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THE CHEMICAL EFFECTS ON CEMENT MORTAR OF SOLUTIONS (two different concentrations) OF CALCIUM MAGNESIUM ACETATE AND SODIUM CHLORIDE

Olof Peterson

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Preface

This work has been proposed by professor Göran Fagerlund. The aims are two:

- 1. To test a quality of calcium magnesium acetate that is claimed to generate less dust and to be less deleterious against cement mortar (and concrete).
- 2. To test, whether calcium magnesium acetate, or sodium chloride, possibly is more deleterious against cement mortar when used in solutions with moderate or low concentration, than the salts are in maximally concentrated solution.

The previous test (Olof Peterson, 1991) showed that a saturated, or nearly saturated (4,5 mol/liter) solution of calcium chloride does not damage cement mortar. A moderately concentrated (3,0 mol/liter) solution destroys the mortar within a few days.

Experience seems to show that a certain concentration of a sodium chloride solution is more deleterious against concrete in freezing tests than a lower, or higher, concentration. For certain concrete qualities, this pessimal concentration is claimed to be about 3 % by mass, and this concentration is chosen as the lower for sodium chloride solution. For calcium magnesium acetate, a concentration was chosen (6,5 gram per 100 gram water), at which the freezing point depression was the same as in 3 % sodium chloride solution.

Mr Bo Johansson has prepared the test mortars and casted the test specimens, and he also toke care of their curing, and photographic documentation.

Mr Sture Sahlén acquired the plastic boxes, necessary for storing the test specimens in the different liquids, and Mr Ingemar Larsson arranged with reasonably tight plastic lids for the largest containers.

Mr Wojciech Roszak answered for following the length of the slender test prisms during all the work.

Mrs Britt Andersson shaped the front pages, and she designed the diagrams for elucidating the results.

Professor Göran Fagerlund, finally, has spended much work for detecting and eliminating errors in the logic flow.

I express my thanks to all them who have helped me in this work.

Lund, October, 1992.

	Contents	Page
1.	Summary Introduction	1
2. 2.1 2.2 2.3 2.4 2.5 2.6	Salt solutions Arrangement of the bath Water Sodium chloride, 3 % solution Sodium chloride, saturated solution Calcium magnesium acetate, 6,5 g/100 g water Calcium magnesium acetate, 38,35 g/100 g water	3 4 4 4 4 6
3. 4. 5.	Model concretes Casting of the test specimens Curing of the test specimens Storing of the test specimens	6 8 8
7. 8. 8.1 8.2 8.2.1 8.2.2 8.2.3 8.2.4	in salt solutions at 5 and 20° C Measurements for comparison Length change Method Results and comments	9 10 10 11 12 16 16 17 18
9.2.3	Sodium chloride, 3 % solution Sodium chloride, saturated solution Calcium magnesium acetate, 6,5 g/100 g water	22 22 23 25 25 26 27 28
10.1 10.2 10.2.1 10.2.2 10.2.3 10.2.4	pH Values of the storing baths Method Results and comments Water Sodium chloride, 3 % solution Sodium chloride, saturated solution Calcium magnesium acetate, 6,5 g/100 g water Calcium magnesium acetate, 38,35 g/100 g water	28 29 29 29 30 30 30
11. 11.1 11.2 11.3	Chemical analysis of calcium and magnesium in calcium magnesium acetate solutions Method Results Comments	31 32 33 33
12	Conclusion	35 36

Contents (continued)

APPENDIX

Results in tables

Table A.1	Length changes, mm/m Water Sodium chloride solution, 3 % by mass "- , saturated Calcium magnesium acetate, 6,5 gram per 100 gram water "-, 38,35 gram per 100 gram water	A-1
Part 1 Part 2	Water cement ratio 0,45 + 5 deg. C +20 deg. C Water cement ratio 0,60	A-1 A-3
Part 3 Part 4	<u>-</u>	A-5 A-7
Table A.2	<pre>Mass changes, gram Water Sodium chloride solution, 3 % by mass "- , saturated Calcium magnesium acetate, 6,5 gram per 100 gram water "-, 38,35 gram per 100 gram water</pre>	A-9
Part 1	Water cement ratio 0,45 + 5 deg. C +20 deg. C Water cement ratio 0,60 + 5 deg. C	A-9 A-11
	+20 deg. C	A-15

Summary

Calcium magnesium acetate (CMA) has been tried as a less harmful de-icing agent than sodium chloride to structural steelwork in bridges and to reinforcement steel in concrete. A pre-study was made at which the potential aggressivity of a mixture of calcium acetate and magnesium acetate, or pure calcium acetate, was investigated.

During and after this work for the Swedish National Road Administration, which was reported by O Peterson in 1991, Mr Kent Gustafson at the Swedish Road and Traffic Research Institute in Linköping told us that a less deleterious quality of calcium magnesium acetate was supplied by BP Chemicals in England.

In this work we had the opportunity to test this new quality, and in the same time the following changes were performed in the test program in comparison with the previous study: 1. The calcium acetate and calcium chloride solutions were excluded. 2. The sodium chloride was tested as saturated solution and also as a 3 % solution. 3. The calcium magnesium acetate was tested as maximally concentrated solution, and also at a concentration of 6,5 gram per 100 g water, which solution has the same freezing point depression as 3 % sodium chloride.

The following changes in test results in comparison with the previous study were observed:

- 1. The molar ratio Mg/Ca was 1,10. For comparison, it was only 0,80 for the product from the Netherlands, used in the previous work.
- 2. The expansion of prisms stored in concentrated calcium magnesium acetate solution disappeared for all combinations except for mortar with water cement ratio 0,60 at +20°C.
- 3. When prisms were stored in low concentration solution of calcium magnesium acetate, they expanded, maximally, 0,6 mm/m during six months. Mortar with water cement ratio 0,45 expanded more than 0,60 mortar. Storing at +20°C caused a more rapid expansion than storing at +5°C.
- 4. The prisms, after storing in highly concentrated solution of calcium magnesium acetate, did not lose any mass at +5°C, but their increase in mass was less than during storing in water or sodium chloride solution. At +20°C, the prisms lost in mass during six months much more than in the previous work; about 19 % of their total mass for 0,45 mortar, and 24 % for 0,60 mortar.

5. After storing in the low concentration solution of calcium magnesium acetate for six months, the prisms increased in mass essentially more than after storing in water or sodium chloride solution. Probably, calcium hydroxide in the mortar has formed a chemical compound with calcium acetate, which explains the gain in mass and the expansion.

1. <u>Introduction</u>

In a previous work, "The chemical effects on cement mortar of solutions of calcium magnesium acetate and other deicing salts", the author, Peterson (1991), showed that the grade of calcium magnesium acetate, used in that work (a blend of calcium acetate and magnesium acetate, probably made by dissolving natural dolomite in acetic acid), attacked cement mortar seriously, especially at elevated temperature (+20°C).

In a telephone call on october 20, 1989, Mr Kent Gustafson, the Swedish Road and Traffic Research Institute, in Linköping, told us that BP CHEMICALS in England produced a grade of calcium magnesium acetate that did not stick or dust. It was claimed to contain the same number of molecules of calcium and magnesium and to be much less harmful to concrete than the grade with higher calcium content, which was used in the previous work. However, this favourable experience was gained when using a solution with a concentration of 6 - 8 percent by weight. Mr Gustafson supplied an amount of the BP CMA for our test.

In the previous study we experienced that the concentration of calcium chloride solution strongly influences its ability to attack cement mortar. This observation gave professor Göran Fagerlund the idea to propose that sodium chloride and calcium magnesium acetate should be tested in two different concentrations, one of them equivalent to a 3 per cent solution of sodium chloride, the other saturated (or as high concentration as possible).

As in the previous work, two qualities of cement mortar have been tested at two different temperatures, +5° and +20°C. A reference test was performed with water without any salt addition. Four sets of test specimens were tested in salt solutions in accordance with the following chapter.

Salt solutions

2.1 Arrangement of the bath

All solutions were stored in boxes of polypropene plastic, and the volume of each bath was 20 liters. During the storing, the box was covered with a plastic lid in order to avoid evaporation of water and absorption of carbon dioxide from the air. A rubber seal served to reduce the exchange of air in the space below the lid. The efficiency of this seal was not perfect, but it was very much improved by a set of six clamping screws.

No stirring of the solutions was made during the test time (8 months), except for the examinations of the test specimens, and nor any change of the solutions. The composition of the calcium magnesium acetate solutions were checked before the storing, and after the termination of the test. The results are accounted for in the Chapter 11.

2.2 Water

As an innocuous reference liquid was used normal drinking-water from the Lund network.

2.3 <u>Sodium chloride</u>, 3 % solution

Sodium chloride was used as a reference substance for a commonly used deicing agent.

For the low concentration bath was used 3 gram of sodium chloride in 97 gram of water, because this solution is experienced to have a specially severe action at tests for salt scaling of concrete (Verbeck, Klieger 1957).

0,62 kg of sodium chloride (NaCl) puriss (Kebo 1.3383) was dissolved in 20 liter of water from the Lund network for each of the two boxes (+5 and +20°C).

2.4 <u>Sodium chloride</u>, saturated solution

The high concentration bath was, quite as in the previous study, a saturated solution.

7,03 kg of sodium chloride was dissolved in 20 liter of water for each of the two boxes.

2.5 <u>Calcium magnesium acetate</u>, 6,5 g/100 g water

The concentration was chosen so that the freezing point depression of the solution should be equal to that of the dilute sodium chloride solution (Subchapter 2.3).

The expected depression in a 3 % sodium chloride solution is

 $1,86 \times (1000/970) \times 2 \times 30/58,5 = 1,97 \text{ K}$

where

1,86 is the freezing point depression, in K, for 1 mol in 1000 gram of water

58,5 is the formula mass of sodium chloride

2 is the number of ions generated from one molecule of sodium chloride

If the calcium magnesium acetate is supposed to have the chemical formula

CaMg(OCOCH₃)₄

the formula mass would be

40+24+4(16+12+16+12+3)=300

and each molecule should generate 6 ions after complete electrolytical dissociation.

For a wanted freezing point depression of 1,97 K, a mass of

(300/6)*1,97/1,86=53 gram

would be necessary.

For correcting possible errors as to the exact composition of the calcium magnesium acetate delivered, the following test was performed.

