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16. ABSTRACT

Synopsis

Fourteen (14) cements from 11 mills in California were tested for optimum S03 content by extraction of hydrated mortar, drying shrinkage and, to a limited extent, by compressive strength. Variations in S03 content were obtained by blending pulverized gypsum with base cements. Marked reduction in drying shrinkage resulted when S03 was at its optimum value. Compressive strength of about one-half of the cements was highest when S03 was at the optimum value but the compressive strength of the remainder was not greatly affected by variations in S03.

The extraction test performed at 18 and 24 hours indicated the optimum for least drying shrinkage within about 0.25 percentage point. The S03 content of all but two of the cements was below the optimum in amounts varying from about 0.3 to 1 percentage point. The optimum S03 content for 11 of the cements exceeded the percentage permitted under A.S.T.M. Designation: C 150-52. The proposed revision of this specification will permit manufacturers to adjust the S03 content to the optimum value.

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State of California
Division of Highways
MATERIALS AND RESEARCH DEPARTMENT

THE OPTIMUM GYPSUM CONTENT
FOR
CALIFORNIA PORTLAND CEMENTS

Research No. 00272
October 1, 1953

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THE OPTIMUM GYPSUM CONTENT FOR CALIFORNIA PORTLAND CEMENTS

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SYNOPSIS

Fourteen (14) cements from 11 mills in California were tested for optimum SO_3 content by extraction of hydrated mortar, drying shrinkage and, to a limited extent, by compressive strength. Variations in SO_3 content were obtained by blending pulverized gypsum with the base cements. Marked reduction in drying shrinkage resulted when SO_3 was at its optimum value. Compressive strength of about one-half of the cements was highest when SO_3 was at the optimum value but the compressive strength of the remainder was not greatly affected by variations in SO_3 .

The extraction test performed at 18 and 24 hours indicated the optimum for least drying shrinkage within about 0.25 percentage point. The SO_3 content of all but two of the cements was below the optimum in amounts varying from about 0.3 to 1 percentage point. The optimum SO_3 content for 11 of the cements exceeded the percentage permitted under A.S.T.M. Designation: C 150-52. The proposed revision of this specification will permit manufacturers to adjust the SO_3 content to the optimum value.

INTRODUCTION

The beneficial effect of adding gypsum to portland cement clinker in an amount to yield an "optimum" percentage of SO_3 in the

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finished cement has been reported by Lerch(1)* and confirmed by a working committee of Committee C-1 of the American Society for Testing Materials(2). It has been shown that the optimum percentage of SO_3 varies with each cement and is affected by fineness of grinding, the content of C_3A and alkalies and probably other factors. One method of establishing the optimum value is by the determination of unreacted SO_3 in an aqueous extract of hydrated Ottawa Sand mortar. It has been found that when the extract of the mortar at the age of 18 hours contains an appreciable amount of SO_3 but contains a negligible amount at the age of 24 hours, the SO_3 in the cement is at the approximate optimum value as determined by physical tests of mortar or concrete for drying shrinkage, strength and resistance to freezing and thawing. It has also been shown that setting characteristics of the cement are improved and that expansion under continued storage in water is not excessive.

A.S.T.M. Committee C-1 has recommended to the Society that the maximum limit for SO_3 in portland cement be established by an extraction test of hydrated mortar at the age of 24 hours. This proposal does not set a minimum value for SO_3 but is intended to permit manufacturers to add gypsum up to the optimum value which for many cements is in excess of the limitations of the present specifications (C 150-52). It is anticipated that at some future date, minimum requirements will be established by means of a minimum limit for SO_3 in the extract of the mortar at the age of 18 hours. The Society has published a Tentative Method of Test for Calcium Sulfate in Hydrated Portland Cement Mortars, A.S.T.M. Designation:

*see list of references at end of text

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SCOPE OF TESTS

This report presents results of tests for optimum SO_3 content of samples of Type I or Type II portland cement from each of the eleven mills in California. Two samples, one high in alkalies and one low in alkalies, were included from each of three mills. The total number of cements tested was 14. The cements are identified in this report by letters from A to N.

Variations in SO3 content were obtained by blending five increments of pulverized gypsum with each of the selected cement samples. The prepared cements, each with six percentages of SO3, were tested for:

(1) SO3 in hydrated mortar at $18\pm\frac{1}{4}$ hr. and $24\pm\frac{1}{4}$ hr.

(2) Drying shrinkage of mortar cured wet for 7 days then stored in an atmosphere at 100°F and 70 per cent relative humidity.

(3) Compressive strength of mortar at the age of 7 days.

Test results for items (1) and (2) were the averages of three rounds of tests. Each round was completed before starting the next round. Item (3), compressive strength was not planned originally. One batch of mortar was subsequently mixed from each cement from which six cubes were molded and tested at the age of 7 days.

