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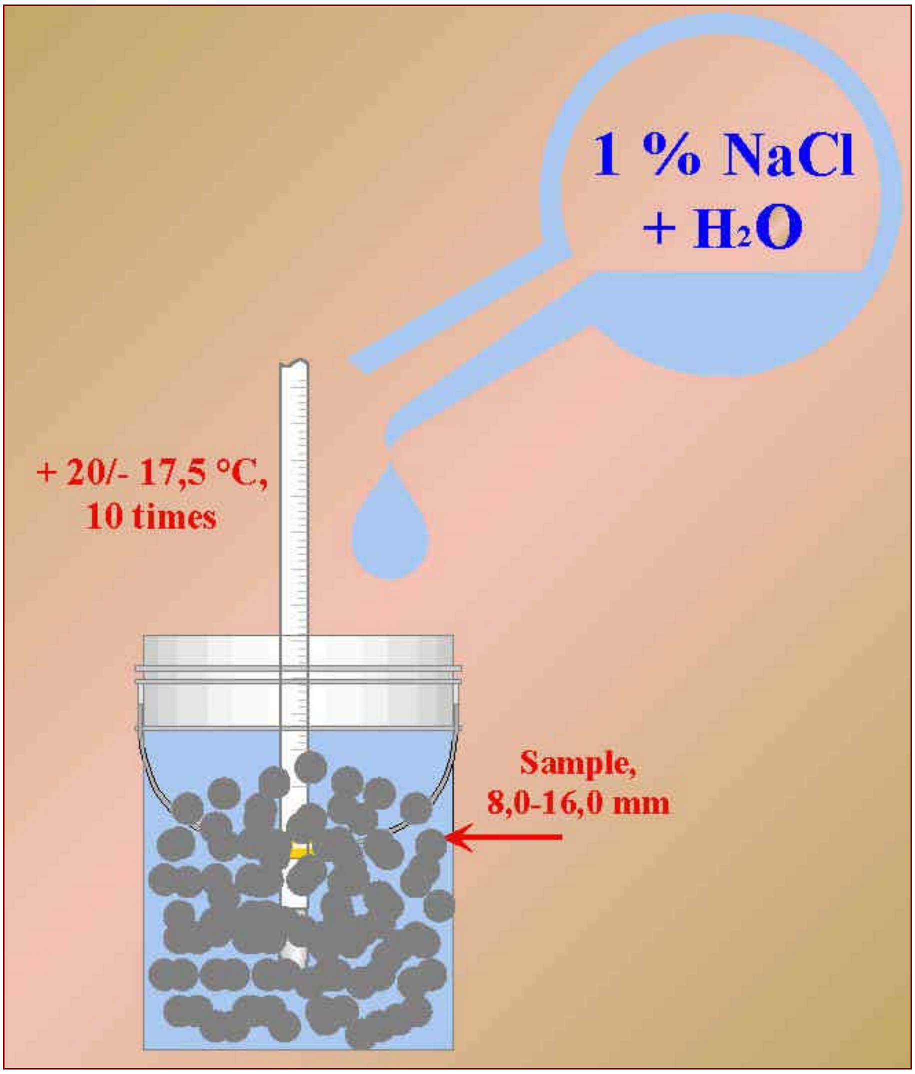
Project no.
NT 1624-03

FINAL REPORT, SEPTEMBER 2004:

NORDTEST PROJECT No. 1624-03

Frost Resistance Test on Aggregates With and Without Salt (FRAS)



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ABSTRACT

The two-year Nordtest project no. 1624-03 has now been completed. It was divided into two main steps, the first one being completed at the end of 2003 with an interim report. Fifteen laboratories in ten European countries participated in this project, including laboratories in all the Nordic countries. The Nordtest proposal and project description are enclosed in Appendix I.

The main objective of the project was to test aggregates from different sources throughout Europe at different laboratories in Europe, with both pure water and salt water freeze/thaw test methods, to widen the applicability of the freeze/thaw test method in 1 % NaCl. A part of this objective was to demonstrate that different freeze/thaw cabinets can be used to obtain comparable results, as long as the desired sample temperature is adhered to. Another objective was to gain reliable repeatability and reproducibility values for frost resistance test methods with and without salt (NT BUILD 485 and EN 1367-1). Thirdly, to produce and suggest to the relevant task group of CEN/TC 154, a revised method for testing aggregate freeze/thaw resistance with 1 % NaCl to be included in the European Standards.

The first step involved inter-comparative, frost resistance testing of eighteen aggregate samples both using fresh water and a 1 % NaCl solution. The second step involved statistical evaluation of the test results for both methods used. Additionally, water absorption and particle density of the aggregates tested was measured to compare with freeze/thaw test results.

The frost resistance of aggregates was tested in pure water and in a 1 % NaCl solution. To secure consistency between laboratories as much as possible, a combined testing procedure was written to include both test methods, EN 1367-1 (pure water) and NT BUILD 485 (salt solution), see Appendix II. The main modifications in the combined test method to the existing test methods (besides giving the option to use a salt solution) are:

1. The option to use air temperature in the cabinet to control the thawing phase of the test specimens as well as thawing, using water circulation in the cabinet.
2. Minor, but necessary, modification to the temperature curve to include the freezing point of the salt solution.

Correlation was established between laboratories in frost resistance test results for both test methods, i.e. with and without salt. Correlation between test results when testing in fresh water and a salt solution was also established, although the numerical values are not comparable between the two test methods. Still, there were considerable differences in test results between laboratories and between individual test specimens in some cases. It is concluded that technically, it is difficult to accurately reproduce the test methods (with and without salt) for several reasons. One reason may be different temperature amplitudes at different locations in the freeze/thaw cabinets. Another cause for inconsistency may be related to the fact that some of the laboratories participating in the project did not have any former experience of freeze/thaw testing according to the prescribed method(s). Yet another reason for inconsistency could be related to undefined factors concerning the temperature curve, sample preparation, equipment used, testing procedure and/or measurements.

The fresh water method (based on EN 1367-1) produced frost resistance values less than 2 % for all the aggregates tested (mean value of all laboratories) and thereof values less than 1 % for 15 out of 18 aggregate samples, including aggregates of known poor quality. In fact, two of the samples having mean values over 1 % were close to 1,2 % and only one had a value close to 2 % (1,9 %). Bearing in mind requirement categories in the product standards of CEN/TC 154, it was concluded that the fresh water method does not distinguish adequately between frost resistant and frost susceptible aggregates.

The salt water method (based on NT BUILD 485) gives a wide spread in values between aggregates, leaving the frost resistant aggregates generally intact but causing degradation of the poorer ones, up to 33 % for the poorest aggregate sample tested (mean value of all laboratories). The ranking of aggregates according to frost resistance values obtained is generally according to predicted quality. Therefore it is possible, by using the salt solution, to introduce new requirement categories in the product standards of CEN/TC 154, based on the connection between test results and known quality of the aggregates tested in this project as well as previous, practical experience.

The precision values are in general acceptable for both test methods. However, some parts of the testing procedure need to be better specified and the laboratories need to gain more experience with the test. This will significantly reduce both repeatability and the reproducibility values.

The evaluation of all test results shows that testing with a 1 % NaCl-solution gives equal or better precision data than when testing in pure water. Introducing salt into the test results also gives a better spread in test results and, for most aggregates, a better correlation with known performance in real constructions.

The evaluation also shows that there is no significant difference when controlling the thawing phase in water or air. Both procedures can therefore be allowed for in the method.

The combined test method that has been drafted is based on EN 1367-1 and NT BUILD 485. No obvious problems occurred using the draft test method, besides problems already existing in the two methods, which the draft is based on. The project group decided to forward the draft test method to CEN/TC 154/SC 6 “Aggregates-Test methods” with the request that the proposed combined method replaces the now existing EN 1367-1 at the 5-year review that is now underway, see Appendix III.

CONTENTS **Page**

PREFACE

This report presents the results of the Nordtest project no. 1624-03, Frost resistance test on aggregates with and without salt – FRAS, see project proposal to Nordtest in Appendix I. This is the second and final report on the two-year project, but an interim report on the results was published in September 2003. The Nordic participants in the FRAS-project are:

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Ten other partners from five European countries participated in the project as well. They are:

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The Nordtest project NT 1624-03 **F**rost **R**esistance test on **A**ggregates with/without **S**alt (**FRAS**) has now been completed. Two project meetings have been held, the first one at RWTH in Aachen on 31 January 2003 and the second one at FEhS in Duisburg on 1 March 2004. The minutes of the meetings are presented in Annex III.

This work was requested by Task Group 12 of CEN/TC 154 and forms an important part of the effort to include a frost resistance test using a salt solution for harsh climatic conditions and in saline environment or where de-icing salts are used. A combined draft frost resistance test method is appended to this report (Appendix II). The draft is being considered by the relevant Sub Committees of CEN/TC 154 to replace the now existing test method, which is due for a 5-year revision (late 2004).

The views and conclusions of this report are the responsibility of the authors. The project group has read the report and their received comments have been taken into account.

Pétur Pétursson

Reykjavík, September 2004

1. INTRODUCTION

It is essential for producers and users of aggregates to have a common test standard for determination of the frost resistance of aggregates for use in various end products. Until such a standard exists, the objective of a free trade of aggregates across all borders of European countries can't be realized.

The existing European standard, EN 1367-1, has not proven to give relevant test result compared to the performance of many aggregates used in the European countries. Harsh climatic conditions, where frequent freeze/thaw cycling occurs and in saline environment, do exist in many places in the Nordic countries, as well as elsewhere in Europe. In such areas it is indeed important to have knowledge of the expected durability of aggregates used for outdoor constructions, whether they would be road surfaces or concrete structures, such as bridges.

A salt-water test method is considered to give such information about aggregates while fresh water methods fail to do so, at least in some cases.

The present European standard, EN 1367-1, has an informative annex which states that *"Until experience with a definitive test method,*....(using a de-icer)...., *giving good precision on a wide range of European aggregates is available, the reference freeze-thaw test method, is as specified in this standard, using distilled water*". The FRAS-project has fulfilled these requirements of the Annex to EN 1367-1 and it is therefore hoped that the results of this research project will be acknowledged to support the implementation of a European frost resistance test using a salt solution.

1.1 Background

One of the most commonly used freeze/thaw tests on loose aggregates has been the German standard DIN 4226. That test method has now been replaced by the European standard EN 1367-1, which is based on the German standard in all major aspects /1/. Based on freezing and thawing in fresh water, Swedish and Icelandic research have demonstrated that the method does not distinguish adequately between frost resistant and frost susceptible aggregates /2 and 3/. It was concluded that the pure water method is therefore of little value in areas, where repeated and severe freeze/thaw cyclic action occurs. A freeze/thaw test method was developed in Iceland in the years 1986 to 1989, using a 1 % NaCl solution during the frost action /3/. The method involved applying 70 freeze/thaw cycles, 10 per 24 hours, with a temperature range of +4 to -4°C. This test method proved to give considerable breakdown of aggregates with a poor service record and showed a strong correlation with actual performance of experimental surface dressing road stretches.

In 1993 the method was introduced to CEN/TC 154/TG 9 (Aggregates-thermal and weathering properties) with a request for a frost resistance test in salt water as an option to the pure water method (EN 1367-1), which was then under consideration by TC 154/SC 6 (Aggregates-Test methods). TG9 wrote a working draft of a freeze/thaw test method, using 1 % NaCl, which is based on the Icelandic method in all major aspects. However, CEN/TC154/SC6 decided not to forward the Icelandic based draft standard, as comprehensive research results were not available and it was stated that not enough data had been collected using cross-European aggregates. Instead, an informative annex was added to EN 1367-1, stating that an undefined procedure with

1 % NaCl could be used in exceptional cases. Iceland has always objected to this conclusion with the support of the other Scandinavian delegates in TC 154/SC 6 ("Test methods").

In 1995-6 a Nordtest project resulted in another method with a better accuracy, but based on the experience obtained earlier and with reference to EN 1367-1 /4 and 5/. Nordic aggregates were tested with the pure water method on the one hand and the salt-water method on the other hand (Nordtest project 1214-95). It was obvious that the difference is very significant. It was demonstrated that the pure water method failed to distinguish between frost resistant and frost susceptible aggregates while the salt water method succeeded to do so. This method has been in use in Iceland since 1996 and became a formal Nordtest standard in 1998 (NT BUILD 485). Again, CEN/TC154/TG9 wrote a new working draft of a freeze/thaw test method using 1 % NaCl, which was based on the Nordtest method in all major aspects this time. However, CEN/TC154/SC6 decided not to forward the Nordtest-based draft standard on the same grounds as before.

In short: Earlier research has demonstrated that frost resistance testing in fresh water does not distinguish between frost resistant and frost susceptible aggregates. Fresh water testing is therefore of little value in areas where frost action occurs and de-icing salts are used or sea spray is abundant. Testing frost resistance in salt water on the other hand distinguishes between frost resistant and frost susceptible aggregates and is therefore of great value for such areas.

It is also important for test laboratories to be able to use the same equipment when testing frost resistance of aggregates, either in pure or salt water. The main difference between the two methods, which were combined into one draft standard and used in the FRAS-project, is the use of a salt solution in the Nordtest method, but pure water in the EN method. Another important difference is that the thawing phase is controlled by air circulation in the Nordtest method but with water circulation in the EN method. These two methods of thawing were also included in the combined draft standard and made optional to use either method. The two methods serve the same purpose, which is to control the thawing phase of the samples. Therefore, it was considered irrelevant which one of the two methods is used to control the sample temperature. By focusing on the sample temperature instead of how it is achieved, unnecessary restrictions on very expensive equipment could be avoided. That would enable laboratories in the Nordic countries as well as elsewhere, to easily adopt the method and run the combined test method. The third item worth mentioning, which was harmonized between the two standards in the combined version is the freezing point, which was given the range between 0 and –1°C instead of a fixed 0°C, to include the freezing point of the salt solution.

Task Group 12 of CEN/TC 154 (TG 12 "Chemical, thermal and weathering properties of aggregates") requested that the Icelandic delegate would draft a new proposal of a test method using a salt solution and based on the Nordtest method NT BUILD 485. This has already been done by combining the existing European and Nordtest methods into a single draft test method, see Appendix II. TG 12 also resolved that *"the organisation of an interlaboratory trial has to be forseen as soon as possible"*. It can therefore be stated that the FRAS-project group was established with the support of TG 12 of CEN/TC 154.

The present Nordtest project accordingly was defined to give test results for a representative selection of European aggregates, using a combined draft test description, using both the salt water and pure water testing procedure, see Appendix II. In that way it is made possible for CEN/TC 154/SC 6 to assess and compare both test methods.

1.2 Objectives and deliverables

Three main objectives are tabulated in the project description:

a) To test aggregates from different sources throughout Europe at different laboratories in Europe, with both pure water and salt water freeze/thaw test methods, to widen the applicability of the Nordtest freeze/thaw test method in 1 % NaCl. A part of this objective is to demonstrate that different freeze/thaw cabinets can be used to obtain comparable results, as long as the desired sample temperature is adhered to.
b) To gain reliable repeatability and reproducibility values for both test methods (NT BUILD 485 and EN 1367-1).
c) To produce and suggest to the relevant task group of CEN/TC 154, a revised method for testing aggregate freeze/thaw resistance with 1 % NaCl to be included in the European Standards. The specified equipment used shall be possible to use in both the salt and the pure water methods.

All three objectives have now been fulfilled. The main activities and deliverables are tabled below.

Year 2003:
January 2003: The project started in January 2003. A meeting was held at the end of January, where a final decision about aggregate samples was taken. The procedure of the whole project was discussed in detail.
February to April 2003: Collection of samples, preparation of samples and shipping of samples to participating laboratories.
May to August 2003: Testing of samples at the national laboratories. This part of the project was completed in early October.
September 2003: Status report to Nordtest.

Year 2004:
January to March 2004: Processing of data and preliminary statistical evaluation. A meeting was held on 1 March in Duisburg to discuss test results. Decisions were made on final conclusions and presentations.
April to September 2004: Statistical evaluation continued. Revision of the standard NT BUILD 485. A final report issued to Nordtest. Proposal concerning inclusion of a CEN/TC 154 standard or a normative annex to an existing standard, which will be based on the Nordtest standard for testing aggregates frost resistance in salt water in 2004.

2. TEST METHODS AND PROCEDURE

One of the main aims of the FRAS project was to compare freeze/thaw test results of aggregates when tested in fresh water according to EN 1367-1 on the one hand and in a 1 % salt solution according to NT BUILD 845 on the other hand. In the effort to ensure consistency, it was decided to combine the two testing procedures into one draft standard, which all the participants would use for their testing procedure, see Appendix II.

As NT BUILD 485 was initially written to harmonize with EN 1367-1 as much as possible, the two standards are identical in many ways. The combined test description is therefore not very different from either of the standards, but the main differences are listed here below:

- In *Foreword*, the two fundamental alterations to EN 1367-1 are described in a short text, i.e. that the samples are tested in either de-ionised water or 1 % solution of NaCl in de-ionised water and that the thawing out sequence is controlled either be air circulation or water circulation in the cabinet to obtain the correct temperature of the reference sample.
- In *1 Scope* there is a statement that in areas where frequent freeze-thaw cycling occurs and seawater sprays or de-icers are abundant it is more appropriate to use a 1 % solution of NaCl in de-ionised water instead of pure de-ionised water.
- In 4 Principle the option to soak test portions in pure water or 1 % NaCl solution is for 24 hours prior to testing given.
- In *5 Apparatus under 5.7* a guidance is given on how to prepare the 1 % NaCl solution.
- In *8 Procedure under 8.1 Soaking* it is stated that soaking should be carried out either in water or 1 5 NaCl.
- In *8.2 Exposure to freezing under water or NaCl-solution* the temperature curve is adjusted to include the freezing temperature of a 1 % NaCl-solution. The freezing temperature of the test liquid is therefore given as 0 to –1 °C instead of 0 °C. *Figure 1* includes the same alteration of the temperature curve.

Three key laboratories were chosen to test all aggregates, both with and without salt to obtain statistically reliable data. The key laboratories are IBRI, SP and RWTH (see preface). Additionally, all the other participating laboratories tested two domestic aggregates, one of assumed poor quality and the other of good quality, as well as one to three reference samples.

The reference samples chosen were expected to have a wide range in quality; one basalt of poor to average quality (Ref. 1), one limestone of very poor quality (Ref. 2) and one granite of very good quality (Ref. 3).

The three key laboratories tested all aggregates with both test methods. This means that 18 samples were tested with two test methods, testing 3 test specimens of each (when possible) = 108 measurements. The other laboratories tested 3 to 5 samples with two test methods, testing 3 test specimens (when possible) = 18 to 30 measurements (depending on the number of reference samples tested). Two of those test samples are therefore generally the ones chosen domestically and the third to fifth sample are one to three reference samples.

3. AGGREGATE SAMPLES

The FRAS-project involved testing 18 different aggregate samples, two from each country, except from UK and Sweden where only one sample was provided. It was decided that one of the materials from each country should have good quality and the other one rather poor quality with respect to frost resistance or evaluated by other means by the participating partner of each country. The UK sample provided was considered of rather poor quality and the Swedish sample provided was considered of good quality.

A careful choice of aggregate samples to be tested in a round-robin project like the FRAS-project, is probably the most important factor for the project to be successful and to give reliable and relevant results. Special emphasis was made regarding the choice of aggregates for the project. The aggregate selection was intended to have a wide spread in geological formations as well as having records of quality or performance. In some cases, the quality recognition of aggregates chosen was based on previous frost resistance testing and/or frost resistance in structures. In other cases the frost resistance of selected aggregates was not known prior to the project and the selection was based on other quality estimations than frost resistance, such as physical strength (for example LA-value) or simply petrographic description. This is especially the case for the countries, which were unfamiliar with frost resistance testing. A summary of compiled information, concerning the chosen aggregate samples is presented in Appendix V. Denomination of the aggregates as being of good or poor quality is therefore a subjective evaluation of each participant to some extent and not directly comparative on a certain scale. Each aggregate sample was given a code to ensure that individual samples would not be traced to a certain producer. The chosen aggregates are as follows in Table 1:

TABLE 1 Aggregate samples used in the FRAS project

No.	Country	Predicted quality	Aggregate sample	Code
1	BE	Poor	Meta-sandstone	BAX[2)]
2	FI	Poor	Granite loose structure	COF[8)]
3	BE	Good	Micro-diorite	DIK[1)]
4	NO	Poor	Mica rich gneiss	FEP[4)]
5	DE	Good	Granite	GAL
6	IS	Good	Glassy basalt	GEB[6)]
7	FR	Good	Granite	HYD
8	NL	Good	Greywacke	MIN
9	FR	Poor	Oolitic limestone	NUC
10	UK	Poor	Greywacke	PEX[9)]
11	DK	Good	Marine low flint gravel	QAB
12	IS	Poor	Altered basalt	Ref 1[5)]
13	DE	Poor	Limestone	Ref 2
14	FI	Good	Granite	Ref 3[7)]
15	DK	Poor	Land-based porous flint	RUN
16	NO	Good	Natural gravel	VUX[3)]
17	SE	Good	Granite	XYD
18	NL	Poor	Gravel	ZIP

Key to the superscript for codes in table 1 above:

1) *Micro-diorit (quartz meta dacit) Belgian porphyry*
2) *Meta sandstone (devonian, famennian arkose)*
3) *Granitic glacio-fluvial aggregate*
4) *Crushed mica gneiss*
5) *50 % fresh, dense, 40 % altered, dense and 10 % very altered basalt.*
6) *60 % fresh, dense, 15 % fresh, porous and 25 % glassy basalt.*
7) *Kinzigite (garnet korderiete gneiss)*
8) *Garnet mica gneiss*
9) *Mixed river gravel*

The name and petrographic description of the aggregates (below table) are those given by the relevant contact person in the FRAS-project, representing the country of origin. Further information about the aggregates as given by contact persons is summarized in Appendix V.

It should be emphasized here that in some cases the quality estimation of the aggregate chosen is not based on any information about the aggregate's potential frost resistance nor actual performance records related to weathering, but other physical features or simply petrographic analysis. This is understandable, when bearing in mind that frost resistance testing is not a common procedure in many of the participating countries. This fact leads to the conclusion that in some cases the labeling, as "good or poor" aggregate does not reflect the frost resistance of the aggregate in question. This is the case for the Finnish granite with loose structure, which is labeled as "poor" on the grounds of an LA-value of 38 %, but proves to have good frost resistance, both with salt (0,17 %) and without salt (0,13 %). The same applies to the Norwegian mica-rich gneiss, which quality was based on petrographic description only, but also proves to have good frost resistance, both with salt (0,36 %) and without salt (0,22 %). On the other hand, the greywacke aggregate from UK is labeled as being of good quality, but the frost resistance is not good according to the salt water method (9,59 %), although it is quite good without salt (0,26 %). It is common practice in the UK to evaluate aggregate quality on the grounds of PSV and AIV, but such tests do not necessarily reflect the frost resistance of aggregates. Previous Icelandic research points in the same direction, i.e. that physical strength and frost resistance are in fact unrelated properties and do not reflect the results obtained from each other /7/.

4. FROST RESISTANCE TEST RESULTS

As has been stated before, two different freeze/thaw testing procedures, combined in one draft testing standard (see Appendix II) were performed on all aggregate samples, one with salt and the other one without salt. Three individual samples (test specimens) were tested in all cases when possible, but in some cases there was not enough material shipped to prepare three test specimens. In those cases, the mean value represents the results of two specimens only. Some laboratories tested four test specimens in the case there was enough material shipped to make up the fourth specimen. The UK partner (University of Ulster) tested their sample and Reference sample 1 by using salt water only (six specimens of each), instead of testing one part (three specimens) with salt and the other part without salt. Apparently, MPA Bau received enough material to be able to run two test runs on each of the tested samples. The test results have been treated as two separate test runs as if performed by two different laboratories in the data matrix.

The freeze/thaw values (individual test values and mean value) for the whole project are presented in tables 2 and 3 a) to c). Table 2 shows the results obtained at the key laboratories for all 18 aggregate samples.

