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Short-Time Tests of Mortars for Controlling S03 in Portland Cement at Optimum Value

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16. ABSTRACT

A cooperative test program was conducted by the Working Committee on SO3 content during 1957. The objective of the program was to determine if short-time tests for (1) compressive strength and (2) expansion in water and contraction in air of Ottawa sand mortars could be used satisfactorily as a means of determining the relationship of the SO3 content of cement to its optimum value.

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A co-operative test program was conducted by the Working Committee on SO3 content during 1957. The objective of the program was to determine if short-time tests for (1) compressive strength and (2) expansion in water and contraction in air of Ottawa sand mortars could be used satisfactorily as a means of determining the relationship of the SO3 content of cement to its optimum value.

Laboratories participating in the test program were as follows:

California Division of Highways Sacramento, California	Bailey Tremper
Ideal Cement Company Fort Collins, Colorado	K. E. Palmer
Medusa Portland Cement Company Wampum, Pennsylvania	J. F. Weigel
Portland Cement Association Chicago, Illinois	William Lerch
Universal Atlas Cement Company Buffington, Indiana	W. C. Hansen

The order of listing these participants is not the same as the order in the presentation of data.

Four portland cements were prepared and shipped to the laboratories. There were two Type III, one Type I, and one Type II cement. The cements were in the following classifications as to chemical composition:

Cement No. 1	Type III	High C_3A, low alkali
Cement No. 2	Type III	Low C_3A, low alkali
Cement No. 3	Type I	High C_3A, high alkali
Cement No. 4	Type II	Low C_3A, high alkali

The chemical analysis and specific surface of these cements are given in Table I. The cements were selected on the basis of preliminary tests to have SO_3 contents below optimum. Tests made in this study indicate that the cements were deficient in SO_3 by 0.40 to 2.15 per cent relative to optimum.

Basis of Test Program

It has been found that in mortars containing portland cements that are relatively low in SO3, progressive additions of gypsum to the cement produce decreasing contraction in air until a minimum contraction is reached. Further additions of gypsum produce increasing contraction. Similarly, additions of gypsum increase the strength of mortars up to a maximum value and further additions produce decreasing strength. The percentage of SO3 that results in the least contraction or highest strength in a given cement is termed the optimum SO3 content.

Curves representing the relationship between SO3 content and contraction or strength vary somewhat in shape between cements but in the region of optimum SO3 the variation in shape is not great. Figures 1 to 4 illustrate the relationships found with the cements under study. The change in magnitude of contraction or strength produced by a suitable, known addition of gypsum provides information on the relationship of the SO3 in the original cement to its optimum value.

When the SO3 content is increased to about 0.75 per cent above its optimum value, significant increases in the expansion in water result.

The plan of the test program was based on the relationships shown above. The methods require the testing of specimens made from mortars containing the original cement and the same cement blended with pulverized gypsum in an amount to increase

the SO_3 content by 0.5 percentage points. Results of the tests are known after 24 hours for strength, 3 days for expansion and 7 days for contraction.

The utility of these tests as a manufacturing guide or as information to the consumer depends on the precision with which they can be made. A study of precision, repeatability and reproducibility, was the main objective of the co-operative program.

General Scope of Tests

Preliminary information indicated that measurable changes in the physical properties of mortars could be obtained by varying the SO_3 content by increments of 0.5 per cent. Therefore, pulverized gypsum was added and blended with the cements to provide increments at this interval. Gypsum having the properties shown in Table I was used by all laboratories.

In order to evaluate the results of the tests it was necessary to establish good values of optimum SO_3 content of each cement. For this reason the cements were blended with gypsum to provide five increments of SO_3 in the range of 1 per cent below to 1 per cent above optimum. Cement 4 however, was received with an SO_3 content of about 0.5 per cent below optimum. The range for this cement extended from this value to 1.5 per cent above optimum.

Detailed Scope of Tests

The complete schedule of tests included the following:

Expansion and Contraction

Expansion in water for 48 hours followed by contraction in air for 4 days. One round consisted of four 1 by 1 by 10-inch gage length bars molded from a batch of 1:2 (by weight) graded Ottawa sand mortar mixed as specified in ASTM C 109. Bars were removed from the molds after 24 hours moist storage at which time the initial measurement of length was made. The bars were then stored in water at 74.3 ± 3F. for 48 hours after which they were again measured for length and the percentage of expansion was computed. The length at the conclusion of the expansion test was taken as the initial length for the contraction test. The bars were stored in a specially designed cabinet described in the appendix. The cabinet was installed in a room maintained at a temperature between 68 and 81.5 F. Relative humidity within the cabinet was of the order of 50 per cent at equilibrium.

The number of rounds made with each cement was as follows:

	Increments of SO_3						
	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement 1			5	5	3	3	3
Cement 2		5	5	3	3	3	
Cement 3	5	5	3	3	3		
Cement 4	5	5	3	3	3		

Except for Cement 4, this schedule provided increments in SO3 content from about minus 1 per cent to plus 1 per cent relative to optimum.

Compressive Strength

Compressive strength by ASTM C 109 using the six-cube batch. Tests were made at 1 and 3 days on Type III cements and at 1 and 7 days on Type I and Type II cements. Compressive strength results at 3 and 7 days are not presented in this report because they were found to be less indicative of optimum SO3 content than were the 24-hour strengths. Three rounds were made at each of the increments of SO3 indicated under Expansion and Contraction.

Calcium Sulfate in Hydrated Cement Mortar

These tests were made in accordance with ASTM C 265-57T, except that tests were made at 18 hours as well as the specified 24 hours.

Single rounds were made with increments of SO3 as follows:

	Increments of SO3						
	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement 1			X		X		X
Cement 2		X		X		X	
Cement 3	X		X		X		
Cement 4	X		X		X		

Laboratories 2 and 3 completed the entire schedule of tests. Laboratory 1 also completed the schedule but used a controlled room in place of the special cabinet for the contraction tests. Laboratory 4 completed two rounds of the expansion-contraction and compressive strength tests. Laboratory 5 varied the designated procedure in a number of ways. The results from this laboratory were used in establishing the best values of optimum SO3 content but they could not be pooled with the others in computations of the precision of the tests.

The SO3 content of each cement was reported by the supplier. Blends containing additional quantities of SO3 were calculated by the following formula:

$$X = \frac{A - B}{C - B} \times 100 \quad \text{and} \quad Y = 100 - X$$

Where X = the per cent of gypsum in the blend

Y = the per cent of cement in the blend

A = the desired SO3 content of the blend in per cent

B = the SO3 content of the cement in per cent

C = the SO3 content of the gypsum in per cent

All laboratories used the same, designated, percentage of water in expansion-contraction and compressive strength tests of mortars containing Cements 1, 2 and 3. Each laboratory independently predetermined the water required in mortars containing Cement 4. In all cases the water was held constant for all increments of SO3. Flow tests indicated that the addition of

gypsum produced only a minor change in consistency. A constant amount of water was used in the test for calcium sulfate in hydrated cement mortar as prescribed in the standard method.

The cement-gypsum blends were prepared in individual lots as required for each batch of mortar. The required amount of gypsum was weighed to the nearest 0.1 gram. Cement was then added to bring the total weight to the required batch quantity. The weighed mixture was then transferred to a bowl and mixed with a rubber spatula preparatory to adding it to the water in the mechanical mixer.

Optimum SO_3 Content of Each Cement

The best value of optimum SO_3 for contraction was determined by pooling the data reported by the five laboratories. Differences in contraction produced by each increment of SO_3 in each round were calculated. The results were then analyzed statistically by the control chart method. The average results of tests that were in control are tabulated in Table VI. Except in one instance, at least four of the five laboratories were found to be in control for each increment of each cement. The cumulative change in contraction was calculated and used to plot the curves shown in Figure 1. This figure also shows the rate of change in contraction for each increment of SO_3. The SO_3 content at which these curves intersect the ordinate of zero change is 0.25 per cent below the optimum value, very nearly. A slight discrepancy results when the upper plotted curves are not symmetrical about the axis of optimum SO_3 but the error is not greater than 0.05 per cent SO_3. The arrows in the upper curves of Figure 1 indicate the position of optimum SO_3.

