THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

U.S. Environmental Protection Agency

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ETV Joint Verification Statement

TECHNOLOGY TYPE:	**Infrastructure Rehabilitation Technologies**		
APPLICATION:	**Grouts for Wastewater Collection Systems**		
TECHNOLOGY NAME:	**Warren Environmental M-301 Epoxy Trowel On Mastic Systems**		
TEST LOCATION:	**University of Houston, CIGMAT**		
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The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The program's goal is to further environmental protection by accelerating the acceptance and use of improved and more cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer-reviewed data on technology performance to those involved in the design, distribution, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations; stakeholder groups, which consist of buyers, vendor organizations, and permitters; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests as appropriate, collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

NSF International (NSF), in cooperation with EPA, operates the Water Quality Protection Center (WQPC), one of six centers under the ETV Program. This evaluation of the performance of the Warren Environmental, Inc. M-301 Epoxy Trowel-On Mastic Grout for wastewater infrastructure rehabilitation was completed under a contract (No. EP-C-05-060) between EPA's Office of Research and Development and RTI International. The testing was performed by the University of Houston's Center for Innovative Grouting Materials and Technology (CIGMAT), with subcontract support by NSF International.

TECHNOLOGY DESCRIPTION

The following description of the M-301 Epoxy Trowel On Mastic System (M-301) was provided by the vendor and does not represent verified information.

The M-301 Epoxy can be used for sealing leaks in sewer pipe joints, and can also be used to control water seepage in cracks and joints in subgrade concrete structures. The grout was formulated with special additives and modifiers to enhance water and chemical resistance, and bond strength to a variety of substrates, as well as its own internal strength. The high thixotropic index allows for build-ups of up to ¾” on vertical surfaces without sag. It has been designed to be applied to a clean surface free of standing water with a notched (toothed) trowel similar to stucco. Alternately, it may be applied using heated tanks, heated lines, and Warren Environmental Inc.’s patented meter, mix, and spray equipment. This Epoxy system utilizes a 2 parts base-to-1 part activator mix ratio by volume. This product is sold and installed only by technicians specifically trained and licensed in the manufacturer's patented techniques.

VERIFICATION TESTING DESCRIPTION - METHODS AND PROCEDURES

Verification testing was conducted to evaluate M-301 Epoxy used in wastewater collection and treatment systems to control leaks into the systems. Specific testing objectives were (1) to evaluate the grout for working properties (setting (gel) time), physical and mechanical properties (unit weight, water absorption, shrinkage, permeability and compressive strength), durability and environmental properties (wet-dry cycle and chemical resistance), (2) evaluate grout-substrate interactions, under both wet and wet-dry conditions, and (3) determine the effectiveness of the grout in controlling leakage in cracked concrete using a model test.

There are no relevant American Society for Testing and Materials (ASTM) methods, so the testing was conducted following methods developed by CIGMAT, including CIGMAT(1) GR 1-04, GR 3-04, GR 7-04, GR 2-04, GR 3-04, CH 2-04 and CT 3-00. Product characterization tests were conducted on specimens of M-301 created for the purpose, and the uncoated concrete specimens used in the grout-substrate interaction tests to assure uniformity prior to their use in the bonding strength tests. Warren Environmental representatives were responsible for coating the concrete specimens and completing the repair of the concrete crack simulations in the model test, under the guidance of CIGMAT staff members. The coated specimens were evaluated over the course of six months.

PERFORMANCE VERIFICATION

<u>Grout Material Evaluation</u>

(a) Working properties

Setting time was determined by the elapsed time from grout preparation until the grout no longer flowed from a plastic cup or beaker inclined slowly (so that if the cup/beaker were filled with liquid, the surface of the liquid would remain level) to 45 degrees. Observations made at periodic intervals determined if the grout exhibited liquid-flow properties, or if the grout sample had gelled and the specimen could no longer flow from the container. Six replicates of grout were observed, with times varying from 38 to 40 minutes, with an average of 39 minutes.

(b) Physical and mechanical properties

Grout test specimens prepared for physical and mechanical tests consisted of cylinders approximately 4.5 inches (in.) high with a diameter of approximately 1.5 in. with a resin-to-water ratio of 9:1. Specimens were cured under ambient room temperature and humidity conditions. Cured specimens were removed from the mold and stored in labeled, sealed plastic bags for identification, protection, and to prevent moisture loss.