TABLE 2 Individual and mean test results, key laboratories

		RB				RWTH				SP			
Type (g=good/p=poor)	**Code**	**spec.**	**No salt**	**spec.**	**Salt**	**spec.**	**No salt**	**spec.**	**Salt**	**spec.**	**No salt**	**spec.**	**Salt**
Meta-sandstone (p)	**BAX**	0,66	**0,91**	10,99	**10,10**	0,38	**0,44**	6,06	**5,55**	0,65	**0,79**	14,24	**13,93**
		0,70		8,95		0,30		6,31		0,89		14,26	
		1,37		10,34		0,63		4,29		0,85		13,31	
Granite loose str.(p)	**COF**	0,04	**0,07**	0,06	**0,10**	0,04	**0,05**	0,10	**0,10**	0,23	**0,22**	0,30	**0,29**
		0,04		0,12		0,06		0,10		0,21		0,29	
		0,12		0,13		0,06		0,09		0,22		0,27	
Micro-diorite (g)	**DIK**	0,11	**0,11**	0,24	**0,18**	0,06	**0,07**	0,06	**0,09**	0,18	**0,11**	0,25	**0,31**
		0,14		0,14		0,08		0,05		0,06		0,34	
		0,08		0,15		0,07		0,14		0,11		0,35	
Mica rich gneiss (p)	**FEP**	0,13	**0,15**	0,22	**0,19**	0,05	**0,07**	0,14	**0,13**	0,39	**0,42**	0,54	**0,53**
		0,16		0,17		0,06		0,11		0,48		0,54	
		0,16		0,18		0,08		0,15		0,40		0,51	
Granite (g)	**GAL**	0,04	**0,05**	0,06	**0,09**	0,03	**0,02**	0,04	**0,03**	0,11	**0,11**	0,16	**0,16**
		0,04		0,10		0,01		0,03		0,13		0,15	
		0,08		0,12		0,02		0,02		0,08		0,17	
Basalt-glassy (g)	**GEB**	0,47	**0,54**	3,73	**4,27**	0,61	**0,72**	3,57	**2,92**	0,80	**0,73**	7,41	**7,59**
		0,63		4,65		0,89		2,60		0,58		8,27	
		0,54		4,44		0,66		2,60		0,82		7,09	
Granite (g)	**HYD**	0,05	**0,04**	0,37	**0,38**	0,09	**0,10**	0,64	**0,85**	0,29	**0,21**	0,65	**0,80**
		0,05		0,23		0,09		1,04		0,18		0,97	
		0,03		0,53		0,12		0,86		0,16		0,77	
Greywacke (g)	**MIN**	0,13	**0,09**	1,04	**0,85**	0,03	**0,03**	0,23	**0,23**	0,13	**0,16**	1,90	**1,61**
		0,08		0,59		0,04		0,21		0,14		1,51	
		0,07		0,92		0,01		0,23		0,20		1,41	
Oolitic limest. (p)	**NUC**	0,43	**0,40**	18,89	**18,77**	0,17	**0,17**	3,24	**3,23**	0,56	**0,55**	25,41	**24,56**
		0,43		17,99		0,17		3,23		0,52		23,31	
		0,33		19,43		X		X		0,58		24,95	
Greywacke (g)	**PEX**	0,27	**0,31**	6,28	**6,31**	0,05	**0,04**	5,24	**3,38**	0,39	**0,43**	15,26	**16,57**
		0,34		6,08		0,02		1,51		0,47		16,29	
		X		6,57		X		X		0,44		18,15	
Marine, low flint (g)	**QAB**	0,06	**0,12**	0,48	**0,52**	0,02	**0,02**	0,39	**0,48**	0,08	**0,08**	1,04	**1,00**
		0,08		0,44		0,01		0,80		0,08		0,99	
		0,22		0,63		0,01		0,26		0,08		0,98	
Basalt-altered (p)	**REF 1**	1,05	**1,06**	9,60	**9,80**	0,87	**1,05**	16,42	**16,00**	1,91	**1,60**	14,28	**13,02**
		1,07		10,00		1,24		15,57		1,28		12,83	
		X		X		X		X		X		11,93	
Limestone (p)	**REF 2**	1,81	**1,72**	28,99	**29,17**	0,87	**1,24**	11,21	**11,27**	2,23	**1,80**	37,16	**39,04**
		1,59		29,68		0,89		11,36		1,69		39,44	
		1,76		28,85		1,95		11,24		1,49		40,52	
Granite (g)	**REF 3**	0,07	**0,08**	0,17	**0,19**	0,06	**0,06**	0,02	**0,03**	0,27	**0,28**	0,34	**0,38**
		0,09		0,20		0,05		0,04		0,29		0,33	
		X		X		X		X		X		0,47	
Landb. por. Flint (p)	**RUN**	1,44	**1,25**	5,04	**5,01**	0,10	**0,41**	1,31	**1,34**	1,48	**1,35**	3,70	**3,56**
		1,10		4,98		0,51		1,43		1,56		3,87	
		1,21		1,98	(not incl.)	0,61		1,26		1,02		3,11	
Natural gravel (g)	**VUX**	0,14	**0,12**	0,16	**0,12**	0,10	**0,09**	0,01	**0,03**	0,21	**0,20**	0,27	**0,29**
		0,12		0,09		0,06		0,02		0,19		0,34	
		0,08		0,13		0,11		0,05		0,19		0,26	
Granite (g)	**XYD**	0,11	**0,13**	2,56	**2,63**	0,10	**0,08**	8,02	**6,86**	0,33	**0,34**	5,10	**5,07**
		0,13		2,99		0,06		6,49		0,31		5,11	
		0,15		2,33		0,09		6,08		0,37		5,01	
Gravel (p)	**ZIP**	0,11	**0,14**	10,20	**8,72**	0,26	**0,21**	10,72	**11,74**	0,19	**0,21**	13,85	**13,37**
		0,17		8,22		0,20		12,86		0,22		11,73	
		0,13		7,72		0,16		11,66		0,21		14,53	

Table 3 (a, b and c) shows the individual and mean values from the remaining participating laboratories.

TABLE 3 a) Individual and mean test results for TUT, MPA Bau and MPA NRW

		TUT				MPA Bau				MPA Bau				MPA NRW			
Type (g=good/p=poor)	Code	spec.	No salt	spec.	Salt	spec.	No salt	spec.	Salt	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt
Granite loose str.(p)	COF	0,17	**0,18**	0,18	**0,19**												
		0,16		0,21													
		0,21		0,21													
		0,17		0,15													
Granite (g)	GAL					0,06	**0,07**	0,18	**0,16**					0,04	**0,04**	0,02	**0,03**
						0,07		0,08						0,04		0,04	
						0,07		0,22						X		0,04	
Basalt-altered (p)	REF 1	1,66	**1,53**	12,37	**13,46**	1,16	**1,17**	12,15	**12,32**	0,25	**0,33**	11,84	**11,76**	0,73	**0,99**	14,78	**15,43**
		1,41		14,55		1,24		12,43		0,39		11,67		1,32		16,48	
		X		X		1,12		12,38		0,34		11,76		0,92		15,04	
Limestone (p)	REF 2	2,65	**2,19**	22,24	**33,92**	2,38	**2,13**	39,22	**38,96**	2,10	**1,99**	34,23	**35,82**	2,79	**2,72**	39,14	**38,99**
		1,77		30,37		1,88		38,88		1,90		37,04		2,81		39,94	
		2,26		41,75		2,13		38,79		1,96		36,20		2,56		37,89	
		2,07		41,32													
Granite (g)	REF 3	0,15	**0,26**	0,18	**0,16**	0,02	**0,01**	0,06	**0,05**	0,02	**0,02**	0,13	**0,08**	0,04	**0,06**	0,09	**0,15**
		0,36		0,14		0,01		0,04		0,02		0,03		0,06		0,09	
		X		X		0,01		0,06		0,01		0,07		0,08		0,27	

TABLE 3 b) Individual and mean test results for BASt, ULSTER, HOLCIM and KOAC

		BASt				Ulster				HOLCIM				KOAC			
Type (g=good/p=poor)	Code	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt
Meta-sandstone (p)	BAX									0,38	**0,44**	11,72	**11,02**				
										0,47		10,96					
										0,48		10,39					
Micro-diorite (g)	DIK									0,25	**0,34**	0,18	**0,16**				
										0,33		0,17					
										0,43		0,13					
Granite (g)	GAL	0,10	**0,14**	0,06	**0,06**												
		0,11		0,05													
		0,21		0,07													
Greywacke (g)	MIN													0,09	**0,11**	1,14	**1,06**
														0,11		1,12	
														0,12		0,91	
Greywacke (g)	PEX					X	**X**	13,31	**12,16**								
						X		9,48									
						X		13,70									
						X	**X**	13,95	**12,11**								
						X		11,45									
						X		10,94									
Basalt-altered (p)	REF 1	0,89	**0,90**	12,09	**12,07**	X	**X**	20,32	**19,61**	1,78	**1,81**	16,69	**16,91**	2,13	**1,87**	9,49	**10,25**
		0,90		12,04		X		17,85		1,68		16,12		1,55		11,02	
		X		X		X		20,65		1,97		17,92		1,93		X	
						X	**X**	18,21	**18,92**								
						X		18,01									
						X		20,54									
Limestone (p)	REF 2	1,25	**1,41**	39,16	**37,79**					0,90	**0,95**	24,82	**24,32**				
		1,14		39,03						1,06		24,55					
		1,83		35,18						0,88		23,60					
Granite (g)	REF 3	0,20	**0,20**	0,16	**0,17**					0,43	**0,33**	0,35	**0,43**				
		0,19		0,17						0,36		0,38					
		X		X						0,19		0,56					
Gravel (p)	ZIP													0,19	**0,17**	10,20	**11,11**
														0,17		12,74	
														0,16		10,41	

TABLE 3 c) Individual and mean test results for FEHs, NBTL, DTI and LRPC

		FEHs				NBTL				DTI				LRPC			
Type (g=good/p=poor)	Code	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt	spec.	No salt	spec	Salt
Mica rich gneiss (p)	FEP					0,24	**0,23**	0,55	**0,57**								
						0,23		0,73									
						0,23		0,44									
Granite (g)	GAL	0,07	**0,22**	0,25	**0,22**												
		0,12		0,21													
		0,47		0,19													
Granite (g)	HYD													0,25	**0,25**	0,06	**0,08**
														0,30		0,09	
														0,20		0,09	
Oolitic limest. (p)	NUC													0,50	**0,47**	17,12	**16,80**
														0,45		16,25	
														0,45		17,03	
Marine, low flint (g)	QAB									0,17	**0,22**	0,84	**0,82**				
										0,31		0,69					
										0,18		0,91					
Basalt-altered (p)	REF 1	0,78	**0,92**	12,13	**10,79**	1,06	**1,23**	10,66	**11,79**	0,89	**0,90**	10,19	**10,05**	1,33	**1,40**	12,69	**11,36**
		1,06		9,45		1,40		12,93		0,71		9,92		1,38		10,56	
		X		X		X		X		1,09		X		1,49		10,83	
Limestone (p)	REF 2	2,39	**2,35**	26,02	**31,03**												
		2,01		34,31													
		2,67		32,77													
Granite (g)	REF 3	0,12	**0,11**	0,08	**0,11**												
		0,11		0,13													
		X		X													
Landb. por. Flint (p)	RUN									1,84	**1,93**	4,68	**4,69**				
										1,86		4,45					
										2,08		4,93					
Natural gravel (g)	VUX					0,13	**0,14**	0,01	**0,02**								
						0,16		0,04									
						0,12		0,01									

In Table 4 the mean values for all the participating laboratories are summarized.

TABLE 4. Total mean values obtained for all the participating laboratories

		RB		RWTH		SP		TUT		MPA Bau 1		MPA Bau 2		MPA NRW		LRPC Lyon	
Type	Code	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt
Meta-sandst. (p)	BAX	0,91	10,1	0,44	5,55	0,79	13,93										
Granite loose str.(p)	COF	0,07	0,10	0,05	0,10	0,22	0,29	0,18	0,19								
Micro-diorite (g)	DIK	0,11	0,18	0,07	0,09	0,11	0,31										
Mica-gneiss (p)	FEP	0,15	0,19	0,07	0,13	0,42	0,53										
Granite (g)	GAL	0,05	0,09	0,02	0,03	0,11	0,16			0,07	0,16			0,04	0,03		
Basalt-glassy (g)	GEB	0,54	4,27	0,72	2,92	0,73	7,59										
Granite (g)	HYD	0,04	0,38	0,10	0,85	0,21	0,80									0,25	0,08
Greywacke (g)	MIN	0,09	0,85	0,03	0,23	0,16	1,61										
Oolitic limest. (p)	NUC	0,4	18,77	0,17	3,23	0,55	24,56									0,47	16,80
Greywacke (g)	PEX	0,31	6,31	0,04	3,38	0,43	16,57										
Mar., low flint (g)	QAB	0,12	0,52	0,02	0,48	0,08	1,00										
Basalt-altered (p)	REF 1	1,06	9,80	1,05	16,00	1,60	13,02	1,53	13,46	1,17	12,32	0,33	11,76	0,99	15,53	1,4	11,36
Limestone (p)	REF 2	1,72	29,17	1,24	11,27	1,80	39,04	2,19	33,92	2,13	38,96	1,99	35,82	2,72	38,99		
Granite (g)	REF 3	0,08	0,19	0,06	0,03	0,28	0,38	0,26	0,16	0,01	0,05	0,02	0,08	0,06	0,15		
Land. por. flint (p)	RUN	1,25	5,01	0,41	1,34	1,35	3,56										
Natural gravel (g)	VUX	0,12	0,12	0,09	0,03	0,20	0,29										
Granite (g)	XYD	0,13	2,63	0,08	6,86	0,34	5,07										
Gravel (p)	ZIP	0,14	8,72	0,21	11,74	0,21	13,37										

MPA Bau delivered two test results for each reference sample, so it apperars that they received two bags of each reference sample

		BASt		Ulster		HOLCIM		KOAC		LfB AG		FEHs		NBTL		DTI	
Type	Code	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt	No salt	Salt
Meta-sandst. (p)	BAX					0,44	11,02										
Granite loose str.(p)	COF																
Micro-diorite (g)	DIK					0,34	0,16										
Mica-gneiss (p)	FEP													0,23	0,57		
Granite (g)	GAL	0,14	0,06							0,09	0,17	0,22	0,22				
Basalt-glassy (g)	GEB																
Granite (g)	HYD																
Greywacke (g)	MIN							0,11	1,06								
Oolitic limest. (p)	NUC																
Greywacke (g)	PEX			X	12,1												
Mar., low flint (g)	QAB															0,22	0,82
Basalt-altered (p)	REF 1	0,90	12,07	X	19,3	1,81	16,91	1,87	10,25	1,32	11,96	0,92	10,79	1,23	11,79	0,9	10,05
Limestone (p)	REF 2	1,41	37,79			0,95	24,32			2,03	38,35	2,35	31,03				
Granite (g)	REF 3	0,20	0,17			0,33	0,43			0,11	0,31	0,11	0,11				
Land. por. flint (p)	RUN															1,93	4,69
Natural gravel (g)	VUX													0,14	0,02		
Granite (g)	XYD																
Gravel (p)	ZIP							0,17	11,11								

UU tested only with salt (Ulster) — *Mixed marking, corrected* (HOLCIM)

The mean values (of all laboratories testing) for each aggregate sample tested with and without salt are presented in Table 5.

Table 5. Mean values for each aggregate sample tested with and without salt

		MEAN	MEAN
Type	**Code**	**No salt**	**Salt**
Meta-sandstone (p)	BAX	0,65	10,15
Granite loose structure (p)	COF	0,13	0,17
Micro-diorite (g)	DIK	0,16	0,19
Mica-gneiss (p)	FEP	0,22	0,36
Granite (g)	GAL	0,09	0,12
Basalt-glassy (g)	GEB	0,66	4,93
Granite (g)	HYD	0,15	0,53
Greywacke (g)	MIN	0,10	0,94
Oolitic limestone (p)	NUC	0,40	9,70
Greywacke (g)	PEX	0,26	9,59
Marine, low flint (g)	QAB	0,11	0,71
Basalt-altered (p)	REF 1	1,21	12,90
Limestone (p)	REF 2	1,87	32,61
Granite (g)	REF 3	0,14	0,19
Land. porous flint (p)	RUN	1,24	3,65
Natural gravel (g)	VUX	0,14	0,12
Granite (g)	XYD	0,18	4,85
Gravel (p)	ZIP	0,18	11,24

Figures 1 and 2 demonstrate the mean frost resistance of the aggregates according to the two methods used. The mean values are calculated on the grounds of all test results obtained from all laboratories involved in testing each aggregate sample.

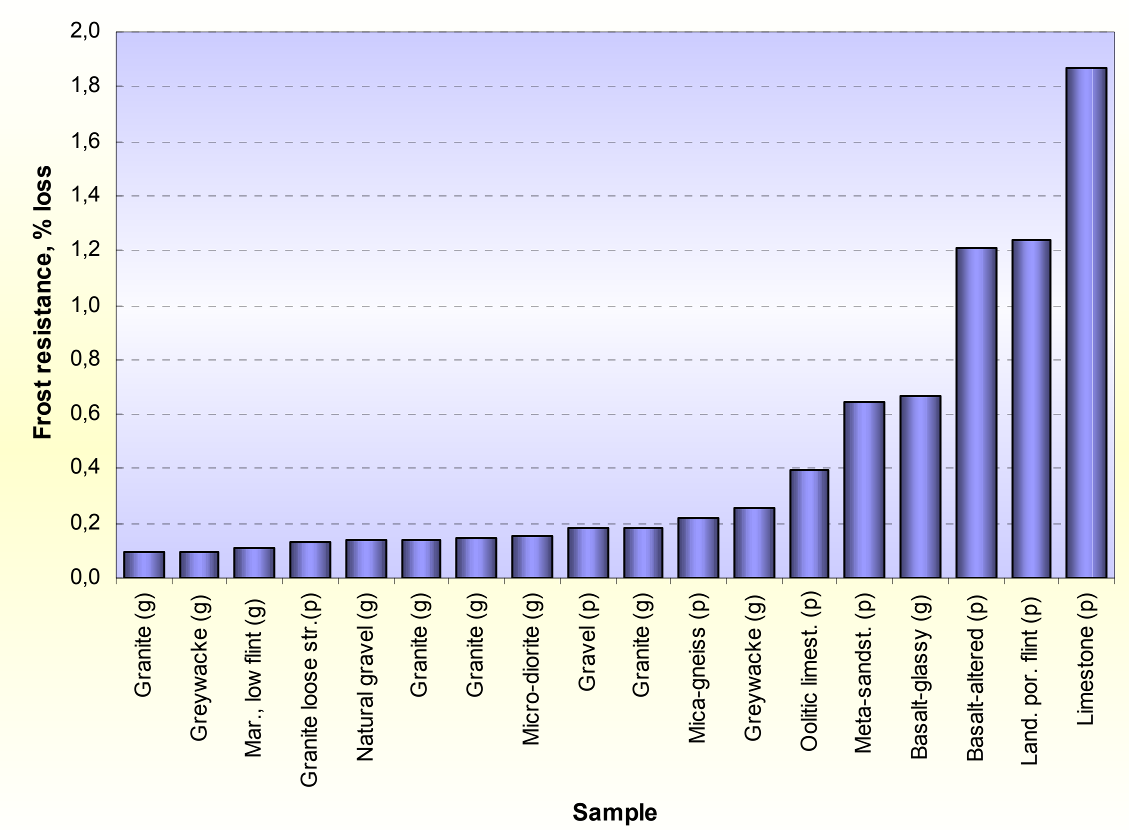

Figure 1 Average freeze/thaw breakdown, combined test draft, without salt

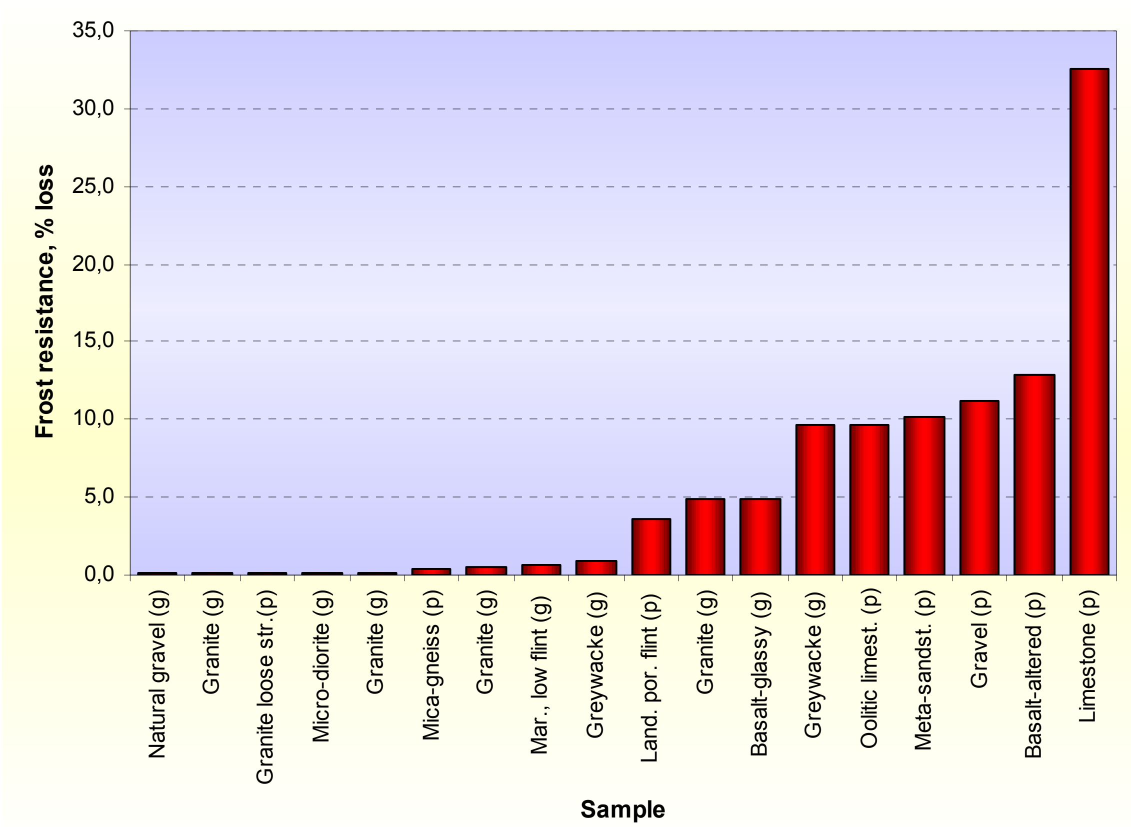

Figure 2 Average freeze/thaw breakdown, combined test draft, with 1 % NaCl

It should be pointed out that the scales on the next two figures are very different from each other; one comprises values up to 2,0 % breakdown, while the other comprises values up to 35 % breakdown.

5. DISCUSSION OF FROST TEST RESULTS

5.1 Results using the fresh water method

It is obvious from figure 1, that the pure water method produces very little breakdown and all of the mean values are below 2 % breakdown and only 3 mean values are more than 1 %. This would mean, according to CEN/TC154/SC´s product standards, that all of the aggregates would be considered, at least, fairly good in respect to freeze/thaw resistance, and 15 out of 18 aggregate samples would be considered as first class material. In fact, two of the samples having mean values over 1 % were close to 1,2 % and only one had a value close to 2 % (1,87 %). Bearing in mind requirement categories in the product standards of CEN/TC 154, it is obvious that the fresh water method does not distinguish adequately between frost resistant and frost susceptible aggregates. In other words: Testing in pure water, does not give a wide spread in values of the aggregates tested, although the aggregates were chosen on the grounds of quality or performance.

The correlation coefficients between individual key laboratories using the fresh water method are shown in figures 3 a) to c).