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TABLE I

SO_3 Content of Selected Prepared Samples

Cement	Computed SO_3	SO_3 Found By Analysis
A-2	1.95	1.92
A-3	2.28	2.19
B-2	2.09	2.09
C-3	2.13	2.16
C-4	2.46	2.50
D-2	2.06	2.09
E-2	2.05	2.05
E-3	2.38	2.43
F-3	2.35	2.42
F-4	2.69	2.72
G-3	2.16	2.30
G-4	2.49	2.59
H-2	1.99	2.08
H-3	2.32	2.37
I-3	2.54	2.78
I-4	2.86	3.02
J-5	3.16	3.02
K-3	2.32	2.62
K-4	2.66	2.92
L-3	2.39	2.66
L-4	2.73	2.92
M-2	2.09	2.09
M-3	2.42	2.61
N-4	2.15	2.10
N-5	2.49	2.47
Average	2.37	2.44

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PREPARATION OF SAMPLES

With the exception of cement "M" the gross cement samples were made up as a composite of a number of routine control samples that had been received in the laboratory during the preceeding few months. Cement "M" consisted of a single sample received directly from the mill. The individual samples were blended by thorough mixing in an open-pan laboratory concrete mixer. Each composite sample weighed about 35 pounds.

Gypsum used in the work was from Gerlach, Nevada, and was furnished by Calaveras Cement Co. The SO_3 content as determined by analysis was 43.3 per cent. The rock gypsum was crushed and then ground in a pebble mill to a fineness of approximately 85 per cent passing the No. 200 sieve. After grinding it was mixed in an open tub mixer to assure uniformity.

From each gross sample of cement, six individual samples weighing approximately 2200 grams were selected. One sample was used without alteration before testing, gypsum was added to the remaining samples in quantities to give a progression of increased SO_3 in the blends of 0.34, 0.67, 1.01, 1.35 and 1.68 per cent respectively. The weighed amounts of cement and gypsum were mixed in a small revolving blade mixer which had been checked previously and found to be efficient in producing a uniform mixture. As soon as the samples were prepared, they were placed in sealed tin cans. Analyses for SO_3 were made on a number of the samples after the addition of gypsum. The analytical determination and the computed content of SO_3 is shown in Table I. It will be noted that the difference amounted to 0.30 per cent in one sample

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but that in general the variation was much less and averaged 0.07 per cent.

TEST METHODS

All tests were performed upon 1:2.75 graded Ottawa sand mortar mixed in accordance with ASTM Designation: C 109-50. Two bars for drying shrinkage and two pats for extraction tests were molded from each batch of mortar.

The bars were 1 x 1 x 11-1/4-in. (10-in. effective gage length) with metal inserts for measuring length. They were cured in the moist room under standard conditions for 48 hours after which they were removed from the molds and stored in water at 73°F for 5 days. The bars were then measured for length to the nearest 0.0001" and transferred to racks in a large oven maintained at a temperature of 100±5°F and 70±5 percent relative humidity. The oven had been checked previously with recording instruments and it was found that it could be depended upon to maintain temperature and humidity within these limits. Other investigators have conducted drying shrinkage tests at 70 to 77°F and 50 percent relative humidity. Cooling equipment was not available in our oven and 100° was selected as the lowest temperature that could be maintained dependably. At this temperature and with a relative humidity of 70 percent the difference in vapor pressure between the bars and the surrounding atmosphere was approximately the same as under 70°F and 50 per cent relative humidity. The bars were removed from the oven and measured for length after storage periods of 7, 14, 28, 56 and 120 days.

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TABLE II

CHEMICAL ANALYSES OF THE BASE CEMENTS

Cmt.	Oxide Analysis, Percent											Computed Compound Composition, Percent				
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Loss	Insol	Na_2O	K_2O	Na_2O Equiv.	C_4AF	C_3A	$CaSO_4$	C_3S	C_2S
A	22.7	3.7	3.1	63.4	3.5	1.61	1.4	0.06	0.24	0.48	0.56	9	5	3	52	26
B	22.5	4.2	2.8	62.4	4.1	1.75	2.0	0.11	0.28	0.32	0.49	9	6	3	45	30
C	21.7	4.1	3.5	62.5	4.1	1.44	1.4	0.19	0.54	0.28	0.72	11	5	2	53	22
D	23.0	3.9	3.1	62.4	3.9	1.72	1.6	0.08	0.46	0.27	0.64	9	5	3	44	33
E	22.4	4.0	2.8	63.5	4.3	1.71	1.0	0.04	0.20	0.39	0.46	9	6	3	52	25
F	22.2	5.9	2.9	64.1	1.5	1.68	1.4	0.59	0.50	0.26	0.67	9	11	3	44	31
G	22.7	4.1	5.3	63.5	1.3	1.48	0.9	0.23	0.33	0.24	0.49	16	2	3	46	30
H	24.6	1.9	3.1	66.2	0.8	1.65	1.0	0.04	0.09	0.19	0.22	9	0.3	3	60	25
I	22.3	5.2	2.6	63.9	1.7	1.85	1.7	0.07	1.06	0.10	1.13	8	9	3	46	29
J	22.6	5.2	4.4	63.3	1.7	1.82	0.9	0.08	0.48	0.09	0.54	13	6	3	39	35
K	22.1	6.0	2.4	62.7	2.5	1.65	1.2	0.06	0.56	0.67	1.00	7	12	3	39	34
L	21.2	5.2	3.4	63.9	2.0	1.72	1.6	0.22	0.52	0.59	0.91	10	8	3	54	20
M	23.6	3.8	3.5	64.0	2.1	1.75	1.1	0.28	0.23	0.24	0.39	11	4	3	46	33
N	24.5	4.2	3.0	64.7	1.4	1.13	1.0	0.11	0.36	0.28	0.54	9	6	2	41	39

