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BMI/ONWI--522

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Thermal Property and Density Measurements of Samples Taken From Drilling Cores From Potential Geologic Media

Technical Report

December 1983

John F. Lagedrost
Webster Capps
of
Fiber Materials, Inc.

prepared for

Office of Nuclear Waste Isolation
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201

MASTER

ONWI
Office of Nuclear Waste Isolation

BATTELLE Project Management Division

BIBLIOGRAPHIC DATA

Lagedrost, John F., and Webster Capps, 1983. *Thermal Property and Density Measurements of Samples Taken From Drilling Cores From Potential Geologic Media,* BMI/ONWI-522, prepared by Fiber Materials, Inc., for Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, OH.

NOTICE

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price codes
Printed copy: A09
Microfiche copy: A01

BMI/ONWI-522
Distribution Category UC-70

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The content of this report was effective as of December 1981. This report was prepared by Fiber Materials, Inc. under Subcontract E515-00800 with Battelle Project Management Division, Office of Nuclear Waste Isolation under Contract Nos. EY-76-C-06-1830 and DE-AC02-83CH10140 with the U.S. Department of Energy.

ACKNOWLEDGMENTS

The authors wish to express appreciation to several persons who provided valuable assistance in the conduct of this work. These include Drs. Gilbert E. Raines and Michael M. Lemcoe of Battelle Memorial Institute, Office of Nuclear Waste Isolation, who were responsible for the technical monitoring of the subcontract. Gratitude is expressed to Edward Eldridge, Cliff Baker, Stephen Michaud, Robert St. John, Sue Joseph and Michael Carter, who performed most of the laboratory measurements, and to Julie Morrison, who typed the many reports and other communications. Finally, the consultation and cooperation of Marvin Morgan, Oak Ridge National Laboratory, is appreciated.

ABSTRACT

Density, steady-state conductivity, enthalpy, specific heat, heat capacity, thermal diffusivity and linear thermal expansion were measured on 59 materials from core drill samples of several geologic media, including rock salt, basalt, and other associated rocks from 7 potential sites for nuclear waste isolation. The measurements were conducted from or near to room temperature up to 500C, or to lower temperatures if limited by specimen cracking or fracturing. Ample documentation establishes the reliability of the property measurement methods and the accuracy of the results. Thermal expansions of salts reached 2.2 to 2.8 percent at 500C. Associated rocks were from 0.6 to 1.6 percent. Basalts were close to 0.3 percent at 500C. Specific heats of salts varied from 0.213 to 0.233 cal $g^{-1}C^{-1}$, and basalts averaged 0.239 cal $g^{-1}C^{-1}$. Thermal conductivities of salts at 50C were from 0.022 to 0.046 $wcm^{-1}C^{-1}$, and at 500C, from 0.012 to 0.027 $wcm^{-1}C^{-1}$. Basalts conductivities ranged from 0.020 to 0.022 $wcm^{-1}C^{-1}$ at 100C and 0.016 to 0.018 at 500C. There were no obvious conductivity trends relative to source location. Room temperature densities of salts were from 2.14 to 2.29 gcm^{-3}, and basalts, from 2.83 to 2.90 gcm^{-3}. The extreme friability of some materials made specimen fabrication difficult.

FOREWORD

This final report is submitted by the Energy Materials Testing Laboratory (EMTL), a Division of Fiber Materials, Incorporated (FMI), to the Office of Nuclear Waste Isolation (ONWI), Battelle Memorial Institute, in partial fulfillment of requirements under Subcontract No. E 515-00800. Staff who may be contacted regarding specific aspects of the work reported herein are:

Technical:	John F. Lagedrost, EMTL Manager, Thermophysics Laboratory Program Manager
	Webster Capps, EMTL Staff Physicist, Thermophysics Laboratory Associate Program Manager
Quality Assurance:	David Loper, FMI Manager, Quality Control
Contracts:	John F. Carr, FMI Manager, Contracts and Finance

The EMTL and FMI central phone number is (207) 282-5911

The address for both EMTL and FMI is:

Energy Materials Testing Laboratories
a Division of Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, Maine 04005

The FMI TWX number is 944480.

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1 INTRODUCTION

This is the final report on work performed under the indicated subcontract with Battelle Memorial Institute, Project Management Division, Office of Nuclear Waste Isolation (ONWI). The objective was thermal property and density evaluation of samples taken from drilling cores from geologic media which are potential storage sites for nuclear wastes. Broad classifications of the media include salt, granite, basalt, caprock, shale and tuff. This work was in support of efforts by ONWI on its Prime Contract EY-76-C-06-1880 with the U.S. Department of Energy.

The thermal properties selected for evaluation included thermal expansion, specific heat, and thermal conductivity, each in the range from approximately room temperature to 500C, or up to the temperature at which specimen integrity is lost due to decrepitation or shattering. Density was to be measured at room temperature only.

The materials evaluated under this subcontract were selected and furnished by the ONWI. The subcontract called for evaluation of fifty-nine (59) materials, plus reporting and technical evaluations. The materials included drilling core samples from the Vacherie Dome in Louisiana, the Cypress Creek Dome in Mississippi, the Salt Valley Dome in Utah, the Palo Duro Basin in Texas, the Richton Dome in Mississippi, the Gibson Dome in Utah, and the Pomona Formation in Washington.

The program was initiated in April, 1979. Sample materials were made available at irregular intervals during the ensuing 2½ years, with the final group arriving in July, 1981. During this period, monthly progress letters were submitted for those periods when work was in progress, and interim reports were submitted for each sample in material groups of four or more. The following is a listing of Interim Reports, showing material group and submittal date.

Material Group	No. of Samples	Reporting Date
Vacherie	4	Sept. 10, 1979
Cypress Creek	4	Sept. 25, 1979
Palo Duro	8	Mar. 7, 1980
Salt Valley	4	Sept. 15, 1980
Richton	6	Sept. 19, 1980
Vacherie	4	Sept. 25, 1980
Richton	4	July 30, 1981
Gibson	12	Sept. 10, 1981
Pomona	11	Sept. 25, 1981
Vacherie-Richton	2	Oct. 7, 1981

These reports presented preliminary results of all measurements in order to assist with early evaluation of the selected sites. This final report summarizes results of all work on the 59 materials, and presents appropriate conclusions and recommendations for future work.

The following sections describe the program materials, the property measurement techniques and procedures, results of all the measurements, and pertinent discussions. Appendices include descriptions of the measurement specifications, and a description of a separate study, not funded by this subcontract, to document the accuracy of our steady-state thermal conductivity measurements.

1.1 MATERIALS EVALUATED

The 59 materials evaluated in this study were selected from seven different sites. Tables 1-7 give details on the identification and location of individual samples from these sites. Note that each sample was furnished as a drilling core nominally 4 inches in diameter by 12 inches long, with the exception of the Pomona Basalt cores which were 1-3/4 inches in diameter by 3 to 6 inches long.

TABLE 1. Core Drilling Samples From the Vacherie Salt Dome, Louisiana

Core Box No.	Depth, Ft.	Description
4-11	681 - 682	Caprock
27-2	1922 - 1923	Salt
28-18	2023 - 2024	Salt
30-12	2122 - 2123	Salt
39-24	2643 - 2644	Salt
43-14	2852 - 2853	Salt
46-21	3045 - 3046	Salt
52-9	3245 - 3246	Salt
27-2*	1922 - 1923	Salt

*New specimen from original billet

TABLE 2. Core Drilling Samples From The Cypress Creek Salt Dome, Mississippi

Boring No.	Depth, Ft.	Description
MCCG-1	1299 - 1300	Caprock
MCCG-1	1599 - 1600	Salt
MCCG-1	1700 - 1701	Salt
MCCG-1	1800 - 1801	Salt

TABLE 3. Core Drilling Samples from The Palo Duro Basin, Texas (all salt samples)

Randall County
Rex White No. 1

Item No	Formation	Depth, Ft
1	Upper Seven Rivers	741
2*	Upper San Andres	1204
3	Upper San Andres	1400
4	Lower San Andres (Cycle 4)	1847
5	Lower San Andres (Cycle 2)	2143
6	Upper Clear Fork (Cycle 2)	2603
7*	Lower Clear Fork (Upper Cycle)	3347

Swisher County
D.M. Grabbe No. 1

Item No.	Formation	Depth, Ft
8	Upper Seven Rivers	1265
9	Upper San Andres	1955
10	Lower San Andres	2525
11*	Upper Clear Fork	3425

*These items not examined, per instructions from ONWI.

TABLE 4. Core Drilling Samples from Salt Valley DOE-3, Utah

Sample No.	Depth, Ft	Description
3-82	567 - 568	Salt
3-77	1952 - 1953	Salt
3-79	2165 - 2166	Salt
3-81	2516 - 2517	Salt

TABLE 5. Core Drilling Samples from Richton Dome, MRIG-9, Mississippi

Core Box No.	Depth, Ft.	Description
7-4	629 - 630	Caprock
11-20	796 - 797	Salt
12-13	858 - 859	Salt
15-21	966 - 967	Salt
16-3	1066 - 1067	Salt
20-9	1250 - 1251	Salt
Unknown	699 - 700	Caprock
Unknown	999 - 1000	Salt
Unknown	1240 - 1241	Salt
Unknown	1259 - 1260	Salt
11-20*	796 - 797	Salt

*New specimen from original billet.

TABLE 6. Core Drilling Samples from Gibson Dome-1, Utah

Core No.	Depth, Ft.	Description
GD-1-43	1299 - 1300	Limestone from Honaker Trail Formation
GD-1-44	2189 - 2190	Limestone with chert from Honaker Formation
GD-1-45	2639 - 2640	Siltstone
GD-1-46	2998 - 2999	Halite with anhydrite bands, Salt No. 5
GD-1-47	3094 - 3095	Silty dolomite
GD-1-48	3100 - 3101	Anhydrite with shale silt
GD-1-49	3111 - 3112	Siltstone
GD-1-50	3184 - 3185	Halite with anhydrite bands (red) Salt No. 6
GD-1-51	3339 - 3340	Halite with anhydrite bands (gray) Salt No. 6
GD-1-52	3369 - 3370	Shale with minor halite
GD-1-53	3438 - 3439	Halite with anhydrite, Salt No. 7
GD-1-54	3446 - 3447	Anhydrite with minor halite

TABLE 7. Core Drilling Samples from Pomona Formation Basalt, Washington (all basalt rock specimens)

Core No.	Depth, Ft.
IE3	20.9 - 21.2
IE3	21.2 - 21.5
IE6	1.5 - 1.9
IE6	11.3 - 11.6
IE6	16.8 - 17.0
IE6	21.0 - 21.5
IE6	24.7 - 25.0
IE7	6.6 - 7.0
IE20	10.7 - 11.1
IE20	17.7 - 18.2
IE20	26.8 - 27.0

1.2 SCOPE OF PROPERTY MEASUREMENTS

The task objectives included measurements of thermal expansion, specific heat, thermal conductivity, and density of specimens representing each of the program samples. Accuracy requirements included ±15 percent for thermal conductivity data, and ±5 percent for the other properties. Conductivity, expansion, and specific heat were evaluated through the range from room temperature to 500C, or up to the temperature at which the specimen integrity was lost due to decrepitation, shattering, etc. The conductivity measurements were made under 1 atm static air; the expansion and specific heat measurements, under flowing argon at 1 atm. In the case of the basalts, diffusivity was measured to determine conductivity. One-half atmosphere of helium was used in the specimen chamber.

Density was measured at room temperature only. Information on changes in density with temperature was not a requirement under this order but can be derived from the measured linear thermal expansion data if isotropic expansion is assumed.

1.3 MEASUREMENT TECHNIQUES

1.3.1 Thermal Expansion

Linear thermal expansion, always in the axial direction of the furnished core drillings, was measured by a recording quartz dilatometer, as described in Appendix A. In this technique, the specimen is supported between members of a fused silica structure. Their relative displacement as the specimen is heated, is recorded using a linear variable differential transformer (LVDT). The signal from this LVDT and

that of a thermocouple measuring specimen temperature are recorded simultaneously on an x-y plot to illustrate continuously the expansion-temperature curve.

1.3.2 Specific Heat

Specific heat was derived from enthalpy data measured in a drop (ice) calorimeter, as described in Appendix B. Sufficient enthalpy data were recorded to establish an enthalpy-temperature curve, the slope of which is specific heat. This slope was evaluated graphically and analytically.

1.3.3 Thermal Conductivity

In most cases, thermal conductivity was measured by the steady-state, comparative technique described in Appendix C. In all cases, measurement was in the axial direction of the furnished core drillings. This steady-state technique involves measurement of the temperature gradient resulting from transfer of a known quantity of heat, one-dimensionally through a slab specimen of known thickness, and calculation of conductivity from the Fourier equation.

For twelve of the fifty-nine materials, it was not possible to fabricate a test specimen of appropriate size to carry out the steady-state measurement. In these cases, conductivity was calculated as the product of thermal diffusivity, density, and specific heat. Thermal diffusivity was measured by the laser-pulse technique as described in Appendix D. This involves measurement of the time required for the transient thermal effect of a short-duration heat pulse to traverse a slab specimen of known thickness. Specific heat is measured as des-

cribed, and density, by the immersion technique as described below.

In both conductivity measurement approaches, data are recorded at a number of temperatures in the range examined to establish a curve of the property versus temperature. In this program, the number of points ranged from five to ten, or more.

