U.S. Air Force Rapid Airfield Damage Recovery (RADR) Modernization Program

**Laboratory Characterization of Rapid-Setting Flowable Fill**

William D. Carruth and Monica A. Ramsey

April 2020
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Laboratory Characterization of Rapid-Setting Flowable Fill

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Abstract

Utility Fill One-Step 750® is a rapid-setting flowable fill product that has previously been validated in numerous full-scale demonstrations as an expedient backfill solution for Rapid Airfield Damage Recovery. Although the field performance of Utility Fill One-Step 750® has been extensively documented, a full laboratory characterization has not been conducted. This report analyzes and documents results from several laboratory tests conducted at two water-to-product ratios. The tests conducted are based on the suite of tests that make up the tri-service spall repair certification program used for rapid-setting concrete products. Tests include strength and set time-related properties, typical parameter control tests for concrete, and tests to determine the mineralogy and chemical makeup of the material. Long-term expansion and contraction properties were also tested. The data presented herein are intended to provide an overall assessment of Utility Fill One-Step 750® and to provide reasonable estimates of various design parameters. The results can be used as a basis for the future development of a formal laboratory certification protocol to down-select other rapid-setting flowable fill products for further evaluation.
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Preface

This study was conducted for the U.S. Air Force's (USAF) pavement evaluation teams, contingency readiness groups, base civil engineers, major command pavement engineers, Rapid Engineer Deployable Heavy Operational Repair Squadron Engineer (RED HORSE) squadrons, and Prime Base Engineer Emergency Force (BEEF) units. Additional users of this report include Army, Navy, and Marine Corps units charged with the repair and sustainment of bomb-damaged airfield pavements.

The project described in this report is part of the USAF’s Rapid Airfield Damage Recovery sponsored by Headquarters, Air Force Civil Engineer Center (AFCEC), Tyndall AFB, FL, under MIPR F4ATA73351G00. The technical monitor for this project was Dr. Craig Rutland of the Air Force Civil Engineer Center. The U.S. Army Engineer Research and Development Center (ERDC) technical manager for this project was Mr. Jeb S. Tingle.

This work was performed by the Airfields and Pavements Branch (GMA) and the Concrete Materials Branch (GMC) of the Engineering Systems and Materials Division (GM), U.S. Army Engineer Research and Development Center, Geotechnical and Structures Laboratory (ERDC-GSL). At the time of publication, Dr. Timothy W. Rushing was Chief, GMA; Mr. Christopher M. Moore was Chief, GMC; Mr. Justin S. Strickler was Chief, GM; and Mr. R. Nicholas Boone, GZT, was the Technical Director for Force Projection and Maneuver Support. The Deputy Director of the ERDC-GSL was Mr. Charles W. Ertle II, and the Director was Mr. Bartley P. Durst.

COL Teresa A. Schlosser was the Commander of ERDC, and Dr. David W. Pittman was the Director.
1 Introduction

1.1 Background

The U.S. Air Force Air Combat Command began the Rapid Airfield Damage Recovery (RADR) Modernization Program to develop technologies to address operational limitations of current RADR equipment, materials, and tactics. The overall objective of the program is to modernize the Air Force’s RADR capability through development of new RADR solutions that are suitable for fighter and cargo aircraft while scalable to the threat or damage. Since 2006, researchers at the U.S. Army Engineer Research and Development Center (ERDC) have been conducting research under the program to develop new, expedient pavement repair techniques in an effort to update repair guidance for military airfields. Damaged airfield pavements must be repaired quickly using suitable materials to reduce the total time that the airfield is removed from service, as well as reduce the need to conduct subsequent repairs to maintain an operable pavement surface, particularly during wartime scenarios. A more complete overview of the RADR modernization program from 2006 forward can be found in Carruth et al. (2015).

Cementitious, rapid-setting concrete repair materials have been successfully demonstrated for capping repairs of bomb-damaged concrete pavements as a part of the RADR Modernization Program. However, before these materials were evaluated on full-scale test sections, they were down-selected in the laboratory using a certification protocol developed by Priddy (2011). The protocol contains not only requirements for early strength gain (e.g., 2-hr compressive strength) but also investigates the long-term durability of rapid-setting concrete materials as well as other properties. The program is now referred to as the tri-service spall repair certification program.

Rapid-setting flowable fill was first evaluated as a backfill material for crater repair in 2009 (Priddy et al. 2013). Utility Fill 1-Step 750® manufactured by Buzzi Unicem is a rapid-setting flowable fill material that has been selected for use as a rapid backfill alternative because it can be easily placed with or without the use of external mixing. In addition, rapid-setting flowable fill has been demonstrated as a surface
capping material capable of withstanding approximately 500 passes of F-15 or C-17 aircraft (Carruth and Howard 2016).

Utility Fill One-Step 750® is traditionally placed by using a standard transit truck or the U.S. Air Force (USAF) simplified volumetric mixer (SVM). This technique is referred to as the “wet method”; it uniformly distributes moisture, resulting in optimal flowability and achievement of ultimate compressive strength (Figure 1). The material can also be placed by using the “dry method” (Figure 2). The dry method is an expedient placement technique in which the pre-blended dry material is dispensed directly into the excavation, alleviating the requirement for a dedicated mixer. Following the placement of thin lifts (4 to 6 in.) of dry material, water is metered onto the surface and allowed to percolate through the dry material. Placement using the dry method sacrifices some of the beneficial properties of flowable fill, including its self-leveling behavior and up to 30% of the compressive strength. However, the material can be placed expediently as a backfill without the use of additional equipment and provides sufficient bearing capacity for heavy aircraft pavement applications. The dry method is typically used when Utility Fill One-Step 750® is used as a base material beneath a rapid-setting concrete cap, as described in Edwards et al. 2013, Bell et al. 2013, and Carruth et al. 2015.

Figure 1. Rapid-setting flowable fill wet placement method.
While Utility Fill One-Step 750® has been successfully demonstrated on numerous occasions via full-scale tests and troop demonstrations, a complete laboratory characterization has not been previously documented. This report describes the results from a suite of laboratory tests conducted on Utility Fill One Step 750® using the tri-service spall repair certification program as a basis for testing. The results obtained from these tests are a first step to developing a laboratory testing protocol to down-select other rapid-setting flowable fill products for full-scale evaluation.

1.2 Objective and scope

The objective of the testing described in this report was to fully characterize Utility Fill One-Step 750® via multiple laboratory tests. This objective was carried out by completing the appropriate testing, documenting the results, and providing pertinent analysis. Conclusions and recommendations were developed based on the analysis and are provided in Chapter 4 of this report.

1.3 Outline of chapters

Chapter 1 provides background information covering previous rapid-setting flowable fill investigations and the specific objectives and scope of the work covered in this report. Chapter 2 presents an overall
description of the laboratory testing conducted and provides pertinent
details of each individual test. Chapter 3 provides the results of each
laboratory test along with the associated analysis, and Chapter 4
discusses the conclusions and recommendations developed. References
used in preparing this report are also provided.
2 Description of Laboratory Tests

This chapter describes how test specimens were fabricated and tested in the laboratory. Various details of each test method are discussed along with the number of replicates and the American Society of Testing and Materials (ASTM) standard used, if applicable.

2.1 Mixing procedure

Each mixture used one 50-lb bucket of Utility Fill One-Step 750® material. The material used was delivered from the manufacturer in buckets since sampling from super sacks can result in segregation of the dry material. The dry materials, water, and equipment were stored at a temperature of 73°F and a relative humidity of 50% for a minimum of 24 hr before testing. Mixing was performed in an aluminum container by using a dual paddle mixer. The dry material was blended first without water to establish uniformity. Water was slowly added to the dry material. Once all the water had been added, mixing commenced for 3 min. After mixing, researchers used the material to fabricate the appropriate specimen or to perform other measurements.

