Rannsóknastofnun byggingariðnaðarins

Rb/Sfb
Yp (J)

UDK: 551

Skýrsla nr. 02-07

EVRÓPUSTÖÐLUN Á NÝJU FROSTÞOLSPRÓFI MEÐ SALTLAUSN

Pétur Pétursson

Keldnaholt, maí 2002

Rannsóknastofnun byggingariðnaðarins
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YRSLA

Skýrsla nr: 02-07	Rb/SfB YP (J)	
Dreifing **Opin** ⊗ **Lokuð** □		

UDK - 551

Heiti skýrslu: Evrópustöðlun á nýju frostþolsprófi með saltlausn	**Dags**: Maí 2002
	Fjöldi síðna: 33
Höfundur: Pétur Pétursson	**Faglega ábyrgur**: PP
Deild: Vegtæknideild	**Rannsóknanúmer**: V-5018

Unnið fyrir:
Rannsókna- og þróunarsvið Vegagerðarinnar og Rannsóknastofnun byggingariðnaðarins

Úrdráttur:
Undirritaður kynnti frostþolspróf Steinefnanefndar árið 1994 fyrir TG 9, en það var sú vinnunefnd innan CEN/TC 154 (Aggregates) sem hafði það hlutverk að setja fram prófunaraðferðir fyrir frostþol steinefna. TG 9 fannst aðferðin athyglisverð og sýndi því skilning að þörf væri á saltvatnsprófi. Á sama tíma vann undirritaður einnig að samnorrænu verkefni varðandi frostþolspróf á steinefnum, en það próf er notað hérlendis nú og er kallað Nordtest-prófið (NT BUILD 485 1998). Reynt var að fá það samþykkt sem Evrópustaðal, en vegna tímatakmarkana og ónógra rannsókna reyndist það of seint fyrir fyrstu kynslóð Evrópustaðla. Það sem fékkst samþykkt í fyrstu kynslóðina var: a) upplýsandi viðauki í ferskvatnsstaðalinn, þar sem gefinn er kostur á að prófa í saltvatni (óskilgreind aðferð) og b) tilvísun í þá prófunaraðferð í framleiðslustöðlum um prófanir á steinefnum til ýmissa nota (malbik, steypu, óbundin o.s.frv.).

CEN/TC 154/SC 6 (Aggregates-test methods) hefur nú nýverið sett á laggirnar nýja vinnunefnd, TG 12, sem meðal annars er ætlað það hlutverk að setja fram nýja prófunaraðferð til prófana á frostþoli steinefna í saltlausn. Nýja nefndin hóf störf á þessu ári og mikilvægt er að hafa áhrif á störf nefndarinnar við stöðlun á frostþolsprófi með saltvatni, þannig að það geti hentað íslenskum steinefnum og aðstæðum. Skýrslan varpar ljósi á þau skref sem tekin hafa verið til þessa af Íslands hálfu innan TG 12, en sú vinna var styrkt af Rannsóknasjóði Vegagerðarinnar og Rannsóknastofnun byggingariðnaðarins.

3 lykilorð: Á íslensku	**Á ensku**
Evrópustaðlar	European Standards
CEN/TC 154/TG 12	CEN/TC 154/TG 12
Frostþolspróf með saltlausn	Frost resistance test with salt

INNGANGUR

Undirritaður hefur haft það verkefni að fylgjast með stöðlunarferli Evrópustaðlanefndar CEN/TC 154/SC 6 “Aggregtates, test methods”. Sú nefnd hefur nú sett á laggirnar nýja vinnunefnd (task group) TG 12 “Test methods for thermal and weathering properties”. Þeirri vinnunefnd hefur verið falið að setja fram tillögu að nýju frostþolsprófi fyrir steinefni, þar sem prófað er í saltlausn. Slíkt próf hefur verið baráttumál okkar í tæpan áratug innan CEN/TC 154 og er þessi vinna afrakstur þeirrar baráttu. Fundur þessi var haldinn í Brussel hjá FEDIEX þann 26. apríl 2002 og var aðalefni fundarins umrætt próf. Undirritaður hafði fengið leyfi hjá formanni nefndarinnar til þess að kynna Nordtest frostþolsprófið (sem fékkst samþykkt sem slíkt 1998) sem tillögu að Evrópustaðli. Erindið fékk góð viðbrögð á fundinum og var ákveðið að leggja til að Nordtest staðallin yrði tekinn upp lítið breyttur sem Evrópupróf, eða a.m.k. sem “Normative Annex” við núgildandi staðal EN 1367-1.

Undirrituðum var falið að útbúa prófið til nánari umfjöllunar á næsta fundi vinnunefndar TG 12 sem haldinn verður í október 2002. Reiknað er með að á þeim fundi verið tekin ákvörðun um frekari prófanir á evrópskum steinefnum með saltvatnsaðferðinni, enda er ljóst að slíkt verkefni er talið nausynlegt til þess að prófunaraðferðin fái brautargengi. Formaður TG 12 mun því leggja til á næsta fundi SC 6, sem haldinn verður á Ítalíu 17. júní næstkomandi, að tímaramminn verði framlengdur, en stefnt var að því að setja prófið í formlega atkvæðagreiðslu í árslok 2003.

Íslenskir hagsmunir felast í því að prófunaraðferðin nái fram að ganga og verði að Evrópustaðli. Það er ljóst að ferskvatnsprófið nýtist okkur ekki til þess að greina á milli frostþolinna og frostnæmra steinefna. Það eru því miklir hagsmunir í húfi fyrir framleiðendur og notendur steinefna að unnt verði að sýna fram á að frostþol þeirra steinefna sem notuð eru sé viðunandi.

Í þessari skýrslu er birt fundargerð fundarins sem haldinn var í Brussel 26. apríl síðast liðinn. Einnig er birt erindi það sem flutt var á fundinum af hálfu Íslands, svo og tillaga að frostþolsstaðli með saltlausn sem undirritaður hefur útbúið og verður unnið með í framtíðinni.

Fundargerð 1. fundar TG 12 sem haldinn var í apríl 2002, ásamt nafnalista

TC 154 TG12 : « Tests Methods for Chemical, Thermal and Weathering Properties »

Present : Dr R. Collins **(RC)** (UK), G. Dirickx **(GD)** (B), Dr M. Kostjak **(MK)** (A), F. Henin **(FH)** (B) Chairman, J. Lay **(JL)** (UK), P. Petursson **(PP)** (IS), Dr A.-M. Marion **(AMM)** (B) Secretary, Dr H. Motz **(HM)** (D), Dr E. Siebel **(ES)** (D), Dr FR Schreiber **(FRS)** (D), J. Vecoven **(JV)** (F) replacing **FHA**, Dr J. Daube **(JD)** (B) former Secretary.

Apologies received from : F. Hawthorn **(FHA)** (F), Y. Houst **(YH)** (CH), Dr V. Jensen **(VJ)** (N), FZW Norman **(FN)** (IRL), Dr Th. Reimer **(TR)** (D), S. Seytre **(SS)** (F)

Minutes of the first meeting held in Brussels on 26th April 2002.

The chairman welcomes the participants, inviting them to introduce themselves.

The list of the documents that are provided in session is quickly examined and the main items to be discussed during the meeting are briefly evoked :

* Discussion of the last prEN 1744-4 draft : Determination of water susceptibility of fillers for bituminous mixtures – German comments and request

* Amendment to EN 1367-1 : Freeze-Thaw with NaCl

* Any other business → Amendment to EN 1744-1 for recycled aggregates :
  * prEN 1744-5 : Determination of acid soluble chloride salts
  * prEN 1744-6 : Determination of the influence of the aggregate extract on the initial setting time of cement

  Amendment to EN 1367-4 for recycled aggregate :
  * Determination of drying shrinkage

No particular comment is made on this agenda.