The curves of the four cements are plotted in Figure 2 so that the point of optimum coincides. The rate of change curves in the lower part of the figure are plotted so that the points of zero change in contraction coincide.

The changes in compressive strength produced by increases of 0.5 per cent SO_3 as reported by four laboratories, were calculated in the same manner as were the changes in contraction. The results

are given in Table VII. Curves of compressive strength versus SO3 content are shown in Figure 3. The precision of the compressive strength test is not as high as that of the contraction test. Consequently, the curves of Figure 3 could not be plotted with the same degree of confidence. The optimum SO3 content for strength was determined in the same manner as that for contraction. The arrows in the upper curves of Figure 3 indicate the optimum determined by both methods. It will be noted that there is no serious discrepancy. This finding is of importance since a number of investigators have reported different values of optimum for contraction and strength. Their conclusions however, were not based on data as comprehensive as those obtained in this co-operative study.

The compressive strength results determined by Laboratory 5 could not be pooled with the others because they were made on 1:2 mortar instead of the designated 1:2.75 mortar. The results of Laboratory 5 are plotted in Figure 4. The arrows of these curves represent the optimum SO3 content for contraction as derived from the test data of the five laboratories. It will be noted that this optimum value is not out of line with the indications of the strength data obtained by Laboratory 5 using the richer mortar.

The effect of SO3 content on expansion is shown in Figure 5. The data were obtained by pooling the results of the five laboratories. The arrows in these charts show SO3 contents of 0.25 and 0.75 percentage points greater than the optimum percentage as determined by the contraction tests. It will be noted that a sharp increase in expansion results when the SO3 content exceeds about

+0.75 percent relative to optimum. The expansion test therefore, may provide an important safeguard against a gross excess of SO_3 in the cement.

For reasons that will become apparent later in this discussion, absolute values of expansion are of greater significance than are the changes produced by increments of SO_3. This report does not contain a table showing the differences in expansion produced by increments of SO_3.

Precision of the Tests

The two measures of precision that are considered are those of repeatability and reproducibility. These measures have been computed in accordance with "Proposed Recommended Practices for Applying Precision Data given in ASTM Methods for Petroleum Products and Lubricants" published as an appendix to "ASTM Standards on Petroleum Products and Lubricants", Nov., 1954. Definitions given in this publication are:

"Repeatability is a quantitative measure of the variability associated with a single operator in a given laboratory, generally with the same apparatus and with a small interval of time. It is defined as the greatest difference between two single and independent results that can be considered acceptable (not significantly different) at the 95 per cent probability level (for methods referring to this recommended practice)."

"Reproducibility is a quantitative measure of the variability associated with operators working in two different laboratories. It is defined as the greatest difference between a single result obtained in one laboratory and a single result obtained in another laboratory that need not be considered suspect (significantly different) at the 95 per cent probability level (for methods referring to this recommended practice)."

In these co-operative tests the reported values of expansion and contraction are the average of four individual specimens made from a batch of mortar. When the average result of a group of specimens is treated as a single observation, it is customary to adopt some criterion for eliminating suspicious results. In these co-operative tests the standard deviation of the individual specimens was computed for each round. A study has been made of these standard deviations for the purpose of determining the limits of variability that can reasonably be expected.

An appropriate value of standard deviation for the expansion test is considered to be 0.0025. The number of tests made by each laboratory and the percentage of results that did not exceed this value of standard deviation is shown below:

Laboratory	No. of Tests	% Meeting Criterion
1	90	100
2	88	95
3	86	56
4	44	98

An appropriate value of standard deviation for the contraction test is considered to be 0.0030. The number of tests made by each laboratory and the percentage of results that did not exceed this value of standard deviation is shown below:

Laboratory	No. of Tests	% Meeting Criterion
1	90	94
2	88	97
3	86	67
4	44	98

In the computation of indices of precision only those results which met the above criteria have been included. Similar criteria were not applied to the tests for compressive strength because they were made in accordance with ASTM C 109 and this method provides rules for rejecting suspected results.

Precision of Compressive Strength Test

Considering first the compressive strength data at 24 hours, the addition of gypsum to increase the SO_3 content by 0.5 per cent when the cement contained optimum SO_3 produced reductions in strength as follows:

Cement 1	200 psi
Cement 2	180 psi
Cement 3	250 psi
Cement 4	40 psi

These values have been scaled from carefully plotted curves similar to those shown in Figure 3. Values of repeatability and reproducibility as shown in Table X are 216 and 214 psi respectively, for the 24-hour compressive strength tests. These measures are nearly as large as the value measured for Cement 3 and are over five times as large as the measured value

for Cement 4. It does not appear, therefore, that compressive strength results can be used advantageously to indicate the relationship of SO_3 content to optimum unless many more than two batches of mortar are tested.

Precision of Contraction Test

Computed values of repeatability and reproducibility for expansion and contraction are given in Tables VIII and IX. There is considerable variation in values for different cements and different increments of SO_3. These variations are believed to result from the relatively small number of results that were available, in comparison with the ideal of an infinite number. For this reason the average values of all cements is considered to be more nearly correct. It is evident that the precision is better when the SO_3 content is near the optimum.

In this study, values of reproducibility are of greater interest than those of repeatability. The average reproducibility of the difference in contraction between cement at optimum SO_3 and at optimum plus 0.5 per cent SO_3 is 0.0046. Average reproducibility for absolute values of expansion are:

At optimum SO_3	0.0041	
At opt. + 0.50% SO_3	0.0060	
At opt. + 1.00% SO_3	0.0102	
At opt. + 0.25% SO_3	0.0050	(interpolated)
At opt. + 0.75% SO_3	0.0076	(interpolated)

These indices, while large with respect to the values to be measured, are more favorable than those of the compressive strength indices.

It should be noted that they represent the range or greatest difference between single, acceptable test results. The departure of a single result from the true value should not be more than one-half of the computed value of reproducibility.

Because of the statistical methods used in deriving the values shown in the curves of Figures 1, 3 and 5, it can be assumed with considerable confidence that these values are very close to the true values, and that single results should not depart from them by more than one-half the value of reproducibility.

Let it be assumed that, by virtue of continuing tests, the cement manufacturer is able to determine the optimum SO_3 content of his cement with relatively high accuracy. Let it be further assumed that he is able to evaluate the effects of variations in chemical composition and fineness on optimum SO_3 content and that knowing these results he is able to proportion clinker and gypsum in his mill to result in a variation in SO_3 not in excess of $\pm$ 0.25 per cent relative to optimum.

If such control were maintained by the manufacturer, it becomes of interest to determine the values of specification limits that could be used to assure the purchaser, as a result of his tests, that the SO_3 content of the cement is within acceptable limits of its optimum value.

For the four cements investigated in this study, it can be determined from the data as shown in Figure 1, that when the

SO_3 content is at optimum minus 0.25 per cent, the true change in contraction is virtually nil. Based on the precision of the contraction test, a suitable value of minimum change in contraction is -0.0025 per cent.

For the cements tested, such an observed result in the contraction test with respect to the apparent relationship to optimum is shown in the following tabulation:

Cement Number	1	2	3	4
Apparent SO_3 content	+1.65	+1.15	+0.63	-0.05
True optimum SO_3	+2.15	+1.55	+0.98	+0.40
Apparent Departure from Optimum	-0.50	-0.40	-0.35	-0.45

These results indicate that the purchaser could be assured that the SO_3 content was not deficient by more than about 0.5 per cent relative to optimum.

If the cement were furnished with an SO_3 content of optimum plus 0.25 per cent, the true change in contraction for the four cements covered in this study are:

Cement 1	+0.0053 per cent
Cement 2	+0.0077 per cent
Cement 3	+0.0080 per cent
Cement 4	+0.0063 per cent

These values have been scaled from carefully drawn curves similar to those of Figure 1.

