Determining Cement Content of Soil-Cement by Heat of Neutralization

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A heat of neutralization test method (as modified by the Bureau of Reclamation) for determining cement content of freshly mixed soil-cement is discussed. Once a calibration curve is established, a cement content determination can be made in approximately 15 min, which in field applications has been found to be accurate to within ±1 percentage point of actual cement content. A sodium acetate–glacial acetic acid buffer solution is used to initiate an exothermic reaction with the calcium hydroxide in the soil-cement test specimen. During construction, test specimens are obtained directly from the spreader as soil-cement is placed, and separation of plus No. 4 (4.75-mm) sieve size material is not required. Results from an interlaboratory testing program were analyzed to establish precision and bias of the suggested test method.

In 1988, the Bureau of Reclamation was placing soil-cement for slope stabilization at Jackson Lake Dam, Wyoming. Because of gradation characteristics of borrow material, a soil-cement mix design having 50 percent plus No. 4 (4.75-mm) sieve size material was the most economical mixture. There was concern that typical titration methods for determining cement content of soil-cement for construction control would be difficult to use and too slow to perform. Titration methods typically require separating the plus No. 4 (4.75-mm) sieve size material and include the time-consuming task of screening and washing the soil-cement mix to obtain test specimens of appropriate size and gradation characteristics. This circumstance prompted initiation of a program to develop a test method for determining cement content of soil-cement with the following objectives: (a) separation of plus No. 4 (4.75-mm) sieve size material not required for obtaining test specimens; (b) cement content determinations in a timely manner, i.e., within 15 to 20 min; (c) durable apparatus for field use; (d) ease of performing test method by field personnel; and (e) accuracy of ±1 percentage point of actual cement content for soil-cement mix designs having cement contents of 3 to 15 percent and up to 50 percent plus No. 4 (4.75-mm) sieve size material.

TEST METHOD Q116B–1978

Test methods used for determining cement content of soil-cement mixtures were reviewed and evaluated against the five requirements. Test Method Q116B–1978, *Cement Content of Cement Treated Materials (Heat of Neutralization)* (*1*), was the test method that came closest to satisfying the five requirements.

In the test method, an acidic buffer solution of glacial acetic acid and sodium acetate with an initial pH of approximately 2 is used to react with the calcium hydroxide in a soil-cement test specimen. This reaction is exothermic and the heat produced is proportional to the quantity of cement present in the test specimen. The nature of this proportionality is linear, which makes it conducive to establishing a calibration curve and determining a corresponding line equation for a given material. Soil-cement test specimens of unknown cement contents can then be tested by obtaining the heat of neutralization, and the cement content can be determined from the calibration curve. Heat of neutralization is defined as the temperature increase resulting from the exothermic reaction in the acidic buffer solution when mixed with a soil-cement test specimen.

The Australian test method specified that a 5-kg soil-cement test specimen be mixed with 1 L of buffer solution consisting of 150 g of sodium acetate, 240 g of glacial acetic acid, and distilled water. A calibration curve is prepared using seven different percentages of cement contents that bracket the cement content specified in the mix design, i.e., if the target figure is 2 percent cement, percentages of cement of 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 are used. Calibration test specimens are prepared at the water content to be used during soil-cement placement. Duplicate heat of neutralization tests are performed at each of the seven cement contents. Temperature increase corresponding to each of the 14 tests is plotted versus cement content and a calibration line is established. Subsequent soil-cement test specimens of unknown cement contents are tested, the heat increase obtained is evaluated using calibration data, and the cement content is determined.

RECLAMATION PROCEDURAL CHANGES

Test Method Q116B–1978 was originally developed for soil-cement mix designs having cement contents of 3.5 percent or less. At cement contents greater than 3.5 percent, the mixture of 1 L of buffer solution with a 5-kg test specimen gels into a solid mass, preventing proper mixing. Recommendations by the Main Roads Department for cement contents greater than 3.5 percent were (a) add additional buffer solution, up to 3 L, to the 5-kg soil-cement test specimen; (b) adjust the mass ratio of buffer solution to test specimen from 1 L to 5 kg for cement contents less than 3.5 percent, to 2.5 L to 4 kg for cement contents between 6.0 and 7.5 percent. It was also stated that any required reduction of test specimen mass should

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be kept to a minimum and under no circumstances should the soil-cement test specimen be less than 4 kg.