2.2 Water-to-product ratios

The proportion of water to the rest of the pre-blended dry material was referred to as the water-to-product (w/p) ratio. Two different w/p ratios were used for testing. The “low” w/p ratio used was 0.11 and required 0.66 gal of water per 50-lb bucket of flowable fill. The “high” w/p ratio used was 0.21 and required 1.25 gal of water per 50-lb bucket. The two different w/p ratios were employed in order to simulate different consistencies of rapid-setting flowable fill placed by using the wet method. The lower w/p ratio would exhibit higher strength but lower workability, while the higher w/p ratio would likely have lower strength and higher workability. The low w/p ratio represents using approximately 40 gal of water per 3,000-lb supersack, while the high w/p ratio represents 70 gal of water per 3,000-lb super sack. All tests conducted were performed for both w/p ratios, with the exception of tests performed on the dry material only (e.g., cement content).
2.3  **Strength**

Strength is a key parameter for rapid-setting flowable fill backfill materials in the RADR base recovery after attack scenario. The material must gain strength quickly so that a capping material can be placed and continue to gain strength during capping material placement and during curing. If asphalt is used as a capping material, early strength is even more critical so that during asphalt compaction the backfill layer can be used to compact against in order to achieve adequate asphalt density. Strength is typically reported as flexural strength and/or compressive strength. Flexural strength can be correlated to compressive strength, although both tests can be required in many cases and were performed in this testing protocol.

### 2.3.1  **Compressive strength**

Minimum compressive strength is a vital parameter for rapid-setting flowable fill backfill to ensure that the crater repair can support heavy aircraft wheel loads. Compressive strength testing was conducted in accordance with ASTM C 39 procedures (2005). The compressive strength was determined by testing 4-in.-diameter by 8-in.-high cylinders. The procedures used for curing and capping the specimens are addressed in section 2.10. The cure times used for compressive strength specimens were 1 hr, 2 hr, 1 day, and 28 days. The cure time was defined as the time from final finishing to testing of a specimen, not the time from initial set of the material to the time of testing. Results were reported as maximum compressive stress (psi) or the maximum applied load (in pounds) divided by the cross-sectional area (in square inches) of the specimen.

### 2.3.2  **Flexural strength**

Flexural strength, or bend strength, is a material property defined as the stress in a material just before it yields in a flexure test. Flexural strength testing occurred in accordance with ASTM C 78 procedures (2016b). The test specimens were rectangular prisms with dimensions of 6 in. by 6 in. by 21 in. long. Tests were conducted on the specimens with an unsupported span equal to three times its depth. Loading was applied at third-points of the span. The beams were tested at the same cure times as the compressive strength cylinders described in the last section with one exception. Beams cured for 1 hr had not reached sufficient strength
to allow for extraction for testing and, thus, could not be tested. The ultimate tensile stress achieved before failure, or the modulus of rupture (psi), was recorded and reported as the flexural strength of the material.

### 2.3.3 Bond strength

Bond strength is important to ensure that the rapid-setting flowable fill will bond to itself, since the material is often placed in layers, and to the rapid-setting concrete capping material that is placed over the material during crater repairs. If the material does not properly bond, cracking or settlement of the repair under heavy aircraft traffic can occur. Bond strength testing was conducted in accordance with the requirements of ASTM C882 (2013a). Cylinder molds 3 in. in diameter by 6 in. in height were used, and a bond line was created at approximately 30° from vertical by first casting wedge-shaped sections of both Utility Fill One-Step 750® and the rapid-setting concrete. After the wedge-shaped sections were cured, the repair material was placed on top of the sections in the mold and cured for 1 and 7 days. Curing and capping methods for the cylinders are described in Section 2.10. Results were reported as maximum bond stress (psi), defined as maximum axial load divided by the area of the bonding surface, which has the shape of an ellipse.

### 2.4 Basic control parameters

#### 2.4.1 Flow consistency

The slump test measures the workability and the consistency of fresh concrete. ASTM C 143 (2015d) is the most generally accepted test method to determine the consistency of concrete. However, the consistency of the rapid-setting flowable fill is not thick enough to perform a slump test. The flow consistency test is used to measure the consistency of flowable controlled low-strength materials (CLSM) with a maximum particle size of ¾ in. CLSM is defined as a blend of aggregates, cementitious material, fly ash, water, and chemical admixtures, which hardens into a material with a higher strength than the soil but less than 1,200 psi. Since rapid-setting flowable fill meets this definition (with the exception of the 1,200-psi maximum in some cases), the flow consistency test was employed to measure consistency. Testing was conducted according to ASTM D 6103 (2017). To perform the flow consistency test, the material is placed into a cylinder with openings on both ends, the material is struck off, and then the cylinder is
lifted to allow the material to spread onto a nonporous surface. The diameter of the spread is immediately measured in two perpendicular directions. The flow consistency is typically reported as the average of the diameters (in.). Both measurements were reported for this study.

### 2.4.2 Density

Understanding density of rapid-setting flowable fill is important for any applications where density can be a limiting factor. The density (unit weight) was determined for freshly mixed flowable fill in the laboratory according to ASTM D 6023 (2016a), which is based on C 138 (ASTM 2014a) for concrete. The density was determined by filling a 0.25-ft³ air meter container with material, consolidating the material by vibration, striking the material off, and weighing the container. The density is expressed in lb/ft³.

### 2.4.3 Air content

The air content was determined by the pressure method in accordance with ASTM C231 (2008b). The fresh flowable fill material was first placed in a standard air-content test device and prepared using the same procedure as that mentioned for density testing. The top of the air-content test device was latched onto the base of the device. The device was then pressurized until the meter had zeroed out. Once the pressure was allowed to stabilize, the pressure was then released, and the air-void content was read from the dial atop the device.

### 2.4.4 Cement content

Typically, the cement content of the CLSM may be calculated in accordance with ASTM D 6023 (2016a), which calculates the cement content by dividing the mass of cement placed into a batch of freshly mixed material by the volume of CLSM produced per batch. This method would suffice if the CLSM was proportioned entirely in-house. However, the CLSM evaluated herein is a manufactured product, and the cement content had to be verified using another technique. The technique chosen to verify the cement content of the pre-packaged material was using a sieve analysis per ASTM C 136 (2014b). In order to comply with ASTM C 136, a 50-lb bucket of dry flowable fill was blended in order to obtain a sample representative of the proportions of all constituent materials present in the bucket. Afterwards, 300 g of the dry material was placed
into a standard set of fine aggregate sieves until 1% or less by mass of the material remaining on any sieve passed that sieve during 1 min of hand sieving. Cementitious material is finer than the majority of the fine aggregate in the prepackaged blend of materials and will pass through all sieves in the set. As a result, the material that passed the #200 sieve was weighed and reported as weight of cementitious material. The weight of the material that collected in the pan during sieving was then taken as a percentage of the overall mass of the material prior to being sieved. That percentage was found to be the cement content.

2.5 X-ray fluorescence and diffraction

X-ray fluorescence (XRF) and x-ray diffraction (XRD) are routinely used by the cement industry as a means of identifying and controlling the composition of raw materials that are used to produce clinker and cement. XRF works by exciting the material using gamma rays and reports any of the approximately 150 elements of interest, provided 0.01% or more is present. XRD is a rapid analytical technique primarily used for phase identification of crystalline materials. Both XRF and XRD were conducted on the cement remaining from each of the 10 cement content tests described in the previous section.

Wavelength Dispersive X-Ray Fluorescence (WDXRF) was performed on each sample to identify the chemical composition of the compounds present in the cementitious material. The loss of ignition was first determined by weighing 1 g (1 ± 0.001) of each cement sample in a platinum crucible and placing it inside a muffle furnace at 950°C for 2 hr to undergo the ignition process. After the ignition, the platinum crucibles were placed inside a desiccator for 1 hr until the crucibles cooled down. Finally, the platinum crucibles were weighed, and the mass loss during ignition was calculated according to ASTM C114 (2015c). Each sample was conditioned by washing the surface that faced the x-ray tube with methanol. The loss-on-ignition value and amount of flux were entered into the software interface of the WDXRF, and the sample was run for analysis. Once completed, all major oxide compositions and spectra were analyzed to verify no overlap was present.

XRD analysis was performed on each bulk sample to identify the phases present in the material. The samples were sieved over a #200 (74 µm) sieve to remove sand and aggregates from the cement. Once the aggregates were removed, samples were prepared into random powder
pack sample holders for XRD measurements. Diffraction patterns to be used for qualitative phase identification were obtained by using a Panalytical X’Pert Pro materials research diffractometer equipped with a Co-Kα X-ray source operated at 45 kV and 40 mA. The run conditions included (1) Co-Kα radiation, scanning from 2 to 70 °2θ with a step size less than 0.02 °2θ at a rate of approximately 0.5 °2θ/min, (2) collection of the diffraction patterns, accomplished by using the PC-based Windows version of X-Pert Pro Data Collector, and (3) analysis of the patterns using the Jade2010 program. Whole pattern fits of the sample provided semi-quantitative analysis of the material.

