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EVALUATION OF TEST METHODS FOR DETERMINATION OF NORMAL CONSISTENCY AND SETTING TIME OF PORTLAND CEMENT PASTE

PART - II

by

N. G. ZOLDNERS AND G. G. CARETTE

Mineral Processing Division

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Mines Branch Investigation Report IR 68-69

EVALUATION OF TEST METHODS FOR DETERMINATION OF NORMAL CONSISTENCY AND SETTING TIME OF PORTLAND CEMENT PASTE PART - II

by

N. G. Zoldners* and G. G. Carette**

SUMMARY

The test results revealed negligible difference in the setting-time values obtained by using either the mechanical- or the delayed mechanical-mixing methods on normal- and false-setting cements. Both the initial and final sets occurred a little earlier when using the delayed mixing, but the difference never exceeded 10 minutes.

The "between-lab" variations were about the same for the two mixing methods and were generally within satisfactory limits. The average C. V. values for both mixing methods and all types of cement were about 6 and 14 per cent for the initial and final sets, respectively.

The "within-lab" variations were also of the same order for the mechanical- and the delayed mechanical-mixing methods. The uniformity of the results for individual laboratories was generally good, most of the C. V. values being under 5 per cent.

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CONTENTS

TABLES

INTRODUCTION

Subcommittee "B" on Physical Requirements and Test Methods, of the Committee on Hydraulic Cements, Canadian Standards Association (CSA), conducted in 1967 a round-robin laboratory investigation. Different test methods for determination of normal consistency and setting time of portland cement paste were evaluated. The Mines Branch was requested by the Committee to act as an impartial centre for processing and analysing the test data submitted by the participating laboratories.

A summary of the test data was presented at the subcommittee "B" meeting in Winnipeg, Manitoba, October 16, 1967 (1). The results indicated that the reproducibility of the setting-time test results was better when the Vicat apparatus and a mechanical mixing procedure was used. It was decided at that meeting that this investigation should be continued and another series of tests should be organized, eliminating hand-mixing procedure and Gillmore test method.

This report covers the second part of the investigation in which the two mechanical mixing methods, using the Vicat apparatus, were studied.

PREPARATION AND DISTRIBUTION OF TEST SAMPLES

10-lb test samples of four types of cements, having different setting characteristics shown below, were prepared by Messrs. C. Beique (Miron Company Ltd) and W.S. Weaver (Canada Cement Company, Ltd) and distributed to the nine participating laboratories (Appendix A).

A. High-Early-Strength Portland Cement (Canada Cement Company Limited, Plant No. 1, Montreal East, Que.)

B. False-Setting Portland Cement (Miron Company Limited, Montreal, Que.)

C. Normal Portland Cement (Canada Cement Company Limited, Plant No. 8, Port Colborne, Ont.)

D. False-Setting Portland Cement (Miron Company Limited, Montreal, Que.)

Care was taken that the distributed test samples were as nearly identical as possible. Preparation of samples was carried out in accordance with the procedure given in Appendix B.

TESTING PROCEDURE

On each of the four cement samples the normal-consistency and setting-time determinationswere made using both normal mechanical- and delayed mechanical-mixing procedures.

1. Normal Consistency

Normal-consistency determinations were made in accordance with CSA Standard A5-1961, Section 7.1.4 in which the Clause 7.1.4.1 was modified as follows:

a) For mechanical mixing - see Appendix C (as per CSA formal revision No. 14, October, 1967).

b) For delayed mechanical mixing (re-mixing), see Appendix D.

2. Time of Setting

Time of setting determinations were made using the Vicat method in accordance with CSA Standard A5-1961 Section 7.5 in which Clause 7.5.2.1 was modified as shown above, for use with mechanical mixing (Appendix C) and delayed mechanical mixing (Appendix D).

Both initial and final setting times were determined using the Vicat apparatus. For each condition of preparation (i.e. mechanical and delayed mechanical mixing), three Vicat tests were made on three different days. For each group of three tests, the first Vicat test was done on the batch used for the normal-consistency determination. The other two were done on pastes prepared with the same water/cement ratio as used in the first test.