After performing a series of tests, the Bureau of Reclamation made the following changes to the original Australian heat of neutralization test method: (a) reduce the number of cement contents required for obtaining a calibration curve from seven to three; (b) perform three heat of neutralization tests at each of the three cement content values selected, to construct a calibration curve using 9 data points, reduced from 14; and (c) use 1.5 L of buffer solution mixed with a 1.5-kg soil-cement test specimen for mix designs consisting of up to 15 percent cement and 50 percent plus No. 4 (4.75-mm) sieve size material.

INTERLABORATORY TEST PROGRAM

After the changes were incorporated into the test method, an interlaboratory test program was performed in accordance with ASTM C802-87, *Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials.* This test program was performed to validate the procedural changes outlined, solicit user comments on the revised heat of neutralization test procedure, and provide reliable information from which precision statements of the type prescribed in ASTM C670-88, *Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials*, could be developed.

The interlaboratory test program was performed on two soil-cement mix designs in two separate phases. Phase I consisted of testing soil-cement specimens of a fine-grained mix design and Phase II used a coarse-grained mix design. Fine- and coarse-grained mix designs were selected to determine if the changes made to the test method were effective both on a fine-grained mix and on one in which a significant percentage of plus No. 4 (4.75-mm) sieve size material was present. The test method changes investigated were (a) reducing the number of cement contents and calibration test specimens required to construct a calibration curve, and (b) reducing the test specimen size from 5 to 1.5 kg.

Testing laboratories from federal, state, and private sectors participated in the test program. Ten testing laboratories participated in Phase I of the test program and eight testing laboratories participated in Phase II. Participating laboratories were provided with a written procedure and individual test specimens of appropriate soil mass, each with a corresponding amount of cement. Each laboratory was required to thoroughly mix the soil, cement, and water, as specified, for each test specimen before performing the heat of neutralization test. A moisture content of 8 percent was specified for each test specimen.

Phase I

For the first phase of the test program, each participating laboratory performed a total of 15 cement content determinations on soil-cement test specimens. The specimens consisted of 5 percent minus ⅜-in. and 95 percent minus No. 4 (4.75-mm) sieve size material. Nine determinations were performed on test specimens of known cement contents—three at 5, three at 7, and three at 9 percent cement—to develop a calibration curve and determine a calibration line equation. The remaining six cement content determinations were performed on duplicate test specimens designated for each laboratory as Test Specimens A, B, and C, but of cement content unknown to the participating laboratories. Test Specimens A were prepared at 6 percent cement, B at 7 percent cement, and C at 8 percent cement.

Phase II

The purpose of the second phase of the interlaboratory test program was to determine the effect that a significant percentage of plus No. 4 (4.75-mm) sieve size material would have on the reliability of the revised heat of neutralization procedure in determining cement content.

Phase II of the test program consisted of the same number of test specimens at the same cement contents as in Phase I. The gradation of Phase II test specimens was as follows:

Material Size	*Percent Retained*
¾ to 1½ in.	14
⅜ to ¾ in.	20
No. 4 to ⅜ in.	16
Minus No. 4	50

Calibration Test Specimen Results

Results obtained from Phase I calibration test specimens (5 percent plus No. 4) are shown in Figure 1. Statistical analysis performed on data from the 90 heat of neutralization tests, 30 at each of the three cement contents, resulted in a correlation coefficient of 0.981 with a corresponding R^2 value of 96 percent. The solid line shown in Figure 1 represents the best-fit line obtained performing a linear regression through all data points. The narrow set of dashed lines indicates the 95 percent confidence limit within which the average value of all data points would fall. The wider set of dashed lines indicates the 95 percent confidence limit within which any data point would fall.

Results obtained from Phase II calibration test specimens (50 percent plus No. 4) are shown in Figure 2. Statistical analysis performed on data from the 72 heat of neutralization tests, 24 at each of the three cement contents, resulted in a correlation coefficient of 0.974 with a corresponding R^2 value of 95 percent. The solid, narrow, and wide sets of dashed lines represent the best-fit line, 95 percent confidence limits for average data points, and 95 percent confidence limits for any data point, respectively, as described for Phase I calibration test specimen results.

Tables 1 and 2 present summaries of Phase I and Phase II calibration test specimen results, respectively. Average, maximum, and minimum values of temperature increase in degrees Celsius, as well as calculated within- and between-laboratory standard deviations, are provided.