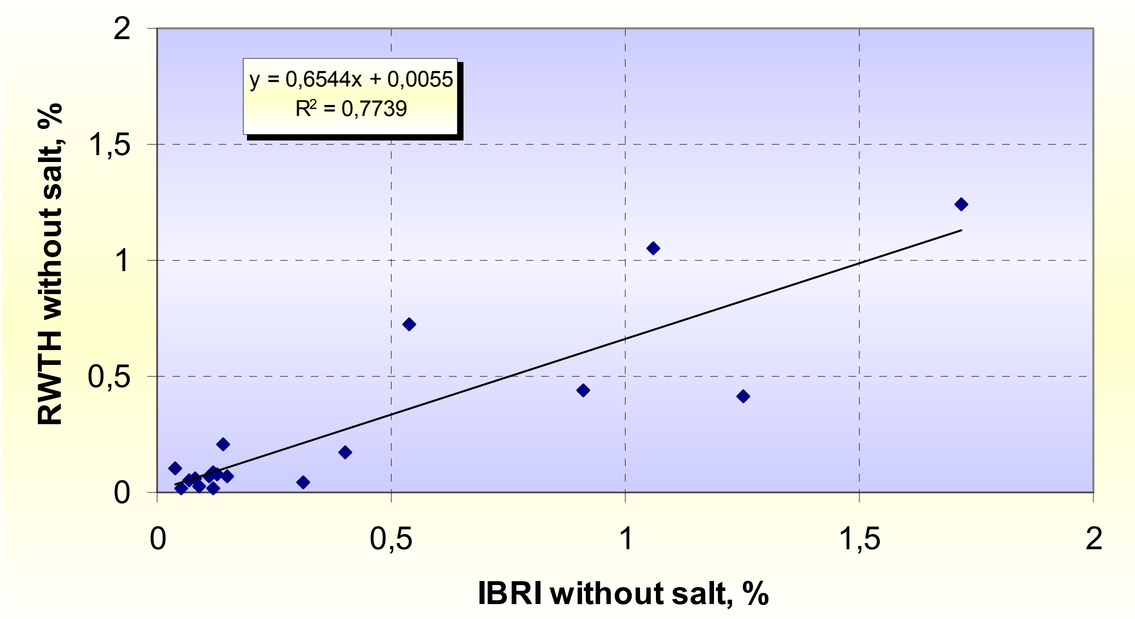

Figure 3 a) Correlation between mean test results without salt from IBRI and RWTH

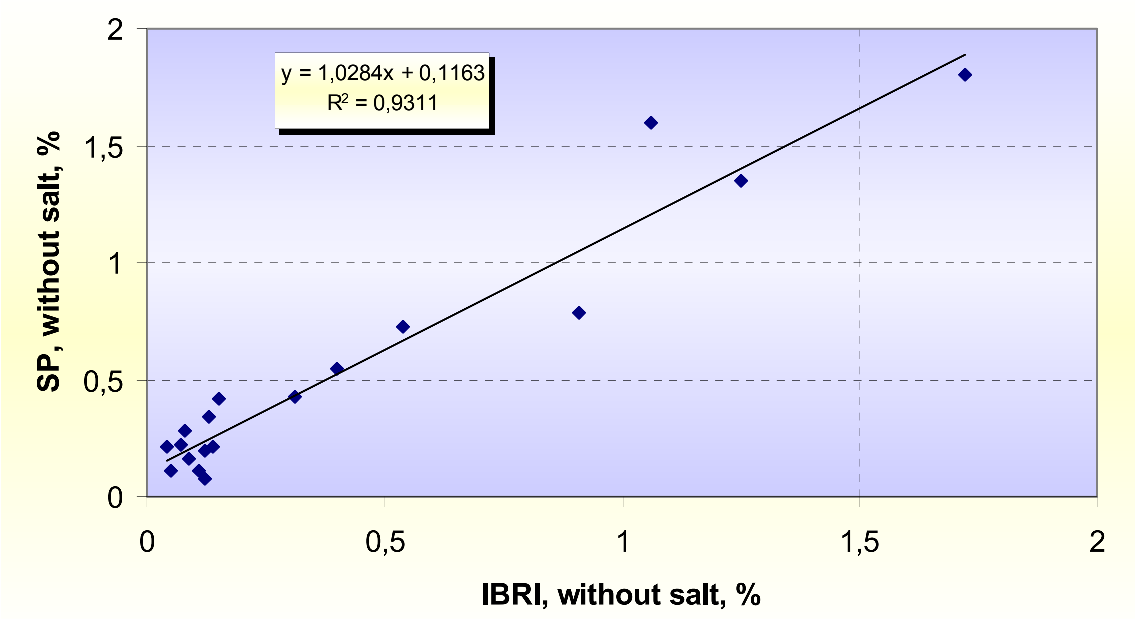

Figure 3 b) Correlation between mean test results without salt from IBRI and SP

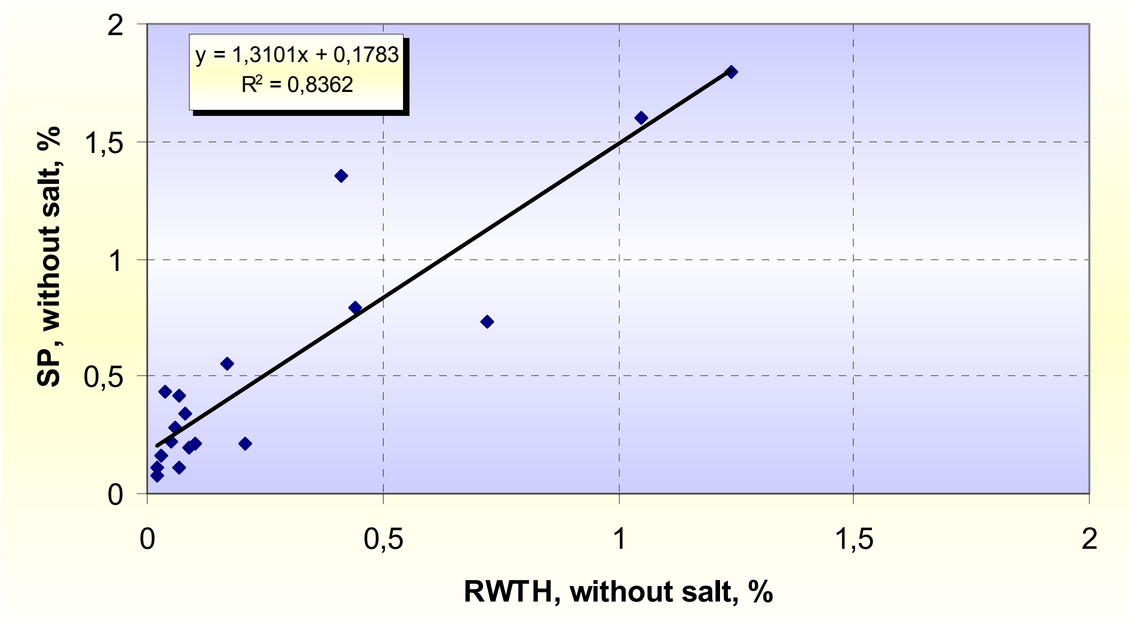

Figure 3 c) Correlation between mean test results without salt from RWTH and SP

The coefficients appear to be significant, although the line of equality is not the same as the trend line. The highest correlation obtained is between results from IBRI and SP without salt.

5.2 Results using the salt water method

Figure 2 shows the mean test results when using 1 % NaCl.

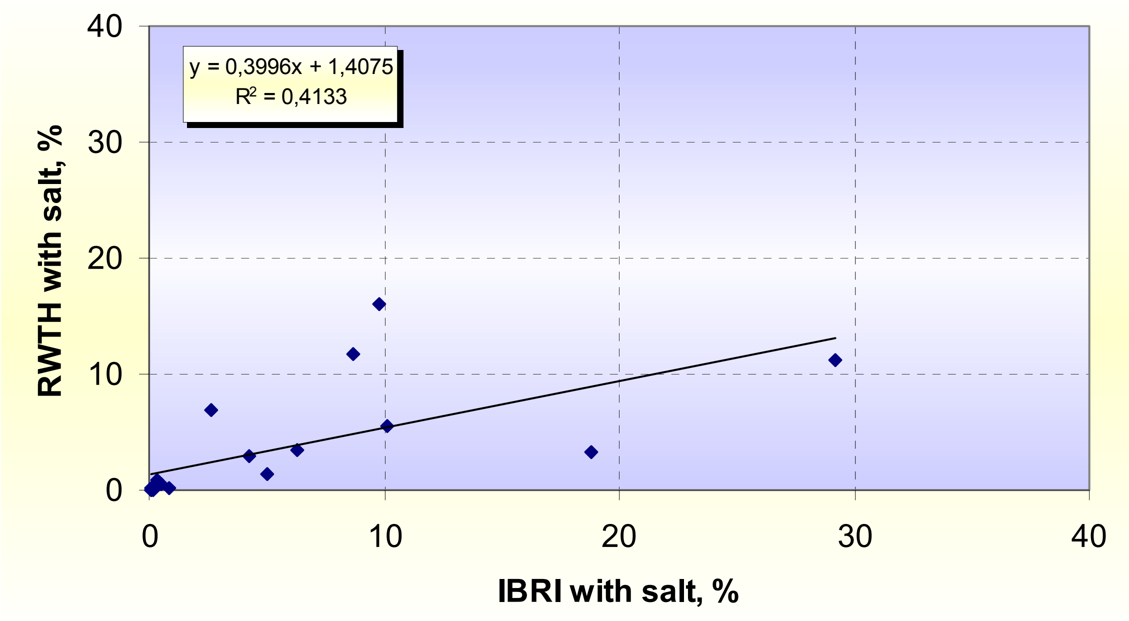

Figure 4 a) Correlation between mean test results with salt from IBRI and RWTH

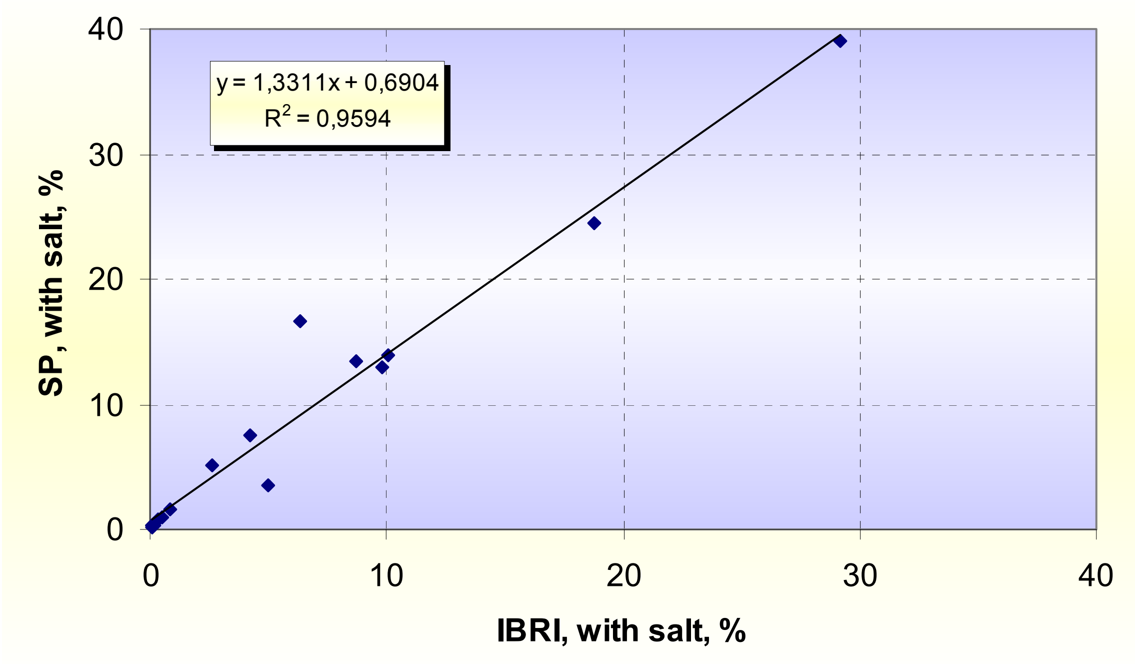

Figure 4 b) Correlation between mean test results with salt from IBRI and SP

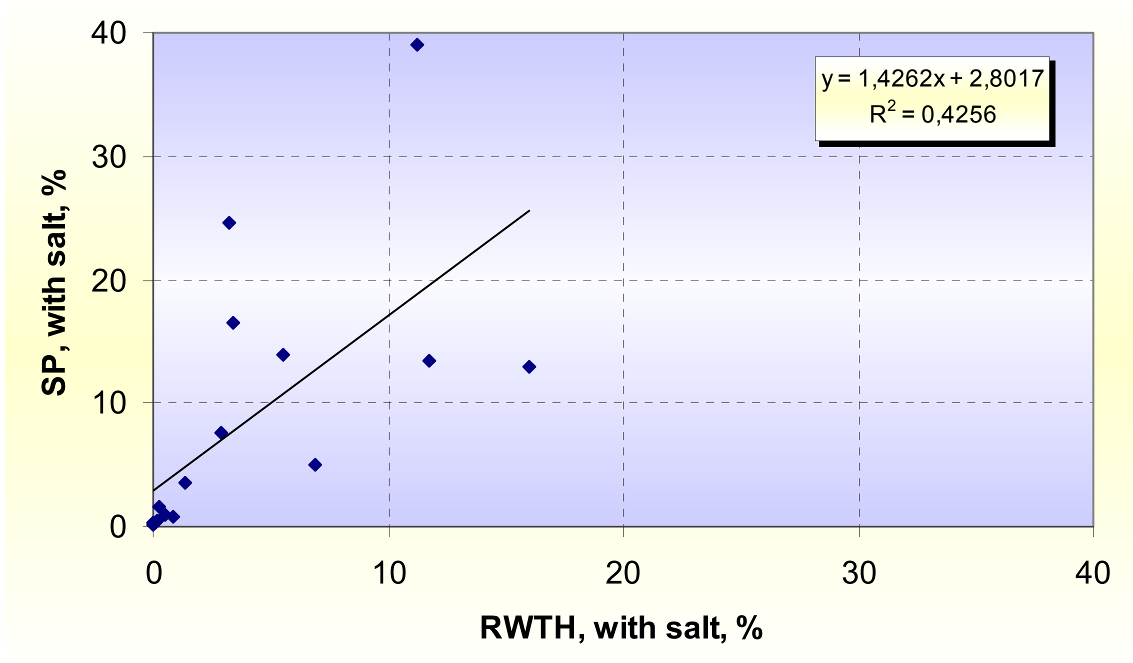

Figure 4 c) Correlation between mean test results with salt from RWTH and SP

The correlation coefficients between individual key laboratories using the salt-water method, which are shown in figures 4 a) to c), appear to be significant, although the line of equality is not the same as the trend line. The highest correlation is between results from IBRI and SP obtained when using salt water, as was the case also without salt.

5.3 Comparison between the two test methods

Figure 5 shows the correlation between all the mean frost resistance values (of each laboratory) obtained in the project with and without salt.

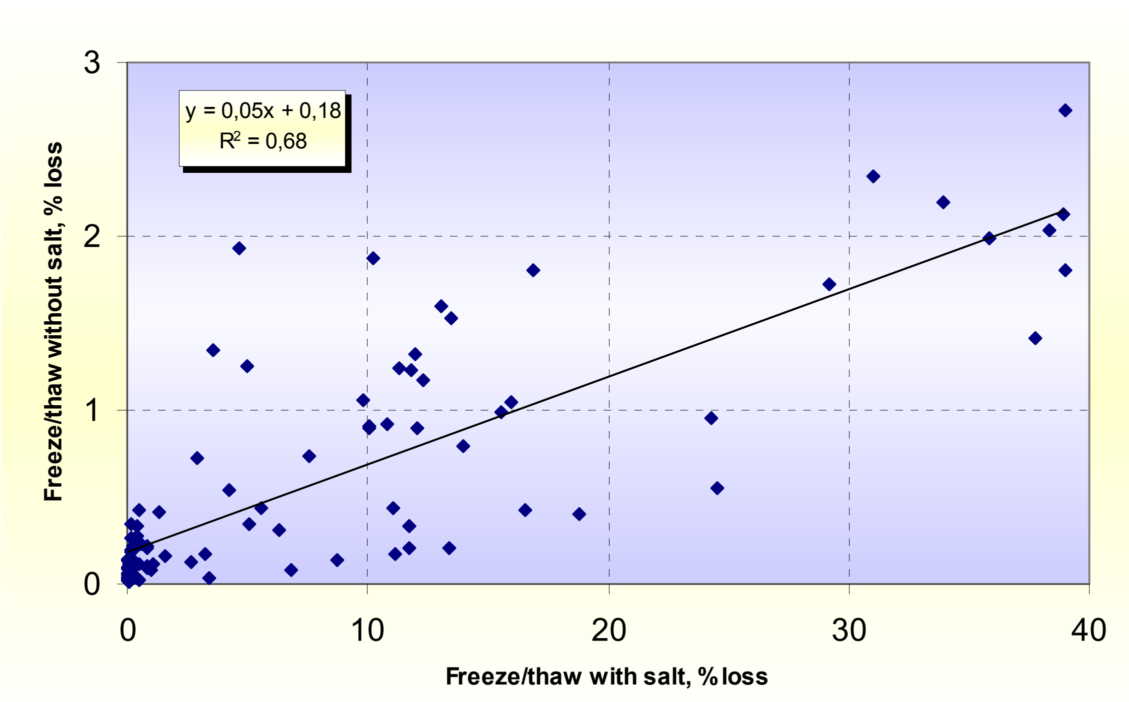

Figure 5 Mean test results (all laboratories)

The figure clearly demonstrates the difference in the magnitude of freeze/thaw breakdown according to the two test methods, when plotting the mean test results of all participating laboratories, see also table 4. The trend is that when using salt, the loss in mass is ten to twenty times the loss in pure water.

Figure 6 draws the attention to test results of the three key laboratories, when testing all 18 aggregate samples with and without salt.

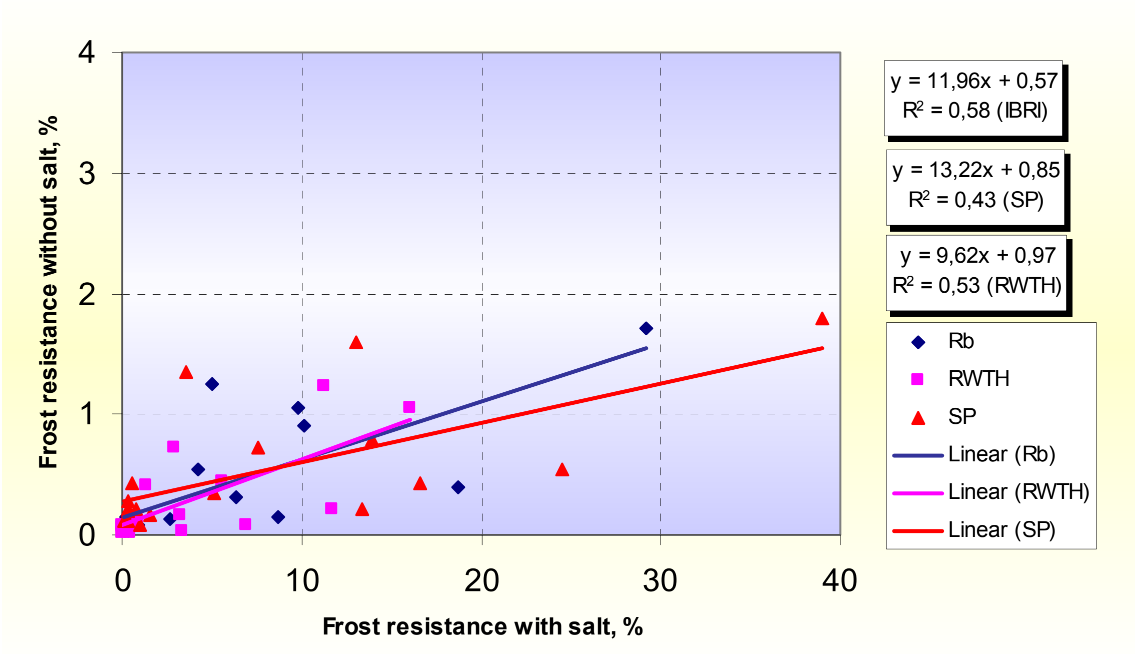

Figure 6 Test results of the three key laboratories

The trend is similar for all three laboratories although the magnitude is considerably lower in general, both with and without salt at RWTH than at IBRI and SP.

Figure 7 shows the test results when testing the reference samples at all the laboratories that tested all three reference samples.

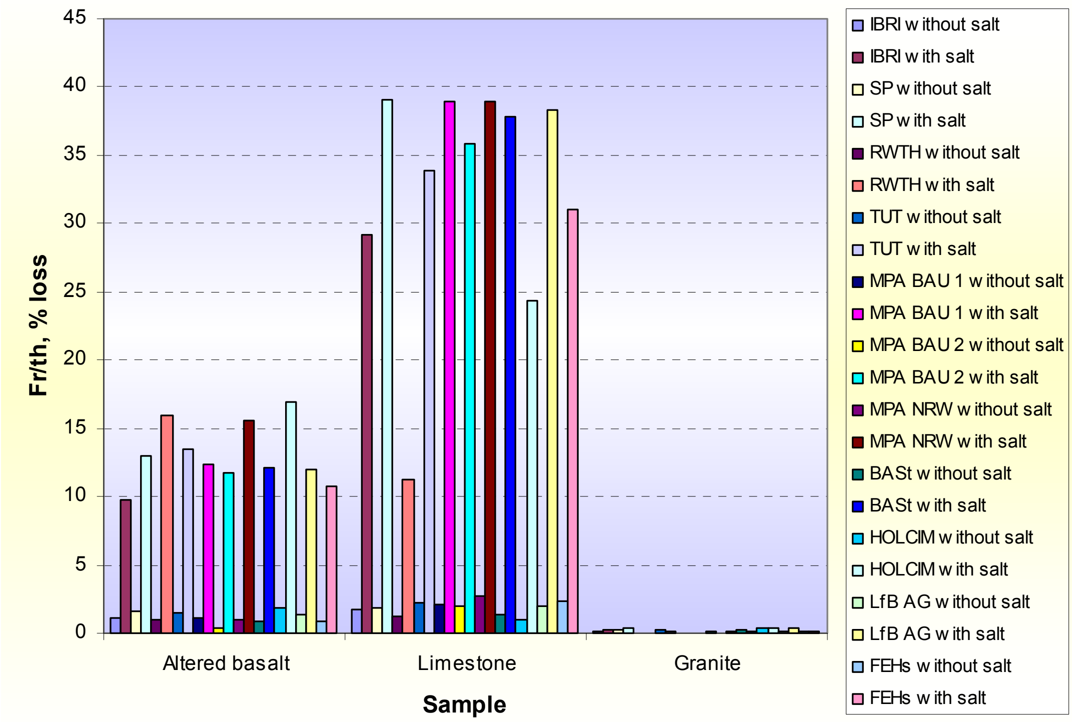

Figure 7 Test results when testing the reference samples at all the laboratories that tested all three reference samples.

The figure shows that there is a distinct difference in the test values between aggregate sources. The altered basalt (Reference sample no. 2) has values less than 1 % when tested in pure water, but generally between 10 and 15 % when tested with salt.

The extremely poor limestone has values generally between 1 and 3 % in pure water, but 30 to 40 % when tested with the salt solution. There is, however, one test result in salt water, which is distinctly lower than all the others, the 11,3 % loss obtained at RWTH. All the three test specimens have similar values, between 11 and 11,5 % loss, so it is not obvious at this stage what causes this relatively low test result.

The good granite sample presented in figure 7 receives test results, which are very low and similar regardless of testing with or without salt. Obviously the salt solution does not affect the measured frost resistance of this aggregate and is therefore not harmful in any way when testing frost resistant aggregates. Figure 8 confirms that one half of the aggregates tested are not affected by the salt solution to any extent.

Figure 8 shows the mean test results for all the aggregates tested in the FRAS-project.