In summary, 12 Vicat tests were made (initial and final, repeated on three days and for two methods of mixing).

TEST RESULTS

After completion of all tests, the test data were forwarded to the Mines Branch in Ottawa for evaluation. The data are compiled in Tables I to IV, Appendix E.

The data were analysed using standard statistical methods. The summary of statistical analyses of setting time is given in Tables 1 and 2 showing "between-lab" and "within-lab" variations for each type of cement and mixing method.

TABLE 1

Summary of Statistical Analyses of Results of Tests for Setting Time

(between-laboratory variations)

Type of Cement	Mixing Procedure	Setting Time	N*	Maximum	Minimum	Average	Standard Deviation	Coefficient of Variation, %
				hr:min	hr:min	hr:min	hr:min	
A (H.E.S.)	Mechanical	Initial	9	2:55	2:25	2:40	0:11	6.6
		Final	9	4:50	3:35	4:18	0:27	10.4
	Delayed	Initial	8	3:00	2:30	2:40	0:12	7.5
		Final	8	4:35	3:35	4:10	0:23	9.2
B (False-Setting)	Mechanical	Initial	9	3:25	2:45	3:08	0:12	6.2
		Final	9	6:05	4:00	5:01	0:44	14.7
	Delayed	Initial	9	3:20	2:55	3:05	0:09	5.1
		Final	9	5:55	3:45	4:53	0:46	15.6
C (Normal)	Mechanical	Initial	9	2:55	2:25	2:41	0:10	6.2
		Final	9	5:25	3:25	4:29	0:43	15.8
	Delayed	Initial	9	2:50	2:25	2:38	0:09	5.5
		Final	9	5:10	3:25	4:24	0:36	13.7
D (False-Setting)	Mechanical	Initial	9	3:25	2:50	3:09	0:10	5.5
		Final	9	6:00	3:40	4:59	0:45	15.1
	Delayed	Initial	9	3:20	2:50	3:05	0:10	5.4
		Final	9	5:50	3:30	4:49	0:43	15.0

* Number of Participants

TABLE 2

Summary of Statistical Analyses of Results of Tests for Setting Time

(within-laboratory variations)

Type of Cement	Setting Time	Average Coefficient of Variation, %	
		Mechanical Mixing	Delayed Mixing
A (H.E.S.)	Initial	3.7	4.8
	Final	4.0	3.6
B (False-Setting)	Initial	3.1	3.2
	Final	4.4	1.8
C (Normal)	Initial	5.4	3.6
	Final	3.3	2.1
D (False-Setting)	Initial	4.3	4.4
	Final	3.4	4.6

DISCUSSION

For each of the four samples of cement, both the mechanical- and the delayed-mixing methods, produced similar setting-time values. As observed previously (1), the delayed-mixing method generally showed a slight decrease in both the initial and final times of set. However, the maximum difference between average values of setting time for each method was only 10 minutes and it is doubtful if decrease of this magnitude is of any practical significance.

Coefficients of variation (C.V.) for "between-lab" test results were excellent for the initial set, using either mixing procedure; the maximum C.V. value for any type of cement was 7.5 per cent (Table 1). For the final set, the variations were, however, larger; the values of C.V. ranged from 9.2 per cent for cement "A" (H.E.S.) to 15.8 per cent for cement "C" (Normal). The false-setting characteristics of a cement were found to have no appreciable effect upon the "between-lab" variations. The C.V. values for each mixing method did not differ by more than 1.1 per cent for the initial set and 2.1 per cent for the final set.

"Within-lab" variations were also generally small for each type of cement and mixing method. The individual C.V. values were generally below 5 per cent and never exceeded 10 per cent, except for one set of tests with cement "A" where the C.V. values ranged between 10 and 20 per cent for both methods (Appendix E, Table I, lab No. 7). The "within-lab" variations of the results are not appreciably affected by the mixing procedure. The maximum difference between the two methods was found for cement "C" (Normal) where the average C.V. values for the initial and final sets were 5.4 and 3.3 per cent, respectively, for the mechanical mixing compared with 3.6 and 2.1 per cent for the delayed mixing. However, no specific trend was observed for any cement. Since the magnitude of variation was relatively low in all instances, the difference may be considered insignificant.