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Before measurements were made the bars were cooled to room temperature over a saturated solution of sodium bichromate in order to maintain a relative humidity of 50 per cent.

Extraction tests, except for cement "M" were made during the first round in accordance with the Tentative Method of Test for Calcium Sulfate in Hydrated Portland Cement Mortar, ASTM Designation: C 265-51T and tests were made at $18\pm\frac{1}{4}$ hr. as well as the specified $24\pm\frac{1}{4}$ hr. For all rounds of cement "M" and the remaining two rounds of the other cements the procedure was modified slightly in accordance with suggestions made by C. E. Wuerpel, a member of the Working Committee on the Effect of SO_3 in Cement of ASTM Committee C-1. Modifications consisted of heaping the mortar above the top of the mold in order to provide a larger quantity for test. Four hundred (400) grams (instead of 250-300 grams) were pulverized and mixed with 100 ml. (instead of 50 ml.) of water. The mixture was stirred mechanically (instead of occasional hand stirring) and an aliquot of 40 ml. (instead of 20 ml.) was taken for determination of SO_3.

Compression tests were made in accordance with ASTM Designation C 109-50.

DATA OF THE TESTS

Data obtained during the tests are presented in the following tables and figures.

Table I.	SO_3 Content of Selected Prepared Samples
Table II.	Chemical Analyses of the Base Cements
Table III.	Average Results of Shrinkage, Strength and Extraction Tests. The indicated percentage of SO_3 shown in the second column is that computed from the weighed proportions of gypsum and the base cement.

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TABLE III

(Continued)

Cement	SO_3 Percent	Drying Shrinkage Percent					Compr. Str. P.S.I.	SO_3 in Hardened Mortar Gms./Liter	
		7 Days	14 Days	28 Days	56 Days	120 Days	7 days	18 Hours	24 Hours
H	1.65	0.058	0.064	0.067	0.071	0.078	2870	0.277	0.129
	1.99	0.059	0.065	0.068	0.072	0.079	2750	0.844	0.307
	2.32	0.060	0.067	0.071	0.076	0.083	2690	1.017	0.750
	2.66	0.067	0.073	0.076	0.081	0.089	2500	1.080	1.078
	3.00	0.071	0.076	0.080	0.085	0.092	2250	1.089	1.068
	3.33	0.074	0.078	0.083	0.088	0.095	2070	1.087	1.087
I	1.85	0.083	0.091	0.096	0.101	0.111	2490	0.007	0.013
	2.19	0.073	0.081	0.085	0.088	0.100	2600	0.010	0.015
	2.54	0.067	0.074	0.079	0.084	0.095	2600	0.280	0.092
	2.86	0.065	0.072	0.077	0.083	0.091	2590	1.030	0.297
	3.19	0.067	0.074	0.079	0.084	0.097	2360	1.728	1.212
	3.53	0.069	0.074	0.079	0.086	0.101	2090	1.912	1.942
J	1.82	0.082	0.088	0.091	0.094	0.102	3320	0.007	0.008
	2.16	0.073	0.078	0.081	0.085	0.092	3150	0.009	0.005
	2.49	0.065	0.071	0.075	0.078	0.086	3320	0.017	0.009
	2.83	0.059	0.066	0.068	0.075	0.082	3180	0.244	0.026
	3.16	0.059	0.066	0.068	0.074	0.082	3050	0.675	0.025
	3.50	0.062	0.067	0.071	0.078	0.087	3010	1.054	0.705
K	1.65	0.091	0.096	0.100	0.105	0.114	3460	0.015	0.004
	1.99	0.080	0.086	0.090	0.095	0.105	3360	0.024	0.007
	2.32	0.074	0.077	0.085	0.090	0.100	3260	0.286	0.047
	2.66	0.067	0.073	0.078	0.084	0.094	3200	0.954	0.126
	3.00	0.069	0.074	0.079	0.085	0.096	3200	1.771	0.776
	3.33	0.069	0.075	0.080	0.087	0.098	3110	2.448	1.540
L	1.72	0.078	0.079	0.084	0.087	0.094	3940	0.015	0.002
	2.06	0.069	0.072	0.076	0.079	0.087	3950	0.107	0.015
	2.39	0.065	0.067	0.074	0.078	0.086	3700	0.413	0.090
	2.73	0.065	0.069	0.074	0.077	0.087	3650	0.882	0.364
	3.06	0.065	0.069	0.075	0.080	0.092	3510	1.460	0.983
	3.40	0.071	0.073	0.079	0.085	0.095	3120	1.489	1.537
M	1.75	0.060	0.063	0.066	0.072	0.078	3010	0.215	0.068
	2.09	0.060	0.063	0.067	0.071	0.080	3020	0.504	0.198
	2.42	0.060	0.067	0.071	0.077	0.085	2970	0.956	0.650
	2.76	0.069	0.071	0.075	0.080	0.090	2830	1.105	1.282
	3.09	0.076	0.078	0.083	0.088	0.098	2260	1.114	1.121
	3.43	0.080	0.083	0.087	0.093	0.103	2120	1.128	1.164
N	1.13	0.077	0.084	0.090	0.095	0.104	2880	0.003	0.002
	1.47	0.070	0.074	0.080	0.085	0.094	2940	0.007	0.006
	1.81	0.064	0.068	0.074	0.078	0.088	3030	0.030	0.015
	2.15	0.060	0.065	0.071	0.077	0.086	3200	0.344	0.070
	2.49	0.061	0.064	0.072	0.078	0.088	3220	0.842	0.299
	2.83	0.063	0.067	0.073	0.079	0.090	3030	1.109	0.811