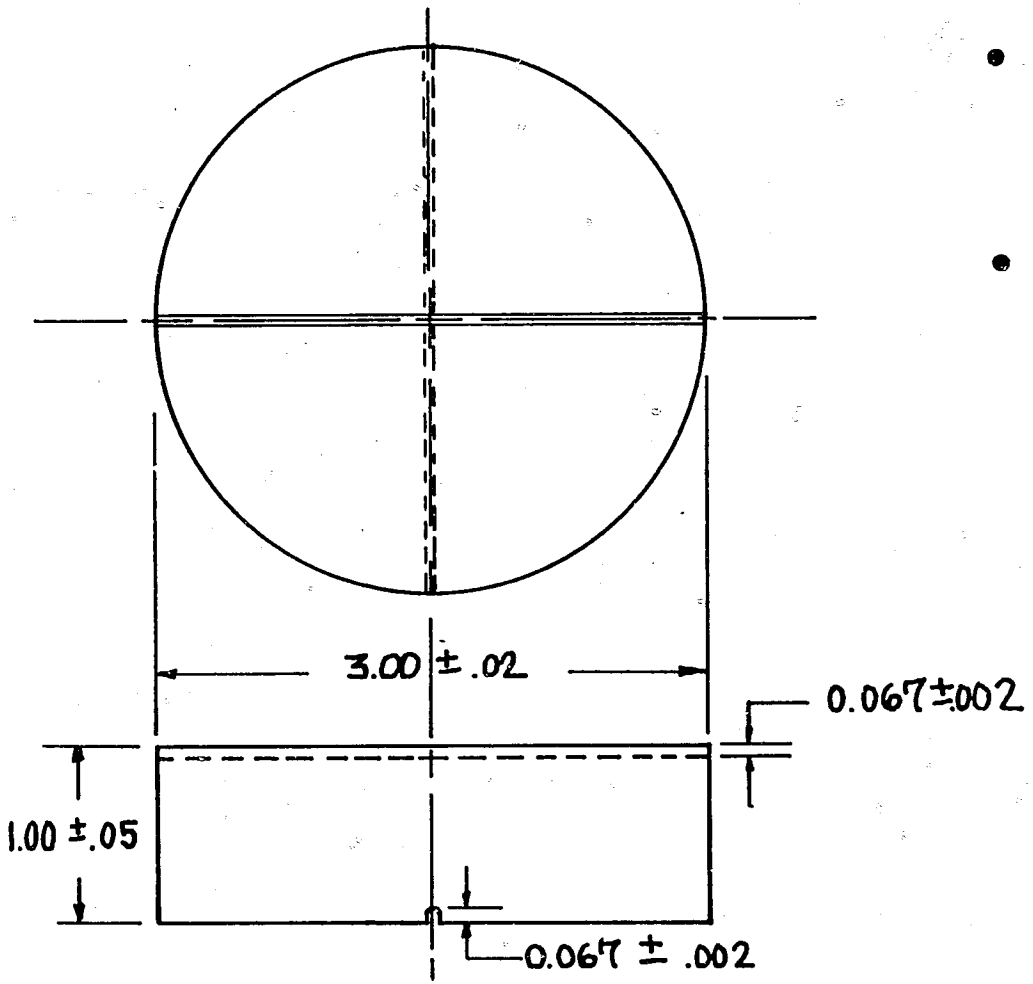
1.3.4 Density

In all cases, densities of samples of each material were measured by the immersion technique as described in Appendix E. This technique utilizes the Archimedes principle of buoyant force in a fluid of known density; the measurement is made at nominally room temperature.

1.4 SPECIMEN PREPARATION

Specimens for the measurement of steady-state thermal conductivity, and linear thermal expansion, were fabricated from the drilling samples according to the drawings illustrated on Figures 1 and 2. Figure 3 illustrates the thermal diffusivity specimen, as was fabricated to derive conductivity in 12 of the 59 samples.

No detailed specimen fabrication was necessary for the specific heat and the density specimens. An appropriate specimen for each property was parted from a representative section of the furnished core for each of these measurements.

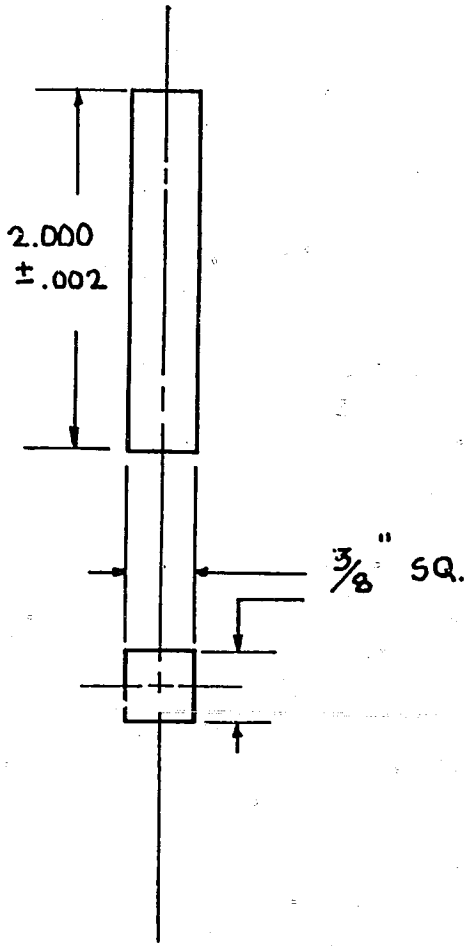


- FACES TO BE GROUND FLAT AND PARALLEL TO WITHIN 0.001 in.
- AXIS TO BE PARALLEL TO THAT OF BILLET

FIGURE 1. Specifications of Thermal Conductivity Specimen

FIBER MATERIALS, INC.	
THERMAL CONDUCTIVITY SPECIMEN	
DR. BY: JFL	CH. BY:
SCALE: 1:1	MAT'L:
REF. DWG:	DATE: 6-29-79
DWG. NO: EMTL-TP-1637-λ	SIZE A

FMI for materials ingenuity

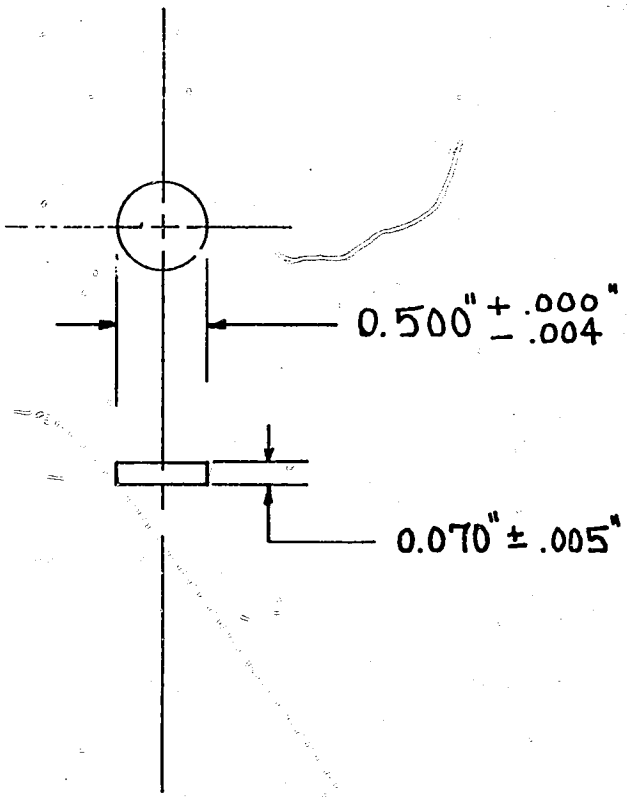


- ENDS TO BE FLAT AND PARALLEL
- AXIS TO BE PARALLEL WITH BILLET AXIS

FIGURE 2. Specifications of Linear Expansion Specimen

FIBER MATERIALS, INC.	
THERMAL EXPANSION SPECIMEN	
DR. BY: JFL	CH. BY:
SCALE: 1:1	MAT'L:
REF. DWG:	DATE: 6-29-79
DWG. NO: EMTL-TP-1637-LE	SIZE A

for materials ingenuity



- FACES TO BE GROUND FLAT AND PARALLEL
- AXIS TO BE PARALLEL TO THAT OF BILLET

FIGURE 3. Specifications of Thermal Diffusivity Specimen

FIBER MATERIALS, INC.	
THERMAL DIFFUSIVITY SPECIMEN	
DR. BY: JFL	CH. BY:
SCALE: NTS	MAT'L:
REF. DWG:	DATE: 6-29-79
DWG. NO: EMTL-TP-1637-α	SIZE A

for materials ingenuity

All machining was done dry, i.e., without any cutting lubricant. In most cases, the cutting was done with diamond abrasive disks.

Insofar as difficulty of specimen fabrication is concerned, the most prominent characteristics of the materials are the friable nature of many of the salt samples, and the hardness of many of the rock samples. Cracks, or fractures in some of the billets limited the available stock for machining and caused failure of some of the materials during machining. Following are comments on the fabrication of test specimens from the samples of the eight sites investigated.

Vacherie Dome

The material from the 2023-foot depth crumbled on unwrapping the billet. It was so friable that all attempts to fabricate expansion and conductivity specimens failed. Also, no conductivity specimen could be made from the 2851-foot depth, and no expansion specimens could be prepared from the 2643 and 3045-foot depths.

Cypress Creek

There were no machining problems associated with these materials.

Randall and Swisher Counties

No machining problems.

Salt Valley

The conductivity specimens from 1952 and 2165-foot depths showed some smearing on the machined faces.

Richton Dome

The caprock from the 629-foot depth peeled or delaminated perpendicular to the axis of the billet. No expansion specimen was fabricated. The caprock from the 699-foot depth chipped badly in machining the conductivity specimen and broke trying to make a 2-inch expansion specimen. It was necessary to use 3 smaller pieces for expansion measurements.

The salt from 1250 feet crumbled so badly that no conductivity specimen was machined.

Pieces broke off or crumbled so that the expansion specimen from the 1000-foot sample was in two pieces; the one from the 1240-foot depth was sawn by hand and finished by hand-filing. Pieces broke off the conductivity disks from the 1240 and 1260-foot depths. These two billets were broken on receipt.

Gibson Dome

Chips and fragments broke off of the conductivitiy disks from the 1300, 2639 and 3369-foot depths during fabrication.

Pomona Basalt

The billets from this source were too small for conductivity specimens. Instead of three-inch diameter disks for conductivity, it was necessary to make thin half-inch diameter "buttons" for diffusivity measurements. Hairline cracks in the billets caused a number of the buttons to fail during machining. Duplicates had to be made from a number of these materials.

2 MEASUREMENT RESULTS

The interim reports of this subcontract presented preliminary data on the pertinent properties for all program materials. This report summarizes and combines appropriate bodies of these data according to site. Combined plotting of the data is used to illustrate trends and to facilitate comparisons.

2.1 VACHERIE DOME, LOUISIANA

There are two groups of materials from the Vacherie Dome. They were tested separately but the results are combined in this report. Figure 4 presents linear thermal expansion data for all samples from this site. All are reproductions of the original curves recorded by the dilatometer, and indicate the depths from which the cores were removed.

Table 8 lists thermal conductivity data for all specimens of this site. They are co-plotted on Figure 5.

Table 9 lists all enthalpy data for specimens of the Vacherie Dome group. Specific heat values are derived from linear regression analysis of the caprock separately, each salt separately, and all of the salt data together.

Table 10 lists room temperature densities of all the specimens which were examined from this site.

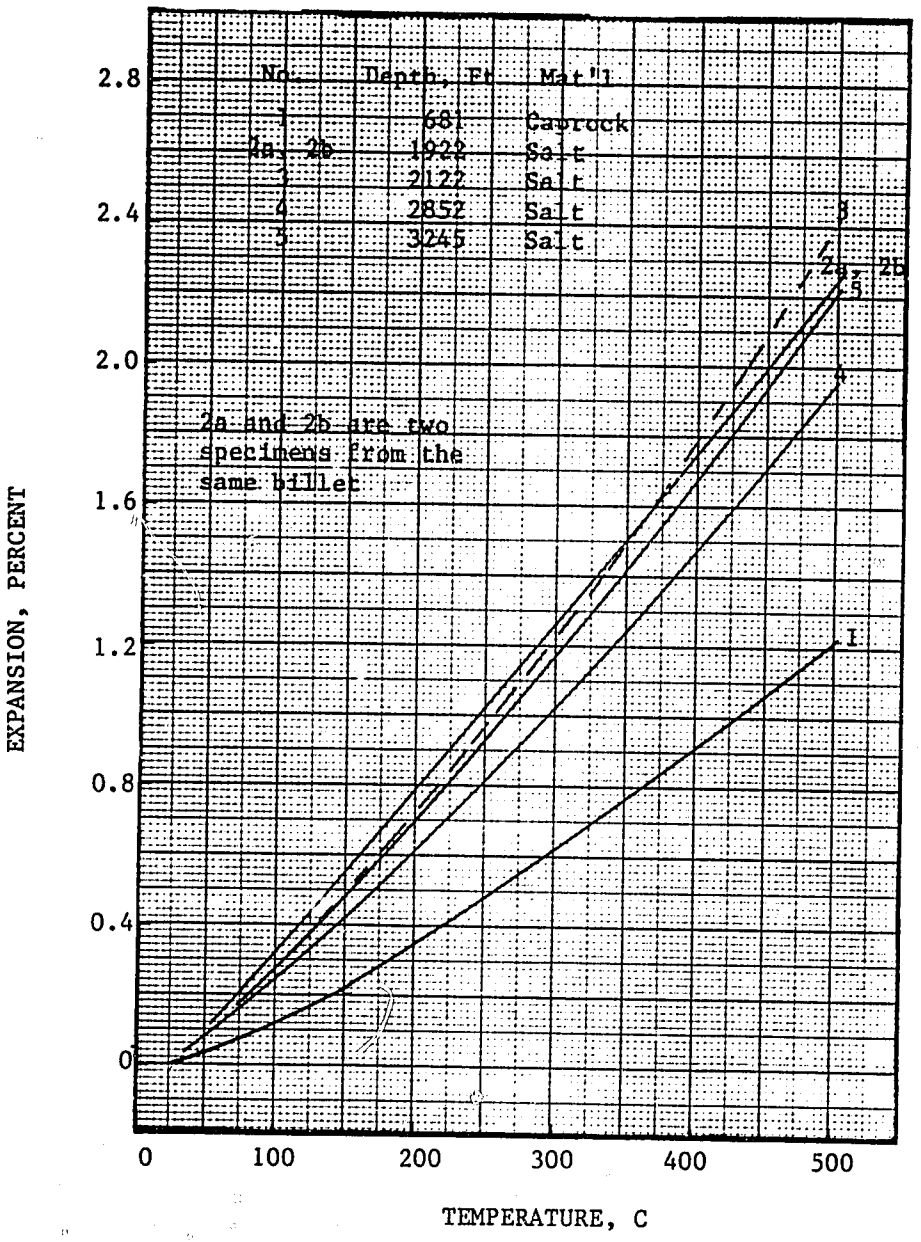


FIGURE 4. Axial Thermal Expansion of Specimens From Vacherie Salt Dome, Louisiana

