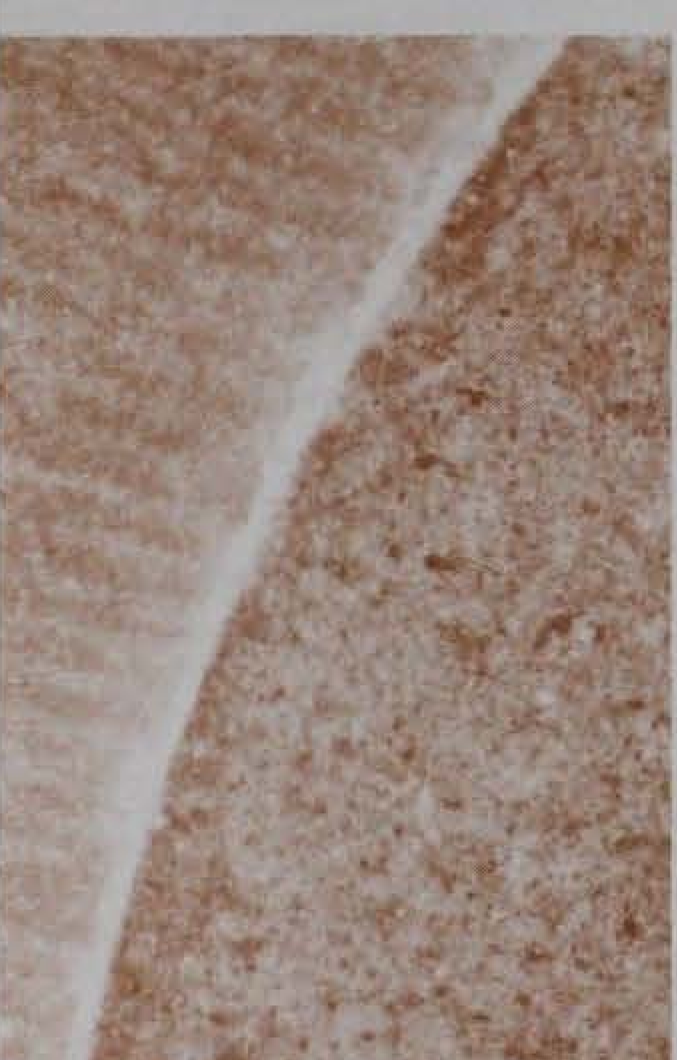
US Army Corps
of Engineers



STRUCTURES LABORATORY



MISCELLANEOUS PAPER SL-85-6

# DEVELOPMENT OF A SANDED NONSALT EXPANSIVE GROUT FOR REPOSITORY SEALING APPLICATION

by

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Waterways Experiment Station, Corps of Engineers
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Final Report





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A sanded version of expansive BCT 1-FF nonsalt grout was developed for use in the 1984 Terra Tek test in Salt Lake City, Utah. For that reason this grout is also known as TT84. Ability to maintain workability (flow) over a 2-hr time period was required in addition to adequate strength, adequate expansion, and low permeability of the hardened material. Laboratory testing also included

(Continued)

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20. ABSTRACT (Continued).

sealing and exposure to elevated temperature as variables for expansion testing; permeability testing of a combined grout-anhydrite specimen to evaluate this contact surface was also done.

Since the test specimen did not perform as well as expected in the Terra Tek test, a fragment of that grout was also examined in the laboratory.

All test data generated at WES were satisfactory for this grout.

Concern was raised about whether in field use the compressed wall rock will elastically deform so as to accommodate slight contraction of the expansion grout mixture that will probably occur due to cooling after the rise in temperature due to heat of hydration or shrinkage due to drying or both in an actual field installation.

It was recommended that periodic monitoring for expansion be continued and should be started to follow changes, if any, in phase composition and microstructure of the grout with time.

## Preface

This report was prepared for the US Department of Energy (DOE) under continuing contract DE-AI97-81ET 46633. It was a milestone for FY 84 which was prepared as a draft report in September 1984; it has been revised to include additional data.

Mr. Steve Webster of the DOE-Columbus was Project Manager when this report was being prepared for publication.

This report was prepared in the Concrete Technology Division (CTD) of the Structures Laboratory (SL) of the US Army Engineer Waterways Experiment Station (WES) by Mr. A. D. Buck under the direction of Mr. J. M. Scanlon, Chief, CTD, and Mr. B. Mather, Chief, SL. Mr. Buck was Project Leader in the CTD and prepared this report.

COL Robert C. Lee, CE, was Commander and Director of WES during the conduct of this study and the preparation of this report. COL Allen F. Grum, USA, was Director of WES during publication. Mr. Fred R. Brown and Dr. Robert W. Whalin were Technical Directors.

# Contents

## Conversion Factors, Non-SI to SI (Metric) Units of Measurement

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

| Multiply | By | To Obtain |
|---|---|---|
| cubic feet | 0.02831685 | cubic metres |
| inches | 25.4 | millimetres |
| Fahrenheit degrees | 5/9 | Celsius degrees or Kelvins* |
| feet | 0.3048 | metres |
| miles (US statute) | 1.609347 | kilometres |
| pounds (force) per square inch | 0.006894757 | megapascals |
| pounds (mass) per cubic foot | 16.01846 | kilograms per cubic metre |

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: C = (5/9)(F - 32). To obtain Kelvin (K) readings, use: K = (5/9)(F - 32) + 273.15.

# DEVELOPMENT OF A SANDED NONSALT EXPANSIVE GROUT FOR REPOSITORY SEALING APPLICATION

## Background

1. The Concrete Technology Division (CTD) of the Structures Laboratory (SL) of the U. S. Army Engineer Waterways Experiment Station (WES) is doing research and development work for the U. S. Department of Energy (DOE) for its repository sealing program. For this work the DOE is considering a repository site in bedded or dome rock salt. The rock called "salt" consists primarily of the mineral halite (NaCl). Other work is underway to evaluate other rock types and locations. Task 84-1 for FY 84 is to develop a nonsalt grout considered suitable for sealing if the actual host rock contact is not salt. This report covers the development of a grout mixture for immediate use in a large-scale test at the Terra Tek facility in Salt Lake City; this grout mixture is designated TT84; it is a sanded version of the nonsalt grout BCT 1-FF and differs from sanded salt grout TT-83 mainly in the absence of salt.

## Materials

2. The following materials were used in the grout mixture that was developed, and most of these were also used in the sanded salt grout (TT83) developed in 1983:

a. Cement. This was Class H (high sulfate resistant) portland cement from the Lone Star Industries, Inc., Maryneal Plant at Sweetwater, Texas. Work was started with sample RC-881 and was completed with new sample RC-911.

b. Fly ash. This was a Class C ash from the Harrington Plant of Southwest Public Service in Amarillo, Texas; the serial No. was AD-592(2).

c. Plaster. This was plaster of Paris (calcium sulfate hemihydrate) obtained under the trade name Cal-Seal. A new sample was obtained during the course of this work.

d. Chemical admixtures.

(1) Melgran 1 and 2. These are trade names for melamine-based high-range water reducers (HRWR) that differ only in particle size. Equal amounts of these two are combined to a total 0.75 percent of the cementitious materials (cement, fly ash, plaster) by mass.

(2) De-Air No. 1. This is the trade name for a material used to minimize foaming tendencies over extended periods of mixing; dosage was 0.25 percent by mass of the cementitious materials.

e. Fine aggregate. Two fine natural sands and a silica flour were used as combined fine aggregates. They were as described below:

(1) NTS 20-40 sand. The original source of this material is Crystal Silica Co. of Oceanside, California, but the material used at the CTD was obtained at the Nevada Test Site (NTS). It is silica sand characterized by being finer than a 1.18-mm (No. 16) sieve; most of it is retained on a 600-μm (No. 30) sieve.