2.6 Time of setting

Working time for the materials in this study was defined as the elapsed time between the initial contact of water with the repair material and the initial set of the material. Because of the short working times associated with rapid-setting cementitious materials, it becomes an important consideration in selecting a material to accomplish a repair. Testing was accomplished in accordance with ASTM C403 (2006). After the material was mixed in accordance with the testing plan, the mortar was placed in cylinders with a diameter of 6 in. by a height of 6 in. Periodic tests were performed on this mortar by penetrating a needle of known bearing area vertically downward into the mortar 1 in. and recording the force produced. The penetration resistance was calculated by dividing the recorded force by the bearing area of the needle. The initial time of setting resulted when the penetration resistance equaled 500 psi, and the final time of setting was when the penetration resistance equaled 4,000 psi. Time of setting was reported in minutes.

2.7 Modulus of elasticity

Modulus of elasticity is an important design parameter that is often used by pavement engineers when designing pavement structures. Modulus of elasticity testing was accomplished in accordance with ASTM C 469 (2002) procedures using 4-in.-diam by 8-in.-high cylinders. An unbounded sensing device was attached to the cylinder at midheight for measuring vertical deformation. The modulus of elasticity (psi) was calculated as change in stress divided by change in strain, where strain was calculated as vertical deformation divided by the gage length. The calculation produced a chord modulus of elasticity where the cord line is drawn between the stress at 50 millistrain and the strain at 40% of the
compressive strength. Tests were conducted at the same intervals as flexural strength testing, since 1-hr specimens did not gain enough strength to be tested. Curing and capping methods for cylinders are addressed in Section 2.10.

2.8 Volumetric expansion or contraction

Excessive expansion or contraction of backfill materials can result in cracking or settlement of the crater repair surface. Also, for larger size crater repairs, excessive expansion of the backfill material can result in the deterioration of the surrounding pavements. Thermal expansion is the tendency of matter to change in volume in response to the change in temperature through heat transfer. It is important to know the thermal response of the material to know of possible distress.

2.8.1 Coefficient of thermal expansion

Coefficient of thermal expansion is important, since a backfill material with a coefficient of thermal expansion considerably greater than the parent material will exhibit greater volume changes with changes in temperature. Differential movements for the backfill material versus the parent material or capping material tend to deteriorate the bond between the two materials and cause pavement distresses. Coefficient of thermal expansion testing was conducted in accordance with ASTM C 531 (2012). These procedures involved the production of mortar bars (1 in. by 1 in. by 11.25 in. long) with metal studs on each end. The studs were used for length measurements. Linear expansion/contraction was measured with a comparator daily for two weeks with bars stored at 73°F, then after 3 days at 210°F, and finally after being cooled overnight at 73°F. This heating and cooling cycle was repeated until a constant length was measured. The coefficient of thermal expansion was calculated as strain per degree F, and reported in units in./in./°F.

2.8.2 Length change

Since volumetric expansion of repair materials can result from causes other than applied force or temperature changes, testing for expansion due to internal forces was conducted according to ASTM C 157 (2008a) procedures. If the rapid-setting flowable fill material experiences length changes greater than +0.03% (expansion) or less than -0.04% (contraction), deterioration of the bond with the capping material can result, which can cause distresses to become apparent in the crater.
repair surface. Test specimens were 3-in. by 3-in. by 11.25-in.-long prisms with metal gage studs embedded into each end to facilitate length change measurements. The prisms were demolded at 23.5 hr and soaked in lime-saturated water for 30 min before an initial length measurement was recorded. All specimens were stored in lime-saturated water at 73°F for 28 days. At the end of the curing period, a second comparator reading was taken. Specimens were then stored both in the lime-water and in an air storage drying room at 73°F and 50% relative humidity. Comparator readings after curing were taken at 4, 7, 14, and 28 days then at 8, 16, 32, and 64 weeks. The length change at each age was calculated as a percentage (change in length/original length)/ (gage length) × 100%.

2.9 Methods of curing and capping

The methods used to cure and cap the laboratory specimens are described in the following paragraphs. These methods are important considerations for purposes of accomplishing accurate and consistent testing, since the methods used can affect the results.

Curing affects many of the tests described above and reflects laboratory curing as recommended by the repair material’s manufacturer. All specimens were air cured at 73°F and 50% relative humidity to represent a less than ideal curing environment. To reflect field performance, curing duration for this report was the time elapsed from the final finishing of a test specimen to the time of testing.

Capping affects compressive strength, bond strength, and modulus of elasticity testing. Capping was required in order to provide flat ends that were perpendicular to the sides of the cylinders to ensure proper loading during testing. Capping was accomplished with either bonded caps (ASTM 2015b) or unbonded pad caps (ASTM 2015a). For this protocol, compressive strength and bond strength testing were accomplished with unbonded neoprene pad caps. Modulus of elasticity testing required the use of bonded caps.

2.10 Replicates

Three replicates were required for each of the tests described in this report, except where exceptions are noted. Three replicates provide a small measure of repeatability, which is imperative when considering
rapid-setting materials. The average result, calculated from the three replicates, is reported in Chapter 3 of this report.
3 Test Results and Analysis

3.1 Strength and modulus results

Table 1 displays the compressive and flexural strength results at both the high and low w/p ratios. For flexural strength testing, specimens cured for 1 hr did not gain sufficient strength for testing at either w/p ratio. As expected, a considerable decrease in compressive strength was observed when the higher w/p ratio was used. An unexpected decrease in the flexural strength was reported on 28 days in both the low and high w/p materials. Furthermore, the flexural strengths at the high w/p ratio were greater than those at the low w/p ratio. A second set of tests was conducted, and both results showed similar strengths and the same pattern of strength loss at later ages. This could be explained as a possible phase transformation occurring in the material over time.

<table>
<thead>
<tr>
<th>Cure Time</th>
<th>Low w/p ratio</th>
<th>High w/p ratio</th>
<th>Low w/p ratio</th>
<th>High w/p ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 hr</td>
<td>430</td>
<td>253</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>2 hr</td>
<td>830</td>
<td>477</td>
<td>181</td>
<td>177</td>
</tr>
<tr>
<td>1 day</td>
<td>1,467</td>
<td>1,035</td>
<td>288</td>
<td>302</td>
</tr>
<tr>
<td>28 days</td>
<td>1,657</td>
<td>1,077</td>
<td>222</td>
<td>255</td>
</tr>
</tbody>
</table>

Each result presented is an average of three tests.

Modulus of elasticity results are displayed in Table 2. As shown, the modulus values for both w/p ratios were very similar at 2 hr of cure time, but the effects of an increased w/p ratio were more noticeable at later cure times. These modulus values could be useful if rapid-setting flowable fill is used in a pavement design, but clearly some field information concerning the w/p ratio to be used is needed. In general, a conservative estimate of flowable fill modulus would be $1 \times 10^6$ psi at any cure time within the tested of w/p ratios tested.
Table 2. Modulus of elasticity results.

<table>
<thead>
<tr>
<th>Cure time</th>
<th>Low w/p ratio (psi)</th>
<th>High w/p ratio (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 hr</td>
<td>$1.25 \times 10^6$</td>
<td>$1.32 \times 10^6$</td>
</tr>
<tr>
<td>1 day</td>
<td>$1.73 \times 10^6$</td>
<td>$1.35 \times 10^6$</td>
</tr>
<tr>
<td>28 days</td>
<td>$1.96 \times 10^6$</td>
<td>$1.42 \times 10^6$</td>
</tr>
</tbody>
</table>

*Only two replicates used for this combination

3.2 Basic control parameter results

Flow consistency, density, and air content results are provided in Table 3. Workability is clearly improved with an increasing w/p ratio, since the flow consistency spread was considerably increased. This agrees with field observations reported in Carruth and Howard (2016), which describes the use of a higher w/p ratio to increase workability when placing large volumes of flowable fill at moderately high ambient field temperatures.

Limited literature is available on air content for flowable fill materials, but the expected air content was anticipated to increase as the water content increased. However, three separate, independent air content tests were conducted in accordance to the pressure methods described in ASTM C231 (2008b), and the average low w/p air content was 17% while the average high w/p air content was 4%. However, visual observations of companion 3-in.-diam x 6-in.-high cylinders showed clear air voids in the low w/p ratio specimens as compared to the high w/p ratio specimens. This would be consistent with a high air content reported on the low w/p ratio and a low air content reported with the high w/p ratio. Figure 3 and Figure 4 document these observations. It is possible that the increased water took up space that normally would be taken up by air.