TABLE 8. Thermal Conductivity Data for Specimens from Vacherie Dome, Mississippi

Specimen Identification Location	Depth, Ft	Temperature C	Conductivity, W $cm^{-1}C^{-1}$
4-11	681 - 682	60	0.0450
		98	0.0443
Caprock		134	0.0374
		236	0.0274
		380	0.0192
		515	0.0149
27-2	1922-1923	58	0.0289
Specimen #1		120	0.0284
Salt		165	0.0256
		232	0.0242
		303	0.0246
		382	0.0238
		465	0.0241
27-2	1922-1923	79	0.0299
Specimen #2		146	0.0272
		225	0.0242
Salt		303	0.0232
(New specimen from original billet with modified apparatus)		377	0.0225
		442	0.0232
		508	0.0241
30-12	2122-2123	63	0.0392
		106	0.0334
Salt		164	0.0304
		250	0.0269
		336	0.0233
		440	0.0229
		493	0.0230

Table 8, cont'd

39-24	2643-2644	66	0.0299
Salt		115	0.0259
		165	0.0248
		213	0.0236
		259	0.0230
		302	0.0228
		343	0.0235
		388	0.0236
		430	0.0240
46-21	3045-3046	67	0.0284
		116	0.0249
Salt		167	0.0235
		218	0.0223
		264	0.0216
		307	0.0214
		348	0.0218
		394	0.0219
		437	0.0221
46-21	3045-3046	136	0.0246
		139	0.0235
Rerun of specimen in modified apparatus		248	0.0223
Salt		294	0.0212
		424	0.0209
		432	0.0220
52-9	3245-3246	67	0.0380
		115	0.0323
Salt		167	0.0296
		216	0.0277
		264	0.0267
		309	0.0264
		356	0.0262
		403	0.0266
		448	0.0268

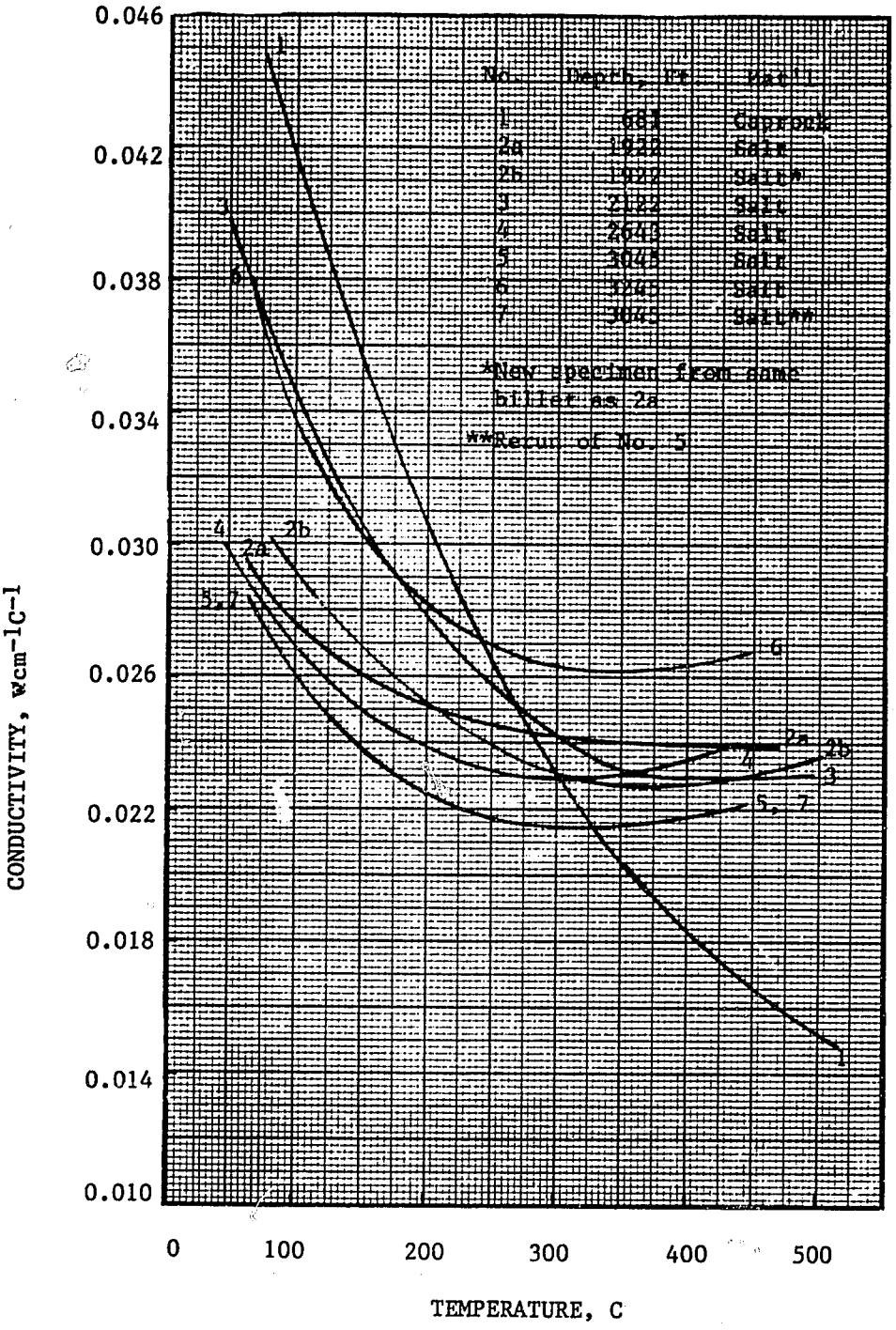


FIGURE 5. Axial Thermal Conductivity of Specimens from Vacherie Salt Dome, Louisiana

TABLE 9. Enthalpy and Specific Heat Data for Specimens from Vacherie Salt Dome, Louisiana

Specimen Location	Identification Depth, Ft.	Temperature C	Enthalpy cal g^{-1}	Specific Heat cal $g^{-1}C^{-1}$
4-11	681-682	0	0	0.214
	Caprock	99	17.943	
		268	55.424	
		390	82.735	
	A = -1.4642 B = 0.2139 Corr. Coeff. = 0.9993			
27-2	1922-1923	0	0	0.219
	Salt	99	20.002	
		267.4	56.151	
		390.5	85.740	
	A = -0.9827 B = 0.2191 Corr. Coeff. = 0.9994			
28-18	2023-2024	0	0	0.218
	Salt	98	19.918	
		266.7	58.501	
		390.0	84.027	
	A = -0.4348 B = 0.2176 Corr. Coeff. = 0.9998			
30-12	2122-2123	0	0	0.216
	Salt	98.0	19.810	
		266.4	57.662	
		390.3	83.696	
	A = -0.4820 B = 0.2161 Corr. Coeff. = 0.9998			
43-14	2852-2853	0	0	0.217
	Salt	22	4.728	
		60	12.426	
		95	19.926	
		142	30.051	
		275	59.029	
		341	74.196	
	A = -0.3879 B = 0.2172 Corr. Coeff. = 0.9999			

Table 9. cont'd

46-21	3045-3046	0	0	0.218
	Salt	22	4.877	
		59	12.426	
		96	19.939	
		142	30.045	
		275	59.526	
		340	73.934	
	A = -0.3481	B = 0.2175	Corr. Coeff. = 0.9999	
52.9	3245-3246	0	0	0.218
	Salt	23	4.796	
		59	12.389	
		96	20.023	
		143	30.078	
		274	58.942	
		340	74.026	
	A = -0.4473	B = 0.2175	Corr. Coeff. = 0.9999	

$H_o^T = A + BT$, where H_o^T is enthalpy from 0 to temperature T_c cal g^{-1}

Specific Heat = B, cal $g^{-1}C^{-1}$

Combined data for all Vacherie Salt Specimens

A = -0.4287 Specific Heat = 0.217 cal $g^{-1}C^{-1}$

B = 0.2173 Correlation Coefficient = 0.9998

TABLE 10. Room Temperature Density Data for All Specimens from Vacherie Salt Dome, Louisiana

Specimen Identification Location	Depth, Ft	Density g cm^{-3}	Material
4-11	681-682	2.93	Caprock
27-2	1922-1923	2.15	Salt
28-18	2023-2024	2.14	Salt
30-12	2122-2123	2.18	Salt
39-24	2643-2644	2.17	Salt
43-14	2852-2853	2.17	Salt
46-21	3045-3046	2.17	Salt
52-9	3245-3246	2.18	Salt

2.2 CYPRESS CREEK DOME, MISSISSIPPI

Figure 6 presents linear thermal expansion data for all specimens from this site and indicates the depths from which they were obtained.

Table 11 lists thermal conductivity data for all specimens from this site.

Figure 7 shows individual plots of the data for each specimen.

Table 12 lists all of the enthalpy data for specimens from this group and also the derived specific heat value representative of each specimen and a specific heat value for the entire group.

Table 13 lists room temperature densities of the specimens from this site.

2.3 PALO DURO BASIN, RANDALL AND SWISHER COUNTIES, TEXAS

Materials in this group were obtained from two sites, one in Randall County and the other in Swisher County, Texas. Although the specimens came from different core-holes, they were treated as one group of materials. The sources, however, are identified in the tables and figures.

Figure 8 presents linear thermal expansion data for all specimens and indicates the depths from which they came.

Table 14 lists thermal conductivity data, and Figure 9 shows plots of conductivity versus temperature for all specimens.

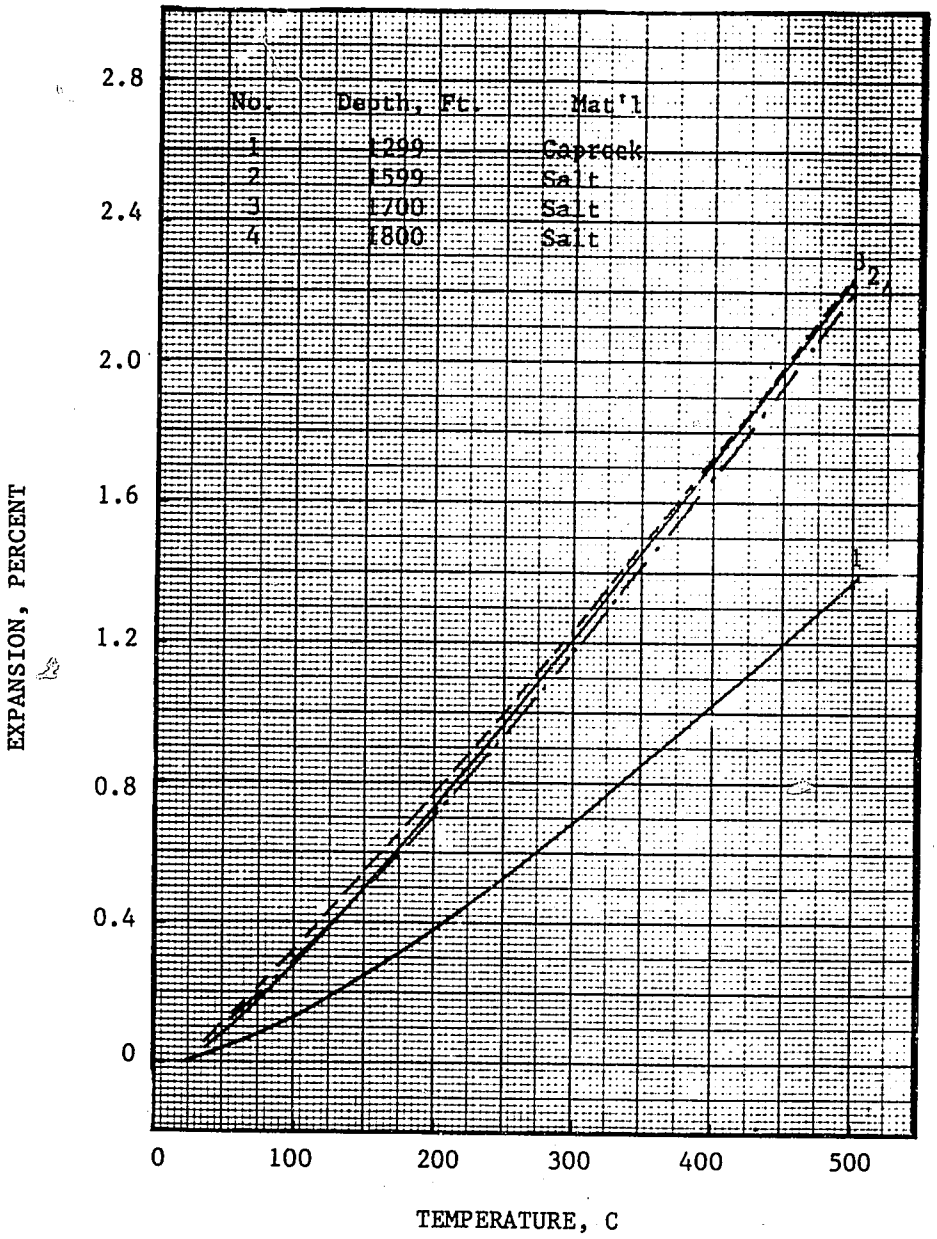


FIGURE 6. Axial Thermal Expansion of Specimens From Cypress Creek Salt Dome, Mississippi