CONCLUSION

The mechanical- or delayed-mixing method has no significant effect upon the determination of setting time of normal- and false-setting cements. The "within-lab" and "between-lab" variations are within acceptable limits for both mixing methods. Comparison of the test results with those obtained in a previous investigation on setting time (1) shows that mechanical mixing does eliminate some large variations experienced in hand mixing for cements having false-setting characteristics. However, from this present investigation, it does seem that either of the two mechanical mixing methods is equally suitable.

REFERENCE

1. N.G. Zoldners and G.G. Carette, "Evaluation of Test Methods for Determination of Normal Consistency and Setting Time of Portland Cement Paste - Part-I", Mines Branch Investigation Report IR 68-57, 1968.

APPENDIX A

PARTICIPATING LABORATORIES

1. Canada Cement Company, Limited, Plant No. 1, Montreal East, Que.
   (C.J. Fremming, Chemist)

2. Inland Cement Industries Limited, Regina, Sask.
   (L. Marchinko, Chemist)

3. Lake Ontario Cement Limited, Box 620, Picton, Ont.
   (R.A. Dersnah, Mgr., Technical Development)

4. Miron Company Ltd., 2201 Jarry Street East, Montreal 38, Que.
   (C. Beique, Director of Research)

5. Ocean Cement Limited, Bamberton Road, Cobble Hill P.O., B.C.
   (F.A. DeLisle, Chemist)

6. St. Lawrence Cement Co., Box 520, Clarkson, Ont.
   (J. Laneuville, Chemist)

7. Ontario Hydro, 200 Kipling Ave. S., Toronto 18, Ont.
   (T.G. Clendenning, Masonry Research Engineer)

8. Department of Public Works, Ottawa, Ont.
   (N.E. Laycraft, Chief, Testing Laboratories)

9. Ontario Research Foundation, Sheridan Park, Ont.
   (R.A. Kuntze, Assistant Director, Dept. of Materials Chemistry)

APPENDIX B

PROCEDURE FOR PREPARATION OF CEMENT SAMPLES

(as prepared by Mr. R.W. Smith)

1) Secure two* bags of the cement to be issued.

2) Spread a clean, dry, rubber sheet, or equivalent (approximately 6' square) on the laboratory floor.

3) Screen the contents of the two bags through a 20-mesh sieve onto the rubber sheet in alternate sequence, that is, sieving approximately five-pound increments of cement from one bag and then the other in alternate sequence.

4) Blend the cement on the sheet by rolling the cement from one side of the sheet to the other and then in a horizontally opposite direction and subsequently from one corner to the other and then in the diagonally opposite direction. Employ rotary sequence of blending for ten minutes.

5) Heap the cement in the centre of the sheet and then divide the mound into quarters.

6) Remove diagonally opposite quarters placing this cement into a clean dry container.

7) Reblend the remaining half for five minutes.

8) Form a mound of cement diagonally across the sheet and divide into eight portions.

9) Approximately half fill (about six pounds) eleven 1-gallon cans with small increments taken in sequence from the eight portions.

10) Place the cement previously set aside (operation 6) on the rubber sheet together with the residue after half-filling eleven cans.

11) By repeating operations 7, 8 and 9, complete the filling of the cans so that each can contains a sample weighing approximately twelve pounds.

*If more than ten samples are to be issued, three bags of cement should be secured for preparation of the samples. In such instances, divide the mound mentioned in operation five into sixths and proceed to fill the cans in micro-increments of thirds rather than halves as detailed above.