Griffin and Brown (2011) tested many different flowable fill mixtures, and the freshly mixed density results ranged from 102 to 132 lb/ft$^3$. The results obtained for the rapid-setting flowable fill tested in this study fall in the middle of that range. However, it is somewhat unexpected that the two w/p ratios would result in very similar density values. Three separate, independent tests were conducted comparing the densities, and all tests showed consistency. The similarities in the densities could be due to the balance of the high air content in the low volume of water ratio mix compared to the low air content and high volume of water ratio mix.
Figure 3. Low w/p ratio 3-in.-diam by 6-in.-high cylinders.

Figure 4. High w/p ratio 3-in.-diam by 6-in.-high cylinders.
Table 3. Basic control parameter test results.

<table>
<thead>
<tr>
<th>Test Method</th>
<th>ASTM</th>
<th>Low w/p Ratio</th>
<th>High w/p Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow Consistency</td>
<td>D6103</td>
<td>8.0 by 8.0</td>
<td>16 by 16</td>
</tr>
<tr>
<td>(in. by in.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fresh Density</td>
<td>D6023</td>
<td>116</td>
<td>119</td>
</tr>
<tr>
<td>(pcf)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Air Content (%)</td>
<td>C231</td>
<td>17.0</td>
<td>4.0</td>
</tr>
</tbody>
</table>

3.3 Cement content results

Cement content tests were conducted on 10 randomly selected buckets of Utility Fill One-Step 750® material from the same lot in order to establish a range of cement contents that is to be expected. These results are displayed in Table 4. As shown, a considerable range (9.99%) and standard deviation (3.31%) were observed between buckets. The reason for the high range of values is believed to be the manner in which buckets are filled by the manufacturer. Previous full-scale tests and demonstrations reported no noticeable changes in set time or strength between 3,000-lb super sacks of dry material.

Table 4. Cement content test results.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement Content</td>
<td>8.07</td>
<td>5.37</td>
<td>9.63</td>
<td>7.60</td>
<td>9.89</td>
<td>7.84</td>
<td>15.36</td>
<td>7.22</td>
<td>6.57</td>
<td>14.54</td>
</tr>
</tbody>
</table>

Note: Average = 9.21%, Standard Deviation = 3.31%, Range = 9.99%

3.4 XRF results

The bulk chemistry composition of 10 randomly selected buckets of material from the same lot was analyzed by using wavelength dispersive X-ray fluorescence (WDXRF). Average amounts of each compound are shown in Table 5. The results of each of the 10 tests can be found in Appendix A. The results show consistency among all tests with low standard deviations and ranges. The percentages of compounds are as expected with a predominately manufactured pre-blended calcium sulfoaluminate (CSA) cementitious material.
Table 5. XRF results for rapid-setting flowable fill.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Average Weight %</th>
<th>Standard Deviation</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>12.89</td>
<td>0.65</td>
<td>1.99</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>18.20</td>
<td>1.02</td>
<td>2.93</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>1.81</td>
<td>0.11</td>
<td>0.35</td>
</tr>
<tr>
<td>CaO</td>
<td>46.24</td>
<td>1.33</td>
<td>4.05</td>
</tr>
<tr>
<td>MgO</td>
<td>1.40</td>
<td>0.11</td>
<td>0.28</td>
</tr>
<tr>
<td>SO₃</td>
<td>15.26</td>
<td>0.86</td>
<td>2.62</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.43</td>
<td>0.01</td>
<td>0.04</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.15</td>
<td>0.01</td>
<td>0.05</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.18</td>
<td>0.02</td>
<td>0.05</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.66</td>
<td>0.05</td>
<td>0.18</td>
</tr>
<tr>
<td>Mn₂O₃</td>
<td>0.06</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td>SrO</td>
<td>0.09</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>ZnO</td>
<td>0.03</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>0.05</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>Equivalent Alkalis as Na₂Oeq</td>
<td>0.44</td>
<td>0.01</td>
<td>0.05</td>
</tr>
<tr>
<td>Loss on Ignition</td>
<td>2.53</td>
<td>0.22</td>
<td>0.70</td>
</tr>
</tbody>
</table>

3.5 XRD results

Ten samples were examined to ensure mineralogy of the cement was consistent between buckets. In Figure 5, all ten x-ray patterns can be seen with the minerals identified. Figure 6 is sample 1 with the d-spacings and minerals identified. Table 6 is a summary of the weight percentages of the minerals identified in the whole pattern fits for each sample. The highest weight percentages of minerals are anhydrite (42.79%), followed by yeelimite (23.1%), then larnite (15.68%). This was to be expected as this is predominately manufactured pre-blended CSA cementitious material. The average percentage of quartz (2.92%) is possibly due to trace amounts of the aggregate. While most of the samples showed consistency, it is observed that some of the samples showed a considerable range in the weight percentage of anhydrite (notably sample 6 and sample 9). The weight percentages for all samples can be found in Appendix A.
Figure 5. X-ray pattern for all 10 samples.
Figure 6. X-ray pattern with d-spacings for sample 1 only.
### Table 6. XRD summary of mineral weight percentages.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Average Weight %</th>
<th>Standard Deviation</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anhydrite</td>
<td>42.79</td>
<td>4.16</td>
<td>12.7</td>
</tr>
<tr>
<td>Bassanite</td>
<td>3.5</td>
<td>2.28</td>
<td>7.2</td>
</tr>
<tr>
<td>Gypsum</td>
<td>0.49</td>
<td>0.58</td>
<td>1.9</td>
</tr>
<tr>
<td>Yeelimite (C₄A₃S)</td>
<td>23.1</td>
<td>2.54</td>
<td>9.2</td>
</tr>
<tr>
<td>Larnite (C₂S)</td>
<td>15.68</td>
<td>3.42</td>
<td>10.6</td>
</tr>
<tr>
<td>Alite (C₃S)</td>
<td>8.33</td>
<td>2.10</td>
<td>8.0</td>
</tr>
<tr>
<td>Calcite</td>
<td>3.18</td>
<td>1.19</td>
<td>4.1</td>
</tr>
<tr>
<td>Quartz</td>
<td>2.92</td>
<td>0.88</td>
<td>2.7</td>
</tr>
</tbody>
</table>

### 3.6 Bond strength results

Bond strength results are shown in Table 7. Bond strength tests were conducted on Utility Fill One-Step 750® bonded to itself and Utility Fill One-Step 750® to rapid-setting concrete for reasons discussed previously in Section 2.3.3 of this report. There is currently no minimum requirement for bond strength of rapid-setting flowable fill material. These tests were conducted to characterize the bond strength in general and to observe changes between 1 and 7 days of cure time and at varying w/p ratios.

### Table 7. Bond strength results.

<table>
<thead>
<tr>
<th>Material</th>
<th>Cure time (d)</th>
<th>Low w/p ratio</th>
<th>High w/p ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>FF/FF</td>
<td>1</td>
<td>655</td>
<td>235</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>780</td>
<td>350</td>
</tr>
<tr>
<td>RS/FF</td>
<td>1</td>
<td>525</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>620</td>
<td>520</td>
</tr>
</tbody>
</table>

As shown, the bond strength for rapid-setting flowable fill bonded to itself (FF/FF) was higher than the rapid-setting concrete to rapid-setting flowable fill (RS/FF) for the low w/p ratio, but the opposite was true for the high w/p ratio. Also, the decrease in bond strength with increasing w/p ratio was more dramatic for FF/FF than for RS/FF. The bond strength did
continue to increase between 1 and 7 days of curing, but all 1-day bond strengths were at least 65% of their corresponding 7-day bond strengths.

3.7 Time of set results

The time of set results are displayed in Table 8. Both the initial and final set times were higher for the high w/p ratio, as expected. The initial set requirement for using flowable fill as a backfill for crater repairs is 15 min for the dry method (low w/p ratio) and 30 min for the wet method (high w/p ratio). The laboratory results shown met this requirement. Final set is not required until the overlying capping material has cured for 120 min, so the laboratory final set times observed easily met this requirement.

