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# A NEW CEMENT FOR COLD WEATHER CONSTRUCTION

by

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October 1976

**Final Report** 

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### 20. ABSTRACT (Continued)

A recently developed cement, called regulated-set cement, is capable of developing very high strengths at ages of 1 to 2 hours. This strength development is accompanied by a substantial release of internal heat during the hydration of the cement. The combination of these two characteristics provides a potential for the elimination of the elaborate and expensive curing procedures now used for cold weather concreting. This potential was examined both in the laboratory and field. The heat development in 3-, 6-, and 12-in.-thick slabs exposed at 15 F immediately after casting peaked in 1 to 2 hours at 46.5, 58, and 69 F, respectively, and remained above freezing long enough to gain considerable strength. Specimens protected 1 hour before exposure exhibited almost as much strength at 28 days age as specimens cured at 70  $\pm$  3 F the full time. Other factors such as concrete temperature at placing and construction procedures were also evaluated. The use of the regulated-set cement in cold weather situations should reduce the cost of the concrete construction and extend the construction season when cold weather sets in.

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PREFACE

This paper was prepared for the 1976 Army Science Conference held at the US Military Academy, West Point, New York, 22-25 June 1976. The information contained in the paper was developed as a part of the project, Military Construction and Maintenance in Cold Regions; work unit, Evaluation of Innovative Concepts for Structure and Materials in Cold Regions, undertaken for the Directorate of Military Construction, Office, Chief of Engineers, by the US Army Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire, and the US Army Engineer Waterways Experiment Station (WES), Vicksburg, Mississippi.

The work reported herein was conducted at WES and CRREL under the direction of Messrs. B. Mather, J. M. Scanlon, G. C. Hoff, and B. J. Houston of WES and Mr. Francis Sayles of CRREL. The paper was prepared by Mr. Hoff.

Directors of WES during the investigation and the preparation and publication of this report were COL G. H. Hilt, CE, and COL J. L. Cannon, CE. Technical Director was Mr. F. R. Brown.

# CONTENTS

	Page
PREFACE	2
CONVERSION FACTORS, US CUSTOMARY TO METRIC (SI) UNITS OF MEASUREMENT	4
COLD WEATHER CONCRETE CRITERIA	5
REGULATED-SET CEMENTS	7
OBJECTIVE AND SCOPE	10
EFFECT OF PLACEMENT TIME	11
EFFECT OF SPECIMEN THICKNESS ON HEAT DEVELOPMENT	
AND STRENGTH	12
FIELD EVALUATION	15
CONCLUSIONS	15
ACKNOWLEDGMENTS	16
REFERENCES	17

# CONVERSION FACTORS, US CUSTOMARY TO METRIC (SI) UNITS OF MEASUREMENT

US customary units of measurement used in this report can be converted to metric (SI) units as follows:

Multiply	By	To Obtain	
inches	25.4	millimetres	
pounds (mass)	0.4535924	kilograms	
pounds (mass) per cubic yard	0.5933	kilograms per cubic metre	
pounds (force) per square inch	0.006894757	megapascals	
calories/gram	4184	joule per kilogram	
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*	

\* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: C = (5/9)(F-32). To obtain Kelvin (K) readings, use K = (5/9)(F-32).

# A NEW CEMENT FOR COLD WEATHER CONSTRUCTION

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#### ·COLD·WEATHER CONCRETE CRITERIA

Some of the principal problems associated with winter concreting are the protection of the freshly mixed concrete from freezing and the provision of a curing environment conducive to the development of sufficient strength in the concrete so that subsequent freezing and thawing will not damage the concrete. If the concrete is kept sufficiently warm (>32 F) so that it does not freeze when made and placed, it will continue to develop strength with the rate of strength gain depending on the temperature at which it is cured.

Standard cold weather concrete protection consists of preventing loss of heat from concrete placed at a safe initial temperature and supplying any additional heat necessary for continued curing and strength development. If fresh concrete is frozen while there is considerable free water in the mixture and the paste has not attained sufficient strength to resist the expansive forces associated with an increase in volume of the water as it goes from its liquid to solid phase, the internal structure of the concrete can be disrupted with corresponding decreases in strength. The 28-day compressive strength may be reduced to 30 to 50 percent of strength of concrete which had not been frozen. (1)

