

FREEZE/THAW TEST WITH 1 % NaCl

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1. A BRIEF OVERVIEW.

The Icelandic Aggregate Committee decided in 1985 to develop a new freeze/thaw test method for use in Iceland. The intention was to develop a method which would reflect actual number of freeze/thaw cycles as well as the most frequent minimum temperature. With respect to field measurements, 70 cycles 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 (Aggregates-thermal and weathering properties) to demonstrate that Iceland would require a frost resistance test in salt water as an option to the pure water method (prEN 1367-1), which was under consideration at TG 9. In 1995, 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 replaced the older method. It also became a formal Nordtest standard in 1998 (NT BUILD 485).

Overview:

- The Icelandic Aggregate Committee (IAC) developed a frost resistance test during the years of 1985 to 1990.
- The IAC test involved 70 freeze/thaw cycles, 10 per 24 hours with the temp. range from $+4^{\circ}\text{C}$ to -4°C .
- A strong correlation was established between test results and performance of aggregates in surface dressing.
- The test method was introduced to CEN/TC 154/TG 9 in the early 1990's as a response to prEN 1367-1, which was considered unsuitable for Icelandic aggregates and conditions.
- A Nordic test method was developed in 1995-1996, based on Icelandic experience and with reference to prEN 1367-1.
- The Nordic test method was issued as NT BUILD 485 standard in 1998.

2. DIFFERENCES BETWEEN THE TWO TEST METHODS.

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. This is the main difference between the two methods, but Icelandic research has shown that testing in pure water fails to distinguish between frost resistant and frost susceptible aggregates, while testing in 1 % NaCl does. Another difference is that two parallel specimens of each sample 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. 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 equipment can be avoided. That would enable laboratories in the Nordic countries as well as elsewhere, to easily adopt either method and run the Nordic test or the pure water method when appropriate.

There are other slight differences between the two methods, one of them being that the Nordtest method requires to calculate the % loss of each of the test specimens and then give the final test

result as the mean value, which is not the case in the EN method. By looking at the mean value and comparing it with the partial values, it is immediately observed whether it is likely that mistakes have occurred in the sample preparation or at another stage in the procedure.

Differences between the two freeze/thaw test methods:

NORDTEST:

- 1 % NaCl.
- Two test specimens.
- Thawing phase controlled by air circulation.
- Applicable to all coarse aggregates having a particle size between 8,0 and 16,0 mm.
- No guidance on interruption of test.
- Calculate the loss of mass of each specimen and the mean value as the test result.

EN 1367-1:

- Pure water.
- Three test specimens.
- Thawing phase controlled by immersion in 20°C water bath.
- Applicable to single sized aggregates having a particle size between 4,0 and 63,0 mm.
- Guidance on interruption of test.
- Combine the three specimens before calculating the total loss of mass as the test result.

3. SIMILARITIES BETWEEN THE TWO TEST METHODS.

The Nordtest method was developed and written to be as similar to the pure water method as far as was considered acceptable. *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. *Temperature curve:* Same requirements and tolerance limits (thawing out sequence not specified). *Number of cycles* 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 of the lower limit of fraction.

Similarities between the two freeze/thaw test methods:

- Sample preparation.
- Soaking period.
- Sample containers.
- Sample temperature control.
- Temperature curve.
- Max. and min. Temperature.
- Number of freeze/thaw cycles.
- Duration of test.
- Expression of test results.

4. TEMPERATURE CURVE REQUIREMENTS.

Figure 1 shows two read-outs of temperature curves, as well as the required curve for both freeze/thaw test methods (Nordtest and EN). The so-called German read-out is from a printer connected to a cabinet at the Bundesanstalt für Straßenwesen (BASt) in Germany. This is since 1992, when tests were carried out on Icelandic aggregates with the proposed EN freeze/thaw method at the BASt. The thawing out phase was controlled by water bath circulation. The

Icelandic read-out is a typical one from the cabinet used at the Icelandic Building Research Institute, where air circulation is used to control every phase of the cycle. It is interesting to see that it is possible to control every phase of the test with air circulation (as done in Iceland), not only the freezing phase, but also the thawing out phase.

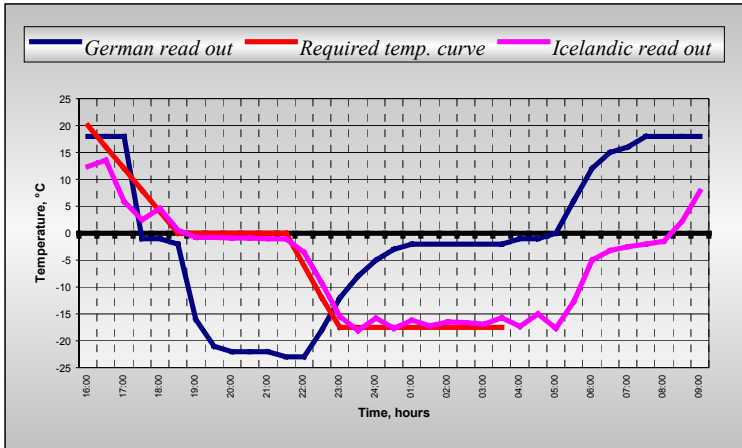


Figure 1 Read-out of temperature curves.