<table>
<thead>
<tr>
<th>w/p ratio</th>
<th>Set time</th>
<th>Initial (min)</th>
<th>Final (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>low</td>
<td>14</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>high</td>
<td>24</td>
<td>40</td>
<td></td>
</tr>
</tbody>
</table>

3.8 Coefficient of linear thermal expansion results

Expansion problems are known to occur with some rapid-setting repair materials; so it is recommended that rapid-setting flowable fill has a coefficient of thermal expansion close to that of conventional PCC pavements. Excessive expansion will result in the loss of bond to the parent material and any overlying material. The coefficient of linear thermal expansion upper limit is typically 8 to 12 × 10^{-6} in./in./ºF for normal PCC. The current requirement is for a rapid-setting concrete to have a value of ≤12 × 10^{-6} in./in./ºF. The results of the rapid-setting flowable fill testing indicated linear thermal expansion values of 4.26 × 10^{-6} in./in./ºF for the low w/p ratio and 3.49 × 10^{-6} in./in./ºF for the high w/p ratio. Both of these values were well below the limits suggested for normal PCC and the current requirement for rapid-setting concrete. The results were intuitive, since the larger amount of expansion was observed in the lower w/p ratio. Since more cement is available for hydration for the low w/p ratio, there is more opportunity for the material to expand.
3.9 Length change results

Volumetric expansion of repair materials can result from forces other than temperature changes; therefore, testing for expansion due to internal forces was conducted according to ASTM C 157 (2008a) procedures. The upper limit for repair materials is +0.03% (expansion), and the lower limit is -0.04% (contraction). Following ASTM C 157 procedures, test specimens were prismatic bars with dimensions based on maximum aggregate size. The bars were then cured in either water at 73°F or air at 73°F with a 50% relative humidity. Specimens were removed from the molds 2.5 to 2.75 hr after the addition of mixing water. Initial observations of length were made 3 to 3.25 hr after the addition of mixing water according to ASTM C 928 standard (2013b). The length change at each age was calculated as a percentage (change in length/original length × 100%). Readings were at 4, 7, 14, and 28 days and after 8, 16, 32, and 64 weeks.

Results are displayed in Table 9. The results indicate that none of the average length change results approached either the upper (expansion) or lower (contraction) bounds described in the previous paragraph. Also, as with coefficient of linear thermal expansion testing described in the previous section, the results were intuitive in that the larger values of expansion and contraction were observed for the lower w/p ratio.

<table>
<thead>
<tr>
<th>Cure Conditions</th>
<th>Low w/p ratio</th>
<th>High w/p ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>In water</td>
<td>0.0153</td>
<td>0.0075</td>
</tr>
<tr>
<td>In air</td>
<td>-0.0060</td>
<td>-0.0034</td>
</tr>
</tbody>
</table>

Note: Upper limit = 0.03% Lower Limit = -0.04%
4 Conclusions and Recommendations

The primary objective of the testing described herein was to fully evaluate the properties of Utility Fill One-Step 750® by using a suite of laboratory tests based on those developed for certifying rapid-setting concrete materials (Priddy 2011). Since this product has exhibited excellent performance during full-scale testing, the results described herein can be used as a starting point in the development of a laboratory testing protocol to down-select other rapid-setting flowable fill products for further evaluation. Tests were completed and results provided previously in Chapter 3. The following sections provide the conclusions and recommendations that were developed from testing.

4.1 Conclusions

- Compression strength results indicated that the material gained strength rapidly within the first 2 hr for both w/p ratios. After one day of curing, compressive strength had reached over 1,000 psi for both w/p ratios, and compressive strength increased slightly from 1 day to 28 days.
- As expected, the low w/p ratio produced a much higher compressive strength at early curing stages than the high w/p ratio.
- The material had not achieved sufficient strength for a flexural strength test to be conducted at 1 hr of curing time.
- Flexural strength test results were not intuitive. Results were similar for both w/p ratios after 2 hr of cure time, but the high w/p ratio exhibited a higher flexural strength after 1 day and 28 days of curing.
- Modulus results were similar for both w/p ratios at only 2 hr of cure time, but the modulus for the lower w/p ratio was higher after 1 day and 28 days of curing.
- Flow consistency for the higher w/p ratio was much higher than for the low w/p ratio, as expected.
- A higher density and lower air content were observed for the higher w/p ratio, which was somewhat unexpected. Air voids were visually apparent for the low w/p ratio but not for the high w/p. The increase in water could have enabled the high w/p ratio material to fill this air space, resulting in an increase in density.
- Cement content tests on ten separate buckets of material showed a considerably high range and standard deviation. These results are
believed to have been caused by the method in which the manufacturer filled the buckets.

- XRF results showed a small range and standard deviation of the various chemical compounds detected.
- XRD results indicated that most of the minerals detected were consistent between tests with the exception of anhydrite, which exhibited a considerable range.
- Both the XRF and XRD results were typical for that of a calcium-sulfoaluminate (CSA)-based cementitious material.
- Bond strength was greater for the lower w/p ratio and increased with cure time for both the low and high w/p ratios.
- Both initial and final set times were lower for the low w/p ratio, as expected. Both initial set times were under 30 min.
- The low w/p ratio produced a higher coefficient of thermal expansion than the high w/p ratio, but both were well below the requirement used for rapid-setting concrete materials.
- The lower w/p ratio exhibited slightly more expansion and contraction during length change tests than the high w/p ratio, but none of the results were close to the limits imposed for rapid-setting concrete materials.
- In general, strength properties were higher for the low w/p ratio compared to the high w/p ratio, but workability was reduced, as expected. Laboratory results concur with the high-quality performance observed for rapid-setting flowable fill during numerous full-scale field tests and troop demonstrations.
- Overall, the flexural strength and air content results were the only results that were somewhat questionable. None of the other results disputed the well-documented high performance of rapid-setting flowable fill in full-scale testing.

## 4.2 Recommendations

- Development of a formalized protocol of laboratory tests using the results described in this report as a starting point is recommended so that other rapid-setting flowable fill products can be down-selected in the laboratory before moving on to further evaluation via full-scale testing. The flexural strength test procedure requires additional investigation before it can be included in a formal protocol.
- Further investigation is recommended to study the microstructural characterization of the material over time to determine possible
phase transformations at later ages. It is speculated that the strength decrease in the flexural beams is attributed to this reason.

- Other w/p ratios should be investigated to address the abnormalities observed for density and air content.
- When material is obtained for laboratory testing, it is recommended to instruct the manufacturer to fill buckets directly from the processing equipment in lieu of sampling from larger packages of material.
- Additional methods for reproducing the dry method in the laboratory should be investigated in order to accurately represent the placement process as much as possible.
References