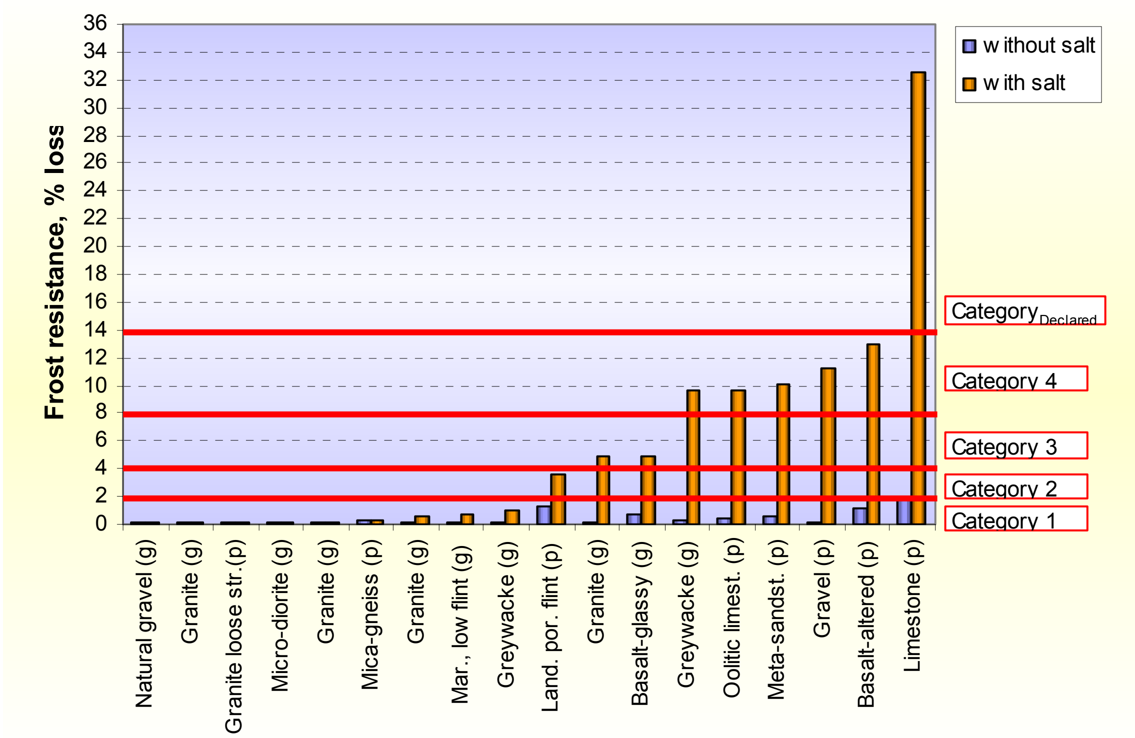

Figure 8 Mean test results for all the aggregates tested in the FRAS-project

It is obvious from this picture that when comparing the mean numerical values received for all the aggregates with and without salt the difference is striking, as stated before. To evaluate the general picture when using the salt-water method, Icelandic requirement categories have been drawn on the figure to give a graphical impression of how the FRAS aggregates would be ranked according to the existing categories (see Chapter 9). The categories have been in use in Iceland since 1995, mainly for aggregates for road construction. It is evident that nine out of the 18 aggregates would be classified as first class material for all purposes, according to the categories presented on the figure. Three additional aggregate samples would fall into categories 2 and 3, and could therefore be considered intermediate concerning frost resistance. The remaining aggregates would be considered of poor frost resistance and thereof one (limestone) extremely poor. This is in general harmony with what might have been expected in accordance with former knowledge of the general quality of the aggregates tested.

Although the general picture is rather convincing regarding quality classification when using the salt water method, there are a few anomalies regarding the quality assessment (good or poor) and results from freeze/thaw testing. This applies for both test methods, with and without salt. This has been discussed and explained in chapter 3 of this report.

6. WATER ABSORPTION AND PARTICLE DENSITY MEASUREMENTS (SSD)

Testing the water absorption is in many cases used as a screening method for assessing the frost resistance of aggregates and natural stones. It was therefore decided to determine the water absorption of the test materials and evaluate the potential correlation.

The water absorption and particle density (ssd) of the tested aggregates were measured according to EN 1097-6, using the pyknometer method /6/.

Table 6 shows the results of all the water absorption and relative density (ssd) measurements. It can be stated here that correlation between water absorption and particle density is not observed, nor between particle density and frost resistance (regardless of salt). Therefore, particle density is of no importance to this project and is not discussed further.

Table 6 Water absorption and particle density measurements

Aggregate sample	Label	Measuring laboratory	Water absorption %	Particle Density Mg/m^3
Meta-sandstone, poor	BAX	HOLCIM	**3**	**2,65**
Granite loose str., poor	COF	TUT	**0,5**	**2,75**
Micro-diorite, good	DIK	HOLCIM	**1**	**2,7**
Mica rich gneiss, poor	FEP	NBTL	**0,7**	**2,79**
Granite, good	GAL	BASt	**0,46**	**2,68**
Glassy basalt, good	GEB	IBRI	**1,67**	**2,85**
Granite, good	HYD	IBRI	**0,53**	**2,79**
Greywacke, good	MIN	IBRI	**0,54**	**2,71**
Oolitic limestone, poor	NUC	IBRI	**1,28**	**2,64**
Greywacke, good	PEX	IBRI	**1,34**	**2,67**
Marine, low flint, good	QAB	IBRI	**0,66**	**2,64**
Altered basalt, poor	Ref 1	IBRI/BASt*	**2,39**	**2,83**
Limestone, poor	Ref 2	BASt	**0,99**	**2,68**
Granite, good	Ref 3	TUT/BASt*	**0,26**	**2,63**
Landbased por. flint, poor	RUN	IBRI	**3,65**	**2,43**
Natural gravel, good	VUX	NBTL	**0,5**	**2,69**
Granite, good	XYD	SP	**1,0**	**2,63**
Gravel, poor	ZIP	IBRI	**1,5**	**2,61**

**mean value*

Figure 9 shows the test results when comparing the water absorption with the mean frost resistance with salt water of all the aggregates tested.

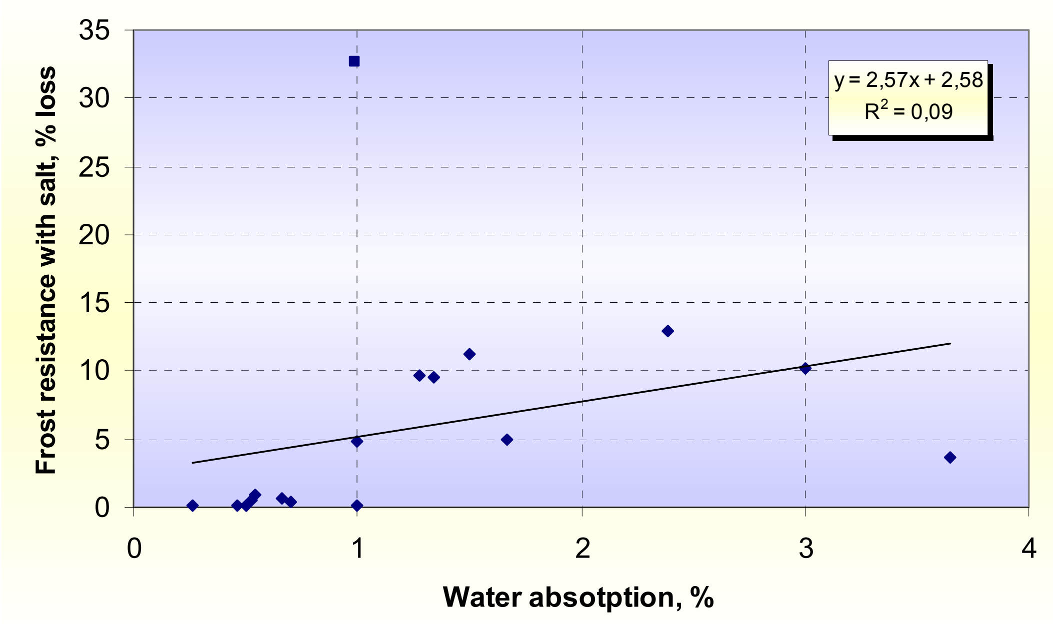

Figure 9 Test results when comparing the water absorption with the mean frost resistance with salt water

It can be seen that most of the aggregates with water absorption values below 1 % are indeed frost resistant according to the freeze/thaw test results. This is however not the case for the extremely poor limestone aggregate, with water absorption just below 1 %, but frost resistance value of over 30 %. Figure 10 shows the test results when comparing the water absorption with the mean frost resistance without salt water of all the aggregates tested.

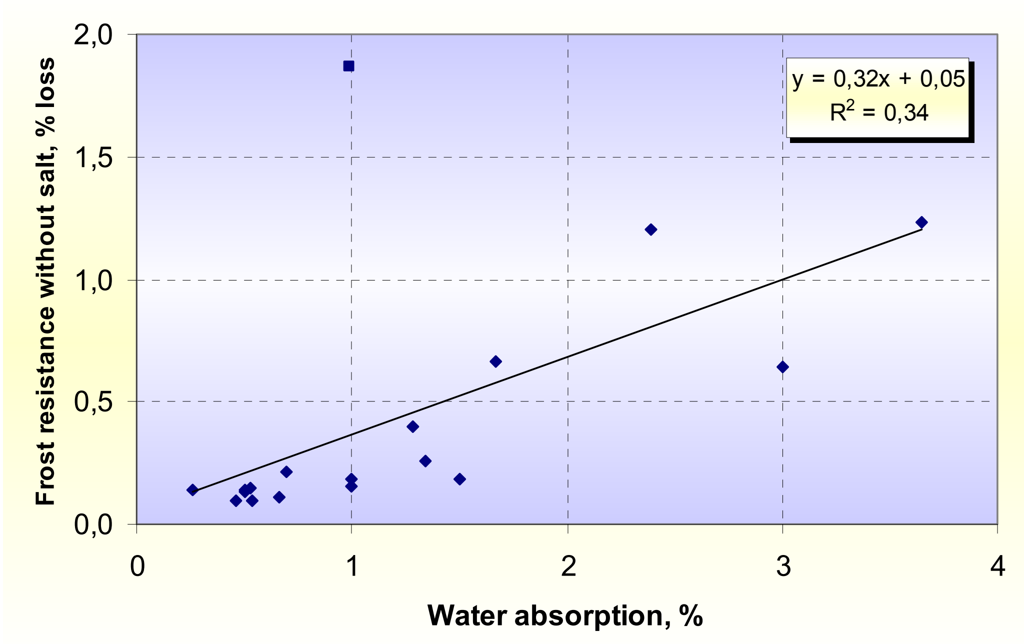

Figure 10 Test results when comparing the water absorption with the mean frost resistance without salt water

It is more difficult to conclude similarly about figure 10 as figure 9, as almost all the frost resistance test values are low. Still, it can be seen from the figure that low water absorption generally indicates low frost resistance values.

The two figures above demonstrate a few things, which are important to point out.

- The correlation between water absorption and frost resistance without salt is higher than when using salt, although it is not very strong.
- One sample, the extremely poor limestone aggregate (Ref. 2) according to freeze/thaw testing results, has water absorption just below 1 % and therefore the water absorption, screening procedure would overlook this aggregate as frost susceptible.
- Eleven of the aggregates tested have water absorption values of ≤1 %, but three of these have been evaluated as being of poor predicted quality. Two of the seven aggregates with water absorption values >1 % were considered to be of good quality.

7. TEMPERATURE MEASUREMENTS

It is very important to have information about the sample temperature in the testing process of the aggregates. One part of the questionnaire, which was sent to all participating laboratories, was to have detailed information on a typical temperature curve, i.e. temperature vs. time. A summary of the answers to the questionnaire is given in Appendix V, but figure 11 below shows typical temperature curves as given by most of the participants in a graphical manner.

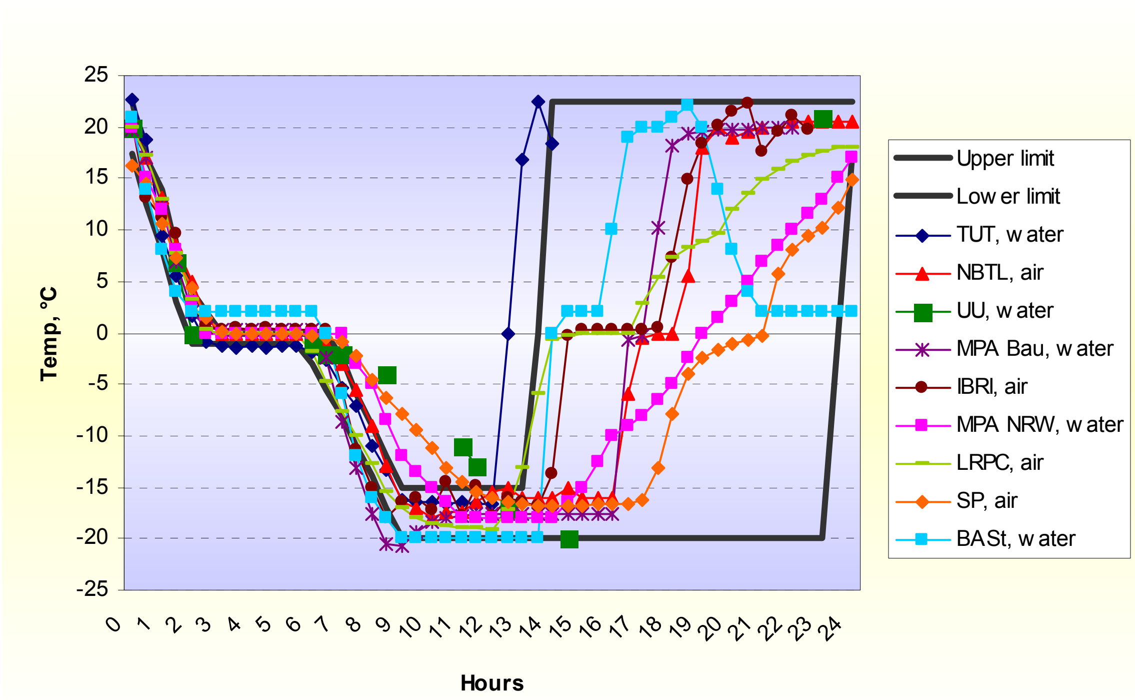

Figure 11 Typical temperature curves obtained from individual laboratories participating in the FRAS project

Upper and lower temperature limits on figure 11 are according to the combined test method. It is evident from the figure that the cooling phase from +20°C, through 0 to –1°C and down to –17,5°C, is fairly consistent for most of the laboratories. The thawing phase from –17,5°C to

+20°C is on the other hand quite different from one laboratory to the other, but still within limits. The concluding points that can be drawn to attention are:

- It is evident that both thawing out methods used (water bath or air temperature control), are equally accurate (see Chapter 8).
- It is evident that the lack of specification of the thawing out temperature causes undesirable differences between laboratories in the thawing phase.
- It is likely that temperature sensors used may be inaccurate in some cases. An example of that is the BASt sensor, which gives readout of several degrees, plus, when the ice is forming, but should be between 0 and –1°C (ice-water temperature). Another example in the other direction (below -1°C in the ice-forming phase) is the TUT sensor.

8. PRECISION EVALUATION

According to ISO 5725 at least 8 laboratories shall participate in a precision trial. The laboratories shall normally be chosen at random among those having different experience with the method. At least 3 different materials of different qualities have to be used.

Since the procedure is rather new to a number of laboratories, it was difficult to gather 8 laboratories with experience in the method. The answers to a questionnaire concerning the experience can be found in Annex V.

Another difficulty concerned the selection of the three reference materials. Pre-existing knowledge about them came from their performance in construction application and petrographic analyses, not by testing in accordance with the frost resistance test methods. These constraints shall be born in mind when assessing the test results. Still, it is evident that the goal to choose one poor, one average and one good reference sample with respect to frost resistance was successful.

8.1 Objectives for the precision trial

The primary objective with the precision trial was to establish the repeatability and reproducibility of the proposed salt-water method and the present reference method. Secondly, to evaluate the possible difference in test results when using different thawing procedures (thawing in air versus thawing in a water bath).

8.2 Statistical evaluation - results

The results of the statistical computation of the test results are summarized in the table 6 and the diagrams below. The statistical data are given in full in appendix IV.

Table 6 shows the relationship between the mean test result and the precision. It also gives the correlation coefficient for each relationship. The equations in bold are those normally used as representing the precision of a test method, i.e. with outliers excluded.

The diagrams display the test results when testing in pure water and a 1 % NaCl solution, without outliers. Outliers are defined in accordance with Cochran's test, ISO 5725-94. Diagrams showing the evaluation of the thawing procedure, whether thawing in air or water has any influence on the final test result or the precision, are given in Appendix IV.

The results of the statistical computation of the test results are displayed in the diagrams below. The statistical data are given in full in appendix IV. The repeatability (r), and the reproducibility (R) can be retrieved by multiplying the sr and sR, respectively, with the factor 2,78.

Table 6 Results of the statistical analysis

Test procedure and calculations	Repeatability standard deviation (sr)	Correl. coeff. (R^2)	Reproducibility standard deviation (sR)	Correl. coeff. (R^2)
Pure water (Water)	sr = 0,170X + 0,012	0,85	sR = 0,580X + 0,097	0,87
1 % NaCl (Salt)	sr = 0,104X + 0,026	0,91	sR = 0,650X + 0,772	0,70
Water, outliers deleted	**sr = 0,167X + 0,007**	0,96	**sR = 0,580X + 0,070**	0,86
Salt, outliers deleted	**sr = 0,048X + 0,215**	0,58	**sR = 0,676X + 0,674**	0,72
Water, thawing in air	sr = 0,150X + 0,020	0,81	sR = 1,700X + 0,050	0,97
Salt, thawing in air	sr = 0,130X + 0,120	0,78	sR = 1,860X + 0,470	0,99
Water, thawing in water	sr = 0,170X + 0,020	0,69	sR = 1,620X + 0,030	0,99
Salt, thawing in water	sr = 0,067X + 0,210	0,67	sR = 1,620X + 3,690	0,92

Table 7 shows the coefficient of variation when testing in pure water and a salt solution. Outliers are included but don't significantly influence the result. It can be seen that the coefficient of correlation are in the same order of magnitude for both tests, although somewhat smaller when using salt.

Table 7 Coefficient of variation when testing in pure water and a salt solution

Testing conditions		Salt solution		Pure water	
		coeff variation (%)		coeff variation (%)	
Type	Code	sr	sR	sr	sR
Meta-sandstone (p)	BAX	8,6	59,9	23,4	67,6
Granite loose str.(p)	COF	15,1	96,5	20,7	125,8
Micro-diorite (g)	DIK	26,2	92,5	35,6	137,8
Mica rich gneiss (p)	FEP	8,2	132,6	9,0	96,8
Granite (g)	GAL	34,6	112,1	25,1	98,2
Basalt-glassy (g)	GEB	11,2	85,2	18,6	33,2
Granite (g)	HYD	25,4	71,3	29,0	112,1
Greywacke (g)	MIN	19,9	107,7	28,1	100,9
Oolitic limest. (p)	NUC	5,4	141,3	9,7	89,9
Greywacke (g)	PEX	17,2	143,4	15,7	146,3
Marine, low flint (g)	QAB	11,5	55,9	53,0	147,1
Basalt-altered (p)	REF 1	9,0	70,7	17,2	58,4
Limestone (p)	REF 2	3,6	55,3	16,5	55,8
Granite (g)	REF 3	37,2	130,1	11,4	104,2
Landb. por. Flint (p)	RUN	6,9	57,6	18,4	89,9
Natural gravel (g)	VUX	28,0	187,5	17,0	60,4
Granite (g)	XYD	12,8	77,0	13,4	127,5
Gravel (p)	ZIP	11,8	32,0	16,8	36,3
Mean		**16,3**	**94,9**	**21,0**	**93,8**

Figures 12 to 15 show the results of the precision evaluation in a graphical manner. In all cases, the outliers have been deleted.

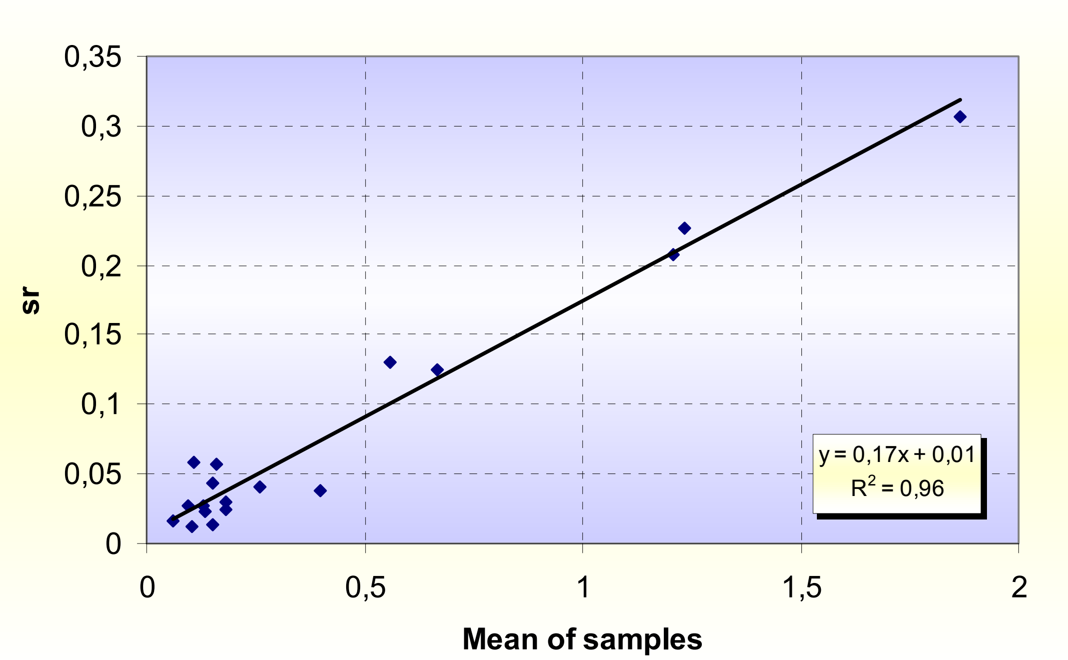

Figure 12 The diagram shows the repeatability standard deviation (sr) for all mean values when testing in pure water. Outliers have been deleted.

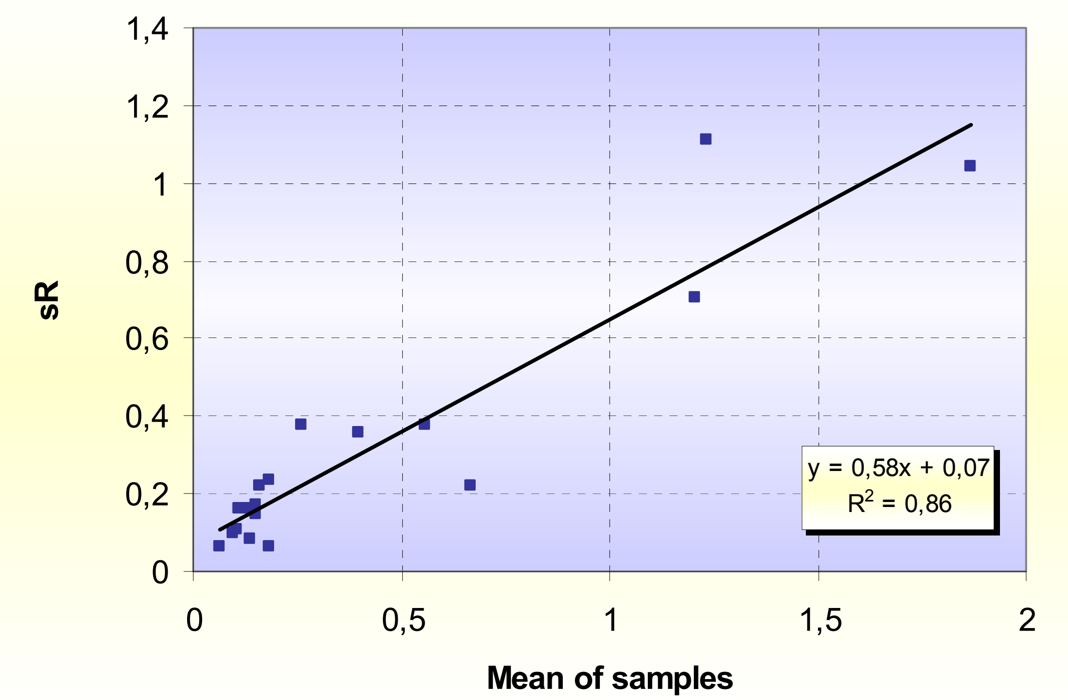

Figure 13 The diagram shows the reproducibility standard deviation (sR) for all mean values when testing in pure water. Outliers have been deleted.