TABLE 11. Thermal Conductivity Data for Specimens from Cypress Creek Salt Dome, Mississippi

Specimen Identification Location	Depth, Ft	Temperature C	Conductivity W $cm^{-1}C^{-1}$
MCCG-1	1299-1300	57	0.0417
Caprock		90	0.0362
		147	0.0298
		255	0.0250
		322	0.1900
		395	0.0180
		407	0.0185
		436	0.0167
MCCG-1	1599-1600	57	0.0397
Salt		88	0.0352
		158	0.0292
		177	0.0268
		258	0.0245
		268	0.0232
		335	0.0212
		342	0.0210
		425	0.0198
MCCG-1	1700-1701	50	0.0400
Salt		79	0.0380
		119	0.0332
		160	0.0292
		230	0.0258
		275	0.0238
		328	0.0220
		432	0.0200
		480	0.0198

TABLE 11. cont'd

MCCG-1	1800-1801	50	0.0383
Salt		72	0.0355
		104	0.0322
		175	0.0275
		214	0.0257
		257	0.0237
		336	0.0220
		392	0.0200
		462	0.0218

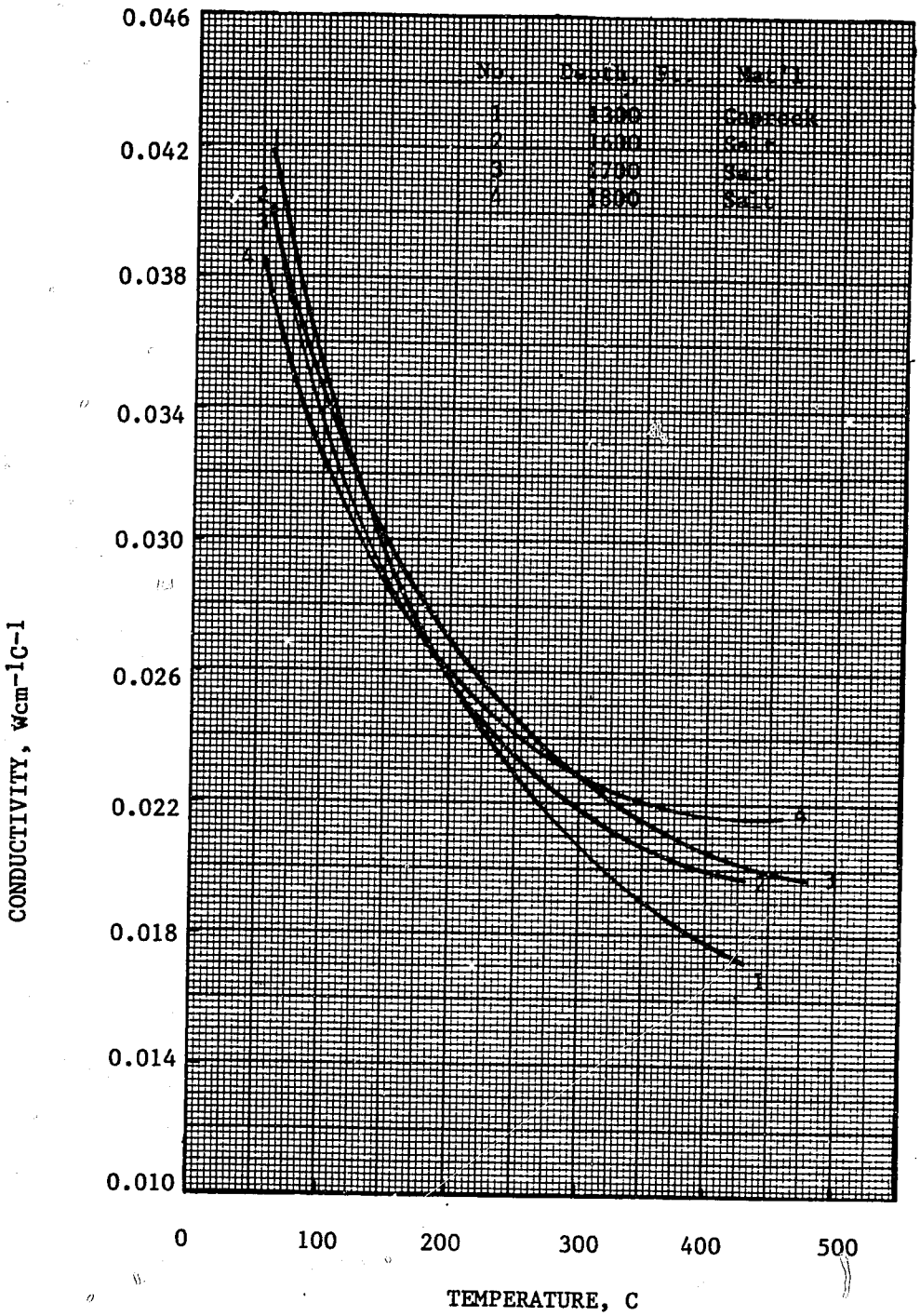


FIGURE 7. Axial Thermal Conductivity of Specimens from Cypress Creek Salt Dome, Mississippi

TABLE 12. Enthalpy and Specific Heat Data for Specimens from Cypress Creek Salt Dome, Mississippi

Specimen Identification		Temperature	Enthalpy	Specific Heat
Location	Depth, Ft.	C	cal g^{-1}	cal $g^{-1}C^{-1}$
MCCG-1	1299-1300	0	0	0.267
Caprock		50.0	9.717	
		121.5	27.342	
		210.3	51.318	
		297.8	78.143	
		373.1	97.576	
A = -2.8146 B = 0.2669 Corr. Coeff. = 0.9986				
MCCG-1	1599-1600	0	0	0.217
Salt		50.0	10.990	
		121.0	26.431	
		209.3	44.367	
		297.1	64.924	
		380.8	82.577	
A = -0.0259 B = 0.2168 Corr. Coeff. 0.9999				
MCCG-1	1700-1701	0	0	0.213
Salt		50.0	10.755	
		121.0	25.814	
		209.5	44.358	
		297.2	63.318	
		385.5	82.451	
A =-0.0389 B = 0.2134 Corr. Coeff. = 1.0000				
MCCG-1	1800-1801	0	0	0.216
Salt		50.0	11.069	
		121.0	25.777	
		209.6	43.886	
		296.3	63.138	
		385.3	83.835	
A = -0.2316 B = 0.2157 Corr. Coeff. 0.9997				
Combined Data for all Cypress Creek Salt Specimens				
A = -0.0967		Specific Heat = 0.215 cal $g^{-1}C^{-1}$		
B = 0.2153		Correlation Coefficient = 0.9998		

TABLE 13. Room Temperature Density Data for Specimens from Cypress Creek Salt Dome, Mississippi

Specimen Identification Location	Depth, Ft	Density g cm^{-3}	Material
MCCG-1	1300	2.96	Cap Rock
MCCG-1	1600	2.18	Salt
MCCG-1	1700	2.21	Salt
MCCG-1	1800	2.14	Salt

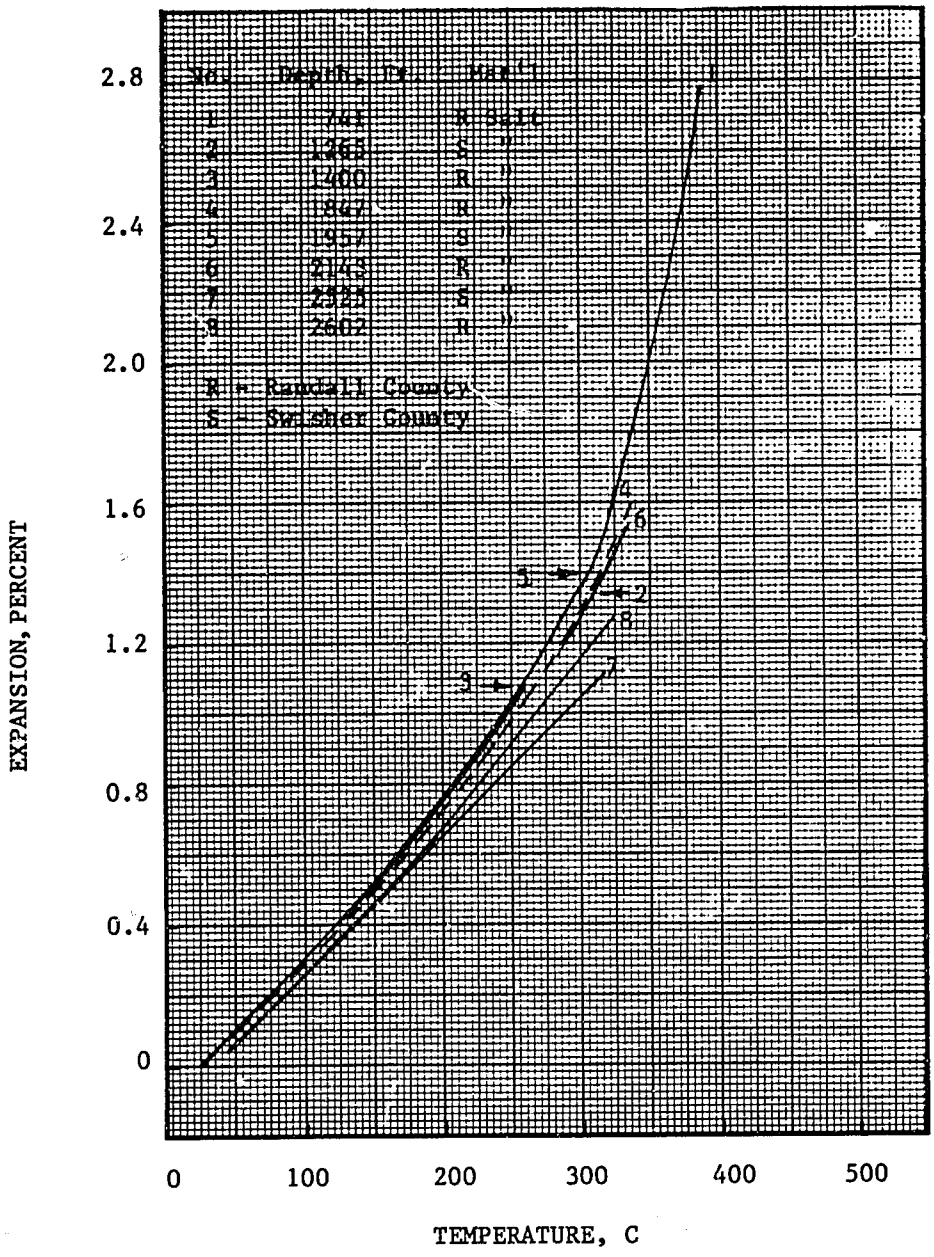


FIGURE 8. Axial Thermal Expansion of Specimens from Palo Duro Salt Dome, Texas

TABLE 14. Thermal Conductivity Data for Specimens from Palo Duro Salt Dome, Texas

Specimen Identification Location	Depth, ft	Temperature C	Conductivity W cm-1C-1
Randall County			
Upper Seven Rivers	741-742	53	0.0299
		78	0.0305
Salt		63	0.0294
		75	0.0299
		117	0.0261
		156	0.0237
		155	0.0242
		30	0.0377
		196	0.0213
		45	0.0370
		251	0.0187
		309	0.0174
		380	0.0153
		221	0.0200
		443	0.0135
Upper San Andres	1400-1401	25	0.0372
		39	0.0382
Salt		68	0.0372
		99	0.0322
		152	0.0287
		196	0.0261
		241	0.0240
		303	0.0226
Lower San Andres	1847-1848	26	0.0433
		43	0.0384
(Cycle 4)		80	0.0411
Salt		119	0.0335
		171	0.0300
		217	0.0263
		298	0.0228
		363	0.0206

TABLE 14. cont'd

Lower San Andres	2143-2144	24	0.0466
		47	0.0422
(Cycle 2)		75	0.0395
Salt		112	0.0347
		158	0.0304
		217	0.0267
		269	0.0243
		351	0.0200
		419	0.0174
Upper Clear Fork	2602-2603	29	0.0218
		47	0.0233
(Cycle 2)		76	0.0228
Salt		108	0.0204
		164	0.0174
		210	0.0161
		168	0.0176
		319	0.0137
Swisher County			
Upper Seven Rivers	1265-1266	28	0.0332
		41	0.0321
Salt		84	0.0297
		135	0.0258
		213	0.0227
		215	0.0227
		172	0.0248
Upper San Andres	1957-1958	36	0.0403
		80	0.0380
Salt		135	0.0299
		228	0.0256
		300	0.0226
		368	0.0190
		397	0.0186

