SPECIAL REPORT 96-28

CRREL

Freezing Temperature Protection Admixture for Portland Cement Concrete

Charles J. Korhonen and John W. Brook

October 1996

Abstract: A number of experimental admixtures were compared to Pozzutec 20 admixture for their ability to protect fresh concrete from freezing and for increasing the rate of cement hydration at below-freezing temperatures. The commercial accelerator and low-temperature admixture Pozzutec 20 served as the reference admixture for this project as it has been a successful product of Master Builders for winter concreting during the past several years. Over thirty-five experimental admixture candidates were tested. Of these, one experimental admixture, code-named EY-11, a nonchloride admixture, outperformed all the others and was selected as the admixture to be considered for future commercialization. It was demonstrated by laboratory evaluation that the Pozzutec 20 admixture did not contribute to corrosion of embedded steel reinforcement. The EY-11 admixture, although still under examination, also did not contribute to corrosion in a newer and different laboratory test. Based on a knowledge of its constituents, EY-11 is not expected to contribute to corrosion under laboratory conditions or in the field. The low and medium dosages (60 and 100 mL/kg [90 and 150 fl oz/cwt]), of EY-11 produced freeze–thaw-durable concrete, but the highest dosage examined, 160 mL/kg (240 fl oz/cwt), did not. The middle dosage (100 mL/kg) protected concrete down to the low-temperature goal of this project, –5°C (23°F). The prototype admixture, EY-11, affords superior low-temperature protection compared to existing accelerating admixtures, as well as good durability. Unfortunately, it did not provide the desirable rapid setting and strength gain of concrete at above-freezing temperatures that field engineers and concrete technicians would like.

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US Army Corps
of Engineers
Cold Regions Research &
Engineering Laboratory

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October 1996

Prepared for
OFFICE OF THE CHIEF OF ENGINEERS

PREFACE

This report was prepared by Charles J. Korhonen, Research Civil Engineer, Civil and Geotechnical Research Division, Research and Engineering Directorate, U.S. Army Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire, and by John W. Brook, Senior Research and Development Scientist, retired, Master Builders, Inc. (MB), Cleveland, Ohio.

The investigation was conducted under the authority of the Corps Construction Productivity Advancement Research (CPAR) program. Project approval was received in August 1991 and work began in April 1992. The research was completed in December 1994.

Technical review of this report was provided by Edel R. Cortez, CRREL, and by Brian E. Caine, Manager, Chemical Laboratory, and Dr. Charles Nmai, Manager, Engineering Group, MB. The authors acknowledge the support of Brian Caine, Ray Giangiacomo, James Zupanic (Youngstown State University), Robert Ryan, Jerry Lewis, Matt Miltenberger, R. Davis, and Jesse Osborne of MB, and Brian Charest, Edel Cortez, Charles Smith, and Christopher Berini of CRREL.

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Freezing Temperature Protection Admixture for Portland Cement Concrete

CHARLES J. KORHONEN AND JOHN W. BROOK

INTRODUCTION

Background

Development of an admixture capable of allowing fresh concrete to gain strength at below-freezing temperatures without causing detrimental effects to the final product has long been a goal of the concreting industry. Work on the problem began several decades ago, with contributions made by researchers from the former Soviet Union, Scandinavia, and elsewhere (Korhonen 1990) who showed that certain chemicals can significantly depress the freezing point of the concrete mix water, and that other chemicals can accelerate the hydration rate of cement at very low temperatures. To date, however, there has been no comparable advancement of these or other chemicals in the United States. Concerns over their potential adverse effects, such as increased risk of corrosion or chemical reaction with aggregate, have discouraged serious consideration.

As a result, current U.S. winter concreting practices have remained unchanged for the past several decades. Concrete ingredients such as stone, sand, and water must still be heated to melt all ice, but not heated so highly as to cause rapid set within the concrete mixing and handling equipment, and to create a mix temperature that is well above freezing. The substrate on which fresh concrete is placed must be thawed, and the concrete must be kept warm and moist long enough to ensure adequate strength to allow early removal of forms for their reuse.

The American Concrete Institute (ACI) sets the standards for winter concreting. It recommends that freshly placed concrete must be protected from freezing by maintaining its temperature at or above 5°C (40°F), preferably at or above 10°C (50°F) (ACI 1988) until it has sufficiently cured to serve its intended purpose. Finishing operations take longer as temperatures dip to 5°C (40°F) and below, and forms cannot be stripped as fast as they can during the summer. The rate of concrete strength gain is slowed. At a few degrees below zero, the hydration rate of cement continues to slow and the mix water begins to turn into ice; at –3°C (27°F), 90% of the water will freeze (Korhonen 1990). If freezing occurs, upon thawing the concrete may lose half its strength.

There are procedures today to protect newly placed concrete from freezing and to ensure adequate strength to produce concrete that meets construction needs for strength and durability. However, this protection is costly. It has been estimated that the U.S. construction industry spends $800 million (Civil Engineering 1991) every year on measures to protect fresh concrete from freezing. An admixture that would alleviate this expense would be of great economic benefit.

Master Builders (MB) established renewed interest in this topic in the late 1980s by marketing this country's first nonchloride, low-temperature admixture: Pozzutec 20. Though Pozzutec 20 depresses the freezing point of water a few degrees, its major cold weather advantage is that it has been specially formulated to accelerate setting time and strength gain in concrete. When used at recommended dosages, Pozzutec 20 greatly increases the rate of cement hydration, generating more heat earlier than would be generated by normal concrete, even those containing conventional accelerators. This extra heat usually provides enough protection to prevent concrete from freezing until it has developed sufficient strength to resist ice damage. After the concrete has reached this level of self-protection, it continues to gain strength even if its internal temperature should fall below freezing. Pozzutec 20 is recommended for use at ambient temperatures down to –7°C (20°F) with an application dosage of up to 60 mL/kg (90 fl oz/cwt).

In an effort to expand upon the success of Pozzutec 20 and to develop the long-sought freezing protection admixture, Master Builders and the U.S. Army Cold Regions Research and Engineering Laboratory entered into a cooperative research project. This project was conducted under the authority of the Corps of Engineers Construction Productivity Advancement Research (CPAR) program. Because the Federal Government is a big buyer of construction services and the Corps of Engineers uses a lot of concrete, a new winter admixture would produce savings for the Government and provide a benefit to the U.S. economy. This is the final report of Fiscal Year 1990 project "Freezing Temperature Protection Admixture for Portland Cement Concrete."

Objectives

The two prime objectives of this study were to explore the low-temperature performance of Pozzutec 20 and to develop a prototype admixture that would protect fresh concrete from freezing while increasing the rate of cement hydration when the internal temperature of the concrete is below 0°C (32°F).

One important constraint in developing low-temperature admixtures for concrete is that no standards of acceptance criteria are available. Chemical admixtures are currently classified by ASTM C 494 into seven categories of set-controlling and water-reducing admixtures. The categories include Type C, accelerating, and Type E, water reducing and accelerating admixtures, each tested at 23 ± 1.7°C (73 ± 3°F), well above freezing. It was therefore necessary at the start of this project to define a freezing protection admixture. Freezing protection admixtures were defined as chemicals that should:

Depress the freezing point of water

Promote strength gain of concrete at low temperatures

Not interfere with concrete strength gain at normal, above-freezing temperatures

Maintain workability of the concrete in freezing conditions

Achieve reasonable concrete set times (this does not necessarily mean accelerated set times)

Produce freeze–thaw-durable concrete

Not react unduly with silica aggregate

Not contribute to corrosion of embedded steel reinforcement, or to steel on which concrete is placed

Be cost-effective

Further, to avoid the necessity of conducting long-term testing of experimental admixtures to determine that they meet these requirements, the decision was made that only chemicals currently being used in concrete be considered for initial evaluation. This decision provided us with reasonable assurance that the chemicals have already been tested for their effect on concrete. As experience was gained with this new technology, other chemicals could be added to the study. It was also decided that the initial low-temperature goal would be set at –5°C (23°F), with –10°C (14°F) being a possible ultimate objective, and that the concrete cured at these low temperatures should gain strength at least as rapidly as normal concrete at 5°C (40°F), the accepted low-temperature limit for winter concreting in the United States (ACI 1988).

Finally, to ensure reasonable continuity during the nearly two years of laboratory testing, both MB and CRREL used the same cement, air entraining agent, and plasticizer. The cement selected was an ASTM Type I cement from Blue Circle Cement, Tulsa, Oklahoma, with a Blaine fineness of 3460 cm^2/g (Table 1). A Type III cement was used at CRREL for some Phase I mixtures (Table 1). The air entraining agent was a neutralized vinsol resin, MB-VR, and the plasticizer was a high-range water reducer, Rheobuild 1000 (naphthalene sulfonate-formaldehyde condensate, calcium salt), both from Master Builders. Each party used its local aggregates and water. The coarse and fine aggregates used by CRREL

Table 1. Chemical composition of Type I and Type III cement.

Compound	*Type I (%)*	*Type III (%)*
SiO_2	20.85	20.95
Al_2O_3	4.75	5.44
Fe_2O_3	2.26	2.36
CaO	63.92	62.57
K_2O	0.70	0.75
MgO	2.34	2.16
SO_3	3.14	4.20
C_3S	58.0	43.6
C_2S	16.0	27.2
C_3A	9.0	10.4
C_4AF	7.0	7.2
LOI	1.18	1.09
Na_2O (Eq)	0.87	0.80

Table 2. The four phases of work.

Phase	Description
I	Evaluation of Pozzutec 20
II	Development of improved admixture
III	Evaluation of improved admixture
IV	Field application

Table 3. Phase I tasks.

Task	Description
1	Strength vs. temperature
2	Corrosion potential
3	Durability
4	Equivalent insulation
5	Critical strength

had bulk specific gravities of 2.89 and 2.67 and an absorption of 0.5 and 1.1 percent, respectively. The coarse aggregate was crushed ledge with a gradation that fit between ASTM sizes no. 6 and 7. The fine aggregate was a natural sand with a fineness modulus of 2.80. The coarse and fine aggregate used by MB had specific gravities of 2.84 and 2.58, respectively. The coarse aggregate was a Drummond Island limestone while the fine aggregate was a Hugo sand. Tap water was used for the mix water at each lab.

Scope

A series of laboratory and field tests was conducted to evaluate the effect of various chemicals on properties of concrete. Master Builders developed chemical formulations for testing and conducted the laboratory studies aimed at defining strength and chemical reactions of the formulations. CRREL conducted the low-temperature laboratory and field studies to verify expected performance of the admixtures.

This project consisted of four phases of experimental work (Table 2). Phase I involved a comprehensive laboratory testing of Pozzutec 20. Phase II conducted a laboratory screening of numerous potentially new freezing protection admixtures, selecting the best for further testing and evaluation. Phase III used a series of tests similar to those performed on Pozzutec 20 in Phase I on the best admixture developed in Phase II. Phase IV consisted of two cold weather field trials.

PHASE I: EVALUATION OF POZZUTEC 20

Procedure

The objective of Phase I was to characterize the low-temperature performance of Pozzutec 20 and, in the process, establish a test protocol for Phase III. Phase I was divided into five experimental tasks (Table 3).

Task 1: Strength vs. temperature

The objective of this task was to develop a relationship between the strength gain of concrete and its curing temperature. The test procedure consisted of mixing and casting the concrete at room temperature. A few minutes after casting, the cylinders were placed into one of several curing rooms set at prescribed temperatures. Concrete temperatures in each of the rooms were monitored for the first seven days by thermocouples cast into dummy cylinders. A data logger recorded the temperatures in each dummy cylinder as well as the ambient temperature. All cylinders were sealed to prevent evaporation from the concrete. At various ages, sets of three cylinders were removed from the curing rooms, allowed to warm up to 10°C (50°F), if necessary, and tested for unconfined compressive strength according to ASTM C 39.

The concrete was prepared according to ACI 211.1 standards. Fourteen mixes, each with a volume of 0.057 m^3 (2.0 ft^3) were batched, twelve corresponding to three cement factors and four admixture dosages for Type I cement, and two for one cement factor with Type III cement and two dosages of admixture (Table 4). Sixty-five cylinders (75 × 150 mm [3 × 6 in.]) were cast per mix (4 ages × 5 temperatures × 3 replicate specimens + 5 dummies).

Each cylinder was identified by three numbers (Table 5): cement factor, admixture dosage, and curing temperature. For example, mix (2,0,–5) contained the cement factor 2 (365 kg/m^3 [611 lb/yd^3]) and no admixture cured at –5°C. The mixtures containing Type III cement were identified by an asterisk (*) preceding the three-digit label. This scheme is used throughout this report.

Once cast, the cylinders were placed into 20, 5, –5, –10, and –20°C (70, 40, 23, 14, –4°F) rooms within 30–45 min of addition of the mix water. This ensured that essentially no strength gain took place at anything but the appropriate curing temperature. The cylinders remained in each room

Table 4. Phase I test variables.

Variable	*Quantity*
Cement factors	308, 365, 420 kg/m^3 (517, 611, and 705 lb/yd^3)
Pozzutec 20	0, 40, 60, 100 mL/kg (0, 60, 90, and 150 fl oz/cwt†)
Test ages	7, 14, 28, and 56 days
Curing temperatures	20, 5, –5, –10, and –20°C (70, 40, 23, 14, –4°F)
w/c ratios	0.44, 0.48, and 0.52 for the 308, 365, and 420 cement factor mixtures, respectively
Cement types	I and III (Type III w/mix [*2,2] and [*2,0])
Plasticizer	For the 308 factor mixture only

† cwt denotes 100 lb of cement.
* Denotes Type III cement.

Table 5. Phase I mixture identification.

Cement factor	*kg/m^3 (lb/yd^3)*	*Admixture dosage*	*mL/kg (fl oz/cwt)*
1	308 (517)	0	0 (0)
2	365 (611)	1	40 (60)
3	420 (705)	2	60 (90)
—	—	3	100 (150)

until tested or until 28 days. After 28 days, all untested cylinders were placed in the 20°C (70°F) room for 28 days of additional curing. This additional curing showed whether any permanent strength loss was caused by the freezing temperatures.

Task 2: Corrosion potential

The potential of Pozzutec 20 to corrode reinforcing steel was tested according to two different procedures: initially via the well-known procedure reported in FHWA/RD-86/193 of the Federal Highway Administration (this method was the predecessor of ASTM G 109, a modification of and more reliable one than that of the FHWA) and the MB-labeled "Lollipop Microcell Corrosion Test." The latter test, based on several references (Sagues 1987, Dawson and Langford 1988, Aguilar et al. 1990, and Tourney and Berke 1993) uses a lower w/c ratio than the ASTM method, thereby providing a better quality concrete. The lollipop procedure uses 75- × 150-mm (3 × 6 in.) cylindrical mortar specimens, each fitted with an axially located No. 4 reinforcing bar positioned 31.8 mm (1.25 in.) off the bottom of the cylinder. The rebar protrudes out from the top of each specimen. In the test, six specimens were cast from two mortar mixtures: one mixture with no admixture, and one with Pozzutec 20 dosed at 60 mL/kg (90 fl oz/cwt). Three of the six specimens from each of the two mixtures were submerged to a depth of 75 mm (3 in.) in a 3% sodium chloride solution, and the other three specimens were partially submerged in deionized water. Another mixture was also prepared with a Pozzutec 20 dose of 100 mL/kg (150 fl oz/cwt), from which only three specimens were cast and placed in the sodium chloride solution. All specimens were made with standard ASTM C 109 mortar with a 0.485 w/c. They were cured at 100% relative humidity according to normal ACI accepted practice. The deionized water provided a nonaggressive environment and the sodium chloride solution an aggressive one. The specimens were monitored for corrosion by regularly recording the reinforcing bar's half-cell potential using ASTM C 876, and periodically running impedance spectroscopy to approximate the corrosion rate. Testing, which was expected to run for up to two years, began during April 1994 and was completed after 1 1/2 years in October 1995, when all specimens in chloride solution began corroding. Specimens in sodium chloride solution were found to have corroded only under an epoxy coating upon final inspection.

Task 3: Durability

The resistance of concrete beams to deterioration from repeated cycles of freezing and thawing was tested according to ASTM C 666, Procedure A. Pozzutec 20 was tested at two dosages: 60 and 100 mL/kg (90 and 150 fl oz/cwt). The concrete for the beams was made with a cement factor of 365 kg/m^3 (611 lb/yd^3), a w/c of 0.434 for the concrete made with Pozzutec 20 (for the admixture provides water reduction) and 0.45 for plain concrete, and an entrained air content of 6%. Three beams were made from each mix, each beam measuring 75 × 102 × 406 mm (3 × 4 × 16 in.). They were moist-cured for 14 days, then wrapped in plastic and stored in a freezer until tested. All beams were cycled through 300 freezing and thawing cycles or until failure, whichever occurred first. Changes in relative dynamic modulus derived from resonant frequency readings were used to monitor the deterioration. Criteria of ASTM C

494 indicate that adequate F/T durability is expected of concrete that provides a durability factor (DF) of 80 or greater.

Task 4: Equivalent insulation

The American Concrete Institute (ACI 1988) specifies that concrete placed during cold weather should be maintained at a certain temperature for a given amount of time. For example, ACI provides a series of tables outlining the amount of insulation that is needed to maintain concrete at 10°C (50°F) for up to seven days. The amount of insulation required is related to the ambient temperature, the shape of the structure, and the cement factor of the concrete. Because Pozzutec 20 accelerates the generation of heat from cement during the first few days, concrete made with this admixture should require less thermal protection than admixture-free concrete. The objective of this task was to determine the minimum ambient temperature at which an uninsulated cylinder of concrete made with Pozzutec 20 can be cured to produce a compressive strength equal to that of admixture-free concrete cured at 10°C (50°F). This minimum curing temperature could then be compared to the ACI tables to determine the amount of insulation that would have been necessary to protect normal concrete if cured at that same low temperature. This insulation value was termed "equivalent insulation," signifying the amount of insulation that Pozzutec 20 could safely replace.

