

~~111105 157412~~

NBS  
PUBLICATIONS

NATL INST. OF STAND & TECH R.I.C.  
A11105 157412

NBSIR 80-2099-2

# Testing Geothermal-Well Cements: Standard Practice

---

Ralph F. Krause, Jr.  
Edwin R. Fuller, Jr.

Fracture and Deformation Division  
Center for Materials Science  
National Bureau of Standards  
U.S. Department of Commerce  
Washington, DC 20234

July 1979  
Interim Report  
Issued July 1980

Prepared for  
Division of Geothermal Energy  
Department of Energy  
Washington, DC 20461

QC  
100  
U56  
80-2099-2  
1980  
c.2



NBSIR 80-2099-2

# TESTING GEOTHERMAL-WELL CEMENTS: STANDARD PRACTICE

---

NATIONAL BUREAU  
OF STANDARDS  
LIBRARY

SEP 19 1980

100-01-312  
2000  
100  
80-2099-2  
1050  
0.2

Ralph F. Krause, Jr.  
Edwin R. Fuller, Jr.

Fracture and Deformation Division  
Center for Materials Science  
National Bureau of Standards  
U.S. Department of Commerce  
Washington, DC 20234

July 1979

Interim Report

Issued July 1980

Prepared for  
Division of Geothermal Energy  
U.S. Department of Energy  
Washington, DC 20461