TABLE 14, cont'd — Pg 3 of 3

Lower San Andres	2525-2526	34	0.0382
		80	0.0361
Salt		139	0.0297
		238	0.0248
		308	0.0224

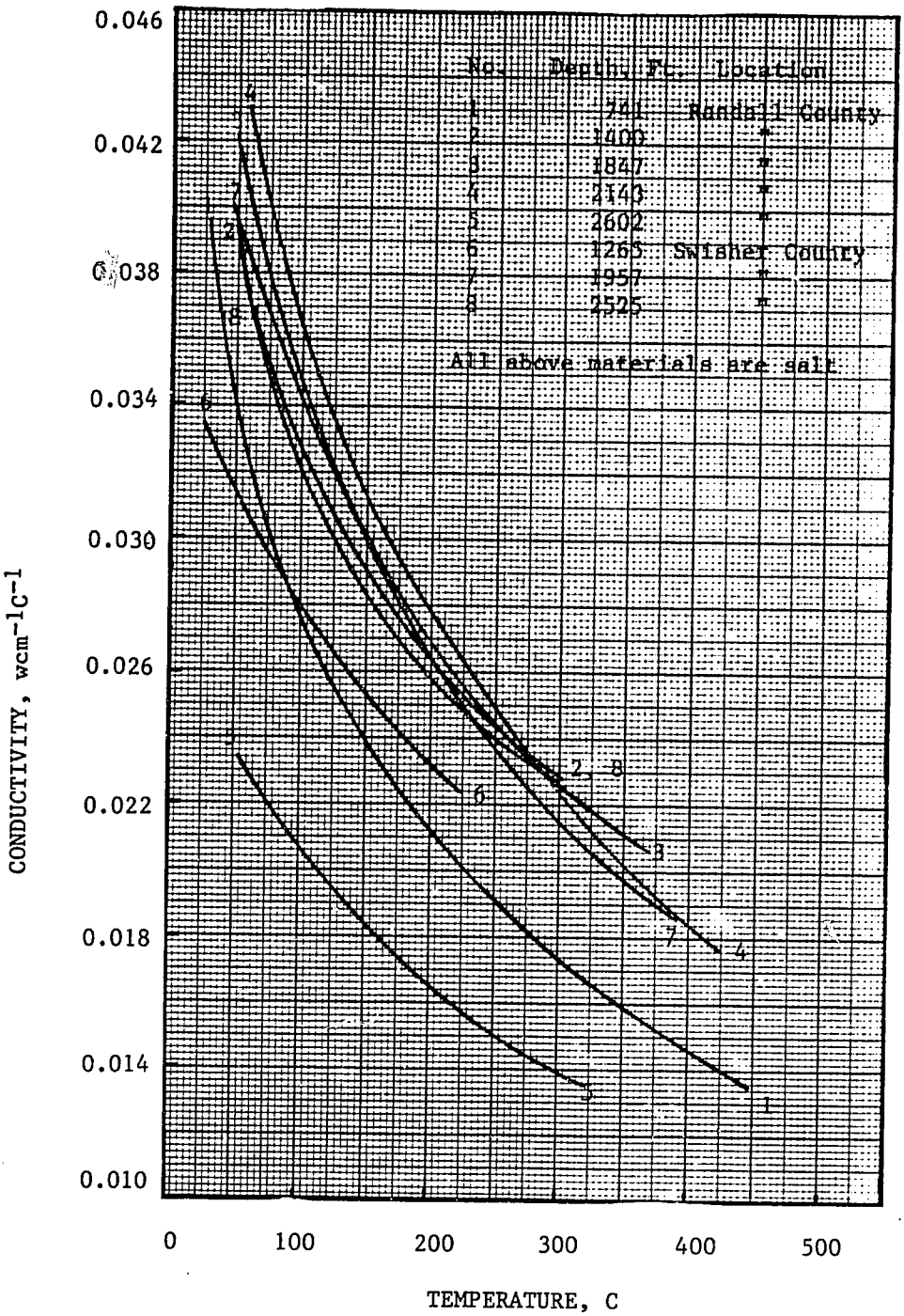


FIGURE 9. Axial Thermal Conductivity of Specimens from Palo Duro Salt Dome, Texas

Table 15 lists the enthalpy data and the derived average specific heats for each material. Also, an average specific heat value representing all of the Palo Duro specimens is given.

Table 16 lists room temperature density values for all Palo Duro specimens.

2.4 SALT VALLEY, UTAH

Linear thermal expansion data are shown in Figure 10.

Table 17 lists thermal conductivity data, and Figure 11 shows plots of conductivity versus temperature for all Salt Valley materials.

Table 18 gives enthalpies and derived specific heats. The average specific heat for each specimen is shown, and a specific heat value for the whole Salt Valley group is shown also.

Table 19 lists density values for all specimens.

2.5 RICHTON DOME, MISSISSIPPI

Materials were received in two separate shipments, but came from the same site. All materials are presented here as one group of materials.

Figure 12 presents linear thermal expansion data for each specimen.

Table 20 lists thermal conductivity data for each material (some salt, some caprock), and Figure 13 shows a separate conductivity curve for each material.

TABLE 15. Enthalpy and Specific Heat Data for Specimens from Palo Duro (Randall and Swisher Counties), Texas

Specimen Identification Location	Depth, Ft.	Temperature C	Enthalpy, cal g^{-1}	Specific Heat cal $g^{-1}C^{-1}$
Randall County	741-742	0	0	0.217
Upper Seven Rivers		22.3	4.751	
Salt		55.4	11.589	
		115.7	24.921	
		157.6	33.564	
		232.1	50.580	
		318.0	68.813	
A = -0.2059 B = 0.2171 Corr. Coeff. = 0.9999				
Upper San Andres	1400-1401	0	0	0.215
		21.5	4.342	
Salt		56.0	11.662	
		113.9	24.005	
		156.3	32.809	
		231.6	49.391	
		322.7	69.539	
A = -0.3623 B = 0.2154 Corr. Coeff. = 0.9999				
Lower San Andres	1847-1848	0	0	0.216
(Cycle 4)		22.3	4.719	
Salt		55.6	11.573	
		115.0	24.324	
		156.8	32.927	
		231.8	49.578	
		319.4	68.957	
A = -0.3055 B = 0.2156 Corr. Coeff. = 0.9999				

Table 15, cont'd

Lower San Andres 2143-2144	0	0	0.214
(Cycle 2)	21.5	4.374	
Salt	56.0	11.809	
	114.6	24.192	
	156.3	33.087	
	231.6	49.509	
	322.3	68.807	

A = -0.1792 B = 0.2139 Corr. Coeff. = 1.0000

Upper Clear Fork 2602-2603	0	0	0.217
(Cycle 2)	20.8	4.202	
Salt	56.0	11.499	
	113.7	23.764	
	156.3	32.673	
	231.8	49.774	
	322.7	69.889	

A = -0.5087 B = 0.2168 Corr. Coeff. = 0.9998

Swisher County			
Upper Seven Rivers 1265-1266	0	0	0.215
Salt	21.2	4.404	
	56.0	11.595	
	113.7	23.897	
	156.3	33.100	
	231.5	50.155	
	326.0	69.686	

A = -0.2448 B = 0.2150 Corr. Coeff. = 0.9999

Upper San Andres 1957-1958	0	0	0.214
Salt	21.5	4.604	
	56.0	11.943	
	113.3	23.822	
	156.9	33.005	
	231.5	48.991	
	324.4	69.571	

A = -0.1527 B = 0.2136 Corr. Coeff. = 0.9999

Table 15, cont'd

Lower San Andres 2525-2526	0	0	0.216
Salt	21.5	4.278	
	56.0	11.559	
	113.3	23.774	
	157.1	33.057	
	231.8	49.623	
	323.6	69.570	

A = -0.3953 B = 0.2155 Corr. Coeff. = 0.9999

Combined Enthalpy Data for all Palo Duro Salt Specimens:

A = -0.3031 Specific Heat = 0.215 cal $g^{-1}C^{-1}$

B = 0.2154 Correlation Coefficient = 0.9999

TABLE 16. Room Temperature Density Data for Specimens From Palo Duro (Randall and Swisher Counties), Texas

Specimen Identification Location	Depth Ft	Density g cm^{-3}	Material
Randall County			
Upper Seven Rivers	741-742	2.14	Salt
Upper San Andres	1400 - 1401	2.16	Salt
Lower San Andres	1847 - 1848	2.17	Salt
Lower San Andres	2143 - 2144	2.16	Salt
Upper Clear Fork	2602 - 2603	2.33	Salt
Swisher County			
Upper Seven Rivers	1265 - 1266	2.18	Salt
Upper San Andres	1957 - 1958	2.21	Salt
Lower San Andres	2525 - 2526	2.15	Salt

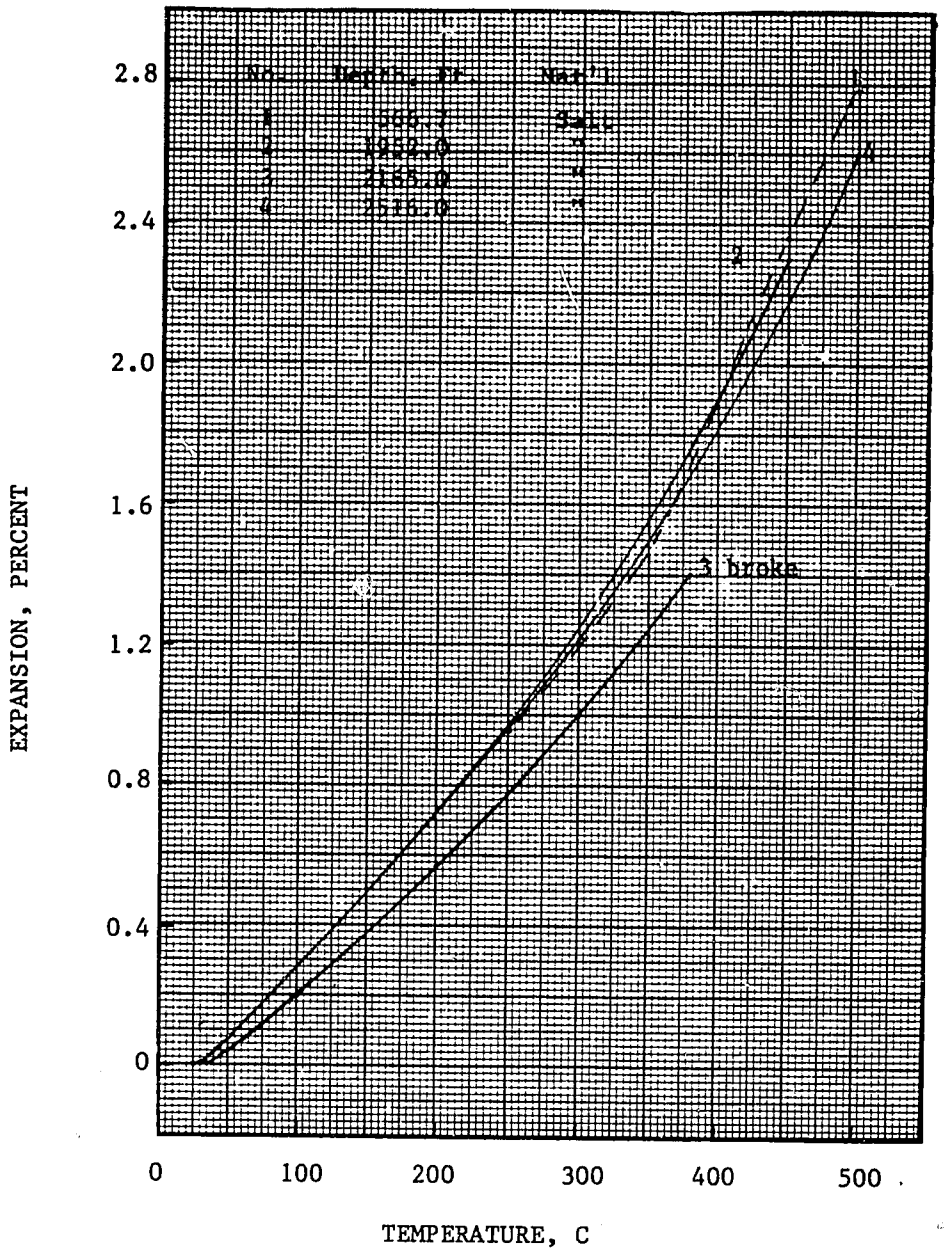


FIGURE 10. Axial Thermal Expansion of Specimens from Salt Valley, Utah

TABLE 17. Thermal Conductivity Data for Specimens from Salt Valley, Utah

Specimen Identification Location	Depth, Ft	Temperature C	Conductivity W cm^{-1}C^{-1}
3-82	566.7-567.7	46	0.0406
		90	0.0362
Salt		132	0.0322
		175	0.0300
		219	0.0282
		259	0.0262
		295	0.0252
		331	0.0247
		370	0.0244
3-77	1952-1953	46	0.0324
		86	0.0290
Salt		127	0.0263
		168	0.0253
		208	0.0233
		246	0.0219
		283	0.0210
		318	0.0205
		354	0.0203
3-77	1952-1953	69	0.0279
		80	0.0274
Salt		139	0.0244
		144	0.0246
		187	0.0232
Rerun with modified apparatus		236	0.0214
		285	0.0206
		293	0.0202
		372	0.0201
		436	0.0200

TABLE 17. cont'd

3-79	2165-2166	48	0.0383
		86	0.0338
Salt		127	0.0300
		171	0.0278
		211	0.0257
		262	0.0239
		287	0.0233
		320	0.0222
		358	0.0193
3-81	2516-2517	46	0.0393
		85	0.0347
Salt		125	0.0308
		168	0.0289
		210	0.0275
		247	0.0255
		283	0.0244
		320	0.0237
		355	0.0230