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TABLE III

AVERAGE RESULTS OF SHRINKAGE, STRENGTH, AND EXTRACTION TESTS

Cement	SO_3 Percent	Drying Shrinkage Per Cent					Compr. Str. P.S.I.	SO_3 in Hardened Mortar Gms./Liter	
		7 Days	14 Days	28 Days	56 Days	120 Days	7 days	18 Hours	24 Hours
A	1.61	0.061	0.066	0.072	0.079	0.085	2120	0.022	0.008
	1.95	0.054	0.059	0.065	0.073	0.079	2310	0.182	0.041
	2.28	0.057	0.061	0.067	0.075	0.083	2470	0.689	0.438
	2.62	0.063	0.067	0.073	0.079	0.089	2450	1.183	0.726
	2.96	0.064	0.069	0.076	0.083	0.093	2110	1.320	1.234
	3.30	0.076	0.081	0.085	0.096	0.102	1760	1.345	1.368
B	1.75	0.053	0.059	0.063	0.069	0.072	2940	0.135	0.023
	2.09	0.053	0.058	0.062	0.069	0.075	2770	0.534	0.162
	2.42	0.054	0.059	0.063	0.071	0.078	2780	1.099	0.560
	2.76	0.057	0.063	0.068	0.075	0.084	2710	1.215	1.039
	3.09	0.060	0.067	0.073	0.082	0.089	2430	1.233	1.317
	3.43	0.071	0.077	0.086	0.095	0.104	1850	1.240	1.292
C	1.44	0.067	0.070	0.076	0.079	0.085	2490	0.005	0.007
	1.78	0.055	0.059	0.064	0.069	0.075	2700	0.069	0.014
	2.13	0.051	0.057	0.064	0.068	0.075	2730	0.352	0.100
	2.46	0.053	0.058	0.066	0.070	0.078	2690	0.858	0.388
	2.79	0.055	0.061	0.070	0.074	0.084	2580	1.233	0.903
	3.13	0.060	0.065	0.073	0.078	0.088	2360	1.343	1.293
D	1.72	0.052	0.059	0.063	0.067	0.074	2530	0.208	0.074
	2.06	0.052	0.058	0.063	0.067	0.075	2630	0.619	0.227
	2.39	0.051	0.059	0.063	0.068	0.078	2550	1.076	0.789
	2.73	0.057	0.063	0.069	0.074	0.084	2520	1.117	1.068
	3.06	0.064	0.069	0.076	0.082	0.093	2240	1.144	1.201
	3.40	0.070	0.076	0.083	0.089	0.099	1890	1.200	1.233
E	1.71	0.058	0.064	0.067	0.070	0.076	2700	0.032	0.012
	2.05	0.054	0.059	0.063	0.066	0.070	2840	0.228	0.051
	2.38	0.056	0.062	0.066	0.071	0.077	3050	0.731	0.261
	2.72	0.058	0.063	0.066	0.072	0.079	2970	1.203	0.719
	3.05	0.061	0.066	0.070	0.077	0.083	2710	1.370	1.200
	3.39	0.067	0.074	0.078	0.084	0.093	2320	1.371	1.460
F	1.68	0.069	0.076	0.076	0.082	0.087	3130	0.008	0.003
	2.02	0.066	0.073	0.075	0.080	0.085	3330	0.010	0.009
	2.35	0.062	0.070	0.071	0.077	0.082	3230	0.024	0.005
	2.69	0.059	0.067	0.068	0.075	0.081	3100	0.402	0.033
	3.03	0.057	0.065	0.068	0.074	0.081	3040	0.925	0.405
	3.36	0.060	0.069	0.071	0.078	0.085	2850	1.169	0.954
G	1.48	0.070	0.073	0.078	0.083	0.091	2740	0.009	0.009
	1.82	0.065	0.069	0.073	0.080	0.086	2890	0.012	0.006
	2.16	0.056	0.060	0.064	0.070	0.079	2870	0.154	0.011
	2.49	0.052	0.057	0.061	0.067	0.076	2870	0.473	0.113
	2.83	0.050	0.057	0.061	0.067	0.079	2640	0.953	0.422
	3.17	0.055	0.060	0.063	0.071	0.080	2670	1.129	0.793