(2) D-30 silica sand. This is a trade name for material commonly used in oil field sealing applications. Our past supply came from a supplier in southeastern New Mexico. However, a new supply was obtained from the same supplier at Hattiesburg, Mississippi, during the course of this work and was given serial No. AD-791. Most of this material is retained on the 150-μm (No. 100) sieve.

(3) Silica flour. Trade name for ground quartz ($SiO_2$) usually obtained locally as material supplied for the oil industry in Mississippi and Louisiana. A new supply during the course of this work was obtained in Hattiesburg, Mississippi, and was given SL serial No. AD-790. It is characterized by being finer than a 300-μm (No. 50) sieve with most of the material retained on a 150-μm (No. 100) sieve.

Developmental work

3. The new mixture was basically developed by leaving out the salt from the sanded salt grout TT83. This meant that the viscosity of the new grout was significantly less. General control parameters were for the grout to have a flow time of approximately 30 sec and remain workable for at least 2 hr; hardened properties were for it to be expansive and to have low permeability (a few microdarcies or less). If a trial mixture lacked the proper flow or workability, it was discarded without having test specimens cast and a batch number assigned. This effort started in March 1984.

4. The suitable TT84 mixture was designated batch 3; 1-cu ft batches of it were cast at the CTD on 16 and 18 April 1984 (batches 3 and 3A), and specimens were made for testing. A modification containing more plaster to see if it would increase expansion was also made on 18 April, and a few bars were cast for expansion testing; it was identified as batch 4 and is not the TT84 grout that was used.

5. Due to time limitations the decision to use the batch 3 composition as the grout for the Terra Tek test had to be made when laboratory test specimens were only a few weeks old. Therefore, about the second week in May 1984, Don Walley of the CTD prepared five separate dry batches of TT84 grout. Three of these were immediately sent to Terra Tek; one was sent to the Materials Research Laboratory (MRL) of the Pennsylvania State University (PSU) for such use as Dr. Della Roy chose, and the final sample was retained in the CTD. This latter sample was batched on 14 May 1984, and specimens were cast and identified as batch 3B.

6. Messrs. Walley and Winters of the CTD went to Terra Tek on 17 May 1984; they performed a dry-run batching test with one of the three TT84 samples on 18 May 1984; unhardened properties were as they had been in the CTD and four 4- by 8-in. cylinders were cast and later returned to the CTD for testing as needed. On 21 May 1984, they mixed the actual test batch at Terra Tek

and assisted in placing it for the 1984 test which was to start 28 days later (18 June). Three 4- by 8-in. cylinders from this batch were sent to the CTD. This action of having CTD staff members actively involved in this batching was taken to try to avoid the flow-time problem that had occurred during the 1983 test with the TT83 sanded salt grout. The word that was received indicated that the TT84 grout had the expected properties when mixed for the Terra Tek test.

Samples

7. The following specimens were cast in the CTD from the batch 3 and 3A mixtures made on 16 and 18 April 1984:

a. Twenty-three bars for expansion testing.

b. Fifteen 2-in. cubes for compressive strength determinations.

c. Three 3- by 6-in. cylinders. These were later used for determination of compressive strength at their 56-day age.

8. Batch 3B specimens cast in the CTD on 14 May 1984 were as follows:

a. Twenty bars for expansion testing; two were defective so 18 were tested.

b. Thirty 2-in. cubes for compressive strength determinations through 1 year.

c. Three 3- by 6-in. cylinders for use as needed.

d. One simulated borehole (SBH) sample for permeability testing.

A short piece (approximately 3 in.) of nominal 4-in.-diameter anhydrite ($C\bar{S}$) core from the 4317-ft depth of hole AEC-7 (southeast New Mexico) had been sawed longitudinally and placed in a cardboard cylinder mold reinforced with metal hose clamps; grout was cast against this sawed surface to make this SBH sample. After the grout had been allowed to harden overnight, the specimen was removed from the cardboard mold and placed in a Hassler cell under about 300-psi confining pressure. It was then tested for permeability at 200-psi driving pressure at ages of 1, 2, 6+, 7+, 8, and 9 weeks.

9. As indicated earlier, this batch 3B was one of the five dry batched earlier in preparation for the actual test at Terra Tek in Salt Lake City.

10. Batch 4 specimens cast in the CTD on 18 April consisted of three expansion bars that contained extra plaster. This was not the TT84 mixture.

11. Seven 4- by 8-in. grout cylinders from the dry run (18 May 1984) and test batch (21 May 1984) TT84 mixtures mixed at the Terra Tek facility were returned to the CTD. They are identified as:

a. CL-43 GRT-(2 through 5); these are the four 18 May cylinders.

b. CL-43 GRT-(6 through 8); these are the three 21 May cylinders.

They were received in the CTD when 16 to 19 days old and were left in their sealed plastic cylinder molds in laboratory air until they were 56 to 59 days old. At that time the tops of all but cylinders 2 and 6 were removed, and the cylinders were placed in the moist storage room at about 73° F. Cylinders 2 and 6 were left sealed and stored as before. A wedge-shaped portion of the grout that had been placed in contact with anhydrite for the Terra Tek test was received 17 August 1984 from Mr. R. Lingle of Terra Tek. It was approximately 5 in. high and 4 in. deep and showed some of the surface that had been in contact with the rock plus two fracture surfaces through the grout.

Test procedure

12. The new cement sample, RC-911, was analyzed.

13. The new sample of "Cal-Seal" plaster was analyzed. This included examination by X-ray diffraction (XRD) to determine actual phase composition.

14. The grading, absorption, and specific gravity of the three fine aggregates were determined.

15. Compressive strength of 15 cubes and 3 cylinders was determined through 56 days for the mixtures cast 16 and 18 April 1984. This exhausted the supply of specimens for this test. Similar data through 56 days were determined for the batch 3B data; this testing is scheduled to continue through at least the 1-year age. It could be extended by testing fewer than three cubes at each test age.

16. Expansion data for 21 bars from batches 3 and 3A grout were determined through 90 days, and this testing will continue. Testing environments included two temperatures (approximately 73 and 86° F) and moist or sealed storage. Sealing was done by coating bars with wax and storing them dry or over water. In general, this sealing procedure was not satisfactory as most specimens started shrinking after a couple of weeks; this was taken to mean the bars dried due to an imperfect seal and therefore shrank in response to this drying.

17. Expansion data for 18 bars from the batch 3B grout were determined through 56 days and will be continued. As before, they included the same two temperatures and moist or sealed storage. The sealing procedure was changed to try to obtain a better seal. After initial coverage with plastic while in moist storage for about 24 hr to harden, the bars were demolded. They were then tightly wrapped in plastic and taped with the metal inserts free. The bars were weighed each time a length measurement was made in an effort to evaluate seal effectiveness.

18. The SBH sample was demolded on 15 May 1984 when it was 1 day old. It was immediately photographed and placed in a Hassler cell, and confining pressure

of 300 psi was applied to maintain the grout-rock contact; this pressure was not released or reduced until testing had stopped. A driving pressure of 200 psi was applied periodically to determine permeability. After the permeability test at the 63-day age the specimen was removed from the Hassler cell and thus from any confining pressure. The specimen was intact at this time and did not show any tendency to separate along the grout to rock contact. The sample was inspected and photographed at this time. Shortly thereafter it did separate along the contact surface. Inspection of this exposed surface showed the presence of a thin layer (<1 mm thick) of soft powdery white material over most of the rock surface and a small amount of the grout surface; some of this material was probably also on the end of the specimen exposed to the driving force of the water. Some of this white material was removed from the contact surface by scraping and was examined by XRD. A small portion of it was placed in dilute hydrochloric (HCl) acid and observed for reaction with a stereomicroscope; another portion was prepared as an immersion mount and examined with a polarizing microscope. The former contact surfaces of grout and anhydrite were photographed as was the companion untested anhydrite surface.

