RI 9124

Bureau of Mines Report of Investigations/1987

Development of Rapid-Setting Inorganic Grouts

By J. E. Fraley and J. E. Bevan

UNITED STATES DEPARTMENT OF THE INTERIOR

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Donald Paul Hodel, Secretary

BUREAU OF MINES
David S. Brown, Acting Director

Library of Congress Cataloging in Publication Data:

Fraley, J. E., 1947-
Development of rapid-setting inorganic grouts.

(Report of investigations/Bureau of Mines; 9124)

Supt. of Docs. no.: I 28.23: 9124.

1. Mine roof bolting. 2. Grouting. I. Bevan, J. E. (John E.). II. Title. III. Series: Report of investigations (United States. Bureau of Mines); 9124.

TN23.U43 [TN289.3] 622 s [622'.28] 87-600144

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°F	degree Fahrenheit	μm	micrometer
ft	foot	min	minute
g	gram	pct	percent
h	hour	rpm	revolution per minute
in	inch	s	second
lb	pound	st	short ton

DEVELOPMENT OF RAPID-SETTING INORGANIC GROUTS

By J. E. Fraley[1] and J. E. Bevan[2]

ABSTRACT

This report describes the preliminary results of a Bureau program to develop and test an inexpensive grout for full-column support of roof bolts. Various inorganic grouts were tested in both cartridge and slurry form. Tests were also performed on methods of water packaging and on the effect of fillers on the grout strength. Results showed that some grouts had poor early strength and exceptional mid- to long-term strength.

Laboratory tests have shown that rapid-setting inorganic grout could be an economical replacement for resin bolts if the short-term strength could be increased. Further development to improve its early strength would need to be tested in the laboratory and underground to prove its potential for the mining industry.

[1]Chemical engineer.
[2]Mechanical engineer.
Spokane Research Center, Bureau of Mines, Spokane, WA.

INTRODUCTION

In most U.S. coal mines, and in many metal mines, rockbolts are the primary means of roof support. The majority of roof bolts can be divided into two major categories: (1) those using mechanical anchorage and (2) those using grouted anchorage. Mechanically anchored bolts use a slot, wedge, or an expansion shell that physically locks the anchor to the rock as the bolt is tensioned. Grouted bolts use a grouting medium to attach the bolt to the rock. The upper end of the bolt may be grouted (point anchorage), or the entire length may be grouted (full-column anchorage).

Grouted bolts are made of reinforcing steel (rebar) and usually have a bolthead forged at one end. Cement has been used to anchor the rebar, but it has a slow set time. To overcome the slow set time, resins (usually polyester, with fillers) have been developed for use as a grouting material. The resins and fillers are blended to achieve an acceptable compromise between performance and cost. Because the resin is a petroleum derivative, it will be sensitive to future oil price increases. Also, there are potential hazards from toxicity during handling and from accidental ignition of the resin. To counter these problems, the Bureau began investigating fast-setting inorganic grouts as replacements for resins.[3]

The need for roof support to protect miners and equipment has motivated Bureau work on automated ground support placement. Examples of Bureau-developed automated ground support include the longer-than-seam-height (LTSH) drill, the roof bolt inserter (RBI), and a slurry injection device.

The LTSH drill drills a hole from a short seam height or a limited space, often the case in coal mines. The RBI takes a roof bolt from a horizontal orientation, bends it into a vertical orientation, and drives the bolt into a predrilled bolthole. The slurry injection device measures the proper amount of bulk inorganic cement, mixes it with the desired amount of water, and injects the paste into the hole via a pneumatic injection tube. Combination of the LTSH drill, the RBI, and the slurry injection device on a roof bolter will provide safe, automated placement of roof support.

Originally, the slurry injection device was designed to use gypsum as a grouting medium. However, because of the limitations of gypsum, such as strength and water solubility, research continues on other injectable inorganic grouts. This report provides preliminary results of this research.

FACTORS AFFECTING BOLT PERFORMANCE

PHYSICAL CHARACTERISTICS OF GROUTED BOLTS

Fully grouted bolts react differently under load than point-anchored bolts. When a mechanical bolt is loaded, the entire bolt is elongated and the load is distributed along the bolt. With a full-column grouted bolt, the load is transferred through the grout and into the bolt. The load is distributed over a shorter length (than in a mechanical bolt), so the total elongation is localized and usually results in less roof movement.

Usually, the shorter the anchorage length needed to make the bolt yield, the better the grout. If this takes 0.5 ft of bond as opposed to 2 ft, the ground support is better (unless a yielding support is desired). New inorganic grouts with rapid set times are being tested to obtain yielding of the bolt with shorter anchorage lengths. If the cartridged grout ingredients are mixed "in the hole," the mixability (the ease with which they combine with water) of the

[3]Simpson, R. E., J. E. Fraley, and D. J. Cox. Inorganic Cement for Mine Roof-Bolt Grouting. BuMines RI 8494, 1980, 32 pp.

ingredients is very important. The energy to break the cartridge and mix the ingredients is supplied by the bolt thrust and bolt rotation. If this energy is insufficient or is not imparted uniformly, a nonhomogeneous product with poor bonding characteristics results. Two of the parameters that affect the mixability are the fluidity of the mixed material and the annular distance between the bolt and hole wall.

GROUT STRENGTH

Effective ground control can require rapid placement of support and high early strength for a grouted material. As the grout hardens, it passes through several stages. These stages are defined in table 1 and illustrated in figure 1, which plots hardness versus time. The method of grouting, cartridge installation, or slurry injection can have a major effect on the rate of strength gain. The ambient temperature, the work energy imparted to the grout during mixing and injection, and the moisture content of the surrounding medium can also have an effect on the grout set time.

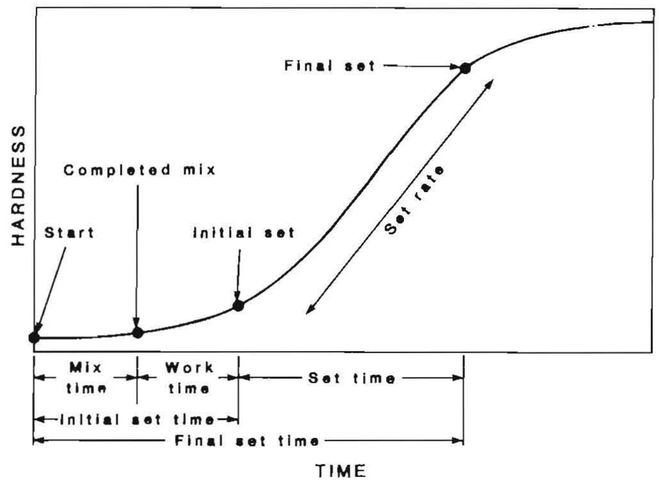

FIGURE 1.—Stages of inorganic grout hardening.