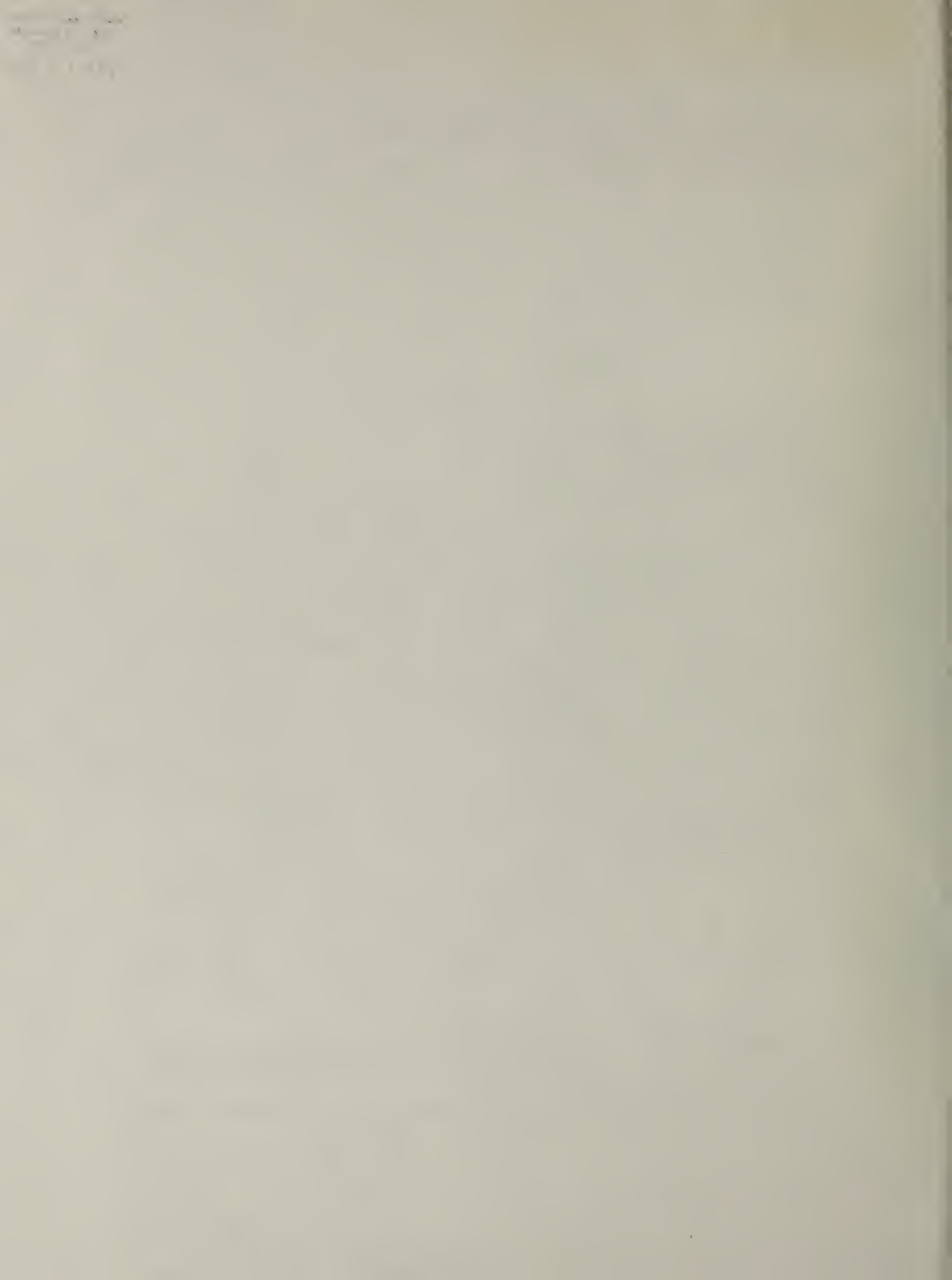
---

**U.S. DEPARTMENT OF COMMERCE, Philip M. Klutznick, *Secretary***

**Luther H. Hodges, Jr., *Deputy Secretary***

**Jordan J. Baruch, *Assistant Secretary for Productivity, Technology, and Innovation***

**NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director***



## I. Scope

The National Bureau of Standards is under contract with the U.S. Department of Energy to verify certain properties of cementing materials which are submitted as candidates for use in the finishing operations of geothermal wells. Specimens will be set-cured in molds under water for two days at elevated temperature and pressure. Subsequently, specimens will be exposed demolded to light and heavy simulated geothermal fluids for periods of one week or one month. Following each of these treatments, the following properties will be measured at room temperature and pressure: compressive strength, splitting tensile strength, shear-bond strength of the cement-steel interface, and cement permeability to water. Upon the basis of this survey of properties at room temperature, a priority of cementing materials will be established for further testing of select physical properties while the specimens are at elevated temperature and pressure.

## II. Candidate Cementing Materials

### A. Participants

Several laboratories, some of whom are under contract with the U.S. Department of Energy, have been developing cementing materials for geothermal wells. Any investigator who has a candidate cementing material to submit to this property testing program will be deemed a participant.

## B. Placement Ability

1. Participants will provide written certification that the slurry of each cementing material that they submit has passed the API thickening time requirement for a class J cement in a 3050 m (10,000 ft.) well depth (Table 2.2 of reference 1). This requirement specifies that the slurry must have a consistency less than 30 Bc during the initial 30 minutes of the stirring period and a consistency less than 100 Bc up to 3 hours of the stirring period.

2. The thickening time of a slurry will be measured according to the API thickening time test (section 7 of reference 2). The pressure and temperature control will follow the casing cement schedule 6g (p. 34 of reference 2) which has been extended to provide that the slurry be heated from 27 to 150 °C in 36 min (average rate of 3.4 °C/min). [Note: the task group may wish to consider a less severe pressure condition, such as the 36 MPa (5300 psi) used by Dowell (p. 105 of reference 3), rather than the 51.7 MPa (7500 psi) given in schedule 6g].

3. Participants may have these tests performed by Dowell under the conditions described in reference 4 (see attached).

## C. Mixing Proportions

1. Participants will give the mass ratio of the cementing components which are to be mixed, for example: 1.00 cement, 0.40 filler, 0.025 additive, and 0.60 water.

2. They will give the slurry density, which may, for example, be determined by the method in Appendix B of reference 2.

3. They may provide the details of any special slurry mix instructions which differ from those described in section IV A below.

#### D. Description

1. Participants will identify each cementing component by a convenient name, for example: API class H cement, Lone Star, Dallas, Texas. (A manufacturer's name is given to identify the material and does not imply that this material is the best available).

2. Unless a cementing material is proprietary, its chemical composition and preparation should be indicated for future reference and identification, for example: 1.00 cement (0.23 SiO<sub>2</sub>, 0.65 CaO, 0.04 Fe<sub>2</sub>O<sub>3</sub>, 0.05 Al<sub>2</sub>O<sub>3</sub>, 0.02 SO<sub>3</sub>, and 0.01 MgO by mass fraction), clinkered at 1500 °C, and pulverized (200 mesh); 0.40 silica flour (200 mesh); etc.

#### E. Shipment

1. Participants should submit before January 1, 1980 cementing materials which meet the above criteria of placement ability. Send the cementing materials, excluding water, to the attention of Ralph Krause, A355 Materials, National Bureau of Standards via, either of the following:

Commercial Carrier:       Route 270 and Quince Orchard Road  
                                  Gaithersburg, Maryland 20760

or U.S. Mail:                Washington, D. C. 20234

2. Preferably, the respective cementing components should be packaged separately; and sufficient quantities should be provided to prepare at least eight liters of slurry.

3. Alternatively, if some participants prefer to premix the dry cementing components themselves or if these components might tend to segregate in shipping, they should provide at least eight packages of cementing material each of which will form one liter of slurry.

### III. Apparatus

#### A. Balances

1. The prescribed quantities of cementing components will be weighed upon a pan balance which has a one kg capacity and an accuracy within  $\pm 0.1$  g.

2. The quantities of salts which are used to prepare the simulated geothermal fluids will be weighed upon a balance which has a 200 g capacity and an accuracy within  $\pm 0.1$  mg.

#### B. Mixing Device

1. The components of a given cementing material will usually be blended in a Waring Blender which has a two liter container and a two-speed propeller-type mixer of stainless steel, capable of rotating 4000 rpm or 10,000 rpm.

2. Also available is a Hobart N-50 mixer which has a 5 L stainless steel bowl with a flat beater as described in ASTM Method C305 for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.

#### C. Molds

1. Specimens for the compressive and splitting tensile strength tests will be set-cured in Pyrex tubes (20 mm OD, 1.2 mm wall thickness, and 150 mm length). Each tube will be thinly coated with silicone grease and capped with a Teflon plug, which has a one mm hole to equalize the pressure and to accommodate any thermal expansion of the slurry.

[Note: This specimen diameter is deemed appropriate due to the prospect of having to handle many specimens. A constraint is the 5 cm ID of the pressure vessels that are available for the geothermal exposures.]



