

STUDY ON ANALYTICAL TECHNIQUES FOR IMPROVING
THE CHEMICAL METHOD

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ABSTRACT: ASTM C 289, Standard Test Method for Potential Reactivity of Aggregates (Chemical Method), has been widely used as a method of evaluating alkali reactivity of aggregates for concrete since it was first approved as a Tentative Method in 1952. This method has the merits that test results can be obtained quickly and that testing can be done using a small sample of aggregates. However, a problem with this method is that there is scatter of test results. It has often been experienced that when the same aggregate samples were tested, the results obtained at different laboratories did not match each other.

The authors have devised a method modifying a number of parts of ASTM C 289 with the purpose of improving the testing precision. Tests were performed at pairs of laboratories using 371 varieties of aggregate samples, and as a result of investigating errors in testing, it was found that the accuracy had been improved. This study was made as a part of a comprehensive technology project of the Ministry of construction, and the method proposed herein is being used as a tentative method of the Ministry of Construction. It has also been incorporated in JIS A 5308-1986. [1]

1. INTRODUCTION

The authors, in 1984, conducted a joint test of ASTM C 289 -81, Standard Test Method for Potential Reactivity of Aggregates

(Chemical Method), distributing aggregates of the same varieties to laboratories at five places. An example of test results obtained then is given in Table 1.[2]

Table 1. Example of results on joint tests according to ASTM C 289-81

Samples of aggregate	Rc mmol/l			Sc mmol/l		
	Max.	Min.	Standard devi.	Max.	Min.	Standard devi.
Pyroxene andesite	175,	108	29	598,	424	75
Bronzite andesite	232,	171	26	756,	597	68
Pyroxene andesite	162,	60	44	534,	303	99

As this table shows, the scatter in test results with this method was fairly great. Factors to increase scatter in test results may be broadly divided into the three below.

- (1) The non-uniformity of rock composition of aggregates
- (2) Deviations in rock compositions produced in sample preparation
- (3) Testing errors occurring in chemical analyses

Regarding (1) of these factors, it is a difficult matter to obtain uniformity of composition since aggregates are in lump form. With regard to (2), it can be expected that scatter can be reduced to a certain extent. As for (3), the most effective improvement can be looked forward to through standardization in detail of analyzing operations.

On reviewing ASTM C 289, the authors became aware that there were parts in which descriptions were not detailed. Therefore, the respective procedures were defined in detail as much as possible. Furthermore, changes to methods thought to be better were made on procedures in a number of parts.

In proposing it, this improved testing method was called the Tentative method of Ministry of Construction.

2. IMPROVEMENTS CONTAINED IN THE TENTATIVE METHOD
OF MINISTRY OF CONSTRUCTION

The Tentative Method of Ministry of Construction is essentially based on ASTM C 289-81. This method differs from ASTM C 289 regarding the following parts:

- (1) The method of sampling was specified in as much detail as possible. The procedure of this is shown in Fig.1.