VOLUMETRIC CONSIDERATIONS

To fully grout a bolt, sufficient quantity of material is needed to fill the annulus between the bolt and the hole wall. The annular volume is the empty

TABLE 1. - Definitions relating to inorganic grout hardening

Plaster	$CaSO_4 \cdot 1/2H_2O$, which forms gypsum ($CaSO_4 \cdot 2H_2O$) when combined with water.
Grout (as a noun)	A mixture of cements, fillers, and water used to fill the annulus between the bolthole wall and bolt.
Grout (as a verb)	To fill the annulus between the bolthole wall and bolt with grout.
Completed mix point	The point where all the ingredients are thoroughly mixed.
Mix time	The time required to reach the completed mix point.
Initial set point	The point where the grout is physically or visually perceived to be stiffening from its original liquid state.
Work time	The time between the completed mix point and initial set point.
Initial set time	The time from the beginning of mixing to the initial set point.
Final set point	The point where little increase in hardness can be perceived with the penetrometer test. This is somewhat subjective as the grout continues to harden and gain strength.
Final set time	The time from the beginning of mixing to the final set point.
Set rate	The increase in hardness as time passes between the initial set point and final set point.
Set time	The time from the initial to final set points.
Short-term	Less than 15 min.
Mid-term	15 to 30 min.
Long-term	More than 30 min.

hole volume minus the bolt volume. When the grout is mixed outside the hole, filling the annular volume is easy; but when the grout is mixed in the hole, as with cartridge insertion, volumetric shortages can occur.

In a cartridge installation system, the maximum premixed volume can only be the volume of the hole (actually slightly less to allow insertion). The cement and filler particle densities can vary because of voids between the particles. These voids are influenced by particle size, shape, and the compaction of the materials. When the dry materials and water are mixed, the aftermixed volume can be less than the premixed volume.

Definitions of the pertinent volumes are given in table 2. If the aftermixed volume of the cartridge is less than the annular volume, the hole does not get completely filled. Compacting the dry powder in the cartridge can help, but may not be enough. Fillers such as sand and larger aggregate can be added to reduce the voids between particles. These additives have also shown to be helpful in improving the mixing.

To provide good anchorage, the grouting material should not shrink during curing. If shrinkage does occur, the grouting material can pull away from the bonding surfaces.

ECONOMICS

The total cost of ground support can be determined in many different ways. The final cost is influenced by material costs, packaging, handling, storage, capital costs, labor, long-term integrity of the support, etc. The authors make no attempt to model these costs. This report only discusses the cost of different types of grouts.

WATER SOLUBILITY AND PERMEABILITY

The integrity of the bolt bond requires a grouting material that does not have a high water solubility and that has low water permeability. A mine can have roof conditions that vary from dry to extremely wet. A soluble material could be washed away, thereby destroying the bond integrity. Grout permeability can allow moisture to reach a steel bolt and cause it to corrode.

WATER PACKAGING

A specified volume of water contains a specific mass. Because of its variable density, dry powder does not have this relationship, and it is more difficult to use a volumetric procedure to obtain a specified mass of powder.

In the operation of the bulk injection device, the water demand is easily met; but in making cartridges containing dry powder and water, it is difficult to find enough space to package a sufficient quantity of water. The cartridge water must not contact the powder before mixing, yet during mixing it must be distributed through the powder so that it can be well mixed by the mechanical energy from the bolt.

It can be difficult to package sufficient grouting material into a cartridge because of volume differences between dry and wet grout. For adequate mixability, a cartridge or a slurry mixture must contain more water than is chemically required. This surplus or free water

TABLE 2. - Definitions relating to inorganic grout cartridge volumetric considerations

Aftermixed volume (cartridge)....	The volume of the cement, fillers, and water after they are thoroughly mixed.
Annular volume..................	The empty hole volume minus the bolt volume.
Cured volume (cartridge).........	The volume of the cured or hardened grout measured at the final set point.
Hole volume.....................	The volume of the empty hole.
Premixed volume (cartridge)......	The volume of the cement particles, filler particles, air voids, and water.

increases the water packaging problem and leaves voids in the cured cement.

In order to include water with dry powder in the cartridge, the water must be packaged; water capsules, glass water tubes, plastic water tubes, and segmented plastic water tubes have all been tested. The water capsule system was developed by the Bureau and is reported in detail in a Report of Investigations (RI 9067). It uses water that has been encapsulated in wax balls approximately 1,700 µm in diameter. To make the cartridges, the wax balls are mixed with dry gypsum plaster. When a bolt is inserted through a cartridge, the water capsules break and release the water, which is then mixed with the gypsum plaster by the bolt.

The water capsules are the most expensive component of the cartridge. Current estimated costs of the water capsules are too expensive to be competitive. In order to find a substitute for the water capsules and to reduce costs, glass and plastic water tubes were developed and tested. Both released the water too rapidly during bolt installation, which resulted in water loss from the hole and insufficient water for proper mixing. A segmented water tube was developed to release its water sequentially, resulting in less water loss and better mixing. This system was tested and the results are covered further in this report.

FLY ASH FILLER

Portland[4] cement sells from $60/st to $85/st and specialized quick setting cements cost more. Because the cost of a filler such as fly ash is much lower, its use can result in a substantial cost savings.

The addition of byproduct mineral fillers such as fly ash adds a pozzolanic reaction. A pozzolan is a siliceous or siliceous and aluminous material having little or no cementitious value, which will, if finely divided and subjected to moisture, chemically react with calcium hydroxide to form cementitious compounds. Before the use of portland cement, volcanic ash or calcined clays and mixtures of lime were used for mortars and concretes. These materials are examples of pozzolans, and their reaction with lime is a pozzolanic reaction.

It is estimated that roughly one-fifth of the 900 million st of coal used annually is lignite and subbituminous. When burned, these coals produce high-calcium ashes, and the ashes have pozzolanic and cementitious properties. In the United States, 48 million st of fly ash was collected in 1980.

WATER-REDUCING ADMIXTURES

Water-reducing admixtures are used to reduce the water-to-cement (W-C) ratio. The W-C ratio is the weight of the water divided by the weight of the cement. High-range water-reducing admixtures, called superplasticizers, can be used to greatly reduce the W-C ratio. They can also increase the fluidity of water-cement mixtures at a given W-C ratio. It is possible to reduce the W-C ratio, increase the grout strength, and make the grout more mixable by using a superplasticizer.

TESTING PROCEDURES AND EQUIPMENT

The initial grout set time was tested using a handheld, spring-loaded Soiltest model CL700 penetrometer (fig. 2) with a spring-loaded calibration ring to record the approximate force. The penetrometer was fitted with a conical point that was pressed into each grout sample with the same force. The diameter of the depression is correlated with the approximate hardness of the grout. The harder the grout, the smaller the depression. While this test does not provide a precise strength versus time relationship, it allows set times to be compared qualitatively.

During bolt installation in mines, the ambient rock temperature and the rapid heat transfer can significantly affect

[4]Reference to specific products does not imply endorsement by the Bureau of Mines.

the setting characteristics of the grout. To model this heat sink effect, grout samples were mixed and cured in cups that were placed in water baths of different temperatures.

Bolt pull tests were begun in 24-in-deep by 1-3/8-in-diam holes using number 6 rebar. Grout failure, not bolt (rebar) failure (yield) is needed to measure the grout strength. To induce grout failure in the better performing grouts, grout lengths had to be shortened. Six-inch-deep holes in the concrete block

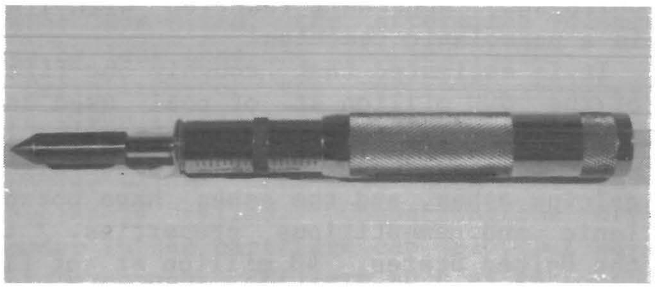

FIGURE 2.—Handheld penetrometer.

were sufficient to show short-term grout failure.