2. Specimens for the shear-bond strength and permeability tests will be set-cured in molds of modified, one-inch-pipe nipples of stainless steel. Commercially available nipples (22 mm ID, 35 mm wrench pad, and 57 mm length) will be sawed in half perpendicular to their longitudinal axes. The inside diameter of the resulting pieces will then be tapered in a conical fashion such that the opening at the pipe threaded end is about five per cent larger than the opening at the wrench pad end. Removeable cover plates (3 mm thick) are machined to fit the ends of these molds, so that the molds may be stacked within a pressure vessel. A one mm groove is machined across the bottomside of the upper cover plates to accommodate any thermal expansion of the slurry. Teflon sheets (0.05 mm thick) will separate the specimen and the cover plates. Some of the cover plates will have recesses to center smooth steel rods (10 mm OD and 28 mm length) inside those molds which will be used for the shear-bond strength tests.

3. A stainless steel rod (3 mm OD and 200 mm length) will be used to puddle slurries in their molds.

#### D. Diamond Saw

A motorized diamond blade (150 mm OD, 0.5 mm width, 1725 rpm) will be used to saw the molded rods of set cement into specimens. Two miniature vises which are attached to a platform will secure a rod on each side of a cut. The platform will move automatically at a constant rate (0.5 mm/s) to feed the rod perpendicular to the blade, rotating in a fixed position through a bath of water.

#### E. Calipers

The lengths and diameters of specimens will be measured with calipers to an inaccuracy within  $\pm 0.02$  mm.

## F. High Pressure Systems

1. A 4 L stirred autoclave of 316 stainless steel, which can be operated up to a maximum pressure of 21 MPa (3000 psi), will be used to set-cure cementing materials in molds which are submerged in distilled water.

2. A series of four pressure vessels of Hastelloy C 276 (50 mm ID, 250 mm internal length, two top ports, and one bottom port) will be used to expose unprotected specimens of set cements in simulated geothermal fluids at pressures up to a maximum operating pressure of 60 MPa (8,700 psi). The wetted parts of all high pressure valves and fittings are also made of Hastelloy C276, except for the pressure lines which are made of Monel tubing.

3. A source of compressed nitrogen or argon will be available to provide pressures up to 40 MPa (5,800 psi) to all five pressure vessels.

4. A liquid metering pump with a Hastelloy C 4 pump head and a tantalum diaphragm will deliver the simulated geothermal fluids with a nominal flow up to 800 ml/hr to any of the four Hastelloy pressure vessels. Being driven by an air motor, the pump can maintain a constant output pressure up to a maximum of 69 MPa (10,000 psi) by adjustment of the pressure of the air supply.

5. Bourdon gauges with Monel coils and sockets will be used to indicate continuously the pressure within each vessel to an accuracy within  $\pm 0.2$  MPa (30 psi). A reference gauge will serve to calibrate periodically the operating gauges. Another bourdon gauge will be used to measure the partial pressure of  $\text{CO}_2$  over the simulated geothermal fluid reservoir to accuracy within  $\pm 0.7$  kPa (0.1 psi).

6. A pressure generator of 316 and 17-4PH stainless steels is being considered to compress water in order to develop a pressure difference across a permeability specimen. This generator is a manually operated piston screw pump which can be operated up to 69 MPa (10,000 psi).

7. Whenever all liquid conditions are used in the Hastelloy pressure vessels at elevated pressure and temperature, relief valves are being considered to dump overflow fluids in the event of an inadvertent over-pressurization. [Note: The use of these valves would possibly avoid abortion of an on-going exposure; nevertheless, safety rupture discs will also be installed on these vessels].

#### G. High Temperature Systems

1. Nichrome wire-wound, tube furnaces are available to surround each of the five pressure vessels, the Hastelloy vessels having furnaces of the split-tube type. These furnaces will be used to heat the vessels and their contents to elevated temperatures at a rate of about 3 °C/min. The maximum operating temperature is 340 °C for the stirred autoclave and 400 °C for the Hastelloy pressure vessels.

2. A temperature controller with an adjustable proportional band, reset, and rate controls will be used to operate each furnace automatically from room temperature to the operating temperature for that furnace, and to maintain the final temperature within  $\pm 2$  °C.

3. Chromel-alumel thermocouples will be used to indicate the temperature of the contents inside of each pressure vessel and the temperature at selected spots along the exterior surface of the pressure vessels. A digital voltmeter which is calibrated in degrees Celsius will be used to read the respective thermocouples to  $\pm 1$  °C.

4. The stirred autoclave is equipped internally with a water-cooling coil; the four Hastelloy vessels have externally applied water-cooling coils.

5. A constant temperature bath of stirred water will be used to hold specimens of set cements at  $25 \pm 1$  °C preceding their property tests at room temperature.

#### H. Fluid Handling Systems

1. Two reservoirs of stainless steel (four liter capacity each) will store at room temperature the two simulated geothermal fluids of different compositions, equilibrated respectively under appropriate pressures of carbon dioxide. Manipulation of stainless steel valves will direct either fluid to the high pressure metering pump.