A suitable specification maximum for change in contraction applicable to these four cements would be 0.0080 + 0.0023 (one-half the value of reproducibility) or 0.010. For the cements tested such an observed result in the contraction test with respect to the apparent relationship to optimum is shown in the following tabulation:

Cement Number	1	2	3	4
Apparent SO3 content	+2.85	2.00	1.48	1.00
True optimum SO3	+2.15	1.55	0.98	0.40
Apparent departure from optimum	+0.70	0.45	0.50	0.60

These results indicate that the purchaser could be assured that the SO3 content was not in excess of optimum by more than 0.75 per cent. The possibility will next be explored that the expansion test is able to afford better assurance to the purchaser. A study of the changes in expansion produced by 0.5 per cent increments of SO3 does not indicate that the desired control can be obtained in this manner.

"True" values of expansion in per cent, scaled from Figure 5, are as follows:

	At optimum +0.25% SO3	At optimum +0.75% SO3
Cement 1	0.0074	0.0130
Cement 2	0.0065	0.0100
Cement 3	0.0045	0.0090
Cement 4	0.0065	0.0095

For cement produced with an SO_3 content of optimum +0.25 per cent a suitable specification limit on expansion would be 0.0075 plus 0.0025 (one-half the value of reproducibility) or 0.010. For the cements tested, such an observed result in expansion with respect to the apparent relationship to optimum SO_3 content is shown by the following tabulation:

Cement Number	1	2	3	4
Apparent SO_3 content	2.75	2.30	1.80	1.25
True optimum SO_3	2.15	1.55	0.98	0.40
Apparent departure from optimum	0.60	0.75	0.82	0.85

The expansion test made on the original cement does not provide as great a degree of assurance to the purchaser as does the contraction test.

When gypsum is added to increase the SO_3 content, 0.50 per cent in the cement containing optimum +0.25 per cent SO_3, the cement tested is at optimum +0.75 per cent. A suitable specification limit for this expansion test is 0.010 plus 0.004 (one-half the reproducibility) or 0.014 per cent. (The use of this value, in effect, would require that the manufacturer control Cement No. 1 below optimum +0.10 per cent).

For the cements tested such an observed result in expansion with respect to the apparent relationship to optimum SO_3 content is shown in the following tabulation:

Cement Number	1	2	3	4
Apparent SO_3 content	2.95	2.60	1.93	2.00
Apparent SO_3 (original cement)	2.45	2.10	1.43	1.50
True optimum SO_3	2.15	1.55	0.98	0.40
Apparent departure from optimum	0.30	0.55	0.45	1.10

These results do not indicate that the expansion test of itself affords greater control than that obtainable by the contraction test, (except in the case of Cement 1, for which the selected limit of expansion was more restrictive than for the other cements). Nevertheless, the expansion test can be made with little additional effort and it is believed that it can serve the purpose of furnishing added assurance to the purchaser.

Precision of Test for Calcium Sulfate in Hydrated Mortar

Tests for calcium sulfate in hydrated mortar at three increments of SO_3 were made by three laboratories. The criterion has been suggested that optimum SO_3 content lies within the range of more than 0.2 grams of SO_3 per liter at 18 hours and less than 0.5 grams at 24 hours. The SO_3 content of each cement that results in each of these values has been computed by straight-line interpolation and the results are shown in Table V. The mean of this range is taken as the optimum SO_3 by this method of test. The range in increments of SO_3 for Cement 2 did not embrace 0.5 grams of SO_3 per liter at 24 hours and therefore the optimum for

this cement cannot be computed exactly. For the remaining cements the data indicate that the three laboratories making this test were able to obtain values of optimum SO_3 within 0.3 per cent of the value established by the co-operative expansion-contraction tests. The results for Cement 2 departed from the expansion-contraction value by more than 0.55 per cent.

Drying Cabinet

In the contraction test, four of the laboratories used drying cabinets constructed in accordance with the details shown in the Appendix. The drying conditions within these cabinets varied somewhat between laboratories. In general, the relative humidity rose to about 75 per cent when specimens were first placed in the cabinet. During the succeeding 14 to 18 hours the relative humidity dropped to values ranging between 45 and 60 per cent. One laboratory conducted the contraction test in a room which was maintained at 73 $\pm$ 2 F and 50 to 57 per cent relative humidity.

Nothwithstanding the apparent differences in drying conditions among the several laboratories, the data indicate that the magnitude of contraction of similar specimens was reasonably constant. A summary of results is given in Table XI. The test procedure required that specimens of the same cement with varying increments of SO3 be molded in close succession on the same day. The differences in contraction for varying increments of SO3 were more nearly constant among laboratories than were the absolute values. It is concluded that the control of temperature and relative humidity provided by the cabinets was satisfactory for the purpose of the test.

Computation of Cement - Gypsum Blends

Computation of the amount of gypsum to be blended with the original cement to obtain the desired increment of SO_3 depends on the SO_3 content of the original cement. The error introduced by assuming a fixed SO_3 content in the original cement, say 2.0 per cent, is not great. Routine test procedure can be simplified by such an assumption to eliminate the necessity of determining SO_3. If this is done, the formula for computing added gypsum to produce an increase of 0.5 per cent SO_3 becomes:

$$X = \frac{0.5}{C-2.00} \times 100$$

$$Y = 100 - X$$

Where X = the percent of gypsum in the blend

Y = the per cent of cement in the blend

C = the SO_3 content of the gypsum

Absolute Values of Optimum SO_3

Throughout this discussion the SO_3 content of the cements and blends has been expressed in terms of increments relative to the original cement. It is of interest to consider the absolute values of optimum SO_3 and these are given below:

Cement 1, Type III, high C_3A, low alkali	3.86 per cent
Cement 2, Type III, low C_3A, low alkali	3.04 per cent
Cement 3, Type I, high C_3A, high alkali	3.26 per cent
Cement 4, Type II, low C_3A, high alkali	2.10 per cent

Duration of Drying

Laboratory 1 submitted data of contraction of the four cements at the ages of 7, 14, 21 and 28 days, during which the specimens were subjected to drying for 4, 11, 18 and 25 days. The data when plotted as change of rate in contraction curves, similar to those shown in Figure 1, indicated optimum SO_3 contents as shown below:

Increment of SO_3 required for optimum after drying for the period indicated

Cement No.	4 days	11 days	18 days	25 days
1	2.12	2.22	2.24	2.32
2	1.57	1.68	1.73	1.77
3	0.97	1.05	1.12	1.15
4	0.40	0.55	0.60	0.62

The data indicate a gradual increase in the amount of SO_3 required for optimum as the period of drying is increased. Optimum SO_3 after 25 days of drying is about 0.20 per cent higher than that after 4 days of drying.

Conclusions

The results of these co-operative tests on four cements, to the extent that they are applicable to all cements, indicate the following with respect to single tests made upon the cement as received and upon the same cement to which gypsum has been added to increase the SO3 content by 0.5 percentage point.

The contraction test can provide assurance to the purchaser that the SO3 content lies within the range of minus 0.50 per cent to plus 0.75 per cent relative to optimum. To provide such assurance it is necessary that the manufacturer control the SO3 content within plus or minus 0.25 percentage point of the optimum. Suitable specification limits to provide such assurance to the purchaser are that the difference in contraction shall not be less than -0.0025 nor greater than +0.010 per cent.

The expansion test while not more discriminating than the contraction test with respect to an excess of SO3, affords additional assurance against such an excess. Suitable specification limits for expansion of the original cement are not more than 0.010 per cent, and for the cement to which gypsum has been added to increase the SO3 content by 0.5 per cent, not more than 0.014 per cent.

Although the relative precision of the compressive strength test is shown in this study to be less than that of the expansion-contraction test, the fact that test results can be obtained within 24 hours suggests the desirability of further investigation.

Although the three laboratories that performed the test for calcium sulfate in hydrated cement mortar (modified to obtain results at 18 and 24 hours) were able to obtain the same order of precision as in the expansion-contraction test, the data are not sufficiently extensive to warrant the conclusion that it possesses the same degree of merit.

