. Some of the discs are shown in FIG 3.6.1.

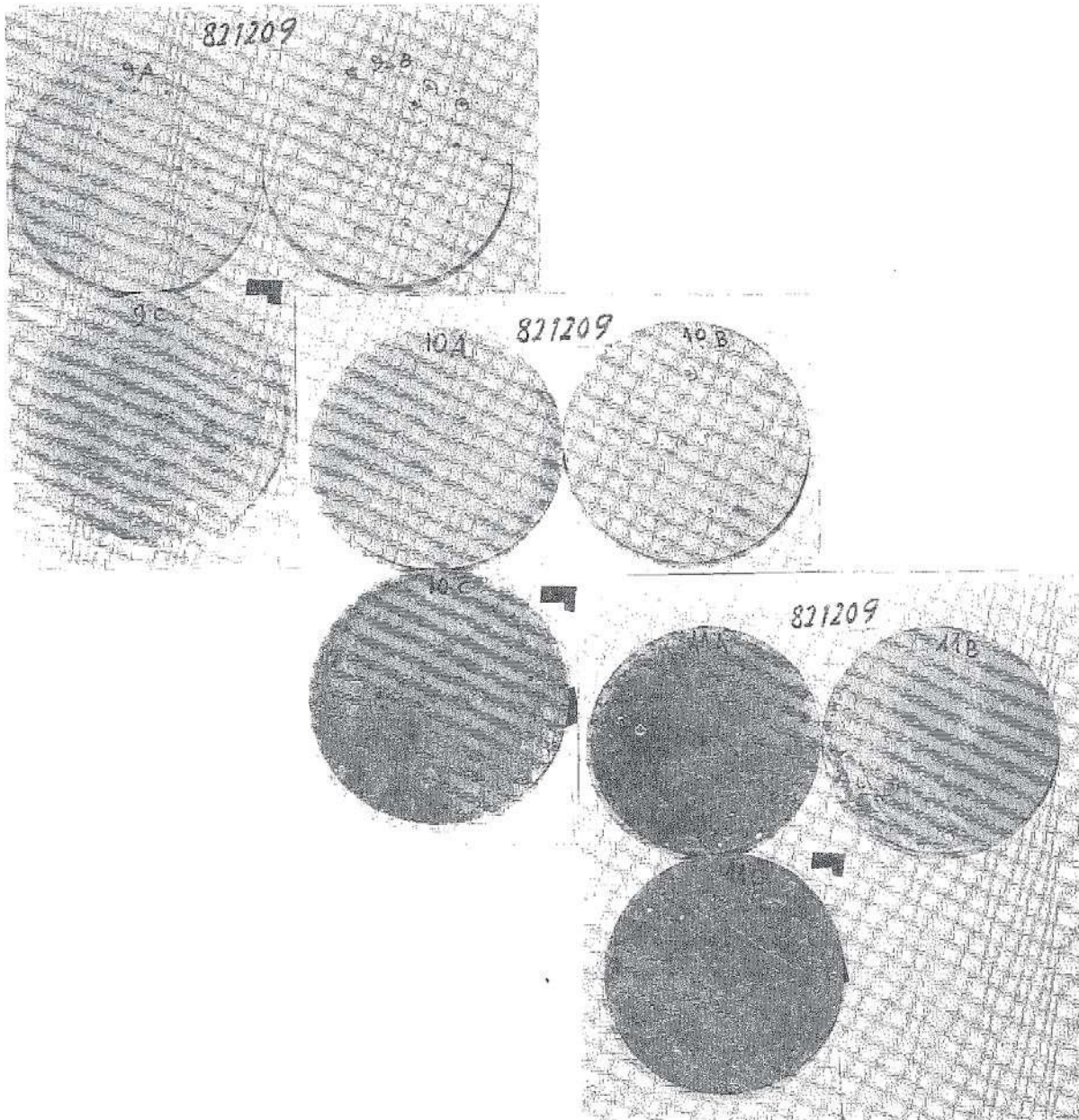


FIG 3.6.1 Discs in series 9-11 after rewetting. Previously stored at +20°C 93%RH (A), +40°C 88%RH (B) and +20°C 100%RH (C)

When the discs were rewetted after a year, more pop-outs were obtained on the discs that had been stored at  $+40^{\circ}\text{C}$  88%RH. With 15 % Si-replacement, however, only small pop-outs occurred with the exception of the part where additional alkalis were present.

The silica-fume N replacing 10 or 15 % of the cement does not seem to consume a sufficient amount of alkalis to protect small grains or larger grains containing small amounts of reactive material. The use of silica-fume N as a potential inhibitor was studied further in series 21-25 and later in series 61-63 another silica-fume was tested.

In series 21-25 the climate  $+40^{\circ}\text{C}$  88 % RH was clearly seen to be the most suitable suited for reproducing pop-outs. Pop-outs occurred only in that climate during the six month testing period and the following rewetting. This is obviously too short a testing period for the climate  $+20^{\circ}\text{C}$  93 % RH.

As can be seen in TABLE XI more than 5 % Si-replacement was needed to prevent larger pop-outs. With 10 % replacement by the silica-fume N only small pop-outs were obtained. This coincides to some extent with the results of series 9-11.

