

Environmental Technology Verification Protocol

Water Quality Protection Center

Verification Protocol for the Verification of Grouting Materials for Infrastructure Rehabilitation

Prepared by



NSF International

Under a Cooperative Agreement with
 EPA U.S. Environmental Protection Agency

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**PROTOCOL FOR THE VERIFICATION OF GROUTING
MATERIALS FOR INFRASTRUCTURE REHABILITATION**

Prepared for:

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Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threaten human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites, sediments and ground water; prevention and control of indoor air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by: developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

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Acronyms

AA	Atomic absorption
ASTM	American Society for Testing and Materials
CIGMAT	Center for Innovative Grouting Materials and Technology
cm	Centimeter
cP	Centi-poise
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
ft	Foot or feet
g	Gram
in.	Inch
I/I	Infiltration and inflow
kPa	Kilopascals
lb	Pound
lb/ft ³	Pounds per cubic feet
mL	Milliliter
mm	Millimeter
min	Minute
MSDS	Material safety data sheet
NSF	NSF International
ORD	EPA Office of Research and Development
psi	Pounds per square inch
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
RH	Relative humidity
RPD	Relative percent deviation
RSD	Relative standard deviation
sec	Second(s)
SD	Standard deviation
SOP	Standard operating procedure
TOC	Total organic carbon
WQPC	ETV Water Quality Protection Center

Chapter 1

Introduction

1.1 Background

A critical issue facing the nation today involves the increased pollution of waterways, shorelines, rivers, bays, and streams. A high percentage of infiltration and inflow (I/I) into sanitary sewer systems is attributable to cracks and leaks from open joints in sewer pipes or service laterals connected to the sewers. Municipalities are discovering that if I/I problems are not adequately controlled, they will lead to frequent overflows and undue burden on treatment facilities.

The primary goal of in-situ grouting, the oldest trenchless technology method still used to control leaks in wastewater systems, is to return the structure to its original working condition. Several types of grout materials have been used to control I/I problems in wastewater systems and storm systems (8,9). These materials have been commonly used for leak control in below-grade wet wells and holding tanks, manholes, sewer and storm lines, and cracked retaining walls and other underground structures (3,5,6,9,10).

However, there is no systematic method for evaluating the performance of grouts under service conditions. In a 2002 infrastructure research needs report (7), EPA identified the need to evaluate the performance of grouts under various environmental conditions, such as humidity, temperature, pH, and wastewater chemistry. The aim of this protocol is to establish a comprehensive approach for testing grouts for leak control applications in old and new wastewater collection systems and other concrete repairs.

1.2 Technical Approach

The overall objective of this protocol is to describe a testing program to systematically evaluate grouts for controlling infiltration to wastewater systems and leaks in concrete structures. Specific verification objectives are to:

- Evaluate the properties (working, physical, mechanical, durability, and leachability) of grouts and grouted sands;
- Characterize the bonding properties of the grout–substrate interaction; and,
- Verify the performance of grouted joints and repaired concrete cracks under hydrostatic pressure up to 5 psi (10 feet of water) and wet/dry cycles over a period of three months.

Testing will use relevant ASTM and CIGMAT standards. A total of 12 different tests will characterize the grouts (Table 3-1); several additional tests will evaluate grouted sand specimens and grout–substrate interaction (Table 3-2). Model tests will be used to evaluate grout effectiveness under various leak control configurations. All CIGMAT standard methods referenced herein are included in Appendix A, while the ASTM standard numbers are referenced in this protocol

A test plan will be prepared for each grout material to be evaluated. The plan will include specific testing procedures and a quality assurance project plan (QAPP) describing the quality systems to be used during the evaluation.

1.3 Roles and Responsibilities

The primary roles and responsibilities of each party involved in the verification process are described in this section.

1.3.1 Verification Organization

The primary role of the verification organization (VO), NSF, is to:

- Coordinate with CIGMAT, the testing organization (TO), and the vendor to prepare a test plan, using this protocol as a template, to meet testing requirements;
- Coordinate with the ETV Grouts Technical Panel, as needed, to review the test plan prior to the initiation of verification testing;
- Review and approve test plan, and sign the test plan signoff sheet;
- Coordinate with the EPA Water Quality Protection Center (WQPC) Project Officer to approve the test plan prior to the initiation of verification testing;
- Review the quality systems of the TO and qualify them to complete the testing;
- Oversee the technology evaluation and associated laboratory testing;
- Review data generated during verification testing;
- Oversee the development of a verification report and verification statement; and
- Provide quality assurance oversight at all stages in the verification process.

1.3.2 EPA

This protocol has been developed with financial and quality assurance assistance from the ETV Program, which is overseen by the EPA's Office of Research and Development (ORD). The ETV Program's Quality Assurance Manager and the WQPC Project Officer will provide administrative, technical, and quality assurance guidance and oversight on all ETV WQPC activities, and will review and approve each phase of the verification project. The primary responsibilities of EPA personnel are to:

- Review and approval of the test plan, including the quality assurance project plan (QAPP);
- Sign the test plan signoff sheet;
- Review and approval of the verification report and verification statement; and
- Posting of the verification report and verification statement on the EPA ETV website.

1.3.3 Technology Panel

A technology panel was formed to assist with the review of this generic grout test plan. Input from the panel ensures that data generated during verification testing are relevant and that the

method of evaluating different technologies is fair and consistent. All grout test plans may be reviewed by representatives of the technology panel and will be approved by the WQPC Program Manager and vendor.

1.3.4 Testing Organization

The TO for verifications completed under test plans developed from this protocol is CIGMAT Laboratories at the University of Houston. The primary responsibilities of the TO are to:

- Coordinate with the VO and vendor relative to preparing and finalizing the test plan;
- Sign the test plan signoff sheet;
- Conduct the technology verification in accordance with the test plan, with oversight by the VO;
- Analyze all samples collected during the technology verification process, in accordance with the procedures outlined in the test plan;
- Coordinate with, and report to, the VO during the technology verification process;
- Provide analytical results of the technology verification to the VO; and
- If necessary, document changes in plans for testing and analysis, and notify the VO of any and all such changes before the changes are executed.

1.3.5 Vendor

The vendor's primary responsibilities are to:

- Provide the TO with grout samples for verification;
- Complete a product data sheet (Appendix B) prior to testing;
- Sign the test plan signoff sheet;
- Provide technical assistance to the TO, as requested, during the verification testing period;
- Provide funding for verification testing;
- Review and concur the test plan, including the QAPP; and
- Review the verification report and verification statement.

Chapter 2

Test Facility

Since 1985, CIGMAT researchers at the University of Houston have been investigating the performance of various grouting materials used in wastewater facilities and sand. The CIGMAT laboratories and associated facilities are located in the College of Engineering complex at 4800 Calhoun Road, Houston, Texas. Several laboratories at the University are available to perform the required physical and chemical testing.

The CIGMAT laboratories and affiliated facilities are equipped with devices that can perform all of the grout, grouted sand, and grout–substrate tests in this test plan. Molds are available to prepare the specimens for testing. Four types of models are available to perform the model tests which simulate the behavior of applied grouts in several different applications: leak control in a pipe joint; leak control in a manhole joint; leak control in a lateral; and concrete leak repair. Hydrostatic test chambers are available to calibrate the leak rates at the test joints.

Chapter 3

Experimental Design

The ETV testing program for grouts will evaluate the performance and characteristics of grout material in three different testing phases:

- The physical properties of the grout and grouted sand will be tested by utilizing test specimens created by the TO;
- The interaction of the grout and substrate will be tested by applying the grout to test substrate material (such as bricks or concrete) and completing a series of performance tests; and,
- Model tests, where grout is applied to laboratory-simulated leaking pipes, pipe joints, and/or manholes will be conducted to test for leak control.

Testing details are given in the following sections.

3.1 Grout and Grouted Sand Physical Property Evaluation

Properties of the neat resin (unsolidified grout), grout, and grouted sand specimen samples to be tested can be grouped as:

- Working properties (resin/grout mix);
- Physical and mechanical properties (grout and grouted sand specimens);
- Durability properties (grout and grouted sand specimens); and,
- Leachability (grout and grouted sand specimens).

The properties to be tested are summarized in Tables 3-1 and 3-2 for grout and grouted sand, respectively.

The physical property evaluation tests consist of creating grout/grouted sand specimens, challenging the specimens to a particular test, and measuring the results. For tests where testing procedures have been developed by the American Society of Testing and Materials (ASTM), the ASTM test procedure will be used. Where no ASTM test procedures exist, CIGMAT has developed their own testing protocols, and these protocols will be used. Where applicable, the ASTM and CIGMAT testing procedures are referenced in the following sections and the CIGMAT procedures are included in Appendix A of this protocol.

3.1.1 Grout and Grouted Sand Specimen Preparation

3.1.1.1 Grout Specimens

ASTM Standard Method C31/C-31M-96 shall be used for making and curing test specimens for cement, acrylic, or acrylamide-based grouts (which typically do not expand while curing). Standard Practice CIGMAT GR 4-00 shall be used for making and curing test specimens for polyurethane-based grout (which may expand while curing). Figure 3-1 shows the molds that

will be utilized to create the test specimens. Specimens to be cured under water shall be completely submerged in a water bath of tap water at room temperature. If a specimen floats, a small amount of force will be applied to keep it submerged. Multiple specimens of the same type of grout or grouted sand material may be placed in the same water bath. After solidification, specimens shall be removed from the mold and stored in labeled, sealed plastic bags for identification, protection, and to prevent moisture loss. The specimens shall be stored in a temperature- and humidity-controlled room at $23 \pm 2^\circ\text{C}$ (room temperature) and 50 ± 5 percent humidity.