The expansion-contraction test with limits open enough to apply to all cements does not appear to afford as high a degree of control as desirable. If suitable test limits are applied to individual cements, or to individual types of cement, it appears possible to use the test to obtain reasonably good control from the standpoint of the consumer as well as the producer. Programs of co-operative testing between producer and consumer of single types of cement produced at individual mills are encouraged as means of determining feasible control limits for the cements under investigation.

TABLE I

Chemical Analyses and Fineness of Cements and Gypsum

Cement Number	1	2	3	4	Gypsum
SiO_2	21.0	21.8	21.0	22.5	0.20
Al_2O_3	6.3	4.4	5.9	4.5	{ 0.20
Fe_2O_3	2.2	3.8	2.8	4.5	
CaO	65.4	64.3	63.1	64.4	33.0
MgO	1.3	2.2	2.7	1.0	0.2
SO_3	1.71	1.49	2.28	1.70	46.4
Loss on Ignition	1.3	1.10	0.85	0.62	
Insol. Res.	0.22	0.19	0.18	----	
Na_2O	0.10	0.35	0.53	0.66	
K_2O	0.64	0.17	0.69	0.23	
Na_2O Equiv.	0.52	0.46	0.98	0.81	
C_4AF	7	12	8	14	
C_3A	13	5	11	4	
C_3S	56	57	46	49	
C_2S	18	20	25	28	
Specific Surface Blaine	4690	5560	3290	2740	
% Passing No. 325					93

TABLE II

AVERAGE EXPANSION OF EACH CEMENT

Values shown are the arithmetical means of all acceptable results, in per cent

Cement Number	Increments of SO3, per cent						
	0	0.5	1.0	1.5	2.0	2.5	3.0
1			0.0069	0.0072	0.0067	0.0076	0.0157
2		0.0058	0.0054	0.0057	0.0074	0.0122	
3	0.0059	0.0045	0.0039	0.0061	0.0197		
4	0.0050	0.0057	0.0085	0.0114	0.0140		

TABLE III

AVERAGE CONTRACTION OF EACH CEMENT

Values shown are the arithmetical means of all acceptable results, in per cent

Cement Number	Increments of SO_3, per cent						
	0	0.5	1.0	1.5	2.0	2.5	3.0
1			0.0695	0.0604	0.0582	0.0572	0.0646
2		0.0721	0.0568	0.0521	0.0564	0.0665	
3	0.0644	0.0478	0.0437	0.0484	0.0589		
4	0.0337	0.0305	0.0376	0.0483	0.0528		

TABLE IV

AVERAGE COMPRESSIVE STRENGTH OF EACH CEMENT

Values shown are the arithmetical means of all results reported by Laboratories 1 to 4 inclusive at the age of 24 hours, in psi

Cement Number	Increments of SO3, per cent						
	0	0.5	1.0	1.5	2.0	2.5	3.0
1			1922	2224	2400	2273	2026
2		1560	1875	2105	2038	1839	
3	1237	1451	1509	1268	1052		
4	955	1137	1099	1001	917		

TABLE V

INTERPOLATED VALUES OF CALCIUM SULFATE IN HYDRATED MORTAR
Values are in per cent of SO3 in excess of that in the original cement required to yield 0.2 gms. of SO3 at 18 hours and 0.5 grams at 24 hrs.

Cement Number	Laboratory Number	0.2 gm. 18 hrs.	0.5 gm. 24 hrs.	Mean	Optimum by Contraction
1	1	2.02	2.42	2.22	
	2	2.12	2.50	2.31	
	3	2.10	2.68	2.39	
	Avg.	2.08	2.53	2.31	2.15
2	1	1.80	2.50+	2.15+	
	2	1.75	2.50+	2.13+	
	3	1.68	2.50+	2.09+	
	Avg.	1.74	2.50+	2.12+	1.55
3	1	0.20	1.20	0.70	
	2	0.20	1.15	0.68	
	3	0.40	1.24	0.84	
	Avg.	0.27	1.20	0.74	0.98
4	1	0.22	1.04	0.63	
	2	0.12	0.68	0.39	
	3	0.20	1.05	0.63	
	Avg.	0.18	0.92	0.55	0.40

TABLE VI

SUMMARY OF DATA USED IN ESTABLISHING OPTIMUM SO3
FOR MINIMUM CONTRACTION

Values shown are change in contraction in per cent
Produced by increasing the SO3 content 0.5 per cent above
the increment shown in each column

Decreasing contraction indicated as minus
Increasing contraction indicated as plus

	Increment of SO3, per cent			
Cement No. 1	1.0	1.5	2.0	2.5
No. of Labs. in Control	4	3	4	5
Avg. Change in Contraction	-0.0080	-0.0044	+0.0011	+0.0065
Cumulative Change	-0.0080	-0.0124	-0.0113	-0.0048
Standard Deviation	0.0022	0.0010	0.0021	0.0023
Cement No. 2	0.5	1.0	1.5	2.0
No. of Labs. in Control	5	4	4	5
Avg. Change in Contraction	-0.0127	-0.0066	+0.0046	+0.0098
Cumulative Change	-0.0127	-0.0193	-0.0147	-0.0049
Standard Deviation	0.0053	0.0017	0.0023	0.0028
Cement No. 3	0.0	0.5	1.0	1.5
No. of Labs. in Control	5	5	4	5
Avg. Change in Contraction	-0.0176	-0.0046	+0.0054	+0.0103
Cumulative Change	-0.0176	-0.0222	-0.0168	-0.0065
Standard Deviation	0.0031	0.0025	0.0013	0.0035
Cement No. 4	0.0	0.5	1.0	
No. of Labs. in Control	4	5	4	
Avg. Change in Contraction	-0.0022	+0.0068	+0.0101	
Cumulative Change	-0.0022	+0.0046	+0.0147	
Standard Deviation	0.0013	0.0019	0.0012	

NOTE: Laboratories in control determined by control chart method of analysis. Individual laboratories reported from two to five rounds. Computations were made to compensate for differences in number of rounds. Standard deviations were computed only from results that were in control.

TABLE VII

SUMMARY OF DATA USED IN ESTABLISHING OPTIMUM SO_3
FOR MAXIMUM COMPRESSIVE STRENGTH
Values shown are change in compressive strength in psi
produced by increasing the SO_3 content 0.5 per cent above
the increment in each column

Decreasing Strength indicated as minus
Increasing strength indicated as plus

	Increment of SO_3, per cent			
Cement No. 1 - 24 hours	1.0	1.5	2.0	2.5
No. of Labs. in Control	3	4	4	3
Avg. Change in Strength	+349	+176	-136	-301
Cumulative Change	+349	+525	+389	+ 88
Standard Deviation	70	56	128	52
Cement No. 2 - 24 hours	0.5	1.0	1.5	2.0
No. of Labs. in Control	4	4	4	4
Avg. Change in Strength	+316	+229	- 66	-198
Cumulative Change	+316	+545	+479	+281
Standard Deviation	72	64	54	72
Cement No. 3 - 24 hours	0.0	0.5	1.0	1.5
No. of Labs. in Control	4	4	4	4
Avg. Change in Strength	+215	+ 62	-250	-216
Cumulative Change	+215	+277	+ 27	-189
Standard Deviation	60	61	63	89
Cement No. 4 - 24 hours	0.0	0.5	1.0	1.5
No. of Labs. in Control	2	4	3	4
Avg. Change in Strength	+152	- 46	- 35	- 84
Cumulative Change	+152	+106	+ 71	- 13
Standard Deviation	35	39	55	51

TABLE VIII

PRECISION OF EXPANSION TESTS
Values are for direct recorded measurements, not for differences between increments of SO_3