Discussion of the last prEN 1744-4 draft : Determination of water susceptibility of fillers for bituminous mixtures – German comments and request

HM specifies that his comments reflect the position of the German delegation. Actually, a German standard is existing and an informative annex in a European standard could not be considered, in Germany, as normative from a legal point of view. The problem as it appears through the vote is that the other countries don't want the Marshall test to be included as a normative step 2.

According to the vote, the Marshall test is authorised to be kept in an annex A: an attempt to try to reconcile both Germany and other countries, could be that the annex A should be designated as « normative » instead of « informative ». **HM** is kindly invited to modify the draft in that way : the revised draft should be transmitted for examination to TG 12 members before to be sent to SC6.
FRS draws attention on item 5.1.3 of the discussed draft and propose to better define the ratio Bitumen / kerosene. **GD** does not agree : it is not possible because bitumen is not standardised (for example, 50/70 does not exist in Netherlands). The chairman will consult Netherlands and Belgium experts to try to find a more precise definition.
Another anomaly concerns item A.1.2. b) which is not correct, because the limit is higher than 4 : to be modified by **HM** during the revision.

Amendments of EN 1367-1 : Freeze-Thaw with NaCl

PP presents the results of an interesting study that was made in Iceland. He recalls that the freeze-thaw with salt was proposed in TG9 as early as 1990 and is became a national standard in Iceland in 1998 (NT Build 485). Some comparative examination of the experimental procedure of both methods is discussed (NT Build 485 with salt versus EN 1367-1 no salt). Some data issued from both methods are also presented : those results show clearly that the freeze-thaw with salt is more aggressive. It appears that in Iceland a method with salt is really necessary to can better appreciate the potential vulnerability of a great number of aggregates. According to the Iceland experience, a value lower than 4 obtained through NT Build 485 would be a reliable indication for a satisfying behaviour of the tested aggregate. **GD** does not totally agree because the behaviour during the lab test is not necessary representative of the behaviour on the field (this is true for each parameter studied with any laboratory procedure). The problem relates also to the definition of limit values : according to **PP** experience, the use of an aggregate for well defined application could be deducted from their behaviour during the NT Build 485 and it should be possible to discriminate between different classes and application on the basis of the behaviour through the NT Build 485.
The final proposition from Iceland delegate is the following :

- The European test with salt should be based on NT Build 485
- The aggregate size should be enlarged to 4-63
- The system used to attain the required temperature should be optional : air or water
- There is a need for introducing a tolerance for acceptable dispersion between 2 simultaneous conducted tests.

According to the other participants, it would appear better to work with three specimens instead of 2. **PP** thinks that the way of calculation of the « final » result as it is used in 1367-1 is not ideal : it is indeed an average value based on the results from three individual measurements which is performed, with no specification on the acceptable dispersion between the three results.
According to the TG 12 working agenda a draft should be produced for the end of 2002 : this is quite impossible because it is necessary, before a draft has to be written, to know how will be the results of such a test in comparison with the result from EN 1367-1. It is very important in terms of future requirements. Therefore, aggregates from other localisation than from Iceland have to be tested and an interlaboratory trial has to be organised in a next future : the chairman has to inform SC6 about this particular item and the delay versus the expected agenda.

In his letter coming with the previous draft from TG9, **FN** mentions that CEN are not likely to accept a second freeze-thaw test : so the question of a normative annex remains open to discussion.
PP is kindly requested for making a proposition for a revised draft of the method with salt.
Some details from the existing draft are examined and discussed in session:
Foreword : 3. Replace by : the sample is tested in a 1% solution of NaCl in de-ionised water instead of de-ionised water.
5. Three test portions are tested and the results are considered separately
Item 5.7 NaCl of analytical grade
8.2.c. Impossible to maintain 0°C with salt→ modify 0°C through 0 to –1°C
Table 1 : to harmonise with EN 1367-1 → 4-63 instead of 8-16 ; suppress «maximum» in «maximum aggregate size»
Requirement for reaching the recommended temperature : optional air or water
PP is in charge of producing a draft to be circulated for agreement in TG12 before to be submitted to SC6.
The organisation of an interlaboratory trial has to be foreseen as soon as possible and SC6 has to be informed by **FH** that the entire process will take a lot of time.

Any other business → Amendments of EN 1744-1 for recycled aggregates
→ EN 1744-5 : Determination of acid soluble chloride salts
→ EN 1744-6 : Determination of the influence of the aggregate extract on the initial setting time of cement

FH informs the members that both drafts previously produced by TG 8 have been sent again to B. Feldman for examination by SC6 and submission to CEN enquiry. This item will be discussed at the SC6 meeting on the 17th June in Milan where **FH** will be present.

Any other business → Amendment to EN 1367-4 for recycled aggregate : Determination of drying shrinkage

As UK seems to have the best experience in that field, **RC** gently proposed to write a draft relating to that point.

TG4 activities
JL gives a brief report on TG 4 activities : no big work at the time, it is in stand-by. He also presents the progress in the voting process of the various EN.

ASR information about the work of Rilem TC ARP and the « Partner » project.
AMM reviews briefly the objectives of the Rilem TC ARP (previous TC 106) and describes shortly the main goals of the European project « Partner » subsidised by CEE. Some more information relating to both points are summarised in annexe 2 document as a support for presentation by **FH** at the next SC6 meeting (17 June 2002).

ES tells about VDZ's experience of a different behavior of concrete with pozzolanic addition, through laboratory testing and outdoor exposure.

The date and venue of the next meeting is discussed. Provisional dates are defined : 31 October 2002 and/or 9 may 2003. **JV** offers to organise the October meeting in Paris and **ES** also proposes Düsseldorf for the May meeting : both kind propositions are immediately accepted with thanks and enthusiasm. These dates and destinations have to be confirmed in relation to the SC6 agenda.

The members of TG12 takes the opportunity to make a standing ovation in honour of Mr Daube and his brilliant participation to the CEN activities.

The chairman thanks all the participants and closes the meeting at 12H00.

Annex 1

List of agreed actions
Meeting held on 26 April 2002

FH Take information on how to deal with the bitumen/kerosene ratio (1744-4)
Participate to SC6 meeting in Milan on 17 June 2002

Inform B. Feldman on the necessary delay (need for an interlaboratory trial) for preparing amendment for EN 1367-1

HM Prepare a revised 1744-4 draft and send it to **FH** for circulation in TG12

PP Prepare a proposition for amendment of EN-1367-1/ Freeze/thaw test with salt and send it to **FH** for circulation in TG12

RC Prepare a proposition for amendment of EN 1367-4 / Drying shrinkage of recycled aggregate and send it to **FH** for circulation in TG12

AMM Prepare a summary about the Rilem TC ARP work and the European « Partner » project (done → see annex 2)

ALL Think to the future interlaboratory trial for Freeze/thaw test with salt (amendment for EN 1367-1) :

- participants at national level
- aggregates to be tested
- financial support from the European Commission

Annex 2

Rilem TC ARP : «Alkali-Reactivity and Prevention : Specification, Assessment and Diagnosis»