19. The fragment of grout from the Terra Tek test was inspected both visually and using an ultraviolet (UV) light; since a fluorescent dye had been used in the water used for the permeability test, it was easy to find where this water had been. The surface that had been in contact with anhydrite and a fracture surface were photographed.

20. Since there had been almost immediate passage of water when pressure was applied for the Terra Tek permeability test, a 4- by 8-in. cylinder of this TT84 grout (CL-43 GRT-7) was tested for permeability with 300-psi confining pressure and 200-psi driving pressure for 14 days; the confining pressure was then increased to 700 psi for four additional days for a direct simulation of part of the Terra Tek test. The specimen was 80 days old when testing was started.

## Results

21. Tables 1, 2, and 3 show analytical data for cement RC-911, plaster, and the three aggregates. XRD indicated plaster (calcium sulfate hemihydrate) to be the only form of calcium sulfate that was detectable; there was a small amount of quartz. Table 3 shows the NTS 20-40 sand being coarsest and silica flour (AD-790) being finest. D-30 (AD-791) sand is intermediate in grading. All three are siliceous materials. The main difference in them aside from grading is the reported high absorption (11.8 percent) of the finer silica flour. Since the individual quartz particles in the silica flour would have no or essentially no absorption, it is likely the reported high absorption for it is really due to failure to obtain a true saturated surface dry condition for this material during the absorption test. Such difficulty is fairly common with materials that are made up of especially small particles like this silica flour. If that is the case, the gradation is actually the only significant difference in the three fine aggregates.

22. The actual mixture proportions for TT84 grout are shown in Table 4 along with other information; the limited range of flow over a 2-hr period of intermittent mixing is considered significant and satisfactory.

23. Compressive strengths of batch 3 and batch 3B TT84 grout specimens cast 16 April and 14 May 1984 are shown in Table 5. Since the strength of this grout exceeds 3,000 psi by 4 days, 10,000 psi by 56 days, and 15,000 psi after 1 year, this parameter is considered more than adequate.

24. Expansion data for the TT84 grout batches are shown in Tables 6 and 7 for different temperatures and storage conditions. These show that this is an expansive mixture with restrained expansions of about 0.1 percent by 28 days and about twice as much unrestrained expansion when stored in a favorable environment (moist) at about 73° F. As can be seen by study of these tables, the data for expansion of sealed bars (i.e., mass curing) are quite variable as they range from shrinkage to expansion over 0.2 percent. The values shown in Table 6 for seals obtained by coating bars with wax should be ignored as unsatisfactory due to drying and shrinkage. All of the batch 3B bars were also weighed each time a length change measurement was made. Sealing had been improved. These data showed progressive increase in mass during exposure to moist storage conditions. Since bars 10, 11, and 12 showed essentially no weight loss or gain during sealed storage at 73° F over 56-days age, the seal was probably effective; length change data for bars 11 and 12 show expansion, but it is erratic and less than that in moist storage. The weight change data for the six sealed bars stored over water at 86° F (Table 7) showed slight gain with time; length changes were positive but erratic and less than for moist storage. The following observations seem warranted from study of the data:

a. Effective sealing of these bars is difficult and generally was not successful.

b. For those few instances where sealing was probably achieved, there was expansion but it was less than for corresponding moist storage.

c. The effect of temperature elevated to about 86° F as seen in Table 6 for bars 3, 4, 24, and 28 shows it inhibited but did not stop expansion.

25. The attempt to evaluate the effect of added plaster on expansion (batch 4) was done using bars sealed by waxing. As already stated, this proved unsuccessful so the data are not shown.

26. Table 8 shows permeability data for the SBH sample that contains the TT84 grout to anhydrite contact and for a sample of TT84 grout (CL-43 GRT-7). Permeability of the SBH sample was extremely low for the first 43 days. It then increased abruptly from 3.5 to 42 microdarcies and increased to 108 at the 63-day age. The water for this permeability test did not have any calcium sulfate added to it. While the basic grout mixture contains about 80 times more plaster than will go into solution with water at 20° C by calculation, it is possible that the test water may have dissolved some anhydrite since sulfate ions from the plaster probably were not available at the test surface when permeability testing was done. Figures 1 and 2 show several views of the SBH sample before and after permeability testing. This sample opened along its contact surface about 2 weeks after testing had been completed. The appearance of these contact surfaces and of the matching surface of untested rock is shown in Figure 3. It is obvious there was change

as development of a thin white powdery material on the contact surface. XRD showed this reaction material was mostly anhydrite plus a trace of calcite and gypsum. The combination of increased permeability after 43 days, the obvious change along the contact surface, and the composition of this reaction product suggest that water slowly worked its way along the contact surface by dissolving anhydrite which later precipitated along the contact with space for some passage of water under pressure (i.e., measurable permeability). It is known from other work (unpublished) that there is a chemical reaction at the contact surface of this or similar grout to anhydrite with formation of gypsum or anhydrite. While the source of calcium sulfate is probably the rock itself, there is a possibility that the plaster in the grout could contribute calcium and sulfate ions. The TT84 grout specimen itself showed essentially no permeability after 18 days of testing (Table 8); this was as expected.

## Discussion

27. As indicated, a workable sanded, nonsalt, grout was developed and used in the 1984 Terra Tek test. One problem was trying to determine expansion levels when bars were sealed to prevent change in moisture levels and the effects of elevated temperature (approximately $86^{o}$ F). As indicated, it is believed from the data that were obtained that both sealing, when effective, and elevated temperature reduce but do not prevent expansion. Since the likelihood of there being a truly dry environment in actual practice is probably slight, this effect of moisture may not be significant. Study of the effect of temperature should probably be continued to verify present data.

28. The grout fragment from the Terra Tek test is shown in Figure 4. It shows vertical flow channels along the grout surface that was in contact with anhydrite and dense impermeable grout on a fractured surface. The basis for this statement is the presence or absence of the fluorescent dye that was used as seen with UV light and examination of the flow channels with a stereomicroscope. Informal telephone conversations with Messrs. Lingle and Bush of Terra Tek and Dr. Licastro of PSU indicated that their samples of the same TT84 grout as cylinder CL-43 GRT-7 all showed similar low or nondetectable permeabilities. The SBH sample tested here showed similar low permeability until the integrity of the contact was destroyed by limited dissolution of the rock after 43 plus days of testing. Testing of an SBH sample for C. W. Gulick, Jr. of Sandia National Laboratories at about the same time showed even lower permeability. The configuration was different with a hole in some of the same anhydrite rock being filled with a nonsanded version of TT84 grout. The significant difference was use of 2000- rather than 300-psi confining pressure. His specimen showed low permeability (approximately 1 microdarcy) until testing was stopped after 83 days. The significance of all this seems to be as follows:

a. The TT84 grout has low permeability as expected.

b. Small (approximately 4- by 4-in.) cylindrical SBH samples of grout and anhydrite rock have similar low permeabilities in the laboratory, at least for limited ages.

c. The significantly larger grout-anhydrite specimen tested at Terra Tek essentially failed when tested by immediate passage of water along the contact surface of grout and anhydrite. While less expansion of this grout due to the higher test temperature may have been a factor, this behavior does seem to raise a question of size effect. What will actually happen when such a contact surface is full scale?