2. A pH meter will be used to monitor the acidity of the fluids in the reservoirs. Both a glass measuring electrode and a reference electrode will be mounted in a stainless steel chamber to allow pH measurement to be made at pressures up to one MPa (150 psi).

#### I. Load Testing Machine

A recently calibrated Tinius Olsen Electromatic Universal Testing Machine (5400 kg load capacity) will be used to apply compressive loading at a constant displacement rate of 0.5 mm/min. A spherically-seated compression tool (79 mm diameter) is suspended from the crosshead of the machine to compensate for possible non-parallel bearing surfaces of a specimen being tested. The maximum load necessary to cause failure of a specimen is indicated by a gauge.

#### J. Permeameter

1. A holder will be provided to receive the modified nipple mold that contains the specimen to be tested.

2. A pair of capacitance-sensing, pressure transducers of 17-4PH stainless steel will be used to measure [to a precision within 3 kPa (0.4 psi)] the pressure difference that is applied to opposite sides of a specimen which is under an ambient pressure up to 60 MPa (8,700 psi).

3. A recording potentiometer will be used to measure the net output of these pressure transducers (14 kPa/mV). The exponential decay of this output is related to the permeability of the specimen being tested (See Section VI A).

#### K. Pressure Transmitter

It is planned tentatively to use a stainless steel bellows to transmit an applied pressure to one end of a steel rod so that the shear-bond strength of the steel-cement interface can be measured at elevated temperature and pressure.

### IV. Preparation of Specimens

#### A. Slurry Mixing

The API recommended practice (section 3 of reference 2) will generally be followed unless otherwise directed by the supplier. In brief, a weighed quantity of distilled water which is required to prepare one liter of slurry will be placed in the mixing container, the mixer turned on the slow speed, and the weighed quantities of dry components added in less than 15 s. Next, the cover will be placed on the container, and stirring will be continued at the high speed for 40 s.

#### B. Molding

1. Prior to mixing a slurry of a given cementing material, silicone grease will be thinly applied to the inside surfaces of ten Pyrex tube molds. Also, seven modified nipple molds of stainless steel and five

smooth steel rods will be washed with trichloroethylene and dried. These molds will provide all the specimens that are needed for the series of property tests which will be conducted at room temperature and pressure on a given cementing material.

2. The slurry of a cementing material which is prepared according to section IV A will be poured into these molds and puddled as needed to remove air bubbles. Five of the modified nipple molds will enclose the smooth steel rods. The excess slurry in the modified nipple molds will be struck off even. The molds will be appropriately covered as indicated in section III C, submerged upright in distilled water at 25 °C, and sealed in the stirred autoclave.

#### C. Set-Curing

1. The set-cure will commence at the application of pressure and temperature. First, the autoclave will be partially pressurized with nitrogen or argon; then it will be automatically heated from room temperature to 200 °C in about 75 min., the temperature rate being fairly constant at 3 °C/min during the first 60 min. The contents of the autoclave will be maintained at 200 °C for the remainder of the cure. The quantity of distilled water and the initial pressure of nitrogen are chosen such that the volume ratio of the final gas phase to vessel capacity is about ten percent and the final pressure will be near, but eventually adjusted to 20 MPa (2900 psi) when the operating temperature is attained.

2. When the set-curing has progressed 48 h, the heating will be terminated and water-cooling through an internal coil of the autoclave will begin to reduce the temperature at a fairly constant rate, ultimately to 60 °C in 45 min; then the pressure in the autoclave will be gradually released.

#### D. Finishing Operations

1. At the conclusion of a set-cure the Pyrex molds of set cement will be removed from the stirred autoclave, and the Pyrex tubes will be broken away by diamond scribing the tubes and gentle tapping.

2. Each of the ten rods of set cement will be sawed perpendicular to its longitudinal axis into three specimens which are twice as long as their diameter. These pieces will be identified as the top, middle, and bottom of a given rod.

3. The seven modified nipple molds of set cement will be removed from the stirred autoclave, and their cover plates and Teflon sheets detached. If the surfaces of the set cement are glazed they will be scraped lightly with a wire brush under a stream of water to remove such glaze.

4. The finished specimens resulting from a set-cure will be submerged in water in a constant temperature bath at 25 °C for storing until some specimens are property tested and the remainder are exposed to simulated geothermal fluids. A weekly schedule which includes specimen preparation is indicated in Table I.

#### V. Exposures in Simulated Geothermal Fluids

##### A. Fluid Compositions

Two aqueous solutions of different concentrations of certain salts and carbon dioxide will be used to represent light and heavy geothermal fluids, respectively. Appropriate quantities of NaCl, KCl, CaCl<sub>2</sub>, and NaHCO<sub>3</sub> will be weighed and dissolved in distilled water to provide the compositions which are shown in Table II. When the solutions A and B are placed in their respective reservoirs, carbon dioxide gas at given partial pressure will be bubbled through the solutions at 25 °C until

they are equilibrated to give a pH near 4.5. Thereafter, this partial pressure of CO<sub>2</sub> will be maintained in the respective reservoirs, also in the Hastelloy pressure vessels while they are at 25 °C. [Note: The compositions of solutions A and B were arbitrarily selected. As reference 5 indicates, geothermal fluids from various locations differ considerably in composition; moreover, the analyses at a given site show varying results. The composition of solution A corresponds to the average of the principal components in 14 analyses of the East Mesa 6-1 wellhead fluid which was collected in the spring of 1977 (reference 6). The analyses of the Salton Sea fluids which are reported in reference 5 served as guidelines in the selection of the composition of solution B. The exposure temperatures given in Table 2 are consistent with the corresponding Na/K mass ratios, which are used to indicate bottom hole temperatures (reference 9). The exposure pressure of solutions A and B corresponds to a well depth of about 2200 m.]