This TC, previously designated as TC106, has been created in the end of the eighties. Its main objective is to try to select harmonised methods, acceptable on an international level, to allow the characterisation of the alkali-sensitivity of aggregates . Up to now, 4 methods have been retained : petrography (AAR-1), as a basic characterisation tool, and 3 expansion methods, to know ultra-accelerated mortar bar test (immersion in 1N NaOH / 80 °C / 14 d) (AAR-2), « normal » concrete prism test (close to 100 % RH, 38°C / 1 y) (AAR-3) and accelerated concrete prism test (close to 100 % RH, 60 °C / 3 to 6 m) (AAR-4). ARR-1should be published in the Rilem publication (Materials and Structures) in the course of 2002 while AAR-2 and –3 have already been published in this Journal in June 2000. An interlaboratory trial using the petrography has revealed how important it is that the petrographer should have experience in the examination of rocks for a correct identification of potentially reactive species. AAR-2 and –3 have been evaluated through interlaboratory trials on aggregate recognised from the field to be non reactive and reactive : about 10 different countries have submitted two local aggregates to both methods and the results suggest that the discrimination power of both expansion tests would be promising. Concerning AAR-4, which is actually an adaptation of the so-called « French performance Test », an interlaboratory test is being organised at the moment : the starting is foreseen for June 2002 at the latest. About 20 participants have offered to participate. The mandatory testing program concerns two « reference aggregate combinations » ; one potentially reactive (Tournasian limestone and Boulonnais limestone as reactive aggregate and non-reactive sand respectively), the other non-reactive (Boulonnais limestone as sand and aggregate). The testing is also opened to other local combinations from which the behaviour on the field is well documented. The Rilem TC ARP has edited his own version of the « French performance test » : this draft includes three different applications of the method, to know its use for investigating the alkali sensitivity of aggregates, its application as a performance test to evaluate the risk of ASR development when using a well defined concrete composition and thirdly the method is usable to determine the « alkali threshold value », that means the maximum alkali content that could be added to the concrete without significant risk for initiating ASR

The TC ARP is also busy with other miscellaneous tasks in relation with alkali pathology : alkali-carbonate reaction (mainly taken into account by experts from Austria and China), releasable alkalis from aggregate (main concern for Canada), and the edition of a guidance for ASR/ACR diagnosis. A lot of work is still to be done on the harmonisation of the already edited drafts relating to the three retained expansion methods (AAR-2, -3 and -4).

European Partner Project

This European subsidised research Project gathers 14 countries and 25 participants. This ambitious project, officially started on 1st march 2002 and running for 4 years, is built on three main objectives :

1. The edition of a geological atlas of the most common alkali reactive aggregate types in Europe : this catalogue is foreseen as a necessary tool for education of « novice petrographer ». The Rilem petrographic method will be the analytical base for conducting this work. The atlas should concern about 40 different aggregate types.

2. The investigation of the discrimination power of the 3 retained Rilem expansion methods : the ultra accelerated mortar bars test will be used as well as the « normal and accelerated » methods on concrete prisms. In parallel, some participants will also test some of the methods currently used in their own country. Some « special » method will also be performed for « special aggregate » :for porous flint notably, where the application of the ultra accelerated mortar bar seems to be inappropriate, a particular chemical test will probably be studied. A tenth of those aggregates investigated in objective 1 will be selected to be submitted to the various expansion tests. Last but not least, this item being of particular interest for correlating the behaviour in the lab and on the field, field test will be conducted with outside exposure of concrete samples : to better delimit the influence of environmental conditions, 3 different exposition sites have been selected with significant variation in climate (south, centre and north of Europe)

3. The study of the statistical parameters is another critical point for assessing the reliability of an expansion method. This important work will be done on 3 to 4 different aggregates, among which one non reactive, in order to investigate the repeatability and reproducibility of each method, judged as reliable from result obtained through objective 2.

4. The data obtained and the newly revised and edited methods, if considered as appropriate for classifying aggregate, will then be disseminated as large as possible at the national and CEN level but also internationally. The final results should constitute an experimental basis for establishing CEN standards describing methods for ASR characterisation and investigation.

The present secretary of TC 154 TG 12 being a Rilem TC ARP member and belonging to the Partner Consortium has actually been sounded out for ensure the liaison between all those experts.

List of assignees (Continued)
CEN/TC 154/SC6/TG12.....
Date: 2002-04-26 Place: ..BRUSSELS.....

Date(s) of meeting: 2002-04-26

Place of meeting .: BRUSSELS.....

*** CEN/TC154/SC6/TG12. Title Test methods for chemical, thermal and weathering properties**

Convenor: (name) **.....HENIN FREDDY..... Country...BELGIUM.....**

Secretariat : (name) **.....Dr.MARION ANNE-MARIE..... Country : BELGIUM.....**

(* Only complete that part which is appropriate)
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CEN/TC 154/SC6/TG12.....
Date: 2002-04-26 Place: ..BRUSSELS.....

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Petursson	PP		26/4	IBRI	I
JAUBE	J.		26/4	IBN	B
Siebel	ES		26.4	VDI Germany	D
LAY	J	J. Lay	26 April 2002	BSI	UK
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HENIN	F		2604	IBN	B

+ present → Dr. COLLINS → not accepted joint b.

Ref. HF/2002/TG12/16 (13/05/2002) TG12 member list .

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List of TG12 documents : (13/05/2002)

HF/2002/TG12/01 : letter 27/02/02 : first meeting 26/04/02 Brussels : first call

HF/2002/TG12/02 : member list (27/02)

HF/2002/TG12/03 : letter 26/03/02 : first meeting 26/04/02 Brussels : second call

HF/2002/TG12/04 : member list (26/03)

HF/2002/TG12/05 : draft agenda first meeting Brussels

HF/2002/TG12/06 : Fediex acces map

HF/2002/TG12/07 : documents from Iceland EN 1367-1 (freeze/thaw with salt)

HF/2002/TG12/08 : pr EN 1744-4 + reaction from Germany

HF/2002/TG12/09 : updated member list (26/04)

HF/2002/TG12/10 : documents on TG12 creation :
Notes of the last meeting of past TG8 (4/05/2001 London) and TG9 (6-7/04/1999 Thessaloniki)
CEN TC154 / SC6 / N776E report of the last SC6 meeting (14/05/2001 London)
CEN TC154 / N655E report of the last TC154 meeting (11-12/10/2001 Maastricht)
CEN TC154 / N665E , N666E and N 662E : pr EN 1744-3 : with the result of formal vote
CEN TC154/ SC6 / N779E (08/11/2001)first report on TG12 activities
Programme of work

HF/2002/TG12/11 : drafts send to SC6 :
prEN 1744-5 : acid-solube chloride salts content (TG12-N1 / TG8-N108)
prEN 1744-6 : influence of recycled aggregate extract on the initial setting time of cement (TG12-N2/TG8-N109)

HF/2002/TG12/12 : CEN TC154 / N618EFD : comments on the first enquiry on prEN 1744-4 Determination of water susceptibility of filler for bituminous mixtures

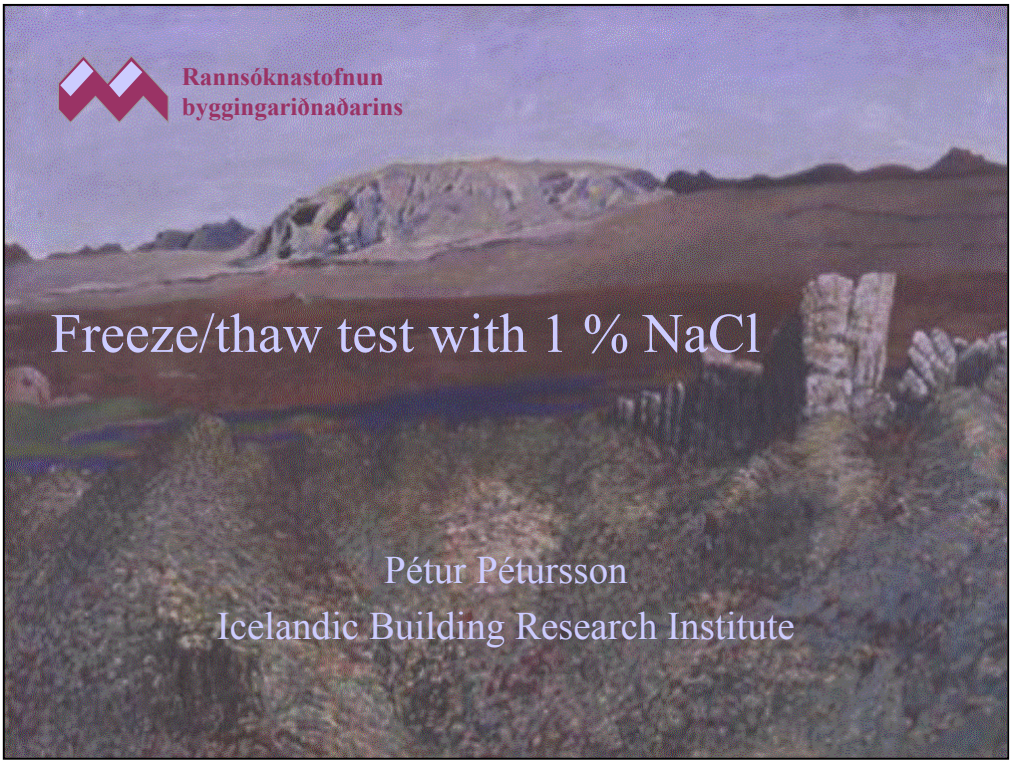
HF/2002/TG12/13 : e-mail from Mr Norman , Irish position on prEn 1744-4 + TG9 380 : first draft on freeze/thaw test with salt