The test consisted of making three batches of concrete, each with a Type I cement and a different dosage of Pozzutec 20. The concrete was mixed and cast into numerous 75- × 150-mm (3 × 6 in.) cylinder molds, and then capped and distributed among various curing rooms, each maintained at a different temperature. At 7, 14, and 28 days, three cylinders were removed from each room and compression-tested after the cylinders were warmed up to 10°C (50°F). Two additional batches of concrete made with Type III cement tested the value of using a high early strength cement. Table 6 gives the test makeup.

Table 6. Equivalent insulation tests.

Mixture ID	*Cure temperature °C (°F)*
2,0	10 (50)
2,1	4, 2, 0, –2 (40, 35, 32, 28)
2,2	4, 2, 0, –2 (40, 35, 32, 28)
2,3	4, 2, 0, –2 (40, 35, 32, 28)
*2,0	4, 2, 0, –2 (40, 35, 32, 28)
*2,2	4, 2, 0, –2 (40, 35, 32, 28)

*Denotes Type III cement.

Task 5: Critical strength

Concrete is susceptible to ice damage at early age because either its pore structure is underdeveloped or its moisture content is too high. As a concrete matures, its water chemically combines with cement, with the result that the concrete increases in strength and decreases in freezable water content. At some strength the quantity of freezable water falls below a critical level, which creates empty space within the concrete, enabling the concrete to accommodate the growth of ice crystals without being damaged. Concrete that attains a compressive strength of 3.5 MPa (500 psi), the critical strength, is expected to be resistant to one cycle of freezing and thawing (ACI 1988). The objective of this test was to determine if Pozzutec 20 affected this value.

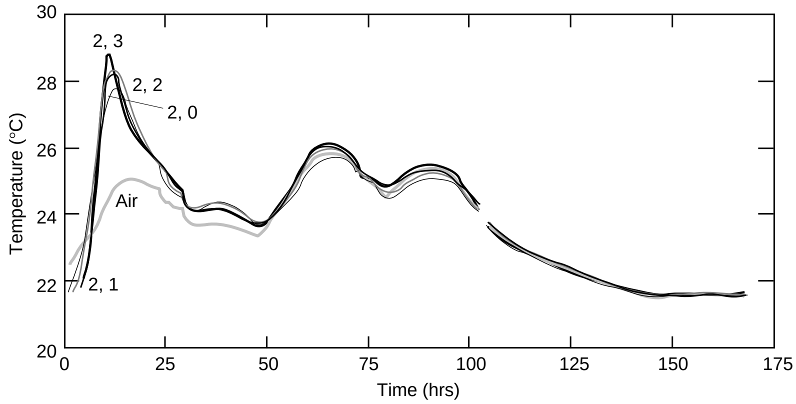
The test was accomplished by allowing 75- × 150-mm (3 × 6 in.) cylinders of fresh concrete to cure at room temperature until they attained a compressive strength of 1.7, 3.4, and 5.2 MPa (250, 500, and 750 psi). They were then transferred to a –20°C (–4°F) freezing room overnight, after which they were returned to room temperature and cured until being strength-tested after 3, 7, and 28 days. The strengths of the once-frozen cylinders were compared to control cylinders that were never frozen to determine if the various freezing scenarios caused a loss of strength.

Results and discussion

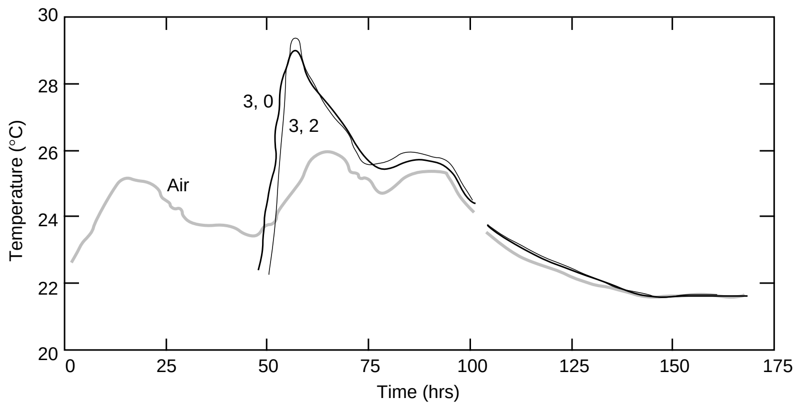
Task 1: Strength vs. temperature

Strength gain of concrete is the result of chemical and physical reactions between cement and water. At room temperature, the reaction process is most easily observed as a rise in temperature of curing concrete. The amount of temperature rise depends on how quickly the cement hydrates and how quickly the generated heat is lost from the concrete to the outside environment. Figure 1 shows typical temperature histories for 75- × 150-mm (3 × 6 in.) cylinders of concrete cured at various temperatures. Results for the 308-kg/m^3 (517 lb/yd^3) mixes are not provided, as these mixes tended to segregate when Pozzutec 20 was added. Because this is considered a low cement content for winter concreting, work with this cement factor was not pursued further.

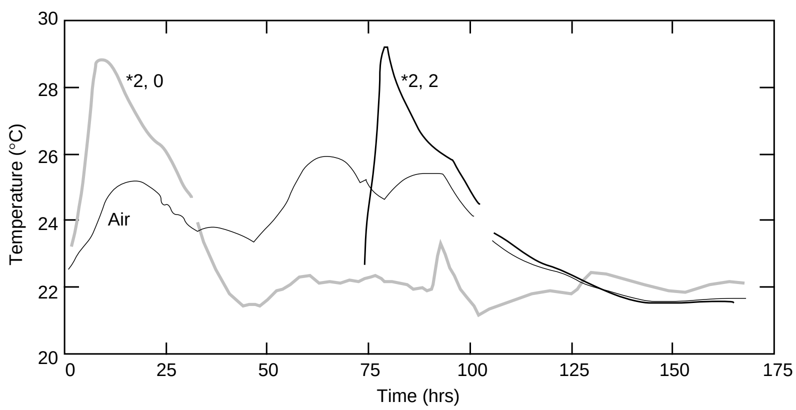
Figures 1a, 1b, and 1c show the effect of cement type, cement amount, and Pozzutec 20 on the temperature of curing concrete. It should be noted that these figures do not represent field conditions, as most field structures are more mas-



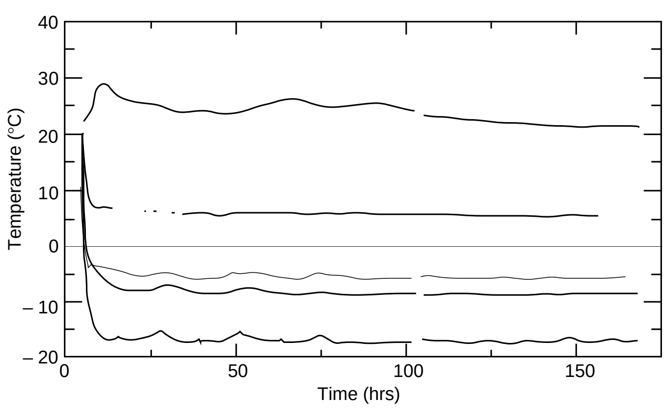
a. Mixtures 2,3; 2,2; 2,1; and 2,0.



b. Mixtures 3,0 and 3,2.



*c. Mixtures *2,0 and *2,2.*



d. All curves represent mixture 2,3.

Figure 1. Temperature histories of concrete with Types I and III cement, various dosages of Pozzutec 20, cured at various temperatures.

sive than the small samples tested in this task and would likely produce higher concrete temperatures. However, the referenced curves clearly demonstrate the accelerating effect of Pozzutec 20. In all three figures, increased dosages of this admixture caused the temperature of the concrete to rise more quickly and attain higher temperatures than did lower dosages. For example, Figure 1a shows that mixture 2,3 produced a concrete temperature that was about 2°C (3.6°F) higher than mixture 2,0 and about 1°C (1.8°F) higher than mixtures 2,1 and 2,2. Comparing Figure 1a to 1b shows that increasing the cement content has the same accelerating effect as does adding Pozzutec 20 to the mix. The 3,0 mixture, containing the high cement factor (420 kg/m^3) and no admixture, produced a concrete temperature that was nearly identical to the 2,3 mixture, containing the middle cement factor (365 kg/m^3) and Pozzutec 20. Comparing Figures 1b to 1c shows that the high early strength cement produced the same temperature that was produced by a higher amount of normal cement.

Figure 1d shows a typical temperature history of samples cured in each of the five curing rooms. Samples stored at room temperature briefly rise in temperature before cooling to room temperature at about 30 hours. The heat loss for the samples in the other rooms was rapid enough to preclude any rise in temperature. The samples quickly cooled from about 20°C (70°F) to ambient temperature. Within eight hours the sample in the 5°C (40°F) room cooled to ambient while those in the three colder rooms cooled to below freezing within five hours, showing that essentially all strength gained by the samples in the cold rooms occurred at the temperature of the particular curing room. Therefore, the cold room temperature can be thought of as the temperature of the concrete.

Figure 2 shows the two most important findings from this task. A complete list of strength results is provided in Appendix A. As was done with the temperature measurements, the strength results for the 308-kg/m^3 (517 lb/yd^3) mixes are not provided due to segregation of this mixture.

The first finding of this task was that Pozzutec 20 not only accelerated early strength gain in concrete but that it also enhanced ultimate strength. This result can be seen in Figure 2 by comparing the room-temperature strength of the control concrete (2,0,20) to those of the three concretes made with Pozzutec 20, cured at room temperature. The low, medium, and high dosages of Pozzutec 20 increased the seven-day strength of concrete by 5, 16, and 17 percent, respectively, and that of the 56-day strengths by 8, 18, and 30 percent, respectively. The second finding was that none of the Pozzutec 20 dosages produced acceptable strengths when cured at –5, –10, or –20°C (23, 14, –4°F); it is probable that mass concrete produced in the field with higher dosages (90 fl oz) of Pozzutec 20 and curing temperatures above 14°F would have acceptable compressive strengths. The initial goal of this project was to produce an admixture that would promote strength in concrete

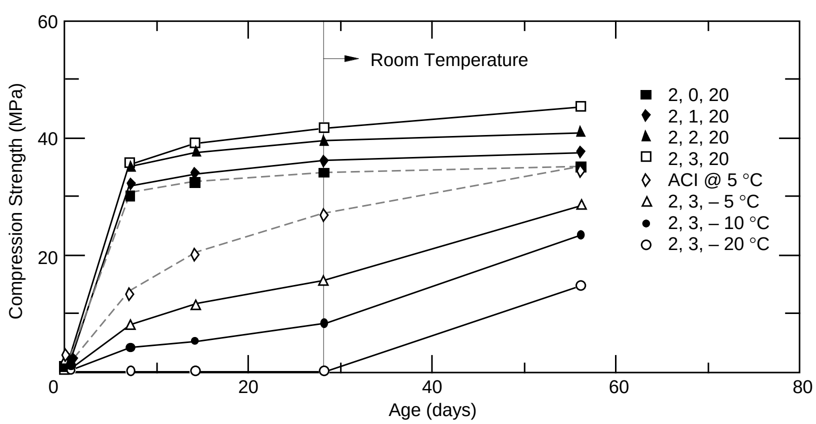


Figure 2. Effect of temperature on strength gain of concrete. The dotted lines show the strength gain of control concrete at 20°C (70°F) and 5°C (40°F). The 5°C (40°F) line is based on guidance from ACI (1988). All results are for concrete made with a 365-kg/m^3 (611 lb/yd^3) cement factor cured at a given temperature for 28 days, followed by 28 days of curing at room temperature.

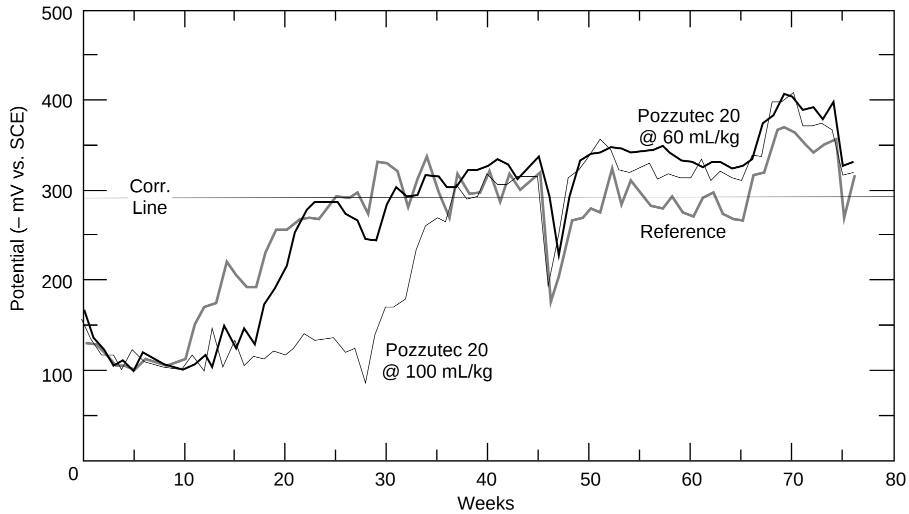


Figure 3. Lollipop specimens submerged half-height in 3% sodium chloride solution.

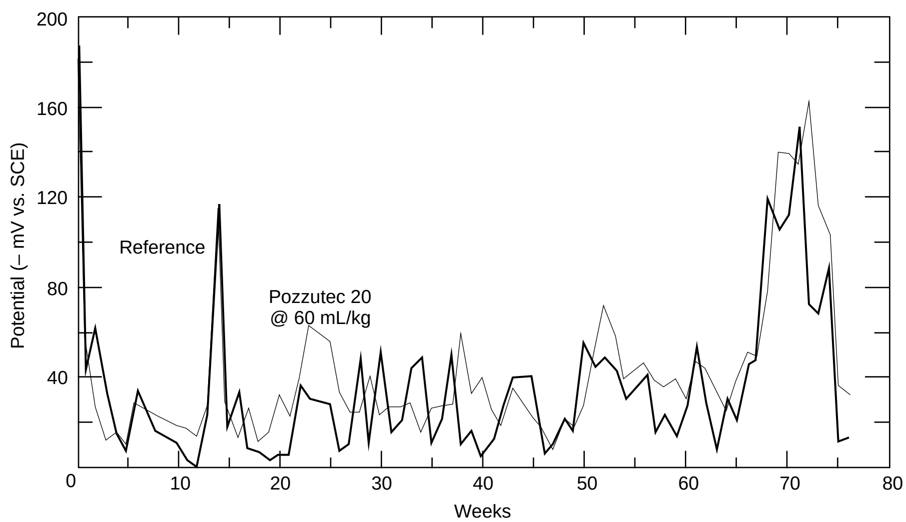


Figure 4. Lollipop specimens submerged half-height in deionized water.

cured at –5°C (23°F) at the same rate as that in control concrete cured at 5°C (40°F). As can be seen, the 7-, 14-, and 28-day strengths of the high dosage concrete cured at –5°C (23°F) were significantly below that of the ACI standard for 5°C (40°F) concrete. Strength gain at –10 and –20°C (14 and –4°F) was even lower (see Fig. 2). This does not necessarily mean that the Pozzutec concrete has been damaged by freezing, as this concrete displayed a remarkable recovery in strength by 56 days when brought back to room temperature. It does suggest, however, that a new admixture would have to be developed to fully satisfy the low-temperature goal of this project.

Task 2: Corrosion potential

The lollipop test results show that mortars treated with 60 mL/kg (90 fl oz/cwt) of Pozzutec 20 are practically identical to admixture-free mortar. Figures 3 and 4 are graphs of the average potentials from three specimens over a 1 1/2-year period. There is no exact potential identifying the initiation of corrosion. ASTM C 876 identifies potentials more positive than –200 mV vs. copper sulfate reference electrodes as passive or noncorrosive behavior. Potentials between –200 and –350 mV are an indication that corrosion has initiated, and potentials more negative than –350 mV indicate a high probability of corrosion. Since our test used a saturated calomel electrode (SCE), 60 mV should by added to the ASTM values to make them useful to our readings (to convert to mV SCE). Based on this guidance, potential indicative of corrosion for a saturated calomel electrode is –290 mV. The admixture-free specimens and the specimens containing both dosages of Pozzutec 20 partially submerged in 3% sodium chloride solution showed beginning signs of corrosion (Fig. 3). The interest, however, is that the Pozzutec 20 did not increase the level of corrosion when compared to the reference. All specimens in deionized water show no indication of corrosion. Thus, the Pozzutec 20 did not adversely affect the mortar from the corrosion point of view.

Previous testing by others (Nmai et al. 1994) corroborates the above results by showing that mortar containing 60 mL/kg (90 fl oz/cwt) of Pozzutec 20 and tested by the aforementioned method of FHWA over the 50-week examination period showed no sign of rebar corrosion. The FHWA test, also known as the modified Southern Climate Accelerated Corrosion Test, subjects the top surface of concrete slabs, embedded with two layers of rebar, to intermittent ponding with 15% sodium chloride solution. The presence of corrosion is determined by the voltage drop between the layers of rebar.