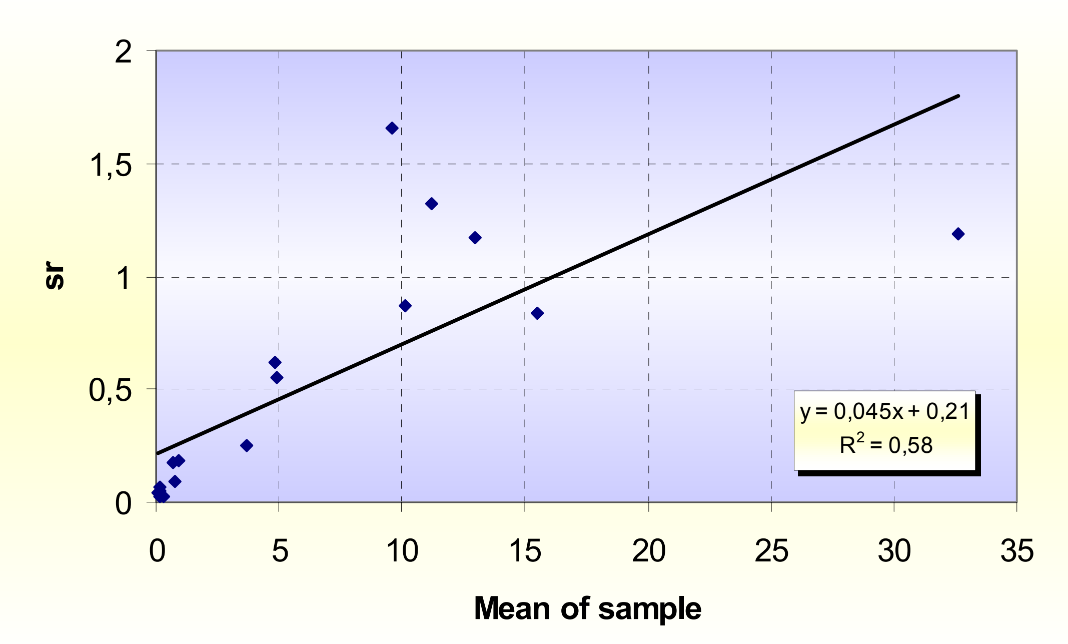

Figure 14 The diagram shows the repeatability standard deviation (sr) for all mean values when testing in a salt solution (1% NaCl). Outliers have been deleted.

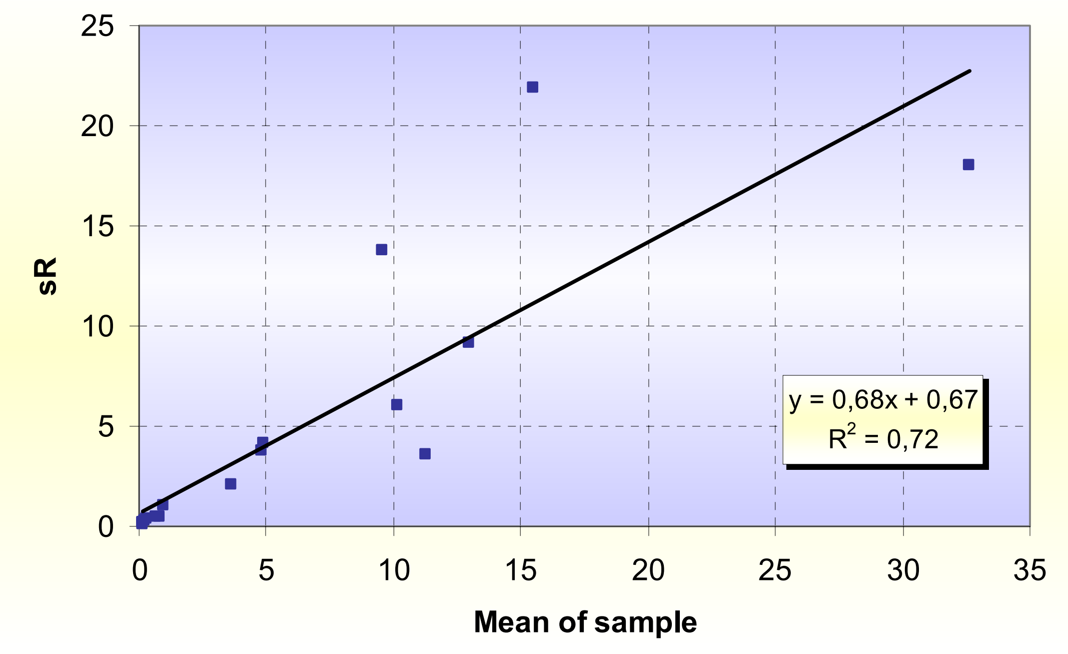

Figure 15 The diagram shows the reproducibility standard deviation (sR) for all mean values when testing in a salt solution (1% NaCl). Outliers have been deleted.

9. DISCUSSION

The Nordtest research project no. 1624-03 is now completed. It involved testing the frost resistance of 18 European aggregate samples at 15 laboratories in 10 European countries. The aggregate samples were tested in accordance with two testing procedures, one with pure water and the other with 1% NaCl solution. One of the main goals of the project was to test the hypothesis that testing the frost resistance of aggregate in fresh water does not distinguish between frost resistant and frost susceptible aggregates. Also, to test the hypothesis that the test method with 1 % NaCl solution distinguishes between frost resistant and frost susceptible aggregates. Thirdly, to test the hypothesis that it does not affect test results whether the thawing out process is controlled with a water bath or air temperature in the freeze/thaw-cabinet. All three aspects have now been confirmed.

Requirement categories for test results when using the salt-water method have been in use in Iceland for almost 10 years. The categories used are ≤ 2 %, ≤ 4 %, ≤ 9 % and ≤ 14 % loss in mass of the original sample. The FRAS-project suggests modifying these requirement categories to 2, 4, 8 and 14 % for consideration in the relevant SC's of TC 154.

Another part of the project was to measure water absorption and relative density of the aggregates according to EN 1097-6 and compare the results with frost resistance test results with and without salt. This was done to evaluate the proposed CEN screening water absorption values for frost resistance of aggregates but the product standards state that if the water absorption is less than 1 %, the aggregates can be considered frost resistance. It is concluded in this project that although low water absorption is connected with low freeze/thaw values in most cases, the screening value does not always give the right picture of the frost resistance of aggregates. As an example of that is the extremely poor limestone aggregate (Reference sample 2), which has a water absorption value of less than 1 %.

The precision evaluation indicates that both test methods, with and without salt, can be considered accurate to some extent, although this project does not demonstrate that either method is very accurate. The explanation for inconsistency is not obvious, but there may be a combination of several influencing factors. One possibility is the result of different sieving procedures at different laboratories or even within the same laboratory. It is quite possible that aggregates of poor frost resistance produce weaknesses, which might break if the sieving procedure is too harsh, but the sieving procedure does not specify precisely the time period of sieving or the harshness of sieving. Another explanation may be due to different amplitudes of freeze/thaw cycles at different places or levels in the freeze/thaw cabinets. Thirdly, the thawing phase of the temperature curve may need to be specified in more detail. The fact that some of the participants were not familiar with the testing procedure and in fact some were testing for the first time, may have caused some errors.

The precision data when testing in pure water is approximately the same when all results are used and when outliers are excluded. In general, the resulting values (comminuting of the particles) are very low. The difference between test results for good aggregates and poor aggregates is very small. This also leads to small precision values, although the method does not adequately discriminate between frost susceptible and frost resistant rock types.

When using pure water, the precision is seemingly better compared to testing in a salt solution. However, the precision is about the same when the order of magnitude of the results is considered (see table 7 with coefficient of variation). In fact, the coefficient of variation, as a whole, is smaller when using salt.

The repeatability when using salt is approximately halved when excluding the outliers, whereas the reproducibility is not affected. The scatter in results is rather high for several laboratories for both methods. This affects the result of the whole precision evaluation. The more scatter in all of the results, the fewer the outliers.

As expected, the reproducibility is larger than the repeatability. However, it is between four and six times higher which clearly indicates one of two things: The laboratories have not strictly followed the method description in detail or several parts of the method need to be better specified. The evaluation also indicates that the precision is approximately the same when controlling the thawing phase in air, as when thawing in water is applied.

10. CONCLUSIONS

- The research project has led to drafting a combined test method for measuring frost resistance of loose aggregate with the options to test in pure water or using 1 % NaCl solution as well as controlling the thawing out sequence either by air temperature or with immersion in 20°C water bath.
- 15 of 18 aggregate samples tested in pure water suffered very little breakdown (< 1 %) and the remaining three aggregates obtained values below 2 %. It can therefore be stated that testing in pure water produces insufficient breakdown to distinguish adequately between frost resistant and frost susceptible aggregates, according to service records or quality rating of the aggregates tested in this project.
- The salt-water method produced considerable difference in test results between aggregate samples, from almost no breakdown up to 35 % for the poorest aggregates.
- The water absorption measurements reflect the frost resistance of aggregates to some extent. However, when bearing in mind the recommended water absorption limit of 1 % in the product standards of CEN/TC 154, some aggregates would be considered frost resistant according to their water absorption, but are not according to frost resistance test results when using a salt solution. The test results when using pure water are not comparable in the same manner as all the test values are too low.
- Although a fairly good correlation exists between test results with and without salt, the magnitude of the test results when testing without salt is too small to distinguish between frost resistant and frost susceptible aggregates. The test method is therefore considered unsuitable for harsh climatic conditions.
- The evaluation of all test results shows that testing with a 1 % NaCl-solution gives equal or better precision data than when testing in pure water. Introducing salt into the test results in a better spread of the test results and, for most aggregates, a better correlation with known performance in real constructions.
- The precision values are in general acceptable. However, some parts of the testing procedure need to be better specified and the laboratories need to gain more experience with the test. This will significantly reduce both repeatability and the reproducibility values.
- The evaluation also shows that there is no significant difference when thawing in water or air. Both procedures can therefore be allowed for in the method.

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/2/ Höbeda, P. & Jacobson, T. 1981: Frystöväxlingsförsök på stenmaterial med svaga lösningar av halkbekämpningsmedel. VTI report no. 244. Linköping.

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/7/ Bjarnason, G., Petursson, P. & Erlingsson, S. 2000: Aggregates resistance to fragmentation, weathering and abrasion. Proceedings of the fifth international symposium on unbound aggregates in roads. Edited by Dawson, A.R. A.A. Balkema/Rotterdam/Brookfield/2000

APPENDIX I - Application for Nordtest funding

Ansøgning om Nordtest projektmidler

Ansøgninger bør være korte og koncise. Ansøgningernes almene kvalitet kan have betydning for bedømmelsen. Ansøgningerne skal helst være på elektronisk form. Efterfølgende skema skal anvendes.

0. Projektnavn: (max 45 anslag)	**Frost resistance test on aggregates with/without salt**

1. Projekttype (check en Box)

Udvikling af metode(r)	☐
Udvikling af guideline	☐
Inter laboratory comparison or round robin	☒**1**
Inspektions eller certificerings projekt	☐
Udviklingsprojekt	☐
Videnoverføring (seminar, workshop etc.)	☐
Anden type	☒**2**

Bemærk at det kan forekomme at projektet er i to grupper. Prioriter hvor det vigtigste resultat ligger.

☒2 Conformity assessment and support to implementation of a Nordtest standard on the European market.

2 Søkeren

Fornavn:	Pétur
Etternavn:	Pétursson
Tittel:	Mr
Firmanavn:	Icelandic Building Research Institute (RB)
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Mobiltelefon:	846 8762

Dato og søkerens underskrift:	14 September 2002, Pétur Pétursson

Ansøgningerne behøver ikke underskrift, når de sendes elektronisk fra ansøgerens virksomhed. Nordtests kvittering for modtagelse af ansøgning sendes til ansøgerens E-post adresse.

3 Prosjektleder

Fornavn:	Pétur
Etternavn:	Pétursson
Firmanavn:	Icelandic Building Research Institute (RB)

Det forudsættes at projektlederen kommer fra ansøgeren, se pkt 2. Ifald dette ikke er tilfældet, så skal adresse og andet skrives som **deltager 1** i pkt 4.

4. PROJEKTDELTAGERE

Følgende skemaer udfyldes således

Fornavn	Etternavn	Firmanavn	
Postnummer	Sted	Land	
Telefon	Mobiltelefon	Deltagelse er bekræftet: Ja	Nej
e-post		Webbadresse	
Rolle i projektet			

DELTAGER 1

Björn	Schouenborg	Sveriges Provnings- och Forskningsinstitut (SP)
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Det er vigtigt at vise at der er deltagere fra mindst to nordiske lande. Nordtest ser gerne at alle nordiske lande deltager i projekter. Det er også muligt for ikke nordiske partnere at deltage i projekter, for eksempel fra baltiske lande.

5. Projektomkostninger for projektet. (see detailed on next page)

Omkostninger i Euro	Omkostninger første år	Omkostninger andet år	Omkostninger tredje år	Totalt
Arbejdsomkostning	15.000 Euro			15.000 Euro
Externe tjenester				
Udstyr og materialer	17.000 Euro	3.000 Euro		20.000 Euro
Rejser og møder	8.500 Euro	8.500 Euro		17.000 Euro
Information	2.000 Euro	14.000 Euro		16.000 Euro
Sum	42.500 Euro	25.500 Euro		68.000 Euro

Nordtest bevilliger kun for et år pr gang. Eventuelle andet og tredie år bliver bevilliget på basis af en statusrapport indsendt senest 15. september i året forud. Der kan i skemaet anvendes en anden valuta. Bevilling vil altid blive i euro. Lønomkostninger er maximalt de nationale forskningsråds godkendte timesatser for teknologiske forskningsinstitutioner.

Finansiering i Euro	Finansiering første år	Finansiering andet år	Finansiering tredje år	Totalt
Nordtest	21.250 Euro	12.750 Euro		34.000 Euro
Egen indsats fra deltagere	21.250 Euro	12.750 Euro		34.000 Euro
Anden finansiering, skriv navn:	Partners outside the Nordic countries will finance their testing and meeting cost, see list of partners above.			
Sum	42.500 Euro	25.500 Euro		68.000 Euro

Når der deltager anden finansiering er det bekræftet eller blot ansøgt/påtænkt ansøgt?

Anden finansiering, navn	Externt ansøgt Ja/nej	Internt ansøgt Ja/nej	Er bevilling givet Ja/nej

A detailed total cost estimate is in the table below, but 50 % of the cost is applied for to Nordtest in a two-year period.

All figures in Euros

	IS*	SE*	NO	FI	DK	DE*	DE	F	BE	NL	Sum
Co-ordination 2003	3000										3000
Collection of samples 2003	1000	1000	1000	1000	1000						5000
Shipment of samples to SP 2003	500	500	500	500	500	500	500	500	500	500	5000
Sample preparation 2003		1000									1000
Shipment of samples to test labs 2003		3000									3000
Testing of samples 2003	6000	6000	1000	1000	1000						1500 0
Meeting in January/February 2003	2500	1500	1500	1500	1500						8500
Collection of data 2003	2000										2000
Data processing and calculations 2004	4000	3000									7000
Co-ordination/status report 2004	3000										3000
Meeting in January/February 2004	2500	1500	1500	1500	1500						8500
Final report 2004	3000	2000									5000
Publishing 2004	2000										2000
Sum	**29500**	**19500**	**5500**	**5500**	**5500**	**500**	**500**	**500**	**500**	**500**	**68000**

*** Key laboratories**

***Key laboratory 1 (RB):** Pétur Pétursson, geology/road research. Experience/competence in this area: Worked at the Bundesanstalt für Strassenwesen in Germany for one month in 1992 using the German equipment for frost testing of aggregates. Co-ordinator of the Nordtest project no. 1214-95, which resulted in the Nordtest standard NT BUILD 485. Member of TC154/SC6 (Aggregates/Test methods), TG 9 (Thermal and weathering properties of aggregates) and later TG 12 (Chemical, thermal and weathering properties of aggregates). See also 6 below.

***Key laboratory 2 (SP):** Björn Schouenborg, PhD geology. Experience/competence in this area: Participated in Nordtest project no. 1214-95. Member of CEN/TC 165/SC 6 (Aggregates/ test methods). Organized the inter-comparison test on the Studded tyre test (Nordic Abrasion value) and the precision data was accepted and included in EN 1097-9 (SP REPORT 1994:21: "*Studded Tyre Test – Precision trials*, Björn Schouenborg & Leif Viman"). Produced a report for Nordtest with guidance of organizing inter-comparison trials: "*Methodology of Inter-comparison Tests and Statistical Analysis of Test Results*" (Tang Lupong & Björn Schouenborg. SP REPORT 2000:35).

***Key laboratory 3 (RWTH):** Cyrus Gharabaghy, Dipl.-Ing., Institut für Strassenwesen Aachen, Erdbau and Strassenbautechnik. Experience/competence in this area:

6 Problembeskrivning med bakgrund inkl. "state of the art" (Hvorfor?)

(*en A4-side*)

Hjælp: …

Hvad er situationen globalt for dette (State of the Art) område? In 1985 a new freeze/thaw test method was developed in Iceland. A correlation between test results and actual performance of aggregates was established. In 1993 the method was introduced to CEN/TC 154/TG 9 (Aggregates-thermal and weathering properties) with a request for a frost resistance test in salt water as an option to the pure water method (EN 1367-1), which was then under consideration by TC 154/SC 6 (Aggregates-Test methods). In 1995-6 a Nordtest project resulted in another method with a better accuracy, but based on the experience obtained earlier and with reference to EN 1367-1. This method has been in use in Iceland since 1996 and has replaced the older method. It also became a formal Nordtest standard in 1998 (NT BUILD 485).

Har projektet en placering i en større sammenhæng? On the request of Iceland, Task Group TG 9 drafted a test standard using salt water and it was circulated to national bodies for comments. The draft did, however, not gain enough support at that time. The main reason was that it was felt that not enough data had been collected using cross-European aggregates. What was achieved is that EN 1367-1 was publisthed with an informative Annex stating that an undefined test method with salt could be considered in certain cases. Also, a clause in the CEN/TC 154 product standards refers to that Annex. Iceland has objected to this conclusion with the support of the other Scandinavian delegates in TC 154/SC 6 ("Test methods").

CEN/TC 154 has recently established a new Task Group, TG 12 "Chemical, thermal and weathering properties of aggregates". On the first meeting of the Task Group in April 2002 it was decided that the Icelandic delegate (the applicant of this proposal) would draft a new proposal of a test method using a salt solution and based on the Nordtest method NT BUILD 485. This has already been done. It was also decided on the meeting that "the organisation of an interlaboratory trial has to be foreseen as soon as possible". The aim of this application is therefore to establish an interlaboratory trial and to support the Scandinavian part of that trial, as well as covering the cost of shipment of samples for other European partners. It is anticipated that they will bear the cost of testing and meetings themselves.

Hvori ligger problemet? The main difference between the two methods is the use of a salt solution in the Nordtest method, but pure water in the EN method. Nordic aggregates have been tested with the pure water method on the one hand and the salt-water method on the other hand (Nordtest project 1214-95). It is obvious that the difference is very significant. It was demonstrated that the pure water method fails to distinguish between frost resistant and frost susceptible aggregates. Experience in Iceland has indicated strongly, that it is very important to obtain freeze/thaw values that classify materials into different frost resistance groups or categories. It is intended to demonstrate that the use of the salt-water method will help to identify frost susceptible aggregates without discriminating frost resistant aggregates.

Another problem is that the thawing phase is controlled by air circulation in the Nordtest method but with water circulation in the EN method. The two methods serve the same purpose, which is to control the thawing phase of the samples. Therefore, it is considered irrelevant which one of the two methods is used to control the sample temperature. By focusing on the sample temperature instead of how it is achieved, unnecessary restrictions on very expensive equipment can be avoided. That would enable laboratories in the Nordic countries as well as elsewhere, to easily adopt the method and run either the Nordic test or the EN 1367-1 test when appropriate. It is intended to demonstrate that thawing out by air and water circulation is equally applicable.

Hvilke brancher/virksomheder/myndigheder/interesseorganisationer etc. påvirkes af problemet og får nytte af resultaterne? Hvilke nordiske interessenter ønske dette projekt gennemført? It is important for aggregate users and producers to have access to a test method, which distinguishes between frost resistant and frost susceptible aggregates. This is especially important in climatic regions where freeze/thaw cycling occurs and in saline environment (use of de-icing salt on road surfacing, bridges over fjords and channels etc.). It is also important for test laboratories to be able to use the same equipment when testing frost resistance of aggregates, either in pure or salt water.

7 Målsetning (hva?)

(en halv A4-side)

Hjælp: Såvel målsætning for hele projektet, som for eventuelle opdelinger af projektet skal der formuleres målbare resultater således at de senere resultater kan sammenlignes med disse mål. Forslag til nogle centrale målbare succeskriterier.

The project is aimed at obtaining frost resistance test results on a representative selection of European aggregates, with both the salt water (NT BUILD 485) and pure water (EN 1367-1) test methods. In that way it will be possible for the relevant Task Group (TG 12) of CEN/TC 154 to assess a test method using a salt solution in comparison with a pure water method. This will hopefully lead to a European standard or a Normative annex to an existing standard, where frost resistance is tested in a salt solution for aggregates with special end-use (de-iced road surfaces, bridges etc.).

The proposed project can be didided into three main objectives:

a) To test aggregates from different sources throughout Europe at different laboratories in Europe, with both pure water and salt water freeze/thaw test methods, to widen the applicability of the Nordtest freeze/thaw test method in 1 % NaCl. A part of this objective is to demonstrate that different freeze/thaw cabinets can be used to obtain comparable results, as long as the desired sample temperature is adhered to.
b) To gain reliable repeatability and reproducibility values for both test methods (NT BUILD 485 and EN 1367-1).
c) To produce and suggest to the relevant task group of CEN/TC 154, a revised method for testing aggregate freeze/thaw resistance with 1 % NaCl to be included in the European Standards.

8 På hvilken måte styrker prosjektet det nordiske næringslivet

(en halv A4-side)

Hjælp: Her beskrives projektets og det forventede projektresultats betydning og værdi for det nordiske næringsliv generelt, og hvordan det relaterer sig til Nordtests mål.

It has been demonstrated that testing the freeze/thaw resistance of construction aggregates in pure water, according to the European Standard EN 1367-1, does not distinguish between frost susceptible and frost resistant aggregates in some cases. On the other hand, the Nordtest method NT BUILD 485, which was published in 1998, appears to do so. It is therefore very important for Scandinavian producers and users of aggregates to have the Nordtest method included in the European Standards to test aggregates, which will be used in harsh climatic conditions where frequent freeze/thaw cycling occurs and in saline environment. These conditions exist in many places in Scandinavia. In such areas it is indeed important to have knowledge of the expected durability of aggregates used for outdoor constructions, whether they would be road surfaces or concrete structures, such as bridges. The NT BUILD 485 test method is thought to give such information about aggregates while EN 1367-1 fails to do so in some cases.

9 Prosjektbeskrivelse og arbeidsplan (Hvordan?)

(to A4-sider)

Hjælp: Her beskrives indholdet I projektet og dets eventuelle delprojekter.

It is intended to have laboratories in ten countries throughout Europe to participate in this project. Each country will choose two aggregates to be tested with the NT BUILD 485 and EN 1367-1 freeze/thaw test methods. It is intended that each country will choose the two aggregates in collaboration with the co-ordinator, bearing in mind that one of the materials has good service records and the other one rather poor service records. The three key laboratories, RB, SP and RWTH in Germany, will test all aggregates with both test methods, which is 20 samples x 2 test methods x 3 test specimens = 120 measurements. It is important that one of the three key labratories is from Germany, as they would be testing in different type of freeze/thaw cabinets then used in Scandinavia. In that way an evaluation of different equipment can be made. The other laboratories will test 3 samples x 2 test methods x 3 test specimens = 18 measurements. Two of those test samples would be the ones chosen from their own country and the third sample would be a reference sample, tested at all laboratories. It is possible that in the case of testing 3 samples, one run in the freeze/thaw cabinet (18 test cans) would be possible in most cases. In the case of testing 20 samples with two methods, multiple test runs would be required.

Besides the actual testing, most of the work required is to collect all the samples from all the countries and prepare them for all the laboratories, packaging and shipping. SP in Sweden will be in charge of that work for three reasons; a) to keep the shipping cost at minimum, b) to divide the cost and labour between participants and c) to have access to the rotary dividing equipment at SP for splitting the samples. The cost of this work is included in the application (shipping of samples to Sweden, washing, drying, splitting, preparing for transport and shipping back to relevant laboratories). It is intended that each participating laboratory will be in charge of collecting the chosen samples.