APPENDIX C

PROPOSED MECHANICAL MIXING PROCEDURE

(as per Mr. C. Beique's letter and enclosure of Dec. 5, 1966)

A-5 Specification

7.1.4 Determination of Normal Consistency

7.1.4.1 Mixing Cement Pastes. A 500-g sample of dry cement shall be mixed with a measured amount of clean water in a mechanical mixer in accordance with the method described below.

a) Mixer. The mixer, paddle, mixing bowl and scraper shall conform with the requirements of clause 7.8.4.3 para. a, b, c, d.

b) Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for a batch into the bowl and mix in the following manner:

1- Place all the mixing water in the bowl,

2- Add the cement to the water and allow 30 sec for the absorption of the water,

3- Start the mixer and mix at slow speed (140 ± 5 rpm) for 30 sec,

4- Stop the mixer for 15 sec. and during this time scrape down into the batch any paste that may have collected on sides of the bowl,

5- Start the mixer at medium speed (285 ± 10 rpm)and mix for 1 min.

APPENDIX D

PROPOSED METHOD FOR THE DELAYED REMIX PROCEDURE

(Mechanical mixing procedure modified as per Mr. R.W. Smith's letter of Nov. 15, 1966)

Modification for C305-59T.

Section 5 -

Steps (1) to (5) - Carry out as detailed.

Add the following:

Step (6) - Determine the penetration for the normal-consistency test (ASTM C187-58) sections 4(b) and (c).

(7) - Within a 30-second interval, return the paste for the N.C. test to the mixing bowl, scrape the sides of the bowl with the rubber scraper.

(8) - Mix at medium speed for one (1) minute.

(9) - Repeat Step (6) for the normal-consistency test (ASTM C187-58).

Significance of N.C. penetration tests.

(1) If the cement has no false set characteristics, the second penetration will probably be the same or about one (1) mm. less than the first penetration test. This seems to have no apparent effect on the Vicat setting times.

(2) If the second penetration test is greater than the first test, this indicates false set and the water requirement should be adjusted to give the proper penetration based on the remix procedure.

APPENDIX E

NORMAL CONSISTENCY AND TIME OF SETTING TESTS
(CSA 2nd Series)

COMPILATION OF TEST RESULTS

Table I - Cement "A" (H.E.S. portland)

Table II - Cement "B" (false-setting)

Table III - Cement "C" (normal portland)

Table IV - Cement "D" (false-setting)

TABLE I

CSA Tests
2nd Series

Cement "A"
(H.E.S. portland)

Lab Code	Normal Consistency			Test No.	Time of Setting (hr:min)			
	Mixing Method	Water-Cement Ratio, %	Rod Penetration (mm)		Mechanical Mixing (a)		Delayed-Mechan. Mixing (b)	
					Initial	Final	Initial	Final
1	a	27.8	9.0	1	2:40	4:40	2:40	4:30
	b	27.4	9.0	2	2:40	4:40	2:25	4:25
				3	2:40	4:40	2:30	4:25
				Average	2:40	4:40	2:30	4:25
2	a	28.6	-	1	2:50	4:45	2:30	4:20
	b	27.8	-	2	2:45	4:40	2:30	4:10
				3	2:55	5:00	2:40	4:40
				Average	2:50	4:45	2:35	4:25
3	a	28.4	-	1	2:30	4:05	2:35	4:10
	b	27.2	-	2	2:35	4:15	-	-
				3	2:30	4:20	-	-
				Average	2:30	4:15		
4	a	27.7	9.0	1	2:50	4:45	2:45	4:35
	b	27.1	9.5	2	2:50	4:50	2:50	4:40
				3	2:55	4:40	2:40	4:30
				Average	2:50	4:45	2:45	4:35
5	a	27.0	9.0	1	2:30	3:40	2:30	3:45
	b	27.0	9.0	2	2:30	3:45	2:30	3:45
				3	2:30	3:35	2:30	3:35
				Average	2:30	3:40	2:30	3:40
6	a	27.5	10.0	1	2:40	4:15	2:40	4:00
	b	27.0	11.0	2	2:35	4:15	2:35	4:00
				3	2:35	4:00	2:35	4:00
				Average	2:35	4:10	2:35	4:00
7	a	-	-	1	2:35	4:30	2:30	4:20
	b	-	-	2	3:15	5:00	3:35	5:00
				3	2:30	4:00	2:40	4:05
				Average	2:45	4:30	2:55	4:30
8	a	28.4	-	1	2:55	3:20	2:55	3:20
	b	28.4	-	2	2:55	3:40	3:05	3:40
				3	3:00	3:40	3:00	3:40
				Average	2:55	3:35	3:00	3:35
9	a	28.0	9.5	1	2:25	4:00	2:30	4:10
	b	28.2	10.0	2	2:15	4:15	2:35	4:15
				3	2:35	4:25	-	-
				Average	2:25	4:15		