Task 3: Durability

Table 7 shows the results from subjecting concrete beams to up to 300 cycles of freezing and thawing according to ASTM C 666, Procedure A. Freeze–thaw deterioration was monitored by measuring the relative dynamic modulus of elasticity of each concrete beam according to ASTM C 215. Criteria of ASTM C 494 indicate that concrete is of adequate durability if it maintains a durability factor of greater than 80 after 300 freeze–thaw cycles. The durability factor is the relative dynamic modulus of elasticity, expressed as percent, at the end of testing multiplied by the fraction of the number of test cycles conducted to the specified number of cycles (300 for this project). As seen in the table, the control and Pozzutec 20 mixture dosed at 60 mL/kg (90 fl oz/cwt) performed well. They both had durability factors of 99 at the end of the test. The 100 mL/kg (150 fl oz/cwt), on the other hand, failed after 204 cycles of freezing and thawing. The lower dosage (90 fl oz) of Pozzutec is the maximum dosage recommended by Master Builders.

Table 7. Durability factors for Pozzutec 20 concrete.

	Pozzutec 20 dosage—mL/kg (fl oz/cwt)		
	None	*60 (90)*	*100 (150)*
Durability factor	99	99	Failed

Task 4: Equivalent insulation

The minimum temperature at which concrete with Pozzutec 20 can be cured to produce compressive strengths equal to that of control concrete cured at 10°C (50°F) was determined. Table 8 shows the strength of the various concrete mixtures studied. As can be seen, the minimum temperature for the 40-mL/kg (60 fl oz/cwt) dosage of Pozzutec 20 (mixture 2,1) was 2°C (35.6°F), where its strength equaled or bettered that of the control at all ages. The 60 (90) and 100 (150) mL/kg (fl oz/cwt) had minimum temperatures of 1 and 0°C, respectively. For the mixture made with high early strength cement, the zero dose and 60 mL/kg (90 fl oz/cwt) dose had minimum temperatures of –2 and –4°C (28.4 and 24.8°F), respectively.

Table 8. Equivalent insulation test results for concrete made with Pozzutec 20.

	Compressive strength—MPa (psi)		
Mixture ID	*7 days*	*14 days*	*28 days*
2,0,10 (control)	*23.5 (3405)*	*28.5 (4131)*	*33.1 (4800)*
2,1,4	23.7 (3442)	29.6 (4291)	33.0 (4791)
2,1,2	24.0 (3475)	30.0 (4357)	34.7 (5027)
2,1,0	22.6 (3282)	27.8 (4037)	33.8 (4899)
2,1,–2	19.9 (2881)	26.1 (3782)	30.5 (4428)
2,2,4	25.5 (3697)	31.7 (4593)	35.5 (5154)
2,2,2	24.4 (3532)	31.1 (4513)	35.8 (5197)
2,2,0	22.4 (3524)	29.1 (4220)	33.4 (4847)
2,2,–2	20.3 (2947)	27.2 (3942)	32.3 (4678)
2,3,4	25.9 (3753)	30.2 (4380)	36.4 (5281)
2,3,2	25.8 (3739)	31.9 (4630)	38.4 (5564)
2,3,0	24.3 (3527)	29.6 (4296)	36.3 (5262)
2,3,–2	19.7 (2862)	28.9 (4186)	33.8 (4899)
*2,0,4	27.2 (3942)	34.8 (5041)	38.7 (5612)
*2,0,2	27.2 (3937)	35.9 (5210)	37.5 (5432)
*2,0,0	26.3 (3810)	33.5 (4857)	32.8 (4763)
*2,0,–2	24.2 (3503)	30.4 (4409)	34.4 (4984)
*2,2,4	30.5 (4418)	35.8 (5197)	41.8 (6059)
*2,2,2	29.8 (4319)	37.0 (5366)	40.0 (5805)
*2,2,0	28.6 (4140)	35.0 (5069)	39.5 (5734)
*2,2,–2	26.9 (3895)	35.0 (5074)	39.4 (5720)

* Denotes Type III cement.

Table 9. Equivalent insulation values for 5.4-cm- (6 in.) thick wall maintained at 10°C (50°F) for seven days.

Mixture	*Air temperature °C (°F)*	*Required thermal resistance m^2 K/W (hr ft^2 F/Btu)*	*Equivalent fibrous glass—mm (in.)*
2,1	2 (37)	1.0 (5.7)	47 (1.8)
2,2	1 (34)	1.1 (6.3)	52 (2.0)
2,3	0 (32)	1.2 (6.9)	56 (2.2)
*2,0	–2 (28)	1.4 (8.1)	66 (2.6)
*2,2	–4 (25)	1.6 (9.2)	75 (3.0)

* Denotes Type III cement.

Table 9 shows the amount of insulation that the various mixtures tested can replace. The table is based on ACI requirements to maintain a 150-mm- (6 in.) thick wall of concrete made with Type I cement at a cement factor of 365 kg/m^3 (611 lb/yd^3) at 10°C (50°F) for seven days. For instance, according to ACI, an ambient air temperature of 0°C (32°F) requires insulation to have a thermal resistance value of 1.2 m^2 K/W (6.9 hr ft^2 F/Btu), which is equivalent to 56 mm (2.2 in.) of fibrous glass insulation. Pozzutec 20 dosed at 100 mL/kg (150 fl oz/cwt) is equivalent to that amount of insulation (Table 9).

Task 5: Critical strength

The objective of this task was to determine if Pozzutec 20 affected the minimum strength at which concrete can be frozen without being frost-damaged. The critical freezing strength of normal air-entrained concrete, according to ACI 1988, is 3.5 MPa (500 psi). A complete list of strength results at all test ages is provided in Appendix B. Figure 5 highlights this data by showing the 28-day strengths for the 365-kg/m^3 (611 lb/yd^3) cement factor of Type I and III cement. These data provide evidence of the effect of Pozzutec 20 on the critical freezing strength of concrete.

Before discussing the effects of Pozzutec 20, it is worth noting in Figure 5 that the three admixture-free concretes, i.e., (2,0), (3,0), and (*2,0), were unaffected by one cycle of freezing and thawing once they had attained a compressive strength of 3.4 MPa (500 psi). The freezing actually produced a slightly stronger concrete for the Type I cement and showed no ill effect for the Type III cement. It is interesting to note that the 3.5 MPa (500 psi) critical strength value is for air-entrained concrete. The concretes in this study were non-air-entrained. Thus, the real critical strength is probably less than that given by ACI.

The addition of Pozzutec 20 to the concrete had a positive effect on when concrete can first be frozen. For both of the Type I cement mixtures (Fig. 5a and b), Pozzutec 20 produced a 28-day strength that exceeded that of the admixture-free control, regardless of the strength at which the concrete was frozen. The exception to this was for the Type III cement mixture (Fig. 5c), where Pozzutec 20 caused a 5% decrease in the 28-

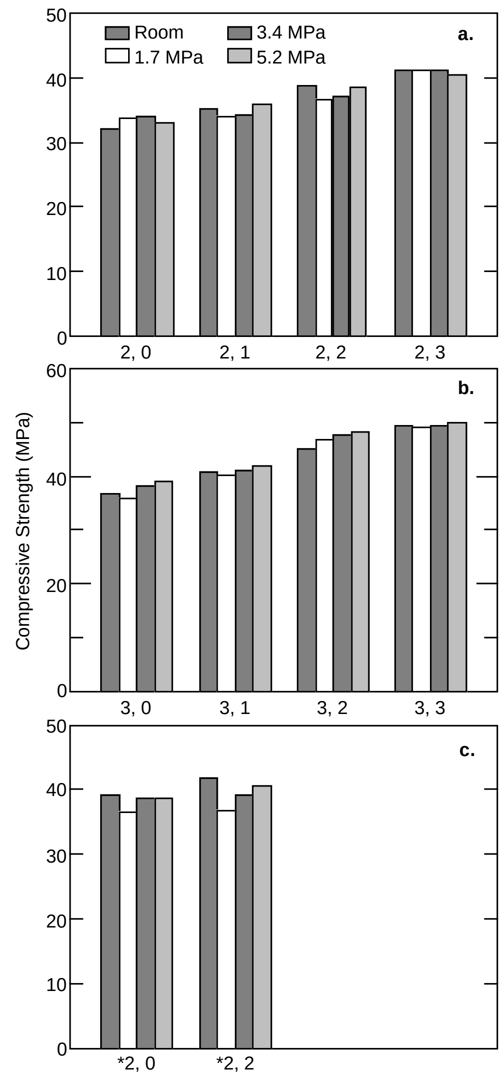


Figure 5. Effect of early age freezing on concrete strength. The concretes were placed in a –20°C (–4°F) room for 24 hours after they attained a specified compressive strength. They were then removed from the cold room and cured at room temperature. This graph compares the 28-day strength of control concrete that was never frozen to those of the concretes that were frozen once.

day strength when the concrete was frozen at the 1.7-MPa (250 psi) strength. The strength of the Pozzutec 20 concrete when frozen after it had attained the 3.4-MPa (500 psi) strength exceeded that of the control by 1% (Fig. 5c).

Based on the data in Table B1 and in Figure 5, it is clear that concrete made with Pozzutec 20 can safely be frozen after it has achieved a compressive strength of 3.5 MPa (500 psi).

PHASE II: DEVELOPMENT OF IMPROVED ADMIXTURE

Procedure

The objective of this phase was to develop a new admixture that would outperform Pozzutec 20 in early strength gain at lower temperatures. This work consisted of creating trial admixtures composed of chemicals in aqueous solution. The raw materials are proprietary information and are not disclosed. Although no listing of individual chemicals is provided, the general categories of chemicals used are given: 1) inorganic salts, 2) organic chemicals containing hydroxy or carboxy groups, and 3) organic surfactants (plasticizer). Phase II was divided into the three tasks indicated in Table 10.

Table 10. Phase II tasks.

Task	*Description*
1	Mortar screening
2	Concrete testing
3	Follow-up testing

Task 1: Mortar screening

Task 1 used mortar as a rapid way to screen the various chemicals. Using mortar instead of concrete simplified mixing operations by reducing material handling and permitting smaller test specimens to be used. The performance of each trial admixture was judged against two references: mortar produced with 60 mL/kg (90 fl oz/cwt) of Pozzutec 20, and plain mortar. The mortars were cured at 10°C (50°F). This temperature was used in the hope that it would yield a reasonable indication of relative admixture efficacy for lower temperatures. The mix proportions are given in Table 11.

Table 11. Phase II, Task 1; mortar mixture proportions.

Ingredient	*Amount*
Type I cement, Blue Circle	500–550 gm (1.1–1.2 lb)
Concrete sand	1375–1513 gm (3.0–3.3 lb)
Tap water—16°C (60°F)	195–213 mL (6.6–7.2 fl oz) (admixture mortar) 225–242 mL (7.6–8.2 fl oz) (plain mortar)
Trial admixture	60 mL/kg (90 fl oz/cwt) 100 mL/kg (150 fl oz/cwt) 160 to 176 mL/kg (245 to 270 fl oz/cwt)

The mortar was prepared according to ASTM C 109 in a Hobart mixer. Set times were obtained with Gillmore needles at 10°C (50°F) ambient temperature. The mortars were tested at a 0.50 w/c ratio so as to provide near-equal flow, or workability, for each mix. The water contents of the mixtures were adjusted for water content of each admixture. Compressive strengths were obtained from 2-in. cubes cast after curing for one, three, and 28 days. This was later changed to three, seven, and 28 days because the one-day strengths were too low to be of value in this screening process.

Three series of trial admixtures were created, coded EX, EY, and EZ, along with two others modeled after Pozzutec 20, for a total of 35 solutions.

Task 2: Concrete testing

The best trial admixtures from Task 1 were tested in concrete. Mixing took place at room temperature in a 0.17-m^3 (6 ft^3) drum mixer rotating at 18 rpm for five minutes. The test specimens, 100- × 200-mm (4 × 8 in.) cylinders, were cast and divided into two groups. One group was cured at room temperature and one at –10°C (14°F) for one, seven, and 28 days. All specimens from the cold room were thawed at room temperature for four to six hours (the amount of time necessary to allow for elevating the concrete specimen temperature to 50°F) before being tested for compressive strength. Set time was determined according to ASTM C 403, air content according to ASTM C 231 (pressure method [Type B]), and slump according to ASTM C 143 (penetrometer). The trial admixtures were added to the mix water before mixing started. Table 12 provides the mixture proportions used in this task.

Task 3: Follow-up testing

Task 3 consisted of follow-up work using the better trial admixture systems found in Task 2.

Table 12. Phase II, Task 2; concrete mixture proportions. A Type I cement with a 365-kg/m^3 (611 lb/yd^3) cement factor was used.

Ingredient	*Control*	*Trial*
Water/cement	0.463	0.438–0.440
Hugo sand, SG 2.58	24.5 kg (53.9 lb)	25.5 kg (56.2 lb)
Coarse agg, SG 2.84	36.4 kg (80.0 lb)	36.4 kg (80.0 lb)
Trial admixture	none none	60 mL/kg (90 fl oz/cwt) 100 mL/kg (150 fl oz/cwt)
Pozzutec 20	60 mL/kg (90 fl oz/cwt)	none

The concrete was mixed at room temperature and cured at –5 and –10°C (23 and 14°F). The best admixtures were selected for further testing in Phase III. The follow-up tests consisted primarily of reexaminations and confirmation testing of the better results.

Results and discussion

Task 1: Mortar screening

There is little to report in this task except to list those admixtures that performed relatively well: ARL-506, EX-3, EX-4, EX-5D, EY-1, EY-3, EY-7, and EY-10. The results from all mortar screenings are provided in Appendix C. Criteria such as set time, both initial and final, compressive strength, and admixture dosage were used in picking the best performances.

Task 2: Concrete testing

Three trial admixtures were found to perform well in concrete. The primary yardstick for admixture selection was compressive strength at –10°C (14°F). The results from all concrete testings are provided in Appendix D. The admixtures chosen for further evaluation were ARL-506, EX-4, and EY-11; ARL-506 is an analog of Pozzutec 20.

Task 3: Follow-up testing

Task 3 further examined the three best trial admixtures from Task 2. It also examined two other groupings of admixtures: two freeze-point depressants in combination with Pozzutec 20, and three new trial admixtures. The results are presented in Tables 13–16. In all cases, the concrete was mixed at room temperature and cured at 20, –5, and –10°C (70, 23, and 14°F).

Table 13 shows the results from a reexamination of ARL-506 and EX-4 in comparison to Pozzutec 20 and admixture-free concrete. All mixtures had a cement factor of 365 kg/m^3 (611 lb/yd^3) and a w/c of 0.48. At –5°C (23°F), the concrete made with high dosages of ARL-506 and EX-4 gained 15 and 17%, respectively, more strength than Pozzutec 20 at 28 days. At –10°C (14°F), these admixtures produced concrete that was significantly weaker compared with Pozzutec 20 concrete cured at that same temperature. At room temperature neither of these two trial admixtures provided as much strength as that recorded for Pozzutec 20; the high doses of ARL-506 and EX-4 gained 16 and 5%, respectively, less strength than Pozzutec 20 at 28 days. Based on the room temperature results, the ARL-506 and EX-4 were excluded from further consideration.

Table 14 shows the results of combining propylene glycol and urea, two freeze-point depressants not previously examined, with Pozzutec 20. The purpose of doing this was to determine if simply adding a freeze-point depressant to Pozzutec 20 would enhance its low-temperature

Table 13. Strength results from two trial admixtures in concrete with a 365-kg/m^3 (611 lb/yd^3) cement factor and a 0.48 w/c.

Admixture code name-dosage mL/kg (fl oz/cwt)	*Curing temperature (°C)*	*Compressive strength—MPa (psi)* 7 days	14 days	28 days
Control	20	28.3 (4102)	31.7 (4593)	34.2 (4960)
EX-4-60 (90)	20	27.2 (3947)	27.6 (4008)	30.1 (4371)
EX-4-100 (150)	20	29.4 (4258)	32.3 (4682)	35.0 (5079)
ARL-506-60 (90)	20	33.4 (4848)	36.0 (5225)	37.8 (5479)
ARL-506-100 (150)	20	33.2 (4810)	37.0 (5362)	39.6 (5748)
Pozzutec 20-100 (150)	20	35.4 (5140)	38.9 (5645)	41.6 (6036)
Control	–5	1.1 (164)	1.2 (180)	1.8 (260)
EX-4-60 (90)	–5	9.0 (1306)	11.4 (1649)	12.5 (1815)
EX-4-100 (150)	–5	12.4 (1797)	16.2 (2348)	19.0 (2761)
ARL-506-60 (90)	–5	8.3 (1202)	11.8 (1707)	13.7 (1985)
ARL-506-100 (150)	–5	8.6 (1254)	13.5 (1964)	18.7 (2716)
Pozzutec 20-100 (150)	–5	8.4 (1211)	12.1 (1752)	16.2 (2349)
Control	–10	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
EX-4-60 (90)	–10	3.4 (492)	3.4 (496)	3.9 (570)
EX-4-100 (150)	–10	3.3 (482)	5.1 (738)	7.0 (1012)
ARL-506-60 (90)	–10	1.8 (259)	2.2 (312)	2.8 (410)
ARL-506-100 (150)	–10	1.4 (203)	2.9 (414)	4.2 (610)
Pozzutec 20-100 (150)	–10	4.4 (645)	5.5 (799)	8.6 (1243)

Table 14. Strength results from Pozzutec 20 (P20) and propylene glycol (PG) and urea with a 420-kg/m^3 (705 lb/yd^3) cement factor and a 0.43 w/c.