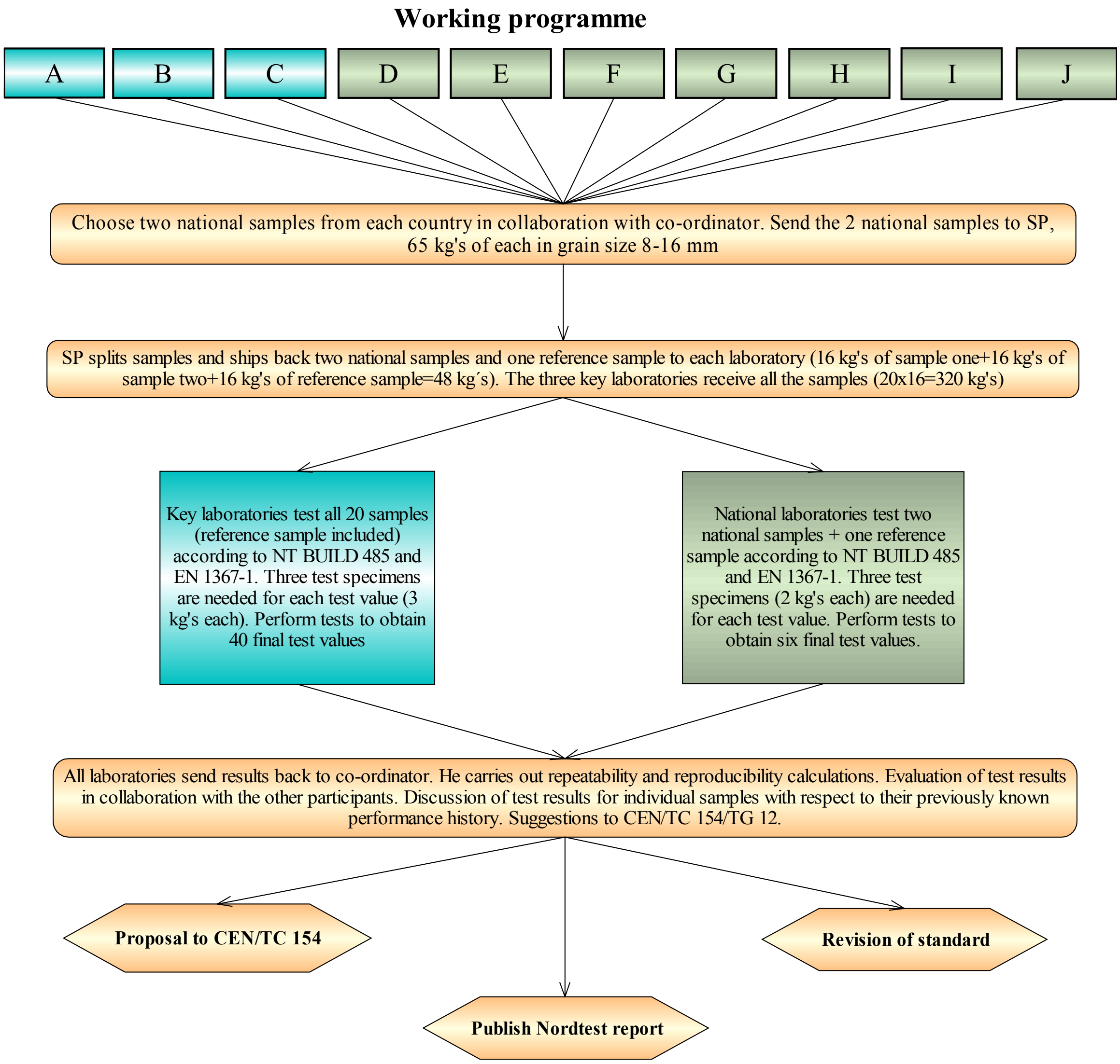
The co-ordinator will collect all the data from all the laboratories. RB and SP will be in charge of data processing, calculations and preparation of a final report in collaboration with the other participants. The project schedule is shown on next page.

Two meetings are scheduled for the proposed project. It is intended to hold both meetings at the RWTH key laboratory. That will enable our European partners to attend the meetings without too much travel expences. On the first meeting, final decisions on aggregates to be chosen from each country for the project will be made. The procedure of the project will be discussed in detail. On the second meeting, test results will be discussed and decisions on final conclusions and presentations will be made.

9 Prosjektbeskrivelse og arbeidsplan (Fortsettelse)

(Forsettelse)

Below is a schematic diagram showing the proposed working programme for the project.



10 Tidsplan

(en halv A4-side)

January 2003: The project will start immediately in January 2003. A meeting will be established in the beginning where final decisions about aggregate samples will be taken. The procedure of the whole project will be discussed in detail.
February to April 2003: Collection of samples, preparation of samples and shipping of samples to participating laboratories. Samples ready for testing.
May to August 2003: Testing of samples at the national laboratories.
September 2003: Status report to Nordtest
January to February 2004: Processing of data. A meeting will be established to discuss test results. Decide on final conclusions and presentations.
March to April 2004: Revision of NT BUILD 485 suggested. Suggest to CEN/TC 154 and publishing of final Nordtest report.

11 Spredning av informasjon og resultater fra prosjektet

(en halv A4-sida)

A status report will be issued to Nordtest at the end of year 2003

A final Notdtest report will be issued in 2004.

Revision of the Nordtest standard NT BUILD 485 will be proposed in 2004.

Proposal concerning inclusion of a CEN/TC 154 standard or a normative annex to an existing standard, which will be based on the Nordtest standard for testing aggregates frost resistance in salt water in 2004.

APPENDIX II - Combined draft EN 1367-1 and NT BUILD 485, May 2003

Combined EN 1367-1 and NT BUILD 485

**Tests for thermal and weathering properties of aggregates:
Determination of resistance to freezing and thawing with/without salt**

To be used in the FRAS intercomparison project

May 2003

Contents

Foreword

This Standard was prepared by TG9 and TG12 of CEN/TC 154, Aggregates at the request of SC6.

The revision of the standard EN 1367-1 has led to the following fundamental alterations:

1. The samples are tested in either pure de-ionised water or 1% solution of NaCl in de-ionised water. The same requirements in the product standards do not apply.
2. The thawing out sequence is controlled either by air circulation or water circulation in the cabinet to obtain the correct temperature of the reference sample.

The frost resistance of the aggregate is determined by subjecting it to the cyclic action of freezing and thawing. The freeze-thaw resistance of aggregate, as measured by the proportion of undersize passing the ½ size sieve as sieved from the test portion, is considered separately for each portion and then expressed as a mean % by mass.

1 Scope

This European Standard specifies a test method to assess the frost resistance of aggregates when it is subjected to the cyclic action of freezing and thawing. In areas where frequent freeze-thaw cycling occurs and seawater sprays or de-icers are abundant it is more appropriate to use a 1 % solution of NaCl in de-ionised water instead of pure de-ionised water.

The test is applicable to aggregates having a particle size between 4 mm and 63 mm.

2 Normative references

This European Standard incorporates by dated or by undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

EN 932-1 Tests for general properties of aggregates
Part 1: Methods for sampling

EN 932-2 Tests for general properties of aggregates
Part 2: Methods for reducing laboratory samples

EN 932-5 Tests for general properties of aggregates – Part 5 : Common equipment and calibration

EN 933-2 Tests for geometrical properties of aggregates Part 2 : Determination of particle size distribution – Test sieves, nominal size of apertures

EN 1097-2 Tests for mechanical and physical properties of aggregates
Part 2 : Methods for the determination of the resistance to fragmentation

Definitions

For the purposes of this standard the following definitions apply.

3.1 Test specimen
Sample used in a single determination when a test method requires more than one determination of a property.

3.2 Laboratory sample
Reduced sample derived from a bulk sample for laboratory testing.

3.3 Constant mass
Successive weighings after drying at least 1 h apart not differing by more than 0,1%

Note: In many cases constant mass can be achieved after a test portion has been dried for a pre-determined period in a specified oven at (110 ± 5) ºC. Test laboratories can determine the time required to achieve constant mass for specific types and sizes of samples dependent upon the drying capacity of the oven used.

4 Principle

Test portions of single sized aggregates, having been soaked in pure water or 1% NaCl solution at atmospheric pressure for 24 h, are subjected to 10 freeze-thaw cycles. This involves cooling to -17,5 ºC under water or in the salt solution and then thawing to 20 ºC.

5 Apparatus

5.1 All apparatus, unless otherwise stated, shall conform to the general requirements of EN 932-5.

5.2 ventilated drying oven, with forced circulation of adequate capacity. The oven shall be capable of being controlled at (110 ± 5) ºC.

5.3 balance, with an accuracy of ± 0,1 g, of adequate capacity.

5.4 low temperature cabinet, (upright or chest) with air circulation. The cabinet shall be automatically controlled to adhere to the temperature curve shown in Figure 1. The sample temperature in the thawing out phase can be controlled either by air circulation or immersion of sample cans in a 20°C water bath. A manual method of control may be used, provided the correct cooling curve, as shown in Figure 1, is adhered to. In the case of a dispute, the automatic control shall be used.

5.5 cans, made from seamless drawn or welded corrosion-resistant sheet metal, with a thickness of about 0,6 mm, having a nominal capacity of 2000 mL, an internal diameter of 120 mm to 140 mm, and an internal height of 170 mm to 220 mm are suitable. Cans shall be covered by suitable lids.
For lightweight aggregates (LWA), cans shall be suitably ballasted.

5.6 test sieves, conforming to EN 932-1.

5.7 Pure de-ionised water or 1% NaCl solution, made by mixing 20,0 g of NaCl of analytical grade in de-ionised water and making up to a volume of 2 litres. If this is insufficient, prepare additional solution at the same concentration.

6 Sampling

Sampling shall be carried out in accordance with EN 932-1.

7 Test specimens

7.1 General

Three individual test specimens shall be used. The test specimens shall be obtained in accordance with EN 932-2 by sample reduction from production single sized aggregates from which oversized and undersized aggregates have been removed.

> NOTE: If it is intended to carry out a strength test after the freeze-thaw cyclic loading, this test should be performed on an appropriate grading sieved out from the laboratory sample, in accordance with EN 1097-6.
>
> In order to do this, a laboratory sample should be taken of twice the mass required for the strength test, plus an allowance for waste. This laboratory sample should then be split into two approximately equal parts. The first part should be used for the strength and density tests, without being subjected to the freeze thaw cycling, and the second part should be subjected to the freeze-thaw cyclic test.

7.2 Size of test specimens

The preferred size fraction shall be within the range 8 mm to 16 mm, but if required, any of the sizes listed in table 1 can be used. The quantities for each of the three individual test specimens are given in table 1, and deviations of ±5 % are permissible.

Table 1: Test specimens required for the freeze-thaw cyclic test

Aggregate size mm	Mass or volume of aggregate required	
	Normal aggregate g	Lightweight aggregate (bulk volume) ml
4-8	1000	500
8-16	2000	1000
16-32	4000[1)]	1500
32-63	6000[1)]	-

[1)] Additional cans will be necessary

7.3 Preparation of test specimens

The test specimens shall be washed and adherent particles removed. They shall be dried to constant mass at (110 ± 5) ºC, allowed to cool to ambient temperature and weighed immediately (M_1).
For lightweight aggregates, dry to constant mass.

Weighing shall be carried out to the following accuracies:-

Aggregates up to 16 mm size, to ± 0,2 g;
Aggregates above 16 mm size, to ± 0,5 g.

8 Procedure

8.1 Soaking

The test specimens prepared in accordance with **7.3** shall be stored at atmospheric pressure for (24 ± 1) h in the cans specified in **5.5** at (20 ± 3) ºC, in water or 1% NaCl solution, the water or solution covering the test portions by at least 10 mm for the full 24 h period of soaking.

8.2 Exposure to freezing under water or NaCl solution

Check that the water or salt solution level in each can is still at least 10 mm above the top of the test portion and place the lids on the cans. Place the covered cans containing the test portions in the cabinet, ensuring that the heat is extracted from them as uniformly as possible from all sides. The distance between adjacent cans and the sidewalls of the cabinet shall be not less than 50 mm and the cans shall not be touching.
The samples in the cabinet shall then be subjected to a series of 10 freeze-thaw cycles as follows:

a) the temperature at the centre of a can, filled with aggregate and water or NaCl solution as specified in **8.2** and situated in the centre of the cooled area, shall be the reference measuring point of temperature.

b) the cabinet shall be controlled so that the temperature follows a cooling curve inside the limits as shown in Figure 1;

c) the temperature shall fall from (20 ±3) ºC to (0 to –1)° C in (150 ± 30) minutes, and shall then remain at (0 to –1)° C for (210 ± 30) min;

d) the temperature shall then be reduced from (0 to –1)° C to (- 17,5 ± 2,5) ºC in (180 ± 30) min;

e) the temperature shall then be held at (- 17,5 ± 2,5) for a minimum of 240 min;

f) at no stage shall the sample temperature fall below – 22 ºC;

g) after the completion of each freezing cycle the cans shall be thawed to (20 ±3) ºC;

h) after the completion of each thawing phase the cans may be held at (20 ±3) ºC for a maximum of 10 h. Each freeze-thaw cycle shall be completed within 24 h.

i) if it is necessary to interrupt the test during the freezing cycle or when under manual control the cans shall remain in the cabinet at (-17,5 ±2,5) ºC. A total interruption of up to 72 h is permitted.

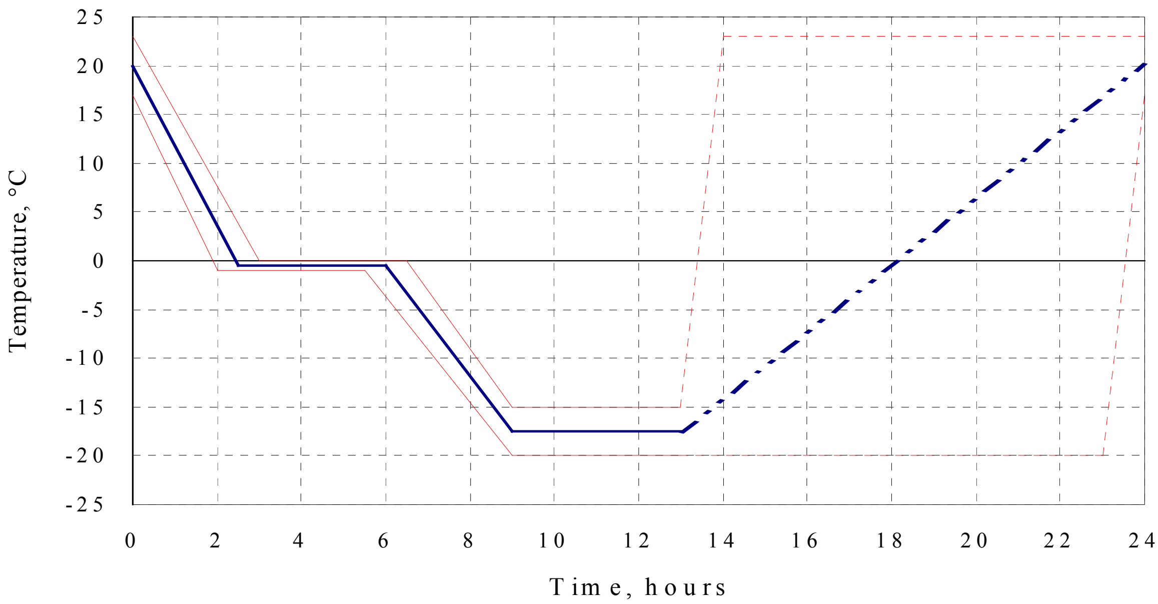


Figure 1: Temperature curve with tolerance limits in the centre of the filled can (reference measuring point) located in the middle of the cabinet

8.2.2 On completion of the tenth cycle, empty each can into a suitable container, and wash thoroughly by hand with water. Dry each test specimen to constant mass and sieve on a test sieve having an aperture size that is half the lower size sieve used to prepare the test portion (e.g. of 4 mm aperture size when testing 8-16 mm sample). Cool the sample to ambient temperature and weigh immediately (M_2).

9 Calculation and expression of results

9.1 Calculate the undersize of the three test specimens, weigh and express the mass obtained as a percentage of the mass of the individual and the combined test specimens.

9.2 Calculate the result of the freeze-thaw test (F) in accordance with the following equation:

$$F = [(M_1 - M_2) / M_1] X100$$

where

M_1 is the initial dry mass of the three test specimens before cycling, in grams;

M_2 is the final dry mass of the three test specimens after cycling, that is retained on the specified sieve, in grams;

F is the percentage loss in mass of the three test specimens after freeze-thaw cycling.

NOTE: A statement on the precision of this test is given below.

10 Test Report

10.1 The test report shall refer to this Standard and contain the following information:

10.1.1 Sampling method if known, and marking, type and origin of the laboratory samples.

10.1.2 Shape, size, grading and number of laboratory samples.

10.1.3 Visual observations of the aggregate retained on the specified sieve. Any unusual disintegration of the aggregate retained on the sieve shall be reported.

10.1.4 Result of the freeze-thaw test, *F* expressed to the nearest 0,1 % by mass.

10.1.5 Date of report and name of test laboratory.

Precision (informative)

The Coefficient of variation for a homogeneous material of a size fraction 8 mm to 16 mm (passing a 4 mm test sieve) is as follows:

Coefficient of variation for:

a) Repeatability *r*: 18 %

b) Reproducibility *R*: 30 %

The results were interpreted in accordance with ISO 5725-2, chapter 7.1-7.4. Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.

APPENDIX III - Minutes of the meetings of the project group

Frost resistance test on aggregates with/without salt (FRAS)

Minutes of the first project meeting at RWTH, Aachen, 31 January 2003

Participants

Pétur Pétursson	IBRI, Icelandic Building Research Institute, Iceland
Cyrus Gharabaghy	Institut für Strassenwesen (Isac) RWTH Aachen, Germany
Peter Arnold	Institut für Strassenwesen (Isac) RWTH Aachen, Germany
Pirjo Kuula-Väisänen	Tampere University of Technology, Finland
Erik Bruun-Frantsen	Danish Technological Institute, Denmark
Viggo Jensen	NBTL Norsk betong og tilslagslaboratorium AS, Norway
Thomas Merkel	Forschungsgemeinschaft Eisenhüttenschlacken (FehS), Germany
Sophie Seytre-DupÊcher	LRPC de Clermont-Ferrand - CETE de Lyon, France
David Woodward	Highway Eng. Research University of Ulster, Northern Ireland
Martijn van Bree	KOAC-WMD BV, The Netherlands

Apologies

Björn Schouenborg	SP
Freddy Henin	HOLCIM

1. Welcome

Pétur Pétursson welcomed all participants to the meeting.

Director of the Institute fur Strassenwesen Dr Steinauer welcomed the participants to Aachen on behalf of the RWTH.

2. Roll call of the delegates

During the roll call all participants introduced themselves and the organisations they are presenting in the project.

3. Adoption of draft agenda

The proposed agenda was agreed.

4. Previous work

Pétur Péturson introduced the literature review done by Peet Höbeda. Björn Schouenbourg has sent a short conclusion paper to all participants by e-mail.

Pétur presented the work done in an earlier Nordtest project and in Iceland, ***Pétur will send his Power Point presentation by e-mail to all participants of the project.***

Erik presented the work done in Denmark. The copies of the presentation were given to the participants of the meeting. The materials tested proved to be fulfilling the requirements of freeze-thaw and magnesium sulphate tests although some of the tested materials were such which would be never used in construction according to the Danish experience.

The discussion over the previous work pointed out following items:

- Freeze-thaw test with water has not proved to be effective to separate the good and bad aggregates according to experiences in some countries
- The RWTH archives contain many old results and for example PhD thesis over the test method, an evaluation must be done over the results.
- In some countries (e.g. Norway, Finland) there is a general opinion that all the aggregates are frost resistant, the problem occurring is mainly subjected to concrete.
- The experience from Iceland with Nordtest method (with salt) is good.
- The purpose of this project is to create an option to freeze-thaw test to be used in severe freeze-thaw conditions.

5. Project description

The project description and the proposed normative annex was evaluated and the following conclusion were made:

The project has at the moment 10 laboratories participating from 9 different countries, a possibility of one laboratory more participating (UK) was informed. Three key laboratories are IBRI Iceland, SP Sweden and RWTH from Germany, these three laboratories will test all the aggregates. The rest of the laboratories will test the two aggregates from their own country and one reference sample.

The amount of reference samples was discussed because there have been proposals for two or even three reference samples due to statistical requirements. ***After the discussion it was concluded that one reference sample is enough*** because the amount of work would be too much with more reference samples. It was also pointed out that the main purpose of this project is to find out the differences between the test methods with and without salt. The statistical information is important but not the main point.

The thawing curve in cabinets using air circulation for thawing must be adjusted to the same thawing curve as in those cabinets using water. ***RWTH will send the thawing curve data with water to all participants. Pétur will send the information about the air circulation cabinet to all participants.***

All laboratories must stick to the temperature curve described in the standard (1367-1). All laboratories must send a temperature printout with the test results. A photograph of the cabinet used would be informative.

6. Equipments and materials in each laboratory

The equipments available in different countries

Iceland	Air thawing cabinet
Northern Ireland	No details yet available.
Norway	The equipment is not ready yet.
Denmark	Both air and water thawing available
Finland	Both air and water thawing available, the experience in Finland is based on the water thawing cabinet
Germany (RWTH)	Water thawing cabinet
The Netherlands	Both water and air thawing available
Germany (FEhs)	Both water and air thawing available
France	Probably air circulation available.
Belgium	No detail available
Sweden	No detail available

The description of the sample cans is in the standard. In Germany Aluminium cans are used. Information about the cans available in the market should be send to all participants.

The salt used in the test must be the same; ***Pétur will send the salt needed to every laboratory.***

7. Choice of aggregates

The aggregates for the test should be selected from those normally used in construction in every country. The size fraction is 8/16 mm, no preparation of the sample is needed, the under and over sizes are taken care of in SP, Sweden when the aggregates are divided to test samples.

The selection of aggregates

Iceland	Two basalts (one good, one poor)
Denmark	1.Land based material, porous flint (poor) 2. Marine based material with lower flint amount (good)
UK	1.Greywacke from Northern Ireland (good/poor) 2. Sandstone from England (good/poor)
Norway	1. Natural gravel (good) 2. Mica rich gneiss (poor)
Finland	1. Natural gravel (good) 2. Granite with loose structure (poor)
Germany and the Netherlands	Will send together four samples, the decision will be done in February
France	1. Chalky oolitic limestone (poor) 2. Basalt (good)
Sweden	1. Limestone (poor) 2. Granite (good)
Belgium	Information not available

Reference sample: It should be logistic to have the reference sample from Sweden; ***the limestone is proposed to be the reference sample.***

The amount of samples to be sent to SP, Sweden is 65 kg of each aggregate selected. The amount of reference sample needed will be 200 kg.

8. Milestones – target dates

The samples should be sent to SP, Sweden until 15 March 2003. The test results should be send to Pétur until the end of August 2003. The next meeting will be in January 2004.

9. Date and venue of the next meeting

The date and venue of the next meeting will be decided later.

10. Closure

Pétur Pétursson closed the meeting with special thanks to Cyrus Gharabaghy and Peter Arnold for their hospitality during the meeting.

Frost resistance test on aggregates with/without salt (FRAS)

Minutes of the second project meeting at FeEhS, Duisburg, 1 March 2004

Participants

Pétur Pétursson	IBRI, Icelandic Building Research Institute, Iceland
Björn Schouenborg	SP, Swedish National Testing and Research Inst., Sweden
Peter Arnold	Institut für Strassenwesen (Isac) RWTH Aachen, Germany
Pirjo Kuula-Väisänen	Tampere University of Technology, Finland
Erik Bruun-Frantsen	DTI, Danish Technological Institute, Denmark
Viggo Jensen	NBTL Norsk betong og tilslagslaboratorium AS, Norway
Thomas Merkel	Forschungsgemeinschaft Eisenhüttenschlacken (FEhS), Germany
Michael Rohleder	BASt, Germany
Dr Koessl	Laboratorium fur Baustoffe AG, Germany

Apologies

Sophie Seytre-DupÊcher	LRPC de Clermont-Ferrand – CETE de Lyon, France
David Woodward	Highway Eng. Research University of Ulster, Northern Ireland
Martijn van Bree	KOAC-WMD BV, The Netherlands
Cyrus Gharabaghy	Institut für Strassenwesen (Isac) RWTH Aachen, Germany
Erhard Westiner	MPA BAU, Germany
Freddy Henin	HOLCIM - France Benelux, Belgium
Patricia Wolfsdorff	Materialprüfungsamt MPA NRW, Germany

4. Welcome

Pétur Pétursson welcomed all participants to the meeting.

