METHOD OF TEST FOR EVALUATION OF SUPERPLASTICIZING ADMIXTURES FOR CONCRETE

1. SCOPE

This method covers physical requirements and qualification tests for superplasticizing admixtures used for:

1.1 High range water reduction and strength increase; and

1.2 Production of flowing concrete.

1.3 These tests are based on arbitrary stipulations that make possible highly standardized testing in the laboratory and are not intended to simulate actual job conditions.

Note 1: This method is based on CSA A266.6-M 85, Superplasticizing admixtures for Concrete which is not currently in existence as a CSA specification.

2. RELEVANT DOCUMENTS

2.1 MTO Test Methods LS-401, LS-407, LS-422, LS-481

2.2 CSA Standards A5, A23.1, A23.2-12A, A23.2-4C, A23.2-5C, A23.2-6C

2.3 ASTM C 157

2.4 ASTM C 403

2.5 ASTM C 457

2.6 ASTM C 666

2.7 ACI Standard Practice 211.1

3. DEFINITIONS

3.1 Non-flowing concrete: Concrete of slump 20 to 180 mm

3.2 Flowing concrete: Concrete of slump greater than 180 mm

3.3 Type SPN superplasticizers: Superplasticizing admixtures with normal setting characteristics.

3.4 Type SPR superplasticizers: Superplasticizing admixtures with set retarding characteristics.

4. PHYSICAL REQUIREMENTS

4.1 The admixture shall be evaluated by comparing the properties of a test concrete containing the admixture with the properties of a reference concrete containing similar materials without the admixture. The test concrete and the reference concrete shall be air entrained. When prepared and

tested in accordance with this test method, the test concrete shall meet the requirements for nonflowing concrete or flowing concrete, or both, as specified in Table 1.

Table 1

Physical Requirements

		Nonflowing concrete		**Flowing Concrete**	
Property	**Calculation**	**SPN type**	**SPR type**	**SPN type**	**SPR type**
Water Content Maximum, %	T/R x 100	88	88	NA	NA
Slump retention of test mix after 20 min	T final / T initial x 100	Final slump of the test mix shall be at least 50 % of initial slump			
Initial Set Minimum, hours:min. Maximum, hours:min.	T - R	-1:20 +1:20	+1:00 +3:00	-1:20 +1:20	+1:00 +3:00
Compressive Strength Minimum, % 1 day 3 days 7 days 28 days 6 months 1 year	T/R x 100 x 1.05	 150 130 125 120 100 100	 130 130 125 120 100 100	 100 100 100 100 100 100	 100 100 100 100 100 100
Length Change	If R exceeds 0.030 %, T/R shall not exceed 1.35. If R is not more than 0.030 %, T-R shall not exceed 0.010 %				
Air-Void Spacing Factor Maximum, mm	T	0.23	0.23	0.23	0.23
Relative Durability Factor Minimum,%	T/R x 100 x 1.10	100	100	100	100

5. CONCRETE MATERIALS

The materials used for the preparation of concrete shall be uniform in quality, properly blended where required and prepared in sufficient quantity so that one lot of material is used for the entire test program.

5.1 CEMENT: Cement shall meet the requirements of CSA Standard A5, Portland Cements, and shall be a blend of equal parts of Normal Portland Cement (Type 10) from three different mills, St. Lawrence, Mississauga, Ontario, St. Marys Cement Company, St. Marys, Ontario, and Essroc

Italcementi Group, Picton. Ontario. When the blend of cements is tested for early stiffening in accordance with CSA Standard A5 (Appendix A), the penetration shall be at least 50 %.

5.2 WATER: The water used in the preparation of mixes shall be drinkable water approved by the Ministry of Health.

5.3 AGGREGATE: All aggregate used in the preparation of concrete mixes shall meet the requirements of CSA Standard A23.1, except as specified herein.

Aggregate shall meet the grading requirements of Table 2 and shall be batched in a water saturated condition which shall be attained by soaking the aggregate, completely submerged, for 24 hours in water. To meet the grading requirements specified, it is necessary to sieve the coarse aggregate through the 26.5, 19.0, 13.2, 9.5 and 4.75 mm sieves and recombine the material to the specified grading.