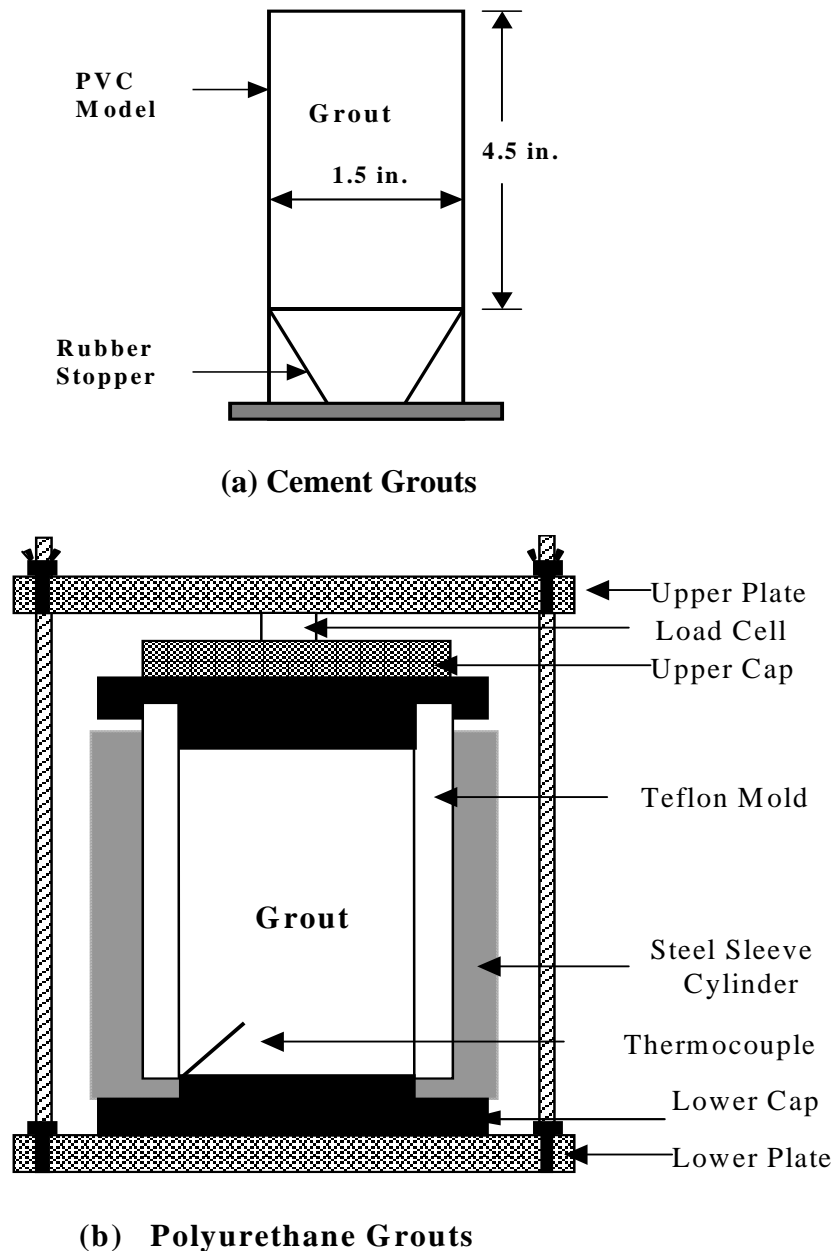


Figure 3-1. Typical models used for preparing grout specimens.

Table 3-1. Grout Tests for Various Applications

Properties	Tests	Conditions	Test Method to be Used	Leak Control Applications				Number of Specimens or Tests
				Pipe Joint	Manhole	Lateral	Concrete Repair	
Working Properties	Viscosity	23°C	CIGMAT GR 6-02	X	X	X	X	3
	Setting (Gel) Time	23°C	ASTM C 191-04 (cement-based) or method defined in 3.1.2.1.2 (chem.)	X	X	X	X	6
Physical and Mechanical Properties	Unit Weight	23°C	CIGMAT GR 1-00	X	X	X	X	3
	Water Absorption	23°C	CIGMAT GR 3-00	NA	NA	NA	X	3
	Shrinkage	Temp, humidity	Method defined in 3.1.3.3	NA	NA	NA	X	3
	Permeability	Water	CIGMAT GR 7-02	NA	NA	NA	X	3
	Compressive Strength	3, 7, 28 days	CIGMAT GR 2-02	NA	NA	NA	X	9
	Tensile Strength	3, 7, 28 days	CIGMAT PC 2-99 (Specimen preparation by CIGMAT CH 2-01)	NA	NA	NA	X	9
	Elongation	3, 7, 28 days	CIGMAT PC 2-99 (Specimen preparation by CIGMAT CH 2-01)	NA	NA	NA	X	9
Durability Properties	Wet-Dry Cycle	Number of cycles	CIGMAT GR 3-00	NA	NA	NA	X	3
	Chemical Resistance	pH = 2, 7, 10	CIGMAT CH 2-01	NA	NA	NA	X	9
Environment. Properties	Leaching	Water	Method defined in 3.1.5.1	X	X	X	X	3

3.1.1.2 Grouted Sand Specimens

Grouted sand specimens shall be prepared according to CIGMAT GS 1-02. The mold to be used to create them is shown in Figure 3-2. Each specimen shall be made in a separate mold and the amount of grout permeated will be recorded by measuring the amount of grout injected. The molds shall be constructed of Plexiglas™ that can be split longitudinally into three equal pieces so that the specimen can be removed after it has been prepared. One edge of each piece of the split tube shall be coated with silicon rubber to form a seal when the three pieces are clamped together with hose clamps. Plexiglas filters with nylon mesh shall be used at the inlet and outlet ends. A half-inch sand filter, separated from the specimen by nylon mesh, shall be used at the inlet to distribute the grout uniformly. The mold shall be filled with sand and another sand filter with nylon mesh shall be used in the outlet (similar to inlet). The sand shall have a particle size in the range of 0.3 to 7 mm, a unit weight in the range of 120 to 130 pounds per cubic foot and a relative density prior to grouting of 85 to 92 percent. Six specimens shall be grouted in parallel at an injection pressure of 2 psi.

After solidification, specimens shall be removed from the mold and stored in sealed, labeled plastic bags in a temperature- and humidity-controlled room ($23 \pm 2^\circ\text{C}$ and $50\% \pm 5\% \text{ RH}$).

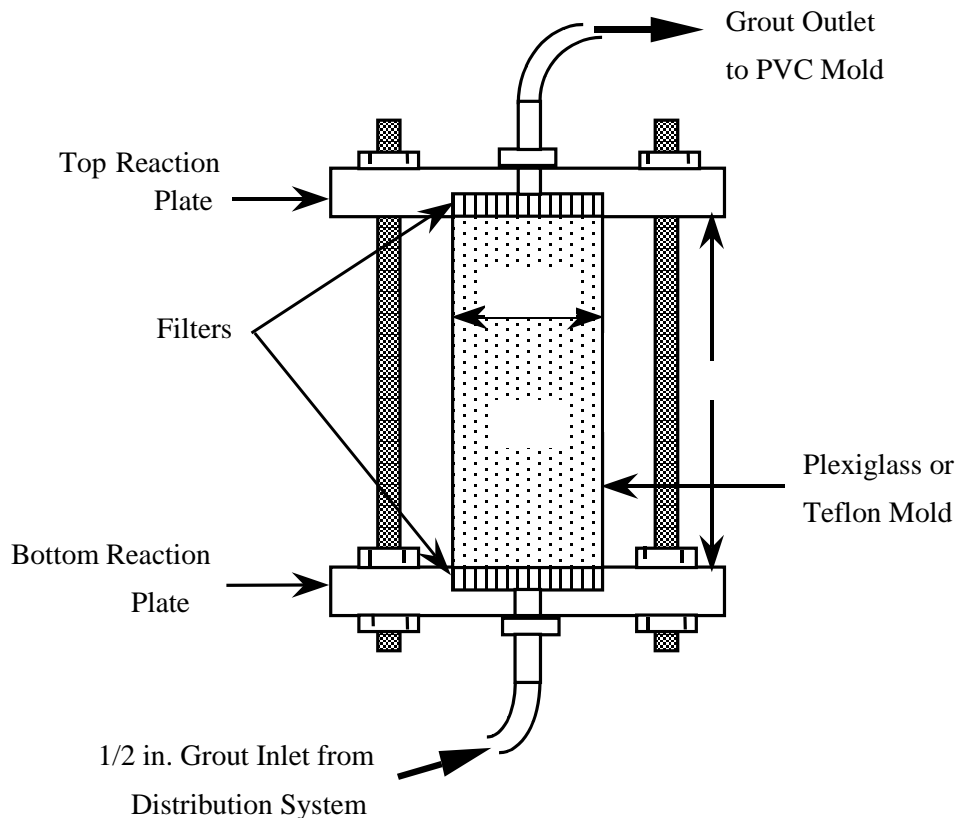


Figure 3-2. Mold for preparing grouted sand specimens.

Table 3-2. Grouted Sand and Grout–Substrate Interaction Tests

Materials	Tests	Conditions	Test Method to be Used	Leak Control Applications			Concrete Repair	Number of Tests
				Pipe Joint	Manhole	Lateral		
GROUTED SAND								
Physical and Mechanical Properties	Unit weight	Cured	CIGMAT GR 1-00	X	X	X	NA	3
	Water absorbance	23°C	CIGMAT GR 3-00	X	X	X	NA	3
	Shrinkage	Temp, humidity	Method defined in 3.1.3.3	X	X	X	NA	3
	Permeability	Water	CIGMAT GR 7-02	X	X	X	NA	3
	Compressive strength	3, 7, 28 days	CIGMAT GR 2-02	X	X	X	NA	9
	Tensile strength	3, 7, 28 days	CIGMAT PC 2-99	X	X	X	NA	9
Durability Properties	Wet-dry cycle	Number of cycles	CIGMAT GR 3-00	X	X	X	NA	3
	Chemical Resistance	pH = 2, 7,10	CIGMAT CH 2-01	X	X	X	NA	9
GROUT-SUBSTRATE INTERACTION								
Bonding Strength	Wet condition	Concrete, clay brick, cured under water	CIGMAT GR 5-00 or CIGMAT CT 3-00	NA	NA	NA	X	12
	Wet-dry cycle	Number of cycles	CIGMAT GR 3-00	NA	NA	NA	X	12

3.1.2 Grout Curing Properties

3.1.2.1 *Viscosity*

Grout viscosity will be evaluated using the procedures described in this section. The viscosity of grouted sand will not be evaluated. Grout viscosity will be evaluated using a procedure outlined in CIGMAT GR 6-02. Using a cylindrical spindle-type viscometer (Brookfield Viscometer with 8 speeds, LVT model with four spindles or equivalent), the initial viscosity and changes in viscosity during the gelling process shall be measured at room temperature at selected strain rates (up to 180 sec⁻¹). The specific strain rates at which viscosity will be measured shall be determined in advance of testing by the TO, with the consent of the vendor. Once the material performs consistently at different viscometer speeds, the test shall be complete. A minimum of three replicate tests shall be conducted.