In series 26 an attempt to test the possible effect of alkali depositions in an evaporation zone was made. The discs were sealed as lids on glass-cups containing pure water, i.e. close to 100 % RH as shown in FIG 3.6.2.

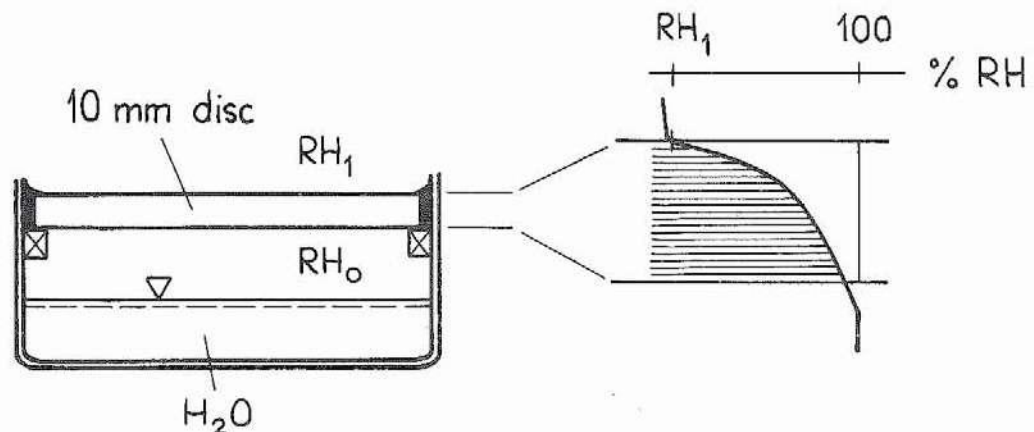


FIG 3.6.2 Method of applying a moisture gradient over the discs.

By placing the cups in boxes with different climates a moisture gradient was obtained over the discs. The moisture distribution over the discs is shown, in principle, in FIG 3.6.2.

The moisture flow should bring alkalis to the evaporation zone, situated at different depths in the different discs.

The upper (dry) surfaces of the discs are shown in FIG 3.6.3, with the continuously used climate indicated.

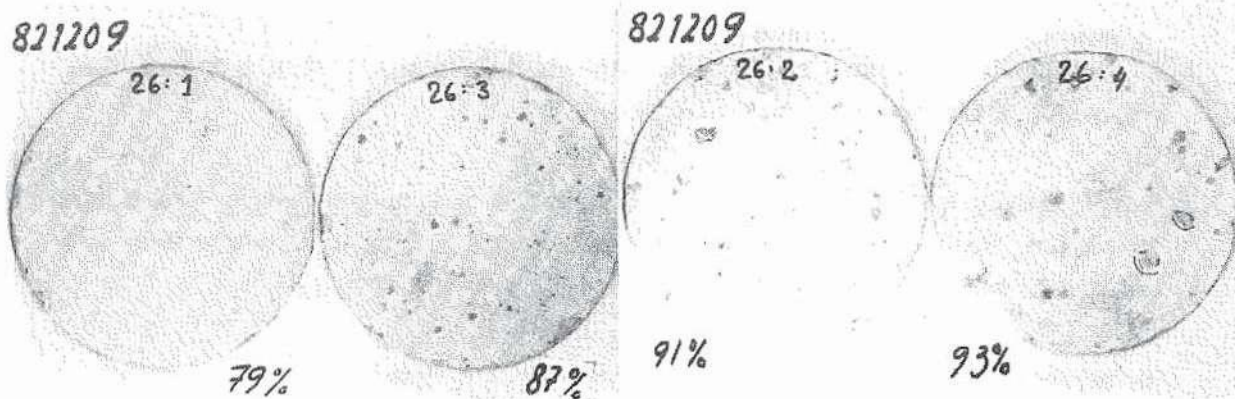


FIG 3.6.3 The upper surfaces of the discs in series 26, showing gel extrusions and pop-outs

With 79 % RH on one side only small signs of reaction are visible at the surface in the form of a few gel spots. With increasing humidity on that side, possibly corresponding to a less deep evaporation zone, the number of gel extrusions and pop-outs increases. On the bottom side no pop-outs at all occurred.

These results, however, coincide with the results from series 16 where the same climates were used on both sides. Consequently the test has not shown an effect of alkali movement on the pop-out formation.

To possibly improve the reproduction of pop-outs in the experiments, two additional moisture histories were used in the following series. Some of the earlier experiments indicated that moisture changes could promote pop-out formation. Therefore another climate at a temperature of +40°C, with 88 % RH

for two months and 100 % RH for one month etc., was used instead of duplicating the number of discs in 93 % RH at +20°C. The climate +20°C 100 % RH that did not produce any pop-outs was at the same time changed to +40°C 100 % RH, i.e. similar to the one used in the ASTM mortar bar method.

In series 27 the specimens were already dried after casting instead of being sealed cured for one week. Greater expansions were of course measured since the prisms dried to some extent before the first length measurement. The occurrence of pop-outs, however, was the same as in the previous series 9 and 21, where some pop-outs were obtained at +40°C 88 % RH. In this case some small pop-outs occurred in the new climate, +40°C 88/100 % RH.