The 6-in-deep hole also simplified slurry installation. Fluid grouts were poured into holes while thicker grouts were tamped. However, cartridge testing was more difficult in these short holes. When the bolt broke the cartridge and mixed its contents, nothing prevented the mixed grout from coming out of the hole. This end effect disturbed the first inch or two of the grout bond.

To solve the problem, a 12-in-deep hole was created with the lower 6 in in the concrete block and the upper 6 in in a plastic tube (fig. 3). The end effect was confined to the section in the plastic tube. When the bolt was pulled, no reactance force was transmitted through the plastic tube. This procedure produced an effective 6-in hole.

Pull tests were used to measure bolt performance. The pull test apparatus (fig. 4) consists of a hydraulic pump, a hydraulic ram, a locking collet, a spacer

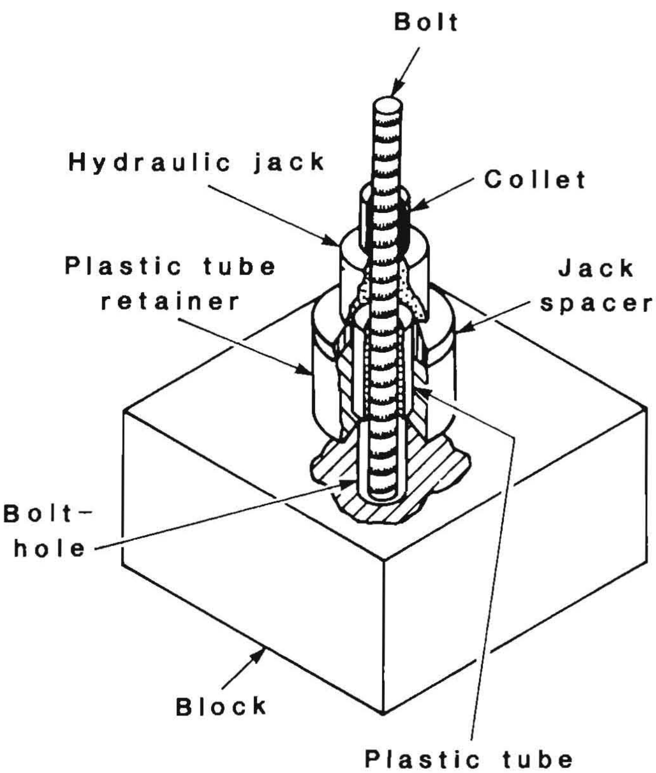

FIGURE 3.—Apparatus for cartridge insertion in short boltholes.

FIGURE 4.—Pull test apparatus.

block, an extensometer, and an X-Y recorder that plots the load versus bolt displacement. The shape of the curve indicates whether the grout failed or the bolt yielded. If the bolt yields, the curve shows the yield point, a decrease in load, and may show a gain in load to the bolt's ultimate strength. With a grout failure, the load builds up to the grout failure level and then diminishes.

It is recognized that bolt failure and grout failure are not as simple as treated in this report. The elastic and plastic characteristics of the bolt and grout are much more involved and may not be fully understood. For relative comparison between different grouts, a more complex evaluation is not necessarily deemed useful for this paper.

GYPSUM GROUT TESTING

The inorganic-grout cartridge was initially developed for 1-in-diam roof boltholes in coal mines. The package consists of a 0.97-in-diam polyethylene wrapper that contains Hydrocal White (U.S. Gypsum Co., Chicago, IL) plaster[5] and water capsules. The packaging system was assigned U.S. Patent No. 4,096,944.[6] The system was modified for use in hard-rock metal mines where boltholes are drilled to 1-3/8-in diam or larger with rotary-percussive machines.

PRELIMINARY TESTS

To determine the effect of hole and rebar size on pullout strength, tests were made in both concrete blocks and a rock face. Three methods of packaging the water in the cartridge were tested: water capsules, glass tubing, and segmented polyethylene tubing.

Water Capsules

Cartridges were handmade using 2-mil-thick polyethylene lay-flat tubing as the outside wrapper. The grout mix consisted of Hydrocal White plaster, sufficient water capsules to make a W-C ratio of 0.5, and potassium sulfate (K_2SO_4) accelerator (1.25 pct of the weight of the Hydrocal White).

Number 6, 7, and 8 (the rebar number refers to the number of 1/8-in diameters, e.g., a number 6 is 6/8 in) rebar (bolts) were grouted in 1-1/8-in, 1-1/4-in, and 1-3/8-in-diam, 18-in-deep holes in concrete blocks. Pull tests were performed 7 min after installation. The results ranged from 3,000 lb (number 6 rebar in a 1-3/8-in hole) to 21,000 lb (number 7 rebar in a 1-1/4-in hole).

Number 6, 7, 8, and 9 rebar were then grouted in 1-1/16-in, 1-1/4-in, 1-3/8-in, 1-5/8-in, and 1-3/4-in-diam holes drilled either 4 or 6 ft deep in a basalt face. Pull tests were performed 10 min after installation. The results ranged from 902 lb (number 6 rebar in a 1-5/8-in, 6-ft-deep hole) to 43,296 lb (number 9 rebar in a 1-3/4-in, 6-ft-deep hole). In several cases, the hydrated grout lacked sufficient volume to fill the annulus between the bolt and hole wall. In the 1-3/8-in-diam holes, it was necessary to use a number 8 rebar so that the hole would be completely filled.

Because making cartridges by hand is time consuming, a vibrator was fabricated (fig. 5). Between 6 and 10 min of machine tamping was required to achieve maximum cartridge density.

To determine the cause of intermittent poor anchorage, 18-in-long number 6 rebar bolts were pull tested in 14-in-long, 1-3/8-in-ID plastic pipes. Examination of the grouted bolts showed that the grout near the perimeter of the hole was poorly mixed. Many of the water pellets had not burst, and there were inclusions of unhydrated grout. The outer plastic wrapper was not completely shredded, and parts of it were lining the hole. Figure 6

[5]The chemical formula for plaster is $CaSO_4 \cdot 1/2H_2O$. When water is added, the plaster becomes gypsum ($CaSO_4 \cdot 2H_2O$).

[6]Simpson, R. E. Cartridge for Grouting an Anchor Element in a Hole of a Support Structure. U.S. Pat. 4,096,944, June 27, 1978.