How long should the concrete be protected from freezing? The duration of protection required to develop strength will vary with conditions under which the concrete is made and placed and also the degree of structural safety that is required. The degree of saturation of freshly placed concrete which has no access to an

external source of water will be reduced as the concrete hardens and water is used in the hydration process. Under such conditions, the time at which the degree of saturation becomes reduced below that level which would cause damage by freezing has been related to both "critical age" and "critical strength." Minimum strengths which should be developed before freezing is allowed have been reported as varying from 350 to 640 pounds per square inch (psi) in Sweden, (2) 1000 to 1500 psi in Canada, (2) 700 to 1280 psi in the Soviet Union, (2) 500 psi in the United States, (3) 850 psi in Japan (by Takahashi and Hayashi), (4) to 2100 psi in Switzerland. (2) Minimum ages have varied from 8 to 48 hours. It has been suggested that the "critical age" concept is more valid than "critical strength." (5) The absolute strength of concrete at the moment of freezing is not as important as the extent of cement hydration that has occurred and the amount of freezable water remaining in the concrete. Strength determinations are simpler to make, however, and the "critical strength" concept is widely used. Most well-proportioned concretes reach 500 psi during the second day when cured at 50 F. Nonairentrained concrete made with Type I cement at a water-cement ratio of 0.57 has been shown to withstand one cycle of freezing after about 18 hours age. (1)

What the above points out is that in cold weather concreting situations, the concrete materials must be of such temperatures that when properly proportioned the resulting concrete will have a temperature sufficiently great enough for the hydration of the cement to begin. Once it starts, it must be sustained for periods long enough for the concrete to develop sufficient strength (or degree of hydration) so that freezing will not damage the concrete. The usual methods to accomplish this can be summarized as follows: (2,4\*)

a. Treatment of the materials.

- (1) Heating of the materials.
  - (2) Heating of the freshly mixed concrete (usually electrically).
  - (3) Use of cement with high heat development.
  - (4) Use of chemical admixtures which either accelerate the reaction of the cement or depress the freezing point of the water in the concrete.
- b. Methods to prevent heat losses.
  - (1) Insulating covering.
  - (2) Heating.

\* Reference 4, Voellmy, A., "High Concrete Quality in Cold Weather."

Considering the economics of winter concreting, the least expensive method would be the one that required the least manpower, time, and equipment. Of the above methods, (a.3) and (a.4) would come the closest to satisfying these criteria. Even the use of Type III high early strength cement, or rapid hardening alumina cement, or an accelerating admixture, calcium chloride (CaCl<sub>2</sub>), does not eliminate the requirement for protection after placing although the times of required protection are greatly reduced. Ideally, a cement which gains strength very rapidly as well as producing quantities of heat sufficient to sustain the development of heat while protecting the concrete from freezing would reduce the cost of winter concreting and possibly extend the winter concreting season for a few more months. The experimental work contained in this report is an examination of a cement, called regulated-set cement, which appears to have great promise in satisfying these criteria.

## REGULATED-SET CEMENTS

Regulated-set cements are manufactured under patents issued to the Portland Cement Association (PCA). (6) Patents exist in Australia, Canada, France, Germany, Great Britain, and South Africa, with patents pending in Austria, Indonesia, Italy, Korea, Mexico, Pakistan, Philippines, Spain, Taiwan, and Switzerland. The PCA patent and a Thailand PCA patent have been sold to the Japanese who market regulated-set cement under the name of "Jet-Set" cement. Regulated-set cement is presently being produced in Austria, Germany, Japan, and the USA.

Regulated-set cements, produced under the PCA patents, can be made to contain 1 to 30 percent by weight of a calcium haloaluminate having the formula  $11CaO\cdot7Al_2O_3\cdotCaX_2$ , in which X is a halogen, or the cement can be a blended cement produced by grinding together portland cement clinker and a clinker containing a calcium haloaluminate. The halogen may be fluorine, chlorine, iodine, or bromine, but fluorine is preferred. Ordinarily the sulfate content of these cements will be 1 to 12 percent SO<sub>3</sub> as calcium sulfate. USAproduced regulated-set cements average approximately 6 percent SO<sub>3</sub>, while Japanese productions average approximately 11 percent with German production being somewhere in between. Some typical chemical analyses for these cements are as follows:

Japanese				German	
	Cemen	t (7)	USA C	USA Cement	
<u>Constituents</u>	<u>A</u>	<u>B</u>	<u>A</u>	В	A
Si0	13.7	13.9	17.1	15.0	12.3
·Alooa	· 10.8	11.0	10.4	10.2	11.4
$Fe_2^{0}$	1.7	1.7	0.9	2.1	2.3
CaO	58.6	58.6	60.9	58.1	57.3
MgO	0.7	0.8	1.0	1.9 ,	0.9
SO3	11.0	11.1	6.0	6.2	8.5
Ignition loss	1.0	0.2	2.7	4.2	5.2
Total	97.5	97.3	99.0	97.7	97.9
Insoluble residue	NG*	NG*	• 0.3	1.11	1.10
Na <sub>2</sub> 0 -	- 0.6	0.6	0.64	0.44	0.07
ĸ	0.4	0.4	0.16	0.98	1.33
Total alkalies as Na <sub>2</sub> 0	0.86	0.86	0.80	1.08	0.95
F	0.9	1.0	NG*	0.9	1.0

NG = not given.