In series 28 an unknown sand S8 was tested. Only a small single pop-out was obtained. A further test was made later, cf. 3.7.

A new cement, a high-alkali cement Sk with 4 % silica-fume added, was tested in series 29. A small expansion was measured but no pop-outs whatsoever were obtained. Further tests were made with this cement, cf. 3.7.

In series 30-32 and 41-43 an attempt was made to determine the effect of lower water-cement ratios and higher alkali contents in the pore solution.

Water-cement ratios of 0.4, 0.55 and 0.7 ("standard") were used and the additions of alkali were chosen to be equal to the amount of alkali present in mortars with the same cement contents but a pore system corresponding to the standard mortar with  $w/C$  for all alkali additions. One of the two contradicting effects of water-cement ratio, increased alkali content and decreased availability of alkalis due to smaller pores, was eliminated in this way in the later series 41-43.

The results are shown in TABLES XIII-XIV. Only when the  $w/C$  was as low as 0.4, or the corresponding alkali content (or higher), did some of the prisms crack.

Pop-outs, however, occurred in all the tested  $w/C$  and alkali contents but no definite differences were visible. A  $w/C$  of 0.4 was tested further in 3.7.

In series 51-53 the reactive sand was "diluted" by replacing some of the fractions < 2 mm with an unreactive quartz sand, "Åstorp". As shown in TABLE XV only when 100 % of the grains < 2 mm was replaced no pop-outs were obtained. This meant that the reactive sand did not show any pop-outs with only the fraction 2 - 4 mm remaining. The number of reactive grains is, however, considerably decreased in this way and perhaps no reactive grains at all were present in the discs. In one of the prisms obviously at least one reactive grain was present, causing a crack.

The results in 3.5 indicated, however, an influence of the presence of reactive material on the behaviour of a reactive grain. Rapidly available silica, e.g. small reactive grains, may promote the formation of a rigid gel, capable of causing a high pressure.

In the last series 61-63 another silica-fume (Dv) was tested. With 5, 10 and 15 % of the cement replaced the result was as shown in TABLE XVI. A Si-replacement of 5 % "prevented" larger pop-outs from occurring and with 10 and 15 % replacement neither pop-outs nor expansion were obtained. This silica-fume is obviously a more promising inhibitor than the one used in series 9-11, cf. TABLE VIII.

TABLE IX Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Series 8-11.

Series	Variable in the composition	Storing climate	Maximum expansion (°/oo)	Number of pop-outs	Size (mm)
8	Sand R ("harmless")	+20 C 93%RH	-(crack)	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	-	-	gel spots
9	Standard mortar	+20 C 93%RH	+0.5	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	-	6 (+ )	2-12
10	10 % Si-replacement	+20 C 93%RH	-	some small	-
		+20 C 100%RH	+0.2	-	-
		+40 C 88%RH	-	- (+ )	-
11	15 % Si-replacement	+20 C 93%RH	- (+0.4)	-	-
		+20 C 100%RH	+0.2	- <sup>*</sup>	-
		+40 C 88%RH	-	small <sup>*</sup> (+large <sup>*</sup> , small)	
*) where alkalis were added (+ ) after rewetting					

TABLE X Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Series 14 & 15: Sands with unknown behaviour in practice.

Series	Variable in the composition	Storing climate	Maximum expansion ( $\frac{\circ}{\infty}$ )	Number of pop-outs	Size (mm)
14	Sand HG ("unknown")	+20 C 93%RH	-	-	-
		+20 C 100%RH	+0.7, crack	-	-
		+40 C 88%RH	-	2	
15	Sand B ("unknown")	+20 C 93%RH	+0.3 (+0.5)	-	-
		+20 C 100%RH	+0.9	-	
		+40 C 88%RH	+0.2	- (+1)	(7)

TABLE XI Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Stored in different climates for 6 months; then rewetted. Series 21-25: Different Si-replacements.

Series	Variable in the composition: Si-replacement	Storing climate	Maximum expansion ( $^{\circ}/\infty$ )	Number of pop-outs	Size (mm)
21	0 %	+20 C 93%RH	(+0.7, crack)	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	(+0.3)	4 (+ )	small (+larger)
22	1 %	+20 C 93%RH	-	-	-
		+20 C 100%RH	(+0.4)	-	-
		+40 C 88%RH	-	4 (+ )	small (+larger)
23	2 %	+20 C 93%RH	-	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	-	few (+ )	larger
24	5 %	+20 C 93%RH	-	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	-	- (+ )	- (larger)
25	10 %	+20 C 93%RH	-	-	-
		+20 C 100%RH	-	-	-
		+40 C 88%RH	-	- (+ )	- (small)



TABLE XII Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Stored in different testing climates for one year.  
Series 27-29

Series	Variable in the composition or treatment	Storing climate	Maximum expansion ( $^{\circ}/_{oo}$ )	Number of pop-outs	Size (mm)
27	Standard mortar, never cured	+20 C 93%RH	+0.2	-	-
		+40 C 88%RH	+0.2	5	2-3
		+40 C 88/100%RH	+0.7	8	3
		+40 C 100%RH	+0.6	1	-
28	Sand Sö	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	-	1	6
		+40 C 100%RH	-	-	-
29	Cement Sk + 4 % Si-fume	+20 C 93%RH	+0.2	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	+0.2	-	-

TABLE XIII Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week.  
Series 30: Different water-cement ratios.