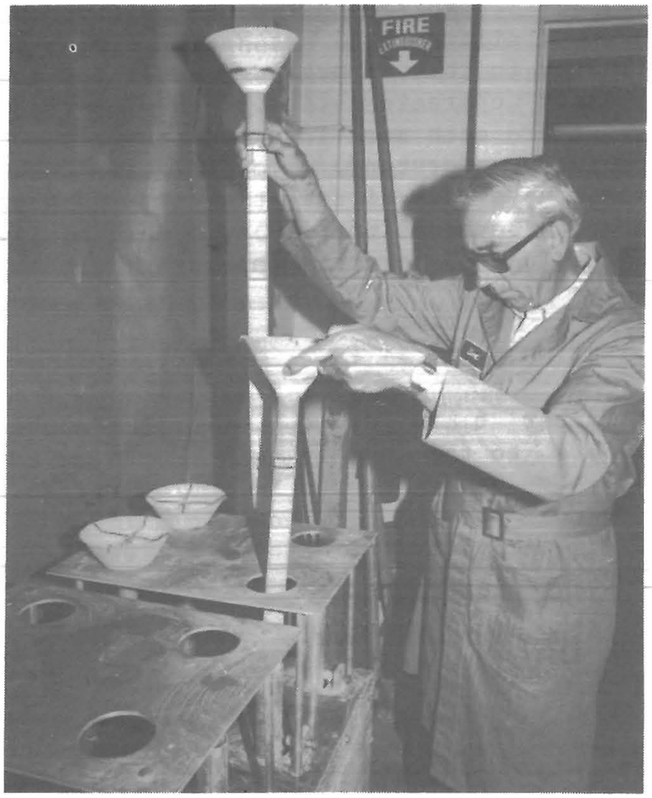

FIGURE 5.—Cartridge vibrator.

FIGURE 6.—Poorly mixed grout in bolthole.

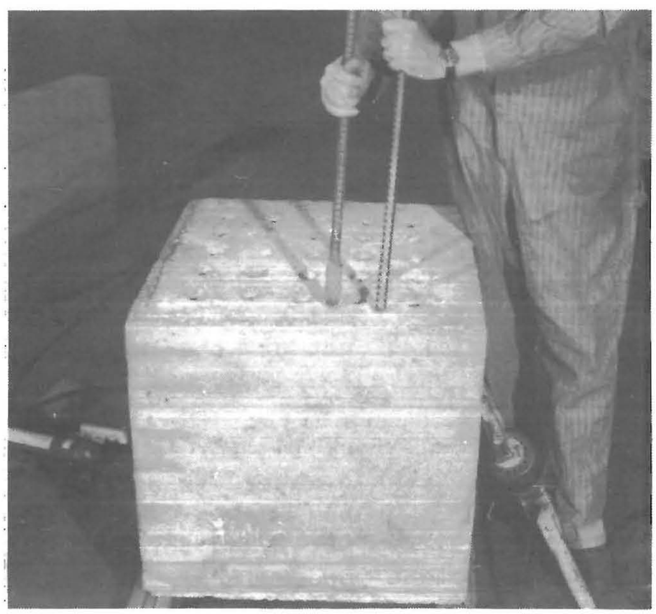

FIGURE 7.—Rebar with spade end (left) and without modification (right).

shows the grout with the uncrushed water pellets and the unshredded wrapper.

Sand was added to the Hydrocal White and the rebar end was flattened to a spade (1-1/4 in wide by 3 in long) to determine if an improvement in grout hydration and wrapper shredding would affect the anchorage strength. The number 6 rebar was grouted in 1-3/8-in-diam, 20-in-deep downholes in concrete blocks using an electric handheld rotary drill. In almost all of the cases, greater anchorage was achieved using the sand and gypsum mix and the modified rebar with spade end (shown along with the regular rebar end in figure 7). The volume of the cured Hydrocal White mix was 2 pct greater with the sand. However, the grout was still about 8 pct short of filling the annulus between the hole and bolt.

Glass Water Tubes

Because of the high cost of water capsules, a more economical way of packaging the water was desired. One-half-inch inside diameter thin-wall glass tubing was cut to length and filled with water. The tubes were sealed at both ends with plastic plugs. A superplasticizer, Pozzolith 400N (Master Builders, Cleveland, OH), was added to the water. This reduced the W-C ratio required for mixability from 0.5 to 0.4. Twenty-two-inch-long cartridges were made containing varying sand-to-cement ratios and 1 to 2 pct Pozzolith 400N (by weight of Hydrocal White). The component weights for one mixture are given in table 3.

Nine number 6 rebar bolts with spade ends were grouted horizontally into 1-3/8-in-diam, 4-ft-long holes. A pneumatically powered jackleg drill was used to drill the holes and install the bolts. Two bolts pulled to 11,750 lb, one after a 3-h cure time and the other after 24 h. The remaining seven pulled to a minimum of 21,150 lb.

The use of glass tubing as the water carrier is expensive. When bolts were installed in upward holes, the tubing broke ahead of the advancing rebar bolt so some water was lost out the hole.

Polyethylene Water Tubes

Because of the problems with the glass tubes, a 1-in lay-flat, 2-mil-thick polyethylene tubing was filled with water and sealed in segments of equal length (fig. 8) using a converted household Seal-A-Meal. The segments are designed to rupture sequentially as the bolt advances. The results are discussed later in this report under individual grout sections.

Gypsum Grout Expansion

When combined with the gypsum plaster, a small amount of aluminum sulfate ($Al_2(SO_4)_3$) causes expansion. The optimum amount was found to be between 1 and 2 pct $Al_2(SO_4)_3$ relative to the weight of the Hydrocal White. This provided a sufficient volume of cured grout to completely fill the void surrounding a number 6 rebar bolt in a 1-3/8-in-diam hole. The $Al_2(SO_4)_3$ also improved the mixability of the grout.

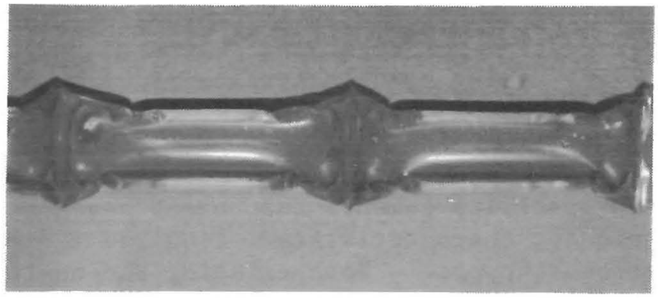

FIGURE 8.—Prepared water tube segments.

TABLE 3. - Components of cartridges made with glass water tubes

Ingredient	Weight, g
Hydrocal White	235
Fine sand	429
K_2SO_4	2.4
Water	95
Pozzolith 400N	3.6

Testing Segmented Polyethylene Water Tube Gypsum Cartridges

A rotary roof-bolter drill was used to drill eighteen 1-3/8-in-diam vertical holes in concrete blocks. The drill was then used to install number 6, grade 50 rebar bolts. Holes were either 44 or 48 in deep, depending upon the size of the block. The cartridges used for grouting contained 1.0 pct K_2SO_4, 2.0 pct $Al_2(SO_4)_3$, 1.5 pct Pozzolith 400N (by weight of the Hydrocal White), and equal parts of coarse and fine sand. The ratio of total sand to cement was 1.1, and the W-C ratio was 0.27.

Nine bolts with slightly bent ends (to improve mixing) registered pulls of 29,000 to 30,000 lb, 2 h after installation, with the exception of one that slipped at 15,000 lb. The yield strength of the rebar varied from 29,000 to 33,000 lb (which showed the rebar stronger than grade 50), as determined from stress-strain graphs made during anchorage pull tests. Three bolts with bent ends registered pulls of 12,000, 25,000, and 25,000 lb, 30 min after installation. The remaining six showed inconsistent results ranging from 12,000 to 22,700 lb after a 2-h set time. The variation in results was probably because of the difficulty in obtaining uniform grout mixing during bolt insertion. Because of the inconsistent results and the weakness of gypsum as a grouting medium, the testing was discontinued.