TABLE II

CSA Tests
2nd Series

Cement "B"
(false-setting)

Lab Code	Normal Consistency			Time of Setting (hr:min)				
	Mixing Method	Water-Cement Ratio, %	Rod Penetration (mm)	Test No.	Mechanical Mixing (a)		Delayed-Mechan. Mixing (b)	
					Initial	Final	Initial	Final
1	a	25.0	9.0	1	3:05	5:00	2:55	4:55
	b	24.8	11.0	2	3:05	5:10	2:50	5:00
				3	3:20	5:40	2:55	4:55
				Average	3:10	5:15	2:55	4:55
2	a	25.2	-	1	3:20	5:55	3:15	5:45
	b	25.2	-	2	3:15	5:50	3:10	5:45
				3	3:20	5:45	3:20	5:45
				Average	3:20	5:50	3:15	5:45
3	a	25.0	-	1	3:20	6:10	3:20	5:40
	b	25.0	-	2	3:10	5:45	3:15	5:40
				3	3:15	5:20	3:15	5:35
				Average	3:15	5:45	3:15	5:40
4	a	24.4	9.5	1	3:30	6:10	3:25	5:55
	b	24.0	9.0	2	3:25	6:05	3:15	5:50
				3	3:20	5:55	3:15	5:55
				Average	3:25	6:05	3:20	5:55
5	a	24.5	9.5	1	3:10	4:15	3:05	4:05
	b	24.5	9.5	2	3:10	4:15	3:00	4:00
				3	3:10	4:10	3:00	4:00
				Average	3:10	4:15	3:00	4:00
6	a	24.5	9.0	1	3:05	4:50	3:00	4:40
	b	24.5	11.0	2	3:10	4:45	3:05	4:30
				3	3:00	4:30	3:00	4:30
				Average	3:05	4:40	3:00	4:35
7	a	-	-	1	2:50	4:30	3:05	4:30
	b	-	-	2	3:00	4:50	3:15	4:50
				3	3:10	4:40	2:55	4:40
				Average	3:00	4:40	3:05	4:40
8	a	23.8	-	1	3:10	4:10	3:05	3:50
	b	23.8	-	2	3:15	4:10	3:05	3:50
				3	2:50	3:40	2:50	3:40
				Average	3:05	4:00	3:00	3:45
9	a	24.5	10.0	1	2:40	4:40	2:40	4:35
	b	24.5	9.0	2	2:45	4:20	3:00	4:45
				3	2:45	5:00	3:00	5:00
				Average	2:45	4:40	2:55	4:45

TABLE III

CSA Tests
2nd Series

Cement "C"
(normal portland)