Series	Water-cement ratio	Storing climate	Maximum expansion (‰)	Number of pop-outs	Size (mm)
30	0.7	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	a few	small
		+40 C 88/100%RH	-	12	5
		+40 C 100%RH	+0.2	1	small
32	0.55	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	12	5
		+40 C 88/100%RH	-	6	5-10
		+40 C 100%RH	+0.2	-	-
31	0.4	+20 C 93%RH	-	1	-
		+40 C 88%RH	-	2	small
		+40 C 88/100%RH	+0.3, crack	10	small
		+40 C 100%RH	+0.4, crack	3	<10

TABLE XIV Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week.  
Series 41-43: Different amounts of alkali added

Series	Amount of alkali added (%Na <sub>2</sub> O <sub>eq</sub> )	Storing climate	Maximum expansion (°/oo)	Number of pop-outs	Size (mm)
40 (=30)	0 % ("standard") (corresponding to wo/C=0.7)	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	a few	small
		+40 C 88/100%RH	-	12	5
		+40 C 100%RH	+0.2	1	small
42	0.3 % (corresponding to wo/C=0.55)	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	7	5
		+40 C 88/100%RH	-	17	<10
		+40 C 100%RH	+0.3	-	-
41	1.0 % (corresponding to wo/C=0.4)	+20 C 93%RH	-	-	-
		+40 C 88%RH	+0.2, crack	6	5
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	+0.5	-	-
43	0.46 % (corresponding to a total of 1.5 %)	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	7	3-10
		+40 C 88/100%RH	+0.2	-	-
		+40 C 100%RH	+0.2, crack	-	-

TABLE XV Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Series 51-53: Different amounts of reactive sand replaced in the fractions 0-2 mm.

Series	Amount of sand replaced (% of fractions <2mm)	Storing climate	Maximum expansion (°/oo)	Number of pop-outs	Size (mm)
50 (=30)	0 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	a few	small
		+40 C 88/100%RH	-	12	5
		+40 C 100%RH	+0.2	1	small
51	25 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	7	2-10
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	-	-	-
52	50 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	3	4
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	-	-	-
53	100 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	+0.1, crack	-	-
		+40 C 100%RH	-	-	-

TABLE XVI Expansion measurements and observations of pop-outs on prisms and discs of different compositions. Sealed cured 1 week. Series 61-63: Different amounts of cement replaced with Silica-fume Dv.

Series	Silica-replacement	Storing climate	Maximum expansion (‰)	Number of pop-outs	Size (mm)
60 (=30)	0 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	a few	small
		+40 C 88/100%RH	-	12	5
		+40 C 100%RH	+0.2	1	small
61	5 %	+20 C 93%RH	+0.2	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	+0.1	4	small
		+40 C 100%RH	+0.1	-	-
62	10 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	-	-	-
63	15 %	+20 C 93%RH	-	-	-
		+40 C 88%RH	-	-	-
		+40 C 88/100%RH	-	-	-
		+40 C 100%RH	-	-	-

### 3.7 Checking of some suitable test methods

The above described experiments have not always given reproducible results for a number of reasons. Obviously expansion measurements are not a suitable tool when quantifying the effects of ASR when the number of reactive grains is as low as in the used aggregate.

Neither are of course small specimens very useful when the occurrence of pop-outs is studied. In practice some 50 pop-outs/m<sup>2</sup> have been observed in some of the case studies. The small discs used in the later series have an area of only 0.023 m<sup>2</sup> and consequently an average of only one pop-out a disc should be expected.

The number of pop-outs on such small discs is not easy to use when quantifying the effects of different variables. Specimens with larger test surfaces have to be used to obtain a quantitative answer. This was done when some preliminary test procedures were to be controlled.

Since the presence of reactive grains of different sizes and types sometimes is very limited and fractions up to 8 mm should also be included, the specimen size chosen was rather large, 300x400x22 mm, which meant a total test surface of approximately 0.25 m<sup>2</sup>.

To test the potential reactivity of combinations of cements and aggregates and the effect of potential inhibitors a test method has been outlined by using a number of testing climates close to the estimated pessimum humidity.

The specimens, the chosen climates and one of the climate boxes are shown in Figure 3.7.1.