29. Present plans are to obtain an initially tight, essentially impermeable contact of a hydraulic cementitious system against wall rock in a repository by expansion of the cementitious system. A general consideration that needs to be addressed for this plan relates to when the cementitious system shrinks or contracts slightly from its maximum volume due to cooling from the temperature maximum it reached due to heat of hydration of the system and due to drying, as it achieves hygrometric equilibrium with its surroundings, or for other reasons, will the elastically compressed wall rock have sufficient elastic deformation to maintain the earlier tight contact it had with the hardened cementitious material? If the answer is yes, fine, then there is no problem. It should be possible to make such calculations using available data or making such assumptions as may be necessary. No such calculations have been called to my attention. On the other hand, if the answer is that the tight contact will not or may not be maintained, then it is time to think about use of a compressible elastic material at the interface to assure that the desired tight impermeable contact is maintained. The thoughts and concern expressed in this paragraph are derived from discussions with Bryant Mather, Chief, Structures Laboratory.

## Conclusions

30. A satisfactory candidate sanded, nonsalt, grout for use in the 1984 Terra Tek test was developed and used for that test. It is identified as TT84.

31. It remained workable over a 2-hr period with a flow rate slightly in excess of 30 sec in the laboratory and nearer 20 sec for the Terra Tek test.

32. Compressive strength, expansion, and permeability are all considered satisfactory.

33. The permeability of a SBH sample that included contact of this grout against anhydrite rock was sufficiently low as to be regarded as excellent until leakage developed after about 6 weeks of testing. It is believed that this leakage was due to partial dissolution of the rock and could be prevented in the future by the use of water saturated with calcium sulfate. This is believed to be a laboratory rather than a field problem since in situ solutions should already be saturated and thus pose no problems of dissolution.

34. Expansion appears to be reduced but not prevented completely by moisture loss and by elevation of the temperature to about 86° F.

35. Use of some new samples of materials from previous sources during development of this grout was not considered significant.

## Recommendations

36. Concern has been expressed herein that the probable inevitable slight volume reduction of any cementitious system due to cooling or drying or both might affect the tightness of the contact with host wall rock. It is recommended that the magnitude of this effect be investigated by calculations and compensated for by use of compressible elastic material at the interface to maintain a tight contact surface if needed.

37. Expansion testing for selected specimens should be continued at 6-month intervals to obtain longer-term data.

38. Phase composition and microstructure of the grout should be determined periodically in the future and compared with those properties for other similar grouts.

Table 1

| TO: | REPORT OF TESTS OF HYDRAULIC CEMENT | FROM: CORPS OF ENGINEERS U.S. ARMY |
|---|---|---|
| Structures Laboratory<br>Waterways Experiment Sta<br>ATTN: Don Walley<br>P. O. Box 631<br>Vicksburg, MS 39180 | RC 911 | Structures Laboratory<br>Waterways Experiment Sta<br>Cement & Pozzolan Unit<br>P. O. Box 631<br>Vicksburg, MS 39180 |

TEST REPORT NO. WES-137-84 | BIN NO. | CWT REPRESENTED: SS | DATE: 15 May 84

SPECIFICATION: A.P.I. Class H | DATE SAMPLED: 2 May 84

COMPANY: Lonestar Ind. | LOCATION Maryneal, TX | BRAND:

THIS CEMENT DOES X MEET SPECIFICATION REQUIREMENTS

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| SAMPLE NO. | 1 | | | | | | | |
| $SiO_2$, % | 21.7 | | | | | | | |
| $Al_2O_3$, % | 3.6 | | | | | | | |
| $Fe_2O_3$, % | 3.8 | | | | | | | |
| MgO, % | 3.2 | | | | | | | |
| $SO_3$, % | 2.0 | | | | | | | |
| LOSS ON IGNITION, % | 0.6 | | | | | | | |
| ALKALIES-TOTAL AS $Na_2O$, % | 0.52 | | | | | | | |
| $Na_2O$, % | 0.14 | | | | | | | |
| $K_2O$, % | 0.57 | | | | | | | |
| INSOLUBLE RESIDUE, % | 0.11 | | | | | | | |
| CaO, % | 63.4 | | | | | | | |
| $C_3S$, % | 58 | | | | | | | |
| $C_3A$, % | 3 | | | | | | | |
| $C_2S$, % | 19 | | | | | | | |
| $C_3A + C_3S$, % | 61 | | | | | | | |
| $C_4AF$, % | 12 | | | | | | | |
| $C_4AF + 2\,C_3A$, % | 18 | | | | | | | |
| HEAT OF HYDRATION, 7D, CAL/G | | | | | | | | |
| HEAT OF HYDRATION, 28D, CAL/G | | | | | | | | |
| SURFACE AREA, $m^2/kg$ (A.P.) | 229 | | | | | | | |
| AIR CONTENT, % | 11 | | | | | | | |
| COMP. STRENGTH, 3 D, PSI | 1750 | | | | | | | |
| COMP. STRENGTH, 7 D, PSI | 2260 | | | | | | | |
| COMP. STRENGTH, 28 D, PSI * | 3050 | | | | | | | |
| Density $Mg/m^3$ | 3.17 | | | | | | | |
| SAMPLE NO. | 1 | | | | | | | |
| AUTOCLAVE EXP., % | 0.02 | | | | | | | |
| INITIAL SET, HR/MIN | 2:00 | | | | | | | |
| FINAL SET, HR/MIN | 6:25 | | | | | | | |
| SAMPLE NO. | | | | | | | | |
| AUTOCLAVE EXP., % | | | | | | | | |
| INITIAL SET, HR/MIN | | | | | | | | |
| FINAL SET, HR/MIN | | | | | | | | |

REMARKS: Job # 441-S866.24SC21 *28 day test results

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Toy S. Poole
for R. E. REINHOLD
Chief, Cement & Pozzolan Unit

Table 2

WES-145-84

| SUBJECT | DATE | PAGE 1 |
|---|---|---|
| Halliburton (Lab Stock) | 3 July 84 | OF 1 PAGES |
| SOURCE OF DATA: Test of Cal-Seal (Plaster) Job #441-S866.14SC51 (AL BUCK) | | |
| COMPUTED BY | CHECKED BY | SECTION |

| CHEMICAL ANALYSIS | METHOD | | % AS RECEIVED | % AS GYPSUM |
|---|---|---|---|---|
| Free Water, % | C 471 | | 1.05 | 0.16 |
| Combined Water, % | C 471 | | 5.84 | 20.49 |
| $SiO_2$+Insol. Residue, % | C 114 | | 0.47 | 0.40 |
| $R_2O_3$, % | C 114 | | 0.58 | 0.49 |
| $Al_2O_3$ (Difference), % | C 114 | | 0.56 | 0.48 |
| $Fe_2O_3$, % | C 114 | | 0.02 | 0.02 |
| CaO, % | C 114 | | 38.16 | 32.52 |
| MgO, % | C 114 | | 0.24 | 0.20 |
| $SO_3$, % | C 471 | | 52.65 | 44.87 |
| $Na_2O$, % | C 114 | | 0.07 | 0.06 |
| $K_2O$, % | C 114 | | 0.05 | 0.04 |
| TOTAL | | | 99.69 | 99.73 |
| CALCULATED COMPOUNDS: | | % | | |
| ($CaSO_4 \cdot ½H_2O$), % | | 82.53 | | |
| Gypsum ($CaSO_4 \cdot 2H_2O$), % | | 97.90 | | |
| $SO_3$ combined as gypsum, % | | 45.53 | | |
| Excess $SO_3$, % | | 7.12 | | |
| Anhydrite ($CaSO_4$), % | | 12.10 | | |
| CaO combined as gypsum, % | | 31.89 | | |
| CaO as anhydrite, % | | 4.98 | | |
| Calcium carbonate ($CaCO_3$), % | | 2.30 | | |
| Magnesium carbonate ($MgCO_3$), % | | 0.50 | | |
| PHYSICAL TESTS | | | | |
| Free Water, % | | 0.95 | | |
| Fineness: | | | | |
| Passing No. 30 Sieve, % | | 99.7 | | |
| Passing No. 100 Sieve, % | | 99.5 | | |
| A.P., C-204, $m^2$/kg, % | | 417 | | |
| Normal Consistency, Paste: N.A. | | 28 | | |
| Time of Set, Paste, Min.: N.A. | | 83 | | |
| Normal Consistency, Mortar | | 10.3 | | |
| Time of Set, Mortar, Min.: | | 10.7 | | |
| Compressive Strength, P.S.I. | | 6,860 | | |
| Density, C-188, $Mg/m^3$, % | | 2.76 | | |
| Mortar Density, Lbs/Cu ft | | 127.18 | | |