Two 200 ml containers of soft plastic were loaded with each 100 gram of deionized water.

In one of the containers 3,1 gram of sodium chloride was dissolved, and in the other 5,3 gram of CMA. The two containers were placed in a freezer, adjusted to a temperature of $-20\,^{\circ}\text{C}$, and the temperature in the solutions were registered.

The sodium chloride solution showed a depression of 2,10 K, and the CMA solution 1,72 K.

In order to get a depression of 2,10 K in the CMA solution

5,3*2,10/1,72=6,47 gram

6,5 gram CMA should be dissolved in 100 gram of water.

Thus, 1,3 kg of granulated CMA, delivered from BP Chemicals, Saltend, Hull, England, was added to 20 liter of water for each of the two boxes.

As the dissolution is rather complete, an analysis of the content of calcium and magnesium gives a relatively good picture of the composition of the BP type of CMA.

2.6 <u>Calcium magnesium acetate</u>, 38,35 g/100 g water

7,67 kg granulated CMA was added to 20 liter of water from the Lund network for each of the two boxes.

At this concentration, part of the magnesium is precipitated as magnesium hydroxide. A filtered and clarified solution was used for analysis of the calcium and magnesium content. By comparison with the low concentration solution it is found out how much of the magnesium content that is precipitated.

3. <u>Model concretes</u>

As in the previous work, test specimens with the moderate sizes $4 \times 4 \times 16$ cm were used. This put a limit for the maximal size of the aggregate grains.

Mortars with the range of 0,08 to 2,0 mm for the aggregate were chosen.

An ordinary Portland Cement was used, Slite Standard Portland Cement from CEMENTA AB. The chemical composition corresponds to:

$$K_2O = 1,25 %$$

$$Na_{2}O = 0,22 %$$

$$SO_3 = 3,1 %$$

The clinker typically contains:

$$CaO = 63,9 %$$

$$SiO_2 = 21,3 %$$

$$Al_2O_3 = 4,6 %$$

$$Fe_2O_3 = 2,4 %$$

$$MgO = 4,2 %$$

The aggregate was composed from three fractions of sand, all of them with rounded quartz grains:

- 1. Fine sand 0,08-0,5 mm
- 2. Medium sand 0,5 1,0 "
- 3. Coarse sand 1,0-2,0 "

We composed the mortar with the water cement ratio 0,45 starting from the CEMBUREAU mortar, the water cement ratio of which, however, is 0,50.

The water content was reduced from 250 to 240 gram, and the cement content was correspondingly increased from 500 to 533 gram. The three sand fractions were kept at each 500 gram, quite like the CEMBUREAU mortar. Thus, we got the desired 0,45 mortar.

For composing the 0,60 mortar, we proceeded in the following manner:

- 1. The contents of water and of the Medium and Coarse sand fractions were similar to the corresponding amounts of the 0,45 mortar.
- 2. The cement contents was reduced from 533 to 400 gram.
- 3. The contents of the Fine sand was increased in order to compensate for the loss in solid volume of the cement. The density of cement is assumed to be 3100 and the density of the sand 2600 kilogram per cubic meter. Thus, the contents of Fine sand was adjusted from 500 to 612 gram.

The principal purpose of this special way to compensate for the reduced cement contents was to get two mortars with different water cement ratios, and still with small, and equal, tendency to separate water on the mortar surface after the casting. Thus, the object was not so much to give the two mortars exactly the same consistency. In this way, there is a fair chance for the specimens to keep their water cement ratio undisturbed by water separation after the casting.

The two batches are presented in the table below:

Water cement	ratio in mortar	0,45	0,60
Water	gram	240	240
Cement	gram	533	400
Fine sand	gram	500	612
Medium sand	gram	500	500
Coarse sand	gram	500	500

Each of the two batches corresponds to a mortar volume of about 0,75 liter.

The mixing was done principally in the same manner as when mixing the CEMBUREAU mortar.

4. Casting of the test specimens

For each mortar, triplicates of test specimens were made for storing in the five test liquids at two different temperatures, Thus, 30 specimens were cast of mortar with water cement ratio 0,45, and 30 specimens of the 0,60 mortar.

For the determination of the mass of the specimens, $4 \times 4 \times 16$ cm test specimens were cast. The mould was filled to somewhat more than half of its height, and the mortar was compacted with a short period of vibration on a vibrating table.

Then the residual volume of the mould was filled, and the mortar was vibrated and steel-trowelled.

The mould was stored for two days in a closed plastic bag, and after this time the test specimens were stripped and weighed for the first time.

A separate series of prisms with the sizes 2 x 2 x 28 cm was cast for measuring the change in length. For this purpose, available moulds were used, some of steel, and some of acrylic plastic. The prisms had dowel rods with plane ends, and the length was checked with a dial indicator between spherical measuring surfaces. 30 slender prisms were cast from mortar with a water cement ratio of 0,45, and 30 from 0,60 mortar.

After two days in plastic bags, the prisms were stripped and their length measured for the first time.

5. Curing of the test specimens

The test specimens were stored for 3-6 days in water from the Lund network. No calcium hydroxide was added. After this, the $4\times4\times16$ centimeter prisms were weighed for the second time, and the length values of the slender prisms were measured.

The specimens were carried over to boxes with air, the relative humidity of which was 75 per cent (wet sodium chloride) at $20\,^{\circ}\text{C}$. They were stored there until a total age of 28 days from casting. The 4 x 4 x 16 centimeter prisms were weighed for the third time, and the length values of the slender prisms were measured.

No analysis was performed as to the degree of carbonation of the cured test specimens.

6. Storing of the test specimens in salt solutions at 5 and 20 °C

The test specimens were immersed in the baths after 28 days of curing.

In the Chapter 2, with its subchapters 2.2 - 2.6, the liquids for the storing test are described.

Each liquid batch of 20 liters was made in duplicate, one was stored in a climate room at +5°C and the other in a room at +20°.

Each plastic box contains:

- One small plastic box containing three prisms 4 x 4 x 16 cm of mortar with the water cement ratio 0,45. This box was intended for taking up material, if any, lost from the prisms during the storing.
- 2. Another small plastic box containing three prisms of the same size of 0,60 mortar.
- 3. Three prisms 2 x 2 x 28 cm with dowel rods. The prisms are made of mortar with a water cement ratio of 0,45.
- 4. Three similar prisms of 0,60 mortar.

7. <u>Measurements for comparison</u>

The purpose of the work was to investigate the effects of the different solutions on the mortars. Therefore, the test specimens were investigated after reasonable time intervals, normally one month.

* Length change. The 2 x 2 x 28 cm prisms had dowel rods with plane ends, and the length was checked with a dial indicator between spherical measuring surfaces.

The absolute length was checked against a calibration rod.

* Mass change.

The 4 x 4 x 16 cm prisms were wiped free from liquid with an absorbing non-wowen cloth, and the mass was determined with a balance, the first two times with a precision of 0,1 gram, but later always with a precision of 0,01 gram.

The mass value was compared with the value, determined after the termination of the water curing. (Wheighing nr 2).

* pH value.

A sample was taken from each 20°C bath, and the pH value was determined at room temperature by means of a glass electrode.

* Chemical analysis

Solutions of calcium magnesium acetate, concentrated as well as dilute, were analyzed for calcium and magnesium, in freshly prepared state and after storing of cement mortar prisms for 9 months.

8. <u>Length change</u>

8.1 Method

In order to follow any length change, a separate set of prisms had been made, see chapter 4. Their size was 2 x 2 x 28 cm, to be compared with 4 x 4 x 16 cm for the main series of prisms.

This choice of size of the measuring prisms was based upon two reasons:

- * If chemical reactions between prisms and bath occur, which cause the length of the prisms to change, the solution will sooner reach the core of each prism, when the prism is slender.
- * The measurements of length changes, made with a dial indicator and presented in millimeter per meter, obtain a better precision with a 28 cm prism than with a 16 cm prism.

The length of the fresh prisms, before storing in the bath, was read three times with a dial indicator, in millimeters:

- 1. Directly after the stripping of the prisms, two days after the casting.
- 2. After curing for six days in water.
- 3. After storing in air with a relative humidity of 75 % to a total age of 28 days, immediately before the immersion in the solutions according to the Chapter 2.

A weak tendency of these three readings was that

- * reading No 2 gave a somewhat greater value than reading No 1. This is a result of a slight water absorption of the prisms.
- * reading No 3, again, gave a somewhat smaller value than reading No 2. This is a result of the evaporation of water that followed storing in air with limited relative humidity. Value No 3 was not far from No 1, but could be slightly less or slightly greater.

Since reading No 2 was done after storing in water, this reading was selected as a reference for all further readings, which were performed with suitable intervals during the immersion period.

The difference, in millimeters, between each of the following readings and the reference, reading No 2, was noted in the table Appendix A.1.

The three values for each set of prisms were divided by 0,28 for transforming the length change from millimeters to millimeters per meter. Then the arithmetic mean and the standard deviation were computed and entered in the table.

8.2 Results and comments

The results are found as Table A.1 in the Appendix. This table is divided into four parts, one for each combination of water cement ratio and storing temperature.

In order to facilitate the understanding, four diagram sets, one for each part of the table, are entered in this subchapter, 8.2.

Not all results from each table are plotted in the corresponding diagram set. A main condition for approving a value for plotting is that its standard deviation must not be greater than the aritmethic means of the three values for each set of prisms.

For each solution the results are collected in a separate Paragraph, designated 8.2.X, where X may be 1, 2, 3, 4, or 5. In those Paragraphs, there is explained why a point, or a whole diagram, is excluded.

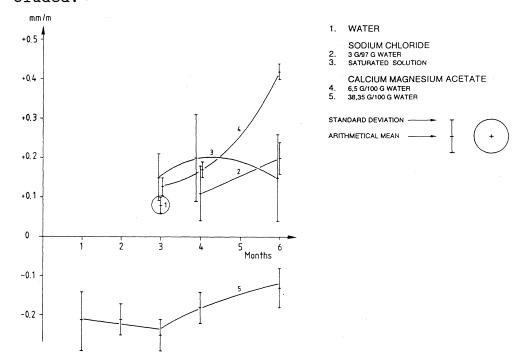


Figure 8.1: Length change in mm/m for mortar prisms; water cement ratio 0,45 stored at +5°C.