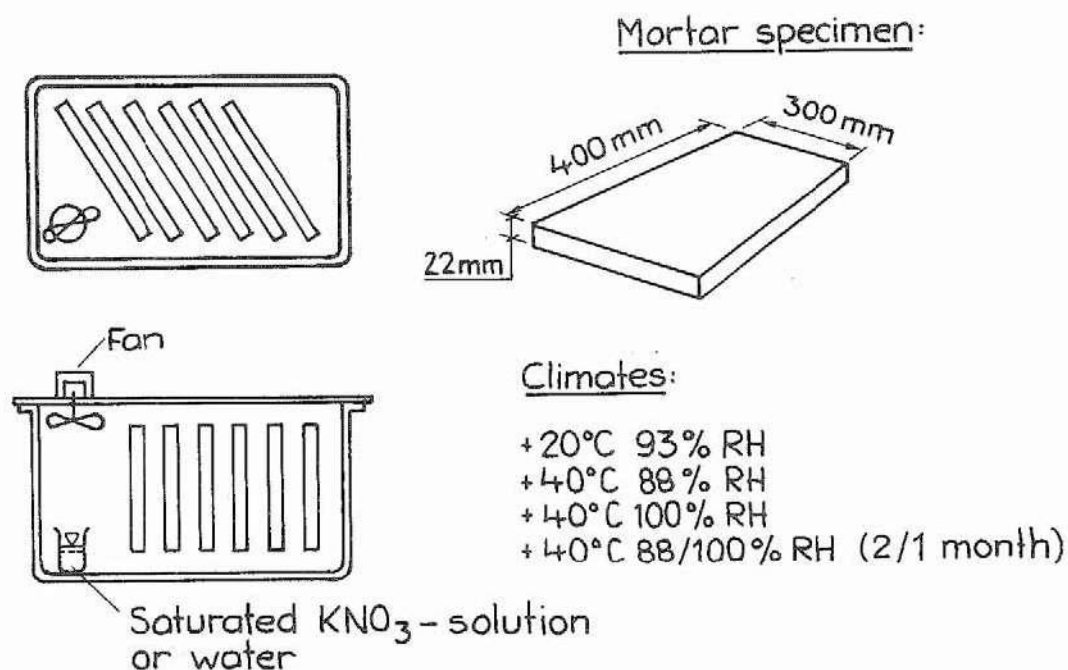


FIG. 3.7.1 Specimen, climate boxes and climates used in the test method.

Eight climate boxes were placed in a large chamber specially made for this experiment. The chamber was heat-insulated with 0.1 m expanded polystyrene and contained a number of heating-lamps and two fans. The climate boxes were placed in the chamber in such a way that they were surrounded on all sides by air at +40°C. Two similar climate boxes were placed in a climate room at +20°C. The temperatures in the boxes were recorded during the first months of the experiment.

Some sands, cements and potential inhibitors have been tested with the suggested method. The results after six months are summarized in TABLE XVII as numbers of pop-outs and their size. The mix proportions were: "cement": water:aggregate equals 1:0.66:4.77, where the amount of "cement" was sometimes a high-alkali cement mixed with various additional materials.

The sand HB is known to produce pop-outs in practice but no pop-outs have ever been reported where the sand R has been used, even though it contains reactive grains. With the HB-sand pop-outs were created in all of the testing climates, but with the unarmful R-sand very few pop-outs were obtained and in one climate only.

Of the three types of sand with unknown behaviour in practice, sand Sö must be classified as "harmless", as no pop-outs whatsoever were created. The other two, B and Hv, can be described as harmful or "possible sources of pop-outs".

All the tested cements have been producing pop-outs in all the testing climates, but different climates result in the greatest number of pop-outs for different cements. This shows why more than one climate should be used in a test procedure.

The potential inhibitors, slag, fly-ash and silica-fume, have been tested with a large amount of the high-alkali cement replaced. The effect may be mainly caused by the decreased total alkali content. Fly-ash and silica-fume seem to be promising inhibitors; only very small or very few pop-outs were created, and only in some of the testing climates. Other mix proportions, taking the required concrete quality into consideration, should also be tested before use in practice.

In FIG 3.7.2 an example of the occurrence of pop-outs measured by time is shown. The pop-outs are marked on the time-scale with circles, the diameters being proportional to the size of each pop-out.