HF/2002/TG12/14 : PWT presentation from Iceland (P.Petursson) : EN 1367-1 (freeze/thaw with salt)

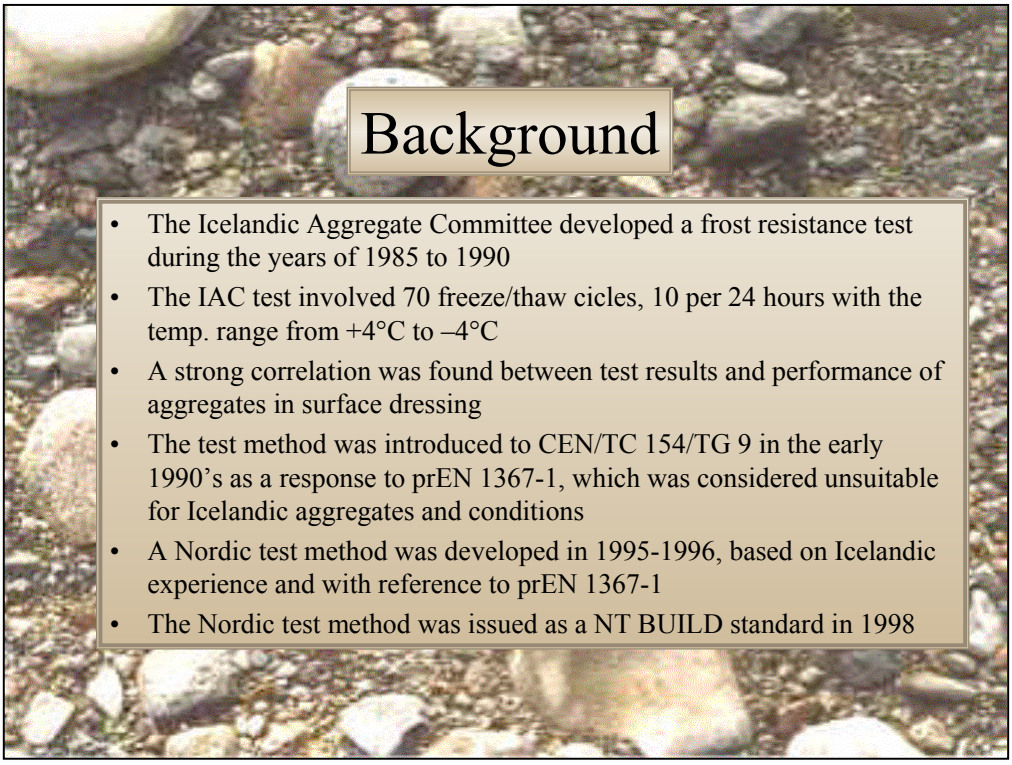
HF/2002/TG12/15 : Minutes of the first meeting held in Brussels on 26th April 2002

HF/2002/TG12/16 : updated member list (13/05)

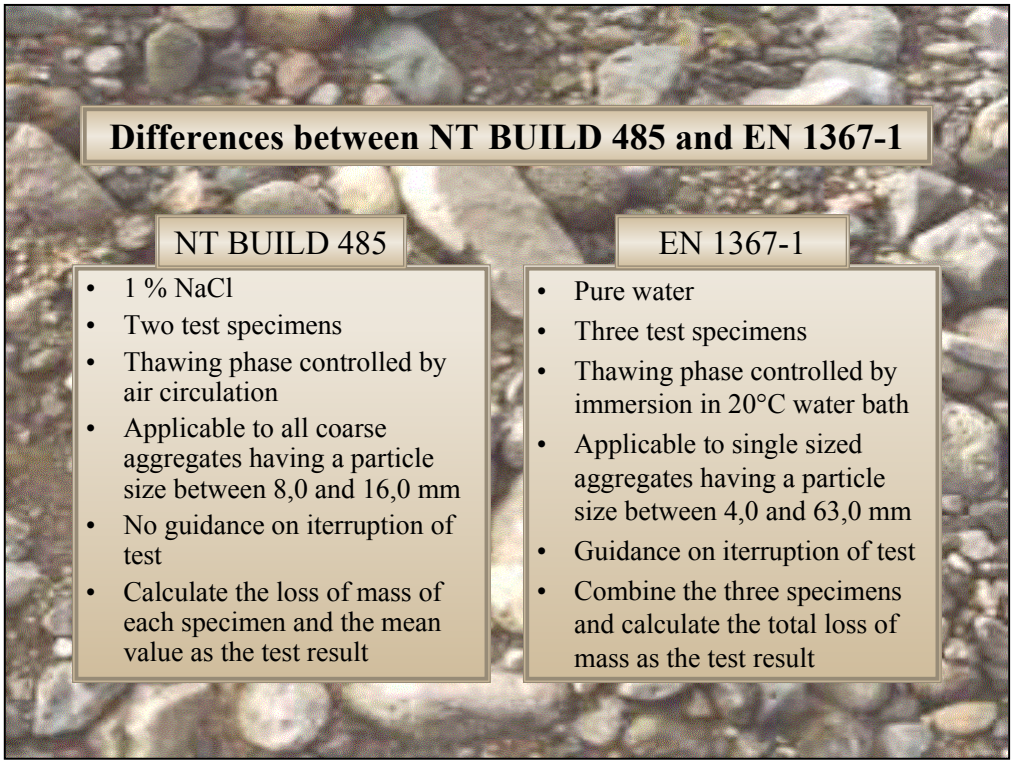
Erindi Péturs Péturssonar sem haldið var
á fundi TG 12 í apríl 2002



I would like to thank Freddy Henin for the opportunity to give this presentation on freeze/thaw test with salt at this first meeting of CEN/TC 154/TG 12 on 26 April 2002. First, I would like to give a brief overview of the develepment in this field for the past 15 years.