Admixture dosed by weight of active ingredient per 100 lbs of cement given in percent.	*Curing temperature (°C)*	*Compressive strength—MPa (psi)* 7 days	14 days	28 days
Control	20	32.2 (4668)	38.5 (5588)	42.1 (6104)
1.5% P20 + 4.5% PG	20	32.6 (4720)	40.0 (5800)	41.8 (6059)
3% P20 + 3% PG	20	35.1 (5088)	41.7 (6048)	44.6 (6470)
4.5% P20 + 1.5% PG	20	36.9 (5357)	42.2 (6126)	46.2 (6705)
1.5% P20 + 4.5% Urea	20	28.1 (4074)	34.9 (5065)	37.6 (5456)
Control	–5	0.7 (101)	2.9 (415)	4.2 (606)
1.5% P20 + 4.5% PG	–5	11.3 (1636)	22.1 (3206)	27.7 (4022)
3% P20 + 3% PG	–5	14.4 (2089)	25.0 (3618)	28.5 (4131)
4.5% P20 + 1.5% PG	–5	16.3 (2365)	27.0 (3908)	32.4 (4697)
1.5% P20 + 4.5% Urea	–5	13.3 (1924)	22.2 (3226)	27.1 (3928)
Control	–10	0.0 (0.0)	0.8 (111)	1.2 (177)
1.5% P20 + 4.5% PG	–10	0.5 (78)	5.2 (751)	8.4 (1223)
3% P20 + 3% PG	–10	1.7 (248)	5.9 (850)	11.6 (1677)
4.5% P20 + 1.5% PG	–10	1.8 (260)	5.7 (825)	9.1 (1318)
1.5% P20 + 4.5% Urea	–10	3.8 (552)	9.3 (1349)	12.9 (1866)

capability without diminishing its room-temperature strength gain. All mixtures used a cement factor of 420 kg/m^3 (705 lb/yd^3) and a w/c of 0.43. At room temperature, the combination of 4.5% Pozzutec 20 plus 1.5% propylene glycol provided the best strength gain compared to the control mixture. It provided a 10% strength gain over that of the control mixture at 28 days, which, unfortunately, was less than the approximately 20% strength increase provided by just Pozzutec 20 in Phase I. At –5°C (23°F), this same combination provided a 28-day strength equal to 77% of the room temperature control mixture. This was better than with Pozzutec 20 alone in Phase I where it provided a –5°C (23°F) strength equal to only 65% of the room-temperature control mixture at 28 days. Since neither of these two freeze-point depressants are routinely used by the concrete industry, they were not considered further in this CPAR project. However, it did appear that the low-temperature range of Pozzutec 20 could be extended by combining it with certain chemicals.

Tables 15 and 16 show the results of three trial admixtures coded EY-11, EZ-3B, and EZ-4B. The Table 15 mixtures had a cement factor of 365 kg/m^3 (611 lb/yd^3) and a w/c of 0.48, while the Table 16 mixtures had a cement factor of 420 kg/m^3 (705 lb/yd^3) and a w/c of 0.43. The Table 15 mixtures were tested at 60 and 100 mL/kg (90 and 150 fl oz/cwt), while the Table 16 mixes were tested at 100 mL/kg (150 fl oz/cwt) only. At room temperature, all three admixtures provided about the same strength results as those attained by the control. They did not enhance strength as much as did Pozzutec 20. Though it did not cause enhanced strength at room temperature at the dosage tested, the EY-11 provided the highest 28-day strength at –5°C (23°F) of all the admixtures tested. (Note that such high dosages will most likely not be used at room temperature.) Consequently, EY-11 was selected as the admixture for continued study in Phase III. Another admixture, EZ-3B, appeared to provide

Table 15. Strength results from three trial admixtures in concrete with a 365-kg/m^3 (611 lb/yd^3) cement factor and a 0.48 w/c.

Admixture code name-dosage mL/kg (fl oz/cwt)	*Curing temperature (°C)*	*Compressive strength—MPa (psi)* 7 days	14 days	28 days
Control	20	35.0 (5074)	40.3 (5843)	41.9 (6073)
EY-11 60 (90)	20	33.3 (5423)	37.4 (5423)	40.5 (5866)
EY-11 100 (150)	20	34.2 (4961)	39.1 (5663)	41.8 (6055)
EZ-3B 60 (90)	20	33.4 (4843)	38.3 (5555)	39.4 (5720)
EZ-3B 100 (150)	20	34.0 (4937)	38.0 (5503)	40.6 (5880)
EZ-4B 60 (90)	20	33.7 (4885)	38.9 (5644)	41.5 (6017)
EZ-4B 100 (150)	20	32.8 (4763)	36.9 (5352)	40.6 (5885)
Control	–5	1.7 (245)	3.6 (521)	3.4 (499)
EY-11 60 (90)	–5	20.2 (2928)	25.1 (3645)	26.5 (3848)
EY-11 100 (150)	–5	24.8 (3598)	32.1 (4654)	35.5 (5154)
EZ-3B 60 (90)	–5	19.1 (2768)	23.5 (3405)	24.9 (3607)
EZ-3B 100 (150)	–5	23.1 (3348)	30.8 (4461)	33.2 (4819)
EZ-4B 60 (90)	–5	19.5 (2829)	23.5 (3405)	23.8 (3452)
EZ-4B 100 (150)	–5	24.4 (3687)	30.9 (4475)	34.6 (5022)
Control	–10	0.0 (0.0)	0.8 (115)	1.0 (144)
EY-11 60 (90)	–10	3.7 (540)	5.8 (842)	4.9 (714)
EY-11 100 (150)	–10	5.3 (763)	8.6 (1242)	6.3 (909)
EZ-3B 60 (90)	–10	3.8 (548)	6.0 (865)	5.6 (811)
EZ-3B 100 (150)	–10	5.4 (790)	8.5 (1228)	7.4 (1078)
EZ-4B 60 (90)	–10	4.2 (602)	6.4 (934)	6.2 (898)
EZ-4B 100 (150)	–10	5.3 (773)	7.9 (1146)	6.9 (995)

Table 16. Strength results from three trial admixtures in concrete with a 420-kg/m^3 (705 lb/yd^3) cement factor and a 0.43 w/c.

Admixture code name-dosage mL/kg (fl oz/cwt)	*Curing temperature (°C)*	*Compressive strength—MPa (psi)* 7 days	14 days	28 days
Control	20	36.0 (5216)	38.4 (5573)	42.0 (6083)
EY-11 100 (150)	20	35.5 (5152)	37.8 (5474)	39.8 (5767)
EZ-3B 100 (150)	20	33.6 (4876)	35.7 (5182)	38.1 (5526)
EZ-4B 100 (150)	20	33.0 (4782)	35.5 (5145)	37.7 (5470)
Control	–5	2.8 (410)	3.9 (562)	4.2 (607)
EY-11 100 (150)	–5	24.0 (3478)	27.2 (3942)	30.5 (4423)
EZ-3B 100 (150)	–5	24.1 (3494)	30.1 (4371)	33.8 (4895)
EZ-4B 100 (150)	–5	21.8 (3160)	28.0 (4060)	28.4 (4117)
Control	–10	0.4 (65)	0.9 (135)	0.9 (127)
EY-11 100 (150)	–10	5.6 (806)	7.6 (1107)	8.7 (1263)
EZ-3B 100 (150)	–10	6.7 (973)	8.0 (1160)	10.0 (1448)
EZ-4B 100 (150)	–10	5.9 (861)	7.7 (1120)	8.1 (1168)

Table 17. Phase III tasks.

Task	*Description*
1	Strength vs. temperature
2	Corrosion potential
3	Durability
4	Equivalent insulation

somewhat higher strengths at –5°C (23°F) than with EY-11, and could have been a prototype alternate for that reason, but it was discovered in time to be relatively unstable (i.e., it tended to precipitate out of solution) and was therefore abandoned.

PHASE III: EVALUATION OF IMPROVED ADMIXTURE

Procedure

The objective of Phase III was to more fully evaluate the best Phase II admixture. Phase III used all Phase I procedures except for one: the critical strength test. Thus, Phase III consisted of four experimental tasks (Table 17). The procedures used in this phase have been explained in Phase I.

Results and discussion

Task 1: Strength vs. temperature

As was done in Phase I, the concrete was mixed at room temperature and immediately after casting placed into 20, –5, and –10°C (70, 23, 14°F) rooms for curing. Table 18 shows the strength results for two cement factors. At room temperature, EY-11 provided concrete of essentially the same strength as that of the control concrete. Though the EY-11 did not enhance strength in the way Pozzutec 20 is capable of, it did not interfere with strength gain at room temperature, which was an important consideration of this project. At –5°C (23°F), EY-11 promoted strength that exceeded that of control concrete cured at 5°C (40°F). Figure 6 illustrates this result. In that figure, the 5°C (40°F) reference strength was based on guidance given in ACI 1988. For the 365-kg/m^3 (611 lb/yd^3) cement factor, the EY-11 concrete exceeded the ACI reference strength at all ages except for 28 days (Fig. 6a). However, this is not considered a problem because the concrete has the potential of recovering full strength when brought back

Table 18. Compressive strength, MPa (psi), of the EY-11 mixtures. The 365-kg/m^3 (611 lb/yd^3) cement factor had a w/c of 0.48 and the 420-kg/m^3 (705 lb/yd^3) cement factor had a w/c of 0.43. The second number of the ID refers to EY-11 dosage.

Mixture ID	*Age—days* 7	14	28
2,0,20	30.5 (4423)	33.0 (4785)	38.4 (5572)
2,2,20	27.3 (3962)	31.4 (4550)	34.6 (5024)
2,3,20	27.5 (3988)	31.7 (4596)	34.0 (4933)
2,2,–5	14.4 (2086)	16.8 (2430)	18.9 (2745)
2,3,–5	16.6 (2406)	25.4 (3690)	27.6 (3998)
2,2,–10	4.8 (699)	5.6 (813)	6.7 (965)
2,3,–10	5.9 (852)	8.8 (1283)	10.6 (1538)
3,0,20	34.5 (5003)	37.4 (5429)	39.4 (5718)
3,2,20	30.7 (4456)	34.5 (4998)	37.8 (5479)
3,3,20	32.2 (4671)	36.6 (5307)	39.7 (5761)
3,2,–5	19.4 (2808)	22.4 (3246)	24.2 (3503)
3,3,–5	22.7 (3288)	27.6 (4005)	33.1 (4795)
3,2–10	6.1 (887)	6.7 (968)	7.8 (1130)
3,2,–10	7.5 (1081)	9.7 (1406)	11.3 (1638)

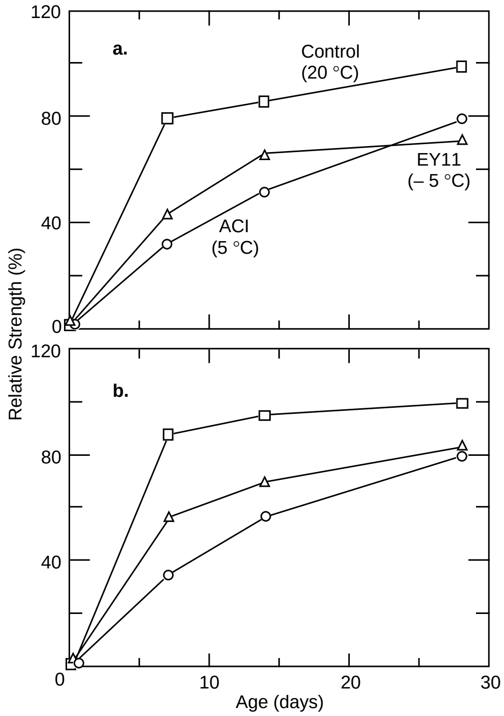


Figure 6. Strength gain of concrete made with EY-11 cured at –5°C (23°F) compared to control concrete cured at two above-freezing temperatures. The line denoted as ACI (5°C) is based on guidance provided by ACI (1988). That line represents the minimum curing condition used by the construction industry today. Figure 6a is for concrete containing a 365-kg/m^3 (611 lb/yd^3) cement factor and a 100-mL/kg (150 fl oz/cwt) EY-11 dosage. Figure 6b contains a 420-kg/m^3 (705 lb/yd^3) cement factor and a 100-mL/kg (150 fl oz/cwt) EY-11 dosage.

to warm conditions. The 100-mL/kg (150 fl oz/cwt) dose with the 420-kg/m^3 (705 lb/yd^3) cement factor exceeded the ACI reference strength at all ages (Fig. 6b).

Task 2: Corrosion potential

The potential of EY-11 to corrode steel reinforcement was tested according to the so-called "Harmlessness Test" (modeled after a German DIN standard according to discussions during meetings of ASTM Committee G-1.14 1994–95). The method employed in this project uses small "lollipop" cylinder specimens measuring 50 × 100 mm (2 × 4 in.). The mortar used Type I cement, an ASTM C 109 sand in a 1:3 cement:sand ratio and a 0.50 w/c. The embedded rebar is a No. 4 axially located 25.4 mm (1 in.) off the bottom of the cylinder and protruding from the top. The test area of the rebar is limited to 30 cm^2 (4.7 in^2) by epoxy paint. The specimens were cured for four days in saturated calcium hydroxide solution to within 12.7 mm (0.5 in.) of the top surface. They were then kept at a potential of +260 mV vs. a saturated calomel electrode. The current flowing through a 1000-ohm resistor placed in the circuit is measured at regular intervals by voltage drop across the resistor. If the current density is below 1 μA/cm^2, the admixture is considered not harmful.

Table 19. Harmlessness corrosion results.

Admixture	*Dosage mL/kg (fl oz/cwt)*	*Current μA/cm^2*
Pozzutec 20	30 (45)	0.539
Pozzutec 20	60 (90)	0.405
EY-11	50 (75)	0.724
EY-11	100 (150)	0.651

Table 19 shows the results for Pozzutec 20 and EY-11. Both admixtures provided results below 1 μA/cm^2, indicating that neither admixture caused corrosion at the dosages used.

One of the corrosion measuring methods used in Phase I to measure the potential of Pozzutec 20 to initiate corrosion damage to embedded steel rebar, the Lollipop Corrosion Test, was again used to measure the potential of EY-11 to initiate corrosion. Two dosages of EY-11 were used (60 and 100 mL/kg [90 fl oz/cwt]), the result being compared in the same test with the same two dosages of Pozzutec 20 and two references without admixture. The specimen size was 75- × 150-mm (3 × 6 in.) cylinders, each concrete mix being prepared, and the concrete specimens cured and otherwise treated, in the same manner as were the earlier lollipop examinations of Phase I, except that 15% sodium chloride solution was used for ponding in place of the 3% solution of Phase I; weekly measurements were taken. Figure 7 shows that EY-11 caused corrosion to be initiated at about week 12 with the higher dosage (100 mL/kg, 150 fl oz/cwt) and around week 43 with the lower dosage (60 mL/kg, 90 fl oz/cwt). Pozzutec 20, on the other hand, was found to initiate corrosion at earlier times, at about week 6 with the higher dosage and week 23 with the lower, each dosage causing initiation to occur earlier by about one-half the time period. Two admixture-free reference specimens were shown to have initiated corrosion at weeks 39 and 43 for an average of 41 weeks for the two references. The trial admixture EY-11, therefore, was found in this 75- × 150-mm

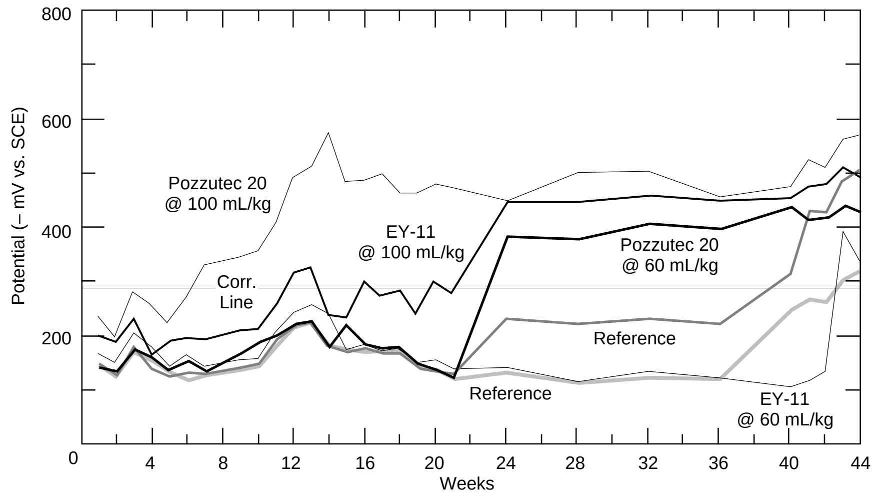


Figure 7. Lollipop specimens, 75- × 150-mm (3 × 6 in.) cylinders ponded half-height in sodium chloride solution.

test specimen not to have initiated corrosion with the lower dosage of 60 mL/kg (90 fl oz/cwt), but to have initiated corrosion at the higher dosage level. Likewise, Pozzutec 20 was found to have initiated corrosion, with the higher dosage causing damage earlier than the lower 60-mL/kg dosage.