#### B. Temperature and Pressure Conditioning

1. The four Hastelloy pressure vessels will be divided evenly to contain the two solutions A and B and will be operated at the temperatures and pressures which are specified in Table II for these respective solutions.

2. In regard to the exposures of specimens which will be property tested at 25 °C, the Hastelloy pressure vessels will be pressurized and heated in a manner similar to that which is described in section IV C for the stirred autoclave. The quantities of exposure solutions which are added to the vessels containing the specimens at room temperature are chosen such that the volume ratio of final gas phase to vessel capacity is about ten percent when the operating temperature is attained. Also, nitrogen or argon will be added to the contents of these vessels



at room temperature such that the final pressure will be near, but eventually adjusted to the value given in Table II. Once a week the vessels will be appropriately cooled and dismantled. Table I gives a weekly schedule for these operations.

### C. Scheduling of Set Cements

1. Specimens of a given set cement will commence being exposed unprotected in the two solutions A and B about 27 and 51 h, respectively, after the conclusion of their set-cure.

2. Eight rods of three sawed specimens each, which are designated for the compressive and tensile strength tests, will be divided evenly and deposited at the appropriate times in two Hastelloy pressure vessels, holding solutions A and B, respectively. Teflon sheets or stainless steel shims (0.05 mm thickness) will be used to separate the sawed ends of these specimens to keep them from sticking together while they are stacked in the vessels during the exposures. Half of these specimens will be exposed one week and the other half, exposed four weeks. Table III shows a schedule of depositing and withdrawing this type of specimen for several set cements in a particular solution.

3. Likewise, the four specimens which are designated for the shear-bond strength test and the two specimens which are designated for the permeability test will be divided evenly and deposited in the other two Hastelloy pressure vessels, holding solutions A and B, respectively. Half of the shear-bond specimens will be exposed one week and the other half, exposed four weeks; the same permeability specimens will be used throughout. Table IV shows a schedule of depositing and withdrawing these types of specimens for several set cements in a particular solution.

## VI. Property Tests

### A. Permeability of Set Cement

1. The measurement technique to be employed for determining permeability is a pulsed technique, similar to that described in reference 10, except that the range of conditions has been extended so that measurements can be made both at room temperature and atmospheric pressure and at elevated pressure and temperature, using the Hastelloy pressure vessels.

2. A mold with its set-cured specimen is sealed in a test holder, using Teflon tape. The holder is filled with the fluid that was used in the set-cure or in the particular exposure that this test follows. When the ambient temperature and pressure are established on both sides of the specimen, a vent valve on the holder is opened momentarily to allow fluid from the fluid reservoir to be pumped through the holder until any air in the holder is displaced.

3. A differential pressure of 2.0 MPa (290 psi) is applied across the specimen of set cement to establish the fluid flow through the cement. The fluid in the holder is then isolated from its reservoir, and the decline of the pressure difference is recorded as a function of time.

4. The fluid permeability ( $k$ ) of the set cement is calculated from the observed logarithmic slope

$$d \ln (P_1 - P_2) / dt = -k A (1/V_1 + 1/V_2) / \mu \beta L$$

where

$P_1$  = pressure of fluid in holder,

$P_2$  = ambient pressure downstream,

$t$  = time,

$A$  = cross-sectional area of specimen,

L = length of specimen,

$\mu$  = fluid viscosity,

$\beta$  = fluid compressibility,

$V_1$  = volume of fluid in holder, and

$V_2$  = ambient volume downstream.

[Note: When measurements are made at room temperature and pressure,  $P_2$  remains constant and  $1/V_2$  is assumed zero.]

#### B. Shear-Bond Strength of Cement-Steel Interface

1. This test will be conducted at room temperature and pressure and also at elevated temperature and pressure, using the Hastelloy pressure vessels.

2. A mold containing a cement specimen with a centrally embedded steel rod is supported in a test holder, and a load is applied to the end of the steel rod which is in the larger opening of the mold. The maximum force required to displace the steel rod is recorded and used to calculate the shear-bond strength.

3. The shear-bond strength ( $\sigma_s$ ) is calculated from

$$\sigma_s = F/\pi DL$$

where

F = maximum force required to displace the steel rod,

D = diameter of steel rod, and

L = length of the cement-steel rod interface.

[Note: The shear-bond strength determined at room temperature can be reduced from its actual value when the thermal expansion of the set-cured cement is less than that of the steel; on the other hand the mold-cement interface might fracture first during a test, despite its larger diameter, if the relative thermal expansion difference is reversed.]

### C. Compressive Strength of Set Cement

1. The testing procedure will generally be in accordance with ASTM Standard Method C-39 (reference 11) except that the specimen dimensions are smaller than those customarily used. The bearing block faces and the test specimens are wiped clean, and the specimen is centered directly under the spherically seated upper bearing block that is able to rotate freely. The universal testing machine will apply a compression load at a constant displacement rate of 0.5 mm/min until the maximum force necessary to cause failure of the specimen is recorded.