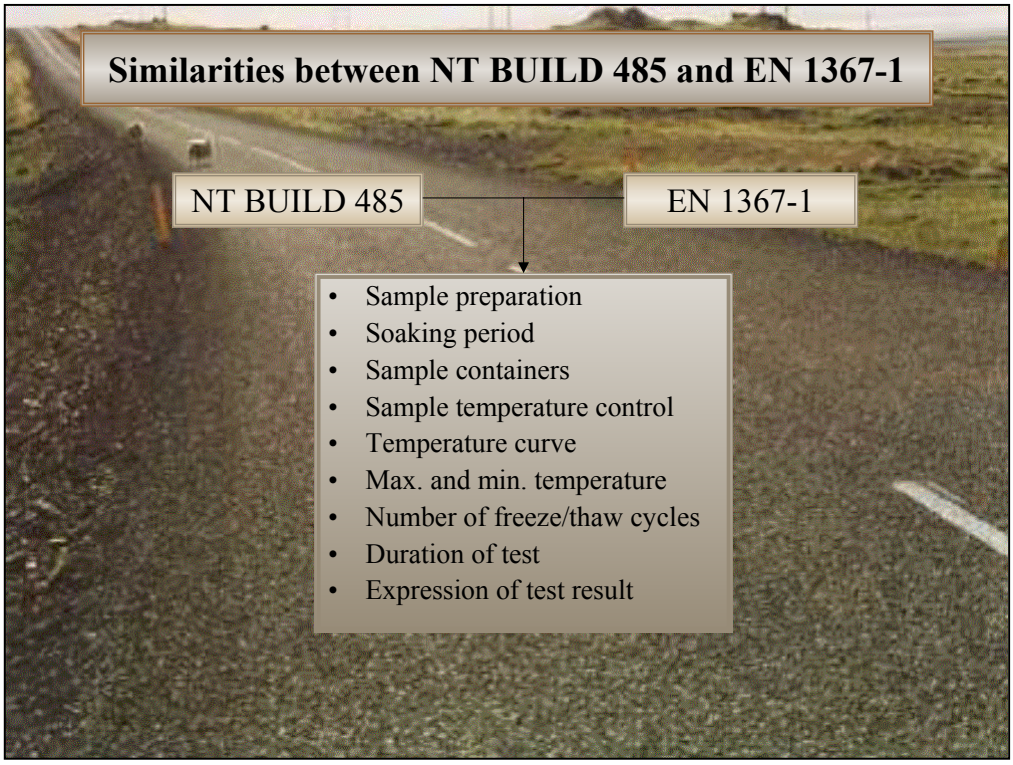


It was decided in 1985 to develop a new freeze/thaw test method in Iceland.. The intention was to develop a method which would reflect actual number of freeze/thaw cicles as well as the most frequent minimum temperature. Therefore 70 cicles were chosen with minimum temperature of –4°C. A correlation between test results and actual performance of aggregates was established. The method was introduced to CEN/TC 154/TG 9 to demonstrate that Iceland would require a frost resistance test in salt water as an option to the pure water method which was under consideration. A co-Nordic 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 repalced the older method. It also became a formal Nordtest standard in 1998.

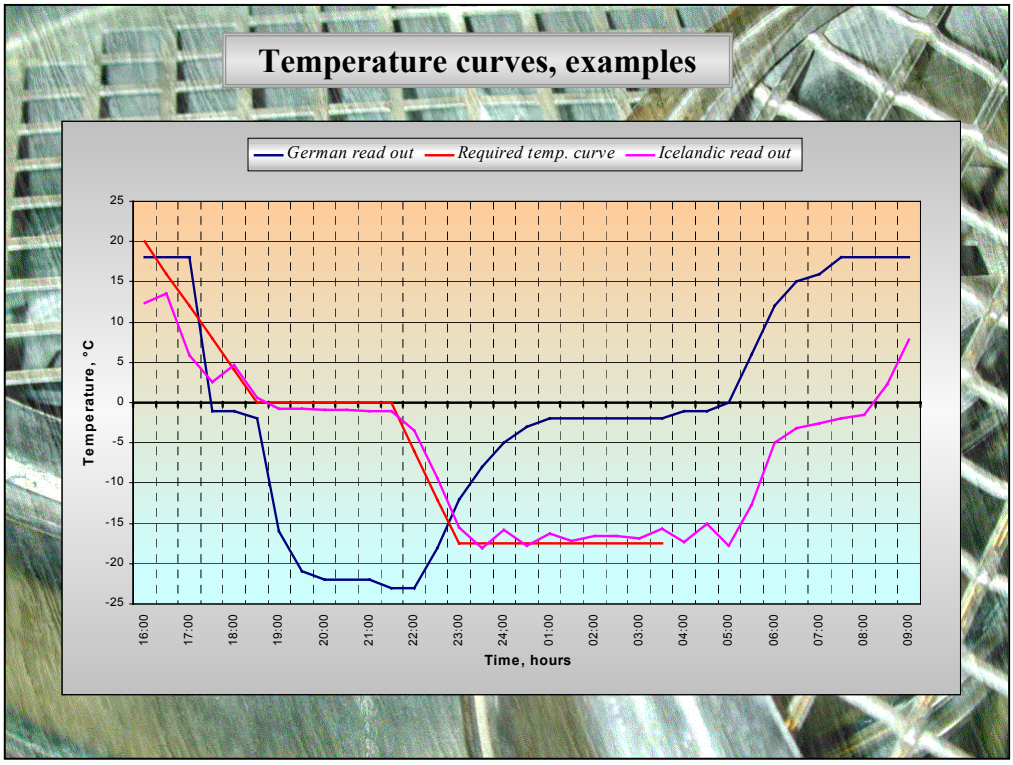


The main difference between the two methods is of course the use of a salt solution in the Nordtest method, but pure water in the EN method. Another difference is that two paralell specimens are tested in Nordtest but three in the EN method. Thirdly, the thawing out sequence is controlled by air circulation 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 sequence of the samples. My statement is that it is irrelevant which one of the two methods is used. By focusing on the sample temperature, unnecessary restrictions on equipment can be avoided. That would enable laboratories in the Nordic countries as well as elsewhere, to easily adopt the method and run the Nordic test.

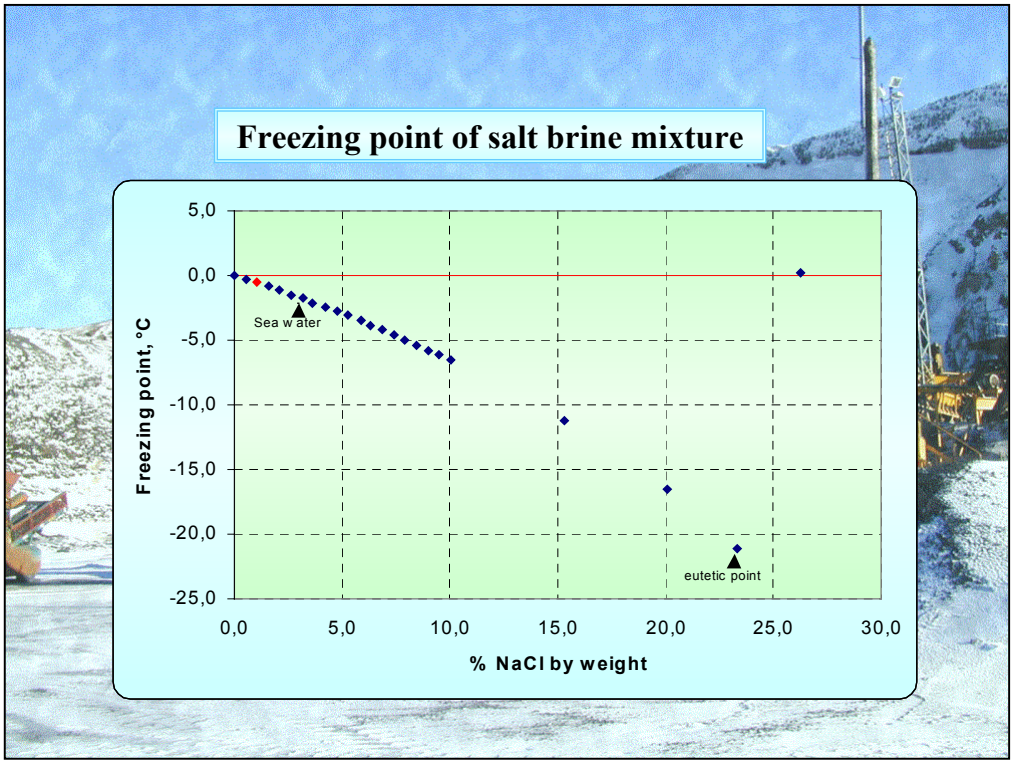
The next two points concerning differences of the test methods are of rather minor importance. The last point, however, is worth some consideration. We would prefer to calculate the % loss of each of the test specimens and then give the result as the mean value. In that way it is immediately observed whether it is likely that mistakes have occurred in the sample preparation or at another stage in the procedure.