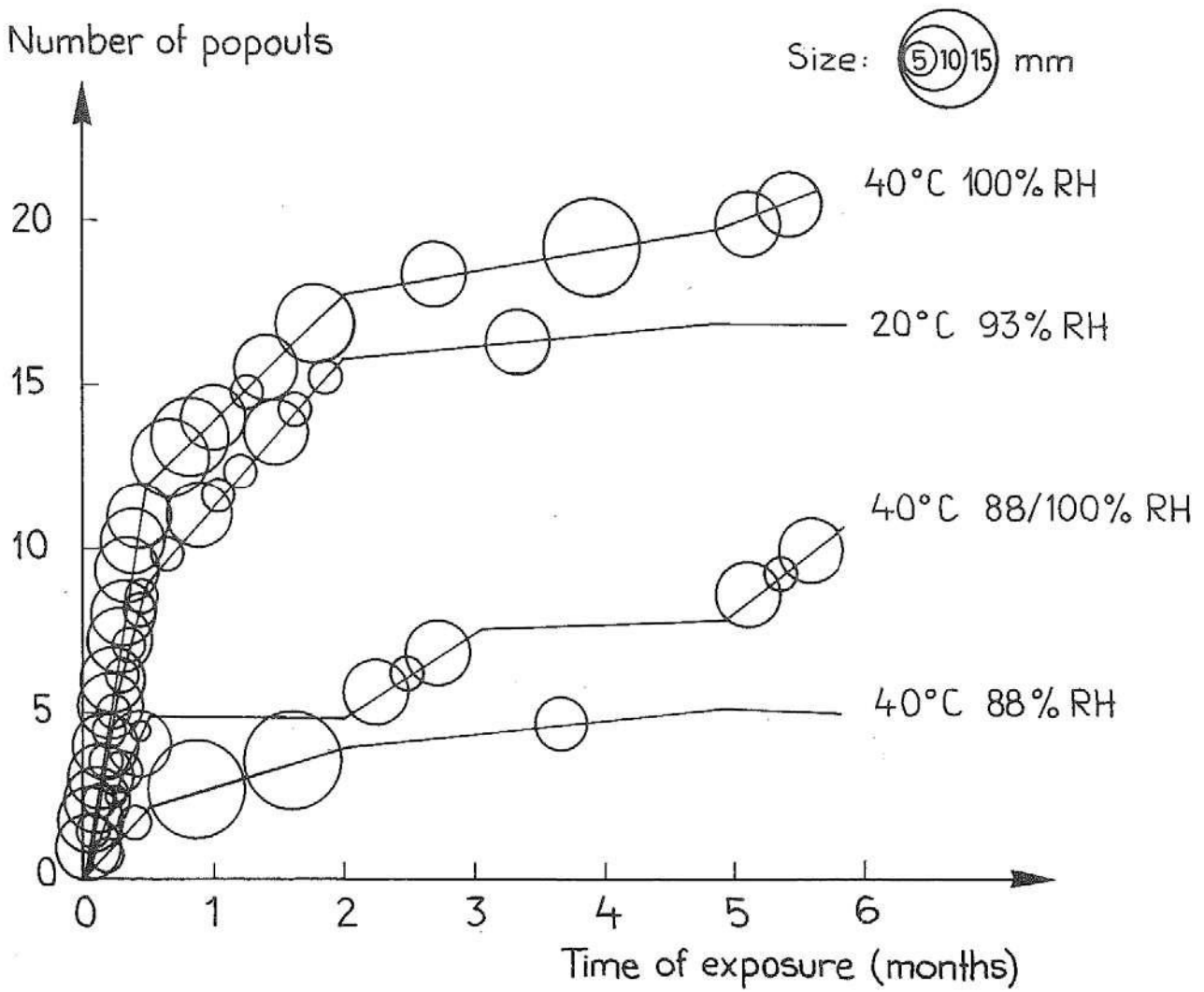


FIG 3.7.2 An example of the occurrence of pop-outs measured by time in different testing climates; cement D and sand HB.

TABLE XVII. Results from checking of the suggested test method

VARIABLES		NUMBER OF POP-OUTS (size in mm) after 6 months				
		Testing climate (after 2 months sealed curing)				
		Sealed at+20 C	93%RH at+20 C	88%RH at+40 C	88/100%RH at+40 C	100%RH at+40 C
<u>Sands: (cement S)</u>						
HB	number size	1 (10)	3 (5-10)	12 (5-10)	19 (5-20)	19 (5-15)
R	number size	0	0	0	3 (5-10)	0
Sö	number	0	0	0	0	0
B	number size	1 (10)	5 (8-15)	3 (5-15)	11 (4-15)	6 (4-20)
Hv	number size	1 (10)	3 (3-10)	1 (16)	17 (8-15)	2 (7-10)
<u>Cements: (sand HB)</u>						
S	number size	1 (10)	3 (5-10)	12 (5-10)	19 (5-20)	19 (5-15)
D	number size	9 (6-15)	18 (5-10)	5 (5-15)	11 (3-10)	23 (5-15)
Ss	number size	11 (5-15)	18 (5-25)	5 (8-30)	13 (5-15)	10 (8-20)
P	number size	1 (8)	3 (5)	11 (5-10)	11 (5-10)	4 (10-30)
<u>Additional materials: (cement S &amp; sand HB)</u>						
40 % slag added	number size	1 (5)	2 (2-5)	15 (3)	20 (3-5)	25 (3-5)
30 % slag -"-	number size	1 (3)	1 (5)	3 (3-5)	16 (5-10)	8 (5-15)
35 % fly-ash -"-	number size	1 (3)	0	0	0	3 (2)
4 % Si-fume -"-	number size	0	0	0	0	2 (15)
w <sub>o</sub> /C = 0.4	number size	16 (3-10)	26 (3-15)	27 (5-8)	40 (3-20)	36 (5-25)

#### 4 CONCLUSIONS

The effect of moisture on the alkali-silica reaction and expansion as concluded from the experimental results and the observations made in experiments and case studies, is briefly described in this chapter.

A test procedure for potential "pop-out capacity" is suggested in 4.2 and a classification system for the test results and their significance in practice is outlined.

##### 4.1 The role of moisture in ASR and the mechanism of pop-out formation

The experimental results and the experiences from case studies have greatly increased the knowledge about the influence of the local climate around the reactive grains. The theoretical assumptions made in 1.5 have been confirmed.

The conclusion is that, in order to reproduce pop-outs, it is of essential importance to have a rather long drying time, at a relatively high humidity. Further, a subsequent rewetting should only be slow or the humidity increase should only be small. Too wet conditions do not create any pop-outs, only gel extrusions. The time needed is shortened very much by using a higher temperature during drying, but the moisture dependence is somewhat changed at higher temperatures.

All the experimental results, as far as moisture effects are concerned, can be summarized in principle as shown in FIG 4.1.1.