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DETERMINATION OF OPTIMUM SO_3

The work of Lerch(1) indicated that the aqueous extract of paste or mortar made from cement containing its optimum percentage of SO_3 would at the age of 18 hours, contain a substantial amount of SO_3 (approaching saturation) but would at the age of 24 hours, contain only a small amount (indicating substantial depletion of the gypsum). In its report of May 5, 1953, to ASTM Committee C-1, the Working Committee on SO_3 content referred to the optimum content of SO_3 as that amount which in the extraction test yielded more than 0.2 gram of SO_3 per liter at 18 hours and less than 0.5 gram per liter at 24 hours. Usually there is a range in SO_3 content that will satisfy these criteria. In this report, the optimum, as determined by the extraction test, is taken as the mid-point of this range. Values were taken from straight-line graphs which are not reproduced in this report.

In tests of drying shrinkage, the percentage of SO_3 giving the least shrinkage is taken as the optimum. Usually the point of lowest shrinkage is clearly defined by the "curves" of Figures 1a and 1b and is consistent at all ages. In a few cases minimum shrinkage did not occur with the same percentage of SO_3 at all ages. The value selected for optimum with these

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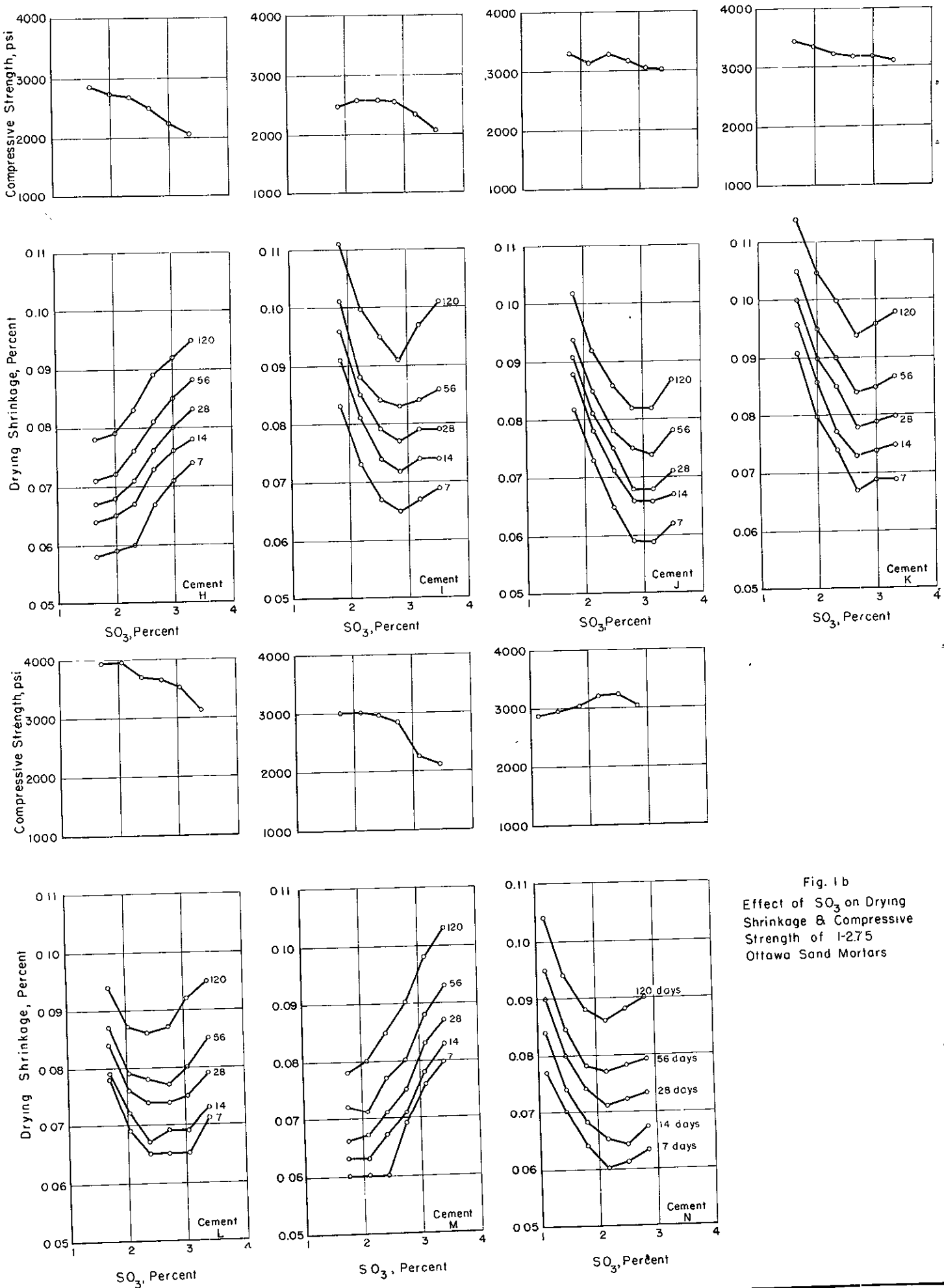