WES FORM NO. 1114
JANUARY 1961

R. E. Reinhold
R. E. Reinhold
Chief, Cement & Pozzolan Unit

Table 3

Physical Data for Three Fine Aggregates

| Grading[(a)] Passing | Grading[(a)] Retained | NTS 20-40 Sand | D-30 Silica Sand AD-791 | Silica Flour AD-790 |
|---|---|---|---|---|
| 1.18 mm (No. 16) | 850 μm (No. 20) | 8 | Not used | Not used |
| 850 μm (No. 20) | 600 μm (No. 30) | 59 | 0 | 0 |
| 600 μm (No. 30) | 425 μm (No. 40) | 25 | Not used | Not used |
| 425 μm (No. 40) | 300 μm (No. 50) | 5 | 4 | Trace |
| 300 μm (No. 50) | 150 μm (No. 100) | 2 | 78 | 66 |
| 150 μm (No. 100) | 106 μm (No. 140) | Not used | 12 | 12 |
| 106 μm (No. 140) | 75 μm (No. 200) | 1 | 5 | 11 |
| 75 μm (No. 200) | 38 μm (No. 400) | Not used | 1 | 7 |
| 38 μm (No. 400) | Pan | Trace | Trace | 4 |
| Total | | 100 | 100 | 100 |
| Specific Gravity[(b)] | | 2.61 | 2.64 | 2.26 |
| Absorption, %[(b)] | | 0.6 | 0.4 | 11.8 |

(a) Done in accordance with CRD-C 103-83 (ASTM C 136-83).
(b) Done in accordance with CRD-C 108-81 (ASTM C 128-79).
Both in U. S. Army Engineer Handbook for Concrete and Cement, with quarterly supplements, Vicksburg, MS, Aug 1949.

Table 4

Mixture Data for TT84 Grout[a]

| Constituents | Amount Required for 1-cu ft Batch |
|---|---|
| Class H Cement[b] | 44.30 lb |
| Class C Fly Ash[b] | 14.90 lb |
| Plaster (Cal-Seal)[b] | 5.23 lb |
| Melgran 1[b] | 0.24 (110 g) |
| Melgran 2[b] | 0.24 (110 g) |
| De-Air No. 1 | 0.16 (73 g) |
| 20-40 Sand | 15.90 lb |
| D-30 Sand | 23.90 lb |
| Silica Flour | 8.00 lb |
| Water | 22.20 lb |
| **Other Data** | |
| Ratio of Water to Cementitious Materials | 0.344 |
| Actual Unit Weight | 136.9 lb/cu ft |
| Air Content | <1.0% |
| Bleeding (ratio of bleed water to mixing water) | 0.03% |
| Ambient Temperature | ~70° F |
| Setting Time | |
| Initial | 9 hr |
| Final | 10-1/4 hr |
| Flow Data:[c] | |
| 29 sec at 5 min | |
| 36 sec at 30 min | |
| 30 sec at 60 min | |
| 34 sec at 120 min | |

(a) This is a sanded modification of BCT 1-FF grout.
(b) Considered to be cementitious material.
(c) Due to a tendency to settle, the batch should be mixed at least 30 min prior to use.

Table 5

Compressive Strength of TT84 Grout Cubes

| Age, days | Compressive Strengths at Ages Shown, psi[a] Batch 3 Cast 16 April 1984 | Batch 3B Cast 14 May 1984 |
|---|---|---|
| 1 | 1,860 | 440 |
| 4 | -- | 4,280 |
| 7 | 6,250 | -- |
| 14 | 7,280 | 6,930 |
| 21 | 9,720 | -- |
| 28 | 10,210 | 9,060 |
| 56 | 10,880 | 12,430 |
| 90 | (b) | 14,530 |
| 180 | -- | 15,020 |
| 365 | -- | 15,780 |

(a) Each value is average of three 2- by 2- by 2-in. cubes except for the 56-day batch 3 data; that value is the average of three 3- by 6-in. cylinders.

(b) Specimens not available.

Table 6

Expansion Data for 21 TT84 Grout Bars* at Different Temperatures and Storage Conditions

| | Age, days | Length Change, % | | |
|---|---|---|---|---|
| | | Restrained[a] | | Unrestrained[b] |
| 73° F | | | | |
| | 4 | 0.042 | | 0.068 |
| Moist | 7 | 0.060 | | 0.112 |
| Cabinet | 14 | 0.076 | | 0.148 |
| Storage | 21 | 0.088 | | 0.164 |
| | 28 | 0.093 | | 0.172 |
| | 56 | 0.098 | | 0.184 |
| | 90 | 0.104 | | 0.190 |
| | 180 | 0.112 | | 0.198 |
| | 365 | 0.127 | | 0.211 |

(a) Average of bars 21 and 23 (16 April).
(b) Average of bars 1 and 2 (16 April).

| | | 3 by 3[c] | (d) | (e) |
|---|---|---|---|---|
| | 4 | 0.043 | 0.043 | 0.071 |
| Sealed | 7 | 0.053 | 0.051 | 0.118 |
| (Waxed) | 14 | 0.044 | 0.046 | 0.154 |
| Storage | 21 | 0.027 | 0.028 | 0.162 |
| | 28 | 0.016 | 0.014 | 0.150 |
| | 56 | -0.003 | -0.004 | 0.110 |
| | 90 | -0.010 | -0.014 | 0.085 |
| | 180 | -0.018 | -0.023 | 0.064 |
| | 365 | -0.025 | -0.028 | 0.052 |

(c) Average of bars 4, 5, and 6 (16 April). Test was stopped after 1 year.
(d) Average of bars 34, 36, 37, and 39 (16 April). Testing stopped after 1 year.
(e) Average of bars UDW 1 and 2. Bar UDW 3 data not used since they indicated obvious drying (18 April). Testing stopped after 1 year.

| 86° F**† | | (f) | (g) |
|---|---|---|---|
| | 4 | 0.044 | 0.038 |
| Not | 7 | 0.060 | 0.042 |
| Sealed | 14 | 0.052 | 0.018 |
| (Storage | 21 | 0.058 | 0.034 |
| over | 28 | 0.063 | 0.038 |
| water) | 56 | 0.064 | 0.045 |
| | 90 | 0.082 | -0.029 |
| | 180 | 0.110 | -0.072 |
| | 365 | 0.134 | 0.037 |

(f) Average of bars 24 and 28 (16 April).
(g) Average of bars 3 and 4 (16 April); shrinkage due to accidental dry storage.

(Continued)

Table 6 (Concluded)

| | Age, days | Length Change, % Restrained(a) | Length Change, % Unrestrained(b) |
|---|---|---|---|
| 86° F**† | | | |
| | 2 | 0.070(h) | |
| Sealed | 5 | 0.089 | |
| (Waxed) | 12 | 0.069 | |
| Storage | 19 | 0.050 | |
| | 26 | 0.043 | |
| | 51 | 0.039 | |
| | 90 | 0.065 | |
| | 180 | 0.038 | |
| | 365 | 0.023 | |

(h) Average of bars UDW 4, 5, and 6 (18 April). Testing stopped after 1 year.