3.1.2.2 *Setting (Gel) Time*

Grout setting or gel time will be evaluated using the procedures described in this section. The setting time of the grouted sand will not be evaluated.

For cement-based grouts, ASTM C 191-04, "Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle," shall be used. The room temperature and relative humidity (23 ± 2°C, 50% ± 5% RH) shall be adjusted as necessary to comply with the ASTM standard.

No ASTM standard method is currently available to determine the gel time for acrylamide, acrylic, or polyurethane-based grouts. Hence it shall be determined by the elapsed time from grout preparation until the grout no longer flows 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. Approximately 50 mL of freshly prepared grout shall be poured into a container. At periodic intervals to be specified in the test plan, the container shall be slowly tipped to approximately 45 degrees, and the analyst shall determine if the grout exhibits liquid flow properties or if the grout sample has gelled and the specimen can no longer flow from the container.

A total of six replicates of each grout shall be analyzed.

3.1.3 Physical and Mechanical Properties

To obtain initial characterization information on the grout and grouted sand specimens, all specimens shall be weighed to 0.1 g using a calibrated digital balance and measured (diameter and height) using a vernier caliper with a least count of 0.01 mm. Measurements shall be taken at the top, middle, and bottom of the specimen, with two measurements taken at 90 degrees from each other at each location to obtain consistent data.

3.1.3.1 *Unit Weight (Density)*

Solidified grout and grouted sand specimens shall be used to determine the unit weight (density) of the grout. The determination shall be completed per CIGMAT GR 1-00 for both grout and grouted sand specimens. Unit weight shall be calculated using the weight and volume of the specimens. A minimum of three replicates will be evaluated for unit weight. Based on the unit weight, the grout shall be reported as lighter or heavier than water.

3.1.3.2 *Water Absorption*

Water absorption characteristics shall be evaluated on both grout and grouted sand specimens as outlined in standard procedure CIGMAT 3-00. A minimum of three solidified grout and grouted sand specimens shall be immersed in tap water (initial pH in the range of 7 to 8) and changes in weight and volume (determined by measuring specimen diameter and height) of the specimens shall be recorded a minimum of once every working day (Monday through Friday, excluding holidays) until the changes in weight and volume become negligible (less than 0.5 percent of the previous weight and volume), or for one week, whichever occurs first. The report for this testing shall include the time of immersion, the initial characteristics of the specimens, the weight and volume change with time, water absorption as a percentage of the initial weight, and volume of grout.

3.1.3.3 *Shrinkage*

This test determines specimen weight and shape changes under various temperature and humidity conditions (Table 3-3) generally encountered during service (16,19). The vendor shall choose one or more of four test conditions (parts), based on the anticipated applications for the grout (crack in wall, sewer, or pipes). For example, Parts A and B specify $10 \pm 2^\circ\text{C}$, similar to the temperature of soils around sewer lines in cooler northern climates, while Parts C and D specify a temperature of $27 \pm 2^\circ\text{C}$, more typical of sewer conditions in warmer southern climates. The vendor may select a different temperature more representative of the application in which the grout is to be used. A controlled temperature chamber shall be used to maintain the specific conditions of temperature, while humidity shall be maintained by enclosing specimens in a sealable plastic bag or glass bottle, or leaving them uncovered. Humidity will be measured using a digital humidity meter. At the onset of the test, specimens shall be prepared in a mold with inner dimensions of 1.5 in. (38 mm) in diameter and 3.5 in. (90 mm) in length. It is anticipated that the grout specimens will shrink during the initial curing. Grout specimens that shrink to a length of 2.6 in. (65 mm) or less shall be rejected and new grout specimens shall be made. A minimum of three specimens shall be tested under each selected test condition. The weight and dimensions of the specimens shall be reported before and after 28 days of conditioning.

Table 3-3. Shrinkage Test Conditions (Vendor-Selected Based on Application)

Parts	Temperature, Duration, and Relative Humidity
Part A	10°C ± 2°C for 28 days @ 90% ± 5% RH
Part B	10°C ± 2°C for 28 days @ 50% ± 5% RH
Part C	27°C ± 2°C for 28 days @ 90% ± 5% RH
Part D	27°C ± 2°C for 28 days @ 50% ± 5% RH

3.1.3.4 *Permeability*

Solidified grout and grouted sand specimens shall be used to determine their permeability. Specimens shall be prepared in 2-in. diameter Plexiglas/glass cylinders and permeated with water under a hydraulic gradient of 100, per CIGMAT GR 7-02. Testing shall be completed at room temperature and humidity unless otherwise specified by the vendor. A minimum of three replicate tests shall be run on both grout and grouted sand specimens. The report for this testing shall include the temperature and humidity at which testing was completed, any changes in the specimens during the testing, and the permeability obtained during the testing.

3.1.3.5 *Unconfined Compressive Strength and Stress/Strain Relationship*

CIGMAT GR 2-02 has been developed for testing grouts and grouted sand specimens in compression under monotonically increasing load (load increasing linearly), based on test methods ASTM D 3574-03 (polyurethane) and ASTM C 109-02 (cementitious materials). Compression tests shall be performed using screw-type machines with capacities up to 5,000 lbs. Specimens shall be loaded at specified rates based on the type of grout or grouted sand, and the loading rate may be determined based on trial tests conducted outside of the ETV test. Cement, acrylic, or acrylamide-based grout and grouted sand specimens 1.5 in. (38 mm) in diameter and 2.6 to 3.5 in. (65 to 90 mm) in length shall be tested. For polyurethane-based grout and grouted sand, the specimens shall be 1.2 to 1.4 in. (30 to 35 mm) long. The specimens shall be trimmed and capped (if necessary, using a sulfur compound commonly used for capping cement concrete) to ensure smooth and parallel surfaces. Specimens shall be tested in triplicate at 3, 7 and 28 days following specimen preparation. The reported data shall include compressive strength, modulus and failure strain, where the modulus is determined from the initial slope of the stress/strain relationship and the failure strain is the maximum loading point before the specimen fails.

3.1.3.6 *Tension Tests*

Direct tension and indirect tension tests shall be performed on grout and grouted sand specimens. Special “dumbbell-shaped” Teflon[®]-lined molds, as described in CIGMAT CH 2-01, shall be used to prepare the direct tension specimens. Direct tension testing shall be conducted on the prepared specimens using the dumbbell-shaped molds, following the procedures outlined in CIGMAT PC 2-99. (Note: CIGMAT PC 2-99 specifies dumbbell-shaped molds with different dimensions than those outlined in CIGMAT CH 2-01. The correct dumbbell-shaped mold dimensions for the ETV test are outlined in CIGMAT CH 2-01.) For the indirect splitting tension

test, specimens 1.5 in. (38 mm) in diameter and 2.6 to 3.5 in. (65 to 90 mm) in length shall be used (ASTM C-496). A screw-type machine shall be used for both the direct and indirect tension tests. Following one or two trial tests, the rate of loading by the machine shall be adjusted to cause failure of the specimens within 20 minutes. The selected test speed shall be included in the final verification report. A minimum of three specimens shall be tested after each curing period of 3, 28, and 180 days for a minimum total of nine replicates for each tension test. The reported data shall include the maximum loads carried by the specimens, the average cross-sectional area, and the calculated direct and indirect tensile strengths, to the nearest 10 psi (69 kPa).

3.1.4 Durability Properties

3.1.4.1 *Wet/Dry Cycle*

During its service life, the grout or grouted sand could be subjected to a number of wet/dry cycles. This test is designed to determine the impact of repeated wetting and drying on the performance of grouts and grouted sand. A minimum of three replicate specimens shall be used for this test. The specimens shall be subjected to 10 wet/dry cycles for a total test time of 140 days, or until failure (unconsolidation). One wet/dry cycle shall be 14 days in duration, consisting of 7 days of water exposure followed by 7 days of dry conditions at room temperature and humidity ($23 \pm 2^\circ\text{C}$ and $50\% \pm 5\% \text{ RH}$). The water exposures shall be completed as described in Section 11 of CIGMAT GR 3-00, using tap water having a pH between 7 and 8. Changes in length, diameter, weight, and volume of the specimens shall be measured daily per Sections 9 and 11 of CIGMAT GR 3-00. At the end of the 10-wet/dry cycles, specimens shall be tested to determine the compressive and/or tensile strengths of the grout, as described in sections 3.1.3.5 and 3.1.3.6. The reported data shall include weight and dimension data collected for the specimens, as well as the data to be reported described in sections 3.1.3.5 and 3.1.3.6.