Fig. 1 b
Effect of SO_3 on Drying Shrinkage & Compressive Strength of 1-2.75 Ottawa Sand Mortars

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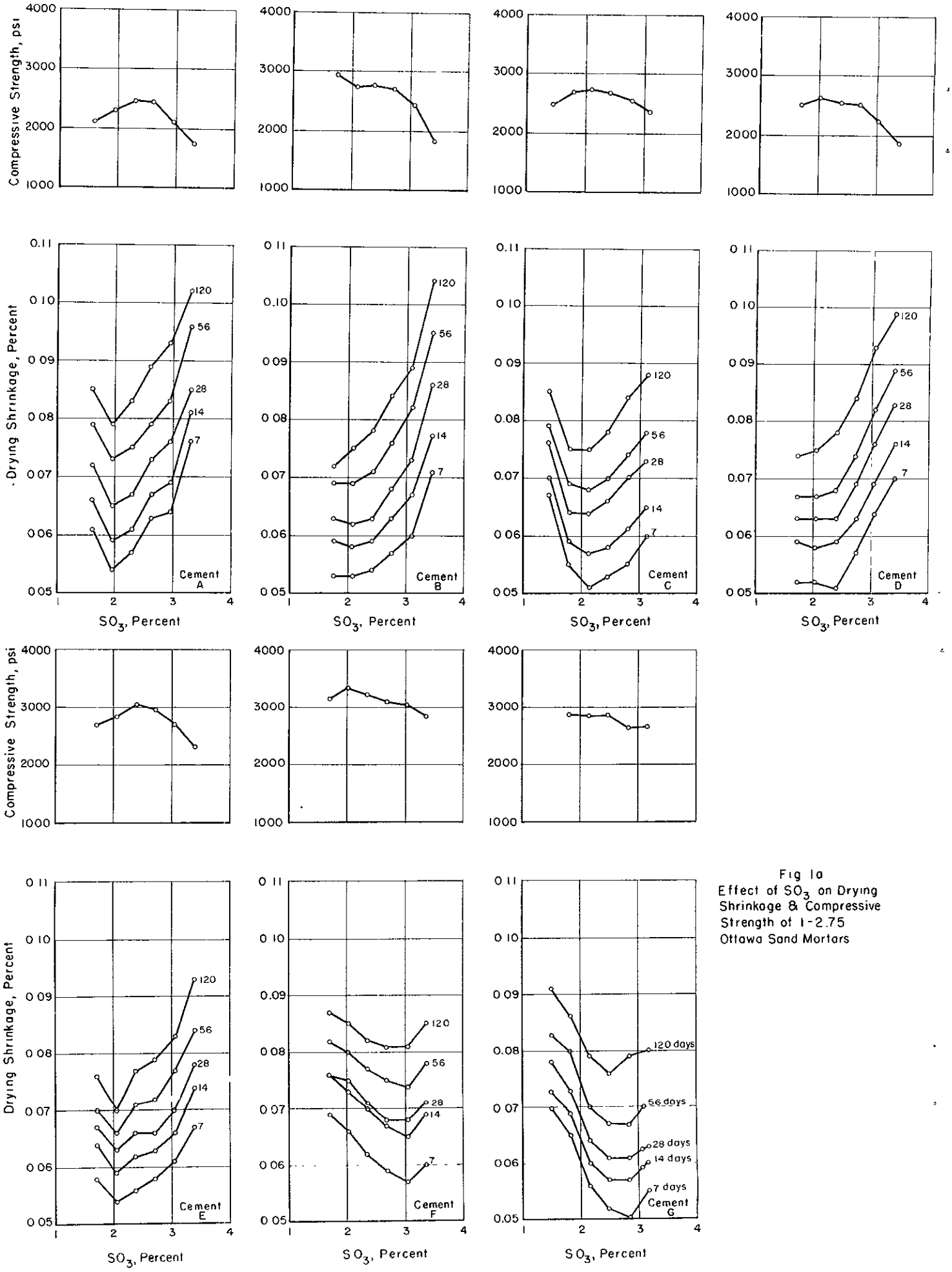