2. The compressive strength ( $\sigma_c$ ) is calculated from

$$\sigma_c = 4F/\pi D^2$$

where  $F$  = maximum force applied to the face of the cement specimen, and

$D$  = diameter of the cement specimen.

### D. Splitting Tensile Strength of Set Cement

1. The testing procedure will generally be in accordance with ASTM Standard Method C496 (reference 12) except that the specimen dimensions are smaller than those customarily used. Unused strips of cardboard (0.5 mm thick) are used as cushions between the specimen and the load bearing surfaces to distribute the load smoothly. Using marked diametral lines, the specimen is centered on its side between the bearing blocks. The testing machine is used to apply diametral compression loading at a constant displacement rate of 0.5 mm/min until the maximum force required to cause failure of the specimen is indicated.

2. The tensile strength ( $\sigma_t$ ) is calculated from

$$\sigma_t = 2F/\pi DL$$

where  $F$  = maximum force applied along length of the cement specimen,

$D$  = diameter of the cement specimen, and

$L$  = length of the cement specimen.

#### E. Tests at Room Temperature and Atmospheric Pressure

1. The property tests listed above will be conducted on moist specimens after the set-cure and after one and four week exposures to the simulated geothermal fluids. Specimens will be stored in the 25 °C constant temperature bath prior to their testing. As Table I indicates, the tests will commence approximately four hours after the set-cure has been terminated and approximately 2 1/2 h after the exposures in solutions A and B.

2. The two permeability specimens for a given set cement will be used in the tests which follow a set-cure; and then these same specimens will be used again after their respective exposures in solutions A and B.

3. Only one shear-bond strength specimen will be tested following each set cure or exposure condition.

4. Three specimens sawed from a rod of a given set cement will be used each time that either a compressive strength test or a tensile strength test is conducted. Tables III and IV indicate the order of weeks when the specimens of the various set cements are removed from their exposures in either of the two simulated geothermal fluids. We plan to duplicate this series of tests at a later time.

#### F. Tests at Elevated Temperatures and Pressures

When the first series of tests at room temperature and atmospheric pressure are concluded, permeability and shear-bond strength test will be conducted at elevated pressure and temperature. Priority for selection of cements for these *in situ* tests will be based on the ranking of materials from the tests at room temperature and atmospheric pressure. Alternate *in situ* property tests are also being considered (for example, shear-bond strength to rock, tensile-bond strength to steel and to rock, and biaxial flexural strength), but we do not presently plan to conduct these tests on all of the candidate cementing materials.