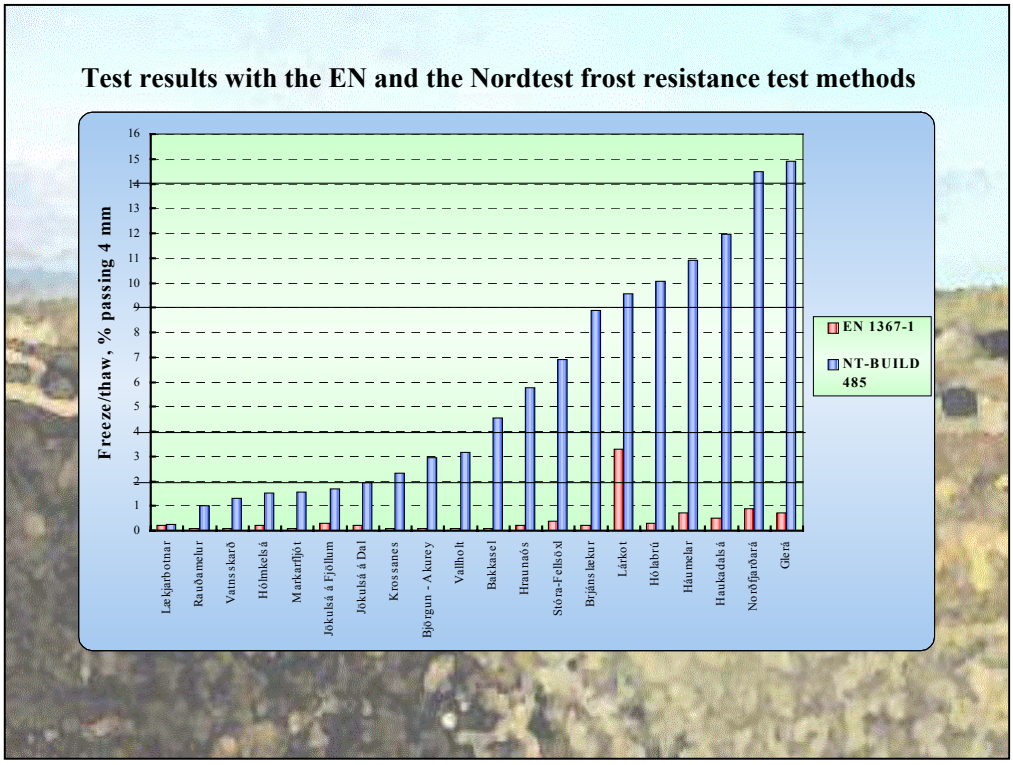
Sample preparation: Washing and drying. Soaking period: 24 hours at atmospheric pressure. Sample container: same dimensions, seamless drawn or welded. Temperature control: reference can in center of cabinet controls the temperature of the sample itself (important). Temperature curve: Same requirements and tolerance limits (thawing out sequence not specified). Number of cicles is the same. Duration of test is the same. Expression of final result is the same, i.e. % loss on a sieve with aperture 1/2 the size og the lower limit of fraction.



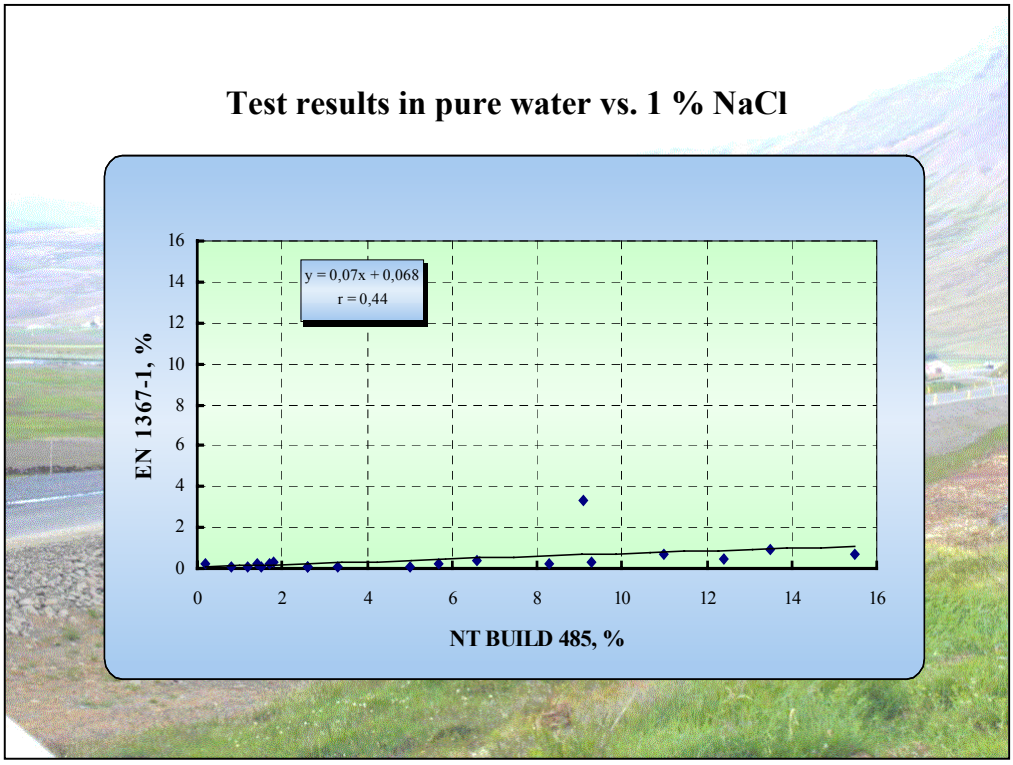
This figure shows two read-outs of temperature curves, as well as the required curve for both test methods. The so-called German read-out is from a printer connected to a cabinet at the BASt. This is since 1992, when I carried out the DIN test at the Bundesanstalt. The Icelandic read-out is a typical one from our cabinet. What is interesting to see is that we are able to control every phase of the test with air circulation, not only the freezing phase, but also the thawing out phase.