8.2.1 <u>Water</u>

The reference bath was water from the Lund network. Water is not expected to cause, neither expansion nor shrinkage.

In the four diagram sets in the Figures 8.1-8.4 there is not much information about the length change on storing in water. The reasons can be traced in the Table A.1. Some comments are collected in the table on next page.

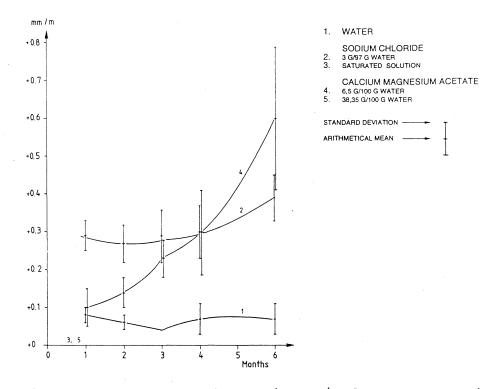


Figure 8.2: Length change in mm/m for mortar prisms; water cement ratio 0,45 stored at +20°C.

- 0,45 +5 The length change did not exceed the standard deviation, the 3 months value excepted. At this time, the change was +0,08 mm/m.
- 0,45 +20 The evolution of the change is well depicted in Figure 8.2. The change stabilized at +0,07 mm/m.
- 0,60 +5 The evolution, after 2 months of storing, is very well depicted in Figure 8.3. The change stabilized at -0,11 mm/m.
- 0,60 +20 During the first four months, the length change did not exceed the standard deviation. The last, 6 months, value was +0,04 mm/m.

For storing in water, the greatest expansion was +0.08 mm/m (wcr = 0.45, $+5^{\circ}$ C), and the greatest shrinkage -0.11 mm/m (wcr = 0.60, $+20^{\circ}$ C).

For computing the compressive load that follows from a restrained expansion or shrinkage, it is necessary to know, at least approximately, the static coefficient of elasticity.

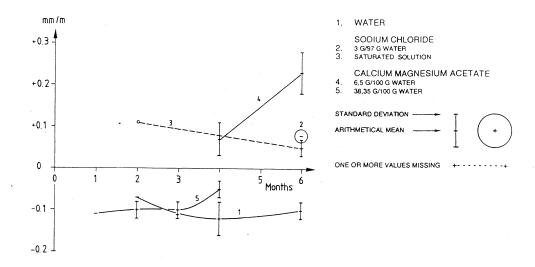


Figure 8.3: Length change in mm/m for mortar prisms; water cement ratio 0,60 stored at +5°C.

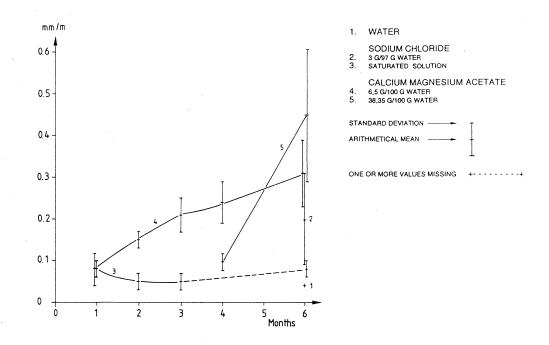


Figure 8.4: Length change in mm/m for mortar prisms; water cement ratio 0,60 stored at +20°C.

The following way has been used for calculating the static coefficient of elasticity. (Hillerborg, Göran Hedenblad)

1.
$$E^{n} = v_{1}E_{1}^{n} + v_{2}E^{n}$$

E Static coefficient of elasticity, mortar. E_1 Static coefficient, cement paste.

E₂ Static coefficient, quarz aggregates (50 GPa).

v₁ Volume of cement paste.

v₂ Volume of aggregates.

$$v_1 + v_2 = 1$$

n The exponent is equal to -0,15.

2. For the paste, the following is valid:

$$E_1 = E_0 * k^2, ^5 = 24$$
 GPa for wcr=0,35

 $k=0,71\alpha/(0,32\alpha+wcr)$

 α Degree of hydration (0,65 for wcr=0,35 and 0,8 for wcr=0,45 or 0,60).

wcr 0,35 0,45 0,60 k 0,8271 0,8045 0,6636 E₀ 38,58 38,58 38,58 GPa E₁ 24 22,39 13,84 GPa

3. For the mortar, the following is valid (compare Chapter 3.):

wcr 0.45 0,60

v₁ 0,417 0,373

v₂ 0,583 0,627

E 35,4 30,1 GPa

For the 0,45 mortar, stored in water, the static coefficient of elasticity is computed to 35,4 GPa. The expansion of 0,08 mm/m, would, if restrained, cause a compressive load of 2,8 MPa. This would cause no damage.

For the 0,60 mortar with the coefficient 30,1 GPa, a shrinkage of -0,11 mm/m would cause a tensile load of 3,3 MPa. Since this load is in "the wrong direction", there is some risk for a tensile fracture.

8.2.2 Sodium chloride, 3 % solution

Prisms of mortar with the water cement ratio 0,45 show an expansion which is well depicted in the Figures 8.1 (after four months) and 8.2 (all the time).

The behaviour of the 0,60 prisms is better shown in the table below than in the Figures 8.3 and 8.4.

- 0,45 +5 Before four months, the standard deviation was about as great as the expansion. After four months, the expansion was +0,1 mm/m, and it reached +0,2 mm/m after six months.
- 0,45 +20 The standard deviation was during all the time fairly low. The expansion reached +0,3 mm/m after one month and remained there until four months had passed. After six months, the expansion was +0,4 mm/m.
- 0,60 +5 During four months, the standard deviation was about as great as the expansion. After six months, the expansion had reached +0,1 mm/m.
- 0,60 +20 The prisms remained neutral during four months. The standard deviation was low, but still higher than the expansion. After six months, the expansion had reached +0,2 mm/m.

For the mortar with water cement ratio equal to 0,45, the dilute sodium chloride solution caused an essentially greater expansion than pure water did. With the static coefficient of elasticity of 35,4 GPa, the compressive load should reach 7 MPa at +5°C, and 14 MPa at +20°C, which should cause no damage.

With the coefficient value 30,1 GPA for 0,60 mortar, the compressive load should reach 3 MPa at +5°C, and 6 MPa for +20°C, which cannot cause any damage either.

8.2.3 Sodium chloride, saturated solution

As can be read in the table on the next page, the mortar with the water cement ratio 0,45 expanded 0,2 mm/m after 4 months at +5°C. At +20°C there was no significant change of length.

The 0,60 mortar prisms did show only some very small expansion, maximally 0,1 mm/m, either at $+5^{\circ}$ C or at $+20^{\circ}$ C.

- 0,45 +5 Until after three months, the standard deviation was as great as the expansion.

 After four and six months, the expansion was +0,2 mm/m and +0,15 mm/m.
- 0,45 +20 During the period 1-6 months, the standard deviation was about as great as the expansion, and this was not entered in Figure 8.2.
- 0,60 +5 During the test period, the expansion normally was very small, and the standard deviation, in most cases, was greater than the expansion figure. However, after two and six months of storing, the expansion of +0,11 and +0,05 mm/m was noted, with low deviation. Those two points were entered in Figure 8.3.
- 0,60 +20 During the period 1-6 months, the expansion was small, in the level +0,05-+0,08 mm/m.

 The standard deviation is well acceptable, the four months point exceptet for. The four accepted values are entered in Figure 8.4.

Normally, the expansion was less for the saturated sodium chloride solution than for the 3 % solution.

The maximal expansion load for 0,45 mortar, with a static coefficient of elasticity of 35 GPa, should be 7 mPa, and for the 0,60 mortar (30 GPa) 3,3 MPa. Those loads should not shape any risk for crack formation.

8.2.4 Calcium magnesium acetate, 6,5 g/100 g water

All mortar prisms became covered with a thin, whitish-grey layer when stored in the dilute CMA solution.

- 0,45 +5° Small standard deviation. After three months the mortar had expanded +0,13 mm/m, and after 6 months +0,42 mm/m.
- 0,45 +20° Greater standard deviation, but also greater expansion: +0,10 mm/m after one month up to +0,6 mm/m after six months.
- 0,60 +5° Up to four months, the standard deviation was almost as great as the expansion.

 After six months, the expansion was +0,23 mm/m.
- 0,60 +20° The mortar expanded gradually from one to six months to +0,31 mm/m.

The maximal expansion load for 0,45 mortar, with a static coefficient of elasticity of 35 GPa, should be 21 MPa, if the expansion would be fully restrained. For 0,60 mortar, with the coefficient 30 GPa, the maximal expansion load should be 9 MPa.

The consequences are to be discussed in the subchapter 9.3, after the account of the mass changes.

8.2.5 <u>Calcium magnesium acetate</u>, 38,35 g/100 g water

At this high concentration of CMA, no whitish-grey cover was formed on the mortar prisms, but the cement paste in the surface of the prisms dissolved, rapidly at +20°C, very slowly at +5°C.

- 0,45 +5° Already after one month, a shrinkage of -0,2 mm/m occured. After three months, the length of the prisms passad a minimum at a shrinkage of -0,25 mm/m. During the period four to six months the shrinkage gradually receded to -0,1 mm/m.
- 0,45 +20° No expansion occurred during the period 0-4 months. During the period 4-6 months, all prisms broke.
- 0,60 +5° After one month, a shrinkage of -0,11 mm/m was noted. After four months, the shrinkage had receded to -0,05 mm/m, and after six months, no shrinkage could be found any more.
- 0,60 +20° During the period one to three months, no length change could be found, greater than the standard deviation. After four months, an expansion of +0,1 mm/m was found, and after six months it had reached +0,45 mm/m.

The maximal shrinkage at +5°C for 0,45 mortar would result in a tensile load of 9 MPa, if the shrinkage is restrained.

At +20°C, no length change was registered, but the dissolution of the paste caused the prisms to break before six months of storing.

The 0,60 mortar prisms showed a very faint shrinkage at +5°C, about 0,1 mm/m. At +20°C, however, an expansion started after four months of storing and reached 0,45 mm/m after six months. At this temperature, the dissolution is the most serious problem.

It is an interesting observation that no other prism than these three did show any expansion. Also at the previous work the 0,60 mortar prisms, stored at +20°C, showed the greatest expansion. However, this time also the 0,45 mortar expanded, but less than the 0,60 mortar, and the +5°C storing developed expansion as well as the +20°C storing, but slower.