Another similar test was run using the same lollipop method, but this time with only 50- × 100-mm (2 × 4 in.) cylinder specimens, to determine if specimen size mattered. The same dosage levels of the two admixtures were repeated, as were the two references. Figure 8 shows that the two admixture-free references initiated corrosion at Weeks 15 and 16, while EY-11 at the high and low dosages initiated corrosion at 8 and 21 weeks, respectively, and Pozzutec 20, again at the high and low dosages, initiated corrosion at two and ten weeks, respectively. Therefore, like the larger cylinders, the lower dosage only (60 mL/kg, 90 fl oz/cwt) of EY-11 did not cause corrosion initiation, and provided evidence that EY-11 was potentially less corrosive to steel rebar. The higher EY-11 dosage (150 fl oz) initiated corrosion at an even later time than the lower dosage (90 fl oz) of Pozzutec 20. Also, as for the specimen size, the smaller the test cylinder, the earlier the initiation of corrosion. It is most important here to restate that the admixtures Pozzutec 20 and EY-11 did not cause corrosion to occur without chloride ions

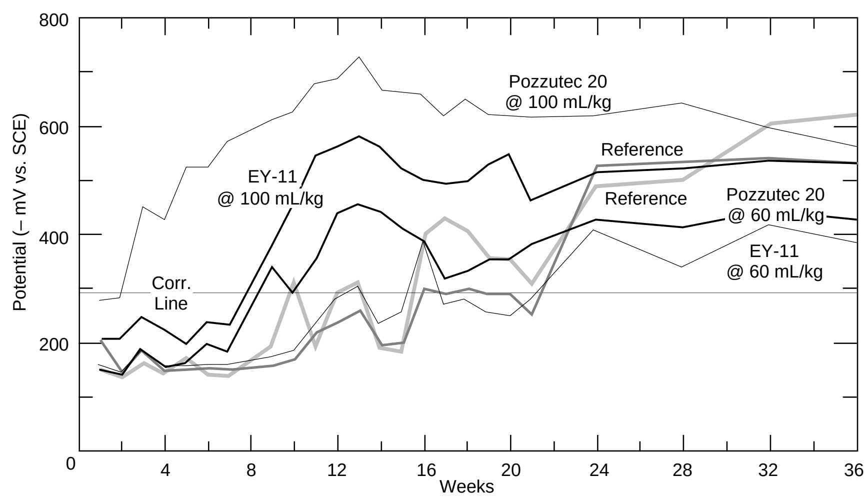


Figure 8. Lollipop specimens, 50- × 100-mm (2 × 4 in.) cylinders ponded half-height in sodium chloride solution.

Table 20. Durability factors for Pozzutec 20 and EY-11 concrete.

Admixture	*None*	*Dosage* 60 (90) mL/kg (fl oz/cwt)	100 (150) mL/kg (fl oz/cwt)
Control	99		
Pozzutec 20		99	Failed
EY-11		98	96

Table 21. Equivalent insulation test results.

Mixture ID	*Compressive strength—MPa (psi)* 7 days	14 days	28 days
3,0,10 (control)	*27.9 (4052)*	*39.8 (5767)*	*43.8 (6348)*
3,2,5	32.4 (4691)	36.7 (5326)	39.8 (5771)
3,2,2	30.6 (4439)	32.3 (4685)	35.3 (5112)
3,2,–2	26.5 (3846)	35.7 (5170)	38.2 (5543)
3,3,5	33.0 (4778)	36.9 (5349)	42.1 (6098)
3,3,2	30.8 (4465)	34.1 (4941)	35.8 (5190)
3,3,–2	26.3 (3817)	34.3 (4970)	38.8 (5628)

Table 22. Equivalent insulation values for 152-mm- (6 in.) thick wall maintained at 10°C (50°F) for seven days.

Mixture	*Air temperature* ℃ (℉)	*Required thermal resistance* m^2 K/W (hr ft^2 F/Btu)	*Equivalent fibrous glass—mm (in.)*
3,2	–1 (30.2)	1.1 (6.5)	50 (2.0)
3,3	–1 (30.2)	1.1 (6.5)	50 (2.0)

present. It appears that the higher dosages of these admixtures may decrease the chloride threshold.

Task 3: Durability

The freeze–thaw durability of concrete made with Pozzutec 20 and EY-11 was tested using ASTM C 666, Procedure A, and evaluated according to ASTM C 494. Table 20 shows the results. As happened in Phase I, Pozzutec 20 passed the durability test at a dosage of 60 mL/kg (90 fl oz/cwt) but not at 100 mL/kg (150 fl oz/cwt). EY-11, on the other hand, showed very high durability at both dosages.

Task 4: Equivalent insulation

The purpose of this task was to determine the amount of insulation that EY-11 can replace in a 420 kg/m^3 (705 lb/yd^3) cement factor mix. Table 21 presents the strength results at various low temperatures. Since EY-11 does not enhance the late age strength of concrete (Phase III, Task 1) when cured at room temperature, the effect of EY-11 was evaluated only at the seven-day strength. EY-11 was found to increase compressive strength relative to 10°C (50°F) down to approximately –1°C (30°F) for both dosages.

Table 22 shows that EY-11 is equivalent to a thermal resistance of 1.1 m^2 K/W (6.5 hr ft^2 F/Btu), or about 50 mm (2 in.) of fibrous glass insulation.

PHASE IV: FIELD APPLICATION

Procedure

The objective of Phase IV was to validate the EY-11 admixture developed in Phase III by means of a field trial. Special attention was given to workability, finishability, temperature records, and strength development.

The prototype admixture (EY-11) was tested outdoors at CRREL, Hanover, New Hampshire, and at the Corps of Engineers Soo Locks, Sault Ste. Marie, Michigan, during February and March 1994. The CRREL site was chosen because of its proximity to testing facilities and because it provided a location convenient for long-term monitoring of the concrete. The Soo Locks was attractive because it provided an opportunity to compare normal winter concreting to concreting with antifreeze admixtures. The timing at each site was determined from weather records and forecasts that promised weather conditions appropriate to the –5°C (23°F) capability of the admixture. A technical representative from MB was on hand to evaluate the effectiveness of the admixture with the cements used at each site. CRREL personnel provided instrumentation for monitoring temperatures and helped measure properties of the fresh and hardened concrete. Pozzutec 20 was used to batch a separate mix of concrete for comparison purposes.

Results and discussion

New Hampshire

Test site. At CRREL, a composting bin consisting of a 16.5-cm- (6.5 in.) thick reinforced slab on grade 3.7 m wide by 4.6 m long (12 × 15 ft) with 1.2-m- (4 ft) high reinforced 203-mm- (8 in.) thick walls on three sides was cast during 17 and 18 February. The bin was oriented such that the long axis of the slab ran east–west, and the three walls formed the east, south, and west sides of the bin. The north wall was omitted. The bin was divided into five sections, three wall sections and two slab sections. Dividing the bin in this manner allowed for five admixtures to be evaluated. This report discusses the two admixtures provided by Master Builders: Pozzutec 20 and EY-11.

Site preparation consisted of removing a meter of snow from the ground, placing about 100 mm (4 in.) of dry sand on the newly exposed frozen ground, and setting the forms and reinforcing steel on the sand. The concrete was placed in the forms, consolidated, and finished as usual. A plastic sheet was placed over the slab and over the top of the wall for three days to minimize water loss. The wooden forms were removed from the walls 20 hours after the concrete was placed. No thermal protection was provided to the concrete. Plastic pullout cylinders, 100 × 150 mm (4 × 6 in.), were cast into the slab and the top of the wall to provide in-situ strength gain results. No control concrete was cast at the site during this study.

Workability/finishability. The initial slump of the EY-11 mix as delivered to the site was poor. The original concern was that the 6% dosage (Table 23) of EY-11 was causing the cement to set up too rapidly but, as explained later, a low water and plasticizer content contributed to this low slump. The Pozzutec 20 mix used for the west half of the slab had good workability, although the concrete workers complained that the concrete seemed to tear when finished with a trowel. There was no apparent reason for this problem as ice was not forming on the bottom of the trowels despite the cold weather. The high slump, as discussed later, may have contributed to this finishing problem. The EY-11 was placed in the west wall and in the west third of the south wall, so finishing characteristics could not be evaluated for this admixture.

Table 23 gives the proportions of the two concrete mixtures used in this study. Table 24 gives the properties of fresh concrete. As previously described, Pozzutec 20 was used in the slab and EY-11 in the wall. The 4% dosage of Pozzutec 20 is equivalent to 60 mL/kg (90 fl oz/cwt) used elsewhere in this report. Likewise, the 6% EY-11 equates to 95 mL/kg (145 fl oz/cwt).

The target water-to-cement ratio was 0.44 with a slump of 100 mm (4 in.). The Pozzutec 20 and EY-11 mixes differed from this target, especially in w/c. The water content of the Pozzutec 20 mix was intentionally reduced below the target value at the mix plant because Pozzutec 20 contains a high-range water reducer and the mix plant normally adds a plasticizer to this mixture. The 0.39 w/c resulted in a relatively high slump of 210 mm (8.25 in.) (Table 24). Based on this result, and because EY-11 also contained a high-range water reducer, the water content of the EY-11 mixture was held to 0.40 at the mix plant. Also, the mix plant was requested not to add plasticizer. The EY-11 concrete unexpectedly arrived at the site with no measurable slump. Thus, water was carefully added to the mix until the concrete in the truck was noticeably looser. The extra water produced slump of 127 mm (5 in.) (Table 24) and a 0.55 w/c (Table 23). The resulting mix was easy to place and consolidate within the wall forms. Note that the concrete temperatures of both placements were above freezing, not the more desirable below freezing.

Thermal record. Five thermocouples were equally positioned through the thickness of the

Table 23. Mixture proportions.

Mix no.	*Rock 3/4-in. crushed ledge, 0.5% abs 2.89 SG kg/m (lb/yd^3) ^{3}*	*Sand natural 1.1% abs 2.71 SG kg/m^3 (lb/yd^3)*	*Cement Type II portland kg/m (lb/yd^3) ^{3}*	*w/c ratio*	*Air entraining agent (Microair) mL/m^3 (fl oz/yd^3)*	*Water reducer added at mix plant (WRDA w/Hycol) mL/m^3 (fl oz/yd^3)*	*Admixture dosage (wgt active ingredient per cement wgt) (%)*
P20	1012 (1700)	788 (1323)	421 (707)	0.39	798 (27)	769 (26)	4
EY-11	1027 (1725)	777 (1305)	420 (705)	0.55	325 (11)	none	6

slab and six through the wall beginning at one surface and ending at the other. An additional thermocouple was positioned away from the concrete out of direct sunlight to record ambient air temperature. A malfunction of the data recorder eliminated some temperature recordings from portions of days two through five. Some thermocouple locations were unable to be read at all due to apparent problems with the sensors themselves.

Figures 9–11 provide the recorded temperature histories. Table 25 gives the approximate times when each concrete was placed. The air temperature (Fig. 9) averaged –1.4°C (29.5°F) over the first five days, with a high of 10°C (50°F) and a low of –16°C (–3.2°F), while the concrete averaged 2.2°C (36.0°F) over that same period. The air temperature on the 17th (day 1) began at –16°C (–3.2°F) at 7:30 a.m., rose to a high of 4.5°C (40.1°F) at 2 p.m., and then dropped off to well below freezing that night. The slab concrete (with Pozzutec 20) temperature (Fig. 10) at placement (12:30 p.m.) was 10°C (50°F). It cooled to about

Table 24. Properties of fresh concrete.

Mix	*Slump mm (in.)*	*Air (%)*	*Unit wgt $kg/m^3(lb/ft^3)$*	*Concrete °C (°F)*
P20	210 (8.25)	4.4	2389 (149)	10 (50)
EY-11	127 (5.00)	4.0	2357 (147)	16 (61)

Table 25. Concrete placement time.

Mix	*Date*	*Start*
P20	17 Feb	12:30 p.m.
EY-11	18 Feb	12:05 p.m.

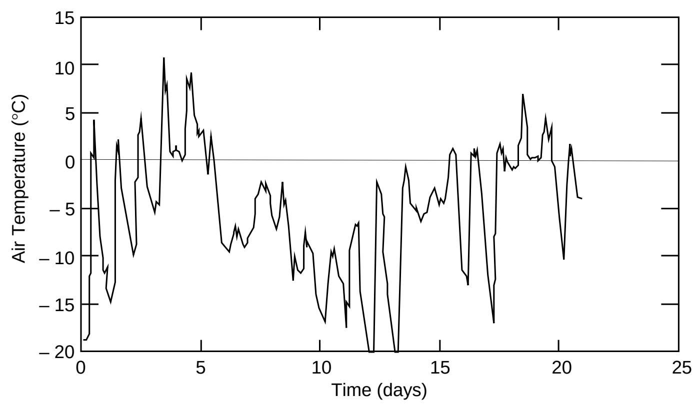


Figure 9. Air temperatures from 7:30 a.m., 17 Feb, through 12:30 a.m., 10 Mar 1994, at Hanover, New Hampshire.

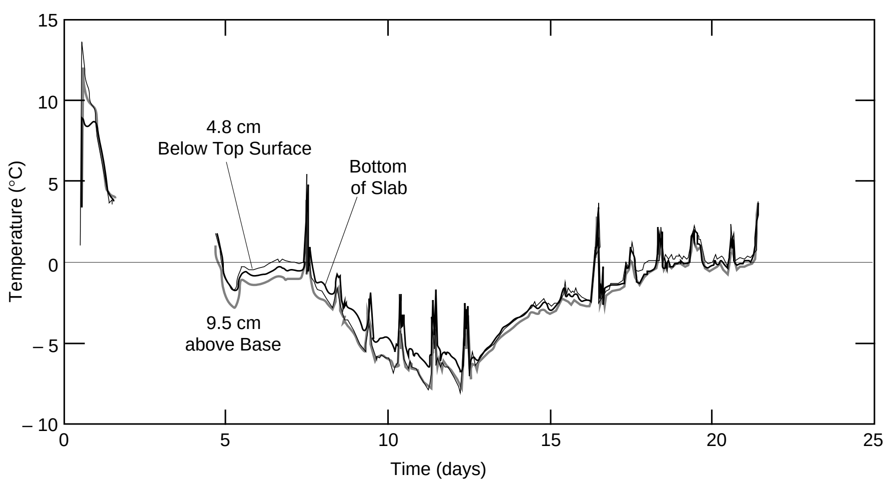


Figure 10. Temperature history of the Pozzutec 20 concrete slab placed on grade at Hanover, New Hampshire. The slab was placed at 12:30 p.m. on 17 Feb (day 1). A malfunction of the data recorder eliminated temperatures from portions of day 2 through day 5.

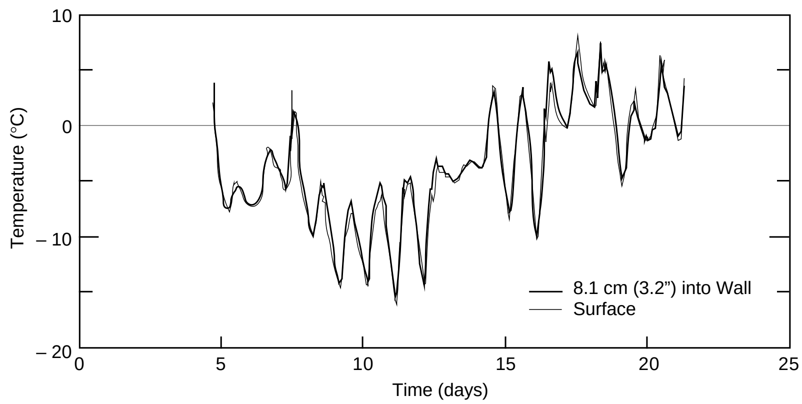


Figure 11. Temperature history of the EY-11 concrete wall placed at 12:05 p.m. on 18 Feb (day 2) at Hanover, New Hampshire. A malfunction of the data recorder eliminated temperature records until 21 Feb (day 5).

3°C (37°F) when it came in contact with the cold ground but quickly rose to 13.2 °C (55.8°F) by 1:00 p.m. and then began to cool. A malfunction of the temperature recorder prevented recordings from 18 Feb at 12:30 a.m. to 21 Feb at 4:30 p.m. Although the air temperature during the first three nights got quite cold, –15 °C (5°F) at 6:30 a.m. on the 18th (day 2), –10.3 °C (13.5°F) at 6 a.m. on the 19th (day 3), and –5.4 °C (22.3°F) at 2 a.m. on the 20th (day 4), the concrete did not freeze. A petrographic examination of core samples drilled from the concrete confirmed this. Data from a separate project show that a slab placed next to this slab at 9 a.m. on the same day dropped to a low of only –1.2°C (29.8°F) on 19 Feb (day 3). This kind of temperature would not have damaged the Pozzutec 20 slab. The Pozzutec 20 slab cooled to below –5°C (23°F) at 3 a.m. on the 26th (day 10), and remained below that temperature until 7 a.m. on 2 March, a five-day period. It then rose slowly the next seven days to near 0°C (32°F) on 10 March. Notice that the slab was close to uniform temperature throughout the recording period. The three temperature recordings (two other thermocouples malfunctioned) nearly overlay one another. Because of the closeness of the recorded temperatures, no attempt was made to distinguish the significance of one line from another.

The wall with admixture EY-11 was placed on 18 Feb (day 2) at 12:05 p.m. at a concrete temperature of 16°C (61°F). Unfortunately, the recorder malfunction prevented any temperature record until 21 Feb (day 4) at 4:30 p.m. Two temperature histories, one on the surface and one internal temperature, are plotted in Figure 11. A petrographic examination of core samples obtained in May shows that the wall did not suffer frost damage.

This was not a severe test of the low-temperature capability of either admixture because the ambient and concrete temperatures both were above freezing.

Strength. Results of the strength tests from the field-cured pullout cylinders taken from each concrete section are presented in Table 26. Though no control concrete was cast at the site for direct comparison to the pullout cylinder strength results, the strength of admixture-free concrete of similar mix design with a 0.44 w/c ratio cured at room temperature is given. As can be seen, the field samples exceeded the 28-day strength of the room-cured concrete. This is remarkable for the EY-11 owing to its relatively high w/c of 0.55.

Michigan

Test site. The second field test was conducted in northern Michigan in March 1994. The Corps' Soo Area Office had scheduled 39 sections of concrete to be replaced because of their advanced stage of freeze–thaw deterioration. The work area was located on the southwest pier, which borders the ship canal of the Poe Lock, the largest of four locks operated and maintained by the Corps of Engineers, Sault Ste. Marie, Michigan. Inspection

Table 26. Strength results, MPa (psi), from pullout cylinders cast into the concrete.