5. ICELANDIC TEST RESULTS.

Figure 2 shows test results on 20 Icelandic basalts with the pure water method (EN 1367-1) on the one hand and the salt water method (NT BUILD 485) on the other hand. It is obvious that the difference is very significant. Long term experience of use of all these materials has been gained in Iceland and it is very important to obtain freeze/thaw values that classify these materials into different frost resistance groups or categories (indicated by the drawn lines on the figure). The pure water method fails to distinguish between frost resistant and frost susceptible aggregates in this case.

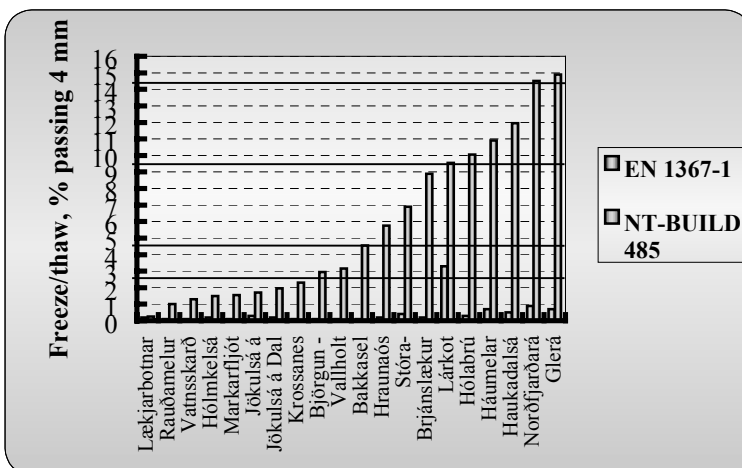


Figure 2 Test results on 20 Icelandic basalts with EN 1367-1 and NT BUILD 485.

Figure 3 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 (dark squares) were therefore valuable to suggest requirements for the Nordtest method by simple reflection and distraction by one unit.

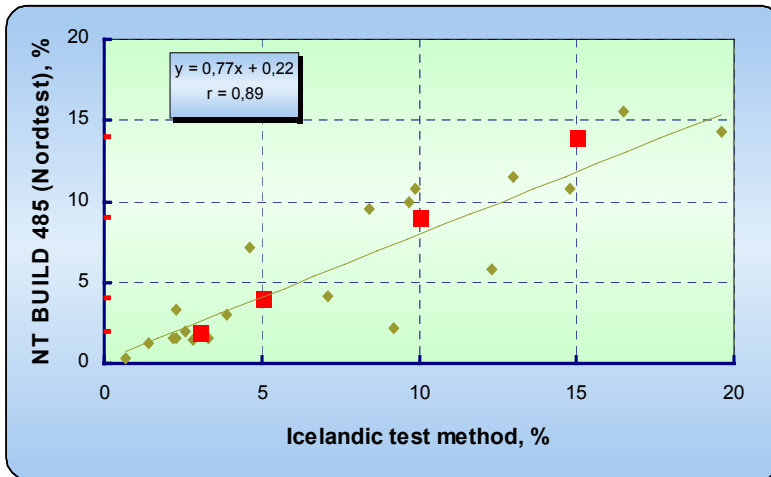


Figure 3 Correlation between the Nordtest results and the Icelandic test results.

Figure 4 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.

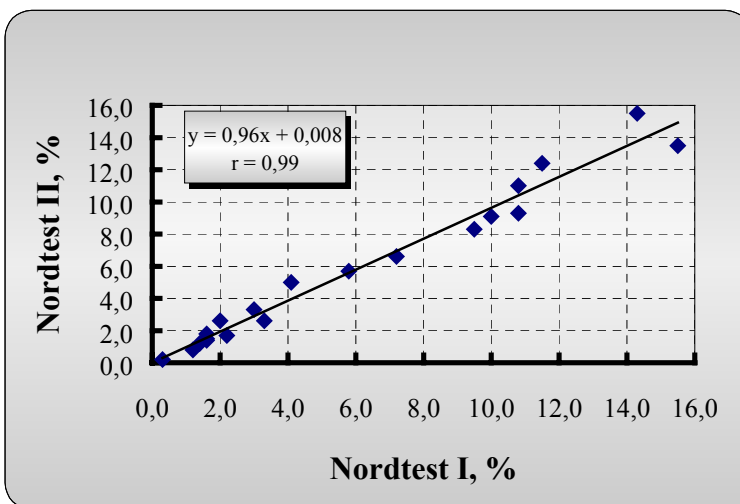


Figure 4 Correlation between two runs with the Nordtest method.

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. Although not many aggregates from other sources than basalt have been tested, it has been indicated that

aggregates with good performance records in Europe would be considered frost resistant on the grounds of the Nordtest method. Good results, when using that method could, in the long run, become a quality assurance for such materials. Further testing and research on European aggregates for more reliable precision data and for category limit is needed to gain European national support for the Nordic test method.

6. CONCLUSIONS.

Based on the correlation between the Icelandic freeze/thaw method and the Nordtest method the following categories have been in use in Iceland for the Nordtest method:

-CategoryNT 2	(loss by mass \leq 2 %)
-CategoryNT 4	(loss by mass \leq 4 %)
-CategoryNT 9	(loss by mass \leq 9 %)
-CategoryNT 14	(loss by mass \leq 14 %)
-CategoryNR	(No Requirement)

7. SUGGESTIONS.

- To base a 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.