SEARCH FOR NEW GROUTS

A telephone search was made to locate new specialty cements that might have the unique characteristics required for ground support. Both existing and newly formulated cements were evaluated. Most commercially available fast-setting cements do not have the required initial set of 2 to 4 min, followed by a rapid set rate. The desired set time varies with the installation method. Because of the time the slurry is in the mixing chamber and injection tube, the slurry injection method of grouting needs more working time than does the cartridge insertion.

Penetrometer tests gave sufficient information to decide whether further testing was justified. The specific criteria and results for each grout are included in the discussion of each grout.

FONDU GROUT

Laboratory penetrometer tests were used to evaluate Fondu (Lafarge Calcium Aluminates, Inc., Chesapeake, VA) which is a calcium aluminate cement. To decrease set time and improve the strength, the samples of Fondu used in these tests were ground to a smaller particle size than the commercial grade of Fondu. In test series A (table 4), lithium carbonate was used as an accelerator. In tests 1 and 2, the lithium carbonate was dissolved in water while in tests 3 through 10, it was mixed in the Fondu powder. Results show that mixing the accelerator in water is more effective than mixing it with the dry ingredients. Increasing the amount of accelerator decreased the set time until the ratio of 1.5 pct by weight of the cement is reached. Beyond this, addition of more accelerator did not further decrease the set time.

In test series B, small amounts of sodium gluconate were added to increase the grout fluidity. Although the fluidity was increased, the set time was retarded.

In test series C, Centralia (Pozzolanic International, Mercer Island, WA) fly ash

TABLE 4. - Fondu penetration diameter tests

Series and test	Ingredients, g: Lithium carbonate	Sodium gluconate	Fondu	Centralia fly ash	Water	Water-Cement ratio	Penetration diameter, in, after-- 3 min	4 min	5 min	6 min
A:										
1...	[1]0.18						0.382	0.086		
2...	[1].31						.129	.095		
3...	.50						.373	.272		
4...	.75						.125	.089		
5...	1.00	None	50.0	None	18.5	0.37	.122	.092	NT	NT
6...	1.50						.113	.092		
7...	2.00						.105	.086		
8...	2.50						.135	.092		
9...	3.00						.106	.093		
10..	4.00						.103	.087		
B:										
11..	[1].18	0.02	50.0	None	18.5	.37	NAs	NAs	NAs	0.278
12..	.20	.005	50.0	None	18.5	.37	NAs	.151	NT	.080
C:										
13..	1.00	None	30.0	20.0	15.0	.50	NAs	.132	0.108	NT
14[2].	1.00	None	30.0	20.0	15.0	.50	NAs	.403	.381	NT

NAs Not applicable, too soft.
NT Not tested.
[1]Dissolved in water.
[2]Cured in 50° F water bath.

TABLE 5. - Inorganic slurry anchorage tests

(1-3/8-in-diam downholes in concrete blocks; all number 6 rebar bolts)

Cement type	Ingredient ratios			Age, h	Anchorage	
	Coarse-fine sand	Total sand-cement	Water-cement		Length, in	Strength, lb
9-41 C..................	NAp	NAp	0.22	16	20	29,500
				66	6	17,500
8-5[1]....................	1.3	1.3	.37	66	6	25,000

NAp Not applicable. [1]1 pct pozzolith by weight of cement.

was added. Centralia fly ash requires the smallest amount of water for fluidity of any fly ash tested. A mixture in which fly ash constituted 66.7 pct by weight of the Fondu, required less water for a given fluidity than a mixture without the fly ash, but the fly ash retarded the set time. This is shown by comparing test 5 with test 13. Because the set time is not rapid enough for mine roof bolting, no further testing was done.

ONE-COMPONENT NEAT CEMENT GROUTS

Two different single-component neat cement grouts, 9-41C and 8-5 (U.S. Grout Corp., Fairfield, CT), were tested as slurries for anchorage strength. Both materials provided excellent pull strength as shown in table 5, but required too much time to develop adequate strength. No further testing was done.

TWO-COMPONENT NEAT CEMENT GROUT

Thoro (Thoro System Products, Miami, FL) component 1 and 2 neat cement was tested for initial set time and strength development using the handheld penetrometer. Table 6 shows the penetration diameters at 6 and 17 min for several mixes with varying weight ratios of component 1 and 2. Each mix contained 18 g of Joliet (American Fly Ash Co., Des Plaines, IL) number 8 fly ash and 20 g of water. The Joliet number 8 fly ash was selected as an inexpensive filler because, for a fine particle size filler, its water demand is low. The fine size filler is desirable to reduce wear on the slurry injection equipment, and the low water demand is necessary for greater

TABLE 6. - Penetration diameters of Thoro two-component (1 and 2) neat cement

Test	Amount, g		Penetration diameter, in, after--	
	Thoro 1	Thoro 2	6 min	17 min
A......	33.75	11.25	([1])	NT
B......	22.5	22.5	0.173	0.156
C......	22.5	22.5	.180	.176
D......	16.87	28.13	.115	.113
E......	11.25	33.75	.114	.079
F......	11.25	33.75	.109	.075
G......	5.63	39.37	([1])	.094
H......	11.25	[2]33.75	.222	.129

NT Not tested.
[1]Set is too slow.
[2]Cured in 1 in. water at 50° F.

strength development. While a fixed price estimate is not available for the cement components, the fly ash priced at \$10/st to \$12/st will lower the total cost.

As the Thoro cement components 1 and 2 weight ratio of 33.75 to 11.25 (table 6), the 6-min set is too slow to measure the penetration diameter. As the 1 and 2 ratio is decreased to 11.25 to 33.75 (test E), the 6- and 17-min penetration diameters decrease, showing an increased set rate. However, continued reduction of the ratio to 5.63 to 39.37 results in a set rate that is too slow to get a good penetration test.

The 1 and 2 ratio of 11.25 to 33.75 appears to produce the fastest 6- and 17-min set as shown by the smaller penetration diameters. The same mix cured in a 1-in water bath at 56° F (test H) had a larger penetration diameter than the mix cured in air at the same temperature. This indicates that a heat sink effect, like that which can be experienced in a coal mine roof, has a significant effect on the set rate.

A 1 and 2 ratio of 11.25 to 33.75, 18 g fly ash, and 20 g water was tested to determine its fluidity after initial mixing. The freshly mixed slurry did not flow after the mixing. The water in the mix was increased to 22 g for greater fluidity, and while easily mixed by hand, the slurry again did not flow. This behavior is in contrast to other grouts where mixing at low W-C ratios is difficult, but after mixing, the grouts are quite fluid. Grout fluidity after mixing is desirable for cartridge and slurry injection to allow the grout to contact the annular surfaces, which will help increase the mechanical bonding.

Testing to determine the pull strength of the grout was not conducted because the grout was not acceptable because of its slow set time.