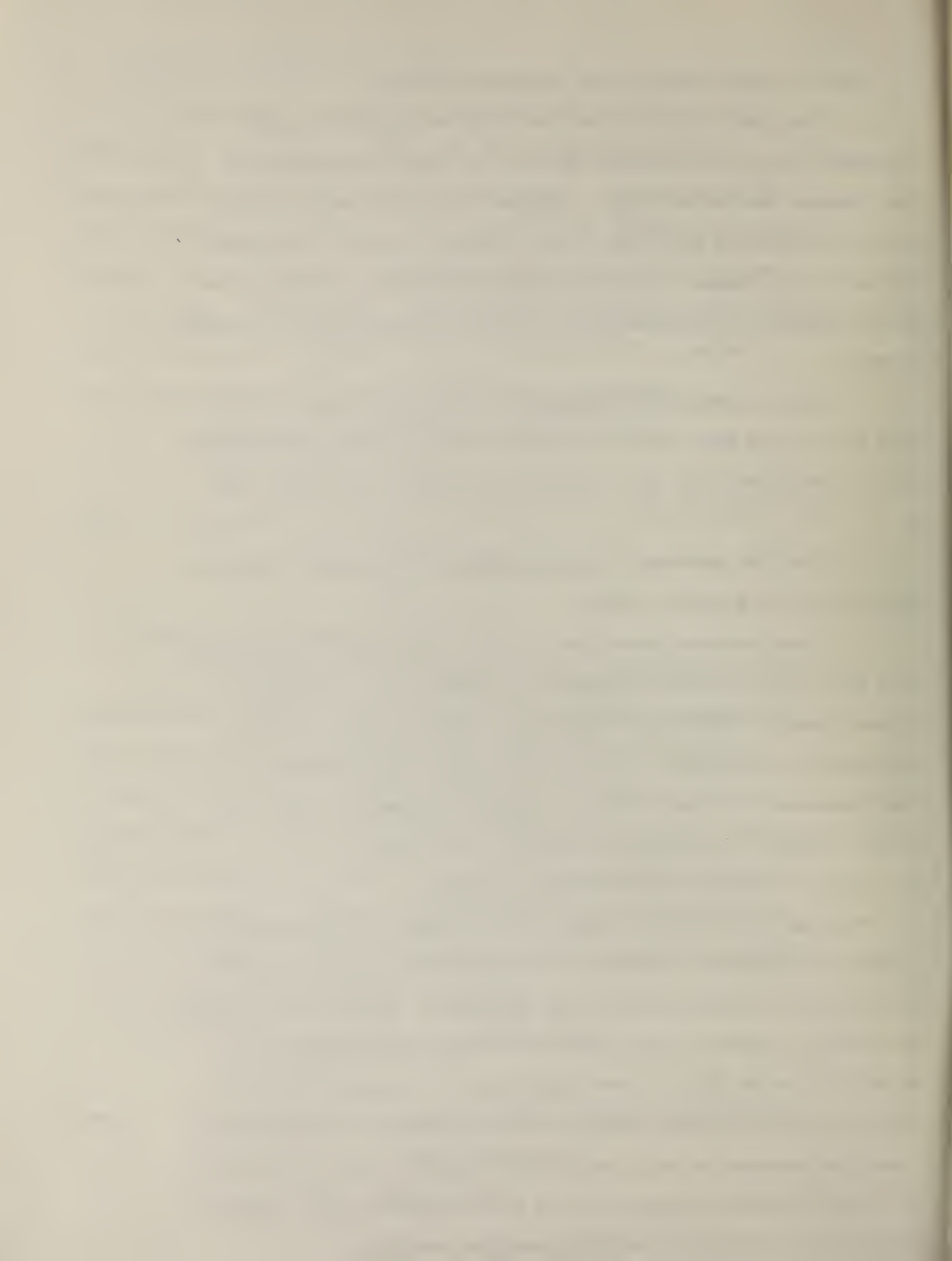


Table I. Weekly schedule of specimen preparation, exposures, and room temperature property testing.

Day	Time	Operation
Monday	8:30 am	Prepare slurry and fill molds.
	10:30 am	Assemble stirred autoclave.
	11:00 am	Commence set-curing.
	1:00 pm	Prepare simulated geothermal fluids.
Tuesday	8:30 am	Calculate results of previous week and prepare report.
Wednesday	11:00 am	Terminate set-curing.
	12:00 noon	Dismantle stirred autoclave.
	12:30 pm	Remove molds from specimens.
	1:30 pm	Saw specimens.
	3:00 pm	Conduct permeability tests on set-cured specimens.
	4:30 pm	Conduct load tests on set-cured specimens.
Thursday	8:30 am	Terminate exposure of specimens in first fluid.
	10:00 am	Dismantle two pressure vessels holding first fluid.
	11:00 am	Conduct permeability tests on exposed specimens.
	1:30 pm	Assemble two pressure vessels holding first fluid.
	2:00 pm	Commence resumption of exposure of specimens in first fluid.
	4:00 pm	Conduct load tests on exposed specimens.
Friday	8:30 am	Repeat Thursday schedule for specimens in second fluid.

Table II. Temperature, pressure, and composition of solutions <sup>a</sup> to be used for exposure of set-cements.

	Units	Solution A	Solution B
T	°C	190	320
P(total)	MPa (psi)	20 (2900)	20 (2900)
P(CO <sub>2</sub> ), 25°C	MPa (psi)	0.2 (30) <sup>b</sup>	0.2 (30) <sup>b</sup>
pH, 25°C		4.5	4.5
NaCl	mol/kg	0.31	2.3
KCl	mol/kg	0.019	0.36
CaCl <sub>2</sub>	mol/kg	0.020	0.66
NaHCO <sub>3</sub>	mol/kg	0.0015	0.0035
Density, 25°C	g/ml	1.01	1.16
Total dissolved solids	mass per cent	2	20

<sup>a</sup>The solutions A and B simulate light and heavy geothermal fluids, which are representative of the East Mesa 6-1 and Salton Sea areas, respectively (reference 6 and 5).

<sup>b</sup>These values are estimates based on data for ideal solutions from reference 7 and corrected by data for the salting out effect from reference 8.



Table III. Schedule of exposing compressive and tensile specimens of set cements in a simulated geothermal fluid at an elevated temperature and pressure.

Week	Cement	Specimens		
		Deposited	Withdrawn	Balance
0	a	12a		12
1	b	12b	6a	18
2	c	12c	6b	24
3			6c	18
4	d	12d	6a	24
5	e	12e	6b, 6d	24
6	f	12f	6c, 6e	24
7			6f	18
8			6d	12
9			6e	6
10			6f	0

Table IV. Schedule of exposing shear-bond (s) and permeability (p) specimens of set cements in a simulated geothermal fluid at an elevated temperature and pressure.

Week	Cement	Specimens				Balance
		Deposited	Withdrawn	Re-deposited		
0	a	2s <sub>a</sub> , P <sub>a</sub>				3
1	b	2s <sub>b</sub> , p <sub>b</sub>	s <sub>a</sub> , P <sub>a</sub>	p <sub>a</sub>		5
2	c	2s <sub>c</sub> , p <sub>c</sub>	s <sub>b</sub> , P <sub>b</sub>	p <sub>c</sub>		7
3			s <sub>c</sub> , P <sub>c</sub>	p <sub>c</sub>		6
4	d	2s <sub>d</sub> , p <sub>d</sub>	s <sub>a</sub> , P <sub>a</sub>			7
5	e	2s <sub>e</sub> , p <sub>e</sub>	s <sub>b</sub> , s <sub>d</sub> , P <sub>b</sub> , P <sub>d</sub>	p <sub>d</sub>		7
6	f	2s <sub>f</sub> , p <sub>f</sub>	s <sub>c</sub> , s <sub>e</sub> , P <sub>c</sub> , P <sub>e</sub>	p <sub>e</sub>		7
7			s <sub>f</sub> , P <sub>f</sub>	p <sub>f</sub>		6
8			s <sub>d</sub> , P <sub>d</sub>			4
9			s <sub>e</sub> , P <sub>e</sub>			2
10			s <sub>f</sub> , P <sub>f</sub>			0

