Freeze–thaw and deicing salt resistance of concrete testing by the CDF method
CDF resistance limit and evaluation of precision

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RILEM TC 117-FDC decided unanimously that the precision of any freeze-thaw and deicing test procedure must be assessed in accordance with ISO 5725. In addition, a resistance limit for concrete should be approved with respect to practical performance. In this article it is shown that these requirements are fulfilled by the CDF test (capillary suction of deicing chemicals and freeze-thaw test). To verify this, different compositions covering the design rules as defined by standards and given by long-term experience have been measured. The repeatability covering the scatter of the materials and the test procedures was calculated using a large data base of 400 tests. To determine the reproducibility, which includes repeatability and between laboratory scatter, 26 comparison tests between two universities, one internal and two European round robin tests were evaluated additionally. The mean scaling after 28 cycles (14 days) at 1500 g m⁻² proved to be a reliable CDF resistance limit. At this level a coefficient of variation for repeatability has been established at 11% and for reproducibility at 18%. An acceptance criterion may be proposed for further discussion at 1800 g m⁻² as the upper 5% fractile. This minimizes the risk for both customer and supplier.

1. INTRODUCTION

Analysis of national and international standards shows clearly that up to now the provision of freeze-thaw and deicing salt resistance for concrete has been achieved mostly by a 'description concept', i.e., by defining the mix design: the water/cement ratio, the minimum content of entrained air, the type of cement and aggregates and the manufacturing process [1, 2]. Direct testing of freeze-thaw resistance is seldom required in standards since the reliability and reproducibility of test procedures reached to date are rather poor, and approval is difficult with respect to the freeze-thaw and deicing salt durability of concrete in practice.

However, due to the rising demands on concrete and the increasing diversity of raw materials, design concepts lose their applicability. To reach an adequate practical performance and to optimize both the cost of manufacture and the utilization of raw materials, a precise and reliable test procedure is needed urgently.

In an effort to fill this gap a test procedure for freeze-thaw and deicing salt attack has been developed, the CDF test (Capillary suction of Deicing chemical and Freeze-thaw test) [3–7]. Relying on basic research work, the significant parameters have been studied and defined as precisely as necessary to increase the reliability. Operational errors are reduced by simple handling aids. Even untrained personnel can run the test with high precision. Additionally, just as in practice, one defined concrete surface is tested. The essential points are found in Section 3 and in [6] and [7]. To evaluate the results of the test in a manner appropriate to practical needs, a limit must be approved which allows a distinction between resistant and non-resistant concrete: This is called the CDF resistance limit.

The precision of a test procedure is the basis of any acceptance criterion for the quality assessment of a product with minimum risk for both the contractor and the supplier. In the autumn of 1993, a meeting of RILEM technical committee TC 117-FDC 'Freeze-Thaw and Deicing Salt Resistance of Concrete' [8] decided unanimously that the precision of any test procedure in this field must be evaluated following the recommendations of ISO 5725 'Accuracy (trueness and precision) of measurement methods and results' [9]. It is the aim of this article to show the precision and reliability of the test and to define an acceptance criterion following an approval procedure. The results show quite clearly that using this test the reliability of freeze-thaw testing is as high as, for instance, for strength testing or testing of other mechanical properties of concrete.

The basic statistical ideas of ISO 5725 are outlined first, and then the precision and approval of the CDF test are dealt with.

2. STATISTICAL BASIS OF PRECISION FOLLOWING ISO 5725

2.1 Basic considerations

During a test procedure a random sample is picked out of the population and is tested. However, even presumably identical materials in presumably identical circumstances do not yield identical results. The reason
is the unavoidable random errors and variations inherent in every test procedure and material, and certainly this is true for concrete.

Various types of measurement error will overlap each other. The systematic error can be controlled: it may be minimized and compensated for using adjustment tables. It is more difficult to separate random errors from the real varieties of various specimens of the same type. In destructive testing this can be reached only approximately. A basis for an assessment of test procedures is ISO 5725, in which the term ‘precision’ refers to the closeness of agreement between test results. The different factors influencing the variability of the results observed are outlined here.

Besides the test procedure the material itself varies. The scatter of data from the two sources overlap, and cannot be measured separately. In a test procedure appropriate for a quality control system the variability of the test procedure should be smaller than the variability of the controlled material, since otherwise the cost of production is increased simply by the inadequate test procedure. On the other hand, in order to assess the precision of a test the opposite is required, i.e., the variability of the material should be inferior. Therefore, to evaluate the precision of a test method major efforts are necessary to achieve specimens which are as homogeneous as possible and to assure almost identical sampling. Selection of materials proves to be the greatest problem in the assessment of precision. This applies especially for concrete, which is inherently inhogeneous and where testing is destructive. The precision is as a rule determined in as many different laboratories as possible by round robin tests, as well as within one laboratory by extensive tests with one or more pieces of test equipment. In a round robin test large quantities of uniform specimens are necessary. The manufacture, transport and storage of the specimens influence the test surface. Even if handled with special care the variability of materials increases significantly with respect to testing in one laboratory, as shown by experience of many round robin projects.

2.2 Statistical analysis by ISO 5725

2.2.1 Definitions

Following ISO 5725 the precision of a test procedure can be described by two aspects.

2.2.1.1 Repeatability. Repeatability refers to measurements which are made under repeated but as constant as possible boundary conditions, i.e., ‘where independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time’ ([9] part 1, page 6). With repeatability one achieves, generally speaking, the minimum of variability. Repeatability derives from the materials variability and the test procedure variability. These cannot be separated by experiment. However, if materials variability can be kept small enough, the variability of the test procedure dominates.

2.2.1.2 Reproducibility. Reproducibility refers to measurements which are made under widely varying conditions, i.e., ‘where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment’ ([9] part 1, page 6). By reproducibility the maximum scatter is found.

2.2.1.3 Between laboratory variability. Reproducibility consists of repeatability and the variation between laboratories. Therefore the between laboratory variability can be calculated from the repeatability and reproducibility.

The repeatability value \( r \) (and the reproducibility value \( R \)) defines the level below which the absolute difference between two single test results is found with a confidence level of 95% certainty and obtained under repeatability (and reproducibility) conditions.

\[
r = 2\sqrt{2\sqrt{s^2}}
\]

hence

\[
r = 2.8s
\]

2.2.2 Statistical measures

The precision, i.e., repeatability and reproducibility, is determined as follows. The standard deviation \( s \), variance \( s^2 \) and coefficient of variation \( v \) of both repeatability and reproducibility are defined in the usual statistical way. The total variance \( s^2 \) can be calculated from the variances \( s^2 \) of the factors contributing to it:

\[
s^2 = \sum s^2_i
\]

The total standard deviation \( s \) is the square root and the total coefficient of variation \( v \), involves the mean value \( \bar{x} \) of the variable:

\[
v = \sqrt{\frac{\sum s^2_i}{\bar{x}}}
\]

2.2.3 Principal steps of the statistical analysis of round robin tests according to ISO 5725

ISO 5725 uses the following model for the calculation. Each single test result is assumed to be the sum of the general average \( m \), the between laboratory variance \( B \) and the random error \( e \) occurring in every test:

\[
y = m + B + e
\]

The between laboratory variance \( B \) includes the between operators and between equipment variability. Under repeatability conditions the scatter is considered as