(i) Unit weight – Unit weight was determined for three specimens by taking the weight of the specimens divided by the volume. The unit weight measurements ranged from 0.91 to 1.09 g/cm^3, with an average of 1.00 g/cm^3.

(ii) Water absorption – Three grout specimens were immersed in tap water (initial pH approximately 7), and changes in the weight and volume (determined by measuring specimen diameter and height) of the specimens were recorded daily over a 4-day period. The weight change of the three specimens varied from 0.09% to 0.17%, with an average of 0.14%. The volume change in the specimen varied from 0.03% to 0.06%, with a mean of 0.04%.

(iii) Shrinkage – Three grout specimens were placed in sealed bags and held for 28 days at 23ºC ± 2ºC and relative humidity (RH) of 90% ± 5%. The weight and dimensions of the specimens were measured before and after the test. It was found that the weight change varied from 0 to 0.09%, with an average value of 0.06%, and the volume change varied from 0 to 0.04%, with an average of 0.01%.

(iv) Permeability – Three grout specimens were prepared in 1.5-in. diameter Plexiglas/glass cylinders and cured for a period of 7 days under room conditions. The cured specimens were permeated with water under a hydraulic gradient of 100 over a period of 72 hours. There was no observed discharge from any of the specimens indicating the permeability of the grout to be zero.

(v) Compressive strength – Compression tests on specimens cured over periods of 3, 7 and 28 days using a screw-type machine. Collected data included the compressive strength, modulus (determined from the initial slope of the stress/strain relationship), and failure strain (the maximum strain before the specimen failed). A total of 11 specimens were tested. The average strength, failure strain, and initial modulus after 3 days of curing were 6591 psi, 9%, and 225,000 psi, respectively; the average strength, failure strain, and initial modulus after 7 days of curing were 6402 psi, 5%, and 225,000 psi, respectively; and the average strength, failure strain, and initial modulus after 28 days of curing were 6180 psi, 5%, and 195,000 psi, respectively.

(c) Durability properties

(i) Wet-dry cycles – This test was designed to determine the impact of repeated wetting and drying on the performance of grouts. Three replicate specimens were subjected to 10 wet/dry cycles for a total test time of 140 days, or until failure (specimen completely deteriorated). One wet/dry cycle was 14 days in duration, consisting of 7 days of water exposure in tap water (pH approximately 7) followed by 7 days of dry conditions at room temperature and humidity (23 ± 2°C and 50% ± 5% RH). Changes in length, diameter, weight, and volume of the specimens were measured daily. At the end of the 10 wet/dry cycles, the specimens were tested to determine the compressive strength of the grout. After the first wet-dry cycle, the average change in weight, length, diameter, and volume was 0.04%, -0.25%, 0%, and -0.25%, respectively. The unit weight of the specimens increased over time, while the length of the specimens decreased. After the tenth wet-dry cycle, the average change in weight, length, diameter, and volume was 0.25%, -0.41%, 0%, and -0.41%, respectively. The unit weight of the specimens increased. The average strength of the grout after 10 wet-dry cycles was also determined and found to be an average of 6,531 psi, approximately equal to the 3-day compressive strength of the grout.

(ii) Chemical resistance – This test evaluates the resistance of grouts when exposed to chemical conditions representing various environmental applications and will help when selecting suitable grouts for use in various chemical environments. A total of nine grout specimens were prepared, and the initial weight, dimensions, color, and surface appearance of the specimens were recorded. Three specimens at each pH were fully immersed in solutions, with pH 2, 7, and 10 maintained at room temperature (23 ± 2°C) for the entire exposure period. The solutions consisted of tap water, with hydrochloric acid or sodium hydroxide added to achieve the pH required for the tests. The weight and volume change were determined and recorded for three specimens at each pH after 30, 90, and 180 days, with the following results:

pH= 2 solution: After 30 days, the average change in weight, volume, and unit weight was 0.32%, 0.59%, and -0.27%, respectively. After 180 days, the average change in weight, volume, and unit weight was 1.38%, 1.70%, and -0.31%, respectively. The weight and volume increased over the 180 days period.

pH= 7 (tap water): After one month, the average change in weight, volume, and unit weight was 0.29%, 0.11%, and 0.18%, respectively After 180 days, the average change in weight, volume, and unit weight was

1.24%, 0.47%, and 0.77%, respectively. The weight and volume increased over the 180 day period, as did the unit weight.

pH= 10 solution: After 30 days, the average change in weight, volume, and unit weight was 0.42%, -0.06%, and 0.48%, respectively After 180 days, the average change in weight, volume, and unit weight was 1.24%, 0.47%, and 0.79%, respectively. The weight and volume increased over the 180 day period, as did the unit weight.