A,B GROUT

A,B (U.S. Grout Corp., Fairfield, CT) grout is a two-component, proprietary, high early strength grout that can be mixed in different ratios to achieve different set times. The A,B grout initially sent to the Bureau for testing contained a mixture of sand and a fixed ratio of A to B. This mixture had a very rapid set rate, but it was necessary to be able to vary the set time according to the differing conditions of use. To provide this control, separate components of A and B without sand were supplied by the manufacturer. This made it possible to mix components in different ratios and to add various fillers, thereby changing initial set times and mixability to suit the requirements of varying bolt installations.

Penetrometer tests (table 7) were made to determine set times for grouts with different W-C ratios. The results showed that a faster set was achieved at lower W-C ratios. Penetrometer tests also verified that the initial set time could be varied by changing the ratio of A to B.

To investigate the effect of heat transfer from the fluid grout to a thermal mass, such as mine rock, a mixture of 18 g of A, 4.5 g of B, 20 g of Wedron 4030 (Wedron Silica Co., Pacific Grove, CA) sand, 30 g of gravel, and 6.3 g of water was cured in air and an identical mix was cured in a water bath. The air-cured mixture set in 2 min, and the reaction was strongly exothermic. In contrast, the mixture in the water bath had not set after 9 min. Because water conducts heat faster than would mine rock, it illustrates the importance of heat transfer from the grout on the set time.

Other factors that can affect the initial set time and the set rate are the grouting method (slurry or cartridge) and the use of fillers. The ability to widely vary the set time of the A,B grout provides the flexibility required to accommodate these factors.

Slurry Pull Tests

The results of pull tests on bolts grouted with A,B slurry are shown in table 8. A commercial ungraded, uncleaned sand was used as a filler in test series A through E, while fly ash was used as a filler in test series F through H. Test series A produced excellent mid-term

TABLE 7. - Penetration diameters for mixtures of A (17.5 g), B (7.5 g), and sand[1] (25 g), with water

6 g water			7 g water			8 g water		
Penetration diameter, in	Time min	Time s	Penetration diameter, in	Time min	Time s	Penetration diameter, in	Time min	Time s
0.400	2	0	0.400	2	15	0.400	2	15
.143	2	15	.400	2	30	.400	2	30
ND	2	30	.047	2	45	.070	2	45
ND	2	45	ND	3	0	.020	3	0

ND Not determined.
[1]Morie No. 50 sand.

TABLE 8. - A,B slurry anchorage tests

Test series	Ingredient ratios: A-B	Coarse-fine sand	Total sand-cement	Fly ash-cement	Water-cement	Age: h	Age: min	Anchorage: Length, in	Anchorage: Strength, lb
SAND FILLER									
A.....	2.3	None	1.0	None	0.16	17	0	12	[1]30,000
						66	0	6	24,500
						72	0	13	[1]29,500
						71	0	24	[1]29,500
B.....	2.1	1.0	2.2	None	.31	0	10	6	10,500
						0	30		27,500
						2	0		[1]29,000
						5	0		[1]29,000
C.....	3.0	1.5	2.2	None	.28	0	30	6	24,500
									26,500
									26,500
									28,000
									30,000
D.....	4.0	1.0	2.0	None	.32	69	0	4	18,000
E.....	5.7	1.0	1.7	None	.32	0	10	6	12,000
FLY ASH FILLER									
F.....	2.3	None	None	0.4	0.27	0	50	6	[1]31,000
						1	30		26,000
						19	0		26,500
G.....	2.3	None	None	1.0	.37	0	50	6	12,000
						1	30		22,500
H.....	2.3	None	None	1.5	.44	0	50	6	20,500
						1	30		17,000

[1]Bolt yielded.

strengths (15 to 30 min). Two examples of good short-term strengths (<15 min) were shown in test series B and E; however, most of the strength development occurred between 10 and 30 min.

Increasing the ratio of A to B reduced the initial set time. The initial set time minus the mix time constitutes the work time (see figure 1). This is the time available for the worker to place the grout and install the bolt. Care must be taken not to decrease the initial set time so much that the working time is inadequate. The required working time will vary depending on the bolting system. The ability to vary the initial set by varying the ratio of A to B is one of the desirable characteristics of the A,B grout. Ratios ranging from 2.1 to 5.7 were used in test series B through E in order to examine the acceleration of the initial set. The tests suggest that a ratio of 3.0 provides the optimum useful acceleration. Any ratio over 3.0 provides a working time that is too short and potentially detrimental to the quality of the hardened grout.

Test series F, G, and H used fly ash as a filler. Because fly ash has a greater surface area than sand, it requires more water to achieve a desirable mixability. In test series F, mid-term pull strengths were excellent. When the fly ash to cement ratio is increased, as in test series G and H, the pull strengths are significantly reduced and less consistent.

Cartridge Pull Tests

Problems were encountered with bolt insertion and with in-hole mixing as the A,B cartridge segmented water tubes did not break in a consistent manner. Consequently, bolt thrust was reduced and its rotation was increased to improve mixing. Also, a rotary impact drill and different bolt tips were tried. However, none of these changes had a positive effect.

The A,B cartridge test results are shown in table 9. Test series A and B show lower mid-term strengths than those obtained with the slurry grout (table 8). Pull test results also varied in test series C.

Cost of A,B Grout

The estimated cost of A,B grout is \$0.292/lb. To anchor a number 6 rebar in a 1-3/8-in-diam, 12-in-long hole with grout containing 40 pct fly ash filler requires about 174.3 g of A, 74.7 g of B, and 99.6 g of filler. If the cost of the filler is estimated at \$0.005/lb, the A,B grout cost per equivalent foot is about \$0.161.

RAPID SET GROUT

Rapid Set grout (Rapid Set Products, Van Nuys, CA) is a single-component, proprietary, high early strength grout. The grout does not contain fillers, but they can be added to reduce the cost.

TABLE 9. A,B cartridge anchorage tests

(1-3/8-in-diam downholes in concrete blocks)

Test series	Ingredient ratios: A-B	Coarse-fine sand	Total sand-cement	Water-cement	Age: h	min	Anchorage: Length, in	Strength, lb
				ROTARY DRILL				
A	2.1	1.0	2.2	0.32	0	30	6	10,000
B	3.0 1.9	([1])	2.2	.30	1	0	6	5,000 12,000
C	3.0	1.5	2.2	.28	0	30	6	17,500
					2	0	6	14,000
					0	30	12	[2]30,000 [2]30,000
					0	30	6	9,500 13,000 15,000 13,000 [3]18,000 26,000
				ROTARY-IMPACT DRILL				
C	3.0	1.5	2.2	0.28	3	30	6	8,000 12,000 13,500 15,000

[1]Well-graded from minus 3/8 in through minus 50 mesh.
[2]Bolt yielded.
[3]No. 7 rebar with chisel-point end; No. 6 rebar used in all other tests.

Slurry Pull Tests

The results of pull tests using Rapid Set are shown in table 10. Pozzolith 400N was added to the Rapid Set in test series A. Pozzolith 400N improves the grout workability at a given W-C ratio, or it can be used to reduce the W-C ratio while maintaining the original workability. Sand was also added to reduce the grout cost. The effects of Pozzolith 400N on set time and strength were not measured. The pull results showed excellent mid-term pull strengths of 20,000 to 30,000 lb for a 6-in anchorage; however, early strength was poor.

Rapid Set A, a formulation designed to improve workability, was used in test Series B. The improved mixability allowed the Pozzolith 400N to be eliminated. Sand was again incorporated as a filler. The pull tests yielded excellent mid-term strengths.