* Batch 3 and batch 3A made 16 and 18 April 1984. All 2 by 2 in. with 10-in. gage length unless shown to be larger.

** First 24 hr moist storage at approximately 73° F.

† Oven went to 135° F between 28- and 56-day ages.

Table 7

Expansion Data for 18 TT84 Grout Bars* at Different Temperatures and Conditions

| | | Length Change, % | |
|---|---|---|---|
| 73° F | Age, days | Restrained[(a)] | Unrestrained[(b)] |
| Moist Cabinet Storage | 3 | 0.027 | 0.051 |
| | 7 | 0.052 | 0.093 |
| | 15 | 0.076 | 0.138 |
| | 21 | 0.086 | 0.166 |
| | 28 | 0.096 | 0.186 |
| | 56 | 0.118 | 0.215 |
| | 90 | 0.128 | 0.230 |
| | 180 | 0.124 | 0.243 |
| | 365 | 0.139 | 0.264 |

(a) Average of bars 1 and 2; bar 3 was defective.
(b) Average of bars 4 through 6 plus a spare.

| | Age, days | (c) | (d) |
|---|---|---|---|
| Sealed | 3 | 0.049 | 0.056 |
| | 7 | 0.081 | 0.084 |
| | 15 | 0.138 | 0.105 |
| | 21 | 0.173 | 0.104 |
| | 28 | 0.200 | 0.088 |
| | 56 | 0.211 | 0.052 |
| | 90 | 0.226 | 0.038 |
| | 180 | 0.236 | 0.023 |
| | 365 | 0.258 | 0.018 |

(c) Average of bars 7, 8, and 9 stored in moist cabinet; bars wrapped in tape.
(d) Average of bars 11 and 12 in container; bar 10 was defective.

| 86° F | Age, days | (e) | (f) |
|---|---|---|---|
| Sealed (Moist) Storage | 3 | 0.088 | 0.084 |
| | 7 | 0.127 | 0.116 |
| | 15** | 0.098 | 0.089 |
| | 21 | — | — |
| | 28 | 0.091 | 0.079 |
| | 56 | 0.125 | 0.110 |
| | 90 | 0.122 | 0.108 |
| | 180 | 0.124 | 0.110 |
| | 365 | 0.154 | 0.160 |

(e) Average of bars 13, 14, and 15 in container over water. Bar 13 missed at 180 days.
(f) Average of bars 16, 17, and 18; same storage condition.

* 2- by 2- by 10-in. gage length bars cast 14 May 1984, batch 3B.
** Temperature went to 135° F between 15 and 21 days.

Table 8

Permeability Data for Simulated Borehole (SBH) Sample (Anhydrite, Rock and TT84 Grout) and Grout Cylinder CL-43 GRT-7[a]

| Age, days | Permeability in Microdarcies[b] SBH Sample |
|---|---|
| 7 | 1.6 |
| 18 | 0.7 |
| 43 | 3.5 |
| 52 | 42 |
| 53 | 72 |
| 56 | 64 |
| 63 | 108[c] |
| | TT84 Grout Sample CL-43 GRT-7 |
| 18 | No Flow |

(a) Tested as the SBH sample for 14 days; confining pressure then increased to 700 psi for 4 more days. The specimen was 80 days old when testing started.
(b) Determined in a Hassler cell with 200-psi driving pressure and 300-psi confining pressure.
(c) Specimen removed from rig after this test.