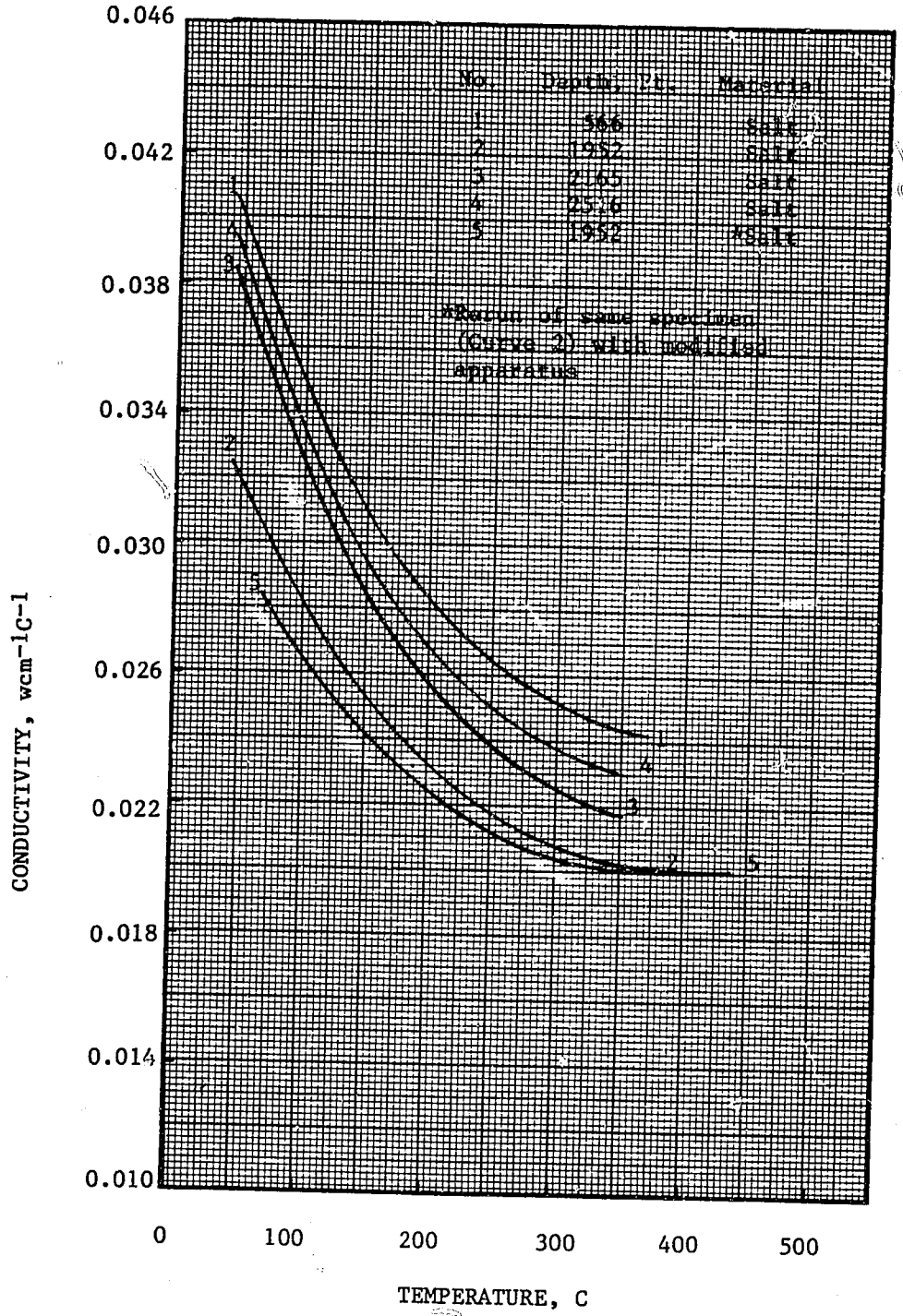


FIGURE 11. Axial Thermal Conductivity of Specimens from Salt Valley Salt Dome, Utah

TABLE 18. Enthalpy and Specific Heat Data for Specimens from Salt Valley, Utah

Specimen Identification Location	Depth, Ft	Temperature C	Enthalpy cal g^{-1}	Specific Heat cal g^{-1} C^{-1}
3-82	566.7-567.7	0	0	0.217
Salt		22	4.675	
		51	10.680	
		91	19.038	
		163	34.289	
		269	57.694	
		361	78.325	
	A = -0.3692	B = 0.2166	Corr. Coeff. = 0.9999	
3-77	1952-1953	0	0	0.217
		22	4.543	
		50	10.606	
		91	18.868	
		165	34.758	
		272	57.646	
		386	84.083	
	A = -0.4551	B = 0.2167	Corr. Coeff. = 0.9998	
3-79	2165-2166	0	0	0.216
		22	4.596	
		50	10.658	
		91	19.065	
		164	34.627	
		271	57.507	
		372	80.950	
	A = -0.3527	B = 0.2164	Corr. Coeff. = 0.9998	

Table 18, cont'd

3-81	2516 - 2517	0	0	0.217
		22	4.636	
		50	10.696	
		91	19.035	
		163	34.513	
		270	57.753	
		365	79.353	
	A = -0.3189	B = 0.2167	Corr. Coeff. = 0.9999	

Combined Data for all Salt Valley Specimens

A = -0.3736 Specific Heat = 0.217 cal $g^{-1}C^{-1}$

B = 0.2166 Correlation Coefficient = 0.9999

TABLE 19. Room Temperature Density Data for Specimens from Salt Valley, Utah

Specimen Identification Location	Depth, Ft	Density g cm^{-3}	Material
3-82	566.7 - 567.7	2.16	Salt
3-77	1952 - 1953	2.17	Salt
3-79	2165 - 2166	2.18	Salt
3-81	2516 - 2517	2.17	Salt

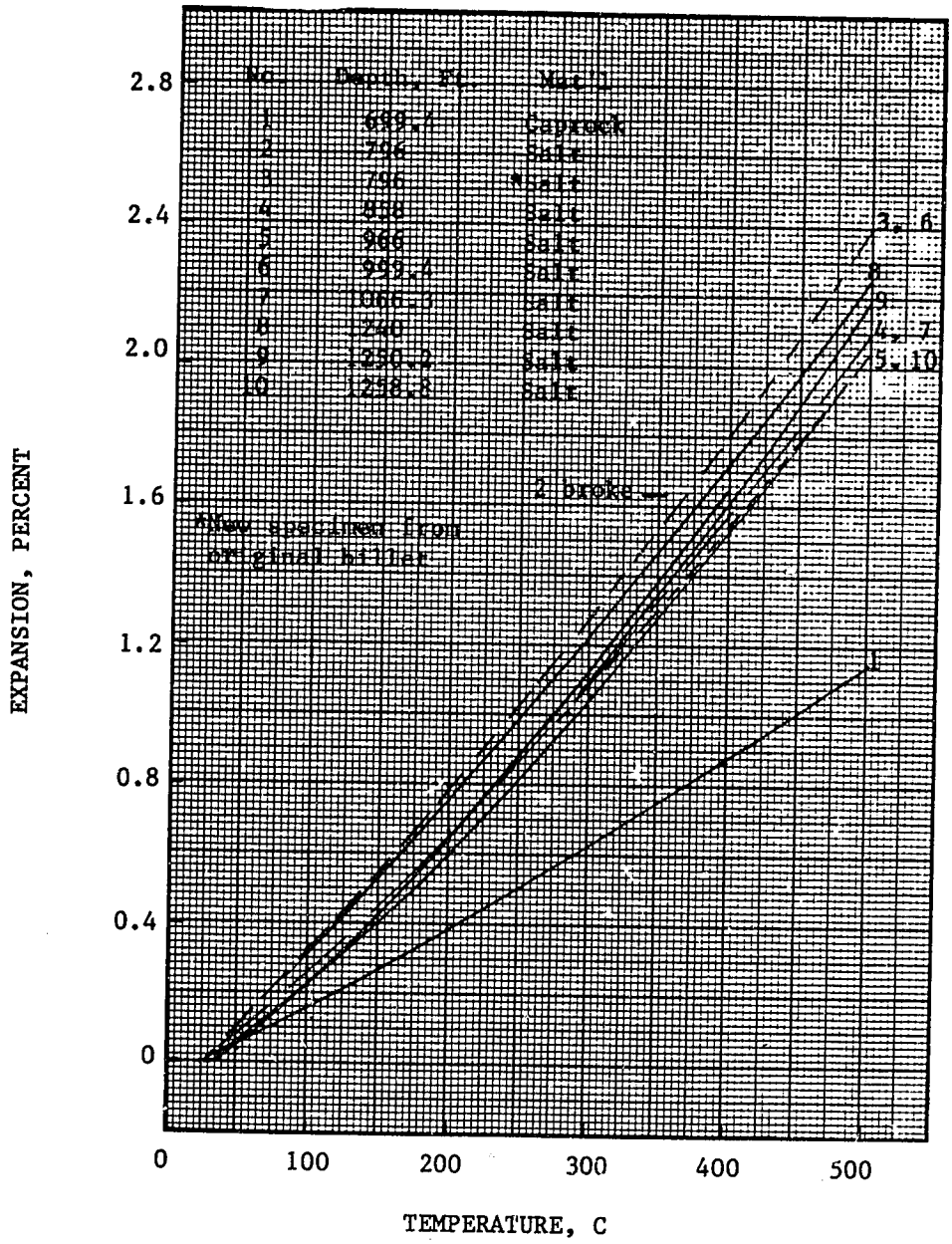


FIGURE 12. Axial Thermal Expansion of Specimens from Richton Salt Dome, Mississippi

TABLE 20. Thermal Conductivity Data for Specimens from Richton Salt Dome, Mississippi

Specimen Identification Location	Depth, Ft	Temperature C	Conductivity W cm-1C-1
7-4	628.6-629.7	46	0.0199
Caprock		93	0.0171
		133	0.0117
		176	0.0090
		221	0.0089
		263	0.0089
		298	0.0087
		336	0.0083
		374	0.0080
-	700	76	0.0362
Caprock		129	0.0325
		216	0.0213
		312	0.0173
		360	0.0158
		435	0.0137
		516	0.0126
11-20	796	48	0.0344
		89	0.0298
Salt		129	0.0271
		175	0.0279
		223	0.0238
		267	0.0221
		314	0.0211
		357	0.0193
		408	0.0197
		454	0.0200

TABLE 20., cont'd

11-20	796	79	0.0252
		144	0.0229
Salt		225	0.0209
		295	0.0196
New specimen from original billet measured with modified apparatus.		375	0.0196
		440	0.0197
		507	0.0202
12-13	858	69	0.0323
		120	0.0285
Salt		174	0.0260
		225	0.0233
		274	0.0219
		318	0.0209
		362	0.0207
		409	0.0203
		454	0.0201
15-21	966	48	0.0324
		99	0.0288
Salt		152	0.0263
		205	0.0240
		254	0.0223
		298	0.0214
		343	0.0208
		390	0.0204
		436	0.0201
-	1000	134	0.0374
Salt		193	0.0324
		269	0.0277
		332	0.0250
		388	0.0238
		444	0.0227
		505	0.0223
		86	0.0434

TABLE 20. cont'd

16-3	1066.3	49	0.0363
		101	0.0322
Salt		155	0.0292
		209	0.0265
		260	0.0243
		306	0.0229
		352	0.0223
		398	0.0216
		444	0.0208
-	1240	64	0.0272
Salt		127	0.0264
		200	0.0246
		262	0.0240
		293	0.0226
		363	0.0216
		436	0.0213
		503	0.0223
-	1260	67	0.0293
		127	0.0289
Salt		201	0.0278
		297	0.0259
		366	0.0266
		440	0.0259
		504	0.0263