In test series C, fly ash was used as the filler. As expected, the W-C ratio required to achieve proper workability was higher than it was when using sand. The mid-term strengths were lower than those obtained with sand as a filler.

Rapid Set and Rapid Set A showed excellent long-term strengths, but poor early strength makes the grouts (in their present form) unacceptable for bolting in most mining applications.

Cartridge Pull Tests

Rapid Set grout was also tested in cartridge form. Results are shown in table 11. The results from test series A

TABLE 10. - Rapid Set slurry anchorage tests

(1-3/8-in-diam downholes in concrete blocks; No. 6 rebar bolts)

Test series	Pozzolith, pct[1]	Ingredient ratios: Coarse-fine sand	Ingredient ratios: Total sand-cement	Ingredient ratios: Fly ash-cement	Ingredient ratios: Water-cement	Age: h	Age: min	Anchorage: Length, in	Anchorage: Strength, lb
A[2]....	2.0	1.3	1.3	None	0.31	0	10	6	None
						0	30		20,000
						2	0		29,000
						5	0		29,000
	1.0	1.3	1.3	None	.31	66	0	6	[3]30,000
						69	0	4	28,000
B[4]....	None	1.3	1.3	None	.31	2	30	6	[3]29,500
						2	30		28,000
						0	45		20,000
	None	1.5	2.2	None	.33	0	30	6	26,000
									27,500
									27,500
									[3]30,000
									[3]31,500
FLY ASH FILLER									
C[4]....	None	None	None	0.4	0.36	0	45	6	17,500
						1	30		22,500
						19	0		26,000
				1.0	.47	0	45		18,000
						1	30		18,500
				1.5	.53	0	45		9,000
						1	30		18,000

[1]By weight of cement.
[2]Rapid Set cement used.
[3]Bolt yielded.
[4]Rapid Set A cement used.

TABLE 11. - Rapid Set cartridge anchorage tests

(1-3/8-in-diam downholes in concrete blocks; No. 6 rebar bolts)

Test series	Pozzolith, pct[1]	Ingredients ratios: Coarse-fine sand	Ingredients ratios: Total sand-cement	Ingredients ratios: Water-cement	Age: h	Age: min	Anchorage: Length, in	Anchorage: Strength, lb
ROTARY DRILL								
A[3]..........	2.0	1.3	1.3	0.35	23	0	22	[2]28,500
								[2]28,500
								[2]29,000
				.31	67	0	4	25,000
							10	28,500
							11	29,000
B[4]..........	None	1.5	2.2	.38	2	30	6	10,000
C[4]..........	1.4	([5])	1.8	.31	0	30	5	2,500
D[4]..........	None	([5])	1.8	.34	1	45	6	20,000
						20		13,500
F[4]..........	None	1.5	2.2	.33	0	30	6	[6]8,000
								[6]9,500
								[6]10,000
ROTARY IMPACT DRILL								
E[4]..........	None	1.5	2.2	0.33	0	30	6	3,000
								4,000
								7,500
								8,500
								[7]6,500
								[7]7,000
								[7]8,000
G[4,8]........	1.2	1.5	2.2	.32	0	30	6	5,500
								6,000
								6,000
					1	0		4,500
								5,500
								5,500
					2	0		11,500
								13,000
								13,000
					4	0		10,000
								11,500
								12,500
					24	0		13,000
								15,000
								17,500

[1]By weight of cement.
[2]Rebar yielded.
[3]Rapid Set cement.
[4]Rapid Set A cement used.
[5]Well graded from minus 3/8 in through minus 50 mesh.
[6]No. 7 rebar; end of bolt ground to chisel point.
[7]End of bolt flattened into spade.
[8]End of bolt ground to chisel point in all tests.

show adequate long-term strengths with various anchorage lengths. Both anchorage length and cure times were shortened in test series B, and the results showed that mid-term strengths were reduced. In test series C, the cure time was reduced to 30 min for a 5-in anchorage length; and as a result, there was a further decrease in pull strength. The strengths obtained in test series B, C, and D are significantly lower than those obtained in similar tests using slurries (table 10). Visual observations during mixing and a comparison of pull strength test results indicate that the lower strengths are due to inadequate mixing.

The rebar ends were modified and impaction was added to the insertion rotation to improve mixing. The results were better than before, as shown in series E, F, and G, but the strengths obtained were still significantly less than those of the slurry test series B.

In Rapid Set test series E, the results were again very erratic (table 11). The variability in strength corresponded with that observed as a result of inadequate mixing and difficult bolt insertion. It appears that the segments of the water tubes were not breaking uniformly. Evidently, the rotating bolt could not impart sufficient in situ energy to the grout to adequately mix the ingredients. More energy was added to the system by impacting, and this energy was distributed with modifying the bolt tips, but this did not eliminate the problem.

In an attempt to improve mixing by reducing the annulus between the bolt and the hole wall, a number 7 rebar was used instead of a number 6 rebar (test series F, table 11). The results showed some improvement but significantly less strength than those of similar slurry tests.

Cost of Rapid Set Grout

Rapid Set was combined with sand and fly ash fillers to reduce the cost and improve mixability. The estimated cost of Rapid Set is $0.15/lb; whereas that of fly ash is $0.005/lb. To fill the annulus of a 1-3/8-in-diam hole, 12 in long, using a number 6 rebar and a 50-50 mixture of Rapid Set and fly ash, requires 170.9 g of Rapid Set and 170.9 g of fly ash. This mix cost is $0.058. The estimated cost of a polyester resin cartridge for a 1-3/8-in hole using a number 7 rebar is about $0.38/ft.

These costs are not directly comparable because the amount estimated for the Rapid Set includes only material costs, whereas the polyester cost is for a marketed product. Reducing the hole diameter to 1 in and using a number 6 rebar reduces the annular volume and resin cartridge cost to about $0.19/ft. A reduction in the annular volume would also decrease the cost of a Rapid Set grouted bolt, but drilling a 1-in-diam hole is more difficult than drilling a standard 1-3/8-in-diam hole.

ORGANIC CARTRIDGES

Grouting bolts with polyester resin is the standard practice in the mining industry. The use of any new grouting material would be compared with the use of the standard practice. Therefore, in order to provide comparative data, resin cartridges were tested. In table 12, test series A, two 0.9-in-diam cartridges were placed side by side in a 1-3/8-in-diameter hole and the bolt was then inserted. Anchorage strengths were erratic because of poor mixing caused by the nonstandard installation. Test series B used the 1-1/4-in-diam resin cartridge that is designed to be used in a 1-3/8-in-diam hole. Pull test results were excellent.

Results of pull tests on a new epoxy slurry grout, developed under contract by the Bureau, are shown in test series C. Results for long-term strength were excellent but no tests of mid- or short-term strengths were made.