3.1.4.2 *Chemical Resistance*

This test will evaluate the resistance of grouts and grouted sands when exposed to chemical conditions representing various sand and groundwater environments. The test results will help when selecting suitable grouts for use in various chemical environments. Cylindrical grout specimens shall be prepared as described in Section 3.1.1.1, and the initial weight, dimensions, color, and surface appearance of the specimens shall be recorded. Nine specimens at each pH shall be fully immersed in solutions with pH 2, 7, and 10 maintained at room temperature ($23 \pm 2^\circ\text{C}$) for the entire exposure period. The solutions shall consist of tap water with hydrochloric acid or sodium hydroxide added to achieve the pH required for the tests. The vendor may specify a different temperature, depending on the application. The weight, volume, color, and surface appearance of the specimens shall be determined and recorded for three specimens at each pH after 30, 90, and 180 days, as described in Section 7.3 in CIGMAT CH 2-01. The pH, clarity, and color of the exposure solution shall also be recorded at each evaluation time. During the evaluation, if the pH changes by more than ± 2 units, additional hydrochloric acid or sodium hydroxide shall be added to the solution to return it to its original pH. The analyst shall note in the project logbook the quantity of chemical and revised pH during each adjustment. At each evaluation, compression testing shall be completed for the specimens in accordance with Section 7.4 of CIGMAT CH 2-01. All data and observations shall be reported, along with the

calculations described in sections 8.1, 8.3, and 8.4 of CIGMAT CH 2-01. The appearance of specimens and immersion solutions shall be reported as described in sections 9.2 and 9.3 of CIGMAT CH 2-01.

3.1.5 Environmental Properties—Leaching Test

Potential contaminant leaching from solidified grout shall be determined by analyzing water exposed to the grout for total organic carbon (TOC) and lead. A minimum of three test replicates, using cylindrical grout specimens, will be prepared as described in Section 3.1.1.1. The specimens will be immersed in three individual exposure jars, each containing tap water (pH = 8 ± 0.5; TOC < 1 mg/L; total lead < 0.3 mg/L). One blank container containing only the exposure water shall be prepared and held under the same conditions as the specimen exposure jars. The exposure jars and blank jar will be held at room temperature for seven days.

The test shall be conducted with the appropriate number of grout specimens and water volume so that there is an adequate volume of exposure water to conduct the required analyses. A liquid-to-solid ratio of 1:1 (by volume) shall be selected as the default, but other ratios may be used, as requested by the vendor and approved by the TO. If a different liquid-to-solid ratio is used, it shall be reported in the verification report.

At the beginning and end of the exposure period, samples of the exposure water will be analyzed to determine the presence of organic compounds or metals that have leached from the grout. The samples will be analyzed for total lead and TOC. Other analyses may be included, as determined by the testing organization, based on the MSDS sheet provided by the grout manufacturer. If other analyses are included, the concentration of the analyte in the exposure water shall not be greater than the method detection limit.

The water in the blank container shall be sampled at the beginning and end of the exposure period, and analyzed for the same constituents as the grout specimen exposure water. This will provide a baseline concentration of constituents in the tap water.

Details of the analytical methods, required sample volumes, and sample holding are provided in Section 4.

3.2 Grout–Substrate Bonding Strength

Interaction between the grout and a concrete substrate shall be evaluated by testing the bonding strength and type of failure (bonding failure, substrate failure, or a combination) under different service conditions, as specified in sections 3.2.1 through 3.2.3. Testing of wet grout/concrete substrate specimens shall be conducted over a period of six months in accordance with CIGMAT GR 5-00 (where two cylinders are bonded with grout) or CIGMAT CT 3-00 (where the area between concrete bricks/prisms is grouted), as selected by the vendor prior to the ETV verification. In addition, bonded configurations prepared according to either CIGMAT GR 5-00 or CIGMAT CT 3-00 shall be subjected to wet/dry cycle testing, as described in Section 3.1.4.1.

3.2.1 Cylinder Bonding (CIGMAT GR 5-00)

This test configuration may be used to determine the bonding strength of various grout materials (15,23). The test consists of sandwiching a layer of grout between flat surfaces of concrete (the ends of concrete cylinders) and then loading the test specimen in tension. Details of specimen preparation are in CIGMAT GR 5-00. The Grout-Rock (Soil) Test outlined in Section 7.2 of CIGMAT GR 5-00 will not be conducted as part of the ETV testing. The reported data shall include all collected data, the bonding strength, and the type of bonding failure.

3.2.2 Brick/Prism Bonding (CIGMAT CT 3-00)

Although CIGMAT CT 3-00 was developed for coating materials, it may be adopted for grouts. As described in CIGMAT CT 3-00, the grout shall be sandwiched between a pair of rectangular concrete block and clay brick specimens and then tested for bonding strength and type of failure. Even though CIGMAT CT 3-00 specifies the use of dry bricks, for the purposes of ETV testing, wet specimens shall be used to simulate extreme grouting conditions. The bonded wet specimens shall be immersed in water until testing begins. The reported data shall include the type of brick used, number of specimens tested, age of specimen at time of test, average bond strength, standard deviation, type of failure, and the bond strength.

3.2.3 Wet/Dry Cycle

During its service life, a grouted concrete joint could be subjected to a number of wet/dry cycles. Hence, each bonded configuration will be tested for performance by subjecting it to 10 wet and dry cycles, where one wet/dry cycle takes 14 days, for a total test time of 140 days, or until failure. Changes in the length, diameter, weight, and volume shall be measured daily, as described in Section 3.1.4.1. Following the wet/dry cycles, a minimum of three test specimens shall be retested to determine the compressive strength of the grout to quantify possible changes. Compressive strength will be determined using the apparatus and procedure described in Sections 5 and 10 of CIGMAT GR 2-02. The reported data shall include testing conditions, number of specimens tested, individual and average compressive strength and modulus, and/or tensile strength.

3.3 Model Tests

Since water leaks can occur under different conditions, four model tests are available to represent different field situations. Depending on the application for the grout, the vendor shall select one or more of these model tests to be completed:

- Model Test 1: Leak Control in Pipe Joint
- Model Test 2: Leak Control in Manhole Joint
- Model Test 3: Leak Control at a Lateral Joint
- Model Test 4: Concrete Leak Repair

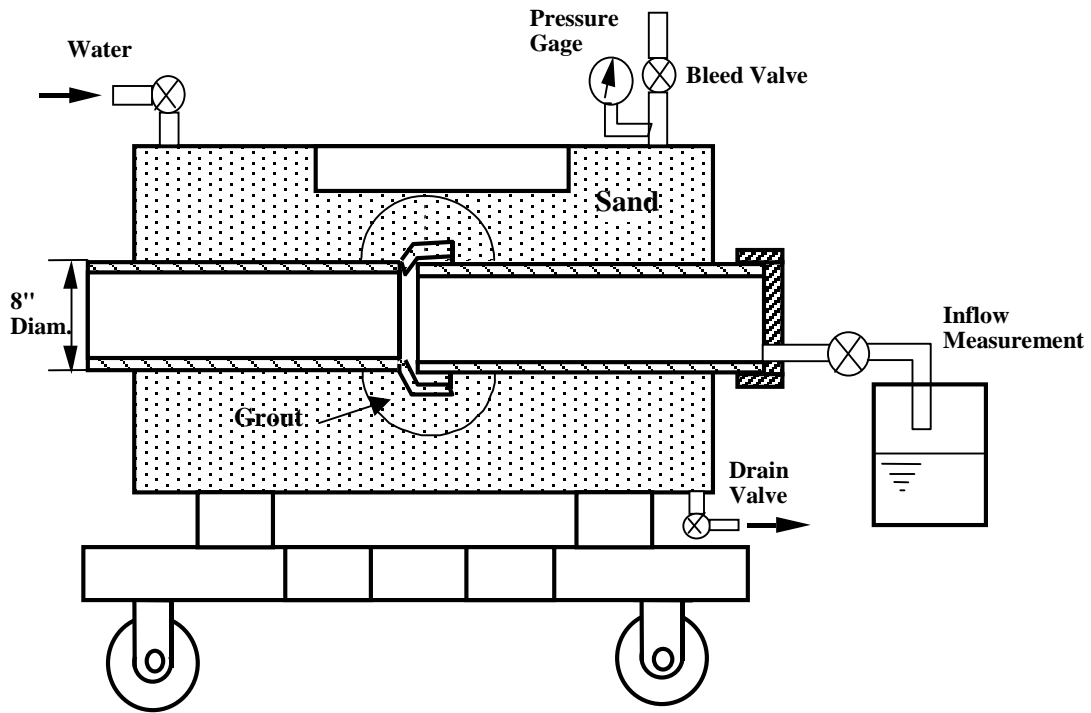
The tests are fully explained in the following sections, and details of the model test procedures are provided in Section 3.3.5.

3.3.1 Model Test 1: Leak Control in Pipe Joints

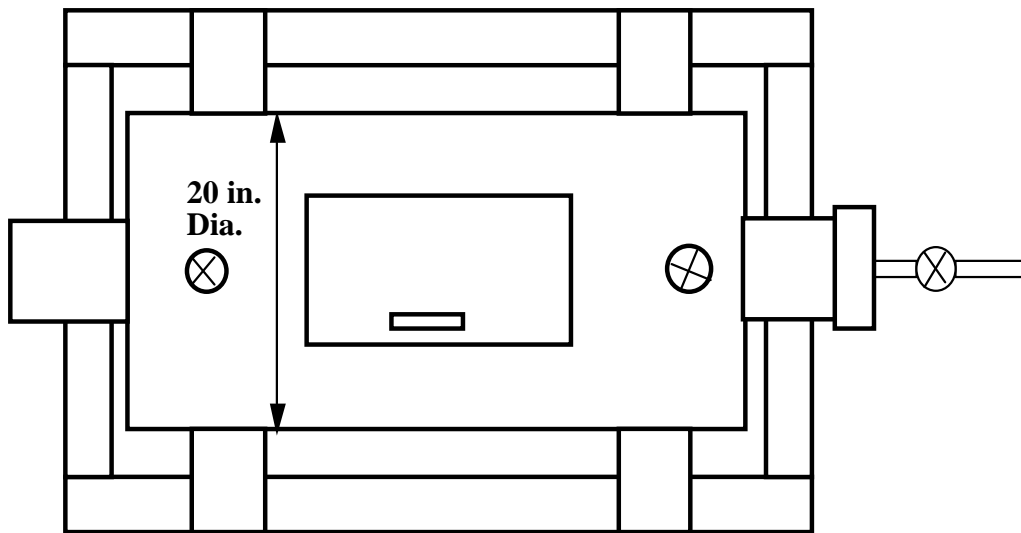
This model test simulates a situation in which grout is applied to an 8-in. (20-cm) diameter leaking pipe joint (Figure 3-3). The leaking pipe joint is placed in a cylindrical steel chamber filled with sand. The sand shall have the same specifications as described for the preparation of grouted sand specimens in Section 3.1.1.2. The steel chamber is 22.5 in. (57 cm) in diameter and 34 in. (86 cm) long, and both ends of the chamber have an opening of 8.5 in. (22 cm) for holding the pipe. This allows access to the leaking pipe joint from either end of the chamber. Valves on the outside of the test chamber enable the TO to saturate the sand and bleed the air from the system.