In comparison to ordinary portland cement, regulated-set cement contains a reduced portion of dicalcium silicate and no tricalcium aluminate. The tricalcium aluminate has been replaced by a new ingredient called calcium fluoroaluminate. Like tricalcium aluminate, the fluoroaluminate hydration imparts considerable strength to a paste or mortar immediately after it sets and also requires a retarder to control setting time. Both citric acid and calcium sulfate hemihydrate have been used as set retarders, (7) but citric acid is most commonly used. The level of strength developed at early ages is somewhat proportional to the amount of fluoroaluminate contained in the cement. Compressive strengths of mortars at one hour age can exceed 1000 psi when the calcium fluoroaluminate content is 12 to 13 percent. Approximately 20 percent calcium fluoroaluminate is present in most USA regulated-set cements.

Associated with the development of very high early strength is the liberation of large quantities of heat. This heat is a byproduct of the reaction of the cement and water and is described by an index called the heat of hydration. The heat of

hydration is expressed as the total heat liberated per gram of cement up to any specified age. After 28 days curing, a typical Type I portland cement will have produced 90-100 cal/g whereas a regulated-set cement will have typically produced 140-150 cal/g with 60-70 cal/g of this being produced in the first hour of curing For most portland cements, the temperature at which hydration takes place affects the rate of heat development. (10) The effects of curing temperature on the rate of hydration of regulated-set cement are not known to the author. Other factors, such as cement fineness and water content, also influence the amount and rate at which heat is liberated.

Initial and final set occur very soon after mixing for regulated-set mixtures. The term "handling time" is defined as the maximum time before the concrete must be in its final position. At 70 F, regulated-set concrete has a controlled handling time which can be varied between 2 and 45 minutes. This regulation can be achieved when the cement is manufactured by blending different proportions of the early strength component and a set-control component, and also in the field by the use of retarding additives or by changing the temperature of the mixture. Citric acid has been found to be a most effective retarder for use in the field. Additions of 0.1 percent citric acid by weight of the cement can extend handling time at 70 F by 25 to 30 minutes.

At 70 F and without additives, regulated-set concrete will develop a compressive strength of 1000 psi or more as early as 1-1/2 hours after mixing. This early strength is directly proportional to the percentage of calcium fluoroaluminate in the cement. An increased percentage of the high early strength component causes a higher early strength and a shorter handling time. After this rapid early strength gain, the strength development of the calcium fluoroaluminate is nearly complete, and little or no strength gain occurs until the normal silicate hydration becomes effective after about one day. The long-term strength, the rate of gain of strength after one day, and other physical properties are then comparable to those of concrete made with Type I and Type III cements. The various cement companies which have manufactured regulated-set cement have formulated their cement not to any national standard but to their own specifications. Thus, the behavior and properties of regulated-set cement vary according to its source.

The relationship of strength and durability to watercement ratio and air content and the response to additives are similar to portland cement; however, regulated-set cement is considerably • more sensitive to variations in these parameters. With regard to handling and setting time, regulated-set cement is particularly sensitive to the temperature of the mixture and to certain retarders such as citric acid. Handling time increases with: lower temperature, addition of citric acid, higher water-cement ratio, lower cement content, and continued mixing. The sulfate resistance of regulated-set cement is reported to be about the same as any portland cement with a high alumina content.

### OBJECTIVE AND SCOPE

The objective of this study is to evaluate the feasibility of using regulated-set cement as a binder in concrete when the concrete is placed at low temperatures. The regulated-set concrete should be able to be placed in the field at temperatures as low as 15 F, and require a minimum of attention after placed.

A typical concrete was selected for evaluation. The concrete mixture used contained regulated-set cement (specific gravity = 3.02), 3/4-in. maximum-size limestone coarse aggregate, and limestone sand. Saturated-surface-dry batch weights were as follows:

Material	Saturated-Surface-Dry Batch Weights, 1b		
Cement	152.5		
Fine aggregate	385.9		
Coarse aggregate	565.7		
Water	80.8		

The mixture contained an actual cement factor of 500 lb/cu yd, a water-cement ratio of 0.53, entrained air content of 8.5 percent, and a slump of 2-1/2 in. All materials and molds were stored at 35 F until immediately prior to mixing. Mixing and molding were done at  $73 \pm 2$  F. The cement was introduced into the mixer as the last step in the charging process, and the concrete mixed for 1-1/2 minutes.