TABLE 12. - Organic resin anchorage tests

(1-3/8-in-diam downholes in concrete blocks; No. 6 rebar bolts)

Test series and cement type	Age h	Age min	Anchorage[1] strength, lb	Test series and cement type	Age h	Age min	Anchorage[1] strength, lb
A: Resin[2]........	0	10	10,000	B: Resin[4]-Con....	0	15	22,500
	0	10	6,000		0	30	21,200
	0	30	18,500		1	0	17,200
	0	30	10,000		0	5	20,500
	2	0	18,000		0	10	27,000
	2	0	[3]6,500		0	15	28,500
B: Resin[4]........	0	15	29,000	C: Epoxy[5]........	19	0	25,000
	0	30	23,500		19	0	25,000
	1	0	24,500		2	30	26,000

[1]6-in anchorage length.
[2]Two 0.9-in-diam Celtite cartridges split and inserted side by side; No. 6 rebar bolts.
[3]Poor mixing.
[4]1-1/4-in-diam Celtite cartridge, 12 in long, 37-s gel; No. 7 rebar bolts.
[5]Amicon 2-component.

SOAKABLE CARTRIDGES

Packaging the cement, water, and fillers in a cartridge that will fit in a bolthole does not often provide a sufficient volume of mixed grout to fill the annulus between the bolt and the hole wall. Cartridges that are permeable to water have been commercially available, but with a much slower set time; they absorb water to allow the water and cement to combine prior to being inserted into the hole. If the cartridge volume is not used for water, it can be used for needed cement and fillers. The elimination of water packaging in the cartridge can also reduce the cost of the cartridges.

The water must be mixed with the cement uniformly along the cartridge length. The permeability of the cartridge wrapper would control the seepage rate so that soaking for a specified period of time will achieve the required W-C ratio. However, a mixture of cement and fillers that would absorb only the needed quantity of water would eliminate the need for timed soaking. The cartridge must also be strong enough to take the abuse of handling, yet tear easily when the bolt is inserted through it during installation.

The energy derived from the rotating bolt during installation is limited. Presoaking should simplify mixing and ease the bolt installation.

A few tests of this concept were conducted using Rapid Set A, Centralia fly ash, and different sands. The Centralia fly ash was used because of its low water requirement. The 1.25-in-diam wrapper was made with a 4.5-in-wide knit fibrous paper that was rolled loosely around a tube; the overlapping edges were glued. The wrapper was filled with a mixture of 71.6 g of Rapid Set A and 71.6 g of fly ash, making a 6-in-long cartridge. The cartridge used 32.2 g of water because earlier tests showed that amount of water as allowing hand mixing. The water and the cartridge were placed in a water-filled plastic container for 40 s to soak. The cartridge and the bolt were then installed in a 1-3/8-in-diam by 6-in-deep hole using a 3/4-in, 375-rpm handheld drill. The mixed material was not sufficient to fill the hole. It appeared that this was caused by the lack of compaction or consolidation of the dry cartridge material.

Another cartridge was made using 71.6 g of Rapid Set A, 64.5 g of Centralia fly ash, and 20.0 g of Morie number 4 sand. It was soaked with 30.5 g of water, and it and the bolt were installed; but the results were unsatisfactory because the mix was too dry.

In the next cartridge, the fly ash was replaced by different sizes of sand. Another 6-in-long cartridge was made, this time using 60.72 g Rapid Set A, 40.5 g Morie number 50, 53.98 g Morie number 0, and 47.23 g Morie number 2. It was soaked in 24.32 g of water and installed with the bolt, but again the hole was not completely filled.

Despite these problems, compared with performance of previous water-based cartridges, soakable cartridges are promising for reducing costs and for providing superior in-hole mixing. However, additional tests are needed to formulate cartridges and further develop packaging. Also, additional work will be needed to improve the wet strength of the cartridge.

SUMMARY

Inorganic cements were evaluated in slurry and cartridge tests. An important part of this work was the evaluation of different cartridge types and installation procedures. Packaging water inside the cartridge remains a problem; none of the systems performed satisfactorily in the tests, and none performed even as well as the previously tested water capsules. To improve performance, various installation methods, bolt modifications, and admixtures were tried, but the results were unsatisfactory.

Several inorganic grouts were tested, but because of better performance, the major testing effort centered on A,B and Rapid Set grouts. A,B grout and Rapid Set grout both had good mixability and good mid- and long-term pull strength, but marginal early strength.

Tests showed that 6-in anchorage lengths of fully cured A,B grout and Rapid Set grout both resulted in bolt yielding. For an inorganic grout to be competitive with resin, it must outperform resin at the same cost or perform as well at a lesser cost. After achieving their mid-term strength, A,B and Rapid Set grouts were able to outperform resin; but during the early set, the resin was superior.

The ability to vary the set time is important for both cartridge and slurry installation. Both the set and the strength gain must be rapid enough to give adequate early roof support, but must not take place before or during bolt installation or the integrity of the bond will be compromised. Resin can be formulated to accommodate ambient temperatures, energy imparted during installation, bolt length, etc. The results described here showed that A,B grout and Rapid Set grout set rates can be varied by changing the proportions of the ingredients. Formulation of the grout to achieve an early set is a principal area of future A,B and Rapid Set research.

The research into inorganic grouts is primarily motivated by economic factors. A superior full-column inorganic support that is less costly than mechanical point anchorage and resin systems would be desirable for industry use.

Resin bolt manufacturers use fillers to reduce material costs while maintaining adequate anchoring strength. To be competitive, inorganic grout costs must be minimized by using inexpensive fillers. Both A,B and Rapid Set were tested using different fillers. Different applications require different filler characteristics. The coarse sand used in a cartridge system helps to mix the ingredients in the hole; but in a slurry injection machine, a finer size filler is desirable to avoid abrasive wear of machine components.

Cartridge and slurry test grouts were made using different sands and fine gravels; but because of the inconsistent cartridge test results, no comparisons can be made. However, slurry admixture test results indicated that both grouts are amenable to cost-reducing fillers. When sand was used as a filler with the A,B grout, pull strengths were excellent at the maximum tested sand-to-cement ratio of 2.2. With fly ash added to the A,B grout, strengths remained good up to a fly ash-to-cement ratio of 0.4. Beyond this ratio, pull strengths decreased dramatically.

Rapid Set showed similar characteristics when sand and fly ash were used as fillers. Pull strengths were excellent at the maximum tested sand-to-cement ratio of 2.2. With fly ash as a filler, the strength remained excellent up to a fly ash-to-Rapid Set ratio of 0.4. Increasing the ratio resulted in a decrease in pull strength. These tests are preliminary, but they do show that the grouts can be made less expensive by using fillers, yet they retain adequate strength.

CONCLUSIONS

Comparative tests of grouting material should be conducted on bolts short enough so that the grouting material fails, and not the bolt. If the load from the bolt to rock is transferred in the first 6 to 12 in of the hole, testing a longer bond length does not produce relevant data.

The fast-setting inorganic grouts proved to have very good mid- to long-term strength but had low early strength.

The inorganic grout material cost can be less than that for resin, so that inorganic grouts have the potential for providing full-column support more economically than resin anchorage.

Packaging of water and dry grout in a cartridge was unsuccessful because it resulted in poor mixing in the hole and volumetric shortages. A few tests of rapid-setting, soakable cartridges showed that water can be introduced prior to installation by presoaking the cement cartridge.

Laboratory tests have shown that rapid-setting, inorganic grout could be an economical replacement for resin bolts if the short-term strength could be increased. Further development to improve its early strength would need to be tested in the laboratory and underground to prove its potential for the mining industry.

U.S. GOVERNMENT PRINTING OFFICE: 1987 605-017/60 100

INT.-BU.OF MINES,PGH.,PA. 28557