Test Specimen Results

Interlaboratory test program results for Test Specimens A, B, and C (5 percent plus No. 4) and Test Specimens D, E,

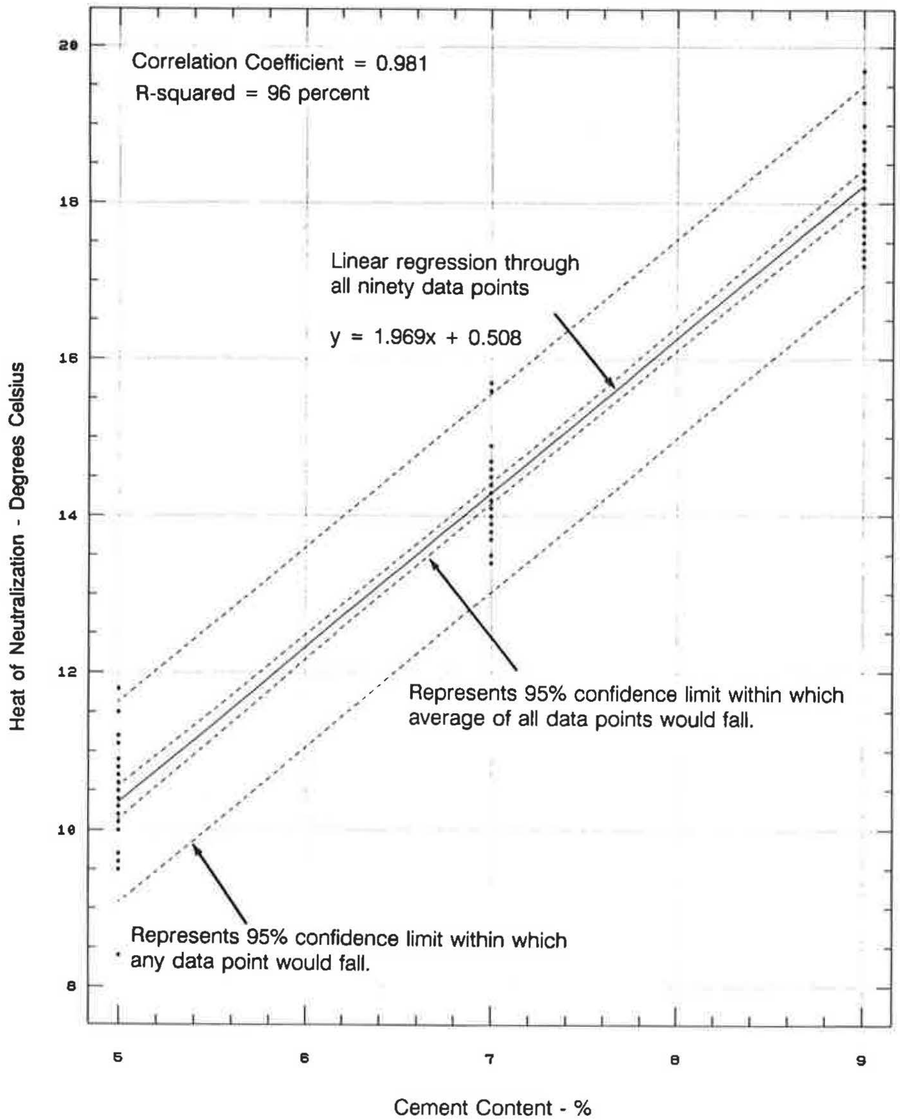

FIGURE 1 Phase I calibration test specimens.

and F (50 percent plus No. 4) are presented in Tables 3 and 4, respectively. Average, maximum, and minimum values of cement content, as well as calculated within- and between-laboratory standard deviations, are provided.

Precision and Bias Statements

Results obtained from the interlaboratory test program were used to develop precision and bias statements as prescribed in ASTM C670–88 for the heat of neutralization test procedure as revised by the Bureau of Reclamation. Table 5 presents a summary of single-operator and multilaboratory precision and bias values obtained for the calibration test specimens. Values shown in Rows 2, 3, and 5 of Table 5 are calculated in accordance with ASTM C670–88 and are in degrees Celsius.

Table 6 presents a summary of single-operator and multilaboratory precision and bias values obtained for Test Specimens A, B, C (5 percent plus No. 4) and Test Specimens D, E, F (50 percent plus No. 4). Values shown in Rows 2, 3, and 5 of Table 6 are calculated in accordance with ASTM C670–88 and are in percentage points of cement content.

From the single-operator and multilaboratory precision and bias values presented in Tables 5 and 6, precision statements can be written for specimens containing from 3 to 15 percent cement and up to 50 percent plus No. 4 (4.75-mm) sieve size material as described in the following section.