Lab Code	Normal Consistency			Time of Setting (hr:min)				
	Mixing Method	Water-Cement Ratio, %	Rod Penetration (mm)	Test No.	Mechanical Mixing (a)		Delayed-Mechan. Mixing (b)	
					Initial	Final	Initial	Final
1	a	24.0	10.0	1	2:40	5:05	2:25	4:40
	b	22.6	9.0	2	2:40	5:00	2:25	4:40
				3	2:50	5:00	2:25	4:40
				Average	2:45	5:00	2:25	4:40
2	a	23.4	-	1	3:00	5:25	2:35	5:00
	b	23.0	-	2	2:50	5:00	2:35	4:45
				3	2:35	5:00	2:35	5:00
				Average	2:50	5:10	2:35	4:55
3	a	23.0	-	1	2:45	5:15	2:40	5:00
	b	23.0	-	2	2:55	5:30	2:50	5:15
				3	3:00	5:30	-	-
				Average	2:55	5:25		
4	a	22.8	9.5	1	3:00	5:10	2:45	4:55
	b	22.2	9.5	2	2:40	4:55	2:50	5:00
				3	2:55	5:00	2:55	4:55
				Average	2:50	5:00	2:50	4:55
5	a	22.5	9.0	1	2:30	3:35	2:25	3:40
	b	22.5	9.0	2	2:25	3:40	2:20	3:40
				3	2:30	3:35	2:25	3:35
				Average	2:30	3:35	2:25	3:40
6	a	23.0	9.5	1	2:35	4:05	2:40	4:05
	b	22.5	10.5	2	2:50	4:15	2:45	4:15
				3	2:40	4:15	2:40	4:15
				Average	2:40	4:10	2:40	4:10
7	a	-	-	1	2:25	4:15	2:25	4:05
	b	-	-	2	2:50	4:20	2:55	4:10
				3	2:25	3:50	2:35	4:00
				Average	2:35	4:10	2:40	4:05
8	a	22.0	-	1	2:45	3:30	2:50	3:30
	b	22.0	-	2	2:45	3:30	2:45	3:20
				3	2:35	3:20	2:35	3:20
				Average	2:40	3:25	2:45	3:25
9	a	23.0	9.0	1	2:15	4:05	2:25	4:40
	b	23.0	11.0	2	2:20	4:25	2:40	4:50
				3	2:35	4:50	2:40	4:25
				Average	2:25	4:25	2:35	4:40

TABLE IV

CSA Tests
2nd Series

Cement "D"
(false-setting)

Lab Code	Normal Consistency			Time of Setting (hr:min)				
	Mixing Method	Water-Cement Ratio, %	Rod Penetration (mm)	Test No.	Mechanical Mixing (a)		Delayed-Mechan. Mixing (b)	
					Initial	Final	Initial	Final
1	a	25.5	11.0	1	3:10	5:20	2:50	5:00
	b	24.8	10.0	2	3:10	5:20	2:50	5:00
				3	3:25	5:35	2:55	5:05'
				Average	3:15	5:25	2:50	5:00
2	a	24.6	-	1	3:15	5:30	3:00	5:10
	b	24.6	-	2	3:10	5:40	3:05	5:15
				3	3:10	5:45	3:10	5:50
				Average	3:10	5:40	3:05	5:25
3	a	24.8	-	1	3:15	5:25	3:10	5:15
	b	24.8	-	2	3:05	5:35	3:15	5:40
				3	3:00	5:35	3:10	5:35
				Average	3:05	5:30	3:10	5:30
4	a	24.4	10.0	1	3:30	6:00	3:20	5:50
	b	24.2	9.5	2	3:20	5:55	3:15	5:55
				3	3:25	6:05	3:25	5:40
				Average	3:25	6:00	3:20	5:50
5	a	24.5	9.5	1	3:15	4:10	3:10	4:10
	b	24.5	9.5	2	3:10	4:10	3:10	4:10
				3	3:10	4:05	3:05	4:05
				Average	3:10	4:10	3:10	4:10
6	a	24.5	10.0	1	3:15	5:00	3:15	4:45
	b	24.5	10.0	2	3:00	4:30	2.55	4:15
				3	3:10	4:30	3:00	4:30
				Average	3:10	4:40	3:05	4:30
7	a	-	-	1	3:35	4:50	3:25	4:45
	b	-	-	2	3:00	4:50	3:35	4:50
				3	3:20	4:35	2:50	4:20
				Average	3:20	4:45	3:15	4:40
8	a	23.6	-	1	3:05	3:40	3:05	3:40
	b	23.6	-	2	3:10	3:45	2:55	3:35
				3	2:45	3:35	2:45	3:10
				Average	3:00	3:40	2:55	3:30
9	a	24.5	9.0	1	2:50	4:50	2:40	4:50
	b	24.5	10.0	2	2:45	4:35	3:00	4:25
				3	3:00	5:30	3:00	5:05
				Average	2:50	5:00	2:55	4:45