## Appendix A: XRF and XRD Full Results

Table A1. XRF chemical composition results for all tests.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Test 1</th>
<th>Test 2</th>
<th>Test 3</th>
<th>Test 4</th>
<th>Test 5</th>
<th>Test 6</th>
<th>Test 7</th>
<th>Test 8</th>
<th>Test 9</th>
<th>Test 10</th>
<th>Avg</th>
<th>SD</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃ (%)</td>
<td>18.83</td>
<td>18.69</td>
<td>16.21</td>
<td>18.84</td>
<td>17.79</td>
<td>18.96</td>
<td>16.34</td>
<td>18.6</td>
<td>19.14</td>
<td>18.6</td>
<td>18.20</td>
<td>1.02</td>
<td>2.93</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>1.74</td>
<td>1.78</td>
<td>2</td>
<td>1.76</td>
<td>1.86</td>
<td>1.72</td>
<td>2.05</td>
<td>1.77</td>
<td>1.7</td>
<td>1.76</td>
<td>1.81</td>
<td>0.11</td>
<td>0.35</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>45.49</td>
<td>45.66</td>
<td>48.41</td>
<td>45.64</td>
<td>47.06</td>
<td>45.13</td>
<td>48.92</td>
<td>45.75</td>
<td>44.87</td>
<td>45.5</td>
<td>46.24</td>
<td>1.33</td>
<td>4.05</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>1.48</td>
<td>1.24</td>
<td>1.45</td>
<td>1.24</td>
<td>1.42</td>
<td>1.5</td>
<td>1.42</td>
<td>1.24</td>
<td>1.52</td>
<td>1.49</td>
<td>1.40</td>
<td>0.11</td>
<td>0.28</td>
</tr>
<tr>
<td>SO₃ (%)</td>
<td>15.59</td>
<td>16.07</td>
<td>14.04</td>
<td>16.09</td>
<td>14.69</td>
<td>15.56</td>
<td>13.47</td>
<td>16</td>
<td>15.73</td>
<td>15.36</td>
<td>15.26</td>
<td>0.86</td>
<td>2.62</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.42</td>
<td>0.45</td>
<td>0.42</td>
<td>0.45</td>
<td>0.41</td>
<td>0.42</td>
<td>0.45</td>
<td>0.42</td>
<td>0.42</td>
<td>0.42</td>
<td>0.43</td>
<td>0.01</td>
<td>0.04</td>
</tr>
<tr>
<td>Na₂O (%)</td>
<td>0.16</td>
<td>0.13</td>
<td>0.16</td>
<td>0.13</td>
<td>0.14</td>
<td>0.16</td>
<td>0.16</td>
<td>0.18</td>
<td>0.16</td>
<td>0.15</td>
<td>0.15</td>
<td>0.01</td>
<td>0.05</td>
</tr>
<tr>
<td>P₂O₅ (%)</td>
<td>0.19</td>
<td>0.15</td>
<td>0.18</td>
<td>0.16</td>
<td>0.17</td>
<td>0.19</td>
<td>0.18</td>
<td>0.15</td>
<td>0.2</td>
<td>0.19</td>
<td>0.18</td>
<td>0.02</td>
<td>0.05</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.67</td>
<td>0.72</td>
<td>0.54</td>
<td>0.72</td>
<td>0.64</td>
<td>0.67</td>
<td>0.61</td>
<td>0.72</td>
<td>0.67</td>
<td>0.66</td>
<td>0.66</td>
<td>0.05</td>
<td>0.18</td>
</tr>
<tr>
<td>Mn₂O₃ (%)</td>
<td>0.05</td>
<td>0.06</td>
<td>0.07</td>
<td>0.06</td>
<td>0.06</td>
<td>0.06</td>
<td>0.06</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td>0.06</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td>SrO (%)</td>
<td>0.10</td>
<td>0.09</td>
<td>0.09</td>
<td>0.09</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.09</td>
<td>0.10</td>
<td>0.09</td>
<td>0.09</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>ZnO (%)</td>
<td>0.034</td>
<td>0.026</td>
<td>0.035</td>
<td>0.026</td>
<td>0.033</td>
<td>0.034</td>
<td>0.033</td>
<td>0.026</td>
<td>0.034</td>
<td>0.034</td>
<td>0.03</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>Cr₂O₃ (%)</td>
<td>0.055</td>
<td>0.05</td>
<td>0.05</td>
<td>0.049</td>
<td>0.051</td>
<td>0.051</td>
<td>0.049</td>
<td>0.05</td>
<td>0.051</td>
<td>0.05</td>
<td>0.05</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>Equivalent Alkalis as Na₂Oeq (%)</td>
<td>0.44</td>
<td>0.43</td>
<td>0.44</td>
<td>0.43</td>
<td>0.41</td>
<td>0.44</td>
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<td>2.41</td>
<td>2.27</td>
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<td>2.53</td>
<td>0.22</td>
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</table>

*Average of all 10 tests*
Table A2. XRD mineral weight percentages for all tests.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Anhydrite</th>
<th>Bassanite</th>
<th>Gypsum</th>
<th>Yeelimite (Ca₃S)</th>
<th>Larnite (CaS)</th>
<th>Alite (CaS)</th>
<th>Calcite</th>
<th>Quartz</th>
</tr>
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<tr>
<td>1</td>
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<td>0.6</td>
<td>25.8</td>
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<td>4.7</td>
<td>1.3</td>
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<td>2</td>
<td>45.1</td>
<td>2.1</td>
<td>0.0</td>
<td>23.8</td>
<td>12.8</td>
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<td>3.2</td>
<td>3.6</td>
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<td>16.3</td>
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<td>1.1</td>
<td>12.1</td>
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<td>0.1</td>
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<td>1.4</td>
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<td>5</td>
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<td>0.7</td>
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<td>3.6</td>
<td>1.4</td>
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<td>7</td>
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<td>0.8</td>
<td>22.7</td>
<td>23.4</td>
<td>12.1</td>
<td>5.2</td>
<td>1.6</td>
</tr>
<tr>
<td>8</td>
<td>47.4</td>
<td>2.5</td>
<td>0.0</td>
<td>23.5</td>
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<td>6.7</td>
<td>3.7</td>
<td>1.5</td>
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<td>9</td>
<td>70.7</td>
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<td>0.0</td>
<td>14.0</td>
<td>7.7</td>
<td>4.1</td>
<td>1.7</td>
<td>0.9</td>
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<tr>
<td>10</td>
<td>45.1</td>
<td>2.0</td>
<td>0.8</td>
<td>23.9</td>
<td>13.0</td>
<td>8.5</td>
<td>3.4</td>
<td>3.3</td>
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<td>Average (weight)</td>
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<td>15.68</td>
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</table>
Figure A1. XRD whole pattern fit - sample 1.

Scan ID: 160109-S1V1.xaml

Scan Parameters: 2.511*69.99°/0.01671*219.71(s), e=1.255678°/34.99573°, e=0.0°, f(p)=3307.0690, Co(45kV,40mA), Friday, J

Generator=PW3050/60 (Theta/Theta). Minimum step size 2Theta:0.001; Minimum step size Omega:0.001
Sample stage=Reflection Transmission Spinner: PW3050/60; Minimum step size Phi:0.1
Diffractometer system=XPERT-PRO
Measurement program=Chris 2.79 scan time 2h, Owner=Ueyasu, Creation date=5/23/2011 8:43:04 AM

☑️ Kα2 Peaks Present
☐ Variable-Slit Pattern
☐ Displacement = 0.0
☐ Kα2/α Ratio = 0.5

Geometry: Diffractometer LP Fitted-Range: 2.5° - 70.0° BG-Model: Polynomial (7) λ: 1.78899 Å (Co)

PSF: Pearson-VII Broadening Individual FWHM Curve Instrument: Constant FWHM = 0.1°

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<thead>
<tr>
<th>Phase ID</th>
<th>Chemical Formula</th>
<th>PDF-#</th>
<th>NA</th>
<th>NR</th>
<th>NP</th>
<th>Wt% (asd)</th>
<th>RIR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anhydrite</td>
<td>CaSO4</td>
<td>01-072-0616</td>
<td>24</td>
<td>12</td>
<td>25.8</td>
<td>0.6</td>
<td>7.77</td>
</tr>
<tr>
<td>Yeelimite</td>
<td>Ca4Al2(OH)12(SO4)</td>
<td>99-000-0467</td>
<td>5</td>
<td>52</td>
<td>10</td>
<td>25.8</td>
<td>0.3</td>
</tr>
<tr>
<td>Larnite</td>
<td>Ca2SiO4</td>
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<td>78</td>
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<td>12</td>
<td>4.7</td>
<td>2.60</td>
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<tr>
<td>Alite Monoclinic CJS</td>
<td>Ca3SiO5</td>
<td>99-000-043</td>
<td>155</td>
<td>621</td>
<td>13</td>
<td>7.0</td>
<td>0.3</td>
</tr>
<tr>
<td>Calcite</td>
<td>CaCO3</td>
<td>00-005-0565</td>
<td>11</td>
<td>12</td>
<td>6</td>
<td>3.7</td>
<td>2.60</td>
</tr>
<tr>
<td>Quartz</td>
<td>SiO2</td>
<td>01-070-0388</td>
<td>12</td>
<td>6</td>
<td>13</td>
<td>1.3</td>
<td>3.04</td>
</tr>
<tr>
<td>Bassanite</td>
<td>CaSO4.10H2O</td>
<td>98-000-0108</td>
<td>24</td>
<td>288</td>
<td>8</td>
<td>1.8</td>
<td>0.2</td>
</tr>
<tr>
<td>Gypsum</td>
<td>CaSO4.2H2O</td>
<td>00-003-0311</td>
<td>38</td>
<td>8</td>
<td>6.6</td>
<td>0.6</td>
<td>1.83</td>
</tr>
</tbody>
</table>

XRF(Wt%): Ca=15.9%, S03=30.6%, SiO2=6.3%, Al2O3=12.9%, CO2=2.0%

Refinement halted (R/E=1.39), m=Round=4, Iter=8, P=1, R=7.47%, E=5.36%, EPS=0.5

Observed Pattern

WP Fitted Pattern

160109-S1V1

C:\ WD0337ER\G0Desktop\160109 Batch\Samples\160109-S1V1.xaml

Wednesday, July 08, 2015, 11:50 AM • US Army Corps Engineers
Figure A2. XRD whole pattern fit - sample 2.
Figure A3. XRD whole pattern fit - sample 3.
Figure A4. XRD whole pattern fit - sample 4.

Scan ID: 160109-S4V1.xml

Scan Parameters: 25117/69.99/0.01671/219.71(s), w=1.256678*34.99573°, q=0.0°, l(p)=4433.0/101.0, Co(45kV,40mA), Friday.