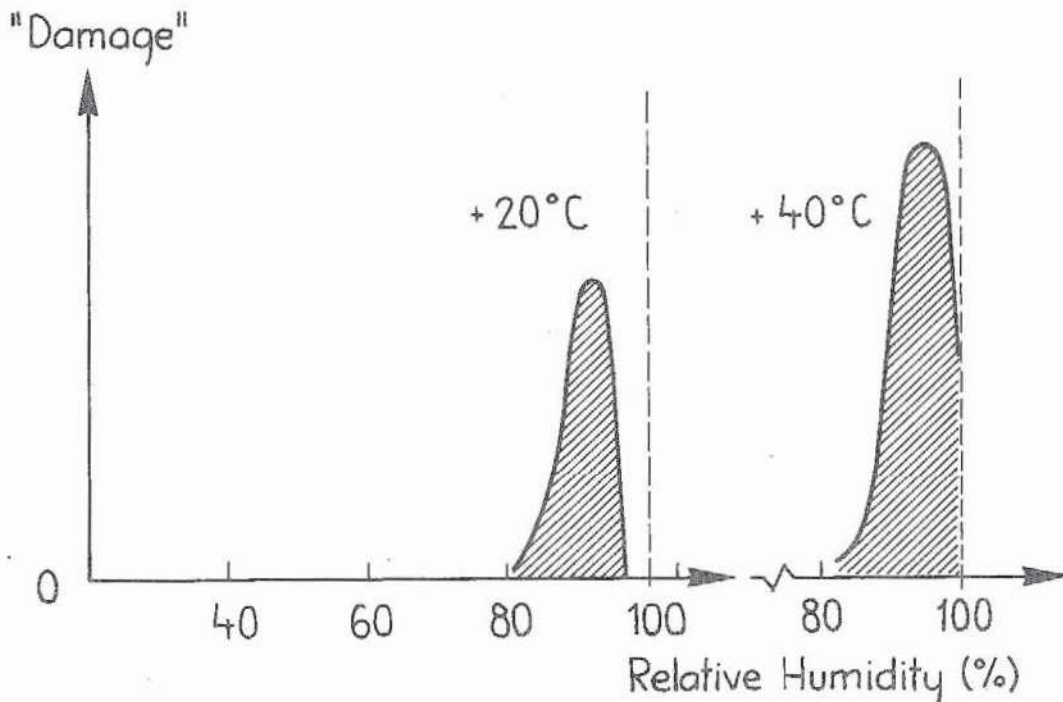


FIG. 4.1.1 The pessimum effect of moisture, in principle, at room temperature and elevated temperature

Some of the experiments indicate that the reaction can actually continue at such a low humidity as 80 % RH. The reaction is slow due to the limited availability of alkalis as there is hardly any water in the capillary pores. The number of paths for the diffusion of alkalis in the pore solution is few. The reaction product, the gel, is rigid but cannot absorb any water at this low humidity. Consequently the "damage", expansion or pop-outs, in FIG 4.1.1 is zero. At least part of the reaction may, however, take place during the accommodation phase, when the mortar specimens contain more water than in equilibrium with 80 % RH. It is only a matter of one or two weeks before the specimens reach moisture conditions very close to equilibrium.

At an increasing humidity the reaction rate increases due to the increased availability of alkalis. The increased amount of water in the pore system should dilute the concentration of alkalis in the pore solution, as estimated in 1.5, but no measurements confirming this have been found in the literature.

At +40°C a higher relative humidity is needed to fill the pore system to the same degree as that at +20°C in order to maintain the availability of alkalies by a continuous water phase. At a constant humidity this effect opposes the effect of increased temperature.

Simultaneously the reaction product of the rapid reaction will be more fluid when the humidity increases. The creation of a rigid gel ought to have a pessimum somewhere between 85 and 95 % RH depending on the temperature and the alkalinity of the pore solution in the vicinity of the reactive grain.

The absorption of water by the gel increases with an increasing humidity and consequently the gel ought to expand. The viscosity of the produced gel, however, decreases when the humidity increases. The gel becomes more fluid and penetrates the surrounding cement matrix without causing any expansion or pop-out if the humidity becomes too high. The "damage" in FIG 4.1.1 is also zero between 95 and 100 % RH at +20°C.

A small humidity increase or a slow humidification, however, will be able to create the highest pressure, provided the properties of the gel are suitable. The effect of moisture obviously has a pessimum close to 90 % RH at room temperature, cf. Figure 4.1.1.

The pessimum effect at +40°C seems to be present at a somewhat higher humidity than at room temperature as shown in the Figure. Very wet conditions may create pop-outs at elevated temperatures as shown in the experiments. A rigid gel with a high molar ratio of silica/alkali metal oxides has a lower concentration of hydroxide ions and is normally created slowly. At a higher temperature this formation will be more rapid. A gel with such a composition will be less fluid in wet conditions and consequently still maintains its capacity to create a high pressure.

Of course the effect of moisture is more complicated than shown in FIG 4.1.1. It is not only the continuously used humidity that matters, but the variation with time. Changes sometimes result in more pop-outs than a constant climate especially if the changes are from a humidity lower than the pessimum humidity to a humidity just above the pessimum.