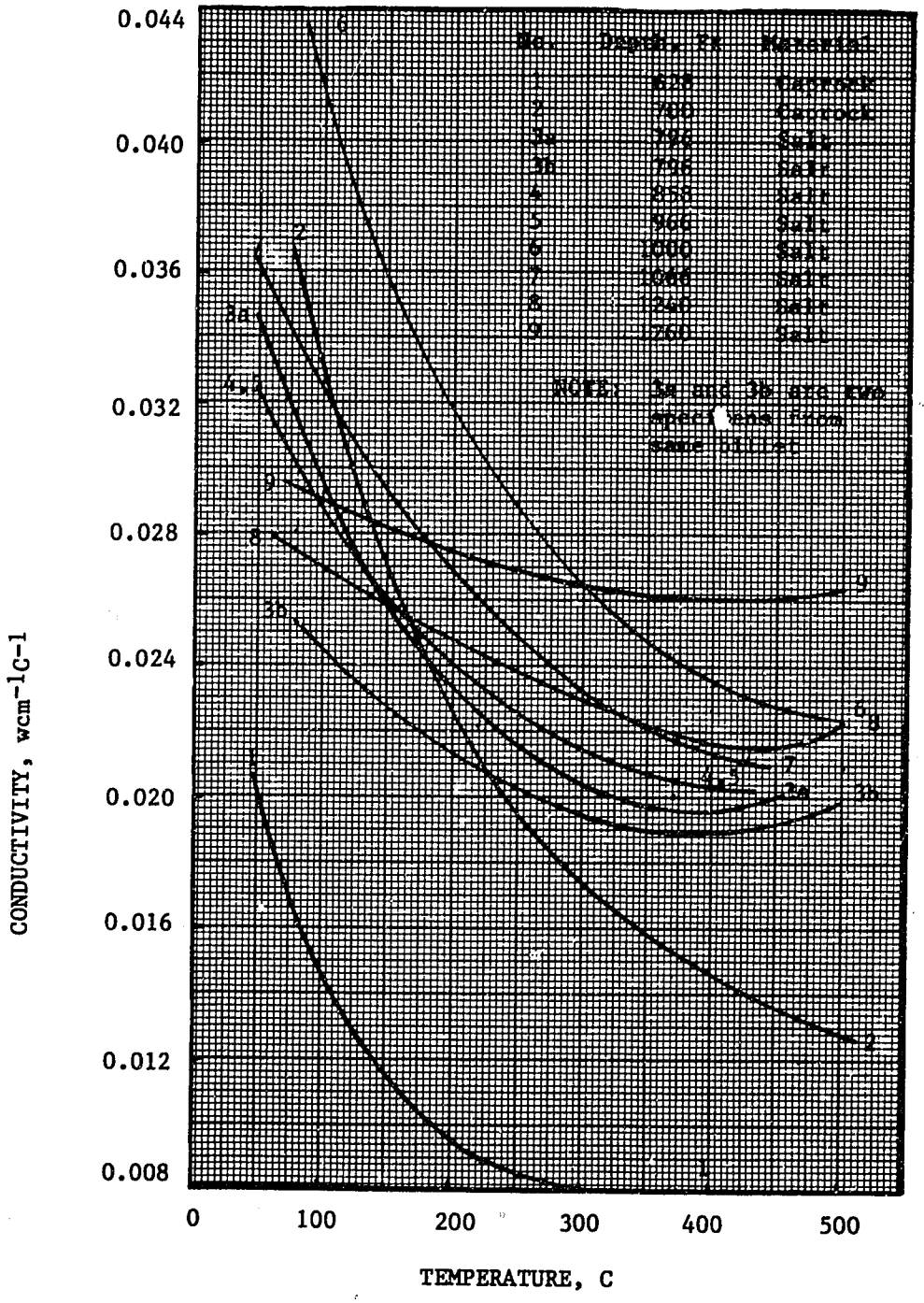


FIGURE 13. Axial Thermal Conductivity of Specimens from Richton Salt Dome, Mississippi

Table 21 lists enthalpy data for each material and the derived average specific heat for each as well as an average specific heat value for all of the salt specimens. The caprock specimens were treated separately.

Table 22 lists room temperature densities for all Richton specimens.

2.6 GIBSON DOME, UTAH

There are four predominantly salt specimens in this group of twelve materials. Each contains some anhydrite. Two rock specimens contained minor amounts of halite and the remaining six materials contained no salt.

Figure 14 presents linear thermal expansion data for the Gibson Dome materials.

Table 23 lists thermal conductivity data and Figure 15 the family of conductivity curves for this group.

Table 24 lists enthalpy data and derived specific heat values for each material and a combined specific heat value representing the four salt specimens.

Table 25 lists room temperature densities for all materials.

2.7 POMONA MEMBER BASALT

There are eleven basalt specimens in this group, but no salt specimens.

TABLE 21. Enthalpy and Specific Heat Data for Specimens from Richton Salt Dome, MRIG-9, Mississippi

Specimen Identification Location	Depth, Ft.	Temperature C	Enthalpy cal g^{-1}	Specific Heat cal $g^{-1}C^{-1}$
7-4	628.6 - 629.7	0	0	0.208
Caprock		23	4.208	
		59	10.728	
		96	17.709	
		143	29.409	
		274	54.147	
		340	71.530	
	A = -1.0021 B = 0.2083		Corr. Coeff. = 0.9988	
-	699.4 - 700.4	0	0	0.223
Caprock		44	7.186	
		167	31.338	
		278	57.022	
		416	90.188	
		499	110.571	
	A = -2.8200 B = 0.2231		Corr. Coeff. = 0.9987	
11-20	796.0 - 797.3	0	0	0.218
Salt		24	5.135	
		52	10.817	
		94	19.673	
		150	31.664	
		245	52.490	
		365	79.723	
	A = -0.4612 B = 0.2180		Corr. Coeff. = 0.9999	

Table 21, cont'd

12-13	858.0 - 859.0	0	0	0.218
Salt		24	5.030	
		57	11.900	
		92	19.204	
		150	31.769	
		243	51.777	
		362	79.065	
	A = -0.4850	B = 0.2178	Corr. Coeff. = 0.9998	
15-21	966.0 - 967.0	0	0	0.219
Salt		24	4.902	
		54	11.357	
		94	19.632	
		150	31.637	
		244	52.238	
		363	79.525	
	A = -0.5485	B = 0.2187	Corr. Coeff. = 0.9998	
-	999.4 - 1000.4	0	0	0.223
Salt		44	8.864	
		167	34.485	
		276	59.366	
		417	92.274	
		501	110.921	
	A = -1.1518	B = 0.2226	Corr. Coeff. = 0.9997	
16-3	1066.3 - 1067.3	0	0	0.218
Salt		24	5.056	
		50	10.453	
		91	19.004	
		149	31.575	
		245	52.440	
		363	79.266	
	A = -0.4503	B = 0.2179	Corr. Coeff. = 0.9999	

Table 21, cont'd

-	1240.0 - 1241.2	0	0	0.222
Salt		44	8.969	
		168	34.640	
		275	59.084	
		415	91.206	
		501	110.890	
	A = -1.1006	B = 0.2219	Corr. Coeff. = 0.9997	
20-9	1250.2 - 1251.2	0	0	0.218
Salt		24	5.072	
		49	10.179	
		90	18.863	
		149	31.533	
		244	52.360	
		362	78.938	
	A = -0.4337	B = 0.2178	Corr. Coeff. = 0.9999	
-	1258.8 - 1260.1	0	0	0.221
Salt		44	8.726	
		168	34.388	
		274	58.672	
		414	90.658	
		501	110.486	
	A = -1.2067	B 0.2214	Corr. Coeff. = 0.9997	

Combined Enthalpy Data for all Richton MRIG-9 Salt Specimens

A = -0.7516 Specific Heat = 0.220 cal $g^{-1}C^{-1}$

B = 0.2202 Correlation Coefficient = 0.9998

TABLE 22. Room Temperature Density Data for Specimens from Richton MRIG-9, Mississippi

Specimen Identification		Density	
Location	Depth, Ft	g cm^{-3}	Material
7-4	628-6 - 629.7	2.64	Caprock
-	700	2.84	Caprock
11-20	796	2.17	Salt
12-13	858	2.20	Salt
15-21	966	2.21	Salt
-	1000	2.23	Salt
16-3	1066.3	2.22	Salt
-	1240	2.26	Salt
20-9	1250.2	2.17	Salt
-	1260	2.22	Salt

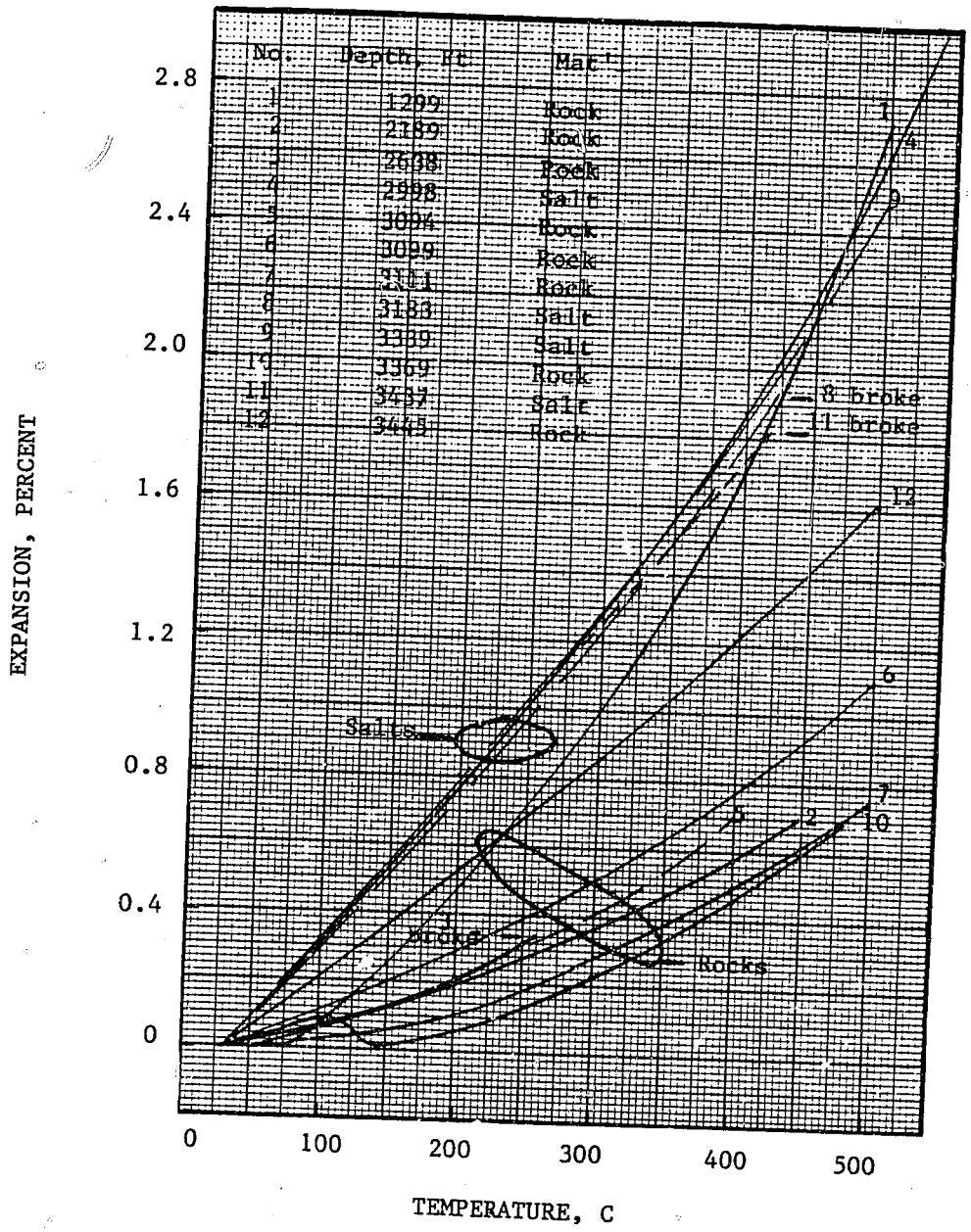


FIGURE 14. Axial Thermal Expansion of Specimens from Gibson Salt Dome, Utah

TABLE 23. Thermal Conductivity Data for Specimens from Gibson Salt Dome, Utah

Specimen Identification Location	Depth, Ft	Temperature C	Conductivity W cm-1C-1
GD-1-43	1299.2-1300.2*	21	0.0450
Rock		53	0.0433
*Attempts to fabricate this conductivity specimen were not successful. Data derived as product of diffusivity, specific heat and density.		99	0.0362
		209	0.0279
		303	0.0230
		408	0.0195
		505	0.0175
GD-1-44	2189.1-2190.1	80	0.0337
		135	0.0230
Rock		186	0.0201
		273	0.0187
		346	0.0175
		391	0.0168
		447	0.0162
		511	0.0157
GD-1-45	2638.9-2639.9	79	0.0169
Rock		133	0.0161
		195	0.0153
		264	0.0120
		328	0.0117
		383	0.0116
		441	0.0110
		509	0.0105
GD-1-46	2998.1-2991.1	78	0.0378
		132	0.0334
Salt		197	0.0277
		275	0.0225
		330	0.0213
		385	0.0201
		442	0.0182
		509	0.0172

TABLE 23. cont'd

GD-1-47	3094.4-3095.4	81	0.0280
		132	0.0254
Rock		196	0.0216
		270	0.0172
		330	0.0162
		386	0.0153
		443	0.0144
		509	0.0136
GD-1-48	3099.9-3100.9	79	0.0435
		134	0.0372
Rock		199	0.0287
		269	0.0229
		331	0.0206
		387	0.0187
		444	0.0169
		512	0.0162
GD-1-49	3111.2-3112.2	77	0.0201
		132	0.0181
Rock		193	0.0145
		266	0.0124
		320	0.0119
		326	0.0122
		385	0.0119
		441	0.0118
		505	0.0109
GD-1-50	3183.5-3184.5	80	0.0327
		143	0.0298
Salt		224	0.0263
		301	0.0218
		378	0.0192
		443	0.0177
		508	0.0169

TABLE 23. cont'd

GD-1-51	3339.1-3340.1	79	0.0387
		142	0.0333
Salt		232	0.0280
		303	0.0254
		405	0.0216
		498	0.0195
GD-1-52	3369.2-3370.2	75	0.0178
		135	0.0138
Rock		216	0.0115
		296	0.0107
		370	0.0103
		434	0.0097
		498	0.0087
GD-1-53	3437.6-3438.6	82	0.0358
		146	0.0302
Salt		225	0.0254
		300	0.0223
		372	0.0198
		436	0.0174
		504	0.0166
GD-1-54	3445.6-3446.6	80	0.0332
		144	0.0284
Rock		224	0.0230
		298	0.0199
		373	0.0170
		439	0.0152
		504	0.0136