(d) Environmental properties

Potential contaminant leaching from solidified grout was determined by analyzing water exposed to the grout for total organic carbon (TOC). Three test replicates, using cylindrical grout specimens of equal volumes and approximate equal weights, were exposed to tap water in individual exposure jars for 7 days. One blank container containing only the exposure water was prepared and held under the same conditions as the specimen exposure jars. The test was conducted with three grout specimens and water volume so that there was an adequate volume exposed to water to conduct the required analyses. A liquid-to-solid ratio of 1:1 (by volume) was used. The results found from 0.057 to 0.185 g of TOC/L, which translated to 0.0007 to 0.0023 g TOC/L/g grout, with a mean of 0.0017 g/L/g. These data should be considered estimated values because of data uncertainty arising from incomplete QA/QC, as discussed in the QA/QC section of this statement.

Grout-Substrate Interaction

Interaction between the grout and a concrete substrate was evaluated by testing the bonding strength and type of failure (i.e., bonding failure, substrate failure, or a combination) of prepared concrete bricks to which grout was applied under different service conditions. The test sandwich specimens were formed by applying grout material to an area of one concrete brick, then overlapping a second brick in a perpendicular direction to the first to allow the grout to form a filling between the overlap areas, as shown in Figure 1.

Figure 1. Formation of sandwich specimens.

Grout/concrete substrate specimens were exposed to (1) wet conditions and (2) a wet/dry cycle test. Four sandwich specimens were evaluated at each of 30, 90 and 180 days following water curing, and three were evaluated after 180 days of wet-dry cycle curing. The cured specimens were tested on a load frame to determine the break strength of the grout-brick bond. The break type was evaluated to determine where the failure occurred as described in Table 1. The results of the testing are summarized in Table 2. The failures observed in the specimens were all either Type 1 or Type 4, without a significant change in the break strength over the 180 day period, averages ranging from 257 to 280 psi. The specimens exposed to the wet/dry cycle showed lower break strengths, averaging 233 psi.

Table 1. Bonding Failure Types

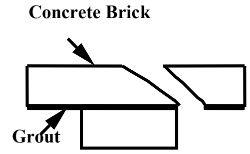
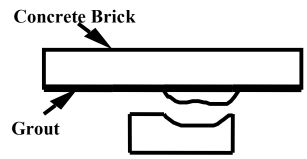
Failure Type	Description	Sandwich Test
Type 1	Substrate Failure	
Type 4	Bonding and Substrate Failure	

Table 2. Summary of Bonding Strength Tests

Exposure Time (days)	Exposure Conditions	Failure Type [1] – Number of Failures					Failure Strength (psi)	
		1	2	3	4	5	Range	Average
30	Water	3			1		228–288	260
90	Water	2			2		247–304	280
180	Water	3			1		204–329	257
	Wet-Dry Cycles	3					204–278	233

[1] See Table 1.

Model Test Evaluation

For this study, Warren Environmental, Inc. selected the model test related to leak control in cracked concrete. The model test uses 10-inch. (25-cm) diameter circular concrete disks with six-inch (15-cm) openings at the center (each disk is donut-shaped) to simulate a leak in a concrete structure. The two disks were placed one-inch apart and the opening was grouted by the vendor, as shown in Figure 2. After the vendor-specified curing period, the grouted joint was placed in a Plexiglas chamber (Figure 3), which was sealed to allow water to completely surround the grouted joint. Hydrostatic pressures of 3, 4, and 5 psi were applied through the inlet to the Plexiglas enclosure for 5 minutes at each pressure, and the water leaking through the grouted joint was collected and recorded. After two wet-dry cycles, the hydrostatic pressure tests were repeated.

Two replicate model tests were completed. As shown in Table 3, the model tests showed that the grouting with M-301 was effective in significantly reducing or eliminating the leak volume in the cracked concrete (0 to 19.3 gallons/day at 5 psi water pressure) immediately after grouting and after two wet and dry cycles over period of one month (0 to 16.2 gallons/day at 5 psi water pressure).