Director of the Institute Dr Heribert Motz welcomed the participants to Duisburg on behalf of the FEhS.

5. Roll call of the delegates

During the roll call all participants introduced themselves and the organisations they are presenting in the project.

6. Adoption of draft agenda

The proposed agenda was agreed.

7. Test results and statistical calculations

Pétur gave a general presentation of the results. There was some scattering in the results between different laboratories and even within some laboratories. A clear tendency was shown about the effects of the salt on test results. The use of salt affects some aggregates dramatically. All the test result means with fresh water are < 1,9 %.

Reference sample 1 (altered basalt) showed clear results about the effects of salt. When the test is done without the salt the test results (mean 1,2 %) fall in the first category according to the Icelandic classification, when salt is used the mean is 13 % and the class is C ("poor but not hopeless"). This sample is a good example of why the test with salt is necessary.

Also the gravel sample 2ZIP is a good example, without salt the mean is 0,18 % and with salt 11,2 %.

The results showed clearly that the salt does not affect the good aggregates at all.

Björn Schouenborg presented the statistical calculations. In evaluation of repeatability (r) and reproducibility (R) it is better to use the linear correlation than exponential correlation because the amount of data is relatively small. There was no good correlation between the results with salt and without salt. When the results were compared to the earlier results without salt (round-robin test in 1995) it was noticed that the values of r and R in our FRAS-project are better than earlier. The effects of salt on the earlier test results had the same tendency as in this project. Björn stated also that mixing all the different rock types in statistical calculations is affecting the quality of the results.

Björn and Pétur will send their presentations after the meeting to all participants. All participants should check the results and if any mistakes are noticed in the test results, the corrections must be sent immediately to Björn and Pétur.

8. Discussions

The following items were discussed:

What is the difference between thawing with water compared to thawing with air? The thawing with water seems to be quicker than with air.

How is the experience of different laboratories affecting the test results? Some laboratories were using the test method for the first time and others are very experienced in this test.

Why 1 % salt totally destroys some aggregates? There is no direct answer, the porosity is not the answer , and the pore size distribution can explain some of the phenomena. When there is room for the water to expand during freezing the damage is not happening. Also the lower freezing point can explain something but not everything.

The Finnish and Norwegian poor aggregates are not poor according to freeze-thaw test as their poorness is based on other tests e.g. Nordic ball mill.

During the test there might be differences in the temperature in different sample cans. The effect of these differences on test results is not known.

Correlation with the field experiences is very important when evaluating the test results.

Also, the magnesium sulphate soundness test was discussed: In Denmark 6 materials were tested and the highest value detected was 13,5 while the limit value for aggregates in product standards is 18. In Iceland 20 aggregates were tested and all of them passed the magnesium sulphate test although some of the materials were not good.

9. Conclusions

All participants must send the following information to Pétur as soon as possible:

- Any mistakes in the test results.
- The temperatures in freeze-thaw cycles.
- Information about the aggregates: petrographic description, results of previous freeze-thaw tests, water absorption, specific gravity (saturated surface dried ρ_{ssd}) physical and mechanical properties and field experience if available.
- If the water absorption value is not available, each partner should perform the test with their national aggregates if they have any of it left.
- Details of the test cabinet: air or water thawing, how the water is led to the cabinet etc.
- Experience of the laboratory in the freeze-thaw testing.

The results showed clearly that the test method with fresh water is less informative and selective than the method with salt.

The results showed clearly that the salt does not affect the good aggregates at all.

The participants agreed that the test with of salt as an alternative method should be added in the standard 1367-1:1999. The standard is soon coming to five-year review. **It was also agreed that the use of salt should be added directly to the standard text (no normative annex).** The main changes are the revision of the standard scope and adding some text in the test procedure. The national mirror groups of TC154 finally accept of the standard revision.

10. Co-operation in the future

Some further co-operation possibilities were discussed, for example, a larger project concerning the collaboration with field and laboratory tests in freeze-thaw phenomena.

After the meeting Dr Thomas Merkel introduced the participants through FEhS laboratories.