Table 2

Aggregate Grading Requirements

Type	**Sieve Size**	**Percent Passing**	**(Alternative)**
Fine Aggregate	4.75 mm	95 -100	
	1.18 mm	65 - 80	
	300 µm	10 - 30	
	150 µm	2 - 10	
Coarse Aggregate	26.5 mm	100	(100)
	19.0 mm	80	(75)
	13.2 mm	50	(50)
	9.5 mm	30	(25)
	4.75 mm	0	(0)

5.4 AIR ENTRAINING ADMIXTURE: The air entraining admixture shall conform to the requirements of MTO Laboratory Test Method LS-422, Method of Test for Evaluation of Air Entraining Admixtures for Concrete.

5.5 MIXTURE TEMPERATURE: The materials shall be conditioned so as to ensure a uniform concrete temperature in the range of 17 - 22°C.

6. CONCRETE MIX PROPORTIONS

6.1 PROPORTIONING: The same mix proportions shall be used in all batches of both test and reference concrete. MTO Laboratory Test Method LS-481, Selecting Proportions for MTO Structural and Pavement Concrete (ACI Standard 211.1, Recommended Practice for Selecting Proportions for Normal and Heavyweight Concrete) shall be used as a proportioning guide.

6.2 NONFLOWING CONCRETE: The water content of the reference concrete shall be that which produces a slump in the range of 80 - 100 mm. The water content of the test concrete shall be not more than 88 % of the water content of the reference concrete, and the slump of the test concrete after addition of the superplasiticizing admixture shall equal or exceed the slump of the reference concrete, but shall not exceed 180 mm.

6.3 FLOWING CONCRETE: The water content of the reference concrete shall be that which produces a slump in the range of 80 - 100 mm. The test concrete shall have the same water content as the reference concrete and, after addition of the superplasticizing admixture, its slump shall be in the range of 180 - 240 mm. If these proportions result in segregation of the test concrete, the fine aggregate content shall be progressively increased until a satisfactory mix is obtained. This mix becomes the test concrete and the proportions of the reference concrete remain unchanged.

6.4 AIR CONTENT: The air content range shall be 5 - 7 %, and the air contents of the test and reference concretes shall not differ by more than 0.5 %. The admixture under test shall be added in the amount necessary to comply with the applicable requirements for water reduction, initial set, and slump.

7. MIXING OF CONCRETE

The following mixing procedure shall be followed:

7.1 Ensure that the mixer is buttered either by mixing a butter batch similar to the reference concrete or through having discharged another test batch just previously.

7.2 Prior to starting the mixer, place about 1/2 the mixing water, the saturated coarse aggregate, the solution of air entraining admixture (but not the superplasticizing admixture), the saturated fine aggregate, and the cement in the mixer in that order. The air entraining admixture shall be diluted with part of the mixing water.

7.3 Start the mixer and mix for 30 ± 3 seconds.

7.4 During the next 90 ± 3 seconds mixing shall be continued, while, in the case of reference or flowing test concrete, the remainder of the mixing water is being added (see note). For a nonflowing, water-reduced test concrete, water shall be added to bring the total amount to about 70 % of the total used for the reference concrete. If this fails to achieve complete wetting of the cement, which is essential to the proper functioning of a superplasticizing admixture, the amount of water shall be increased gradually until the cement is wetted to the point where all the ingredients can properly mix.

Note 2: In the case of reference concrete, an experienced operator may add water incrementally during mixing to adjust to the desired slump.

7.3 The superplasticizing admixture shall be added to the test concrete over a period of 10 to 20 s and mixing continued for a further 120 ± 3 seconds.

7.4 The mixer shall then be stopped for 180 ± 3 seconds. During this rest period the mixer shall be covered with damp burlap or other suitable material to minimize evaporation.

7.5 Mixing shall be resumed for 60 ± 3 seconds.

7.6 The mixer shall then be stopped and the concrete left undisturbed for 120 ± 3 seconds before removing any for testing. During this period the mixer shall be covered as during the previous rest period. The total mixing time shall be 5 minutes, with 2 rest periods totalling approximately 5 minutes.

8. TEST PROGRAM

8.1 Each of the properties listed in Table 1, except the air-void spacing factor, shall be determined from each round of at least three rounds of tests. A round of tests shall consist of sufficient batches to provide the required number of test and reference specimens. Reference and test mixes for comparison purposes shall always be made on the same day. When the repeatability within a round of tests or the reproducibilty over three consecutive rounds of tests is judged unsatisfactory, additional rounds of tests shall be performed until satisfactory repeatability and reproducibility are obtained.

8.2 The values for water content, compressive strength, time of initial set, length change, and relative durability factor shall be those obtained by averaging the results from three consecutive rounds of tests and applying the appropriate correction factors.