Figure 2. Repaired cracked concrete.

Figure 3. Model test setup.

Table 3. Model Test 4 Leak Rate Results (gallons/day)

Hydrostatic Pressure (psi)	Replicate 1		Replicate 2	
	Initial Condition	Wet-Dry Cycle Condition	Initial Condition	Wet-Dry Cycle Condition
3	11.5	7.6	0.0	0.0
4	13.9	12.6	0.0	0.0
5	19.3	16.2	0.0	0.0

Summary of Verification Results

The tests resulted in the following conclusions for Warren Environmental's M-301Epoxy Mastic Grout:

- The setting time of the grout at room temperature (70^{o}F) varied from 38 to 40 minutes, and the average unit weight of the solid grout was 62.6 pcf.
- During the water absorption test (under saturated conditions), the weight change in the specimens varied from 0.09% to 0.17%, with an average of 0.14%. The volume change in the specimen varied from 0.03% to 0.05%, with a mean of 0.04%.
- The shrinkage at 23^{o}C and 90% RH after 28 days of testing resulted in an average weight and volume change of 0.03% and 0.01%, respectively.
- The permeability of the grout was zero (impermeable) under a hydraulic gradient of 100 held for 4 days.
- The average strength, failure strain, and initial modulus after 3 days of curing were 6,591 psi, 9%, and 225,000 psi, respectively. The average strength, failure strain, and initial modulus after 28 days of curing were 6,180 psi, 5%, and 195,000 psi, respectively.
- After ten wet-dry cycles, the average change in weight, length, diameter and volume was 0.25%, - 0.41%, 0%, and -0.41%, respectively. The unit weight of the specimens increased by 0.99%. The average strength of the grout after 10 wet-dry cycles was 6,531 psi; hence, the specimen strength was not affected after 10 wet-dry cycles.
- After 180 days in a pH =2 solution (acid), the average change in unit weight and volume was -0.31% and 1.70%, respectively. After 180 days in a pH =7 solution (water), the average change in unit weight and volume was 0.77% and 0.47%, respectively. After 180 days in a pH =10 solution (base), the average change in unit weight and volume was 0.79% and 0.47%, respectively.

- The average total organic content (TOC) in the leaching water was 0.0017 g/L/g of grout; however, because of incomplete QA/QC, these results should only be considered estimated values.
- The bonding strength for water cured grout-concrete specimens did not vary significantly over a 180 day exposure period, with average strengths ranging from 257 to 280 psi; most of the specimen failures were Type 1 (substrate), with others being Type 4 (substrate failure with the grout intact).
- After 180 days of the grout-substrate test, following 10 wet-dry cycles, the average bonding strength was 233 psi, and all (100%) of the failures were Type 1.
- Model tests showed that grouting with M-301 significantly reduced or eliminated the leak in a simulated one-inch concrete crack (0 to 19.3 gallons/day at 5 psi water pressure) immediately after grouting, and after two wet and dry cycles over period of 30 days (0 to 16.2 gallons/day at 5 psi water pressure). Prior to grouting all of the water leaked out of the open pipe joint.

Quality Assurance/Quality Control

A technical systems audit prior to the start of testing to ensure that CIGMAT was equipped to comply with the test plan, along with a data quality audit of at least 10% of the test data to ensure that the reported data represented the data generated during testing. The documentation submitted by CIGMAT for the working properties, physical and mechanical properties, and durability properties support the findings as described in this report. The documentation provided by CIGMAT for the TOC analyses showed that the laboratory did not produce sufficient QC documentation to provide traceability to back up the TOC analytical results. Overall, the TOC data does not have the QA/QC support to validate or refute the reported values. Further explanation is provided in the verification report.

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Availability of Supporting Documents

Referenced Documents:

1) CIGMAT Laboratory Methods for Evaluating Grout and Coating Materials, available from the University of Houston, Center for Innovative Grouting Materials and Technology, Houston, TX.

Copies of the *Test Plan for Verification of Warren Environmental Inc. Mastic 301-04 for Infrastructure Rehabilitation* (September 2009), the verification statement, and the verification report (NSF Report Number 10/34/WQPC-SWP) are available from:

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