Geometry: Diffractometer Lp

Fitted-Range: 2.5 - 70.0°

BG-Model: Polynomial (7)

λ: 1.78809 Å (Co)

PSF: Pearson-VII

Broadening: Individual FWHM Curve

Instrument: Constant FWHM = 0.1°

<table>
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<th>Chemical Formula</th>
<th>PDF-#</th>
<th>NA</th>
<th>NR</th>
<th>NP</th>
<th>Wt% (est)</th>
<th>R/R</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anhydrite</td>
<td>Ca(SO4)</td>
<td>01-072-0916</td>
<td>---</td>
<td>24</td>
<td>12</td>
<td>47.2 (0.5)</td>
<td>1.77</td>
</tr>
<tr>
<td>Yeelimite</td>
<td>Ca4(Al6O12)(SO4)</td>
<td>98-000-0467</td>
<td>5</td>
<td>52</td>
<td>10</td>
<td>22.7 (0.3)</td>
<td>3.11</td>
</tr>
<tr>
<td>Larnite</td>
<td>Ca2SiO4</td>
<td>01-070-0388</td>
<td>---</td>
<td>76</td>
<td>10</td>
<td>15.3 (0.5)</td>
<td>0.76</td>
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<tr>
<td>Quartz</td>
<td>SiO2</td>
<td>01-075-0443</td>
<td>---</td>
<td>12</td>
<td>4</td>
<td>1.4 (0.1)</td>
<td>3.04</td>
</tr>
<tr>
<td>Alite Monoclin C3S</td>
<td>Ca3SiO5</td>
<td>98-000-0043</td>
<td>155</td>
<td>821</td>
<td>13</td>
<td>8.0 (0.2)</td>
<td>0.77</td>
</tr>
<tr>
<td>Bassanite</td>
<td>CaSO4H2O</td>
<td>98-000-0198</td>
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<td>208</td>
<td>4</td>
<td>2.9 (0.4)</td>
<td>1.19</td>
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<tr>
<td>Calcite</td>
<td>CaCO3</td>
<td>00-005-0586</td>
<td>---</td>
<td>12</td>
<td>8</td>
<td>2.3 (0.1)</td>
<td>2.00</td>
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<tr>
<td>Gypsum</td>
<td>CaSO4H2O</td>
<td>00-033-0311</td>
<td>---</td>
<td>38</td>
<td>3</td>
<td>0.1 (0.0)</td>
<td>1.83</td>
</tr>
</tbody>
</table>

Refinement Converged (R/E=1.38), ∙ Round=4, iter=3, P=69, R=7.39% (E=5.36%, EPS=0.5)

Anhydrite • Ca(SO4)
Yeelimite • Ca4(Al6O12)(SO4)...
Larnite • Ca2SiO4
Alite Monoclin C3S • Ca3SiO5
Bassanite • CaSO4H2O
Calcite • CaCO3
Quartz • SiO2
Gypsum • CaSO4H2O

Observed Pattern
WP Fitted Pattern
160109-S4V1
Figure A5. XRD whole pattern fit - sample 5.

Scan ID: 160109-S5V1.xml

Scan Parameters: 2.511^0/99.99^0/0.01671^0/219.71^0, ω=1.255678^0/34.999573^0, φ=0.0^0, I(p)=3073.0/103.0, Co(45kV,40mA), Friday.

Goniometer=PW2559/65 (Theta/Theta). Minimum step size 2Theta:0.001; Minimum step size Omega:0.001
Sample stage=Reflection Transmission. Spinners PW2559/65. Minimum step size Phi:0.1
Diffractometer system=XPERT-PRO
Measurement program=Chris 2.70 scan time 2h. Owner= User-1, Creation date=8/23/2011 8:43:04 AM

☑ Ka2 Peaks Present
☑ Variable-Slit Pattern
☑ Zero Offset = -0.00181 (0.00467)
☑ Displacement = 0.0
☑ Background = 0.5
☑ X-Ray Polarization = 1.0
☑ Ka2/Ko1 Ratio = 0.5

Geometry: Diffractometer Lp
Fitted-Range: 2.5 - 70.0
BG-Model: Polynomial (7)
λ: 1.78890 Å (Co)

PSF: Pearson-VII
Broadening: Individual FWHM Curve
Instrument: Constant FWHM = 0.1

Phase ID (8)  Chemical Formula  PDF-#  NA  NR  NP  Wt% (sd)  R/R
Anhydrite  Ca(SO4)  01-072-0916  ---  24  12  34.7 (0.8)  1.77
Yeelinite  Ca4(Al6O12)(SO4)  98-000-0467  5  52  10  24.0 (5.5)  3.11
Larnite  Ca2SiO4  01-070-0388  ---  76  13  20.4 (0.8)  0.76
Bassanite  CaSO4·1.5H2O  98-000-0108  24  268  8  6.9 (1.3)  1.19
Alite Monoclinic C3S  Ca3SiO5  98-000-0043  155  821  13  8.3 (0.4)  0.77
Calcite  CaCO3  00-005-0063  ---  12  11  3.6 (0.5)  2.00
Quartz  SiO2  01-075-0443  ---  12  6  1.4 (0.2)  3.04
Gypsum  CaSO4·2H2O  00-033-0311  ---  38  10  0.7 (0.2)  1.83

XRF (%Wt): CaO=47.4%, SiO2=27.7%, Al2O3=10.7%, Fe2O3=12.0%, CO2=1.5%

Refinement Halted (R(E)=1.84). • Round=4, Iter=5, P=52, R=9.91% (E=5.39%, EPS=0.5)

Yield 1.84% 4.7% 9.91%

Anhydrite  Ca(SO4)
Yeelinite  Ca4(Al6O12)(SO4)
Larnite  Ca2SiO4
Bassanite  CaSO4·1.5H2O
Alite Monoclinic C3S  Ca3SiO5
Calcite  CaCO3
Quartz  SiO2
Gypsum  CaSO4·2H2O

Revised 10/10/2011 8:15 PM

Data: 2K06SOURG/DESKTOP/160109 Batch Samples/160109-S5V1.xml

Wednesday, July 04, 2018, 12:17 PM • US Army Corp Engineers
Figure A6. XRD whole pattern fit - sample 6.
Figure A7. XRD whole pattern fit - sample 7.

Scan ID: 160109-S7V1.xml
Scan Parameters: 2.511/59.99°/0.01671°/219.71°(e), um=1.25556°/34.95537°, ϕ=0.0°, Ip=2655.0/100.0, Co(45kV,40mA), Saturday
Configuration=Sample Spinner Reflection Transmission, Owner=EE-1, Creation date=12/23/2009 3:41:59 PM
Goniometer=PW3050/60 (Theta/Theta), Minimum step size 2Theta=0.01°, Minimum step size Omega=0.001°
Sample stage=Reflection Transmission Spinner PW3050/60, Minimum step size Phi=0.1°
Diffractometer system=XPERT-PRO
Measurement program=Chris 2.70 scan time 2h, Owner=EE-1, Creation date=6/23/2011 8:43:04 AM

- Ka2 Peaks Present: True
- Zero Offset: 0.00529 (0.00391)
- X-Ray Polarization: 1.0°
- Ka2/Ka1 Ratio: 0.5

Geometry: Diffractometer Lp
Fitted Range: 2.5 - 70.0°
BG Model: Polynomial (7)
λ: 1.78859 Å (Co)

Phase ID (8) | Chemical Formula | PDF# | NA | NR | NP | W% (est) | R/R |
--- | --- | --- | --- | --- | --- | --- | --- |
Yeelimite | Ca4Al6O12(SO4) | 98-000-0467 | 5 | 52 | 10 | 22.7 (4.4) | 3.13 |
Anhydrite | CaSO4 | 01-072-0916 | --- | 24 | 12 | 28.8 (0.6) | 1.77 |
Larnite | Ca2SiO4 | 01-070-0388 | --- | 76 | 13 | 23.4 (0.8) | 0.76 |
Bassanite | CaSO40.5H2O | 98-000-0108 | 24 | 286 | 10 | 5.3 (0.4) | 2.00 |
Alite Monoclinic CSS | Ca3SiO5 | 98-000-0043 | 155 | 821 | 13 | 12.1 (0.4) | 0.78 |
Calcite | CaCO3 | 00-005-0586 | --- | 12 | 11 | 5.2 (0.5) | 2.00 |
Quartz | SiO2 | 01-075-0443 | --- | 12 | 6 | 1.6 (0.2) | 3.04 |
Gypsum | CaSO4.2H2O | 00-033-0311 | --- | 38 | 13 | 0.7 (0.1) | 1.83 |

XRF (W%): CaO=49.6%, SO3=31.2%, SiO2=13.0%, Al2O3=11.4%, CO2=2.3%

Refinement Halted (R/E=1.57), R=8.53%, RP=8.53%, R=8.53%, RP=8.53%, R=8.53%, RP=8.53%

Anhydrite ● CaSO4
Larnite  ● Ca2SiO4
Yeelimite  ● Ca4Al6O12(SO4)
Alite Monoclinic CSS  ● Ca3SiO5
Bassanite  ● CaSO40.5H2O
Calcite  ● CaCO3
Quartz  ● SiO2
Gypsum  ● CaSO4.2H2O

Observed Pattern
WPFFitted Pattern
160109-S7V1
Figure A8. XRD whole pattern fit - sample 8.