8.3 <u>Discussion of length change</u>

Generally, the precision of the measurements was low. The standard deviation was very often so great that points must be discarded. Still, some conclusions can be drawn:

1. Water

0,45 mortar: less than +0,1 mm/m expansion 0,60 mortar: +5°C: -0,1 mm/m shrinkage +20°C: Hardly any expansion

2. Sodium chloride, 3 g/97 g water

0,45 mortar: +5°C: +0,2 mm/m expansion +20°C: +0,4 mm/m - rather strong expansion * 0,60 mortar: +5°C: less than +0,1 mm/m expansion

+20°C: +0,2 mm/m expansion

3. Sodium chloride, saturated solution

0,45 mortar: +5°C: +0,2 mm/m, great standard deviation
+20°C: No expansion observed
0,60 mortar: +5°C: +0,1 mm/m expansion
+20°C: less than +0,1 mm/m expansion

4. Calcium magnesium acetate, 6,5 g/100 g water

0,45 mortar: +5°C: +0,4 mm/m expansion * +20°C: +0,6 mm/m expansion * 0,60 mortar: +5°C: +0,2 mm/m expansion +20°C: +0,3 mm/m expansion

5. Calcium magnesium acetate, 38,35g/100g water

0,45 mortar: +5°C: -0,2 mm/m shrinkage +20°C: No length change observed, but all prisms broken after 6 months 0,60 mortar: +5°C: -0,1 mm/m shrinkage

,60 mortar: +5°C: -0,1 mm/m shrinkage +20°C: expansion started after 4 months, reached +0,45 mm/m after 6 months * In the table on page 19, four sets of mortar prisms expanded 0,4 mm/m, or more:

- a. Prisms of mortar with water cement ratio 0,45 stored in sodium chloride solution, 3 g NaCl/97 g water, 20°C, expanded 0,4 mm/m.
- b. Prisms of the same type of mortar stored in calcium magnesium acetate solution, 6,5 g CMA/100 g water, expanded 0,4 mm/m at +5°C, and 0,6 mm/m at +20°C. This solution had the same freezing point depression as the sodium chloride solution.
- c. Prisms of mortar with water cement ratio 0,60 stored in calcium magnesium acetate, 38,35 g CMA/100 g water at +20°C expanded 0,45 mm/m.

In the case (a.), why did the dilute solution of sodium chloride cause an expansion, when the saturated solution did not?

When a sodium chloride solution penetrates the mortar, a chemical compound, *Friedel's salt*, will form:

(1)
$$2Na^{+} + 2Cl^{-} + 3CaO.Al_{2}O_{3}.Ca(OH)_{2}.12H_{2}O =$$

= $2Na^{+} + 2OH^{-} + 3CaO.Al_{2}O_{3}.CaCl_{2}.12H_{2}O$
Friedel's salt

The reaction implies that chloride ions from the solution replace the same number of hydroxide ions from one of the hydration products of tricalcium-aluminate. As result, the solution will be more alkaline.

If the sodium chloride solution is very strong, more hydroxide ions are supplied to the solution and to the pore liquid in the hardened cement paste. Provided that a solubility product

(2)
$$(Ca^{2+}) \times (OH^{-})^{2} = L$$

is valid for calcium hydroxide, an increase of the hydroxide ion concentration will cause a decrease of the calcium ion concentration. Thus, an increase of the hydroxide ion concentration will decrease the solubility of calcium hydroxide, eventually making the calcium hydroxide the stable phase instead of the Friedel's salt.

This would mean that a saturated sodium chloride solution has less capacity to shape Friedel's salt, and also less capacity to cause an expansion of the cement paste.

The length changes of mortar prisms stored in water and saturated sodium chloride solution were not much different from those reported 1991 (O Peterson).

In the case (b.), why did the *dilute* solution of calcium magnesium acetate cause a relatively great expansion, when the *concentrated* did not?

The most plausible explanation may be that the dilute solution lost its limited contents of magnesium ions very rapidly:

(3)
$$Mg^{+2} + Ca^{+2} + 2OH^{-} = Mg(OH)_{2} + Ca^{2+}$$

magnesium hydroxide
 brucite

Thus, the dilute solution of calcium magnesium acetate in contact with cement mortar rather rapidly changes to calcium acetate. The previous report (O Peterson 1991) revealed that a saturated solution of calcium acetate caused an expansion of 0,45 mortar of more than 15 mm/m within about one year. Still, no macro-cracks formed.

The present report indicates that also the dilute solution of calcium magnesium acetate, after conversion to calcium acetate, causes 0,45 mortar to expand, but naturally to a less degree.

The exact explanation is not clear. Probably, the calcium acetate forms a double salt with the solid calcium hydroxide of the hardened cement paste, and this double salt takes more space than the original calcium hydroxide phase. Its consistency seems to be pasty, which prevents the formation of cracks, at least before the carbonation of the expanded mortar.

The 0,60 mortar did not expand so much as the 0,45 mortar. The probable explanation may be that the pore volume in 0,60 mortar is great enough to allow formation of the calcium acetate hydroxide double salt without any need of more space.

The concentrated solution of the BP quality of CMA did not cause any expansion, except in 0.60 mortar at $+20\,^{\circ}\text{C}$, 0.45 mm/m. This should be compared with 1.2 mm/m in the previous work. In Chapter 11 is described some analyses, which aimed at elucidating the differences between the two types of calcium magnesium acetate.

In the case (c.) the expansion started after 4 months and reached 0,45 mm/m after 6 months. The 0,6 mortar has a greater pore volume than the 0,45 mortar. Thus, more of the CMA solution converts to calcium acetate in the pore liquid, causing the moderately greater expansion.

9. <u>Mass change</u>

9.1 Method

Each of the $4 \times 4 \times 16$ cm prisms was weighed at several occasions:

- 1. Just after stripping, at an age of two days.
- 2. At an age of 8 days, after completed wet curing.
- 3. At an age of 28 days, after completed curing in air with a relative humidity of 75 %, just before transferring to the test bath.
- 4. After a storing time in the test bath of one month the weighing was repeated, normally with an interval of one month, until the test was terminated after six months.

Of the weighings No 1-3, No 2 was most comparable with the weighing of the prisms in the test bath, and was chosen as reference for the later weighings.

The three prisms in each set have not necessarily the same mass. In order to get the mass change determined with the best precision possible, the mass of each prism always was compared with the mass of the same prism at weighing no 2.

The standard deviation was counted from the values of mass changes of the three prisms in a set.

9.2 Results and comments

All results are reproduced in four tables in the Appendix A.2, one table for each mortar and each storing temperature.

Each table contains five columns, one for each liquid:

- 1. Water
- 2. Sodium chloride solution, 3 g/97 g of water
- 3. "- , saturated
- 4. Calcium magnesium acetate (CMA) solution, 6,5 g/100 g of water
- 5. CMA solution, 38,35 g/100 g of water

In the four figures below (Figure 9.1-9.4), the mass changes in gram, (+) for increase, and (-) for decrease, are divided by the total surface of the prisms and set off versus the time of storing in the five liquids. Thus, the mass change is given in kilograms per square meter.

For the two sets which were stored in the liquid No 5 at +20°C, the losses of mass were so great that it should be considered more reasonable to divide the value with the total mass at weighing No 2. The results are presented as percentage of mass.

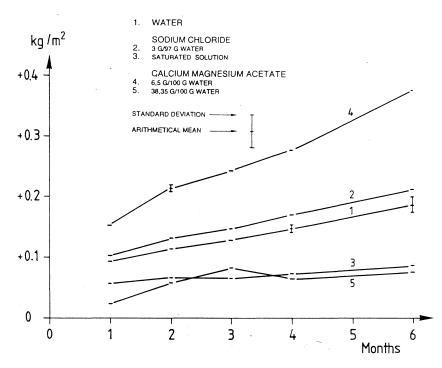


Figure 9.1: Mass change in kg/m² for mortar prisms water cement ratio 0,45 stored at +5°C.

9.2.1 <u>Water</u>

During six months of storing, the prisms gradually increased in mass. At +5°C the prisms increased in mass at a rate of 0,15 kilogram per square meter. At +20°C the rate was 0,2 kilogram per square meter. The rate was independent of the water cement ratio of the mortar.

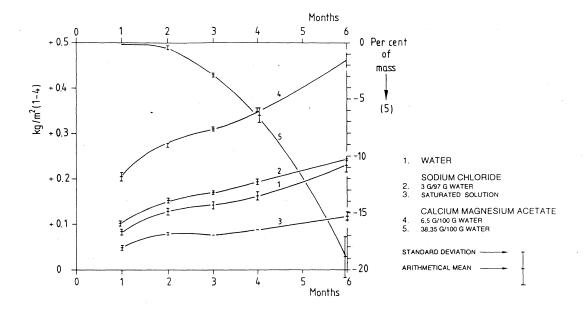


Figure 9.2: Mass change

in kg/m^2 for mortar prisms Nos 1-4 as percentage by mass for prism No 5

water cement ratio 0,45 stored at +20°C.

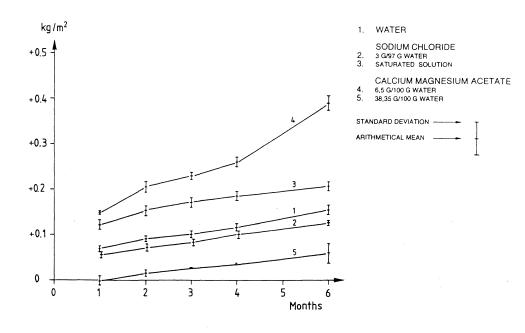


Figure 9.3: Mass change in kg/m² for mortar prisms water cement ratio 0,60 stored at +5°C.

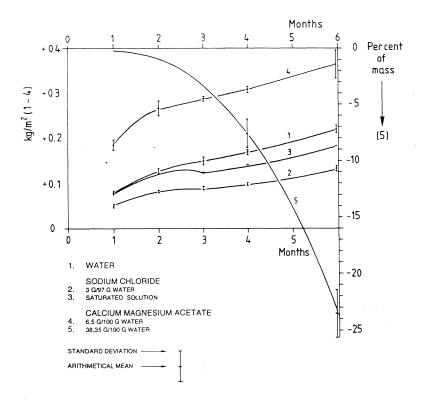


Figure 9.4: Mass change

in kg/m^2 for mortar prisms Nos 1-4 as percentage by mass for prism No 5

water cement ratio 0,60 stored at +20°C.