Mixture	*7 days*	*28 days*
Pozzutec 20	27.4 (3975)	48.3 (7010)
EY-11	20.3 (2949)	38.1 (5526)
Room-cured admixture-free	30.9 (4480)	37.6 (5451)

and repair of the locks themselves is normally done during the winter months, January through March, when shipping is stopped. Other repair work, such as the replacement of the slabs described here, is also most conveniently done during the winter nonshipping season, making this test particularly relevant.

For this test, four reinforced slabs on grade were selected for testing two admixtures. Each slab measured 5.5 m wide by 6.1 m long by 150 mm thick (18 × 20 ft × 6 in.). The two admixtures tested were EY-11 and Pozzutec 20. The EY-11 admixture was used in two dosages: low and high, designated EY11L and EY11H. The Pozzutec 20 admixture was used in a single dosage. The four test slabs were cast between 15 and 16 March.

Site preparation consisted of jackhammering out alternate sections of concrete, replacing 150 mm (6 in.) of base material with an equal amount of coarse crushed stone, and setting forms and reinforcing steel. The slabs that remained between the removed sections provided work space for finishing operations. A temporary heated enclosure was erected over one slab to serve as a control section and to provide a comparison between normal and antifreeze concrete operations. A second enclosure, unheated, was used to cover the EY11L admixture section as a secondary test. Admixture-free concrete was placed in the heated shelter while concretes made with the EY11H and Pozzutec 20 admixtures were placed in sections exposed to ambient air outside the shelter.

The concrete was placed and finished in the normal fashion. Except for the heated control section, the concrete remained thermally unprotected. A plastic sheet was placed over the two exposed concrete sections for seven days to minimize water loss. The concrete in the two shelters was left uncovered. Thermocouples connected to data loggers monitored concrete and air temperatures. Numerous 75- × 150-mm (3 × 6 in.) cylindrical samples were cast from each concrete section and stored in two locations next to the slabs on grade and overhead in the heated enclosure. A concrete testing laboratory in northern Michigan tested the cylinders for compressive strength at regular intervals.

The concrete was transported by rotary-drum truck from a ready-mix plant 8 km (5 mi) from the job site. The concrete was mixed with unheated aggregate and heated water. The ingredients, including all admixtures, were mixed before being added into the truck. The mix proportions are given in Table 27. Table 28 gives the concrete placement times. The concrete was delivered 30 to 45 minutes after water was added to the mix, and placed within another 30 minutes. Consolidation and finishing operations took another 45 to 60 minutes. Table 29 gives the properties of the fresh concrete.

Workability/finishability. The concrete for all sections was placed and finished in the normal fashion. No extra effort or skill was required to work outdoors compared to doing the same work in-

Table 27. Mix proportions.

Mix	*3/4 in. maximum size coarse aggregate kg/m^3 (lb/yd^3)*	*Sand kg/m^3 (lb/yd^3)*	*Cement (Type IA portland) kg/m^3 (lb/yd^3)*	*w/c ratio*	*Admixture dosage (wgt active ingredient per cement wgt) (%)*
Control	1047 (1760)	774 (1300)	392 (658)	0.41	None
EY11L	1047 (1760)	774 (1300)	392 (658)	0.41	3.7
EY11H	1047 (1760)	774 (1300)	392 (658)	0.38	6.3
Pozzutec 20	1047 (1760)	774 (1300)	392 (658)	0.39	4.0

Table 28. Concrete placement time.

Mix	*Date*	*Start*
Control	15 March	11:00 a.m.
EY11L	16 March	9:45 a.m.
EY11H	16 March	11:40 a.m.
Pozzutec 20	16 March	1:27 p.m.

Table 29. Properties of fresh concrete.

Mix	*Slump mm (in.)*	*Air (%)*	*Unit wgt kg/m^3 (lb/ft^3)*	*Temperature °C (°F)*
Control	51 (2)	3.2	2307 (144)	12.2 (54)
EY11L	140 (5.5)	3.2	2307 (144)	3.3 (38)
EY11H	140 (5.5)	4.7	2275 (142)	3.3 (38)
Pozzutec 20	150 (6)	3.4	2330 (145)	4.4 (40)

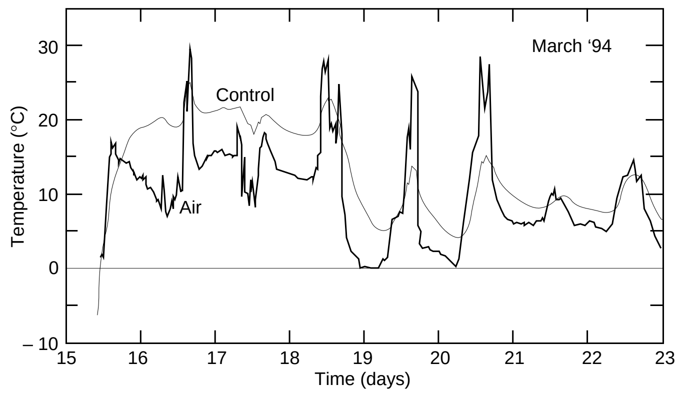


Figure 12. Temperature history of the top surface of the control slab and the heated air in the control shelter at Sault Ste. Marie, Michigan.

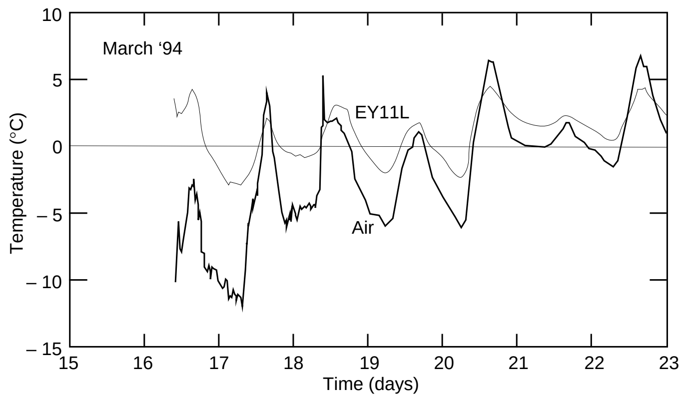


Figure 13. Temperature history of the top surface of the EY11L slab and that of the outdoor air at Sault Ste. Marie, Michigan.

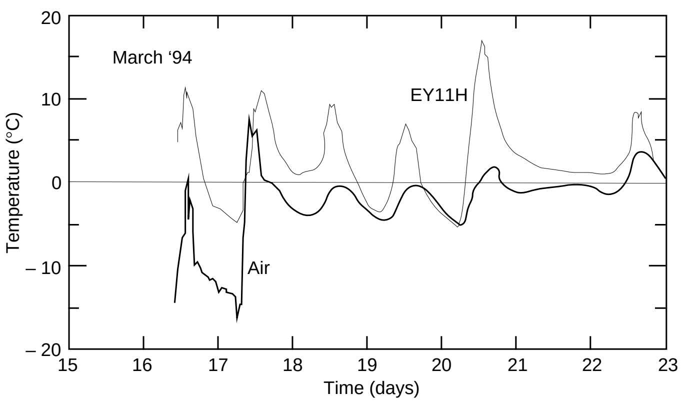


Figure 14. Temperature history of the top surface of the EY11H slab and that of the outdoor air at Sault Ste. Marie, Michigan.

side the heated shelter. The workers found the freedom of movement better outdoors than in a temporary enclosure, while the heated shelter was useful as a warming hut between concrete deliveries. The workers remained outdoors for periods of approximately two hours. The Pozzutec 20 and EY-11 concretes were very easy to place, consolidate, and finish, according to the concrete workers. The concrete maintained its workability throughout the finishing operation, which lasted nearly two hours after water was first added to the mixtures at the mix plant. According to workers' comments, the EY-11 mixture seemed to be somewhat easier to finish compared to the Pozzutec 20 or the control, though no difficulty was noted with working with any of the mixtures.

Thermal record. Thermocouples connected to data loggers monitored concrete and air temperatures. Five thermocouples were equally spaced throughout the thickness of each slab, beginning at the top surface. (The temperature of the Pozzutec 20 was not recorded due to equipment malfunction.) An additional thermocouple was positioned away from the concrete, 150 mm (6 in.) above grade and out of direct sunlight, to record the ambient air temperature. For this report, only the data from the top surface thermocouples are provided because the top surface was the coolest portion of each slab—it cooled quicker and experienced wider temperature excursions than the rest of the slab, including the bottom surface, which was in contact with the cold gravel. Figures 12–14 show the temperatures of the slabs' top surfaces and the temperature of surrounding ambient air. The recording period for each concrete section began at the time shown in Table 28 and extends through midnight, 22 March.

Figure 12 shows the temperatures of the control concrete and the heated air in the shelter. The shelter was heated for several days before 15 March to thaw the frozen ground. To facilitate placement of the control concrete, two walls of the shelter were removed at 10:30 a.m. on 15 March and replaced at noon. The air inside the shelter cooled to –6.6°C (20°F) by the time concreting started, but after the walls were replaced, the shelter warmed up again. However, the shelter temperature fluctuated daily. The maximum of 29.7°C (85°F) occurred at 4:10 p.m. on the 16th, and two lows of –0.2°C (31°F) and 0.4°C (33°F) occurred at 3:30 a.m. on the 19th and at 6:45 a.m. on the 20th, respectively. The two low temperatures were caused by a malfunction of the heating equipment. The heat was turned off about 4 p.m. on 22 March. The average air temperature in the shelter for the recording period was 10.5°C (51°F).

The control concrete was delivered to the site in two separate shipments, at a temperature of about 12°C (54°F) for each shipment. (All other concrete was delivered in one truck per section.) By the time both control shipments had been placed and the shelter walls were reinstalled, the concrete had cooled to 1.3°C (34°F) (Fig. 8). It wasn't until 5 p.m. of that same day that the heat supplied by cement hydration and the shelter warmed the concrete to 12°C (54°F). The concrete continued to warm until it reached 20.3°C (68.5°F) at 7 a.m., 16 March, in spite of the air cooling to 9.4°C (48.9°F). Like the air, the concrete temperature fluctuated throughout the recording period. It reached a maximum temperature of 25.3°C (77.5°F) at 4:10 p.m. on the 16th and a minimum of 3.8°C (38.8°F) at 7:10 p.m. on the 20th, closely corresponding to the high and low shelter air temperatures. The average temperature of the control concrete through 4 p.m. on 22 March was 13.3°C (55.9°F). It never dropped below 0°C during this period.

The EY-11 mixtures were placed on 16 March, the colder of the two days during which concrete was placed. The outdoor air temperature, shown in Figure 13 and again in 14, averaged a chilly –8.7°C (16.3°F) through midnight on the 16th, though it rose to slightly above freezing for a short time by midday, the 17th. The minimum outdoor air temperature of –16.5°C (2.3°F) was recorded at 6:45 a.m. on 17 March. Winds created wind chills down to –28°C (–18.4°F) during the 17th. Thereafter the outdoor air temperature became much milder. The average outdoor air temperature from 16 March through 22 March was –2.4°C (27.7°F).

Figure 13 shows the temperatures of the EY11L concrete and the air inside the unheated shelter. The EY11L mix was placed at 9:45 a.m. on 16 March. It was delivered at a temperature of 3.3°C (37.9°F). As was done with the control section, two walls of the unheated shelter were removed temporarily. When exposed to the –10°C (14°F) (but warming) air, the concrete temperature quickly dropped from its delivered temperature to 2°C (35.6°F), but almost immediately began rising, reaching 4.3°C (39.7°F) by 4 p.m. After that the concrete temperature dropped to –3°C (26.6°F), its lowest recorded temperature, at 3:30 a.m. on 17 March. This concrete contained a low admixture dosage and had an expected freezing point around –3°C (26.6°F). Its average temperature was 0.9°C (33.6°F) through 4 p.m. on 22 March.

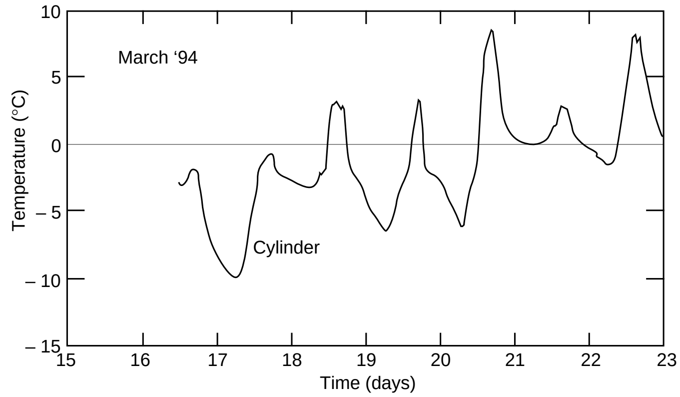


Figure 15. Temperature history of the center of mass of a 75- × 150-mm (3 × 6 in.) cylinder of EY11L concrete stored on grade in the unheated shelter at Sault Ste. Marie, Michigan.

Figure 14 shows the temperatures of the EY11H concrete and the outdoor air. The freezing point of this concrete was –5°C (23°F). The EY11H mix was cast outdoors at 11:40 a.m. on 16 March. It, too, began at 3.3°C (37.9°F). Instead of cooling when exposed to the –7.3°C (18.9°F) air, however, it warmed to 11.8°C (53.2°F) at 2:10 p.m. before dropping to –4.4°C (24.1°F) at 7 a.m. on 17 March. It reached its lowest temperature of –5.5°C (22.1°F) at 7 a.m. on 20 March, four days after being cast. Its average temperature was 2.4°C (36.3°F) through 4 p.m. on 22 March.

Figure 15 shows the temperatures of an EY11L cylinder stored on grade in the unheated shelter. The cylinder's temperature dipped below –5°C (23°F) on several occasions, the first at 8:00 p.m. on 16 March, about ten hours after it was cast. The average temperature of the cylinder through 4 p.m. on 22 March was –1.3°C (29.7°F).

Strength development. Several 75- × 150-mm (3 × 6 in.) cylindrical samples were cast from each type of concrete and stored in two locations on grade next to the slabs and overhead in the heated enclosure. A concrete testing laboratory in Michigan periodically tested the cylinders' compressive strength.

The compressive strengths of the cylinders cannot be used as an indicator of the in-place strength of the antifreeze concrete because, as Figure 15 shows, the cylinders probably froze. Subsequent petrographic analysis of the suspected frozen cylinders at CRREL revealed typical ice lens patterns in the cylinders. Strengths reported by the testing laboratory indicate that the cylinders de-

Table 30. Test results from 92- × 133-mm (4 × 5.25 in.) core samples drilled in July 1994. Densities are based on cylinder dimensions and mass. Minimum design strength was 32 MPa (4640 psi).

Mix	*Compressive strength MPa (psi)*	*Bulk density kg/m³ (lb/ft³)*	*Evidence of past ice?*
Control	46.7 (6770)	2310 (143.7))	No
EY11L	50.6 (7350)	2320 (144.4)	No
EY11H	53.2 (7720)	2290 (142.5)	No
Pozzutec 20	54.1 (7840)	2340 (145.6)	No

veloped only about half their potential strength, which is indicative of concrete that has frozen while curing.

Likewise, the strengths of the cylinders stored on an overhead shelf in the heated shelter were not considered useful information other than to confirm that the admixtures promoted strength in concrete cured at above-freezing temperatures. They shed little light on the in-place strength of the concrete slabs.

The most interesting and useful results came from cores drilled from each slab in the summer (Table 30). The cores showed that the antifreeze concrete was stronger than the control concrete in compression. None of the slabs showed signs of frost damage.

Cost comparison between conventional and antifreeze concrete. As previously mentioned, a heated shelter was used for the control concrete. This provided an opportunity to compare costs between normal winter concreting and concreting with antifreeze admixtures. Based on these field tests it became apparent that the main difference between normal concrete and antifreeze concrete is the heat, shelter, and labor needed to protect normal concrete compared to the chemicals needed to protect antifreeze concrete. The cost to erect, heat, and dismantle the temporary shelter at the Soo Locks was estimated to be $1,079.54 (Table 31). Heating accounted for close to 15 percent of this expense. Since antifreeze admixtures are still prototypes, their market price has not been determined. However, based on the estimate developed for the shelter, the cost of an antifreeze could potentially be as high as $21 per gallon.

Summary of both field tests. The New Hampshire field demonstration was not considered to be a severe enough test of the low-temperature capability of EY-11. The concrete did not freeze until it had gained considerable strength. However, this test showed that no special skills are needed to work with the prototype admixture at near-freezing conditions.

The northern Michigan field demonstration provided a good evaluation of EY-11 under severe conditions. Normal unprotected concrete would have frozen during this test. The freezing point depression and accelerated cure properties of the EY-11 concrete enabled it to resist freezing.

The best evidence that the concrete did not freeze was obtained by examining drilled cores. The core samples taken from each slab four months after construction and examined under a microscope showed no signs of frost damage.

The drilled cores were also tested for compressive strength, thereby providing additional information that the admixtures produced a concrete that was unaffected by the outdoor winter conditions.

Other than the cold weather, the major concern during the test was that concrete was placed on a subgrade that was significantly below the –5°C protection capability of the admixtures at their highest dosage, let alone at the low dosage. The concern was that the bottom of the concrete would be damaged by frost. Gavrish et al. (1974) reported that up to 16 times more heat is lost from a concrete slab to frozen ground than is lost to the air during initial curing. From our data, however, it was clear that the bottom of the concrete was free from frost damage. The lowest slab-bottom temperature of the low-dosage EY-11 concrete 21 hours after placement was about –1.2°C (30°F), and for the high-dosage EY-11 concrete four days after placement, it was –2.6°C (27.3°F). At these

Table 31. Winter cost estimate.