<table>
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<th>Phase ID (8)</th>
<th>Chemical Formula</th>
<th>PDF#</th>
<th>NA</th>
<th>NR</th>
<th>NP</th>
<th>Wt% (esd)</th>
<th>RIR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anhydrite</td>
<td>Ca(SO₄)</td>
<td>01-072-0916</td>
<td>---</td>
<td>24</td>
<td>12</td>
<td>47.4 (0.6)</td>
<td>1.77</td>
</tr>
<tr>
<td>Larnite</td>
<td>Ca₂SiO₄</td>
<td>01-070-0388</td>
<td>---</td>
<td>76</td>
<td>10</td>
<td>14.7 (0.4)</td>
<td>0.76</td>
</tr>
<tr>
<td>Yeelimite</td>
<td>Ca₄<a href="SO%E2%82%84">Al₆O₁₂</a></td>
<td>98-000-0467</td>
<td>5</td>
<td>52</td>
<td>10</td>
<td>23.5 (0.3)</td>
<td>3.11</td>
</tr>
<tr>
<td>Alite Monoclinic CSS</td>
<td>Ca₃SiO₅</td>
<td>98-000-0043</td>
<td>155</td>
<td>821</td>
<td>10</td>
<td>6.7 (0.3)</td>
<td>0.77</td>
</tr>
<tr>
<td>Bassanite</td>
<td>Ca₃SO₄[O₄.5H₂O]</td>
<td>98-000-0108</td>
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<td>268</td>
<td>8</td>
<td>2.5 (0.5)</td>
<td>1.19</td>
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<td>Calcite</td>
<td>CaCO₃</td>
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<td>12</td>
<td>8</td>
<td>3.7 (0.3)</td>
<td>2.00</td>
</tr>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>01-075-0443</td>
<td>---</td>
<td>12</td>
<td>6</td>
<td>1.5 (0.1)</td>
<td>3.04</td>
</tr>
<tr>
<td>Gypsum</td>
<td>Ca₃SO₄[O₄.2H₂O]</td>
<td>00-033-0311</td>
<td>---</td>
<td>38</td>
<td>4</td>
<td>0.0 (0.0)</td>
<td>1.83</td>
</tr>
</tbody>
</table>

XRF (Wt%): CaO=45.7%, SiO₂=23.6%, Al₂O₃=11.6%, CO₂=1.5%
Figure A9. XRD whole pattern fit - sample 9.

Scan ID: 160109-S9V1.xml
Scan Parameters: 2.5111/59.99°/0.01671°/219.71°, u=1.255678°/34.99573°, φ=0°, l(p)=16177/93, Co(45kV,40mA), Saturday, 12/23/2009 3:41:59 PM
Configuration=Sample Spinner Reflection-Transmission, Owner=NTU, Creation date=12/23/2009 3:41:59 PM
Geometer=PW3059/60 (Theta/Theta), Minimum step size 2Theta:0.001, Minimum step size Omega:0.001
Sample stage=Reflection Transmission Spiner PW3059/60, Minimum step size Phi:0.1
Diffractometer system=XPERT-PRO
Measurement software=Crystallography software, Owner=NTU, Creation date=12/23/2011 8:43:04 AM

- Ka2 Peaks Present
- Variable-SIli Pattern
- Displacement = 0.0
- X-Ray Polarization = 1.0
- Ka2/Ka1 Ratio = 0.5

Geometry: Diffractometer Lp, Fitted-Range: 2.5° - 70.0°, BG-Model: Polynomial (8), λ: 1.78899 Å (Co)

PSF: Pearson-VII, Broadening: Individual FWHM Curve, Instrument: Constant FWHM = 0.1°

Phase ID (8) Chemical Formula PDF# NA NR NP %W (est) R/I
Anhydrite Ca(SO4) 01-072-0916 --- 24 12 70.7 (0.5) 1.77
Yeastite Ca4(A6O12)(SO4) 98-000-0467 5 52 10 14.0 (0.1) 3.11
Larinite Ca2SiO4 01-070-3388 --- 76 8 7.7 (0.2) 0.76
Alite Monoclinic CSS Ca3SiO5 98-000-0043 155 821 8 4.1 (0.1) 0.77
Calcite CaCO3 00-005-0586 --- 12 4 1.7 (0.1) 2.00
Bassanite CaSO4.5H2O 98-000-0108 24 208 3 0.9 (0.1) 1.19
Quartz SiO2 01-075-0443 --- 12 4 0.9 (0.1) 3.04
Gypsum CaSO4.2H2O 00-033-0311 --- 38 3 0.0 (0.0) 1.83

XRF(Wt%): CaO=43.6%, SO3=43.9%, SiO2=4.6%, Al2O3=7.0%, CO2=0.8%

Refinement Halted (R/E=1.57), Initial=4, Iter=6, P=4,1, R=8.18% (E=5.22%, EPS=0.5)

Observed Pattern

WP Fitted Pattern 160109-S9V1
Figure A10. XRD whole pattern fit - sample 10.
# Unit Conversion Factors

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<th>Multiply</th>
<th>By</th>
<th>To Obtain</th>
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<td>cubic meters</td>
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<td>cubic yards</td>
<td>0.7645549</td>
<td>cubic meters</td>
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<td>degrees Celsius</td>
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<td>feet</td>
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<td>meters</td>
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<td>gallons (US liquid)</td>
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<tr>
<td>inches</td>
<td>0.0254</td>
<td>meters</td>
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<tr>
<td>mils</td>
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<td>millimeters</td>
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<tr>
<td>ounces (US fluid)</td>
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<td>kilopascals</td>
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<td>pounds (mass)</td>
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<td>kilograms</td>
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<td>pounds (mass) per cubic foot</td>
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<td>kilograms per cubic meter</td>
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<td>quarts (US liquid)</td>
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<td>square inches</td>
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<td>tons (force)</td>
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<td>tons (2,000 pounds, mass)</td>
<td>907.1847</td>
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<td>tons (2,000 pounds, mass) per square foot</td>
<td>9,764.856</td>
<td>kilograms per square meter</td>
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<td>yards</td>
<td>0.9144</td>
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<td>4. TITLE AND SUBTITLE</td>
<td>Laboratory Characterization of Rapid-Setting Flowable Fill</td>
<td></td>
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<tr>
<td>6. AUTHOR(S)</td>
<td>William D. Carruth and Monica A. Ramsey</td>
<td></td>
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</table>
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U.S. Army Engineer Research and Development Center  
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| 14. ABSTRACT | Utility Fill One-Step 750® is a rapid-setting flowable fill product that has previously been validated in numerous full-scale demonstrations as an expedient backfill solution for Rapid Airfield Damage Recovery. Although the field performance of Utility Fill One-Step 750® has been extensively documented, a full laboratory characterization has not been conducted. This report analyzes and documents results from several laboratory tests conducted at two water-to-product ratios. The tests conducted are based on the suite of tests that make up the tri-service spall repair certification program used for rapid-setting concrete products. Tests include strength and set time-related properties, typical parameter control tests for concrete, and tests to determine the mineralogy and chemical makeup of the material. Long-term expansion and contraction properties were also tested. The data presented herein are intended to provide an overall assessment of Utility Fill One-Step 750® and to provide reasonable estimates of various design parameters. The results can be used as a basis for the future development of a formal laboratory certification protocol to down-select other rapid-setting flowable fill products for further evaluation. |
| 15. SUBJECT TERMS | Runways (Aeronautics)—Maintenance and Repair  
Controlled low-strength materials  
Military bases  
Air bases  
Cratering  
Fills (Earthwork) |
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