CALIBRATION TEST SPECIMENS

Single-Operator Precision

The single-operator standard deviation of a single test result (a test result defined in this procedure as the average of three separate measurements) has been found to be 0.5°C. Therefore, results of two properly conducted tests by the same operator (each consisting of the average of three calibration test specimens of the same cement content) should not differ

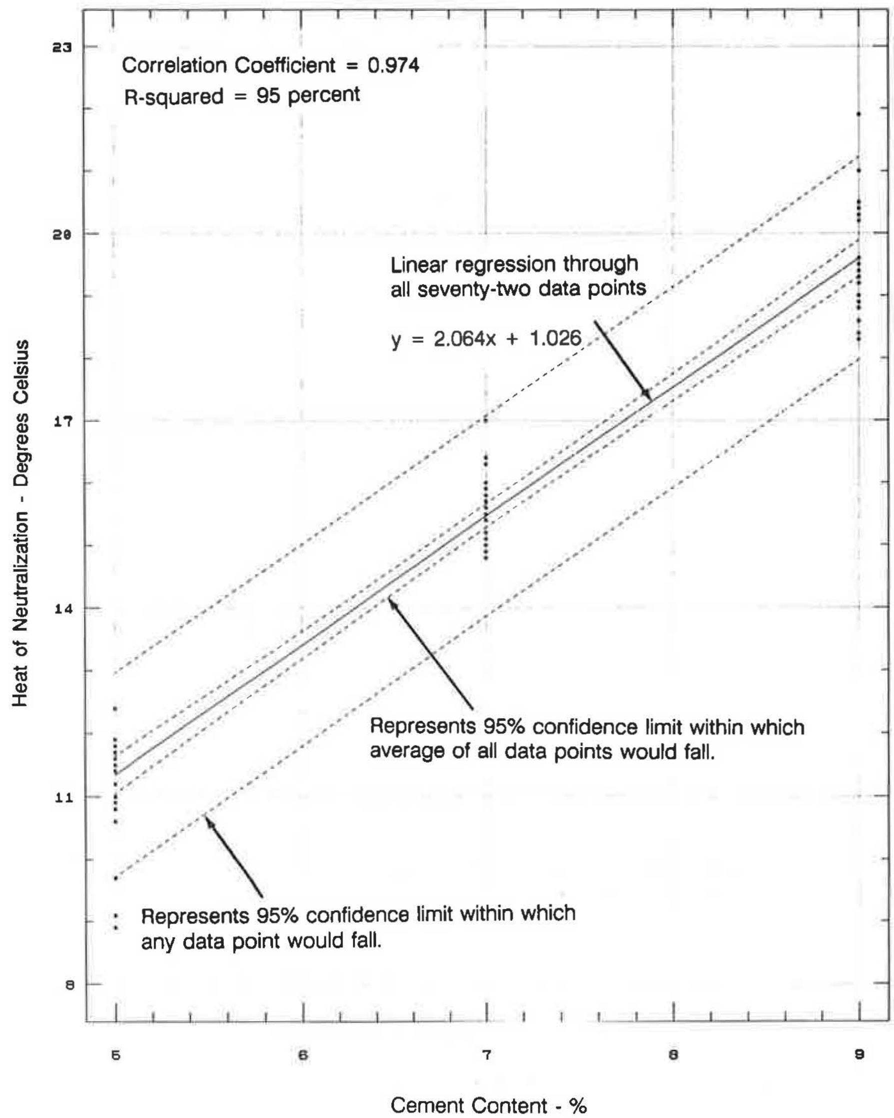

FIGURE 2 Phase II calibration test specimens.

TABLE 1 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR 5 PERCENT PLUS NO. 4 CALIBRATION TEST SPECIMENS

	Temperature Rise				
Cement Content %	Average °C	Maximum °C	Minimum °C	Standard Deviation within-lab	Standard Deviation between-lab
5	10.4	11.8	8.4	0.512	0.659
7	14.3	15.7	13.4	0.321	0.613
9	18.2	19.7	17.2	0.338	0.684
			Average:	0.390	0.652

TABLE 2 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR 50 PERCENT PLUS NO. 4 CALIBRATION TEST SPECIMENS

	Temperature Rise				
Cement Content %	Average °C	Maximum °C	Minimum °C	Standard Deviation within-lab	Standard Deviation between-lab
5	11.2	12.4	8.9	0.573	0.912
7	15.7	17.0	14.8	0.423	0.603
9	19.5	21.9	18.3	0.585	0.879
			Average:	0.527	0.798

TABLE 3 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR 5 PERCENT PLUS NO. 4 TEST SPECIMENS A, B, AND C

Test Specimen	Cement Content Average %	Maximum %	Minimum %	Standard Deviation within-lab	between-lab
A	6.0	6.4	5.7	0.164	0.232
B	7.1	7.5	6.4	0.130	0.313
C	8.2	8.6	7.7	0.130	0.235
			Average:	0.141	0.260