	Increment of SO_3, per cent		
Cement No. 1	2.0	2.5	3.0
Repeatability	0.0036	0.0022	0.0178
Reproducibility	0.0033	0.0067	0.0125
Cement No. 2	1.5	2.0	2.5
Repeatability	0.0085	0.0104	0.0106
Reproducibility	0.0066	0.0020	0.0049
Cement No. 3	1.0	1.5	2.0
Repeatability	0.0040	0.0049	0.0068
Reproducibility	0.0038	0.0069	0.0149
Cement No. 4	1.0	1.5	2.0
Repeatability	0.0024	0.0051	0.0067
Reproducibility	0.0027	0.0085	0.0223

TABLE IX

PRECISION OF CONTRACTION TESTS
Values are in units of per cent contraction computed from change in contraction produced by increasing the SO_3 content 0.5 per cent above the increments shown in each column

	Increment of SO_3, per cent			
Cement No. 1	1.0	1.5	2.0	2.5
Repeatability	0.0072	0.0040	0.0090	0.0063
Reproducibility	0.0122	0.0110	0.0060	0.0108
Cement No. 2	0.5	1.0	1.5	2.0
Repeatability	0.0047	0.0071	0.0059	0.0066
Reproducibility	0.0074	0.0028	0.0047	0.0119
Cement No. 3	0	0.5	1.0	1.5
Repeatability	0.0112	0.0191	0.0024	0.0153
Reproducibility	0.0039	0.0278	0.0050	0.0081
Cement No. 4	0	0.5	1.0	1.5
Repeatability	0.0046	0.0048	0.0039	0.0096
Reproducibility	0.0075	0.0055	0.0040	0.0044
Average repeatability				0.0074
Average reproducibility				0.0085
Average repeatability when SO_3 content is at approximate optimum				0.0055
Average reproducibility when SO_3 content is at approximate optimum				0.0046

TABLE X

PRECISION OF COMPRESSIVE STRENGTH TESTS

Values are in units of psi computed from change in strength produced by increasing the SO3 content 0.5 per cent above the increments shown in each column

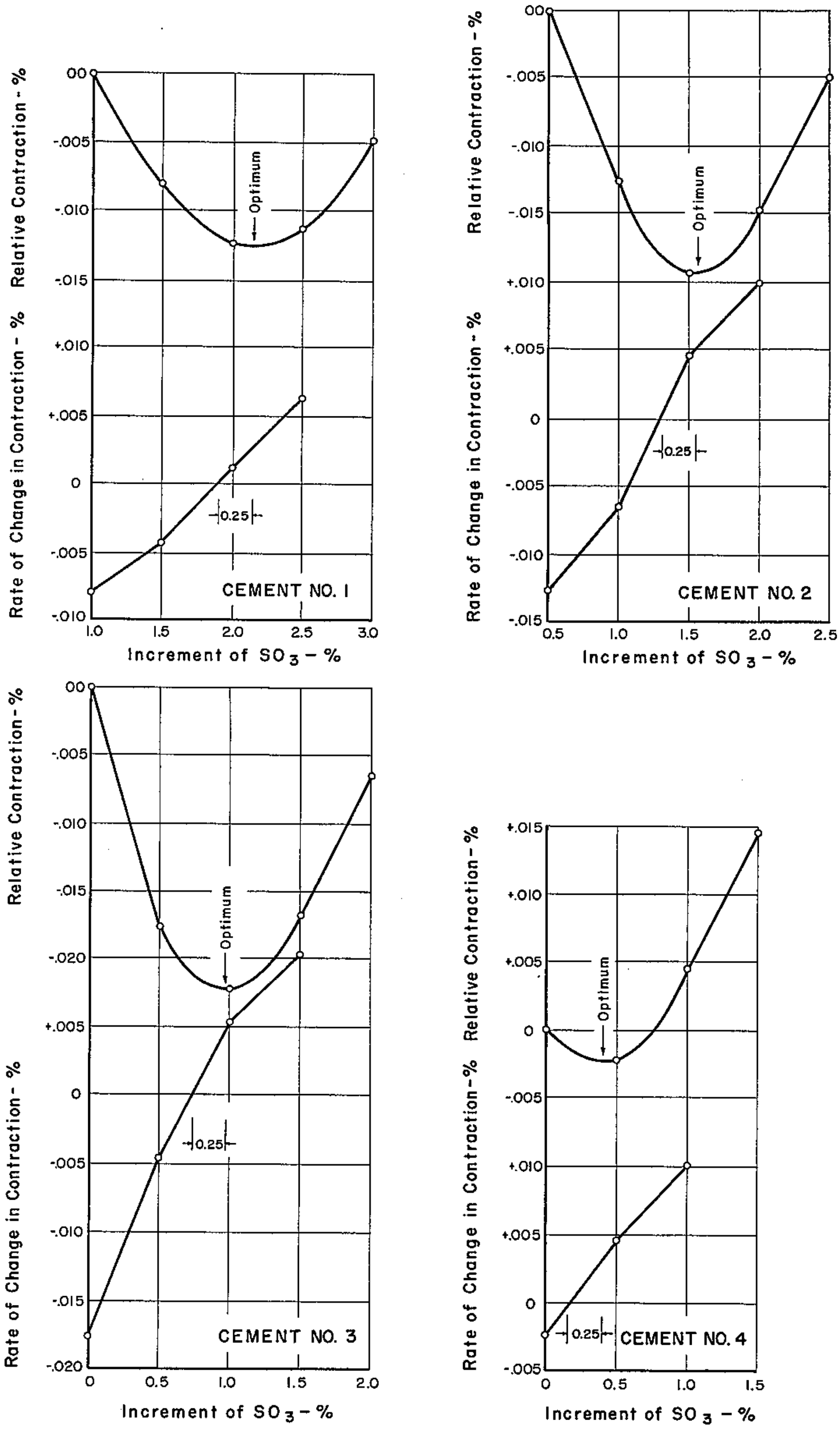
	Increment of SO3, per cent			
Cement No. 1 - 24 hours	1.0	1.5	2.0	2.5
Repeatability	209	200	360	257
Reproducibility	300	111	406	159
Cement No. 2 - 24 hours	0.5	1.0	1.5	2.0
Repeatability	215	175	167	165
Reproducibility	204	177	149	259
Cement No. 3 - 24 hours	0.0	0.5	1.0	1.5
Repeatability	213	209	221	282
Reproducibility	129	131	137	248
Cement No. 4 - 24 hours	0.0	0.5	1.0	1.5
Repeatability	121	116	132	144
Reproducibility	570	162	381	98

Average repeatability	199 psi
Average reproducibility	226 psi
Average repeatability when SO3 content is at approximate optimum	216 psi
Average reproducibility when SO3 content is at approximate optimum	214 psi