Shelter	
Erect shelter	
(6 men, 1/2 day @ $23/hr)	$552.00
Heat shelter - 1 d prior to pour and 7 d after	
(8 d @ 21.4 gal propane/d @ $0.78/gal)	$133.54
Dismantle shelter	$276.00
Materials—assume 9 reuses	
(Total cost estimated at $1,062)	$118.00
Total estimated cost of shelter	$1,079.54
Antifreeze admixture	
Volume of concrete placed inside shelter	6.7 yd³
Dosage of admixture per 100 lb of cement	150 fl oz
Amount of cement per yd³ of concrete	658 lbs
Amount of admixture per 6.7 yd³ of concrete	51.67 gal
Cost of admixture to equal cost of shelter	20.89/gal

temperatures and by these times, even admixture-free concrete may have been able to set and become resistant to freezing.

The test showed that a plastic sheet was capable of providing more than just protection against moisture loss. Figures 13 and 14 show that the concrete under the plastic sheet was actually warmer than the concrete inside the unheated shelter, at least on sunny days. The sheet-covered concrete was 5 to 10°C warmer during the day on all days but 21 March, which was cloudy. On that day, the two concrete temperatures were nearly identical. During nighttime, the opposite occurred: the concrete inside the unheated shelter was up to 1.5°C warmer. These observations can be explained by the effect of the large volume of air within the shelter. The plastic sheet, having essentially no air to heat up and cool off, allowed the concrete to heat and cool faster than could the concrete inside the shelter. The six-day temperature of the concrete under the plastic sheet averaged 2.4°C (36.3°F) compared to only 0.9°C (33°F) for the concrete in the unheated shelter. A blanket of insulation would undoubtedly have performed even more effectively.

Of special interest in these tests was how the work would progress in cold weather. The workers at the Soo Locks stated that working outdoors was much preferred to working in a confining, though heated, enclosure. It was much easier to place and finish the concrete where there was freedom of movement. The consensus was that outdoor concreting was practical down to –20°C (–4°F), possibly lower, provided a heated shelter was available to warm up in periodically. At the Soo, the workers worked outdoors in windy –10°C (14°F) weather for two-hour intervals. The finishing operation required no special tools, skills, or precautions. The antifreeze concrete finished in the same manner as normal concrete. Ice did not build up on the cold metal tools as suspected.

Concreting in winter costs more than during the rest of the year. The extra costs in this test were 113 percent for the enclosure, and up to 43 percent for the admixture. Costs associated with antifreeze admixtures were more than offset by savings on protection requirements.

From a strength development standpoint, the antifreeze concrete was equal to or better than the concrete placed inside a heated enclosure. Dry heat can create problems. In fact, if the temperature of concrete is not closely regulated, high temperatures can cause significant strength loss.

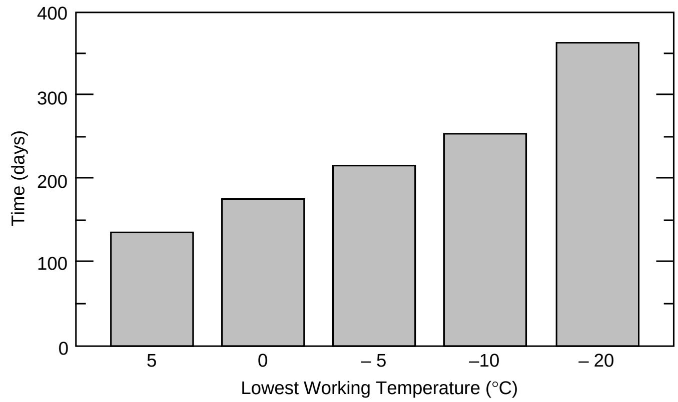


Figure 16. Possible extension of construction season with various low-temperature limits (Horrigan 1995, unpublished).

The potential effect on the length of the construction season of being able to place and keep concrete at –5°C (23°F) instead of at the current limit of 5°C (41°F) can be determined by looking at weather records. The number of days that the maximum air temperature in northern Michigan (at the Soo Locks) exceeded various low temperatures is shown in Figure 16. As can be seen, pushing the temperature envelope to –5°C (23°F) increases the length of the construction season by nearly 80 days. More working days become available at lower temperatures, to the point that concreting is a year-round proposition without the need for heat. The climate at the Soo is similar to that of the coldest areas in the contiguous United States.

CONCLUSIONS

The results from investigating Pozzutec 20 and developing a new prototype admixture indicate the following:

1. Pozzutec 20 accelerates and enhances the strength gain of concrete. When cured at room temperature, Pozzutec 20, used at its maximum permissible dosage of 60 mL/kg (90 fl oz/cwt), improved the seven-day strength of concrete by nearly 20%. A similar result was produced when the concrete was tested after 56 days of room-temperature curing.

2. Compared to the more-than 35 trial admixtures tested, Pozzutec 20 provided the fastest setting concrete. In one test conducted in mortar, Pozzutec 20 shortened the initial set time of concrete from 4 1/6 hours to 2 5/6 hours. The best

trial admixture produced a set time that was 20 min longer than that achieved with the Pozzutec 20 admixture. Other admixtures acted as set retarders, producing concrete set times in excess of those produced by admixture-free concrete. These comparisons were drawn from mortar cured at room temperature and made with 365 kg/m^3 (611 lb/yd^3) of Type I cement.

3. Pozzutec 20 did not contribute to the corrosion of reinforcing steel embedded in concrete submerged in sodium chloride solution. This was true for both the 60- and 100-mL/kg (90 and 150 fl oz/cwt) dosage.

4. At its maximum permissible dosage of 60 mL/kg (90 fl oz/cwt), Pozzutec 20 did not reduce the freeze–thaw durability of standard concrete beams tested according to ASTM C 666, Procedure A. At that dosage, the durability factor of concrete made with Pozzutec 20 following 300 cycles of freezing and thawing was 99 compared to control concrete, which was also 99. A durability factor of 80 is considered passing. At a dosage of 100 mL/kg (150 fl oz/cwt), the durability factor of the concrete dipped below 80 after 204 cycles of freezing and thawing.

5. Pozzutec 20 at a dosage of 60 mL/kg (90 fl oz/cwt) was determined to be equivalent to placing 50 mm (2 in.) of fibrous glass insulation over the concrete. This is the thickness of insulation that admixture-free concrete would require to remain above freezing for seven days at an air temperature very near freezing.

6. The critical freezing strength of concrete made with Pozzutec 20 is considered the same for admixture-free concrete. Pozzutec 20 does not adversely affect the strength at which concrete can first be frozen.

7. When used at its maximum permissible dosage of 60 mL/kg (90 fl oz/cwt), Pozzutec 20 was unable to promote strength in concrete cured at –5°C (23°F) at the same rate as that in admixture-free concrete cured at 5°C (41°F). This finding prompted the search for an improved low-temperature admixture.

8. The prototype admixture, code named EY-11, was selected as the potential improvement to Pozzutec 20 for use in freezing temperatures.

9. EY-11 at a dosage of 100 mL/kg (150 fl oz/cwt) was able to promote strength in concrete cured at –5°C (23°F) at the same rate as that developed in admixture-free concrete cured at 5°C (23°F). This is considered a major advantage over existing admixtures used by the concrete industry today.

10. At the 100-mL/kg (150 fl oz/cwt) dosage, the EY-11 admixture produced a concrete that easily passed the ASTM C 666, Procedure A, freeze–thaw test. The EY-11 concrete had a durability factor of 96 at the end of 300 cycles of freezing and thawing compared to a durability factor of 99 for admixture-free concrete.

11. At dosages of 60 and 100 mL/kg (90 and 150 fl oz/cwt), EY-11 was not found to contribute to corrosion of steel reinforcing embedded in concrete submerged in calcium chloride solution.

12. EY-11 was determined to be equivalent to 55.9 mm (2.2 in.) of insulation when the ambient temperature is as low as –1°C (30°F).

13. The negative aspect of the EY-11 admixture is that it did not promote short set times as effectively as did Pozzutec 20. The set time of EY-11 was approximately half an hour longer than that with Pozzutec 20. Also, the EY-11 admixture did not promote enhanced strengths to the same degree as did Pozzutec 20 when concrete was cured at room temperature. These are considered important productivity factors.

14. The field tests clearly demonstrated that working with EY-11 required no new skills. The concrete was easily mixed at low temperature, the admixture was dosed into the truck, as is normally done with some admixtures today, and the concrete was finished in the usual manner. The major benefit was that, once finished, the concrete was not damaged by exposure to freezing temperatures. The only protection used was a plastic sheet to cover exposed areas to minimize moisture loss during curing. Because external heat was not needed to protect the concrete, a tremendous amount of thermal energy was conserved. The resulting concrete quality was excellent.

15. The potential effect of being able to place concrete at temperatures below freezing is significant. Pushing the winter concreting envelope from the current 5–10°C limit to –5°C (23°F) can extend the "normal" construction season by over two months in northern Michigan, such as at the Soo Locks. Since the climate at the Soo is similar to that of the coldest areas in the conterminous United States, the construction season should be extendible by at least two months in the United States by using an admixture with the low-temperature capability of the experimental admixture EY-11.

RECOMMENDATIONS

A new low-temperature concreting technology was explored with the result that a prototype freezing-temperature-protection admixture has been developed. The resulting EY-11 prototype affords superior low-temperature protection compared to existing admixtures and provides good freeze–thaw durability at high dosages. These are important qualities. However, EY-11 needs further development to improve its ability to accelerate setting and enhance strength at above-freezing temperatures in order to fit into current ASTM (C 494) testing guidelines for concrete admixtures. It is believed necessary and in the best interests of Master Builders to develop an admixture that performs well at both above- and below-freezing temperatures. Consequently, MB has chosen not to market EY-11 until improvements can be made, particularly those of setting and early strength, at temperatures above freezing.

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APPENDIX A: PHASE I, TASK 1 STRENGTHS

Table A1. Compressive strength, MPa (psi), with Type I cement and a 365-kg/m^3 (611 lb/yd^3) cement factor.

	Age—days			
Mixture ID	*7*	*14*	*28*	*56*
2,0,20	30.2 (4385)	33.8 (4898)	33.9 (4916)	34.9 (5057)
2,1,20	31.9 (4621)	33.7 (4881)	36.3 (5258)	37.6 (5447)
2,2,20	35.3 (5116)	37.8 (5484)	39.9 (5788)	41.1 (5965)
2,3,20	35.4 (5140)	38.9 (5645)	41.6 (6036)	45.2 (6557)
2,0,5	25.1 (3636)	29.2 (4239)	33.2 (4810)	39.5 (5725)
2,1,5	25.1 (3640)	28.8 (4183)	33.6 (4869)	37.4 (5423)
2,2,5	27.5 (3985)	30.9 (4485)	36.9 (5352)	41.4 (6007)
2,3,5	28.6 (4154)	34.1 (4951)	40.8 (5918)	47.8 (6932)
2,0,–5	0.8 (123)	0.7 (98)	0.9 (125)	12.8 (1858)
2,1,–5	6.0 (869)	7.1 (1028)	8.5 (1230)	14.4 (2094)
2,2,–5	8.4 (1214)	10.2 (1481)	12.1 (1754)	20.8 (3018)
2,3,–5	8.4 (1211)	12.1 (1752)	16.2 (2349)	28.7 (4168)
2,0,–10	0.3 (38)	0.1 (8)	0.3 (46)	15.5 (2254)
2,1,–10	0.8 (113)	2.8 (402)	2.9 (423)	12.2 (1773)
2,2,–10	1.5 (217)	3.3 (478)	3.9 (562)	14.1 (2042)
2,3,–10	4.4 (645)	5.5 (799)	8.6 (1243)	23.6 (3418)
2,0,–20	0 (0)	0 (0)	0 (0)	18.9 (2735)
2,1,–20	0.1 (16)	0 (0)	0 (0)	14.2 (2066)
2,2,–20	0.3 (49)	0 (0)	0 (3)	14.9 (2160)
2,3,–20	1.1 (159)	1.1 (153)	0.5 (68)	22.9 (3320)

Table A2. Compressive strength, MPa (psi), with Type III cement and a 365-kg/m^3 (611 lb/yd^3) cement factor.

	Age—days			
Mixture ID	*7*	*14*	*28*	*56*
*2,0,20	36.9 (5352)	40.6 (5890)	42.6 (6172)	42.9 (6224)
*2,2,20	39.8 (5772)	43.7 (6338)	47.5 (6892)	46.9 (6802)
*2,0,5	33.4 (4847)	38.3 (5550)	42.8 (6201)	45.5 (6601)
*2,2,5	33.8 (4894)	39.5 (5725)	42.1 (6102)	47.2 (6849)
*2,0,–5	1.0 (146)	1.7 (241)	2.8 (404)	18.1 (2631)
*2,1,–5	10.0 (1451)	14.2 (2056)	19.3 (2796)	29.7 (4305)
*2,0,–10	0 (0)	0.5 (66)	0.5 (69)	19.0 (2749)
*2,1,–10	4.1 (590)	5.5 (797)	5.8 (842)	18.0 (2617)
*2,0,–20	0 (0)	0 (0)	0 (0)	23.3 (3376)
*2,1,–20	0 (0)	0.1 (21)	0 (0)	19.2 (2784)

* Denotes Type III cement.

Table A3. Compressive strength, MPa (psi), with Type I cement and a 420-kg/m^3 (705 lb/yd^3) cement factor.

	Age—days			
Mixture ID	*7*	*14*	*28*	*56*
3,0,20	30.9 (4480)	34.2 (4961)	37.6 (5451)	37.7 (5470)
3,1,20	32.9 (4767)	37.4 (5423)	40.9 (5932)	40.7 (5906)
3,2,20	37.3 (5404)	42.4 (6154)	43.6 (6328)	46.6 (6755)
3,3,20	36.6 (5314)	43.9 (6371)	46.8 (6790)	47.2 (6837)
3,0,5	28.5 (4126)	34.1 (4947)	38.2 (5536)	42.0 (6088)
3,1,5	28.1 (4074)	32.6 (4720)	38.2 (5541)	43.3 (6276)
3,2,5	31.3 (4536)	37.8 (5480)	40.8 (5923)	48.3 (7002)
3,3,5	31.0 (4494)	38.0 (5513)	44.1 (6399)	51.2 (7418)
3,0,–5	0.6 (85)	1.2 (167)	1.6 (237)	14.1 (2042)
3,1,–5	7.8 (1127)	11.0 (1601)	12.4 (1797)	19.8 (2874)
3,2,–5	9.9 (1432)	14.1 (2051)	17.0 (2471)	27.2 (3942)
3,3,–5	9.7 (1401)	17.9 (2598)	24.4 (3532)	40.2 (5823)
3,0,–10	0 (0)	0.4 (52)	0.3 (49)	15.2 (2202)
3,1,–10	2.3 (337)	3.7 (533)	4.6 (672)	13.1 (1905)
3,2,–10	3.7 (537)	5.5 (797)	6.9 (1002)	18.4 (2664)
3,3,–10	3.3 (472)	6.3 (915)	9.9 (1442)	26.8 (3890)
3,0,–20	0 (0)	0 (0)	0 (0)	16.8 (2438)
3,1,–20	0 (0)	0 (0)	0 (0)	15.4 (2240)
3,2,–20	0 (0)	0.4 (52)	0.4 (58)	16.8 (2443)
3,3,–20	0 (5)	1.7 (241)	2.8 (401)	24.5 (3556)

APPENDIX B: PHASE I, TASK 5 CRITICAL STRENGTHS

Table B1. Critical strength results of early age concrete frozen at –20°C (–4°F) overnight, then cured at 20°C (70°F). The control concrete was continuously cured at 20°C (70°F).

Mixture ID	*Age (days)*	*Control at 2°C (70°F)*	*Strength attained before freezing—MPa (psi)* 1.7 (250)	3.4 (500)	5.2 (750)
2,0	3	23.4 (3400)	21.9 (3178)	23.9 (3461)	24.7 (3584)
	7	27.4 (3973)	28.6 (4145)	27.1 (3926)	29.6 (4287)
	28	32.3 (4678)	33.6 (4867)	34.0 (4928)	32.8 (4756)
2,1	3	26.0 (3765)	23.9 (3468)	24.3 (3527)	25.0 (3624)
	7	32.2 (4673)	30.0 (4350)	29.1 (4225)	29.6 (4289)
	28	35.2 (5100)	34.1 (4951)	34.5 (5008)	36.2 (5246)
2,2	3	26.9 (3895)	27.2 (3947)	27.4 (3975)	26.4 (3834)
	7	33.8 (4907)	32.7 (4742)	33.4 (4836)	33.5 (4860)
	28	39.0 (5654)	36.8 (5336)	37.3 (5411)	38.5 (5588)
2,3	3	28.5 (4131)	28.7 (4164)	28.4 (4119)	28.9 (4192)
	7	34.5 (5001)	36.4 (5275)	35.4 (5135)	36.0 (5213)
	28	41.4 (6005)	41.2 (5997)	41.4 (6005)	40.6 (5890)
3,0	7	32.5 (4716)	31.8 (4610)	31.8 (4605)	32.4 (4704)
	14	36.9 (5348)	34.8 (5039)	36.5 (5293)	36.5 (5296)
	29	37.0 (5362)	35.7 (5178)	38.4 (5574)	39.1 (5668)
3,1	7	36.4 (5279)	33.6 (4867)	35.2 (5098)	35.1 (5086)
	14	37.6 (5447)	35.6 (5157)	37.5 (5435)	39.0 (5661)
	29	40.5 (5875)	40.0 (5805)	41.1 (5960)	41.8 (6059)
3,2	7	40.7 (5904)	39.2 (5691)	40.4 (5857)	40.4 (5857)
	14	43.2 (6260)	42.3 (6137)	43.3 (6281)	44.9 (6505)
	29	45.0 (6526)	46.7 (6767)	47.5 (6884)	47.8 (6932)
3,3	7	43.9 (6369)	42.5 (6159)	42.5 (6161)	43.3 (6279)
	14	45.8 (6644)	44.9 (6508)	46.8 (6779)	48.0 (6956)
	29	49.1 (7120)	48.9 (7087)	49.0 (7106)	49.7 (7210)
*2,0	3	29.3 (4251)	18.7 (2714)	30.1 (4367)	28.6 (4152)
	7	34.2 (4966)	33.6 (4874)	32.7 (4739)	36.3 (5270)
	28	39.1 (5668)	36.6 (5308)	39.1 (5675)	39.1 (5666)
*2,2	3	31.9 (4626)	27.7 (4015)	29.4 (4265)	30.7 (4449)
	7	38.6 (5590)	31.1 (4513)	33.9 (4911)	35.5 (5152)
	28	41.8 (6064)	37.1 (5378)	39.5 (5732)	40.7 (5906)

* Denotes Type III cement.