TABLE 4 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR 50 PERCENT PLUS NO. 4 TEST SPECIMENS D, E, AND F

Test Specimen	Cement Content Average %	Maximum %	Minimum %	Standard Deviation within-lab	between-lab
D	5.9	6.5	5.3	0.095	0.298
E	7.1	7.4	6.5	0.167	0.270
F	8.0	8.5	7.8	0.126	0.173
			Average:	0.129	0.247

TABLE 5 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR PRECISION DETERMINATION OF CALIBRATION TEST SPECIMENS

		Phase I 5% plus No. 4	**Phase II 50% plus No. 4**
Single-Operator	**Standard deviation of a single test result [1]**	**0.39 [4]**	**0.53**
	Maximum allowable difference between two test results [2]	**1.1**	**1.5**
	Maximum allowable difference between the highest and lowest individual temperature determinations [3]	**2.2**	**3.0**
Multilaboratory	**Standard deviation of a single test result [1]**	**0.65**	**0.80**
	Maximum allowable difference between two test results from different laboratories	**1.8**	**2.3**

[1] A test result is defined as the average of three separate measurements

[2] Each consisting of the average of three calibration test specimens at the same cement content

[3] The three individual temperature determinations used in calculating a test result

[4] Numbers in table are in degrees Celsius

TABLE 6 SUMMARY OF INTERLABORATORY TEST PROGRAM FOR PRECISION DETERMINATION OF TEST SPECIMENS

		Phase I - 5% plus No. 4 Test Specimens A, B, and C	**Phase II - 50% plus No. 4 Test Specimen D, E, and F**
Single Operator	**Standard deviation of a single test result [1]**	**0.14 [4]**	**0.13**
	Maximum allowable difference between two test results [2]	**0.40**	**0.37**
	Maximum allowable difference between the highest and lowest cement content determinations [3]	**0.55**	**0.51**
Multilaboratory	**Standard deviation of a single test result [1]**	**0.26**	**0.25**
	Maximum allowable difference between two test results from different laboratories	**0.74**	**0.71**

[1] A test result is defined as the average of two separate measurements

[2] Each consisting of the average of two cement content determinations

[3] The two cement content determinations used in calculating a test result

[4] Numbers in table are in percentage points of cement content

by more than 1.5°C. The range (difference between the highest and lowest) of the three individual temperatures used in calculating a test result should not exceed 3.0°C.

Multilaboratory Precision

The multilaboratory standard deviation of a single test result (a test result is defined in this procedure as the average of three separate measurements) has been found to be 0.8°C. Therefore, results of two properly conducted tests in different laboratories on the same soil-cement mix should not differ by more than 2.3°C.

TEST SPECIMENS

Single-Operator Precision

The single-operator standard deviation of a single test result (a test result is defined in this procedure as the average of two separate measurements) has been found to be 0.14 of a percentage point of cement content. Therefore, results of two properly conducted tests by the same operator (each consisting of the average of two cement content determinations) should not exceed 0.40 percentage points of cement content. The range (difference between the highest and the lowest) of the two individual cement content determinations used in calculating a test result should not exceed 0.55 percentage point of cement content.

Multilaboratory Precision

The multilaboratory standard deviation of a single test result (a test result is defined in this procedure as the average of two separate measurements) has been found to be 0.26 of a percentage point of cement content. Therefore, results of two properly conducted tests in different laboratories on the same soil-cement mix should not differ by more than 0.74 percentage point of cement content.

CONCLUSIONS

Results of the interlaboratory test program indicate that changes made to Test Method Q116B–1978 by The Bureau of Reclamation produce single-operator and multilaboratory precision values that are well within acceptable limits for producing a high-quality end product. The Bureau of Reclamation's revised heat of neutralization test method can be used to accurately and quickly determine the cement content of freshly mixed soil-cement containing from 3 to 15 percent cement and up to 50 percent plus No. 4 (4.75-mm) sieve size material. The test method does not require separation of the plus No. 4 (4.75-mm) sieve size material for obtaining test specimens, and cement content determinations can be made in 15 to 20 min. Required apparatus is durable for field use, the test procedure is easily performed by field personnel, and in field applications performed to date the method is accurate to within ±1 percentage point of actual cement content.

A written test procedure has been prepared for use on Bureau of Reclamation projects. The written test method will soon be submitted to ASTM for approval and publication through its normal consensus standards procedure.

REFERENCE

1. *Cement Content of Cement Treated Materials (Heat of Neutralization).* Test Method Q116B–1978, Main Roads Department, Queensland, Brisbane, Australia, 1978.

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