Changes in this way may also make a pop-out "grow". If a rigid gel is created absorption of a small amount of water causes some expansion. If this expansion is not sufficient for a pop-out to occur a following drying to a level below the pessimum means that no gel escaped because it did not become too fluid.

Even if the gel shrinks to some extent when drying the slowly continuing reaction can produce more rigid gel and fill the space that may have been created at the first small expansion. The gel slowly increases its "working capacity" and eventually a visible pop-out has been created.

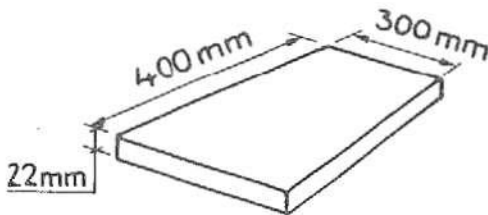
The moisture history required to obtain as much pressure out of a reactive grain as possible is precisely the same as close to the surface of a concrete slab towards the ground, or a slab containing a lot of excess building moisture, if the slab is covered by a vapour tight surface cover. This is also where the pop-outs have been obtained in practice as described in Chapter 2.

#### 4.2 Suggested test method for pop-outs

The potential pop-out capacity of a cement/aggregate combination may be tested with the following test method, suggested on the basis of the results presented above.

##### 4.2.1 Test specimens

With the composition to be tested eight (Simplified test: two) mortar specimens should be prepared by casting in horizontal forms to a specimen size of approximately 400 x 300 x 20 mm. The specimens should be prevented from drying and when the bleeding has stopped the upper surface should be floated and then steel trowelled.



The specimens should be kept wet for one day using e.g. wet sacks arranged in a way that no dripping at the surface can occur.

##### 4.2.2 Curing

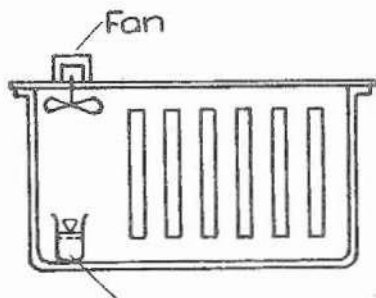
After demoulding the test specimens should be sealed cured in plastic bags of 0.2 mm polyethylene foil. The curing time should be four weeks at +20°C.

##### 4.2.3 Testing climates

Two specimens should be stored continuously for at least six months in each of the following climates (Simplified test: in the third climate only):

- Climate 1) +20°C 93 % RH
- 2) +40°C 88 % RH
- 3) +40°C 88 % RH 2 months + +40°C 100 % RH 1 month etc.
- 4) +40°C 100 % RH

To obtain these climates sealed climate boxes containing a fan should be used. The fan motor must be located outside the box. The different climates



Saturated  $\text{KNO}_3$  - solution  
or water

are obtained by placing a beaker in each box containing a saturated  $\text{KNO}_3$ -solution with an excess of salt or containing pure water.

The climate boxes should be placed in a well-insulated oven, chamber, or room with a constant temperature of  $+20^\circ\text{C}$  or  $+40^\circ\text{C}$ , respectively. Temperature changes must not exceed  $\pm 2^\circ\text{C}$ .

Rewetting after at least six months is carried out by changing the salt solutions to beakers containing pure water. If a longer test period is desired one of the duplicate specimens can be rewetted after six months and the other can continue in the different climates for another six months.

#### 4.2.4 Observations

The specimens should be examined after curing for gel extrusions and the number and amount noted.

The specimens should then be examined once every month and the salt solution checked. The number and sizes of new pop-outs obtained and older ones that have grown should be noted and marked with a lime-proof ink.

The last observation should be made not earlier than one month after the specimens were rewetted.

#### 4.2.5 Evaluation

In the following TABLE XVIII a classification system is suggested for the evaluation of the tests performed. Some comments on the possible behaviour in practice of the tested cement/aggregate combination are made, based on the field experience of some tested material combinations.



TABLE XVIII Suggested classification of the "potential pop-out creativity" of the cement/aggregate combinations tested.

Pop-out creativity	Test result	Possible field behaviour
0	No gel extrusions, no pop-outs	Unreactive
1	Gel extrusions, but no pop-outs	Pop-outs not likely to occur
2	A few very small pop-outs (<5mm)	Pop-outs not likely to be noticed
3	Very small pop-outs only (<5mm)	Pop-outs not likely to be of any great importance
4	A few larger pop-outs (5-20mm)	
5	Several larger pop-outs (5-20mm)	Pop-outs likely to be a problem in "moist indoor structures"
6	Several, some very large, pop-outs	

#### 4.2.6 Precautions

Six months may be a too short a period for large reactive grains to react sufficiently. If an examination of the fraction 4-8 mm indicates possible reactive grains a negative test could be carried out with the gel-pat test. Similar results may be obtained if the specimens are sliced after the test procedure described above. Microscopical examination of larger grains will show the behaviour of the larger grains. If some larger grains have reacted, large pop-outs on certain structures may occur in practice.

Pessimum effects must be taken into account when the result of one cement/aggregate combination is evaluated and translated to field practice. Other cement/aggregate combinations or concrete compositions, using the same constituents, are not necessarily harmless if one combination has been tested and shown to be unreactive.

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