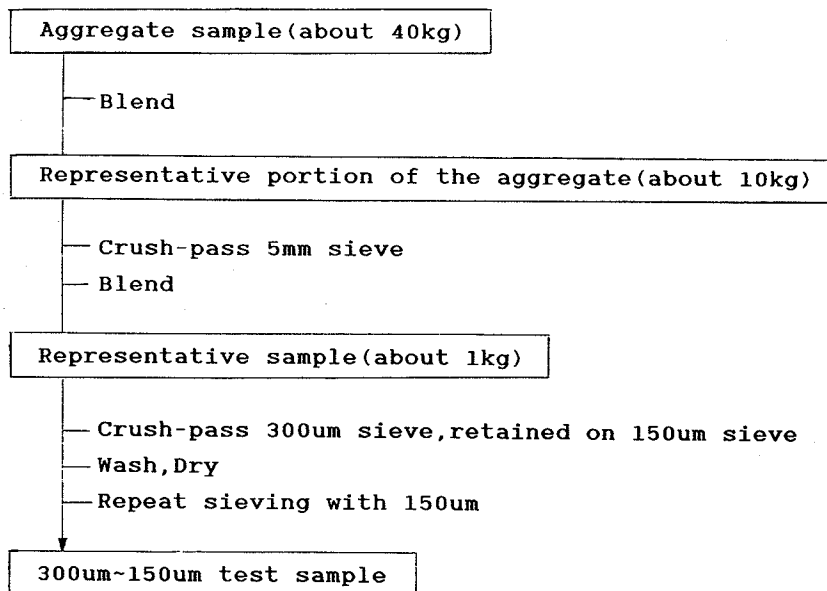


Fig.1 Preparation method of aggregate sample

- (2) Parts of the analysis procedure were modified as follows:

- (a) After the reaction to occur at 80 °C for 24 hours, cooling is done for 15 minutes with running tap water. There will be condensed drops of water adhering to the inner surface of the reaction container and to completely return these water drops to the sample solution, the container is to be rotated twice as shown in Fig.2 before removing the container cover.
- (b) In filtering operations the vacuuming conditions are specified in ASTM in terms of vacuuming pressure, but the Tentative Method specifies in terms of time: 4 minutes of vacuuming.

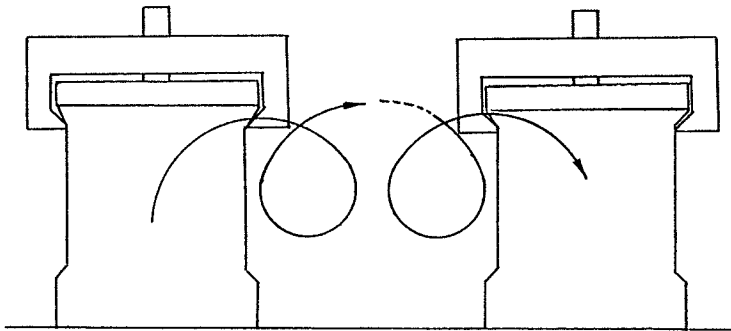


Fig.2 Recovery operation of condensed water

(c) With the objective of accurately performing titration of reduction in alkalinity (R_c), preliminary titration is first done with 0.05N HCl, and using this titration value as reference, a second titration is to be done, and the value from the second titration is to be taken as the proper titration value.

(d) For analysis of dissolved silica (S_c), an atomic absorption method was adopted in addition to the gravimetric method and the photometric method.

In the atomic absorption method, part of the same sample solution as that used in measuring reduction in alkalinity (R_c) is used. A high temperature burner with an acetylene-nitrous oxide flame is to be used for measurement. [3]

An improvement was also made in the gravimetric method. The evaporation-dryness method using HCl is adopted in ASTM C 289, but instead of this a fuming method employing perchloric acid is used.

The photometric method will not be discussed here as it was not touched.

(e) Regarding repeatabilities of measurement of reduction in alkalinity and dissolved silica, the requirements were made difference not more than 10 millimoles per liter when the average is 100 millimoles per liter or less, and not more than 10 percent when the average is higher than 100 millimoles per liter, with retesting to be done when this is exceeded. This is a severer requirement than

presently in ASTM C 289.

3. RELIABILITY ON THE TENTATIVE METHOD
OF MINISTRY OF CONSTRUCTION

The authors conducted tests of the same samples at pairs of laboratories with the purpose of verifying the reliability of this test method, and the errors between the pairs were investigated. Five laboratories cooperated in the joint tests, and 371 varieties of samples were used.

As a result, regarding errors between pairs of laboratories for both R_c and S_c , or for either of the two, there were 59 varieties (16 percent) of samples that differed more than 10 percent from the average of the pair of laboratories, with 312 varieties (84 percent) of samples within 10 percent for both R_c and S_c . In particular, 157 varieties (42 percent) among these were within 5 percent for both R_c and S_c , and the measured values of the laboratories pairs were in agreement. The improvement not only achieved the purpose of enhancing the precision of testing, but also as a result of having adopted the atomic absorption method for measuring S_c , there was the advantage that a large number of samples could be handled in a short period of time.

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