Procedure for preparing a pipe joint for Model Test 1:

- The chamber is placed vertically and the bottom end is sealed using a rubber gasket with one section of the pipe in place.
- The chamber is filled approximately halfway by freely dropping and lightly compacting sand. The spigot part of the pipe is put in place and the rest of the chamber is filled with sand.
- Once the chamber is filled with sand, the top cover is placed on the chamber using a rubber gasket to make the end watertight.
- The chamber is placed horizontally for the test. A calibration curve of the joint leak rate versus pressure shall be developed, as shown in Figure 3-4.
- The vendor has the choice of grouting the joint or having CIGMAT grout the joint per vendor instructions. The grouted joint is then tested for performance as detailed in Section 3.3.5.



(a) Elevation View



(b) Plan View

Figure 3-3. Model configuration for testing leak control at pipe joints (Model Test 1).

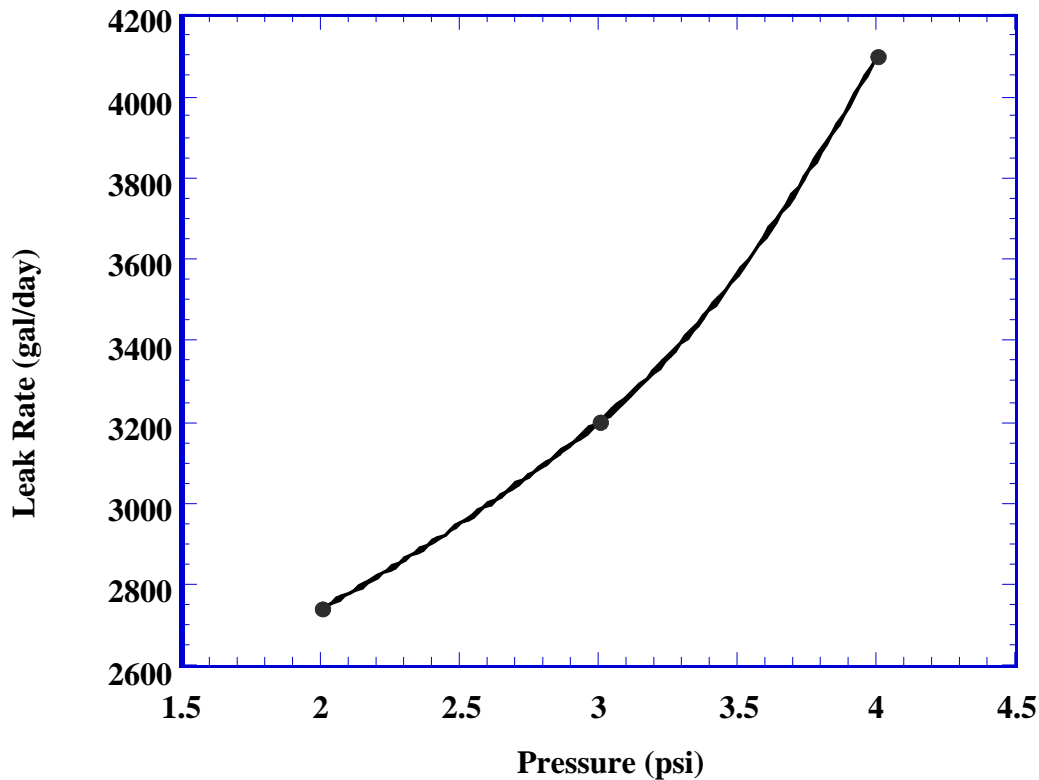


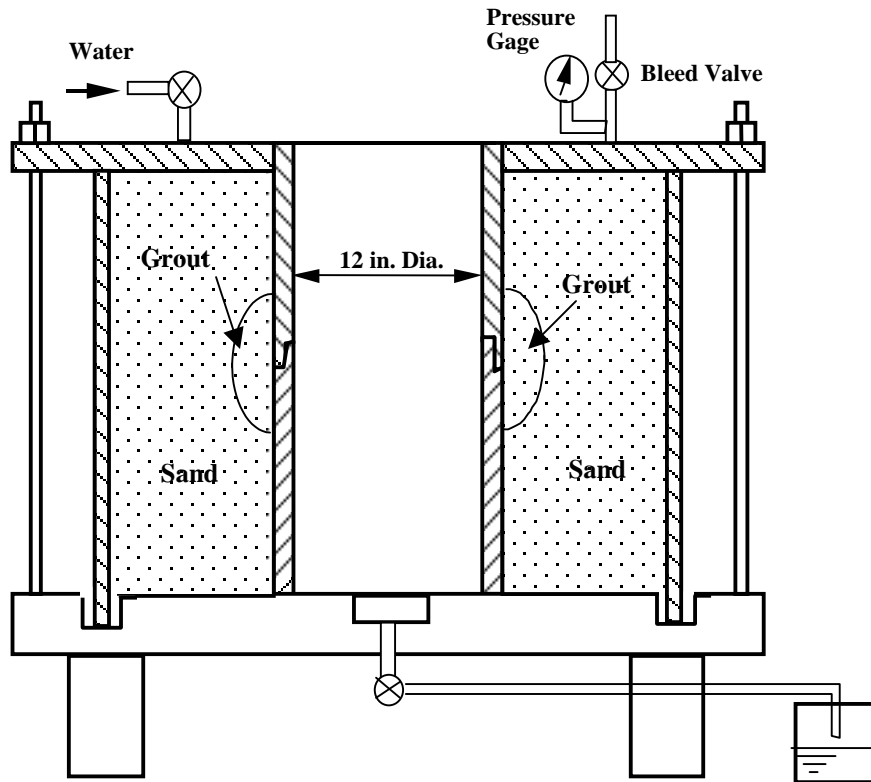
Figure 3-4. Typical calibration of Model Test 1: leak rate versus pressure.

3.3.2 Model Test 2: Leak Control in Manhole Joints

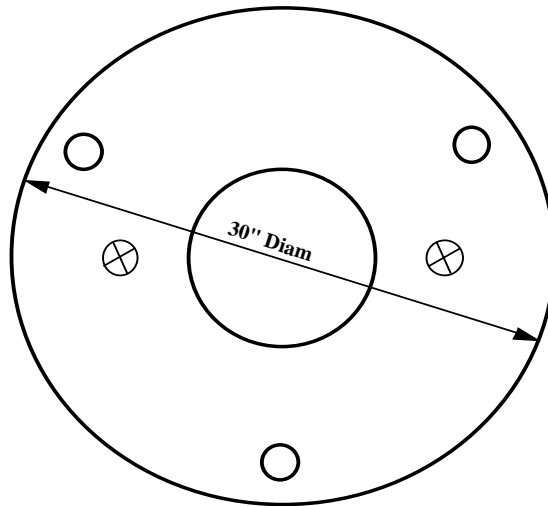
In order to simulate a leaking manhole joint, this model test (Figure 3-5) uses a 10- or 12-in. (25- or 30-cm) diameter vertical pipe. The pipe is enclosed in a cylindrical steel chamber 22.5 in. (57 cm) in diameter and 34 in. (86 cm) long, filled with sand. The only opening in the chamber (other than the valves) shall be from the top, leading into the vertical pipe. Valves on the outside of the test chamber enable the TO to saturate the sand and bleed the air from the system.

Procedure for preparing a manhole joint for Model Test 2:

- The chamber is filled by freely dropping and lightly compacting sand. The spigot part of the pipe is put in place in the test chamber and the rest of the chamber is filled with sand.
- The chamber remains in the vertical position for the test. A calibration curve of joint leak rate versus pressure shall be developed, as shown in Figure 3-4.
- The vendor has the choice of grouting the joint or having CIGMAT grout the joint per vendor instructions. The grouted joint is then tested for performance as detailed in Section 3.3.5.