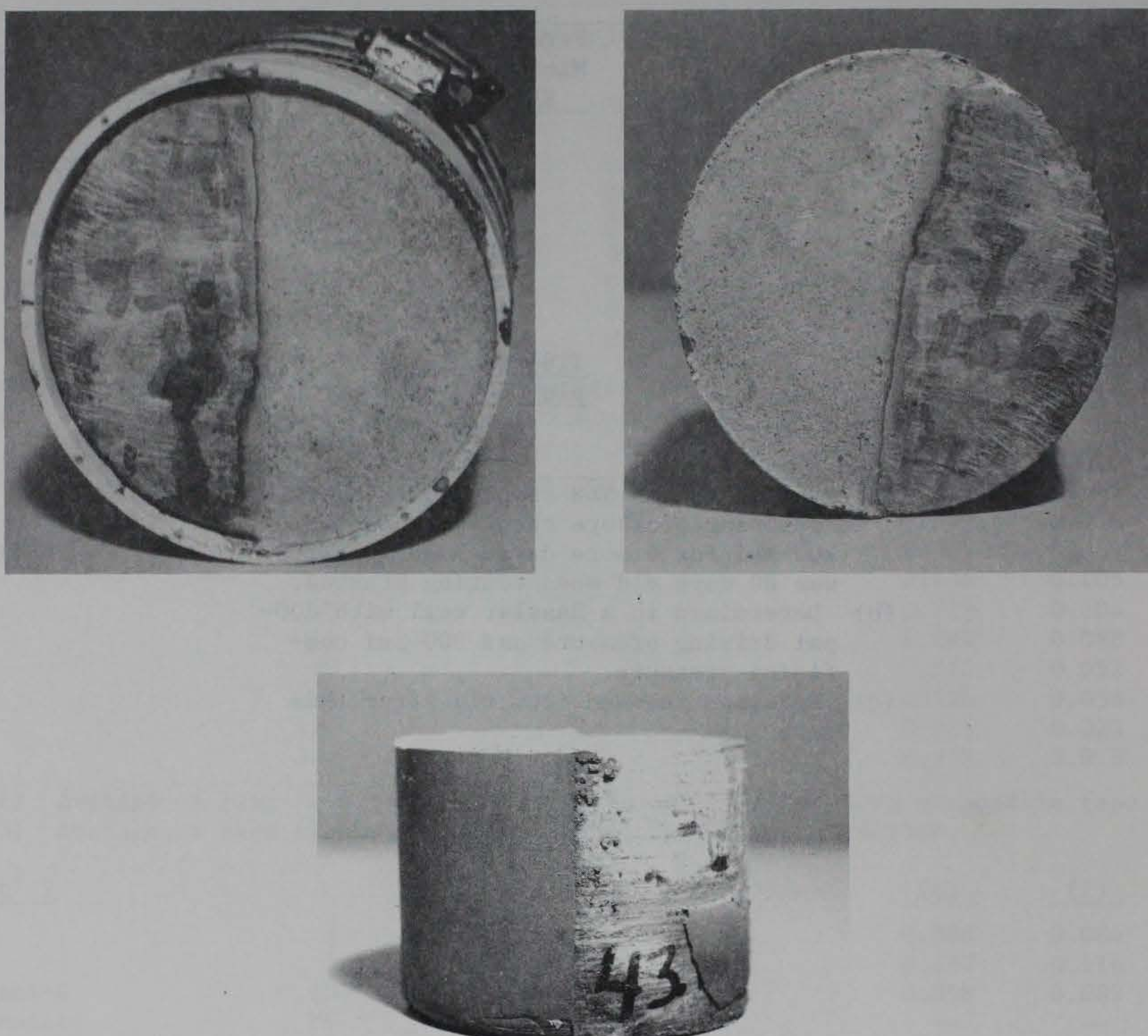
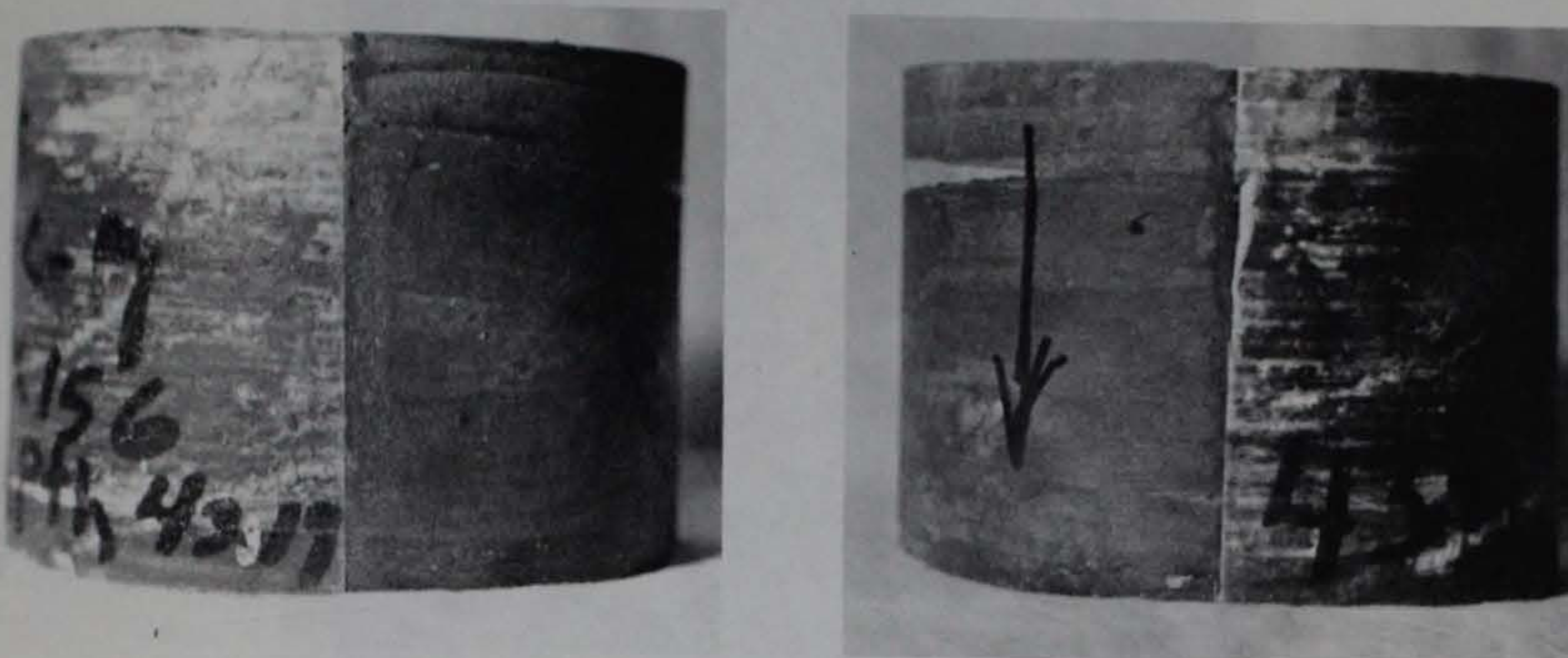
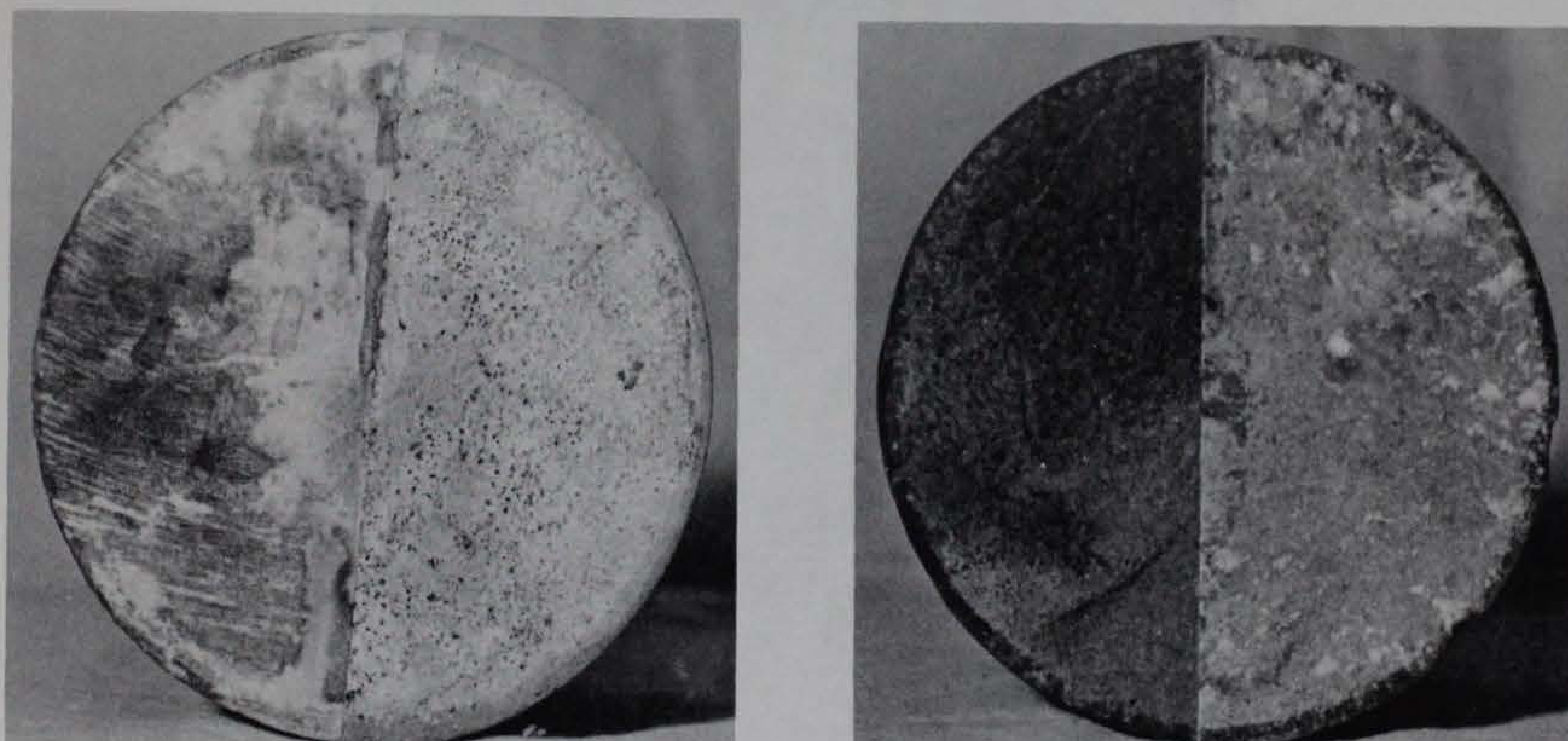



Figure 1. Three views of 1-day old TT84 grout- anhydrite SBH sample before insertion into Hassler cell and subsequent permeability testing. Upper left, sample in mold compressed with hose clamps; upper right and lower picture, unloaded sample. The anhydrite is the material on which markings are visible.



Top



Bottom

Figure 2. Four views of same SBH sample as Figure 1 after 63 days in Hassler cell with periodic permeability testing. No obvious separation at grout-rock contact or other distress. Top is side views of contact surface; bottom is end views. Rock always to outside and grout to inside in this series of pictures; arrow shows direction of testing.