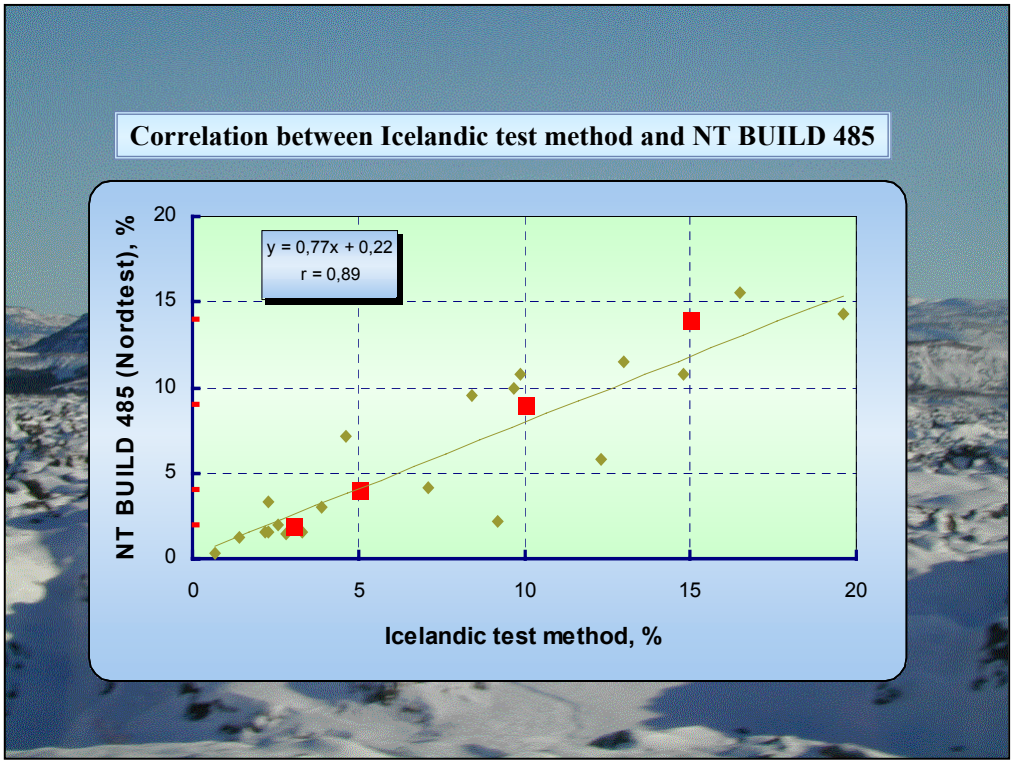
This figure simply shows that a 1 % salt solution freezes at approximately minus 0,4 to 0,5 °C, see the red dot.



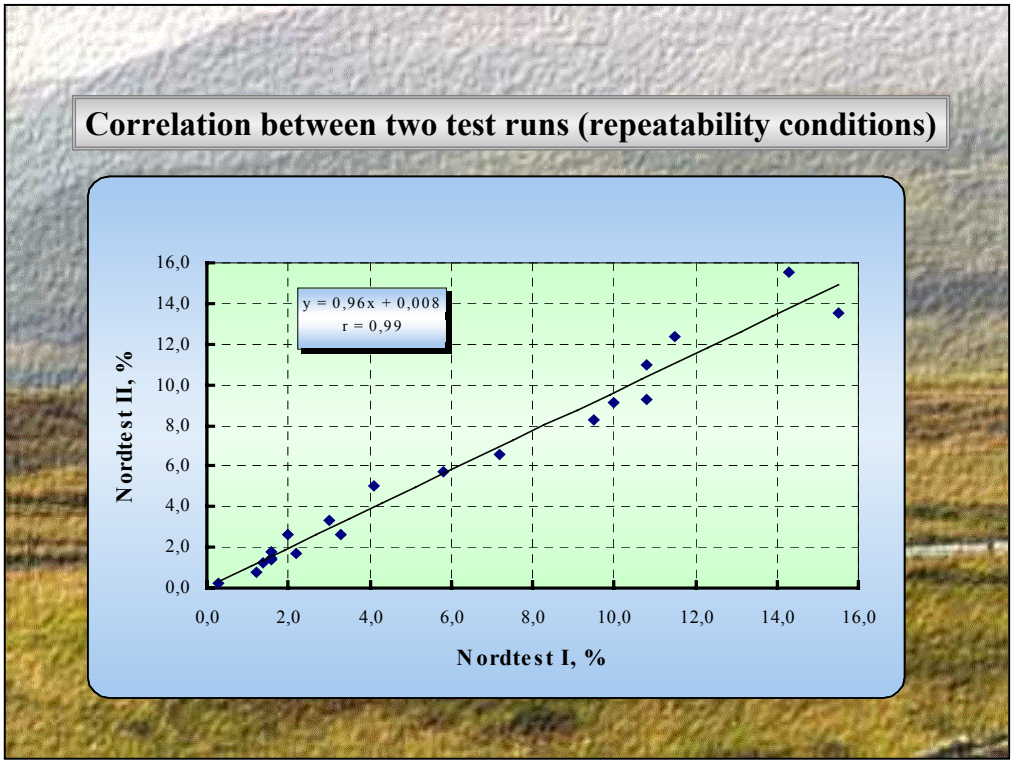
This graph shows test results on 20 Icelandic basalts with the pure water method on the one hand and the salt water method on the other hand. It is obvious that the difference is completely significant. We have long time experince with all these materials and it is very important for us to be able to give values that classify these materials into different frost resistance groups or categories (indicated by the drawn lines). The pure water method fails to distinguish between frost resistant and frost susceptible aggregates in this case.



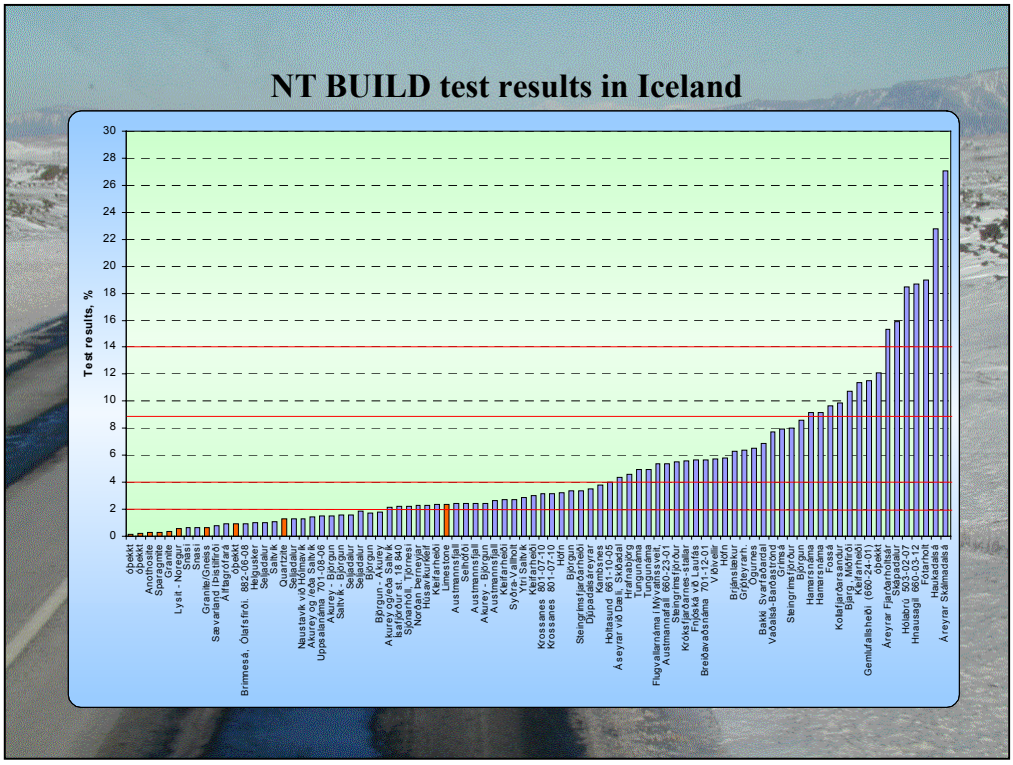
Here is the same data plotted as a correlation diagram. It is obvious that the correlation is rather poor, r being 0,44. Even though the correlation was better, it is still evident that the pure water method does not make it possible to classify the aggregates according to their frost resistance.



This figure shows that the correlation between the Nordtest results and the older Icelandic test results is quite convincing, still using the same 20 aggregate samples. The existing requirements for the Icelandic method are therefore valuable for us to suggest requirements for the Nordtest method by simple reflection and distraction by one unit.