(a) Elevation View



(b) Plan View

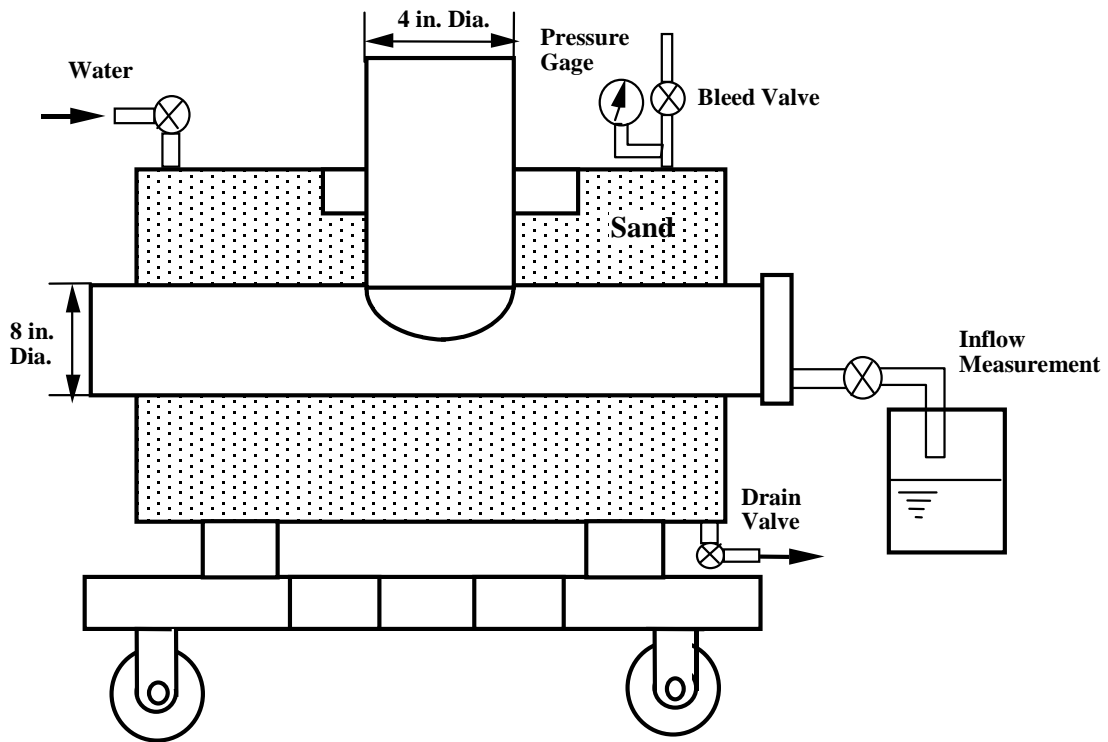
Figure 3-5. Model configuration for testing leak control in manhole joints (Model Test 2).

3.3.3 Model Test 3: Leak Control at a Lateral Joint

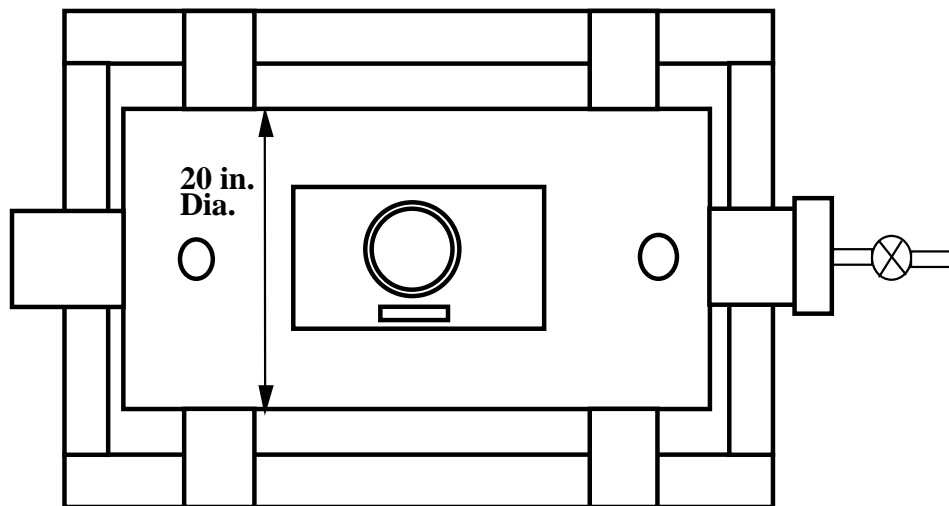
In order to simulate a leaking lateral joint, this model test (Figure 3-6) uses an 8-in. (20-cm) diameter main pipe with a 4-in. (10-cm) diameter lateral pipe. Both pipes are enclosed in a cylindrical steel chamber 22.5 in. (57 cm) in diameter and 34 in. (86 cm) long, filled with sand. Both ends of the chamber have a circular opening 8.5 in. (22 cm) in diameter for the main pipe, and the top of the chamber has a circular opening for the lateral, allowing access to the leaking joint from the main pipe and the lateral. Valves on the outside of the test chamber enable the TO to saturate the sand and bleed air from the system and to apply water under pressure to evaluate the effectiveness of the grout application.

Procedure for preparing a lateral joint for Model Test 3:

- The chamber is placed vertically and the bottom end sealed using a rubber gasket with part of the pipe in place.
- The chamber is filled approximately halfway by freely dropping and lightly compacting sand. The lateral pipe is then inserted in the main pipe and the rest of the chamber shall be filled with sand.
- Once the chamber is filled with sand, the top cover is placed on the chamber with a rubber gasket to make the end watertight.
- The chamber remains in the vertical position for the test. A calibration curve of joint leak rate versus pressure shall be developed, as shown in Figure 3-4.
- The vendor has the choice of grouting the joint or having CIGMAT grout the joint per vendor instructions. The grouted joint is then tested for performance as detailed in Section 3.3.5.



(a) Elevation View



(b) Plan View

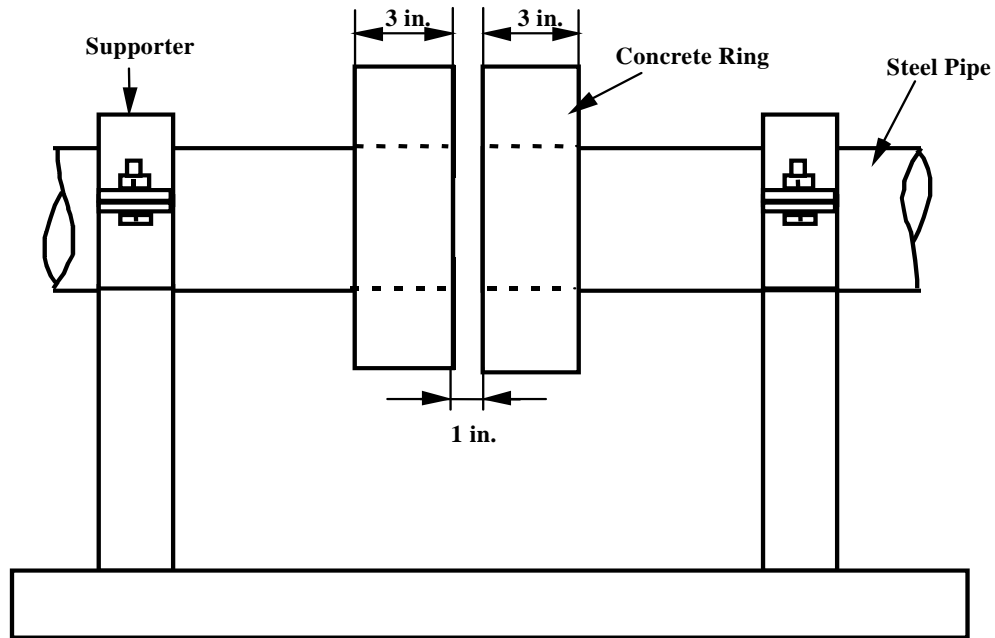
Figure 3-6. Model configuration for testing leak control at a lateral (Model Test 3).

3.3.4 Model Test 4: Concrete Leak Repair

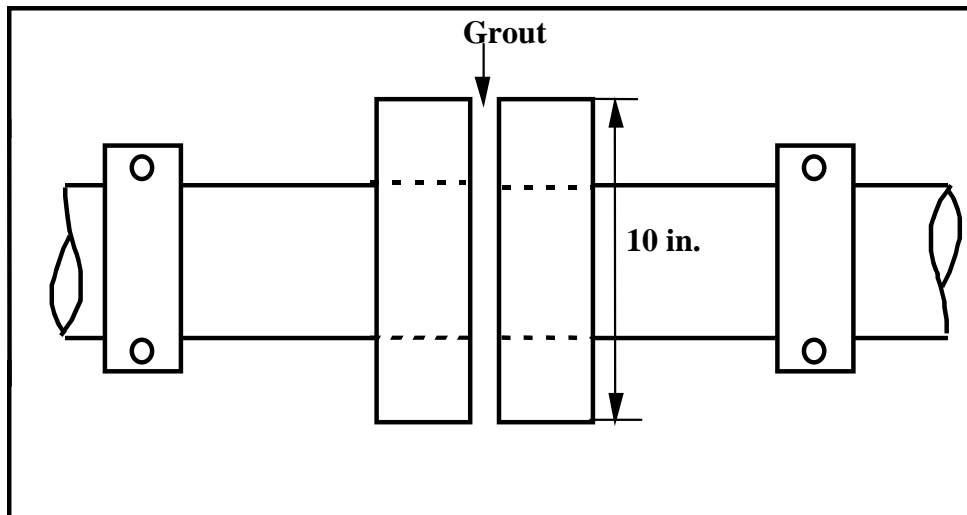
In order to simulate a leak in a concrete structure, this model test (Figure 3-7) shall use 10-in. (25-cm) diameter circular concrete disks with 6-in. (15-cm) openings at the center (each disk is donut-shaped). As a default, the two disks shall be placed one inch apart and grouted by the vendor. The vendor may, however, select the opening size. After the vendor-specified curing period, the grouted joint shall be subjected to hydrostatic pressure testing to determine the leak rate, as detailed in 3.3.5.

Procedure for preparing a concrete leak repair joint for Model Test 4:

- The gap between the concrete rings on the testing rig is set at the appropriate dimension, (one inch or as specified by the vendor).
- The vendor or CIGMAT applies the grout in the gap in accordance with the vendor's standard procedures.
- After the grout has cured, testing will commence using the procedures outlined in Section 3.3.5.



(a) Elevation View



(b) Plan View

Figure 3-7. Model configuration for testing concrete leak repair (Model Test 4).