Pirjo Kuula-Väisänen
Research Scientist
TUT, Laboratory of Engineering Geology

APPENDIX IV - Precision data

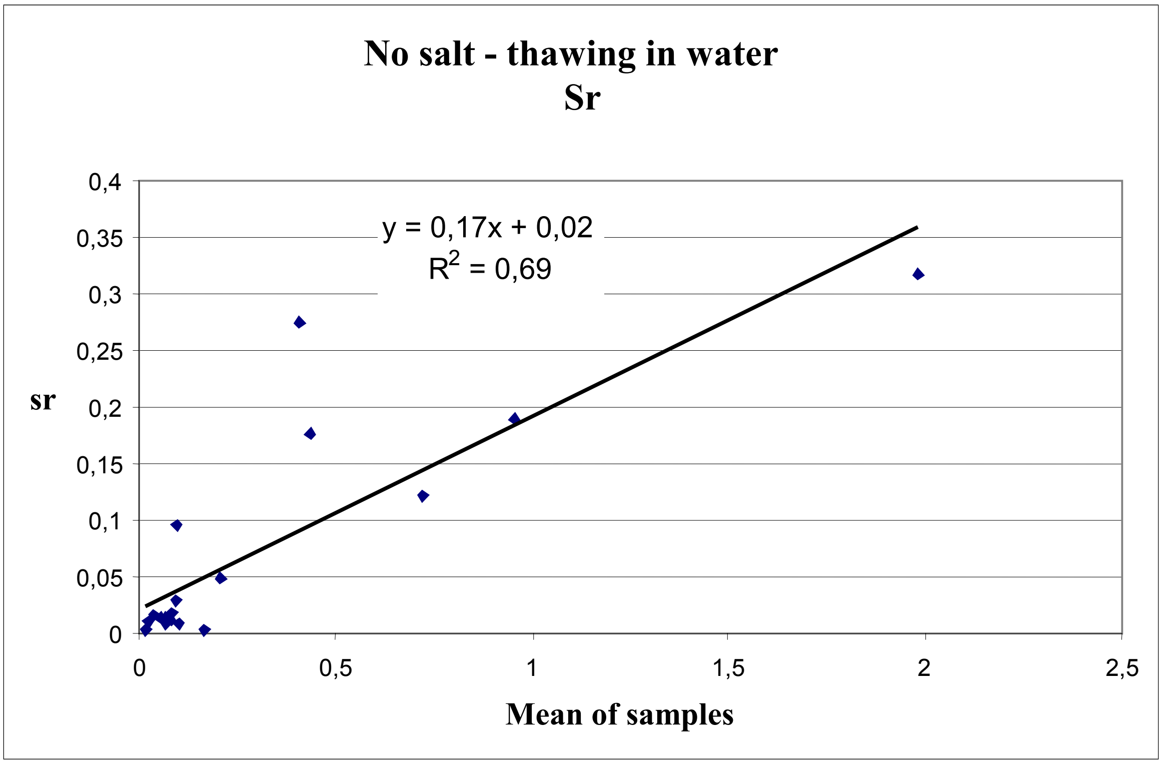


Figure IV 1

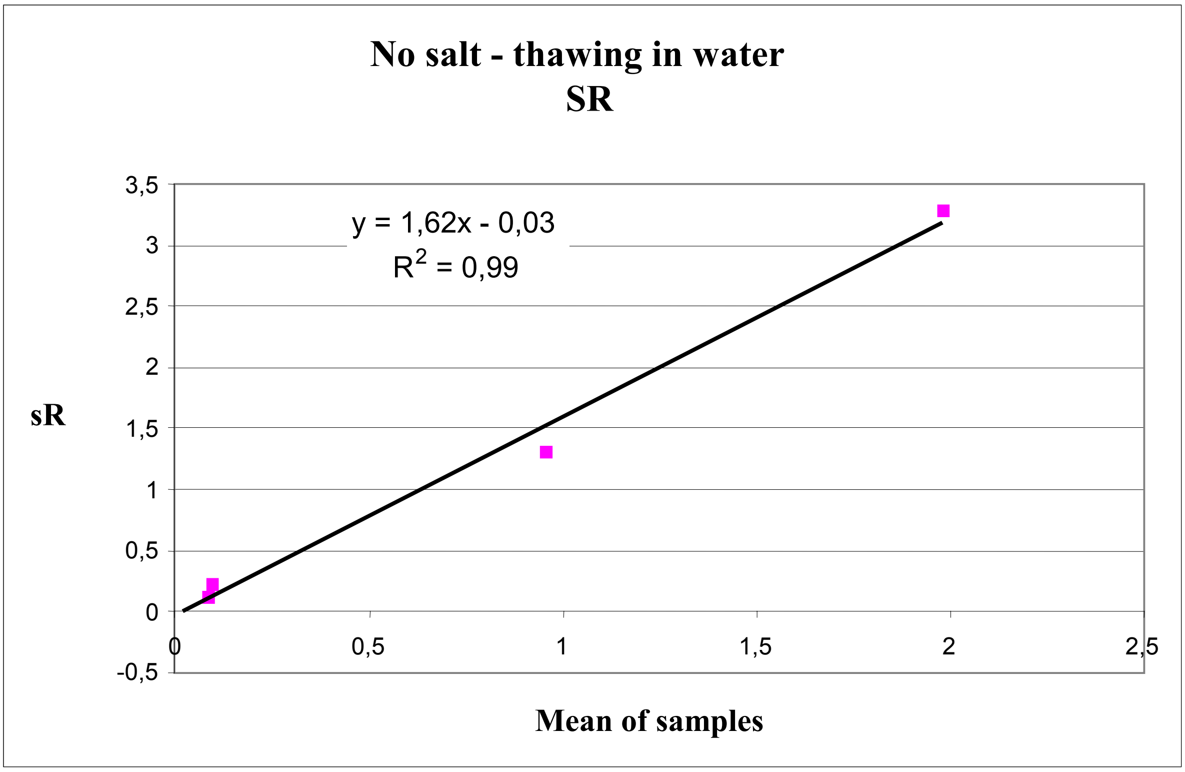


Figure IV 2

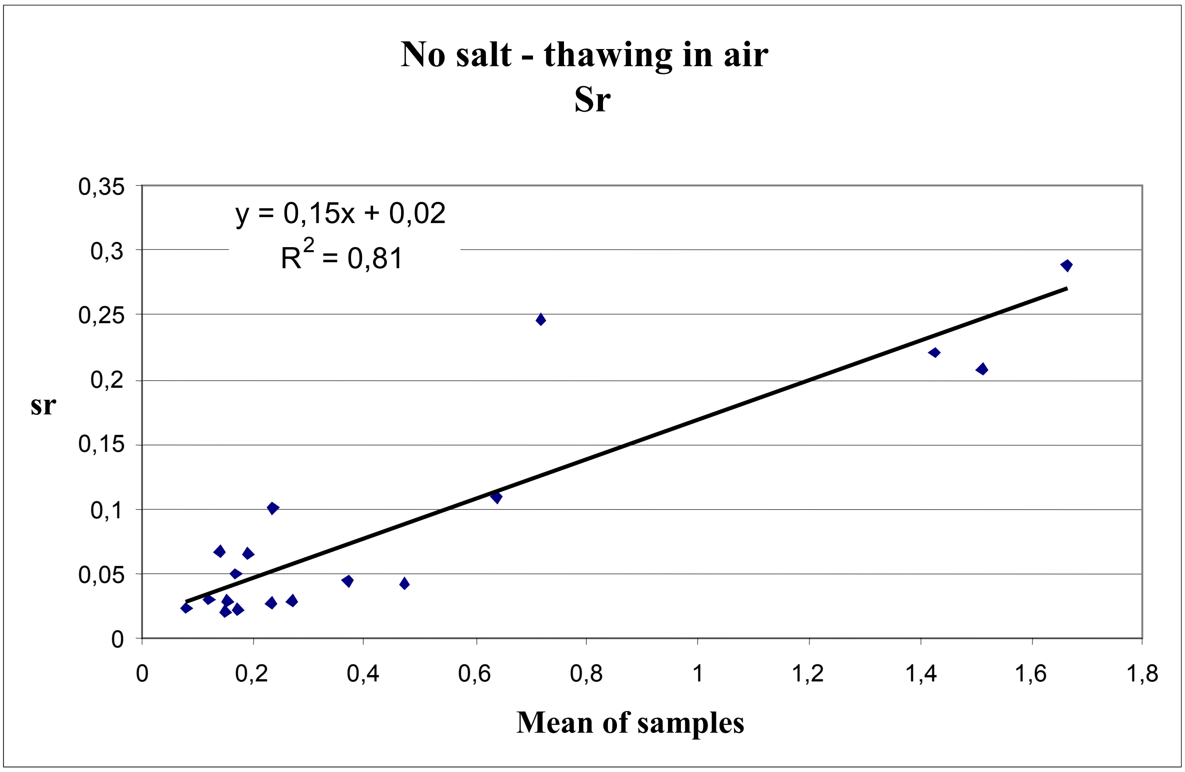


Figure IV 3

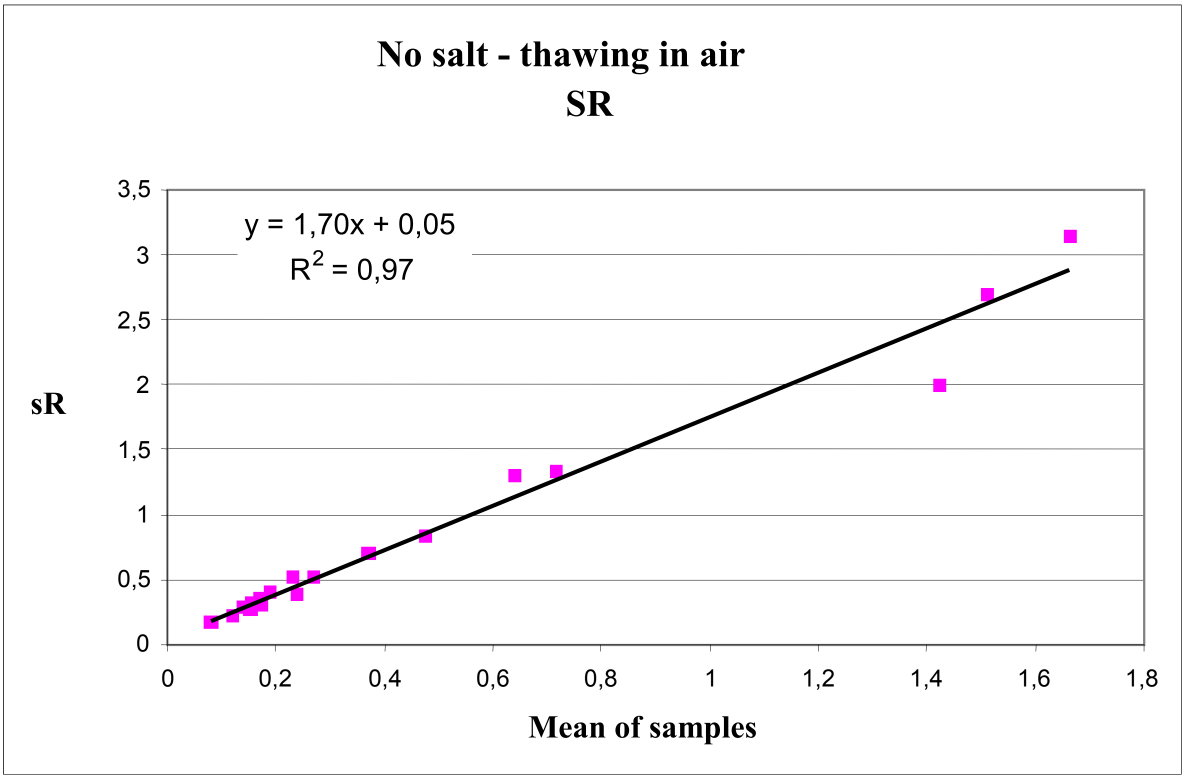


Figure IV 4

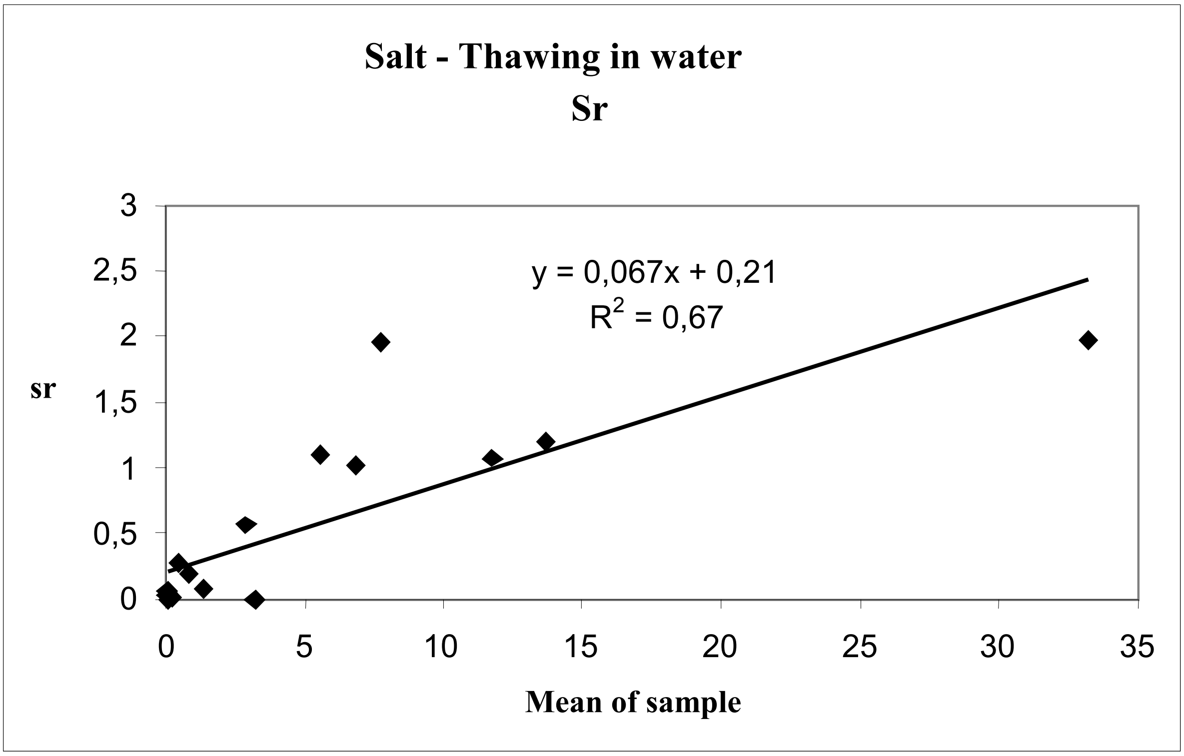


Figure IV 5

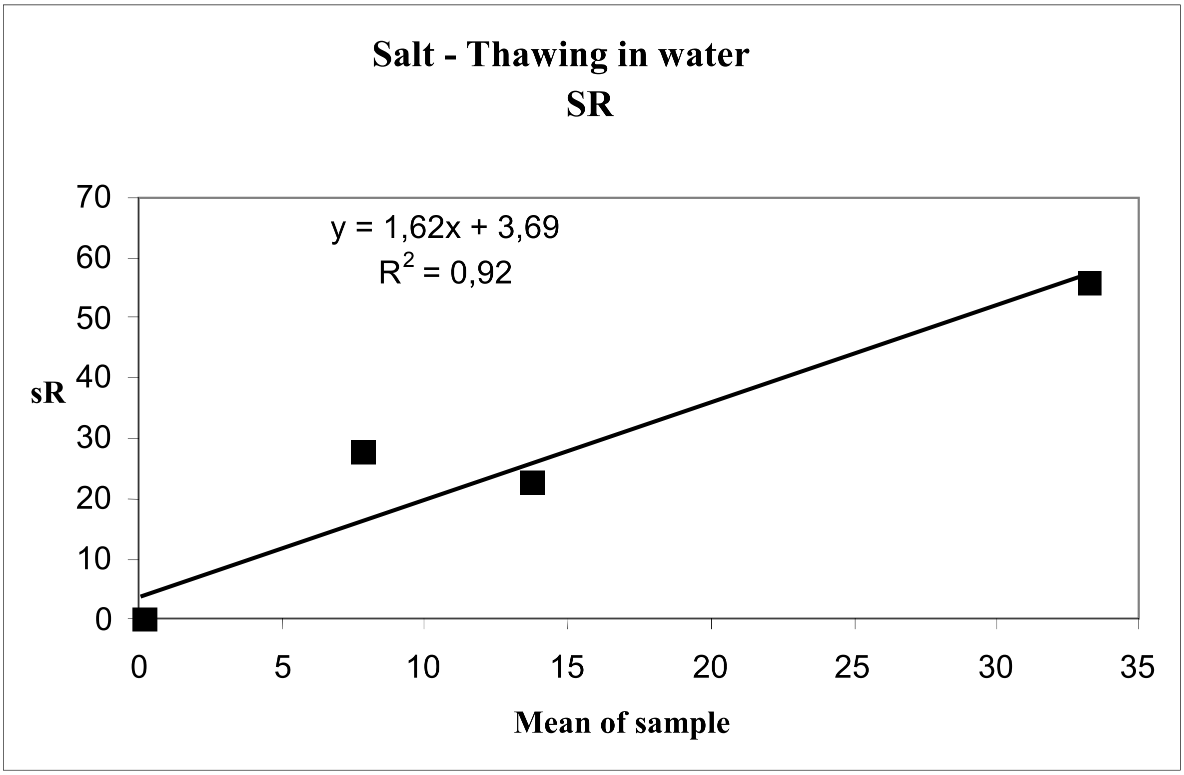


Figure IV 6

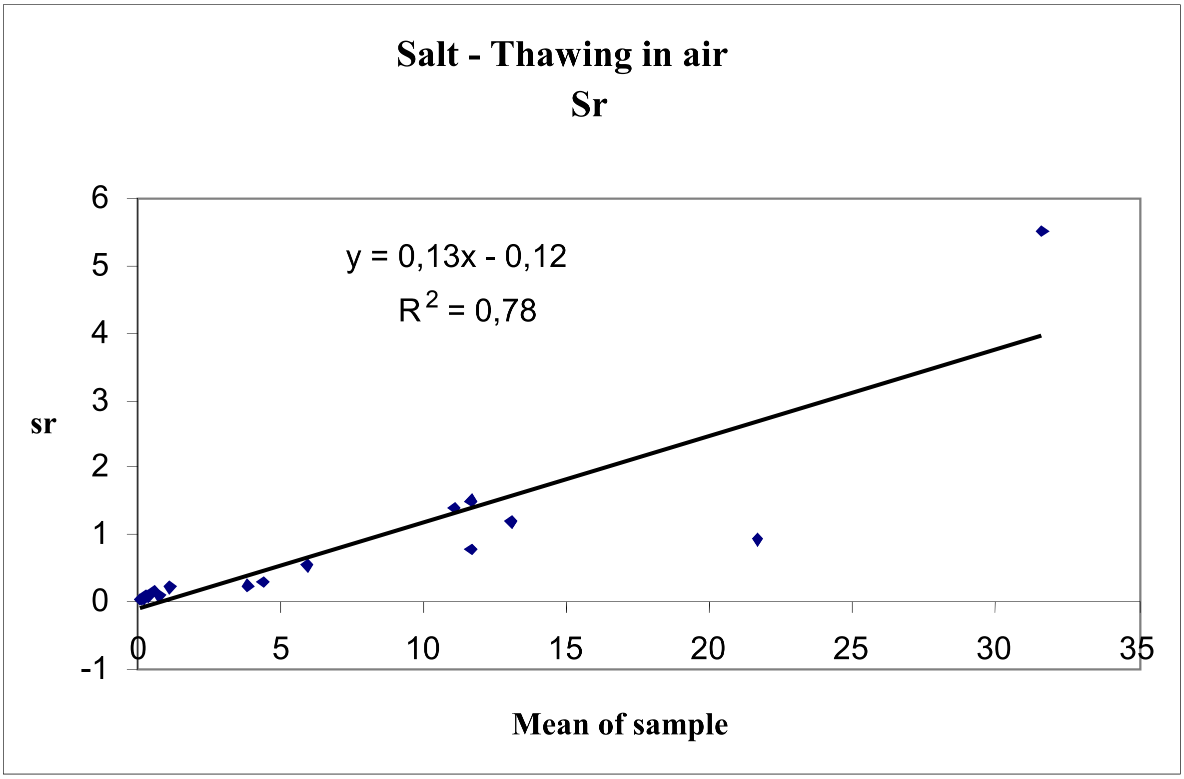


Figure IV 7

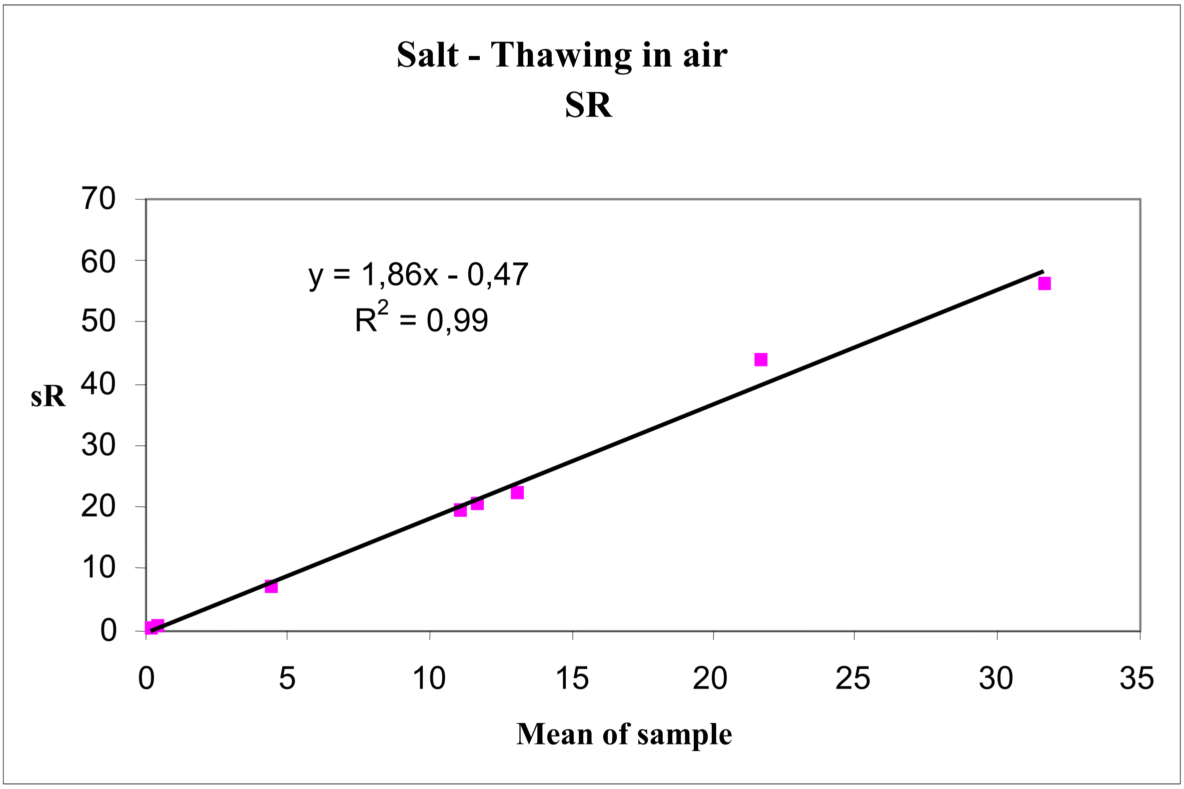


Figure IV 8

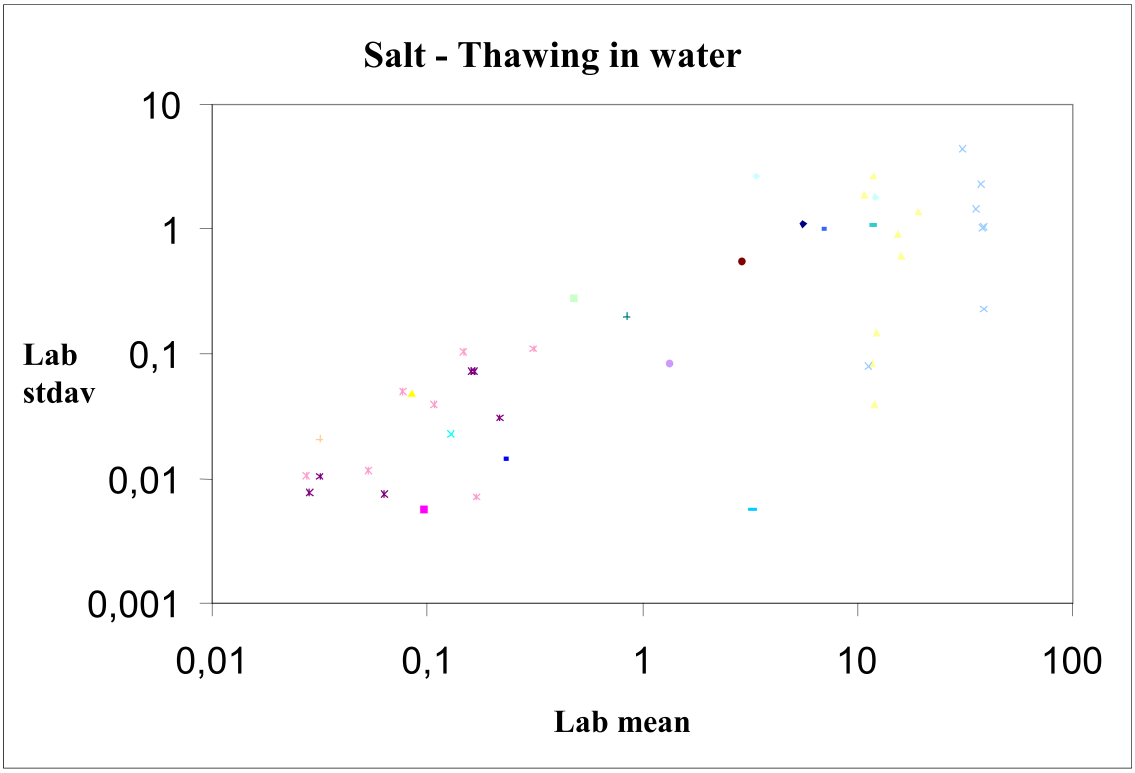


Figure IV 9

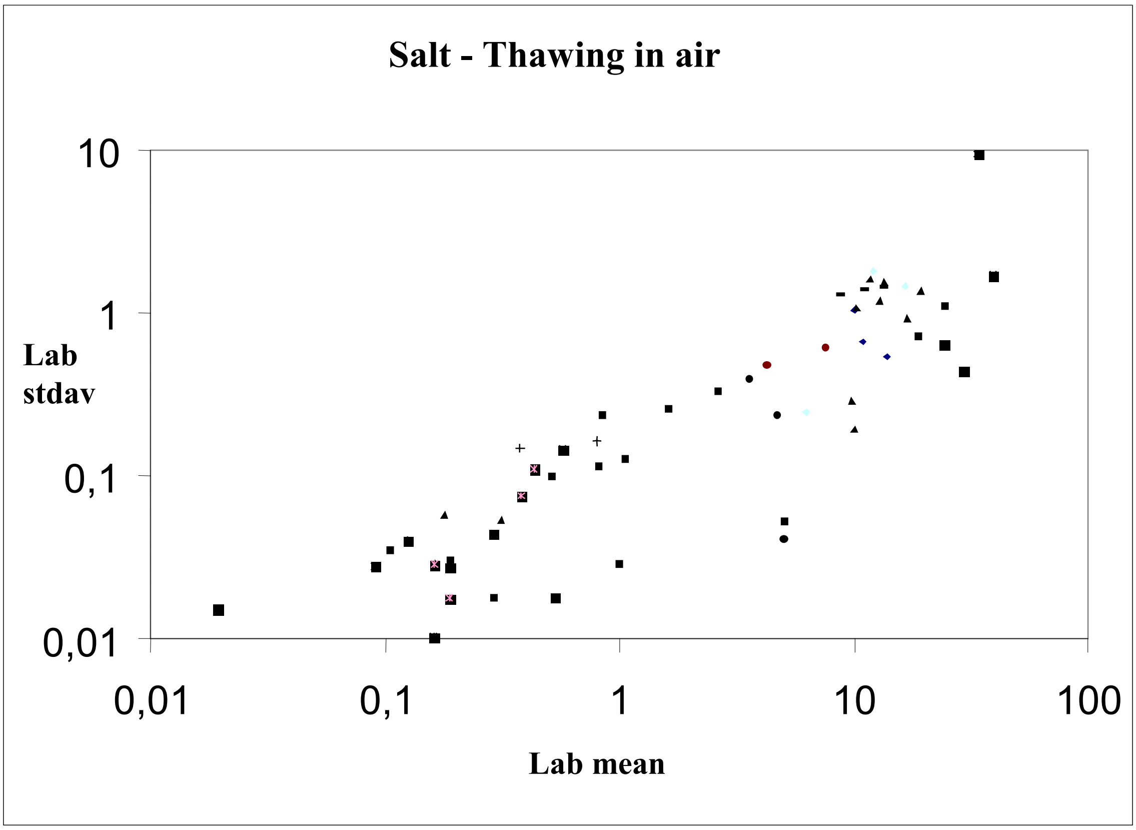


Figure IV 10

Table IV 1. Mean values for each participating laboratory. Salt thawing in water

		RB	RWTH	SP	TUT	MPA Ba	MPA Ba	MPA NF	BASt	Ulster	HOLCIM	KOAC	LfB AG	FEHs	NBTL	DTI	LRPC	Mean	s_r	s_R	No lab
Type	Code	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt	Salt				
Meta-sandstone (p)	2BAX		5,552 1,1014 3															5,552	1,1014		1
Granite loose str.(p)	2COF		0,0966 0,0057 3															0,0966	0,0057		1
Mica rich gneiss (p)	2FEP		0,13 0,0229 3															0,13	0,0229		1
Oolitic limest. (p)	2NUC		3,2331 0,0057 2															3,2331	0,0057		1
Basalt-altered (p)	2REF 1		15,996 0,6006 2			12,32 0,1493 3	11,757 0,085 3	15,43 0,9146 3	12,066 0,039 2	19,264 1,3659 6			11,963 2,634 2	10,788 1,899 2				13,698	1,2101	22,81	8
Limestone (p)	2REF 2		*11,27* 0,0795 3			38,963 0,2268 3	35,823 1,4424 3	38,988 1,0328 3	37,791 2,2609 3				38,351 1,0212 4	31,033 4,4067 3				33,174	1,9765	55,86	7
Landb. por. Flint (p)	2RUN		1,3364 0,0847 3															1,3364	0,0847		1
Gravel (p)	2ZIP		11,744 1,0735 3															11,744	1,0735		1
Micro-diorite (g)	1DIK		0,085 0,0482 3															0,085	0,0482		1
Granite (g)	1GAL		0,0283 0,0076 3			0,16 0,0721 3		0,0317 0,0104 3	0,0633 0,0076 3				0,1667 0,0729 3	0,2167 0,0305 3				0,1111	0,0441	0,2254	6
Basalt-glassy (g)	1GEB		2,924 0,5582 3															2,924	0,5582		1
Granite (g)	1HYD		0,8495 0,2003 3															0,8495	0,2003		1
Greywacke (g)	1MIN		0,2267 0,0144 3															0,2267	0,0144		1
Greywacke (g)	1PEX		3,3759 2,6317 2							12,136 1,7917 6								7,7561	1,9569	28,108	2
Marine, low flint (g)	1QAB		0,4832 *0,282* 3															0,4832	0,2816		1
Granite (g)	1REF 3		0,0275 0,0106 2			0,0533 0,0115 3	0,0767 0,0503 3	0,1471 0,1032 3	0,1699 0,0071 2				0,3125 0,1096 2	0,1075 0,0389 2				0,1278	0,0635	0,2155	7
Natural gravel (g)	1VUX		0,0317 0,0208 3															0,0317	0,0208		1
Granite (g)	1XYD		6,8623 *1,024* 3															6,8623	1,0242		1

Table IV 2. Mean values for each participating laboratory. Salt, thawing in air

Type	Code	RB Salt	RWTH Salt	SP Salt	TUT Salt	MPA Ba Salt	MPA Ba Salt	MPA NF Salt	BASt Salt	Ulster Salt	HOLCIM Salt	KOAC Salt	LfB AG Salt	FEHs Salt	NBTL Salt	DTI Salt	LRPC Salt	Mean	s_r	s_R	No lab
Meta-sandstone (p)	2BAX	10,095 1,0457 3		13,934 0,5423 3							11,024 0,6656 3							11,685	0,7812	20,548	3
Granite loose str.(p)	2COF	0,105 0,035 3		0,2876 0,018 3	0,1889 0,0303 4													0,1938	0,0289	0,3955	3
Mica rich gneiss (p)	2FEP	0,1884 0,0276 3		0,5299 0,0181 3											0,5736 ***0,147*** 3			0,4306	0,0867	0,835	3
Oolitic limest. (p)	2NUC	18,768 0,728 3		24,557 1,1036 3														21,662	0,9349	43,91	2
Basalt-altered (p)	2REF 1	9,7975 0,2864 2		13,015 1,1833 3	13,459 1,5412 2					19,264 1,3659 6	16,91 0,9223 3	10,253 1,0788 2			11,794 1,6113 2	10,053 0,1933 2		13,068	1,1977	22,301	8
Limestone (p)	2REF 2	29,171 0,4427 3		39,043 1,7163 3	33,919 ***9,402*** 4						24,322 0,6425 3							31,614	5,5005	56,556	4
Landb. por. Flint (p)	2RUN	5,0096 0,0415 2		3,5613 0,3969 3												4,6888 0,2375 3		4,4199	0,2931	6,9196	3
Gravel (p)	2ZIP	8,7179 1,3114 3		13,373 1,4604 3								11,114 1,4095 3						11,068	1,3951	19,64	3
Micro-diorite (g)	1DIK	0,1783 0,0577 3		0,313 0,0538 3							0,1631 0,0293 3							0,2182	0,0486	0,4069	3
Granite (g)	1GAL	0,09 0,0279 3		0,1602 0,0103 3														0,1251	0,0210	0,2654	2
Basalt-glassy (g)	1GEB	4,2704 0,4786 3		7,5914 0,61 3														5,9309	0,5483	12,552	2
Granite (g)	1HYD	0,3766 0,1478 3		0,7994 0,1629 3														0,588	0,1555	1,2944	2
Greywacke (g)	1MIN	0,8484 0,2359 3		1,6097 0,2585 3								1,0564 0,1276 3						1,1715	0,2151	2,1512	3
Greywacke (g)	1PEX	6,3072 0,247 3		16,566 1,4627 3						12,136 1,7917 6								11,67	1,5074	26,668	3
Marine, low flint (g)	1QAB	0,5166 0,1002 3		1,0043 0,0291 3												0,8162 0,1151 3		0,779	0,0897	1,4178	3
Granite (g)	1REF 3	0,1875 0,0177 2		0,3814 0,0756 3	0,163 0,0286 2						0,433 0,1108 3							0,2912	0,0787	0,5169	4
Natural gravel (g)	1VUX	0,125 0,04 3		0,2887 0,0443 3											0,0193 0,0152 3			0,1443	0,0355	0,345	3
Granite (g)	1XYD	2,625 0,3357 3		5,0745 0,0532 3														3,8498	0,2403	8,2668	2

Table IV 3. Mean values for each participating laboratory. No salt thawing in water

Type	Code	RB No salt	RWTH No salt	SP No salt	TUT No salt	MPA Ba No salt	MPA Ba No salt	MPA NF No salt	BASt No salt	Ulster No salt	HOLCIM No salt	KOAC No salt	LfB AG No salt	FEHs No salt	NBTL No salt	DTI No salt	LRPC No salt	Mean	s_r	s_R	No lab
Meta-sandstone (p)	2BAX		0,4365 0,1766 3															0,4365	0,17664		1
Granite loose str.(p)	2COF		0,0533 0,0126 3															0,0533	0,01257		1
Mica rich gneiss (p)	2FEP		0,0667 0,0126 3															0,0667	0,01259		1
Oolitic limest. (p)	2NUC		0,1675 0,0034 2															0,1675	0,0034		1
Basalt-altered (p)	2REF 1		1,0548 0,2619 2			1,1733 0,0611 3	0,3267 0,0709 3	0,9883 0,3033 3	0,8998 0,0071 2				1,32 0,2263 2	0,9249 0,1979 2				0,9554	0,18989	1,3069	7
Limestone (p)	2REF 2		1,2397 0,6151 3			2,13 0,25 3	1,9867 0,1026 3	2,7183 0,1377 3	1,4064 0,3708 3				2,0263 0,1652 4	2,3549 0,3311 3				1,9803	0,31799	3,2756	7
Landb. por. Flint (p)	2RUN		0,4067 0,275 3															0,4067	0,27495		1
Gravel (p)	2ZIP		0,2083 0,048 3															0,2083	0,04796		1
Micro-diorite (g)	1DIK		0,07 0,0087 3															0,07	0,00866		1
Granite (g)	1GAL		0,0233 0,0104 3			0,0667 0,0058 3		0,0375 0,0035 2	0,1416 0,0597 3				0,0867 0,0058 3	0,2217 ***0,217*** 3				0,0962	0,09605	0,2184	6
Basalt-glassy (g)	1GEB		0,7231 0,1503 3															0,7231	0,12273		
Granite (g)	1HYD		0,1016 0,0116 3															0,1016	0,00949		
Greywacke (g)	1MIN		0,0267 0,0126 3															0,0267	0,01027		
Greywacke (g)	1PEX		0,0368 0,0223 2															0,0368	0,01578		
Marine, low flint (g)	1QAB		0,0167 0,0029 3															0,0167	0,00236		
Granite (g)	1REF 3		0,0575 0,0106 2			0,0133 0,0058 3	0,0167 0,0058 3	0,06 0,018 3	0,1975 0,0106 2				0,1125 0,0177 2	0,115 0,0071 2				0,0818	0,0117	0,1307	7
Natural gravel (g)	1VUX		0,0933 0,0293 3															0,0933	0,02927		1
Granite (g)	1XYD		0,085 0,018 3															0,085	0,01802		1

Table IV 4. Mean values for each participating laboratory. No salt, thawing in air

Type	Code	RB No salt	RWTH No salt	SP No salt	TUT No salt	MPA Ba No salt	MPA Ba No salt	MPA NF No salt	BASt No salt	Ulster No salt	HOLCIM No salt	KOAC No salt	LfB AG No salt	FEHs No salt	NBTL No salt	DTI No salt	LRPC No salt	Mean	s_r	s_R	No lab
Meta-sandstone (p)	2BAX	0,9114 0,4017 3		0,7945 0,1299 3							0,44467 0,05623 3							0,7169	0,2459	1,3338	3
Granite loose str.(p)	2COF	0,065 0,0477 3		0,2193 0,0101 3	0,1798 0,0214 4													0,1547	0,02958	0,3288	3
Mica rich gneiss (p)	2FEP	0,1516 0,0189 3		0,4209 *0,047* 3											0,2349 0,0061 3			0,2691	0,02955	0,5245	3
Oolitic limest. (p)	2NUC	0,395 0,0564 3		0,5532 0,0339 3													0,469 0,029 3	0,4724	0,04151	0,8307	3
Basalt-altered (p)	2REF 1	1,0599 0,0141 2		1,5965 0,4423 2	1,533 0,1737 2						1,81224 0,14764 3	1,8699 0,2947 3			1,2336 0,2421 2	0,8999 0,1925 3	1,4007 0,0813 3	1,4257	0,2216	1,9939	8
Limestone (p)	2REF 2	1,7169 0,1161 3		1,8041 0,3807 3	2,1884 0,3693 4						0,94774 0,10126 3							1,6643	0,288	3,1426	4
Landb. por. Flint (p)	2RUN	1,2513 0,1711 3		1,3549 0,2894 3												1,9275 0,1319 3		1,5112	0,20851	2,7006	3
Gravel (p)	2ZIP	0,1366 0,0293 3		0,2059 0,0181 3								0,1733 0,0153 3						0,172	0,02173	0,3046	3
Micro-diorite (g)	1DIK	0,1116 0,0301 3		0,1145 0,0609 3							0,33969 0,0902 3							0,1886	0,06518	0,4029	3
Granite (g)	1GAL	0,0517 0,0202 3		0,1089 0,0275 3														0,0803	0,02415	0,1769	2
Basalt-glassy (g)	1GEB	0,545 0,0815 3		0,7328 0,1308 3														0,6389	0,10899	1,3028	2
Granite (g)	1HYD	0,045 0,01 3		0,2103 0,0706 3													0,25 0,05 3	0,1684	0,05029	0,3509	3
Greywacke (g)	1MIN	0,095 0,035 3		0,1579 0,0364 3								0,1066 0,0153 3						0,1199	0,03045	0,2177	3
Greywacke (g)	1PEX	0,3075 0,053 2		0,4328 0,0407 3														0,3701	0,04518	0,6921	2
Marine, low flint (g)	1QAB	0,1184 0,0843 3		0,0795 0,0027 3												0,2233 0,0798 3		0,1404	0,06702	0,2832	3
Granite (g)	1REF 3	0,0825 0,0106 2		0,2808 0,0145 2	0,2576 *0,149* 2						0,32936 0,12068 3							0,2376	0,10165	0,3807	4
Natural gravel (g)	1VUX	0,1166 0,0284 3		0,1995 0,0086 3											0,135 0,0199 3			0,1504	0,02062	0,2719	3
Granite (g)	1XYD	0,1283 0,0252 3		0,3355 0,0292 3														0,2319	0,02725	0,5294	2

APPENDIX V - Compilation of answers to questionnaire

Questionnaire

It is important that you fill in the questionnaire as accurately as possible. Although some of the questions may look odd at a first glance, it is important for the records and the statistical evaluation that all participants answer to their best knowledge. Additional information and/or comments are very welcome. Please note that there are three sheets with questions.

How many test cans did you place in the freeze thaw cabinet at a time?

BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
12	5	9	8	6	20-24	16	12	12

Did you test specimens with and without salt at the same time?

BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
Yes	No	No	Yes/no	Yes/no	Yes	No	Yes	No

How many temperature sensors did you place in sample-cans during testing?

BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
3	0	3	2	1	4	2-4	1	1

How was the thawing process controlled?	BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
Thawing in a 20°C water bath	X			X				X	X
Thawing controlled by air temp.		X	X	X	X	X	X		

Familiarity with the test methods EN 1367-1 and/or TN BUILD 485. Testing according to either method is performed at the laboratory:

	BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
On a routine basis	Yes	Yes	No	No		Yes	Yes	No	No
Once in a while	Yes	No	No	Yes				Yes	No
For the first time in this project	No	No	Yes	No	Yes			No	Yes
Cabinet used for this test procedure before	Yes	Yes	No	Yes			Yes	Yes	No
Cans according to standard	Yes	Yes	Yes	Yes			Yes	Yes	No
Comments	Yes	No	Yes	Yes	Yes	No	No	Yes	Yes

Aggregate properties

Petrographic desctiption of aggregates:

BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
No	DIK	No	No	VUX	REF 1	No	REF 3	PEX
	BAX			FEP	GEB		COF	

How was the experience of the aggregates tested obtained?

	BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
"Poor" aggregate:									
Based on:									
Field performance	No comm.	Yes	No comm.	No comm.	Non	Yes	x	LA=38	
Experimental road sections	No comm.		No comm.	No comm.	Non	No	x		
Petrographic analysis	No comm.		No comm.	No comm.	Yes	Yes	x	Yes	Yes
Previous freeze/thaw testing	No comm.	Yes	No comm.	No comm.	Unknown	Yes	x	No	
"Good" aggregate:									
Based on:									
Field performance	No comm.	Yes	No comm.	No comm.	Good	Yes	Yes	LA=20	x
Experimental road sections	No comm.	Yes	No comm.	No comm.	No	No			x
Petrographic analysis	No comm.	Yes	No comm.	No comm.	Yes	Yes		Yes	x
Previous freeze/thaw testing	No comm.	Yes	No comm.	No comm.	Unknown	Yes		No	x

Values for the water absorption and particle density (on a ssd basis)

BASt	HOLCIM	LRPC	MPA NRW	NBTL	IBRI	SP	TUT	Ulster
Yes	Yes	No	No	No	Yes	Yes	Yes	No