## References

1. API Specification for Oil-Well Cements and Cement Additives, API Spec 10A, 19th edition, American Petroleum Institute, Dallas, Texas, April 1977.
2. API Recommended Practice for Testing Oil-Well Cements and Cement Additives, API RP 10B, 20th edition, American Petroleum Institute, Dallas, Texas, April 1977.
3. L. H. Eilers, E. B. Nelson, L. K. Moran, L. B. Sapngle, and B. E. Simpson, "Development of Geothermal Well Completion Systems", pp 91-106, Cementing of Geothermal Wells, Progress Report No. 8, BNL 50850, Brookhaven National Laboratory, Upton, NY, January-March 1978.
4. B. E. Simpson, Dowell Division of Dow Chemical USA, Tulsa, Oklahoma, 74102, Letters to L. E. Kukacka, Brookhaven National Laboratory, Upton, NY, February 17, 1978 and March 13, 1978.
5. S. R. Cosner and J. A. Apps, A Compilation of Data on Fluids from Geothermal Resources in the United States, Report LBL-5936, Lawrence Berkeley Laboratory, University of California, Berkeley, CA., May 1978.
6. James C. Watson, Battelle Pacific Northwest Laboratories, Richland, Washington, private communication to Ralph Krause, National Bureau of Standards, Washington, D. C., April 19, 1979.
7. D. D. Wagman, et al, Selected Values of Chemical Thermodynamic Properties, Technical Notes 270-3 (Jan. 1968) and 270-6 (Nov. 1971), National Bureau of Standards, Washington, D. C.
8. A. J. Ellis and R. M. Golding, The Solubility of Carbon Dioxide in Water and in Sodium Chloride Solutions, Amer. J. Sci. 261, 47 (1963).
9. A. H. Truesdell, Summary of Section III, Geochemical Techniques in Exploration, Proceedings UNIS Development and Use of Geothermal Resources, San Francisco, 1975.
10. S. K. Sanyal, R. M. Pirnie III, and G. O. Chen, "A Novel Liquid Permeameter for Measuring Very Low Permeability", Soc. Pet. Eng. J., 206-210, June 1972.
11. "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens," ANSI/ASTM C39-72, Annual Book of ASTM Standards, Part 14, American Society for Testing and Materials, Philadelphia, PA., 1977, pp. 25-28.
12. "Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens", ASTM C496-71, Annual Book of ASTM Standards, Part 14, American Society for Testing and Materials, Philadelphia, PA., 1977, pp 313-318.



U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBSIR 80-2099-2	2. Gov't. Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE TESTING GEOTHERMAL-WELL CEMENTS Standard Practice		5. Publication Date July 1980	
7. AUTHOR(S) Ralph F. Krause, Jr. and Edwin R. Fuller, Jr.		6. Performing Organization Code	
9. PERFORMING ORGANIZATION NAME AND ADDRESS NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, DC 20234		8. Performing Organ. Report No.	
12. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP) Dr. Robert R. Reeber Division of Geothermal Energy U.S. Department of Energy Washington, D.C. 20461		10. Project/Task/Work Unit No.	
15. SUPPLEMENTARY NOTES  <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.		11. Contract/Grant No. FA77-A-01-6010	
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)  The National Bureau of Standards is under contract with the U.S. Department of Energy to verify certain properties of cementing materials which are submitted as candidates for use in the finishing operations of geothermal wells. Specimens will be set-cured in molds under water for two days at elevated temperature and pressure. Subsequently, specimens will be exposed demolded to light and heavy simulated geothermal fluids for periods of one week or one month. Following each of these treatments, the following properties will be measured at room temperature and pressure: compressive strength, splitting tensile strength, shear-bond strength of the cement-steel interface, and cement permeability to water. Upon the basis of this survey of properties at room temperature, a priority of cementing materials will be established for further testing of select physical properties while the specimens are at elevated temperature and pressure.		13. Type of Report & Period Covered July 1979, Interim	
17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)  Compressive strength; exposure to geothermal fluids; geothermal-well cements; permeability to water; shear-bond strength to steel; splitting tensile strength.		14. Sponsoring Agency Code	
18. AVAILABILITY <input checked="" type="checkbox"/> Unlimited  <input type="checkbox"/> For Official Distribution. Do Not Release to NTIS  <input type="checkbox"/> Order From Sup. of Doc., U.S. Government Printing Office, Washington, DC 20402, SD Stock No. SN003-003-  <input checked="" type="checkbox"/> Order From National Technical Information Service (NTIS), Springfield, VA, 22161	19. SECURITY CLASS (THIS REPORT)  UNCLASSIFIED	21. NO. OF PRINTED PAGES  24	
	20. SECURITY CLASS (THIS PAGE)  UNCLASSIFIED	22. Price  \$5.00	