9.2.2 <u>Sodium chloride</u>, 3 % solution

During 6 months of storing, the 0,45 mortar prisms gained in mass 0,212 kilogram per square meter at +5°C and 0,243 kilogram per square meter at +20°C. The gain was somewhat greater than after storing in water.

The 0,60 mortar prisms gained in mass 0,125 kilogram per square meter at +5°C, which was somewhat less than the gain after storing in water. At +20°C, the gain was 0,132 kilogram per square meter, which was rather much less than the gain after storing in water.

9.2.3 Sodium chloride, saturated solution

During 6 months of storing, the 0,45 mortar prisms gained 0,087 kilogram per square meter at $+5^{\circ}$ C and 0,117 kilogram per square meter at $+20^{\circ}$ C. This was very much less than after storing in water.

The 0,60 mortar prisms gained 0,205 kilogram per square meter at $+5^{\circ}$ C, which was more than the gain after water storing. At $+20^{\circ}$ C the gain was 0,182 kilogram per square meter, which was insignificantly less than after storing in water.

9.2.4 <u>Calcium magnesium acetate</u>, 6,5 g/100 g water

After 6 months of storing, the 0,45 mortar prisms gained 0,378 kilogram per square meter at +5°C, and 0,462 kilogram per square meter at +20°C.

The 0,60 mortar prisms gained 0,389 kilogram per square meter at $+5^{\circ}\text{C}$, and and 0,365 kilogram per square meter at $+20^{\circ}\text{C}$.

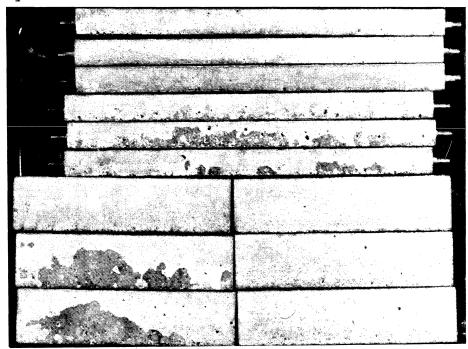


Figure 9.5: Prisms stored for five months at +20°C in CMA solution 6,5 g/100 g water.

The 0,45 prisms were totally covered with a white layer.

The 0,60 prisms had partially lost the white layer.

The white layer, which covered all prisms more or less completely, was examined as a powder with transmitted light under an optical microscope. The crystals were recognized as brucite, which is magnesium hydroxide.

The white layer partly fell away from the 0,60 mortar prisms, as shown in the Figures 9.5 and, as a detail, 9.6.

The formation of magnesium hydroxide as a consequence of chemical reactions between magnesium ions and calcium hydroxide solution can not explain why the prisms gain in mass, because the calcium hydroxide, which is dissolved, has a greater mass than the magnesium hydroxide, which is precipitaded.

A reasonable explanation is that the calcium acetate solution reacts with crystals of calcium hydroxide in the hardened cement paste, thus increasing the total mass of the mortar, and also the total length of the prisms (sub-chapter 8.3).

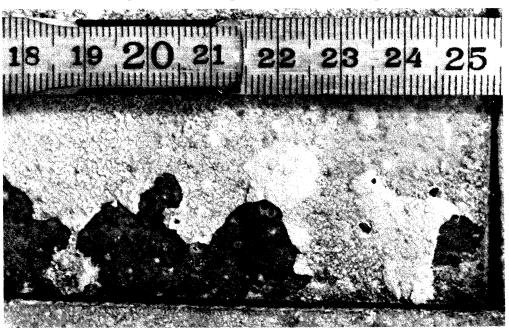


Figure 9.6: Detail of 0,60 mortar prism with partially lost layer of magnesium hydroxide.

9.2.5 <u>Calcium magnesium acetate</u>, 38,35 gram/100 gram water

After six months of storing, the 0,45 mortar prisms gained 0,076 kilogram per square meter at +5°C. At +20°C, the prisms suffered a loss in mass, which was so great that we chose to express the loss as 18,9 procent by mass.

The 0,60 mortar prisms gained 0,059 kilogram per square meter at +5°C. At +20°C, the loss was 23,5 procent by mass.

9.3 <u>Discussion of mass change</u>

As a concentrated solution, the BP quality of calcium magnesium acetate, used in this study, behaved in a much milder manner against both types of mortar at +5°C than the product from the Netherlands used in the previous work.

In the Paragraph 9.2.5 was shown that the prisms gained a little in mass during the storing at +5°C. Still it was found that small amounts of aggregate was lost from the surface, and this means that a corresponding small amount of hardened cement paste was dissolved from the same surface.

At +20°C, however, the BP product had a four times more rapid dissolving action on the 0,45 mortar, and six times on the 0,60 mortar during six months, all compared with the Netherlands product. Only in the 0,60 mortar, the loss of material was accompanied with an evident expansion at the same time, and this expansion started after four months.

The dilute CMA solution, with a freezing point depression comparable with a 3 % sodium chloride solution, caused a greater increase of mass than water or any of the two sodium chloride solution, especially in 0,60 mortar.

It is very reasonable that this increase was caused in the following four steps:

- 1. Magnesium hydroxide is precipitated on the surface of the prisms.
- 2. This changes the solution in the superficial pore system into calcium acetate.
- 3. The calcium acetate seems to form a double salt with the crystalline calcium hydroxide phase in the hardened cement paste. This will increase the mass of the mortar.
- 4. The increase in mass will increase the volume, principally in the 0,45 mortar. The 0,60 mortar seems to have a pore volume, which is sufficient for accepting the increase in mass.

10. pH values of the storing baths

After preparing the test solutions, the two solutions of calcium magnesium acetate were measured with respect to their pH value. The measurement was done only for solutions at +20°C.

The water bath and the two sodium chloride solutions were not measured.

10.1 Method

The calcium magnesium acetate solutions were filtered through a 00R filter before the pH measurement. The purpose was to eliminate slimy precipitations of magnesium hydroxide.

When the water and the sodium chloride solutions were measured, no filtering was necessary.

A Metrohm 691 pH meter was used, and the calibration was done with the two Metrohm buffer solutions for the pH values 7,00 and 9,00.

10.2 Results and comments

The results were collected in the following table, which is divided in five columns:

- 1. Immersion in water
- 2. Immersion a solution of 3 gram sodium chloride solution per 97 gram of water
- 3. Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Column of	immersion, No.	1	2	3	4	5
Date 1991	State of storing	1				
08 JAN 18 FEB 18 MAR 18 APR 15 MAY 26 JUL	Before immersion 1 month 2 months 3 months 4 months 6 months	12,09 12,13 11,89	12,23 12,31 12,09 12,37 12,30	12,07 11,83 12,08	8,89 9,27 9,19	9,23 9,07 9,21

10.2.1 <u>Water</u>

The water reached a pH value of maximally 12,14. The value kept rather constant during the storing time.

10.2.2 Sodium chloride, 3 % solution

The pH value in this dilute salt solution reached a maximum of 12,37 and, during the storing, remained rather well constant.

The increase, compared to water, may be caused by the chemical reaction in Subchapter 8.3 (page 20):

(1)
$$2Na^{+} + 2Cl^{-} + 3CaO.Al_{2}O_{3}.Ca(OH)_{2}.12H_{2}O =$$

= $2Na^{+} + 2OH^{-} + 3CaO.Al_{2}O_{3}.CaCl_{2}.12H_{2}O$
Friedel's salt

When chloride ions react with the tetra calcium aluminate hydrate of the hardened cement paste, hydroxide ions will be emitted, and the pH value of the sodium chloride solution will increase.

10.2.3 Sodium chloride, saturated solution

It would be reasonable to expect that the saturated sodium chloride solution would cause the pH value to increase still more than the 3 % solution does.

Instead, the maximal reading was 12,08, which is slightly lower than the maximal reading for water, which was 12,14.

Probably, saturated sodium chloride solution really gives pH values, which are higher than that of the 3 % solution, and the reading would probably have been higher, if a hydrogen gas electrode system had been used instead of the glass electrode system.

However, the glass electrode is not a quite ideal membrane electrode, which can be penetrated only by hydrogen ions. Sodium ions are essential constituents in the membrane, and thus the high concentration of sodium ions in the saturated sodium chloride solution is "mistaken" for an increase in the hydrogen ion concentration. The error is called the "sodium ion error". Glass electrodes with reduced sodium ion error are manufactured.

10.2.4 <u>Calcium magnesium acetate</u>, 6,5 gram/100 gram water

The pH value of the solution was 9,43 before the prisms of cement mortar had been immersed.

Interesting enough, the pH value during the six months storing of mortar prisms varied irregularily within the range 8,84-9,27, i. e. lower than the value in the solution without mortar prisms!

A possible explanation may be the contents in the last part of the Subchapter 8.3 (page 28). The calcium hydroxide in the hardened cement paste is partly consumed, and so is part of the acetate ions in the solution. Both changes will act in the direction towards lower pH values.

10.2.5 Calcium magnesium acetate, 38,35 gram/100 gram water

The pH value of the solution was 9,16 before the prisms of cement mortar had been immersed. The value is in complicated ways influenced by the equilibrium

- * of acetate and acetic acid in water, and
- * of magnesium ions and magnesium hydroxide in water.

The pH value during the storing of mortar prisms varied irregularily within the range 9,04 - 9,23. This is neither significantly higher, nor lower than the pH value of the original solution.

While the dilute solution precipitated magnesium hydroxide on the surfaces of the prisms, and probably forms a double salt with the calcium hydroxide of the hardened cement paste, the more concentrated solution at + 20°C mainly acts by dissolving the hardened cement paste at the surfaces of the prisms.

Probably, this is a consequence of the higher viscosity of the more concentrated solution, which makes it more difficult to penetrate the interior of the mortar prisms.

11. Chemical analysis of calcium and magnesium in calcium magnesium acetate solutions

The purposes of the analyses were to find out

- * whether the BP quality of calcium magnesium acetate used in this study has a composition that is different to the Dutch product, used in the previous investigation.
- * whether the most concentrated solution (38,35 g/100 g water) had lost some of the magnesium during the preparation because of precipitation of magnesium hydroxide.
- * whether the concentrated or the dilute solution had lost magnesium (and gained an equivalent amount of calcium) during prolonged contact with mortar prisms at 20°C.