The concrete was evaluated to determine: (a) the effect of placement time after mixing on strength development when the concrete was placed at 15 F with no additional heat curing, and (b) the effect of specimen thickness on heat and strength development. Field construction of two large test slabs at 15 F was also attempted.

#### EFFECT OF PLACEMENT TIME

Ten 6- by 6- by 36-in.-long concrete beams were cast from the above mixture. The beams were later sawed into five 6- by 6-in. cubes. The beams were exposed as follows:

- a. Two beams (10 cubes) were exposed at 70 ± 3 F and 90 to 100 percent humidity immediately after casting.
- b. Two beams (10 cubes) were exposed at 15 F immediately after casting.
- c. Two beams (10 cubes) were exposed at 15 F one hour after casting. The beams were covered with damp burlap until placed at 15 F.
- d. Two beams (10 cubes) were exposed at 15 F three hours after casting. The beams were covered as in c above until exposed.
- e. Two beams (10 cubes) were exposed at 15 F 24 hours after casting. The beams were placed at room temperature and 90 to 100 percent humidity until exposed at 15 F.

Two cubes from each of the five exposures were tested in compression at 3, 7, and 28 days age. Frozen cubes were thawed for six hours at room temperature prior to breaking.

The compressive strength test results are shown in Figure 1 and indicate that concrete containing regulated-set cement exhibits the following properties:





- a. Concrete specimens mixed at 35 to 40 F and placed at 15 F immediately after casting had a three-day strength of slightly less than 1000 psi and continued to gain strength while exposed at 15 F.
- b. The longer the mixture remains above freezing before exposure to 15 F the greater the subsequent 28-day strength.
- c. Specimens exposed at  $70 \pm 3$  F until 24 hours old then exposed at 15 F exhibited almost as much strength after seven days age as companion specimens'exposed at  $70 \pm 3$  F the full time.
- d. The 28-day strengths of all specimens kept at room temperature 1, 3, and 24 hours before exposure at 15 F were almost as great as those specimens exposed at 70 + 3 F the full time.
- e. Specimens cured at 15 F with slight delays before freezing gained strength slower than control specimens, but at 28 days age were practically as strong as the specimens exposed at room temperature the full time. Specimens exposed to 15 F immediately after casting were only 60 percent of the control strength at 28 days age.

## EFFECT OF SPECIMEN THICKNESS ON HEAT DEVELOPMENT AND STRENGTH

To investigate the temperature buildup in the concrete caused by hydration of the cement and the transfer of heat to the subgrade, four test slabs were cast. The slabs were 20 by 20 in. square, with one being 3 in. thick, two 6 in. thick, and one 12 in. thick. Four inches of sand were compacted in the bottom of each mold to represent the subgrade except for one of the 6-in. slabs which had no sand base. The molds and sand were brought to 15 F prior to placing concrete in them. Thermocouples for monitoring temperature were placed in the center of each concrete, slab and in the horizontal center and 1 in. deep in the sand.

The storage and testing procedure was as follows:

a. One 20- by 20- by 6-in. test slab was placed at  $70 \pm 3$  F and 90 to 100 percent humidity immediately after casting. This mold did not have a sand base.

- b. All other slabs were placed at 15 F immediately after casting.
- c. The temperature in the concrete and the sand beneath were recorded until ambient was reached for all slabs exposed at 15 F but only in the concrete for the slab exposed at 70  $\pm$  3 F.
- d. At seven days age, the molds were stripped and the slabs sawed into cubes. The 12-in.-thick slab was sawed horizontally into two 6-in.-thick slabs, which were in turn sawed into 6- by 6- by 6-in. cubes. Cubes were broken in compression at 8 and 28 days age.

Results of the temperature study are shown in Figure 2. Strength results of cubes sawed from the slabs are shown in Table 1.