3.3.5 Model Test Procedures

The testing procedure will be essentially the same regardless of the model test selected. Each model test shall be conducted in duplicate. Prior to grouting, each joint to be tested shall be calibrated in order to develop a characteristic leak rate versus pressure relationship (see

Figure 3-4). The grout shall be applied to wet sand (models 1 through 3) or wet concrete disks (model 4) by the vendor. CIGMAT personnel shall supervise the grouting procedures and pictures shall be taken of the joint/concrete disks prior to and after grouting. The time elapsed and volume of grout used during the grouting process shall be recorded. The time period following the application of the grout before testing is initiated shall be determined from the manufacturer's literature and is dependent on the penetrability and setting time of grout. During the grouting of the joints/cracks, at least ten grout samples shall be collected to test the setting time (Section 3.1.2.2), unit weight (Section 3.1.3.1), and compressive properties (Section 3.1.3.5) of the grouts. These analyses are in addition to those specified in Section 3.1.

Once the grouted joint(s)/concrete disks have cured per the manufacturer's instructions, they shall be subjected to the following regimen:

1. Apply hydrostatic pressure of 3 psi and hold for 5 minutes; then measure the leak rate using a graduated cylinder and a stopwatch.
2. Repeat Step 1 at a hydrostatic pressure of 4 psi.
3. Repeat Step 1 at a hydrostatic pressure of 5 psi.
4. Maintain saturated conditions for a period of one week. For model tests 1, 2, and 3, fill the chambers with water with no hydrostatic pressure. In model test 4, soak the joint with water.
5. Drain all water from the test chambers and allow them to stand for one week.
6. Fill the chambers with water and repeat Step 4.
7. Repeat Step 5.
8. Determine leak rates as described in steps 1 through 3.

The reported data shall include the characteristic leak rate versus pressure for each ungrouted joint; lapsed time and volume of grout used during the grouting process; lapsed time between grouting and commencement of testing; data for grout (setting time, unit weight, and compressive properties); initial leak rates at the three hydrostatic pressures; and the leak rates at the three hydrostatic pressures following the saturated/drained cycles.

At the end of each model test, the test chamber shall be emptied, any loose sand shall be carefully removed with a brush, and the grouted region shall be inspected and documented. During the inspection, the distribution of grout around the leaking joint/concrete crack shall be mapped. Photographs shall be taken of the grouted joints during and upon completion of this activity.

Chapter 4

Sampling and Analytical Procedures

Verification of grouts under ETV primarily consists of physical tests performed on prepared specimens as described in Chapter 3, “Experimental Design.” The outlined procedures identify the sampling locations and frequency required for each test.

Further sample preparation and analysis is required only for the leaching test, which is outlined in Section 3.1.5. Exposure water samples will be collected and analyzed, at a minimum, for total lead and TOC. Other analyses may be conducted based on the chemical composition of the tested grout. Tables 4-1 and 4-2 will be modified in the test plan prepared for each grout verification based on the analyses to be completed. The exposure water samples shall be representative grab samples collected from the exposure jar.

The sample handling, analysis and reporting shall be as outlined in Table 4-1.

Table 4-1. Handling Methods and Analyses for Collected Samples

Analysis	Method¹	Bottle Type and Size	Preservation, Holding Time	Reporting Detection Limit
Lead	SM 3310	Plastic, one 125-mL bottle	Cool to 4°C, pH<2 H ₂ SO ₄ , 28 days	0.3 mg/L
TOC	SM 5310 (B or C)	Glass, two 40-mL bottles	Cool to 4°C, pH<2 HNO ₃ , six months	1 mg/L

¹ *Standard Methods for the Examination of Water and Wastewater*, 20th Edition.

Samples shall be delivered to the analytical laboratory following appropriate chain of custody procedures, including use of chain of custody forms. Samples shall be logged in and refrigerated by the laboratory, as described in Table 4-1.

Table 4-2 describes the specific QC checks required for the analytical methods for total lead and TOC used in this project. These checks shall determine when corrective action is needed.

Table 4-2. Scheduled Instrument QC Checks and Corrective Actions for Analytical Methods

QC Procedure	Frequency	Acceptance Criteria	Corrective Action
Calibration curve	Every batch	$\pm 10\%$ of known sample	Find cause, repair, rerun before sample analysis
Analyze blank (deionized water)	Every batch	$\pm 10\%$ of calibration blank sample	Find cause, repair, rerun before sample analysis
Analyze standard	Every batch	$\pm 10\%$ of known sample	Find cause, repair, rerun before sample analysis
Dilute over range samples	When needed	$\pm 10\%$ of original	Find cause, repair, rerun previous samples
Matrix spike	Every batch	80–120% recovery of spike	Find cause, repair, rerun previous samples

Chapter 5

Quality Assurance Project Plan (QAPP)

The QAPP, which is part of the test plan, specifies procedures that shall be used to ensure data quality and integrity arising from the testing. Careful adherence to these procedures will ensure that the data generated from the testing will provide sound analytical results that will indicate the true performance of the grout, and form the basis for the report on the testing.

5.1 Quality Assurance Responsibilities

The TO, in preparing the test plan, shall be responsible for ensuring that the test plan and the QAPP properly implement the requirements of this protocol. The VO is responsible for review of the test plan to assure that all elements required by this protocol are properly addressed.

During testing, the TO shall be responsible for assuring that the elements contained in the test plan are complied with. Written or electronic records shall be maintained for calibrations, sample collection, and data manipulation. In grout testing, sources of error may include instrumentation drift or miscalibration; variations in the grout, sand, and/or substrate; systematic bias of measurements; and/or intrinsically inaccurate instruments. The quality of reference measurements is ensured by frequent instrumentation calibration in accordance with the manufacturer's instructions. The TO shall maintain documentation of instrument calibration.

5.2 Data Quality Indicators

The data obtained during verification testing must be sound for accurate conclusions to be drawn. For all measurement and monitoring activities conducted for grout verification, the VO and EPA require that the data quality parameters be established based on the proposed end-users of the data. Data quality parameters include four indicators of data quality: representativeness, completeness, precision, and accuracy.

5.2.1 Representativeness

Representativeness refers to the degree to which data accurately and precisely reflect the conditions or characteristics of the parameters. In verifications following this protocol, representativeness will be ensured by consistent data acquisition and sample collection (including sample numbering, timing of sample collection, sampling procedures, sample preservation, sample packaging, and sample shipping). Representativeness will also be ensured by using each method at its optimal capability to provide results representing the most accurate and precise measurements possible. Representativeness also implies collecting sufficient data during each operation to be able to detect changes in operation. To achieve this, the following actions will be taken:

Test Materials:

- Test Sand: The test sand batch shall be rejected if the particle-size distribution of the sand exceeds $\pm 20\%$ of the mean particle-size distribution value.

- **Test Concrete:** The test concrete batch shall be rejected if the unit weight and/or water absorption properties exceed $\pm 20\%$ of the mean values.

Laboratory Conditions:

- **Temperature and Humidity:** For those tests where temperature and humidity requirements are specified, temperature and humidity readings shall be recorded daily to ensure that laboratory conditions have not changed.

Equipment:

- **Proper operation:** This shall be verified every morning of active testing.

5.2.2 Completeness

Completeness refers to the amount of data collected from a measurement process compared to the expected amount to be obtained. For this ETV test plan, completeness refers to the proportion of valid, acceptable data generated using each method. The completeness objective for data generated following this protocol is 85%, as calculated by Equation 5-1.

$$\text{Completeness} = \left(\frac{n_{\text{valid and acceptable}}}{n_{\text{total}}} \right) \times 100 \quad (5-1)$$

5.2.3 Precision

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Analytical precision is a measurement of how far an individual measurement may deviate from a mean of replicate measurements. Precision is evaluated from analysis of field and laboratory duplicates and spiked duplicates. Duplicates will be collected at a frequency of one duplicate for every ten samples collected for the laboratory analyses discussed in Chapter 4. The laboratory will run duplicate samples as part of its QA program. The data quality objective for precision is based on the type of analysis performed.

The standard deviation (SD), relative standard deviation (RSD), and/or relative percent difference (RPD) recorded from sample analyses are ways to quantify precision. SD is calculated by:

$$\text{Standard Deviation} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}} \quad (5-2)$$

Where:

- \bar{x} = sample mean
 x_i = i th data point
 n = number of data points

Relative percent difference (RPD) is calculated by:

$$\text{RPD} = \left(\frac{|C_1 - C_2|}{\bar{C}} \right) \times 100\% \quad (5-3)$$

Where:

- C_1 = Concentration of the compound or element in the sample
 C_2 = Concentration of the compound or element in the duplicate
 \bar{C} = Arithmetic mean of the sample and the duplicate

As specified in *Standard Methods* (Method 1030-C), precision is specified by the standard deviation of the results of replicate analyses. For the various tests to be conducted by CIGMAT at its testing facility, precision will be measured by performing duplicate tests and evaluating the resultant data by calculating the SD, RSD, and RPD. Tables 3-1 and 4-1 provide the required number of duplicate tests for the various testing methods.

In situations where the testing procedures specify precision objectives (such as ASTM or *Standard Methods*), the specific precision objectives must be achieved in order for the test to be considered valid. For other situations where specific precision objectives are not required, the precision values shall be reported in the verification report.

5.2.4 Accuracy

For measurements that will be recorded as part of this study, accuracy refers to the difference between the measured reading and an established reference. In order to report accuracy, the instruments used during testing shall be calibrated as required by the analytical method, and the calibration records, which are maintained as a hard copy maintained in the laboratory, shall be made available.

Spiking a sample matrix with a known amount of a constituent and measuring the recovery obtained in the analysis is a method of determining accuracy. Using laboratory performance samples with a known concentration in a specific matrix can also monitor the accuracy of an analytical method for measuring a constituent in a given matrix. Accuracy is usually expressed as the percent recovery of a compound from a sample. The following equation will be used to calculate percent recovery:

$$\text{Percent Recovery} = [(A_T - A_i) / A_s] \times 100 \quad (5-4)$$

Where:

A_T = Total amount measured in the spiked sample

A_i = Amount measured in the un-spiked sample

A_s = Spiked amount added to the sample

During verification testing, the laboratory will run matrix spike samples at a frequency of one spiked sample for every 10 samples analyzed. The laboratory will also analyze liquid and solid samples of known concentration as lab control samples.