TABLE XI

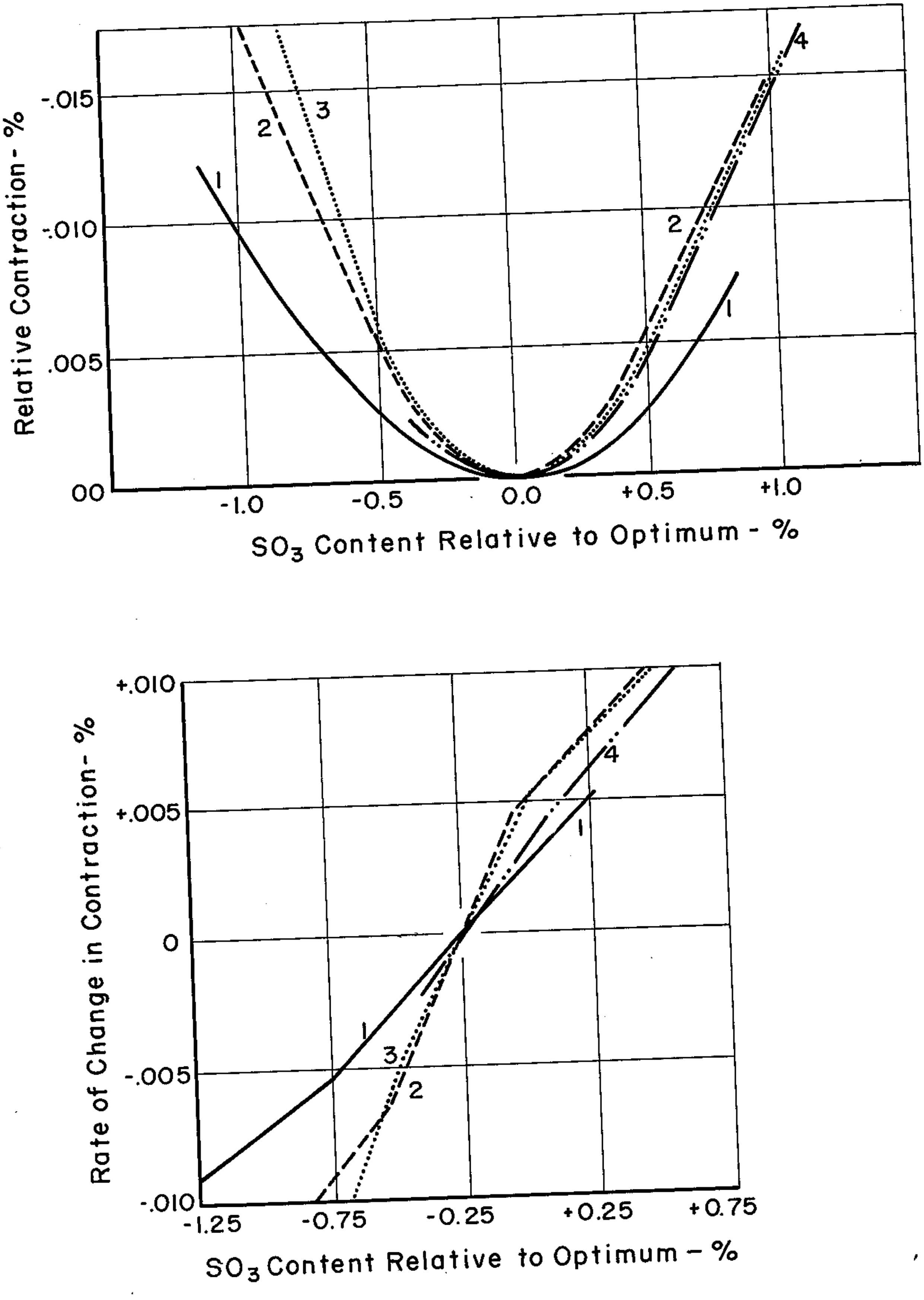
ABSOLUTE VALUES OF CONTRACTION IN PER CENT FOR CEMENTS CONTAINING THE INCREMENT OF SO_3 NEAREST TO THE OPTIMUM VALUE

Laboratory Number	Cement Number 1	2	3	4	Average
1	0.0553	0.0582	0.0435	0.0332	0.0462
2	.0611	.0551	.0439	.0350	.0488
3	.0598	.0546	.0437	.0354	.0484
4	.0552	.0504	.0396	.0286	.0440
5	.0578	.0483	.0432	.0337	.0463
Average	0.0573	0.0533	0.0428	0.0352	0.0470