The results of the temperature study are as follows:

- a. The temperature in a 6-in. slab, with all materials at 35 F prior to mixing and exposure temperature of 70  $\pm$  3 F, rose to approximately 70 F in 12 hours and peaked at 79.5 F in 36 hours. Ambient was reached in approximately 72 hours.
- b. The temperature in the 6-in. slab exposed at 15 F immediately after mixing peaked at 58 F at 1 hour 20 minutes. Ambient temperature was reached in





Slab			Compr	essive
Thickness		Specimen	Strength, psi	
<u>in.</u>	Storage Condition	No.	8 Days	28 Days
3	15 F immediately after casting	1 2 3	950 1550 1790	2100 1620 2460
		Average	1430 .	2060
6	15 F immediately after casting	1 2	2550 2470	2520 2850
· •		Average	2510	2680
12	15 F immediately after casting	1T* 2T*	3940 2950	4290 3390
		Average	3440	3840
12	15 F immediately after casting	1B** 2B**	3570 4060	5040 4630
- -		Average	3810	4840
6	70 <u>+</u> 3 F and 90- 100 percent humidity	1 2	3040 3760	4170 5280
		Average	3400	4730

## Compressive Strength of Cubes Sawed from Temperature Study Concrete Slabs

Table 1

Sawed from top 6 in. of slab. ×

\*\* Sawed from bottom 6 in. of slab.

> approximately 38 hours. The temperature in the sand base peaked at 52 F at 2 hours 20 minute's and reached the slab temperature at 6 hours.

The temperature in a 12-in.-thick slab exposed at 15 F c. peaked at 69 F at 1 hour 40 minutes and reached ambient in about 84 hours. The temperature in the sand base underneath peaked at 57 F at 5 hours and reached the temperature of the slab at approximately 15 hours.

d. The temperature in the 3-in. slab peaked at 46.5 F at 1 hour 20 minutes and reached ambient at about 36 hours. The temperature in the sand base peaked at 43.5 F at 2 hours 20 minutes and also reached the temperature of the slab at that time.

Compressive strength cubes sawed from the above slabs were tested in compression at 8 and 28 days age (Table 1). Compressive strengths were greater in the thicker slabs. The strengths of specimens from the bottom 6 in. of the 12-in. slab exposed at 15 F were greater at 8 and 28 days than specimens from the 6-in. slab exposed at  $70 \pm 3$  F; however, strengths of specimens from the top 6 in. of the 12-in. slab were slightly lower than the  $70 \pm 3$  F specimens at 28 days age.

## FIELD EVALUATION

Two concrete test slabs, 12 ft square by 8 in. thick were constructed with the regulated-set cement in New Hampshire in January, 1975 when the mean ambient temperature was 15 F. Both construction procedures and materials behavior were evaluated in this exercise. The only major difference in the two slabs was the tenperature of the concrete at the time of placing. Slab 1 had a concrete temperature of 33 F while Slab 2 had a concrete temperature of 50 F. The higher temperature in slab 2 was accomplished by heating the water prior to mixing. No extraneous heat curing was provided to either slab. The setting time of the concrete in the test slabs was slower than the laboratory work indicated it would be. Neither slab obtained any appreciable compressive strength at 1 day but slab 1 strengths were approximately 1200 and 2000 psi at 7 and 28 days respectively, while slab 2 strengths were 2200 and 3300 psi respectively. The early indications are that the slab placements were successful and that the use of regulated-set cement accomplished the objectives for no-cure cold weather concreting. Evaluations of the slabs are continuing.

## CONCLUSIONS

The results of the investigation reported herein indicate that concrete containing regulated-set cement will gain strength even when unprotected when placed at temperatures as low as 15 F. This is accomplished because the chemical reaction between the cement and water is accelerated and hydration heat is generated almost immediately after mixing of the cement and water. This heat generation sustains temperatures within the mixture above freezing long enough for considerable strength buildup. The significance of this is that with the use of regulated-set cement the implementation of normal protection measures for concrete placed in cold weather can be delayed both at the start and finish of the winter season and that the period of initial concrete protection can be reduced during any winter concrete placements. In all these instances, this should result in substantial cost savings and reductions in energy requirements normally used in concrete heating.

The following factors affect strength development of concrete containing regulated-set cement when freshly mixed and exposed unprotected at 15 F.

- a. The age of the concrete when placed at 15 F. The longer the mixture remains above freezing before exposure, the greater the subsequent strength gain. The mixtures tested, however, will gain considerable strength even when exposed to 15 F immediately after mixing, and when protected as long as an hour after mixing before exposing at 15 F will exhibit almost as much strength at 28 days age as specimens exposed at 70 ± 3 F the full time.
- b. <u>Thickness of section.</u> The heat development in 3-, 6-, and 12-in.-thick slabs exposed at 15 F immediately
  after casting rose to 46.5, 58, and 69 F, respectively, in one to two hours, then dropped off, indicating enough internal heat was generated to sustain hydration. A 12-in.-thick slab exposed at 15 F will obtain almost as much strength as a slab exposed at 70 + 3 F the full time.

#### ACKNOWLEDGMENTS

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