5.2.5 Measurements

Leaks in the model tests will be measured accurate to ± 2 mL. The weight and dimension during the grout and grouted sand tests will be measured to an accuracy of 0.1 g and 0.01 mm, respectively. The unit weight and strength will be measured to an accuracy of 0.5 lb/ft³ and 2 psi, respectively.

5.2.6 Analytical Quality Control

The quality control procedures for blanks, spikes, duplicates, calibration of equipment, standards, reference check samples and other quality control measurements will follow the guidance of EPA methods and CIGMAT SOPs. Table 5-1 shows the quality control limits that will be used by the laboratory for these analyses to ensure compliance with the data quality indicators for accuracy and precision. Field and laboratory duplicate analyses will be performed at a frequency of one duplicate per ten samples collected. Samples will be spiked for accuracy determination at a frequency of one per fifteen samples analyzed by the laboratory. Accuracy and precision will be calculated for all data using the equations presented in sections 5.2.2 and 5.2.3.

Table 5-1. Summary of Analytical Accuracy and Precision Limits

Analysis	Accuracy (% recovery)	Precision (RPD)
Lead	70–130	0–30
TOC	80–120	0–20

Note: If additional analytical parameters are added to the testing procedures, accuracy and precision limits shall be specified in the test plan.

RPD: Relative percent deviation

Chapter 6

Data Reporting, Data Reduction, and Data Validation

The TO (CIGMAT) is responsible for managing all the data and information generated during the testing program. To maintain quality data, specific procedures shall be followed during data reporting, reduction, and validation. These procedures are discussed below.

6.1 Data Documentation

All field and laboratory activities shall be thoroughly documented by the use of field logbooks, project approval/chain of custody sheets, laboratory notebooks and bench sheets, and instrument records.

A field logbook shall be maintained at the test facility. Daily activity entries shall be made in the logbook documenting operating conditions, observations, and maintenance activities, if any. Each sample collected shall be noted in the logbook and any other pertinent information shall be recorded. Completed pages in the logbook shall be signed and dated.

Original project approval and chain of custody forms shall accompany all samples sent to the analytical laboratory and will be maintained by the TO. The laboratory shall produce a final data report that includes all chemical test results, physical measurements, QA/QC data for blanks, accuracy (recovery), precision (percent difference), and lab control or matrix check samples. Any deviation from standard protocol shall be discussed in a narrative and any data that does not meet the QA/QC requirements shall be flagged. A narrative shall be prepared discussing the findings of any corrective action.

The laboratory shall maintain all logbooks, bench sheets, instrument printouts, and similar materials. The TO shall make these records available for inspection by the VO or EPA upon request.

6.2 Data Reduction

Data reduction refers to the process of converting raw test results into useful data for selecting grout material for wastewater system maintenance and concrete repair. Data shall be obtained from logbooks, data sheets, and computer outputs. While reduced data will be officially reported to the VO upon completion of each evaluation, all raw data shall also be made available to the VO for the QA review of the project and for record keeping.

6.3 Data Validation

The person performing each test shall verify the completeness of the appropriate data forms. The TO Director shall review laboratory logbooks and data sheets on a regular basis to verify completeness. The TO technical staff shall regularly inspect testing equipment and keep it in working order.

6.4 Verification Report

All the data collected during the testing shall be reported as indicated in Chapter 3, processed and analyzed as outlined in Chapter 5, and summarized in a verification report and verification statement following ETV Water Quality Protection Center guidelines.

The verification report shall thoroughly present and discuss the findings of the verification test. It shall contain all raw and analyzed data, all QA/QC data sheets, a description of all types of data collected, a detailed description of the testing procedure and methods, results and QA/QC results. The verification statement shall present a condensed summary of the testing procedure and findings. It is expected that the verification report will contain the following main sections.

- Verification Statement
- Notice
- Forward
- Contents
- Abbreviations and Acronyms
- Introduction and Background
- Testing Procedures and Methods
- Testing Results
- Quality Assurance/Quality Control Summary
- Glossary
- References
- Appendices
 - Raw Data and Testing Logs
 - Laboratory Standard Operating Procedures
 - Test Plan
 - O&M Manual

Chapter 7 Assessments

7.1 Audit Reports

The TO Director or designee shall perform at least one QA inspection of the test facility laboratories during the evaluation of a grout and shall document any and all findings in an audit report, which will be submitted to the EPA Project Officer, EPA Program Quality Assurance Manager, and VO Program Manager for review. The VO Program Manager, Manager, QA and Safety, or other VO designee shall also conduct a technical system audit and a performance evaluation audit of measurement systems used in testing at least once during the verification testing period for a given technology.

7.2 Corrective Action Plan

The test plan shall include the predetermined acceptance limits, the corrective action to be initiated whenever such acceptance criteria are not met, and the names of the individuals responsible for implementation. Routine corrective action may result from common monitoring activities, such as:

- Performance evaluation audits; and
- Technical systems audits.

Ultimate responsibility for project QA/QC during implementation of this test plan rests with the verification organization, specifically with the VO Project Manager, with appropriate input from the VO QA/QC Manager. However, immediate QA/QC for individual tasks (e.g., sample collection, handling, preparation, and analysis) rests with the individuals and organization performing the task at hand. The VO Project Manager will coordinate oversight and/or audits of these tasks with the TO Project Manager to ensure that the test plan is being executed as written, and that nonconformances are appropriately reported and documented.

Corrective action shall be taken whenever a nonconformance with the test plan occurs. Nonconformances can occur within the realm of sampling procedures, sample receipt, sample storage, sample analysis, data reporting, and computations.

Chapter 8

Safety Considerations

Grouting the joints for the model tests shall be done at the covered test facility at the University of Houston, which has adequate ventilation. Grout and grouted sand specimens for testing shall also be prepared in CIGMAT laboratories. The research personnel and technicians on-site will take all necessary precautions to ensure safety and compliance with local and federal regulations.

CIGMAT maintains a health and safety plan, which shall be made available to personnel involved in this project. Adherence to the health and safety plan shall be ensured throughout the duration of the project.

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Glossary

Accuracy—A measure of the closeness of an individual measurement or the average of a number of measurements to the true value and includes random error and systematic error.

Batch—The number of samples analyzed during a period in which an instrument was operated continuously.

Bias—The systematic or persistent distortion of a measurement process that causes errors in one direction.

Comparability—A qualitative term that expresses confidence that two data sets can contribute to a common analysis and interpolation.

Completeness—A qualitative term that expresses confidence that all necessary data have been included.

Precision—A measure of the agreement between replicate measurements of the same property made under similar conditions.

Quality Assurance Project Plan—A written document that describes the implementation of quality assurance and quality control activities during the life cycle of the project.

Representativeness—A measure of the degree to which data accurately and precisely reflect a characteristic of a population parameter at a sampling point, or for a process or environmental condition.

Room Temperature— $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Relative humidity under room temperature conditions is expected to be $50\% \pm 5\%$. This definition of room temperature shall be used for all testing conducted under the ETV *Test Plan for the Verification of Grouts*, even if a referenced SOP or standard defines the term differently.

Standard Operating Procedure—A written document containing specific procedures and protocols to ensure that quality assurance requirements are maintained.

Technology Panel—A group of individuals with expertise and knowledge in grouts.

Test Plan—A written document that describes the procedures for conducting an evaluation for the application of a grout material; the quality assurance project plan is an important part of the test plan.

Verification—To establish the evidence on the performance of grouts under specific conditions, following a predetermined test plan.

Verification Report—A written document containing all raw and analyzed data, all QA/QC data sheets, descriptions of all collected data, a detailed description of all procedures and methods used in the verification testing, and all QA/QC results. The test plan shall be included as part of this document.

Verification Statement—A document that summarizes the verification report reviewed and approved by the verification organization and EPA.

Appendix A CIGMAT Procedures

CIGMAT CH 2-01
CIGMAT CT 3-00
CIGMAT GR 1-00
CIGMAT GR 2-02
CIGMAT GR 3-00
CIGMAT GR 4-00
CIGMAT GR 5-00
CIGMAT GR 6-02
CIGMAT GR 7-02
CIGMAT GS 1-02
CIGMAT PC 2-99

(Questions about CIGMAT procedures may be directed to CIGMAT at the University of Houston)

Appendix B Vendor Data Sheet

GROUT VENDOR DATA SHEET (Attach Additional Sheets if Necessary)**Grout Product Name:** _____**Grout Product Manufacturer Name and Address:** _____
_____**Grout Type:** _____ **Chemical Formula:** _____

TESTING METHOD	MANUFACTURER'S RESULTS
Type of Resin, Initiator and or Promotor	
Grout Mix (by weight or volume)	
Resin Viscosity (ASTM _____)	
Flash Point (ASTM D 93/_____)	
Tensile Adhesion to Concrete and Clay Brick (ASTM _____)	
Chemical Resistance (ASTM _____) (NaOH, 3% H ₂ SO ₄ or others)	
Volatile Organic Compounds - VOC's (ASTM _____)	

WORKER SAFETY	RESULT/REQUIREMENT
Flammability Rating	
Known Carcinogenic Content	
Other hazards (corrosive)	
MSDS Sheets Availability	

ENVIRONMENTAL CHARACTERISTICS	RESULT/REQUIREMENT
Heavy Metal Content (w/w)	
Leaching from Cured Grouts	
Disposal of Cured Grouts	

DATA SHEET ON PROPERTIES OF GROUT (Continued)

APPLICATION CHARACTERISTICS	RESULT/REQUIREMENT
Minimum Application Temperature	
Maximum Application Temperature	
Minimum Cure Time before Immersion into Service	
Type of Preparation Before Grouting	
Grouting Pressure	

VENDOR EXPERIENCE	COMMENTS
Length of Time the Grout in Use	
Applicator Training & Qualification Program	
QA/QC Program for Grouts in the Field	

ADDITIONAL COMMENTS (Including Case Studies on Performance)