APPENDIX C: PHASE II, MORTAR SCREENING RESULTS

Table C1. Mortar mix results, 90 fl oz/cwt dosage.

			Compressive strength					
Admixture	*Initial set*	*Final set*	*1 day*	*Reference mix (%)*	*3 days*	*Reference mix (%)*	*28 days*	*Reference mix (%)*
Plain	4:10	7:20	364	100	520	100	3,201	100
ARL-506	3:25	8:00	259	71	1,134	218	6,251	195
ARL-507	3:15	8:05	144	40	276	53	5,231	163
Pozzutec 20	2:50	7:15	185	51	786	151	5,710	178
EX-1	3:30	8:55	246	68	1,075	207	7,089	221
EX-2	3:40	8:40	228	63	833	160	6,749	211
EX-3	3:55	8:10	216	59	1,018	196	5,840	182
EX-4	3:45	7:40	428	118	1,159	223	5,611	175
EX-5D	3:40	7:25	309	85	1,193	229	6,208	194
EX-6	3:55	8:15	200	55	568	109	6,616	207
EX-7	3:25	7:55	163	45	604	116	5,209	163

500 g cement
1375 g sand
212.7 mL water (reference mix: 242 mL)
29.3 mL (90 fl oz/cwt) admixture
Ambient mix room temperature @ 50°F (10°C)
Ambient curing temperature @ 35°F (2°C)

Table C1a. Mortar mix results, 240 fl oz/cwt dosage.

			Compressive strength					
Admixture	*Initial set*	*Final set*	*3 days*	*Reference mix (%)*	*7 days*	*Reference mix (%)*	*28 days*	*Reference mix (%)*
Plain	3:40	9:00	0	na	1,114	100	4,448	100
ARL-506	3:05	6:00	no samples					
ARL-507	7:10		0	na	943	85	2,055	46
Pozzutec 20	5:40	10:15	0	na	436	39	999	22
EX-1	3:00		1,680	na	3,684	331	5,054	114
EX-2	3:05	6:30	925	na	2,815	253	5,989	135
EX-3	2:20	8:00	1,326	na	4,051	364	7,428	167
EX-4	2:15	7:25	1,115	na	3,721	334	6,693	150
EX-5D	2:15	7:20	1,186	na	2,663	239	6,144	138
EX-6	2:05	10:45	0	na	1,473	132	4,285	96
EX-7	1:55	6:10	788	na	2,569	231	5,466	123

550 g cement
1513 g sand
180.1 mL water (reference mix: 266.2 mL)
86.1 mL (240 fl oz/cwt) admixture
Ambient mix room temperature @ 50°F (10°C)
Ambient curing temperature @ 35°F (2°C)

Table C2. Mortar mix results, 90 fl oz/cwt dosage.

Admixture	Initial set	Final set	Compressive strength: 1 day	Reference mix (%)	3 days	Reference mix (%)	28 days	Reference mix (%)
Plain	2:05	2:45	114	100	1,311	100	4,158	100
Pozzutec 20	2:26	2:48	236	208	1,444	110	4,726	114
EY-1	2:19	2:51	189	166	1,538	117	5,151	124
EY-3	2:40	5:20	153	134	1,471	112	4,995	120
EY-7	3:35	7:30	480	422	1,373	105	4,629	111
EY-8*	5:35		183	160	1,031	79	4,070	98
EY-10	2:10	6:45	240	211	823	63	3,835	92
EY-11*	3:30		550	484	1,243	95	4,389	106

* Denotes Type III cement.

Table C3. Mortar mix results, 90 and 150 fl oz/cwt dosage.

Admixture	Initial set	Final set	Compressive strength: 3 days	Reference mix (%)	7 days	Reference mix (%)	28 days	Reference mix (%)
Plain	3:10	7:40	70	100	760	100	3,525	100
Pozzutec 20 @ 90	2:50	6:20	185	264	1,090	143	3,268	93
EZ-1 @ 90	2:30	5:30	210	300	990	130	2,864	81
EZ-1 @ 150	3:05	5:25	225	321	1,375	181	3,960	112
EZ-2 @ 90	2:15	5:05	325	464	1,325	174	3,094	88
EZ-2 @ 150	2:10	4:45	300	429	1,975	260	4,805	136
EZ-3 @ 90	2:15	4:35	330	471	1,725	227	4,038	115
EZ-3 @ 150	2:05	4:30	165	236	1,585	209	4,725	134
EZ-4 @ 90	2:10	5:05	390	557	1,480	195	4,065	115
EZ-4 @ 150	1:55	4:50	200	286	1,185	156	4,368	124
EZ-7 @ 90	3:45	5:50	525	750	1,345	177	4,214	120
EZ-7 @ 150	3:30	6:10	170	243	1,305	172	4,450	126

APPENDIX D: PHASE II, CONCRETE TESTING RESULTS

Table D1. Mix data and plastic properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*
MB-VR (fl oz/cwt)	0.90	1.30	1.35	1.20	1.35	1.35
Pozzutec 20 (fl oz/cwt)	—	90.00	150.00	—	—	—
ARL-506 (fl oz/cwt)	—	—	—	90.00	150.00	—
ARL-507 (fl oz/cwt)	—	—	—	—	—	90.00
Cement (lb/yd)	612	612	608	619	613	609
Sand (lb/yd)	1,250	1,314	1,305	1,329	1,317	1,308
Stone (lb/yd)	1,801	1,800	1,788	1,820	1,804	1,791
Water (lb/yd)	258	244	243	247	244	243
w/c	0.422	0.399	0.400	0.399	0.398	0.399
Water reducer (%)	—	5.4	5.8	4.3	5.4	5.8
Air (%)	6.2	5.6	6.2	4.5	5.4	6.0
Slump (in.)	5.00	8.00	9.00	7.50	6.25	7.50

Table D1a. Hardened properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*
MB-VR (fl oz/cwt)	0.90	1.30	1.35	1.20	1.35	1.35
Pozzutec 20 (fl oz/cwt)	—	90.00	150.00	—	—	—
ARL-506 (fl oz/cwt)	—	—	—	90.00	150.00	—
ARL-507 (fl oz/cwt)	—	—	—	—	—	90.00
70°F Comp. strength						
1 day	2,320	3,270	2,850	3,240	3,060	2,490
7 days	3,780	4,900	5,200	5,620	5,230	4,720
28 days	4,710	6,550	6,600	6,500	6,250	5,930
14°F Comp. strength						
1 day	NA	190	120	380	250	50
7 days	NA	530	510	470	410	170
28 days	NA	910	1,170	920	1,060	420
70°F Set time (hr:min)						
Initial	3:56	3:41	3:34	2:48	2:30	4:03
Final	5:21	4:36	4:21	3:25	3:09	4:49
14°F Set time (hr:min)						
Initial	NA	9:02	9:10	7:29	7:31	9:27

Table D2. Mix data and plastic properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*
MB-VR (fl oz/cwt)	0.90	1.40	1.55	1.45	1.40	1.40
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—
ARL-507 (fl oz/cwt)	—	—	90.00	150.00	—	—
EX-4 (fl oz/cwt)	—	—	—	—	90.00	150.00
Cement (lb/yd)	615	614	614	605	611	607
Sand (lb/yd)	1,256	1,319	1,319	1,300	1,313	1,304
Stone (lb/yd)	1,812	1,807	1,807	1,781	1,798	1,787
Water (lb/yd)	278	248	248	234	254	246
w/c	0.453	0.404	0.404	0.387	0.417	0.406
Water reducer (%)	—	10.80	10.8	15.8	8.6	11.5
Air (%)	4.5	5.0	5.0	7.0	5.0	6.0
Slump (in.)	4.00	5.50	5.00	6.25	4.25	5.00

Table D2a. Hardened properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*
MB-VR (fl oz/cwt)	0.90	1.40	1.55	1.45	1.40	1.40
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—
ARL-507 (fl oz/cwt)	—	—	90.00	150.00	—	—
EX-4 (fl oz/cwt)	—	—	—	—	90.00	150.00
70°F Comp. strength						
1 day	2,500	3,040	2,580	2,430	2,590	1,910
7 days	4,430	6,360	5,630	5,530	4,820	4,310
28 days	5,520	7,400	6,810	6,490	5,840	5,460
14°F Comp. strength						
1 day	NA	160	120	100	270	150
7 days	NA	840	420	290	870	730
28 days	NA	1,940	1,830	2,280	1,590	1,700
70°F Set time (hr:min)						
Initial	4:07	3:28	3:44	3:44	3:05	2:25
Final	5:15	4:26	4:42	4:56	4:06	3:20
14°F Set time (hr:min)						
Initial	NA	9:02	10:00	9:36	9:03	9:20

Table D3. Mix data and plastic properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*
MB-VR (fl oz/cwt)	1.00	2.00	1.80	1.10	1.80	1.10	2.00	1.60
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—	—	—
EX-3 (fl oz/cwt)	—	—	90.00	150.00	—	—	—	—
EX-5D (fl oz/cwt)	—	—	—	—	90.00	150.00	—	—
EY-1 (fl oz/cwt)	—	—	—	—	—	—	90.00	150.00
Cement (lb/yd)	614	611	609	611	613	615	613	613
Sand (lb/yd)	1,252	1,313	1,309	1,312	1,316	1,320	1,316	1,316
Stone (lb/yd)	1,806	1,799	1,793	1,798	1,803	1,808	1,803	1,803
Water (lb/yd)	279	246	252	242	252	246	254	245
w/c	0.453	0.403	0.413	0.396	0.411	0.400	0.415	0.400
Water reducer (%)	—	11.8	9.7	13.3	9.7	11.8	9.0	12.2
Air (%)	4.8	5.5	5.4	5.8	5.0	5.1	4.8	5.4
Slump (in.)	3.75	5.75	4.50	4.75	4.50	5.00	3.75	5.25

Table D3a. Hardened properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*
MB-VR (fl oz/cwt)	1.00	2.00	1.80	1.10	1.80	1.10	2.00	1.60
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—	—	—
EX-3 (fl oz/cwt)	—	—	90.00	150.00	—	—	—	—
EX-5D (fl oz/cwt)	—	—	—	—	90.00	150.00	—	—
EY-1 (fl oz/cwt)	—	—	—	—	—	—	90.00	150.00
70°F Comp. strength								
1 day	2,460	2,940	2,600	2,390	2,620	1,940	1,970	1,660
7 days	4,330	5,260	3,890	4,450	3,990	4,460	3,610	3,370
28 days	5,060	6,950	4,850	5,380	5,490	5,820	4,530	4,590
14°F Comp. strength								
1 day	NA	210	270	240	300	270	120	70
7 days	NA	1,900	1,790	2,600	2,230	2,280	880	800
28 days	NA	3,700	2,830	3,700	2,630	2,880	1,130	1,460
70°F Set time (hr:min)								
Initial	4:12	3:43	3:14	2:43	3:12	3:06	4:41	5:57
Final	5:20	4:24	4:07	3:35	4:14	3:53	6:20	7:04
14°F Set time (hr:min)								
Initial	NA	7:53	7:41	7:41	8:00	10:58	11:40	13:41

Table D4. Mix data and plastic properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
MB-VR (fl oz/cwt)	0.85	1.30	1.80	2.00	1.10	0.75	1.60	2.35	0.80	0.30
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—	—	—	—	—
EY-3 (fl oz/cwt)	—	—	90.00	150.00	—	—	—	—	—	—
EY-7 (fl oz/cwt)	—	—	—	—	90.00	150.00	—	—	—	—
EY-10 (fl oz/cwt)	—	—	—	—	—	—	90.00	150.00	—	—
EY-11 (fl oz/cwt)	—	—	—	—	—	—	—	—	90.00	150.00
Cement (lb/yd)	611	617	602	603	604	604	601	613	612	620
Sand (lb/yd)	1,281	1,360	1,262	1,265	1,331	1,333	1,260	1,235	1,349	1,367
Stone (lb/yd)	1,748	1,815	1,770	1,776	1,776	1,778	1,769	1,805	1,800	1,824
Water (lb/yd)	261	223	279	283	237	225	293	319	239	236
w/c	0.427	0.361	0.463	0.469	0.392	0.373	0.488	0.520	0.391	0.381
Water reducer (%)	—	14.6	0.0	0.0	9.2	13.8	0.0	0.0	8.4	9.6
Air (%)	5.6	5.8	6.0	5.6	6.6	7.2	5.2	4.6	5.8	5.0
Slump (in.)	5.00	5.75	4.50	4.00	5.00	6.25	4.50	3.00	4.00	4.00

Table D4a. Hardened properties.

Mix #	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
MB-VR (fl oz/cwt)	0.85	1.30	1.80	2.00	1.10	0.75	1.60	2.35	0.80	0.30
Pozzutec 20 (fl oz/cwt)	—	90.00	—	—	—	—	—	—	—	—
EY-3 (fl oz/cwt)	—	—	90.00	150.00	—	—	—	—	—	—
EY-7 (fl oz/cwt)	—	—	—	—	90.00	150.00	—	—	—	—
EY-10 (fl oz/cwt)	—	—	—	—	—	—	90.00	150.00	—	—
EY-11 (fl oz/cwt)	—	—	—	—	—	—	—	—	90.00	150.00
70°F Comp. strength										
1 day	2,440	3,240	1,780	1,790	2,870	2,870	1,580	1,280	2,380	2,140
7 days	4,430	6,190	4,280	4,360	4,480	4,630	4,060	3,820	4,540	5,210
28 days	5,240	7,300	5,090	5,240	5,130	5,550	4,930	4,670	5,410	6,180
14°F Comp. strength										
1 day	NA	110	80	30	150	300	170	130	420	320
7 days	NA	1,620	1,580	260	890	1,680	1,260	40	170	470
28 days	NA	2,370	530	260	1,720	2,480	590	550	2,470	3,680
70°F Set time (hr:min)										
Initial	—	—	—	5:01	—	—	3:02	3:43	2:53	2:19
Final	2:40*	—	—	6:50	3:56	3:23	4:44	5:16	4:24	3:04
14°F Set time (hr:min)										
Initial	NA	8:53	8:00*	11:45	8:00*	7:30*	8:51	8:59	8:45*	8:00*

* Denotes estimated set times.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	October 1996	

4. TITLE AND SUBTITLE

Freezing Temperature Protection Admixture for Portland Cement Concrete

5. FUNDING NUMBERS

6. AUTHORS

Charles J. Korhonen and John W. Brook

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

U.S. Army Cold Regions Research and Engineering Laboratory
72 Lyme Road
Hanover, New Hampshire 03755-1290

8. PERFORMING ORGANIZATION REPORT NUMBER

Special Report 96-28

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Office of the Chief of Engineers
Washington, D.C. 20314-1000

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

For conversion of SI units to non-SI units of measurement consult ASTM Standard E380-93, *Standard Practice for Use of the International System of Units,* published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

Available from NTIS, Springfield, Virginia 22161

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

A number of experimental admixtures were compared to Pozzutec 20 admixture for their ability to protect fresh concrete from freezing and for increasing the rate of cement hydration at below-freezing temperatures. The commercial accelerator and low-temperature admixture Pozzutec 20 served as the reference admixture for this project as it has been a successful product of Master Builders for winter concreting during the past several years. Over thirty-five experimental admixture candidates were tested. Of these, one experimental admixture, code-named EY-11, a nonchloride admixture, outperformed all the others and was selected as the admixture to be considered for future commercialization. It was demonstrated by laboratory evaluation that the Pozzutec 20 admixture did not contribute to corrosion of embedded steel reinforcement. The EY-11 admixture, although still under examination, also did not contribute to corrosion in a newer and different laboratory test. Based on a knowledge of its constituents, EY-11 is not expected to contribute to corrosion under laboratory conditions or in the field. The low and medium dosages (60 and 100 mL/kg [90 and 150 fl oz/cwt]), of EY-11 produced freeze–thaw-durable concrete, but the highest dosage examined, 160 mL/kg (240 fl oz/cwt), did not. The middle dosage (100 mL/kg) protected concrete down to the low-temperature goal of this project, –5°C (23°F). The prototype admixture, EY-11, affords superior low-temperature protection compared to existing accelerating admixtures, as well as good durability. Unfortunately, it did not provide the desirable rapid setting and strength gain of concrete at above-freezing temperatures that field engineers and concrete technicians would like.

14. SUBJECT TERMS

Antifreeze admixture	Freeze–thaw	Winter construction
Cold-weather concrete	Thermal protection	

15. NUMBER OF PAGES 46

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102