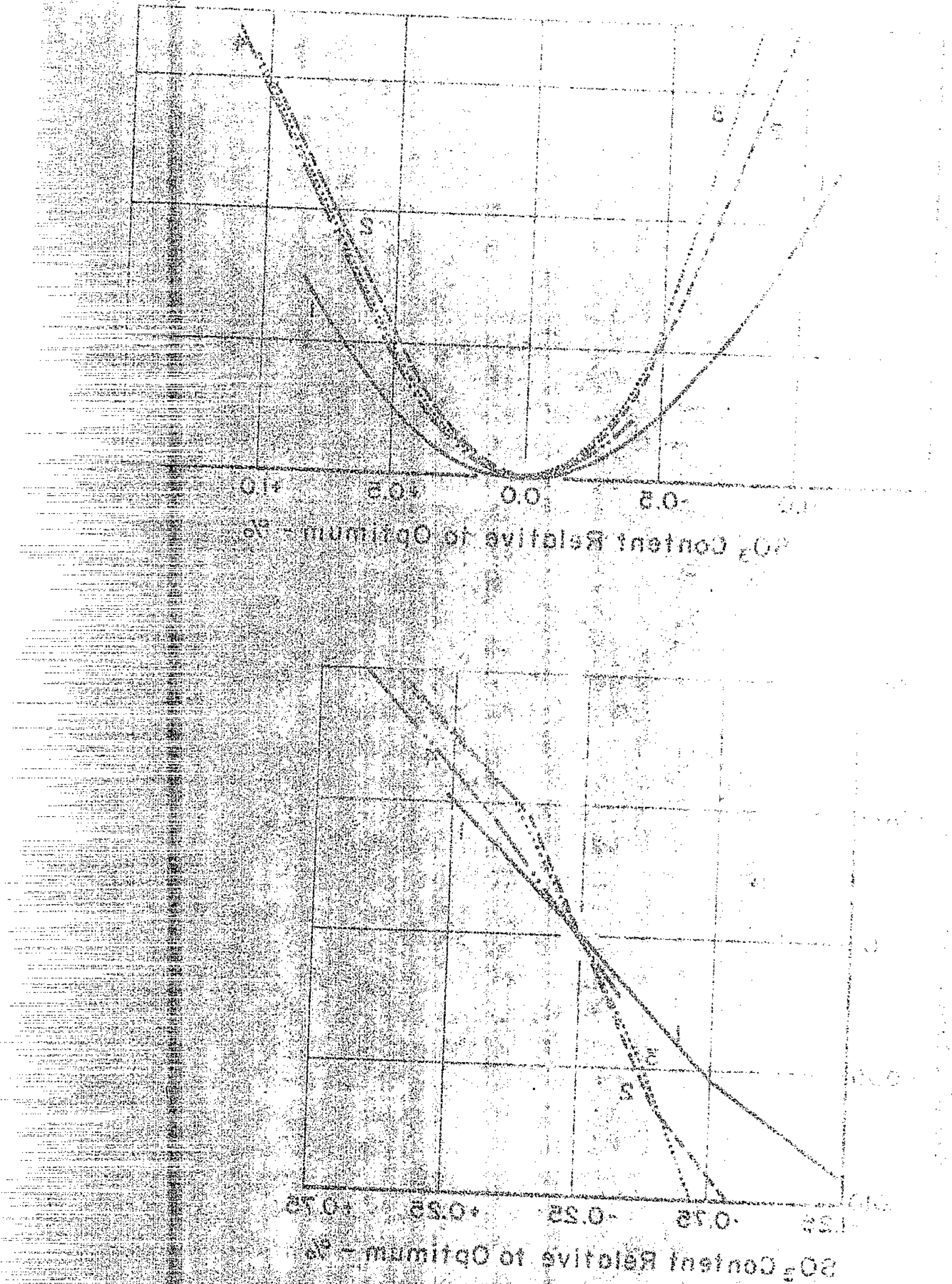


EFFECT OF SO_3 ON CONTRACTION

FIGURE I

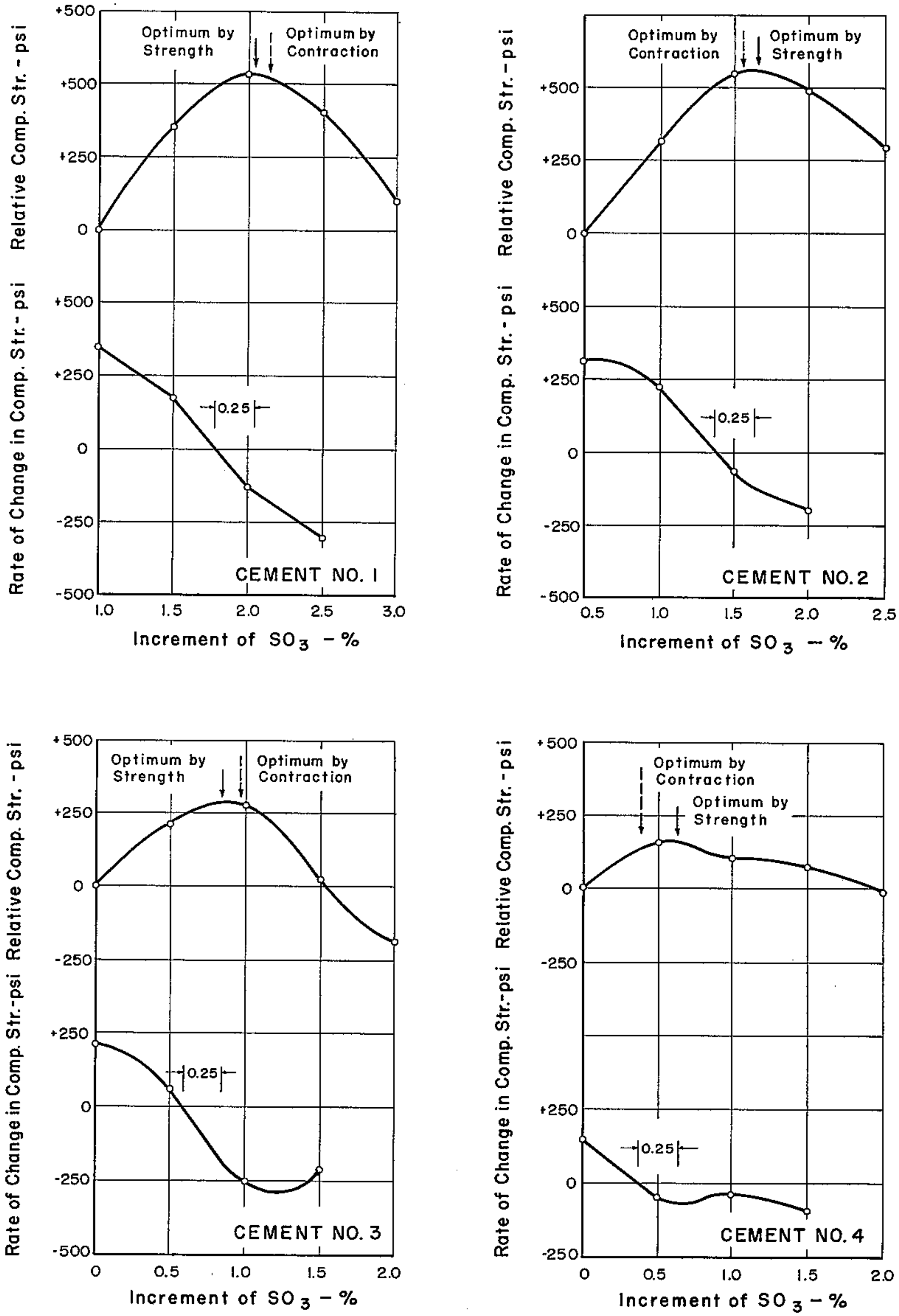


EFFECT OF DEPARTURE FROM OPTIMUM SO_3
ON CONTRACTION
FIGURE 2



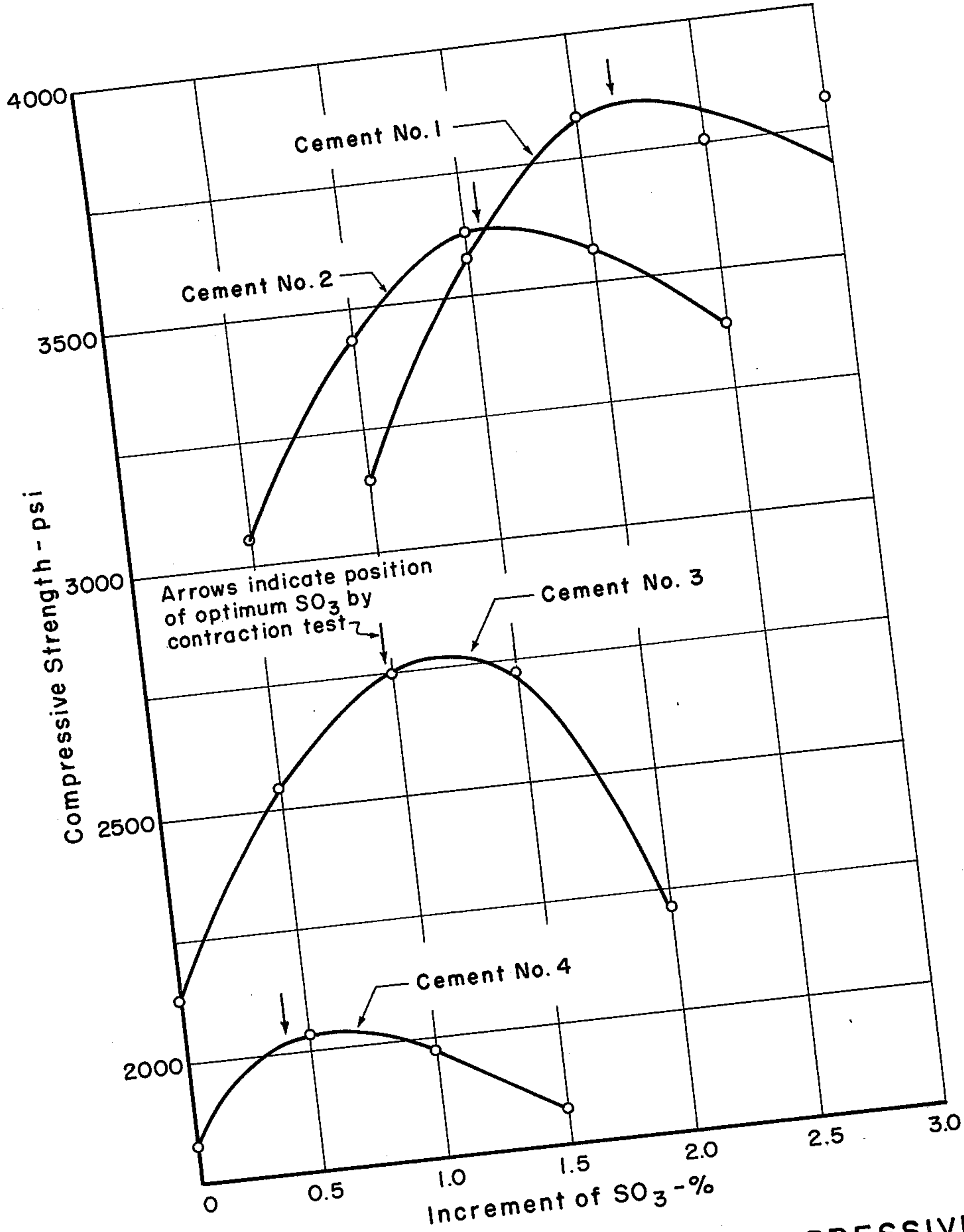
EFFECT OF DEPARTURE FROM OPTIMUM SO_3 ON CONTRACTION