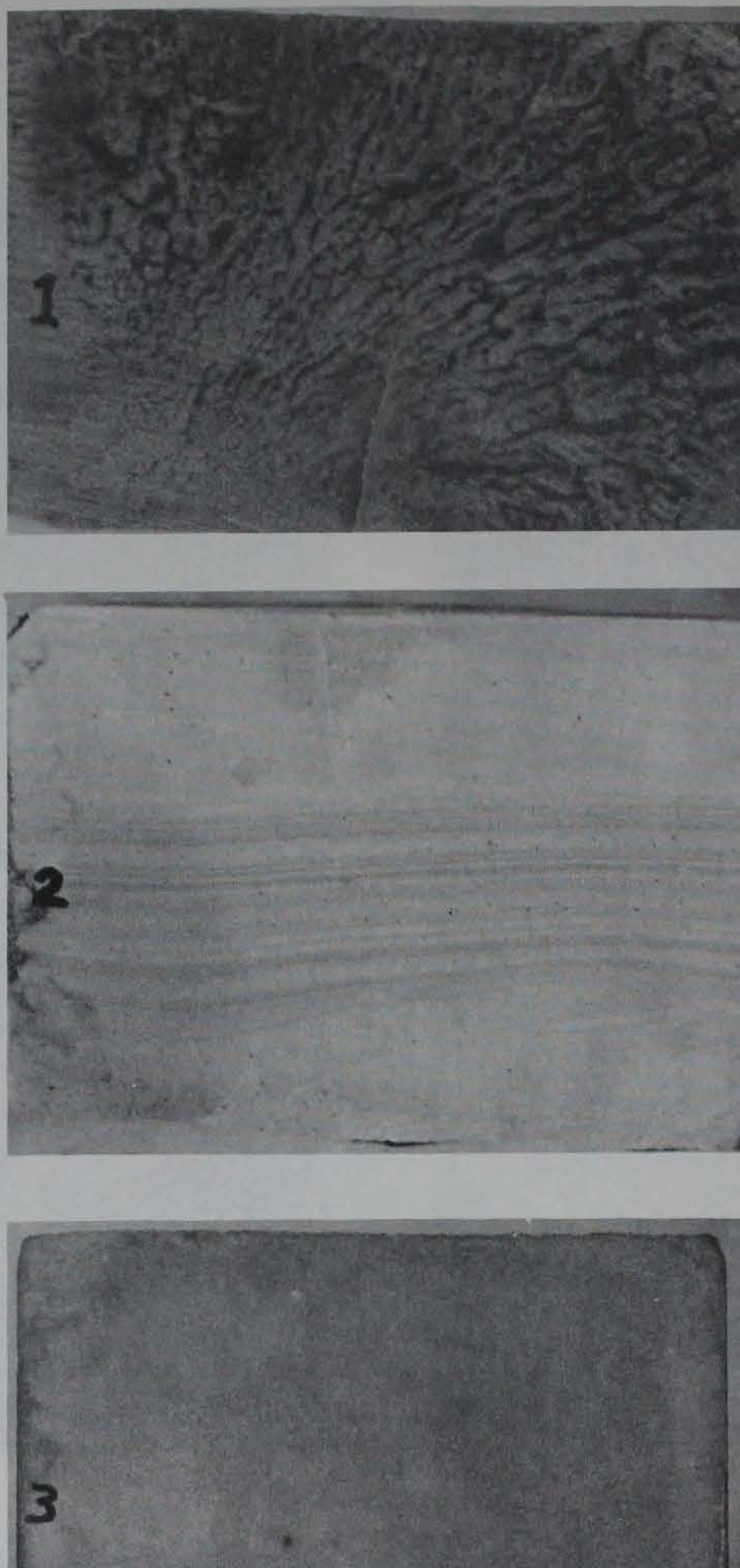


Figure 3. No. 1 and 2 are mirror image surfaces of anhydrite (No. 1 untested, No. 2 after 63 days in Hassler cell as SBH sample). No. 3 is grout surface that had been in contact with No. 2 as the SBH sample.

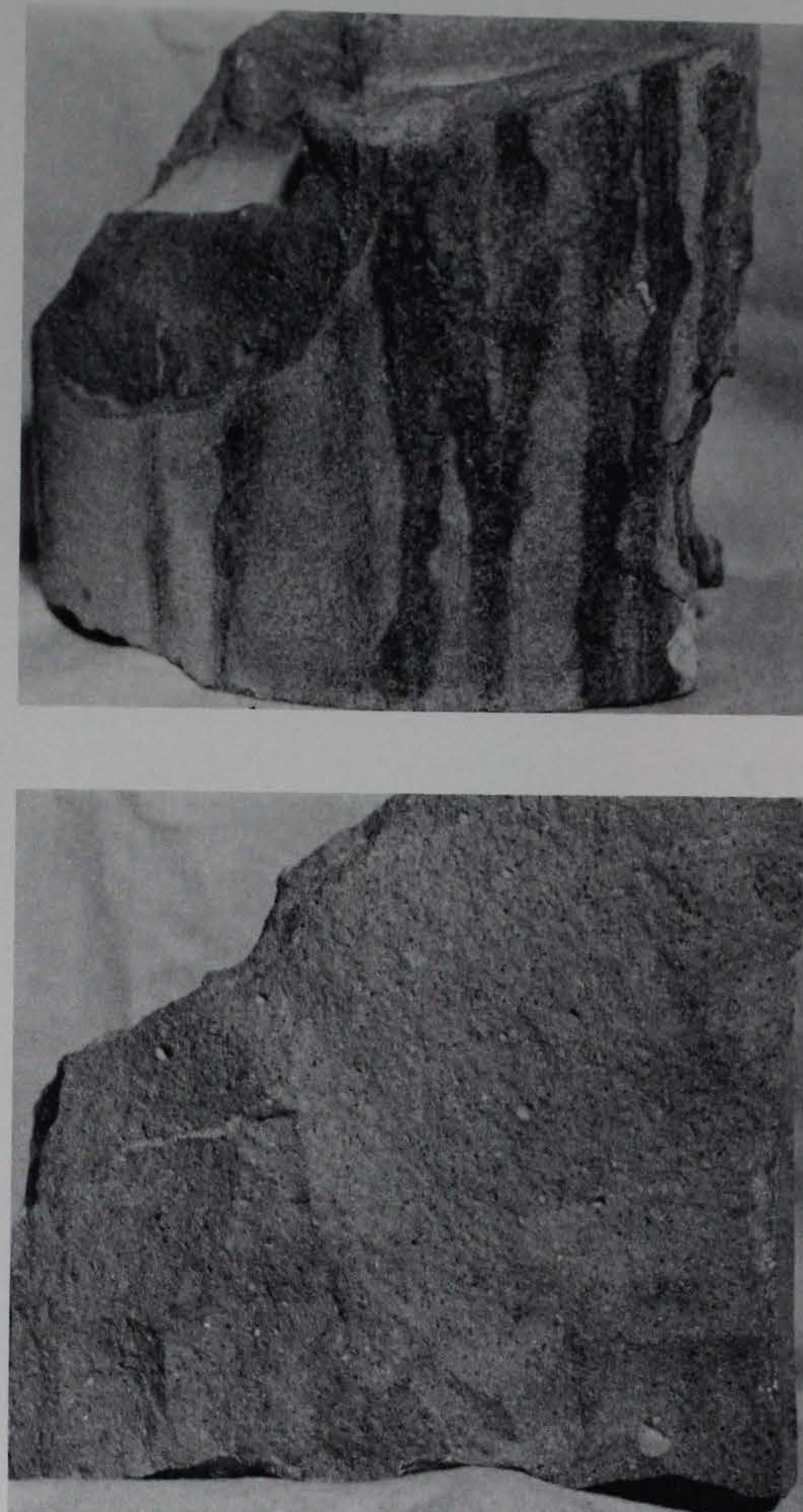


Figure 4. Portion of grout from actual Terra Tek 1984 test. Top is surface (dampened) that had been in contact with anhydrite; dark vertical lineations are flow channels. Bottom is fractured dense surface of grout; no flow channels.