11.1 Method

Five samples were taken:

- Concentrated solution (38,35 g/100 g water), filtered free from precipitates.
- 2. Concentrated solution after 9 months storing of cement mortar prisms at +20°C, and then filtered.
- 3. Solution with the same concentration and the same immersion time as 2., but after storing at +5°C instead of +20°C.
- 4. Dilute solution (6,5 g/100 g water), filtered free from precipitates.
- 5. Dilute solution after 9 months storing of cement mortar prisms at +20°C, and then filtered.

The analyses were performed at the Department of Environmental Engineering at our Institute, and atomic absorption was used. All samples were diluted for a calculated calcium concentration of 5 mg per liter. We chose to do this in two steps:

5 ml of the strong solution was diluted to 500 ml. 3 ml of this intermediate solution was diluted to 500 ml, and this solution has a concentration 0,000.06 of the strong solution.

5 ml of the dilute solution was diluted to 500 ml. 10 ml of this intermediate solution was diluted to 250 ml, and this solution has a concentration 0,000.4 of the dilute solution.

For analysis of magnesium it was necessary to dilute the solutions still more, to 0,5 mg per liter. This was done at the laboratory mentioned above.

The samples 4. and 5. could be analyzed with reasonable results whithout difficulties.

The samples 1., 2., and 3., however, gave irregular results. Another set of samples was prepared according to the following principles:

- 1. The dilution was done with destilled and deionized water instead of water which was only deionized, in order to avoid possible contamination with calcium ions.
- 2. The magnesium was analyzed without adjustment of the pH value, and with adjustment to 2,0.
- 3. The calcium was analyzed without adjustment of the pH value, and with a combination of pH adjustment to 2,0 and addition of lanthanum chloride.

11.2 Results

The results of the analyses are collected in the following table:

Sample of CMA solution	Concentration,	mol/liter
	Calcium	Magnesium
1. 38,35 g/100 g water. No- Adj. 1. only Adj. 1., 2., and 3.	1,12	1,106 2 1,20 2 1,23
2. The same concentration, immersion of cement more specimens for 9 months Adj. 1. only Adj. 1., 2., and 3.	tar	1,06
3. The same concentration, immersion of cement more specimens for 9 months Adj. 1. only Adj. 1., 2., and 3.	tar	5 1,17
4. 6,5 g/100 g water. Not	adj. 0,223	0,245
5. The same concentration, immersion of cement more specimens for 9 months	tar	0,177

11.3 Comments

For the samples 1., 2., and 3., the third line in the table was used for comparison. This means that the distilled and deionized water was used for diluting the samples, and that the pH value was adjusted to 2,0 before atomic absorption analysis, and finally that lanthanum chloride was added to the calcium solutions.

The magnesium content did not change very much on immersion of cement mortar specimens for 9 months. Still, the order of the changes seems reasonable: Storing at $+20\,^{\circ}\text{C}$ causes a greater decrease than storing for the same time at $+5\,^{\circ}\text{C}$.

In the sample 1., in which no specimens of cement mortar had been immersed, the molar content of magnesium was 1,23 mol/liter, which is slightly more than the calcium content, 1,12 mol/liter. The molar ratio Mg/Ca is equal to 1,10.

The corresponding values for the previously used solution, from the Netherlands, was 0,69 mol/liter magnesium and 0,87 mol/liter calcium, molar ratio 0,79.

In the sample 2., where specimens of cement mortar had been stored for nine months at +20°C, the analyzed magnesium concentration was 1,10 mol/liter, to be compared with 1,23 mol/liter before the immersion. The calcium concentration was 3,44 mole per liter, to be compared with 1,12 mol/liter before the immersion. This very great increase is difficult to explain.

As a check on the plausibility of the decrease of the magnesium concentration found in sample 2., a calculation was made from the loss of mass of the three prisms of 0,45 mortar and the three prisms of 0,60 mortar. The losses were 302 gram and 400 gram, in order. Those masses corresponded with 70,83 and 71,07 gram of cement, totally 141,9 gram, which in turn corresponds to 90,67 gram of calcium oxide in clinker, equal to 1,62 mol.

For dissolving this cement, 1,62 mol of magnesium is needed. Assumed that the 20 liters of water, used to prepare the stronger calcium magnesium acetate solution, would give 20 liters of solution, the solution's total content of magnesium was 24,6 mol. After the loss of 1,62 mol, 23,0 mol of magnesium should remain, which makes 93,5 per cent of the fresh solution. After six months of storing, 1,15 mol of magnesium per liter should remain. Thus, 1,10 mol magnesium per liter after nine months of storing seems to be of the proper order of magnitude.

In the sample 3., which is similar to sample 2. whith exception for the storing temperature +5°C, the magnesium concentration was 1,20 mol/liter, to be compared with 1,23 mol/liter before the immersion. The calcium concentration was 2,79 mol/liter and this value is still very great, compared with 1,12 mol/liter before the immersion.

In the sample 4. the magnesium concentration was analyzed to 0,245 mol/liter and the calcium concentration to 0,223 mol/liter, which is very near to the expected concentration of 0,22 mol/liter from the weighing. The molar ratio Mg/Ca was 1,10, which was the same ratio as in the concentrated solution in sample 1.

In the sample 5. the magnesium concentration was 0,177 mol/liter, which is to be compared with 0,245 before the 6 months of immersion of mortar prisms at +20°C. The loss was 0,068 mol/liter. Some of this, or possibly all, may be the magnesium hydroxide, recovered as a white cover on the mortar prisms.

The calcium concentration was 0,274 mol/liter, which is to be compared with 0,223 before the storing period. The gain was 0,051 mol/liter. The difference allows the explanation that some calcium acetate may have been absorbed in the mortar prisms, maybe a reasonable explanation to the expansion and to the gain in mass by storing for six months in this dilute CMA solution.

After the storing, the molar ratio Mg/Ca was decreased from 1,10 to 0,65, which is very near to the ratio of the CMA from the Netherlands, used in the previous work. Probably, this explains the relatively remarkable expansion of 0,6 mm/m in the cement mortar prisms with water cement ratio 0,45, which were stored in this solution. This expansion is just above the limit 0,5 mm/m for accepted expansion of mortar bars in tests for alkali silica reactions. Still, it is very likely that an expansion of 0,6 millimeter per meter, caused by calcium acetate, does not cause cracking of the mortar, as e. g. freezing would do.

The analyses of the samples 2. and 3. also showed a very great decrease in molar ratio Mg/Ca. The change is mainly caused by a strong increase of the concentration of calcium ions. Just now it seems difficult to explain how the calcium concentration of the solution could increase so much.

12. <u>Conclusions</u>

An increase of the molar rate Mg/Ca in calcium magnesium acetate to 1,10 (compared with 0,79 in the previous work, Peterson (1991)) decreases the expansion of mortar in concentrated solution and also the dissolution of the cement paste at +5°C.

The expansion, when it occurred (0,60 mortar, +20°C), did not start until four months had passed. A condition is that some magnesium is replaced by calcium in the pore liquid.

The dissolution rate at $+20\,^{\circ}\text{C}$ did increase, probably because of the increased molar ratio Mg/Ca.

A calcium magnesium acetate solution with the same freezing point depression as 3 % sodium chloride solution caused a moderate expansion, especially in mortar with water cement ratio 0,45. In such a dilute solution, the mortar prisms became covered with a white layer of magnesium hydroxide, and the prisms increased in mass. The solution lost magnesium and toke up calcium, and probably the expansion and the gain in mass are caused by chemical reactions between penetrating calcium acetate and calcium hydroxide crystals in the pore system of the mortar.

With this dilute solution of calcium magnesium acetate, the expansion started as soon as the mortar prisms were immersed in the solution.

Mortar prisms with water cement ratio 0,60 lost their white layer gradually on parts of their surfaces.

Also the solution av 3 gram of sodium chloride and 97 gram of water caused an expansion of 0,45 mortar prisms of 0,4 millimeter per meter after six months at +20°C. The expansion was less at +5°C, in 0,60 mortar prisms, and in saturated solution.

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Table A.1 Length change, mm/m

The results of the length readings are entered in the table below, which is divided in four parts:

- 1. w/c-ratio 0,45 storing temperature +5°C 2. w/c-ratio 0,45 storing temperature +20°C
- 3. w/c-ratio 0,60 storing temperature +5°C
- 4. w/c-ratio 0,60 storing temperature +20°C

Part 1

w/c-ratio 0,45 Storing temperature +5°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution 3 per cent by weight
- 3. Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A.1 Length change, mm/m (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,45 +5°C	Part 1. Reading No. 1. Millimeter	7,18 7,08 6,67	6,58 6,52 6,15	6,92 6,35 6,83	5,20 5,65 4,73	7,10 6,52 6,49
	Reading No. 2. Millimeter	7,24 7,13 6,68	6,62 6,54 6,17	6,95 6,39 6,84	5,22 5,65 4,73	7,13 6,56 6,51
	Reading No. 3. Millimeter	7,22 7,11 6,66	6,62 6,53 6,16	6,91 6,35 6,83	5,20 5,62 4,69	7,10 6,52 6,47
FEB 91	Reading No. 4. Difference to reading No. 2.	+0,02 0 0	0 +0,02 +0,02	-0,01 0 +0,04	+0,01 -0,01 -0,01	-0,08 -0,06 -0,04
	A. M., mm/m Stand. deviation	+0,02 0,04	+0,05 0,04	+0,04 0,09	-0,01 0,04	-0,21 0,07
MAR 91	Reading No. 5. Difference to reading No. 2.	0 -0,05 +0,01	0 +0,03 +0,03	+0,07 +0,01 +0,03	+0,02 0 0	-0,07 -0,06 -0,05
	A. M., mm/m Stand. deviation	-0,05 0,11	+0,07 0,06	+0,1 0,11	+0,02 0,04	-0,21 0,04
APR 91	Reading No. 6. Difference to reading No. 2.	+0,02 +0,03 +0,02	0 +0,03 +0,05	+0,07 +0,01 +0,05	+0,04 +0,04 +0,03	-0,08 -0,07 -0,06
	A. M., mm/m Stand. deviation	+0,08 0,02	+0,1 0,09	+0,15 0,11	+0,13 0,02	-0,25 0,04
MAJ 91	Reading No. 7. Difference to reading No. 2.	-0,01 -0,05 0	+0,01 +0,03 +0,05	+0,07 +0,01 +0,06	+0,05 +0,05 +0,04	-0,06 -0,05 -0,04
	A. M., mm/m Stand. deviation	-0,07 0,09	+0,11 0,07	+0,2 0,11	+0,17 0,02	-0,18 0,04
JUL 91	Reading No. 8. Difference to reading No. 2.	0 -0,04 +0,01	+0,05 +0,05 +0,07	+0,08 +0,02 +0,03	+0,12 +0,11 +0,12	-0,02 -0,05 -0,04
	A. M., mm/m Stand. deviation	-0,04 0,09	+0,20 0,04		+0,42	-0,13 0,05