FIGURE 2



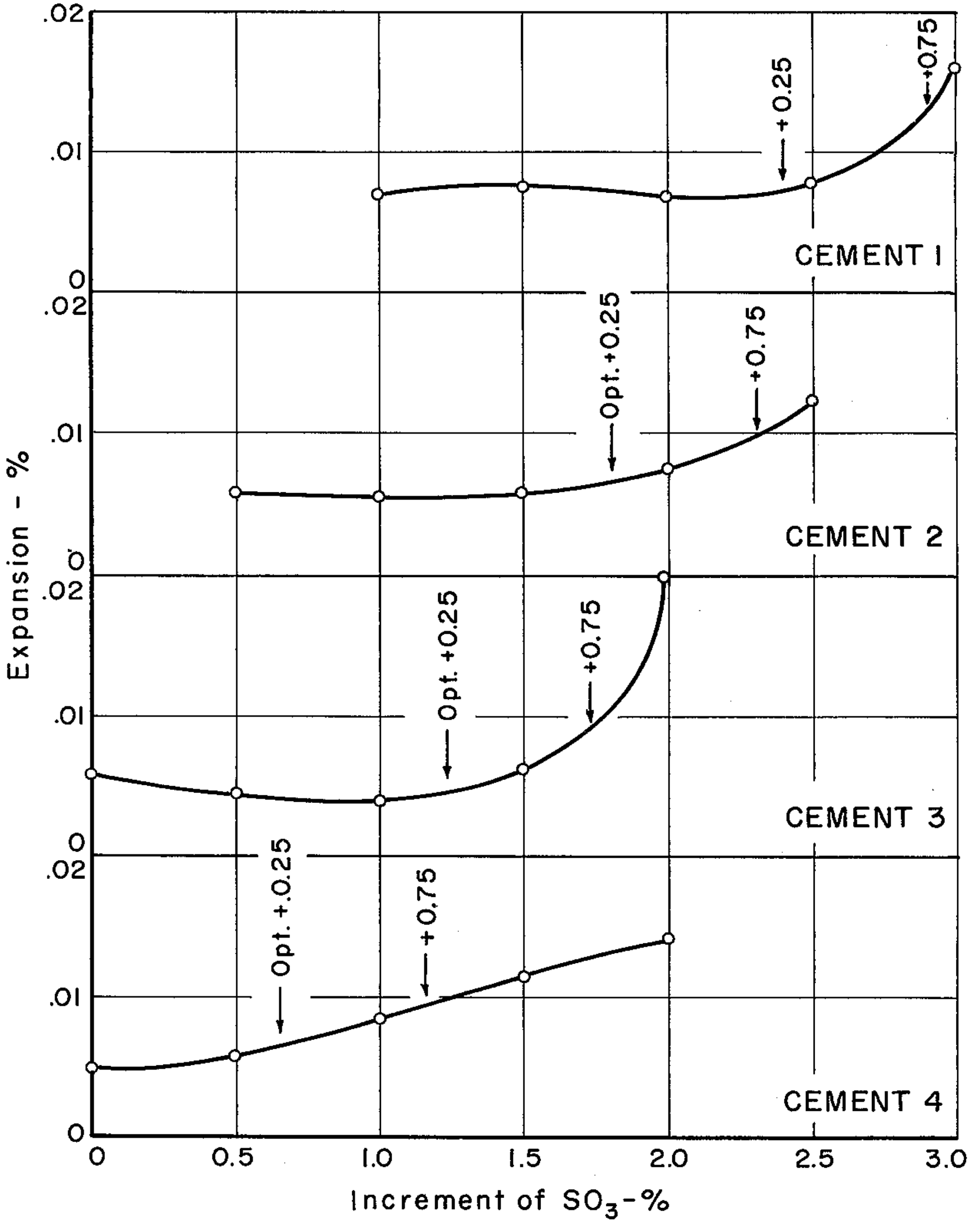
EFFECT OF SO_3 ON 24-HOUR COMPRESSIVE STRENGTH, C 109 MORTAR

FIGURE 3



EFFECT OF SO_3 ON 24-HOUR COMPRESSIVE STRENGTH, 1:2 MORTAR

FIGURE 4



EFFECT OF SO_3 ON EXPANSION

FIGURE 5

APPENDIX

METHOD OF TEST FOR EXPANSION IN WATER AND CONTRACTION IN AIR OF PORTLAND CEMENT MORTAR

SCOPE

1. This method is intended to measure the expansion in water of mortars containing portland cement and a blend of the cement and gypsum. It is also intended to measure the change in contraction in air of mortar produced by the addition of gypsum to the portland cement. The results may be used to indicate the relationship of the SO_3 content of the cement to its optimum content.

APPARATUS

2. Apparatus specified in ASTM Designation: C 109, less specimen molds and testing machine.

3. Molds and comparator as specified in ASTM Designation C 151.

4. Drying cabinet constructed according to the design shown in Figure 1. The pan shall contain a saturated solution of sodium bichromate with an excess of crystals. The depth of solution in the pan shall be about 1-1/2 inches. Spread the sodium bichromate crystals over the bottom of the pan to a loose depth of about 1 inch (about 10 pounds required). Add water to bring the depth to approximately 1-1/2 inches. Stir to effect substantial saturation. Add additional sodium bichromate if necessary to produce or maintain an excess of undissolved salt.

The cabinet shall be installed in a room, the temperature of which is maintained between 68 F. and 81.5 F.

In lieu of the above cabinet, any enclosure equipped with suitable shelves or racks may be used provided the temperature within the enclosure is maintained between 68 F and 81.5 F, and the relative humidity is maintained between 45 and 60 per cent.

5. Device as detailed in Figure 2 to facilitate removal of molded specimens from the base plate.

MATERIALS

6. Graded Ottawa sand, ASTM Designation: C 109.

7. Sodium bichromate, crystals, technical grade.

8. Finely pulverized gypsum of the grade known commercially as "Terra Alba", containing not less than 40 percent SO_3. At least 90 per cent shall pass a U.S. No. 325 sieve.

9. Masking tape, 1 inch wide.

PROCEDURE

10. A batch of mortar shall consist of 750 grams cement or cement-gypsum blend, 1500 grams graded Ottawa sand and water in the quantity required to produce a flow of the straight cement mortar between 100 and 115 when tested in accordance with ASTM Designation C 109. The same amount of water shall be used in the mortar containing the cement-gypsum blend.

11. One batch of mortar shall be mixed with the cement under test. A second batch shall be mixed within 30 minutes with the cement blended with pulverized gypsum in an amount necessary to increase the SO_3 content approximately 0.50 percentage points. The weights of cement and gypsum required to produce the required blend shall be calculated by the following formulas:

$$X = \frac{0.5}{C - 2.00} \times 750$$

$$Y = 750 - X$$

where X = grams of gypsum in the blend
Y = grams of cement in the blend
C = SO_3 content of the gypsum in per cent

12. Prepare the cement-gypsum blend in lots of the size required for a single batch of mortar. Weigh the gypsum to the nearest 0.1 gram using a suitable balance. Add cement to bring the total weight of the blend to 750 grams. Transfer the weighed mixture to a bowl and mix with a rubber spatula until the cement and gypsum appear to be uniformly blended. If the blend is not used at once for mixing mortar, transfer it to a tight container.

13. Mix the mortar according to ASTM Designation: C 109.

14. Mold four 1 by 1 by 10-inch gage length bars from each batch of mortar as provided in Section 4(c) of ASTM Designation C 151.

15. Store the specimens in the moist closet for 24 hours ± 30 minutes, computed from the time of mixing. Remove the specimens promptly from the molds. (The use of the device illustrated in Figure 2 is recommended as a precaution against breakage of the bars.) Measure for length as provided in ASTM Designation: C 151. If not measured at once, protect the specimens by covering them with a damp cloth.

16. Store the specimens in water maintained at a temperature of 73.4 ± 3F. to the age of 72 hours ± 30 minutes, computed from the time of mixing. Remove the specimens from the water, wipe free of surface moisture and measure promptly for length.

17. Place the specimens on shelves of drying cabinet with at least 1 inch clear space between specimens. Close the door of the cabinet and seal with masking tape. Start the fan. Leave the door closed and operate the fan continuously except when adding or removing specimens. Remove specimens when they have reached the age of 168 hours (7 days) ± 30 minutes, computed from the time of mixing. Measure promptly for length.

COMPUTATIONS

18. Compute the expansion in water of each specimen as the length upon removal from water storage less the length upon removal from the molds and express it as a percentage of 10 inches.

19. Compute the contraction in air of each specimen as the length upon removal from water storage less the length upon removal from the drying cabinet and express it as a percentage of 10 inches.

20. The average change in length of four specimens from the same batch of mortar shall be reported as the percent expansion in water or the percent contraction in air provided the variability between specimens does not exceed the limits of acceptability as described below. If the results of four specimens do not meet the criterion for acceptability, but the results of any three of the specimens do meet the criterion, the average of the measured length changes of the three specimens shall be reported as the per cent expansion in water or the per cent contraction in air.

If the length changes of at least three specimens do not meet the criterion for acceptability, the results shall not be reported and the test shall be repeated.