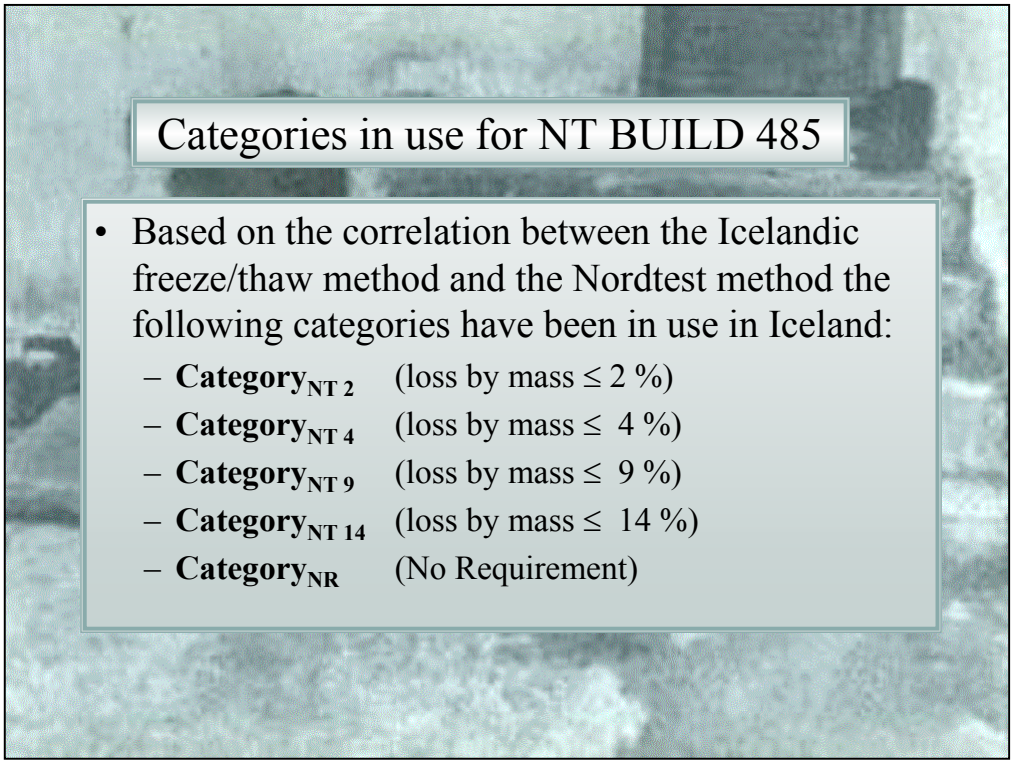


This figure indicates that the Nordtest method is quite repeatable. In fact this correlation is almost too convincing, but the Nordtest I was carried out in 1997 and Nordtest II in 1998. One of the strong reasons for withdrawing the older Icelandic method and replacing it with the Nordtest method was the superior accuracy of the latter one.



Here, test results on various aggregate samples have been collected, but they have been obtained during the last few years. The red lines show the requirement limits for different categories which are in use. Generally speaking, aggregates with less than 4 % loss can be considered quite frost resistant, between 4 and 9 are marginal, 9 to 14 would be considered poor aggregate and above 14 % would hardly be used outdoors, unless as a subgrade material in desolate areas.

The red columns show test results obtained by testing imported aggregates, such as granite, quarzite and limestone. Only the limestone sample shows a result above 2 % loss, but it was considered a rather poor limestone in Sweden. Although we have not tested many aggregates from other sources than basalt, the results indicate that most commonly used aggregates in Europe would survive the Nordtest method. Good results, when using that method, could in the long run become a kind of a quality assurance for such materials.



This table is informative and shows the category limits that are in use in Iceland at present for the Nordtest method

Suggestions...

- To base the new European test for frost resistance in salt water on the existing Nordtest method NT BUILD 485
- To include optional single sized grain sizes of aggregates between 4,0 and 63,0 mm
- To leave open, how the thawing out temperature is controlled (air or water bath)
- To include tolerance between the two individual test values (f.ex. if the difference exceeds X %, two more test specimens should be tested)
- To acknowledge the need for further testing and research for more reliable precision data and for category limits

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Uppkast að frostþolsprófi með saltlausn

May 2002

DRAFT ANNEX B (NORMATIVE) TO EN 1367-1:

Freeze-thaw test with salt water for severe freeze-thaw conditions

Contents

Foreword

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

This test method is based on EN 1367-1, but with fundamental alterations:

3 The samples are tested in a 1% solution of NaCl in de-ionised water.
4 The thawing out sequence is controlled by air 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 in a 1% NaCl solution. 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 and then expressed as a mean % by mass.

1 Scope

This test method can be used to assess the frost resistance of aggregates. It is primarily applicable in areas where frequent freeze-thaw cycling occurs and sea water sprays or de-icers are abundant.

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 a 1% NaCl solution at atmospheric pressure for 24 h, are subjected to 10 freeze-thaw cycles. This involves cooling to -17,5 ºC immersed in the salt solution and then thawing to 20 ºC. After completion of the freeze-thaw cycles, the aggregates are examined for any changes (crack formation, loss in mass and, if appropriate, changes in strength).

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. 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 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 prEN 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 (see table 1), 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 8-16 16-32 32-63	1000 2000 4000[1)] 6000[1)]	500 1000 1500 -
[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.

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 the 1% NaCl solution, the solution covering the test portions by at least 10 mm for the full 24 h period of soaking.

8.2 Exposure to freezing under the NaCl solution

Check that the 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 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.

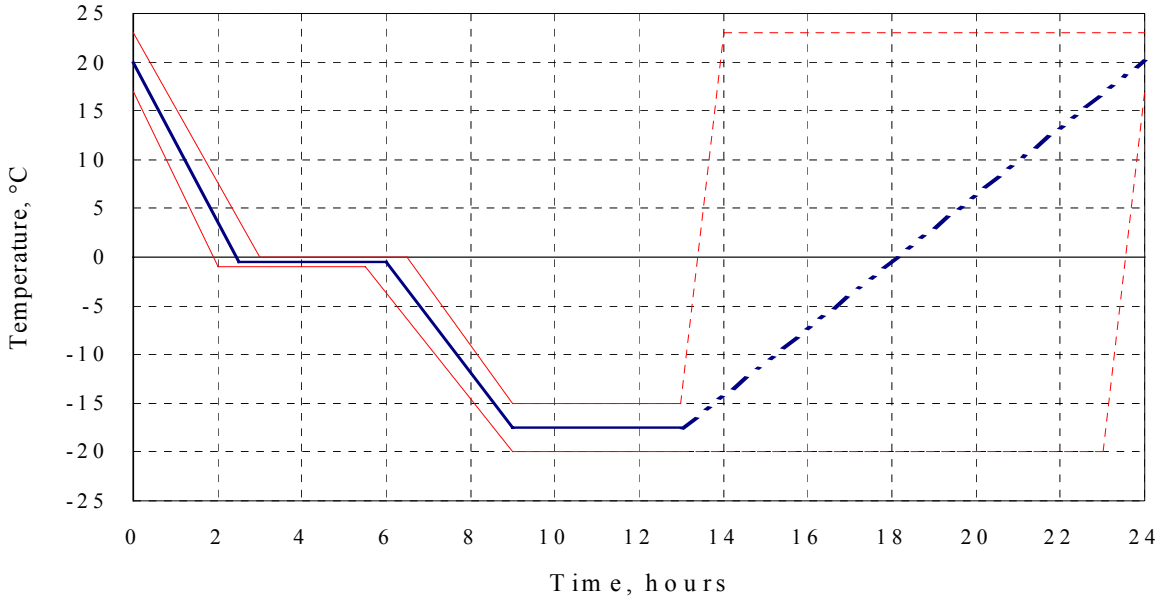

Figure 1: Temperature curve with tolerance limits in the centre of the filled can (reference measuring point) located in the middle of the cabinet

8.2.2 On completion of the tenth cycle, empty each can into a suitable container, and wash thoroughly with de-ionised water to remove the salt solution. 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).

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 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 in 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.