Table A.1 Length change, mm/m (continued)

Part 2

w/c-ratio 0,45
Storing temperature 20°C

- 1. Immersion in water
- Immersion in sodium chloride solution
 per cent by weight
- Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A.1 Length change, mm/m (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,45 +20°C	Part 2. Reading No. 1. Millimeter	7,71 7,27 6,33	6,37 6,71 6,32	7,39 6,92 7,03	6,56 6,83 6,76	6,40 6,93 6,49
	Reading No. 2. Millimeter	7,73 7,30 6,37	6,40 6,74 6,37	7,42 6,95 7,05	6,59 6,85 6,80	6,41 6,96 6,51
	Reading No. 3. Millimeter	7,71 7,29 6,36	6,38 6,72 6,36	7,40 6,93 7,03	6,57 6,82 6,78	6,37 6,94 6,48
FEB 91	Reading No. 4. Difference to reading No. 2.	+0,02 +0,02 +0,03	+0,07 +0,08 +0,09	+0,01 +0,10 +0,03	+0,01 +0,04 +0,03	-0,02 +0,01 -0,02
	A. M., mm/m Stand. deviation	+0,08	+0,29 0,04	+0,2 0,2	+0,10 0,05	-0,04 0,06
MAR 91	Reading No. 5. Difference to reading No. 2.	+0,01 +0,02 +0,02	+0,06 +0,08 +0,09	+0,01 +0,08 +0,02	+0,03 +0,05 +0,04	-0,02 +0,02 -0,02
	A. M., mm/m Stand. deviation	+0,06	+0,27 0,05	+0,1 0,14	+0,14 0,04	-0,04 0,1
APR 91	Reading No. 6. Difference to reading No. 2.	+0,01 +0,01 +0,01	+0,06 +0,08 +0,10	+0,01 +0,08 +0,02	+0,05 +0,08 +0,06	-0,02 +0,02 -0,03
	A. M., mm/m Stand. deviation	+0,04 0,00	+0,29 0,07	+0,1 0,14	+0,23 0,05	-0,04 0,09
MAJ 91	Reading No. 7. Difference to reading No. 2.	+0,01 +0,02 +0,03	+0,06 +0,09 +0,10	+0,02 +0,10 +0,02	+0,06 +0,12 +0,09	-0,01 +0,02 -0,03
•	A. M., mm/m Stand. deviation	+0,07 0,04	+0,30 0,07	+0,2	+0,3 0,11	-0,02 0,09
JUL 91	Reading No. 8. Difference to reading No. 2.	+0,01 +0,02 +0,03	+0,09 +0,12 +0,12	+0,03 +0,11 +0,03	+0,14 +0,16 +0,24	B r o k
	A. M., mm/m Stand. deviation	+0,07 0,04	+0,39 0,06	+0,2 0,16	+0,6 0,19	е

Table A.1 Length change, mm/m (continued)

Part 3

w/c-ratio 0,60
Storing temperature 5°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution
 3 per cent by weight
- Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A.1 Length change, mm/m (continued)

Colu	ımn	of immersion, No.	1 1	2	3	4	5
w/c-ratio 0, +5°		Part 3. Reading No. 1. Millimeter	6,74 6,74 6,35	6,60 6,62 6,64	6,39 6,71 6,46	5,22 6,63 6,68	6,24 6,53 6,35
		Reading No. 2. Millimeter	6,74 6,73 6,34	6,60 6,63 6,64	6,38 6,72 6,45	5,22 6,62 6,69	6,23 6,50 6,36
		Reading No. 3. Millimeter	6,73 6,72 6,33	6,60 6,63 6,63	6,36 6,69 6,43	5,20 6,61 6,66	6,20 6,47 6,32
FEB	91	Reading No. 4. Difference to reading No. 2.	+0,01 +0,03 -0,02	0 +0,01 0	0 -0,01 0	0 0 -0,02	-0,03 -0,03 -0,03
		A. M., mm/m Stand. deviation	+0,02	+0,01 0,02	-0,01 0,02	-0,02 0,04	-0,11 0,00
MAR	91	Reading No. 5. Difference to reading No. 2.	-0,02 -0,02 -0,02	+0,02 +0,02 0	+0,03 +0,03 +0,03	0 +0,01 0	-0,03 -0,02 -0,03
		A. M., mm/m Stand. deviation	-0,07 0,00	+0,05 0,04	+0,11 0,00	+0,01 0,02	-0,10 0,02
APR	91	Reading No. 6. Difference to reading No. 2.	-0,03 -0,03 -0,03	0 +0,02 +0,02	+0,02 0 +0,03	+0,02 +0,02 0	-0,02 -0,03 -0,03
		A. M., mm/m Stand. deviation	-0,11 0,00	+0,05 0,04	+0,06 0,05	+0,05 0,04	-0,10 0,02
MAY	91	Reading No. 7. Difference to reading No. 2.	-0,02 -0,04 -0,04	+0,01 +0,03 0	0 0 +0,02	+0,02 +0,03 +0,01	-0,02 -0,01 -0,01
		A. M., mm/m Stand. deviation	-0,12 0,04	+0,05 0,05	+0,02 0,04	+0,07 0,04	-0,05 0,02
JUL	91	Reading No. 8. Difference to reading No. 2.	-0,03 -0,03 -0,02	+0,02 +0,03 +0,02	+0,01 +0,01 +0,02	+0,06 +0,08 +0,05	-0,02 +0,01 -0,02
		A. M., mm/m Stand. deviation	-0,10 0,02	+0,08 0,02	+0,05 0,02	+0,23 0,05	-0,04 0,06

Table A.1 Length change, mm/m (continued)

Part 4

w/c-ratio 0,60
Storing temperature 20°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution
 3 per cent by weight
- Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A.1 Length change, mm/m (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,60 +20°C	Part 4. Reading No. 1. Millimeter	6,47 5,78 6,58	6,42 6,91 6,89	6,29 6,24 5,97	7,17 6,69 6,61	6,41 6,84 7,29
	Reading No. 2. Millimeter	6,46 5,77 6,60	6,41 6,91 6,89	6,30 6,24 5,97	7,13 6,68 6,61	6,41 6,84 7,29
	Reading No. 3. Millimeter	6,46 5,77 6,59	6,40 6,90 6,89	6,27 6,22 5,95	7,11 6,65 6,60	6,39 6,82 7,28
FEB 91	Reading No. 4. Difference to reading No. 2.	+0,01 +0,01 0	+0,01 -0,02 +0,01	+0,02 +0,03 +0,02	+0,03 +0,03 +0,01	+0,02 +0,02 0
	A. M., mm/m Stand. deviation	+0,02 0,02	0,00 0,06	+0,08 0,02	+0,08	+0,05 0,04
MAR 91	Reading No. 5. Difference to reading No. 2.	+0,01 +0,01 0	+0,01 -0,02 +0,01	+0,01 +0,02 +0,01	+0,04 +0,05 +0,04	+0,01 +0,02 0
	A. M., mm/m Stand. deviation	+0,02 0,02	0,00 0,06	+0,05 0,02	+0,15 0,02	+0,04 0,04
APR 91	Reading No. 6. Difference to reading No. 2.	0 0 -0,01	+0,01 -0,03 +0,01	+0,01 +0,02 +0,01	+0,07 +0,06 +0,05	0 +0,03 +0,01
	A. M., mm/m Stand. deviation	-0,01 0,02	-0,01 0,08	+0,05 0,02	+0,21 0,04	+0,05 0,05
, MAY 91	Reading No. 7. Difference to reading No. 2.	+0,01 +0,01 0	+0,01 -0,02 +0,02	+0,01 +0,01 0	+0,08 +0,07 +0,05	+0,03 +0,03 +0,02
	A. M., mm/m Stand. deviation	+0,02 0,02	+0,01 0,07	+0,02 0,02	+0,24 0,05	+0,10 0,02
JUL 91	Reading No. 8. Difference to reading No. 2.	+0,01 +0,01 +0,01	+0,04 +0,06 +0,10	+0,02 +0,03 +0,02	+0,10 +0,10 +0,06	+0,08 +0,17 +0,13
	A. M., mm/m Stand. deviation	+0,04	+0,2 0,11	+0,08 0,02	+0,31 0,08	+0,45 0,16

Table A. 2 Mass changes, gram

The results of the weighings are entered in the table below, which is divided in four parts:

- w/c-ratio 0,45 storing temperature +5°C
 w/c-ratio 0,45 storing temperature +20°C
 w/c-ratio 0,60 storing temperature +5°C
- 4. w/c-ratio 0,60 storing temperature +20°C

Part 1

w/c-ratio 0,45 Storing temperature +5°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution 3 per cent by weight
- 3. Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A. 2 <u>Mass changes</u>, gram (continued)