Fig 1a
Effect of SO_3 on Drying Shrinkage & Compressive Strength of 1-2.75 Ottawa Sand Mortars

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cements is that which most nearly satisfied the "curves" for all ages. It is evident that since additions of gypsum were arbitrary in amount, the absolute minimum in shrinkage may have occurred at slightly different percentages of SO_3 than those shown by the data. Finally, there is no certainty that cements "H" and "M" did not contain more than optimum SO_3 in the "as received" condition. The optimum values determined by drying shrinkage as shown in Table IV therefore represent the judgment of the writers and could be modified with some justification.

Optimum SO_3 for compressive strength is taken directly from the test data as the percentage showing the highest strength. In a number of cases the plotted "curves" of Figures 1a and 1b are quite flat and other values of SO_3 could have been selected with perhaps equal justification. It is probable that had the test data been more extensive, optimum values would have been indicated with greater certainty.

DISCUSSION

Bearing in mind the uncertainties discussed in the preceeding section, a study of Table III and Figures 1a and 1b indicates in general that there are well defined points of optimum SO_3 for minimum shrinkage and maximum strength of many of the cements.

Table IV lists optimum SO_3 values as determined by the three criteria of extraction, drying shrinkage and compressive strength. There are a few major discrepancies between optimums for shrinkage and strength which it is believed can be discounted because of lack of more precise data for strength.

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TABLE IV

Optimum SO_3 Content by Three Criteria

Cement	Optimum SO_3 in per cent		
	Extraction	Drying Shrinkage	Compressive Strength
A	2.16	1.95	2.28
B	2.09	2.09	1.75
C	2.24	2.13	2.13
D	1.98	2.06	2.06
E	2.29	2.05	2.38
F	2.77	3.03	2.02
G	2.56	2.49	1.82
H	1.89	1.65*	1.65
I	2.70	2.86	(2.19 (2.86
J	3.05	3.16	2.46
K	2.55	2.66	1.65
L	2.49	2.39	2.06
M	2.03	1.75*	2.09
N	2.31	2.15	2.49
Ave.	2.35	2.32	2.13

*The range in SO_3 used in the tests may not have included the percentage giving the lowest shrinkage for this cement.

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TABLE V

Reproducibility of Extraction Test for Optimum SO_3

Cement	Optimum SO_3, Percent Round Number 1	2	3	Avg.	Standard Deviation	Coefficient of Variation, %
A	2.21	2.24	2.02	2.16	0.119	5.51
B	2.07	2.16	2.03	2.09	0.067	3.21
C	2.24	2.27	2.21	2.24	0.030	1.34
D	1.96	2.07	1.92	1.98	0.078	3.94
E	2.26	2.36	2.24	2.29	0.064	2.79
F	2.75	2.80	2.75	2.77	0.029	1.05
G	2.56	2.58	2.53	2.56	0.025	0.98
H	*	1.90	1.88	1.89	0.014	0.74
I	2.78	2.69	2.62	2.70	0.080	2.96
J	3.10	3.04	3.02	3.05	0.042	1.38
K	2.63	2.41	2.60	2.55	0.119	4.67
L	2.58	2.44	2.46	2.49	0.076	3.05
M	2.02	2.03	2.04	2.03	0.010	0.49
N	2.39	2.27	2.27	2.31	0.069	2.99

*Error in chemical analysis

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TABLE VI

Highest Values in 24-hour Extraction Test
For Cements with Optimum SO_3

Cement	Optimum* SO_3 Percent	Maximum Result in 24 Hour Extraction, Grams per Liter
A	2.16	0.53
B	2.09	0.21
C	2.24	0.12
D	1.98	0.18
E	2.29	0.19
F	2.77	0.00
G	2.56	0.19
H	1.89	0.21
I	2.70	0.00
J	3.05	0.12
K	2.55	0.04
L	2.49	0.00
M	2.03	0.13
N	2.31	0.11

*Optimum is average value determined by 18-hr. and 24-hr. extraction tests

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The planned purpose of the study was to determine the effect of SO_3 on drying shrinkage of the cements tested and to determine the correlation with extraction tests. Attention is therefore focused on these results. The average value of optimum for the 14 cements is essentially the same by extraction and drying shrinkage (2.35% and 2.32%). For two cements the difference exceeded 0.25 per cent slightly. For the remainder the difference was less than this amount. It is indicated therefore, that the extraction tests yield dependable values of SO_3 content for minimum shrinkage.

Table V lists values of optimum SO_3 as indicated by individual extraction tests. The coefficients of variation indicate that the extraction test is reasonably reproducible in a single laboratory.