9. TEST PROCEDURE

9.1 FRESH CONCRETE

9.1.1 Water content: The water content shall be defined as the total effective water and shall include added water, surface moisture on aggregate and any water contained in the admixtures. It shall be calculated according to the following equation for each batch:

$$W_T \text{ (or } W_R) = W_A + W_B + W_C - W_D\,(1+A/100) - W_E\,(1+B/100)$$

where:

W_T = water content for test concrete

W_R = water content for reference concrete

W_A = batch mass of saturated aggregate

W_B = batch mass of added water

W_C = mass of water contained in all admixtures

W_D = oven dry mass of coarse aggregate

W_E = oven dry mass of fine aggregate

A = absorption percent of coarse aggregate as defined in CSA Test Method A23.2-12A

B = absorption percent of fine aggregate defined in CSA Test Method A23.2-6C

9.1.2 Slump: The initial and final slump shall be determined for each batch of reference or test concretes in accordance with CSA Test Method A23.2-5C, Slump of Concrete. Initial slump shall be determined not sooner than 3 minutes nor later than 5 minutes after the final rest period following mixing of the concrete. After the concrete has been allowed to remain undisturbed for 20 minutes with the ambient air temperature in the range of 23 ± 2°C, the concrete shall be remixed to ensure uniformity and the final slump determined. The moulding of test specimens shall commence only after the final slump has been determined.

9.1.3 Air content: The air content of the concrete shall be determined for each batch concurrently with the final slump test in accordance with CSA Test Method A23.2-4C, Air Content of Plastic Concrete by the Pressure Method. Meters designed to provide air content measurements as a function of pressure change shall have dials with each division representing not more than 0.1 % air content in the range of 5 - 7 %, and shall be accurately calibrated by positive displacement methods in this range.

9.1.4 Density: The density shall be determined in accordance with CSA Test Method A23.2-6C, Density, Yield and Cement Factor of Plastic Concrete. The volume of concrete tested shall be not less than 14 L.

9.1.5 Time of initial set: The time of initial set shall be determined in accordance with ASTM Standard C403, Time of Setting of Concrete Mixtures by Penetration Resistance. The test shall be conducted at 23 ± 2°C and the penetration resistance apparatus used shall be of the bench or floor mounted hydraulic or spring reaction type, having a frame of sufficient stiffness to prevent lateral movement of the needle during penetration.

9.2 HARDENED CONCRETE

9.2.1 Compressive strength: Compressive strength specimens shall be 150 mm in diameter and shall be prepared and cured in accordance with MTO Method LS-401, Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory. The specimens shall be tested in accordance with MTO Method LS-407, Method of Test for Compressive Strength of Moulded Concrete Cylinders. One test shall consist of two cylinders from each round for each age specified in Table 1.

9.2.2 Air-void spacing factor: The air-void spacing factor shall be determined in accordance with ASTM Standard C457 at a minimum magnification factor of 100. Either the Linear Traverse (Rosiwal) Method or Modified Point-Count Method may be used and the paste content may be either measured

or calculated. A test shall consist of one sample prepared from a 100 mm x 200 mm test. The specimen shall be prepared from a longitudinal slice sawn from the centre of the cylinder.

9.2.3 Relative durability factor: The relative durability factor shall be determined using Procedure A (Freeze in Water, Thaw in Water) of ASTM Standard C666, Resistance of Concrete to Rapid Freezing and Thawing. The relative durability factor (RDF) shall be calculated as follows:

D_T (or D_R) = PN/300

RDF = (D_T / D_R) x 100 x 1.10

where:

D_T = durability factor of the concrete containing the admixture under test

D_R = durability factor of concrete containing the reference admixture

P = relative dynamic modulus of elasticity in percentage of the dynamic modulus of elasticity at zero cycles (values of P will be 60 or greater)

N = number of cycles at which P reaches 60 percent, or 300 if P does not reach 60 percent prior to the end of the test (300 cycles)

A test shall consist of two specimens each of reference and test concrete from each round.

9.2.4 Length change : The determination of length change shall be made in accordance with ASTM Standard C157, Length Change of Hardened Cement Mortar and Concrete. The moist curing period, including the period in the moulds shall be 14 days. The drying period shall also be 14 days. The drying shrinkage shall be considered to be the length change during the drying period based on initial measurement at the time of removal of the specimen from the mould. It shall be expressed as a percent, to the nearest 0.001 percent based on the specimen gauge length.