Column	of immersion, No.	1	2.	3	4	5
w/c-ratio 0,45 +5°C	Part 1. Weighing No. 1.	564,8 560,4 559,7	563,4 559,5 562,9	558,6 556,4 567,9	559,2 561,2 567,6	563,9 563,4 565,5
	Weighing No. 2.	576,2 570,9 568,7	571,2 569,5 574,6	568,0 566,6 577,9	570,6 571,1 578,0	572,9 572,6 575,7
	Weighing No. 3.	575,60 570,02 567,60	571,16 569,54 574,34		567,90 568,33 575,18	569,44
FEB 91	Weighing No. 4. Difference to weighing No. 2.	+2,8 +2,6 +2,7	+3,0 +3,1 +2,9	+1,5 +1,6 +1,8	+4,4 +4,3 +4,5	+0,6 +0,8 +0,7
	Arithm. mean Stand. deviation	+2,7 0,1	+3,0 0,1	+1,6 0,15	+4,4 0,1	+0,7 0,1
MAR 91	Weighing No. 5. Difference to weighing No. 2.	+3,25 +3,15 +3,4	+3,7 +3,9 +3,7	+1,8 +1,8 +2,0	+6,3 +6,4 +5,9	+1,6 +1,8 +1,7
	Arithm. mean Stand. deviation	+3,3 0,1	+3,8 0,1	+1,9 0,1	+6,2 0,3	+1,7 0,1
APR 91	Weighing No. 6. Difference to weighing No. 2.	+3,7 +3,5 +3,8	+4,3 +4,4 +4,2	+1,85 +1,8 +1,95	+7,3 +7,1 +6,6	+3,05 +2,2 +1,9
	Arithm. mean Stand. deviation	+3,7 0,15	+4,3 0,1	+1,9 0,1	+7,0 0,4	+2,4 0,6
MAY 91	Weighing No. 7. Difference to weighing No. 2.	+4,2 +4,0 +4,3	+4,85 +5,0 +4,8	+2,1 +2,1 +2,2	+8,2 +8,05 +7,7	+1,85 +1,9 +1,95
	Arithm. mean Stand. deviation	+4,2 0,15	+4,9 0,1	+2,1 0,06	+8,0 0,3	+1,9 0,05
JUL 91	Weighing No. 8. Difference to weighing No. 2.	+5,8 +5,0 +5,35	+6,0 +6,2 +6,0	+2,5 +2,4 +2,5	+10,9 +11,2 +10,5	+2,9 +1,9 +2,2
	Arithm. mean Stand. deviation	+5,4 0,4	+6,1 0,1	+2,5 0,07	+10,9 0,35	+2,2 0,5

Table A. 2 Mass changes, gram (continued)

Part 2

w/c-ratio 0,45
Storing temperature 20°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution 3 per cent by weight
- Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A. 2 Mass changes, gram (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,45 +20°C	<u>Del 2</u> . Weighing No. 1.	565,3 564,8 575,3	579,2 569,7 576,1	554,8 565,5 569,0	572,6 568,6 566,6	554,8 566,2 568,2
	Weighing No. 2.	571,3 570,1 580,3	587,8 578,1 585,6	565,7 575,3 579,0	581,8 578,0 576,5	562,1 573,9 577,3
	Weighing No. 3.	568,86 568,07 578,56	586,39 577,10 584,61	562,07 572,19 576,20	578,47 574,96 573,53	558,77 570,65 573,92
FEB 91	Weighing No.4. Difference to weighing No. 2.	+2,1 +2,6 +2,4	+2,7 +3,1 +3,0	+1,4 +1,2 +1,5	+5,9 +6,1 +5,6	+0,4 +0,8 +0,4
	Arithm. mean Stand. deviation	+2,4 0,25	+2,9 0,2	+1,4 0,15	+5,9 0,25	+0,5 0,2
MAR 91	Weighing No. 5. Difference to weighing No. 2.	+3,8 +3,7 +3,5	+4,1 +4,55 +4,3	+2,3 +2,4 +2,1	+8,0 +8,2 +7,9	-1,7 -3,5 -1,7
	Arithm. mean Stand. deviation	+3,7 0,15	+4,3 0,2	+2,3 0,15	+8,0 0,15	-2,3 1,0
APR 91	Weighing No. 6. Difference to weighing No. 2.	+4,0 +4,4 +4,0	+4,75 +5,0 +4,9	+2,25 +2,2 +2,2	+9,1 +8,9 +8,65	-15,8 -16,9 -17,5
	Arithm. mean Stand. deviation	+4,1 0,2	+4,9 0,1	+2,22	+8,9 0,2	-16,7 0,9
MAY 91	Weighing No. 7. Difference to weighing No. 2.	+4,6 +5,0 +4,55	+5,4 +5,7 +5,55	+2,5 +2,5 +2,5	+10,2 +10,0 +10,0	-34,1 -34,7 -40,7
·	Arithm. mean Stand. deviation	+4,7 0,25	+5,55 0,15	+2,5 0,0	+10,1 0,1	-36,5 4
JUL 91	Weighing No. 8. Difference to weighing No. 2.	+6,5 +7,1 +6,3	+6,9 +7,2 +7,0	+3,4 +3,4 +3,35		-93,8 -96,4 -111,85
	Arithm. mean Stand. deviation	+6,6 0,4	+7,0 0,15		+13,3 0,5	-108 10

Table A. 2 Mass changes, gram (continued)

Part 3

w/c-ratio 0,60
Storing temperature 5°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution 3 per cent by weight
- 3. Immersion in saturated solution of sodium chloride
- 4. Immersion in calcium magnesium acetate solution, 6,5 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A. 2 <u>Mass changes</u>, gram (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,60 +5°C	Part 3. Weighing No. 1.	560,7 557,4 559,3	550,9 549,7 543,8	554,6 550,5 549,6	555,0 544,2 554,3	556,5 555,0 557,2
	Weighing No. 2.	566,5 562,5 569,0	559,0 558,5 555,5	562,5 557,4 557,9	559,0 548,4 559,7	560,1 558,1 561,3
	Weighing No. 3.	563,18 560,28 566,74		559,87 554,07 554,56	556,67 546,71 557,64	556,38
FEB 91	Weighing No. 4. Difference to weighing No. 2.	+1,8 +2,1 +2,0	+1,4 +1,7 +1,8	+3,3 +3,4 +3,8	+4,2 +4,4 +4,3	-0,4 +0,0 +0,1
	Arithm. mean Stand. deviation	+2,0 0,15	+1,6 0,2	+3,5 0,3	+4,3	-0,1 0,3
MAR 91	Weighing No. 5. Difference to weighing No. 2.	+2,4 +2,7 +2,6	+1,9 +2,2 +2,2	+4,1 +4,4 +4,6	+5,9 +6,3 +5,6	+0,3 +0,35 +0,6
	Arithm. mean Stand. deviation	+2,6 0,15	+2,1 0,2	+4,4 0,25	+5,9 0,35	+0,4 0,2
APR 91	Weighing No. 6. Difference to weighing No. 2.	+2,75 +3,1 +2,85	+2,2 +2,5 +2,6	+4,6 +4,8 +5,2	+6,5 +6,9 +6,5	+0,7 +0,8 +0,8
	Arithm. mean Stand. deviation	+2,9	+2,4	+4,9 0,3	+6,6 0,2	+0,8 0,06
MAY 91	Weighing No. 7. Difference to weighing No. 2.	+3,15 +3,5 +3,2		+5,0 +5,3 +5,6	+7,45 +7,8 +7,3	
	Arithm. mean Stand. deviation	+3,3 0,2	+2,9 0,2	+5,3 0,3	+7,5 0,3	+1,1 0,06
JUL 91	Weighing No. 8. Difference to weighing No. 2.	+4,2 +4,7 +4,2	+3,5 +3,6 +3,8	+5,55 +5,9 +6,2	+11,2 +11,6 +10,7	+2,2 +1,8 +1,1
	Arithm. mean Stand. deviation	+4,4	+3,6 0,15		+11,2 0,45	+1,7 0,6

Table A. 2 <u>Mass changes</u>, gram (continued)

Part 4

w/c-ratio 0,60 Storing temperature 20°C

- 1. Immersion in water
- 2. Immersion in sodium chloride solution
 3 per cent by weight
- Immersion in saturated solution of sodium chloride
- Immersion in calcium magnesium acetate solution,
 gram per 100 gram of water
- 5. Immersion in calcium magnesium acetate solution, 38,35 g per 100 gram of water

Table A. 2 <u>Mass changes</u>, gram (continued)

Column	of immersion, No.	1	2	3	4	5
w/c-ratio 0,60 +20°C	Part 4. Weighing No. 1.	550,0 551,0 558,8	540,8 548,3 550,7	545,8 550,6 561,3	549,1 556,6 557,8	560,9 558,8 566,6
	Weighing No. 2.	559,8 556,9 565,7	550,1 555,4 558,9	549,7 554,2 565,0	554,4 560,4 562,5	564,4 562,4 570,7
	Weighing No. 3.	557,59 554,89 563,39	552,34	547,50 551,97 563,20		562,66 560,64 568,40
FEB 91	Weighing No. 4. Difference to weighing No. 2.	+2,3 +2,4 +2,2	+1,6 +1,4 +1,4	+2,1 +2,1 +2,3	+5,2 +5,8 +5,2	-0,7 -0,8 -1,4
	Arithm. mean Stand. deviation	+2,3	+1,5 0,1	+2,2	+5,4	-1,0 0,4
MAR 91	Weighing No. 5. Difference to weighing No. 2.	+3,7 +3,8 +3,5	+2,4 +2,4 +2,3	+3,4 +3,5 +3,5	+7,2 +8,2 +7,8	-4,4 -6,55 -4,0
	Arithm. mean Stand. deviation	+3,7 0,15	+2,4 0,06	+3,5 0,06	+7,7 0,5	-5,0 1,4
APR 91	Weighing No. 6. Difference to weighing No. 2.	+4,3 +4,4 +4,05	+2,65 +2,6 +2,5	+3,7 +3,6 +3,8	+8,3 +8,5 +8,2	-16,9 -24,4 -16,45
	Arithm. mean Stand. deviation	+4,25 0,2	+2,6 0,1	+3,7 0,1	+8,3 0,15	-19,25 4,5
MAY 91	Weighing No. 7. Difference to weighing No. 2.	+4,9 +5,0 +4,7	+2,9 +3,0 +2,9	+4,2 +3,9 +4,2	+8,8 +8,8 +9,2	-38,0 -50,5 -39,4
•	Arithm. mean Stand. deviation	+4,9 0,15	+2,9 0,06	+4,1 0,2	+8,9 0,2	-43 7
JUL 91	Weighing No. 8. Difference to weighing No. 2.	+6,4 +6,45 +6,0	+3,6 +3,9 +3,8	+5,35 +5,0 +5,4	+9,5	-124,9 -146,55 -128,7
	Arithm. mean Stand. deviation	+6,3 0,2	+3,8 0,15	+5,25 0,2	+10,5 0,9	-133 12