The proposed revision of ASTM specifications limits the SO_3 to a maximum of 0.50 gram per liter in the extract of hydrated mortar at 24 hours. Table VI shows the maximum value in three tests of 24-hour extract for the cements containing the optimum percentage of SO_3 (determined by the average of extraction tests). In only one instance did the result of a single test exceed the tentatively specified value of 0.50 gram per liter and the excess in this case was slight. The probability is high that test results will be well below the specified maximum when the cement contains the approximate optimum percentage of SO_3.

It was stated earlier that the first round of extraction tests (except for cement "M") was made in accordance with ASTM Designation C 265-51T and that the procedure during the second and third rounds was modified slightly. The reason advanced by the sponsor of the modifications was that they resulted in greater

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TABLE VII

Reduction in Drying Shrinkage Resulting from Increase in SO_3 to Optimum Arranged in Order of Increasing Drying Shrinkage at 7 Days Values of Optimum SO_3 Selected from Drying Shrinkage Data at all Ages

Cement	Cement as Received			Optimum SO_3			% Reduction in Shrinkage	
	% SO_3	Percent Drying Shrinkage		% SO_3	Percent Drying Shrinkage			
		7 days	120 days		7 days	120 days	7 da.	120 da.
D	1.72	0.052	0.074	2.06	0.052	0.075	0	--
B	1.75	0.053	0.072	2.09	0.053	0.075	0	--
E	1.71	0.058	0.076	2.05	0.054	0.070	7	8
H	1.65	0.058	0.078	1.65	0.058	0.078	0	0
M	1.75	0.060	0.078	1.75	0.060	0.078	0	0
A	1.61	0.061	0.085	1.95	0.054	0.079	11	7
C	1.44	0.067	0.085	2.13	0.051	0.075	24	12
F	1.68	0.069	0.087	3.03	0.057	0.081	17	7
G	1.48	0.070	0.091	2.49	0.052	0.076	26	17
L	1.72	0.078	0.094	2.39	0.065	0.086	17	9
J	1.82	0.082	0.102	3.16	0.059	0.082	28	20
N	1.13	0.077	0.104	2.15	0.060	0.086	22	17
I	1.85	0.083	0.111	2.86	0.065	0.091	22	18
K	1.65	0.091	0.114	2.66	0.067	0.094	26	18

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reproducibility. These tests do not provide data for computing relative deviations by the two methods. It is interesting to note, however, that the results were affected only slightly by the modifications. The grand average results were as follows:

	SO_3, grams per liter	
	18 hours	24 hours
Standard Method	0.65	0.44
Modified Method	0.72	0.50

Table VII shows the reduction in drying shrinkage after 7 and 120 days resulting from increasing the gypsum to the optimum percentage of SO_3 . All but two of the 14 cements contained less SO_3 than the optimum (determined by consideration of drying shrinkage at all periods). No improvement was found in four of the cements at the 7 and 120-day drying periods. For the remaining nine, the reduction in shrinkage was greater both numerically and in percentage at 7 days than at 120 days. The reduction at 7 days exceeded 20 per cent for 6 cements. Corresponding reductions at 120 days varied from 12 to 20 per cent for these cements. There are many concrete structures in which drying shrinkage would not be expected to approach the values obtained in the 120-day tests of these small mortar bars. The results of these tests at the earlier periods therefore indicate that reductions in drying shrinkage can be of considerable practical importance.

Shrinkage among the cements as received varied over the range of 0.052 per cent to 0.091 percent after 7 days of drying. When the SO_3 content was adjusted to the optimum the range

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in shrinkage was reduced considerably (to 0.51 - 0.067 percent).

The SO_3 content of all but two cements was below that required for least drying shrinkage (considering all periods of test). The deficiency varied from about 0.3 to 1.0 per cent. Optimum SO_3 for 11 of the cements is above that permitted under present specifications (ASTM Designation: C150-52).

CONCLUSIONS

Of 14 cements produced at 11 mills in California, all but two contained less SO_3 than the optimum amount to result in least drying shrinkage. The optimum percentage thus determined is greater for 11 cements than that permitted under ASTM Designation: C150-52.

With SO_3 at the optimum value, variation in drying shrinkage was reduced considerably below the range obtained in the cements as received.

Values of optimum SO_3 determined by extraction tests at 18 and 24 hours may be expected to indicate the optimum for least drying shrinkage within about 0.25 percentage point.

Limited tests indicate that highest compressive strength frequently results when the percentage of SO_3 is at the optimum for least drying shrinkage but with some cements variation in SO_3 content does not result in marked changes in compressive strength.

The proposed revision of ASTM Designation: C150-52 in which the maximum SO_3 content is defined by the 24 hour extraction test will permit manufacturers to increase the SO_3 to its

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optimum value with assurance that the specification limit will not be found to have been exceeded if tests have been made with reasonable care.

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