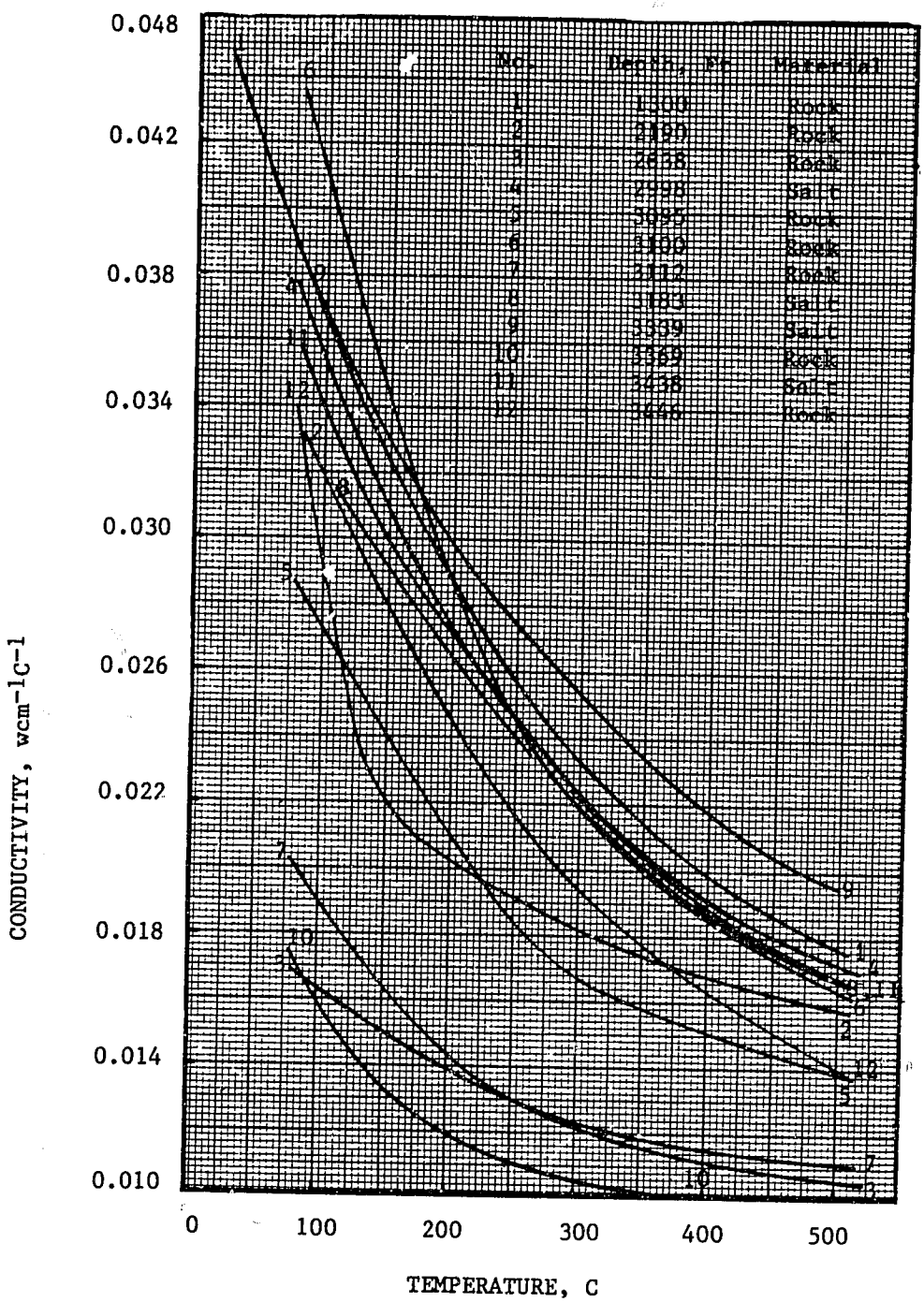


FIGURE 15. Axial Thermal Conductivity of Specimens from Gibson Salt Dome, Utah

TABLE 24. Enthalpy and Specific Heat Data for Specimens from Gibson Salt Dome, Utah

Specimen Location	Identification Depth, Ft	Temperature C	Enthalpy cal g^{-1}	Specific Heat cal $g^{-1}C^{-1}$
GD-1-43	1299.2 - 1300.2	0	0	0.245
Rock		44	8.163	
		168	34.634	
		272	60.894	
		411	97.848	
		501	121.995	
	A = -3.0586 B = 0.2449		Corr. Coeff = 0.9986	
GD-1-44	2189.1 - 2190.1	0	0	0.244
Rock		44	8.345	
		168	34.559	
		271	60.272	
		409	96.914	
		501	121.772	
	A = -3.0037 B = 0.2440		Corr. Coeff. 0.9985	
GD-1-45	2638.9 - 2639.9	0	0	0.243
Rock		51	10.523	
		151	32.647	
		241	53.381	
		325	75.528	
		471	114.624	
	A = -2.3601 B = 0.2428		Corr. Coeff = 0.9987	
GD-1-46	2998.1 - 2999.1	0	0	0.221
Salt		50	11.065	
		151	32.408	
		249	53.522	
		325	70.515	
		469	104.259	
	A = -0.5186 B = 0.2210		Corr. Coeff. 0.9998	

Table 24. cont'd

GD-1-47	3094.4 - 3095.4	0	0	0.242
Rock		49	10.525	
		150	33.129	
		254	57.793	
		325	75.920	
		467	113.535	
	A = -1.7811	B = 0.2422	Corr. Coeff. = 0.9992	
GD-1-48	3099.9 - 3100.9	0	0	0.224
Rock		49	9.152	
		150	29.493	
		256	51.723	
		325	69.571	
		466	104.569	
	A = -2.3298	B = 0.2235	Corr. Coeff. = 0.9983	
GD-1-49	3111.2 - 3112.2	0	0	0.241
Rock		49	10.339	
		150	32.059	
		254	55.709	
		326	75.840	
		469	113.463	
	A = -2.2388	B = 0.2411	Corr. Coeff. = 0.9985	
GD-1-50	3183.5 - 3184.5	0	0	0.222
		53	10.582	
Salt		143	30.027	
		278	59.509	
		365	79.921	
		470	104.309	
	A = -1.0701	B = 0.2221	Corr. Coeff. = 0.9998	

Table 24 cont'd

GD-1-51	3339.1 - 3340.1	0	0	0.223
Salt		53	10.563	
		142	29.631	
		278	59.568	
		365	79.195	
		470	105.387	
	A = -1.2727	B = 0.2232	Corr. Coeff. = 0.9995	
GD-1-52	3369.2 - 3370.2	0	0	0.237
Rock		53	11.066	
		141	31.141	
		275	61.708	
		375	86.832	
		470	111.380	
	A = -1.4343	B = 0.2365	Corr. Coeff. 0.9995	
GD-1-53	3437.6 - 3438.6	0	0	0.221
Salt		53	11.364	
		142	29.132	
		273	58.046	
		371	80.830	
		470	104.441	
	A = -1.0022	B = 0.2214	Corr. Coeff. = 0.9996	
GD-1-54	3445.6 - 3446.6	0	0	0.220
Rock		54	9.891	
		142	27.667	
		272	56.768	
		369	78.164	
		470	103.486	
	A = -1.9224	B = 0.2200	Corr. Coeff = 0.9992	

Combined Enthalpy Data for All Gibson Dome Salt Specimens

A = -0.9625 Specific Heat = 0.222 cal $g^{-1}C^{-1}$

B = 0.2219 Correlation Coefficient = 0.9996

TABLE 25. Room Temperature Density Data for Specimens from Gibson Salt Dome, Utah

Specimen Identification		Density	
Location	Depth, Ft	g cm^{-3}	Material
GD-1-43	1299.2 - 1300.2	2.67	Rock
GD-1-44	2189.1 - 2190.1	2.67	Rock
GD-1-45	2638.9 - 2639.9	2.60	Rock
GD-1-46	2998.1 - 2999.1	2.18	Salt
GD-1-47	3094.4 - 3095.4	2.43	Rock
GD-1-48	3099.9 - 3100.9	2.81	Rock
GD-1-49	3111.2 - 3112.2	2.43	Rock
GD-1-50	3183.5 - 3184.5	2.17	Salt
GD-1-51	3339.1 - 3340.1	2.21	Salt
GD-1-52	3369.2 - 3370.2	2.29	Rock
GD-1-53	3437.6 - 3438.6	2.25	Salt
GD-1-54	3445.6 - 3446.6	2.76	Rock

Figure 16 gives linear thermal expansion curves for all eleven basalts.

Table 26 lists thermal conductivity data and Figure 17 presents the very narrow range of conductivity values by showing upper and lower limits. It is not possible to show a curve for each material distinct from the others in the narrow band shown.

Table 27 lists the enthalpy data and the derived specific heat for each basalt specimen, and also an average specific heat value representative of the group of eleven materials.

Table 28 lists the room temperature density data for each basalt specimen.

3 DISCUSSION OF DATA

3.1 ACCURACY OF THERMAL CONDUCTIVITY DATA

Of the properties evaluated in this study, thermal conductivity is particularly important in assessing the suitability of the various sites for storage. Because of the variability of data on salts, as reported by numerous investigators, several extra-precautionary steps were taken to insure sufficient accuracy in our measurements of this property. These included the following:

1. Modification/Variation of Measurement Procedure
2. Verification of Conductivity Values Used for the Reference Standard

Descriptions and comments on each are presented.

3.1.1 Procedure Modification

The procedure specified for measurement of thermal conductivity in this program is a steady-state comparative approach, using Pyroceram 9606 as the reference standard. In the version used in the early part of this program, and described in Appendix C, only one heat-flow meter with passive guarding was used. The meter was positioned between the specimen and the heat sink. In a modified version, a second heat-flow meter was added, resulting in the opportunity to place one meter on each side of the specimen. This modified arrangement precluded the possibility to achieve passive guarding against radial losses, so active guarding was added.

Following this modification, several salt specimens which had been evaluated earlier in the program, were again measured. One of these was from the Vacherie Salt Dome. Table 8 lists data for

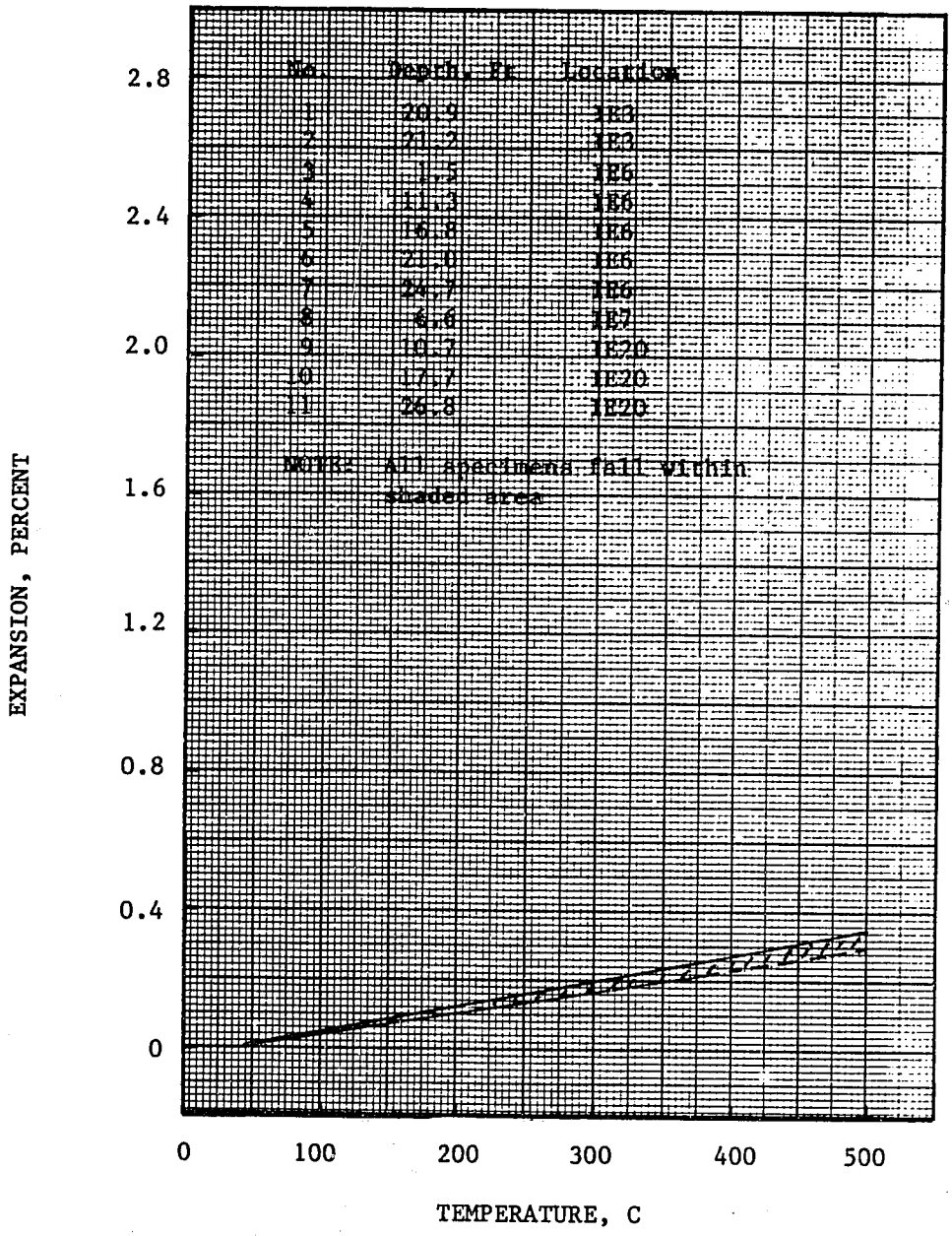


FIGURE 16. Axial Thermal Expansion of Specimens from Pomona Member Basalt

TABLE 26. Thermal Diffusivity and Conductivity Data for Specimens from Pomona Member Basalt, Washington

Specimen Location	Identification Depth, Ft.	Temperature C	Diffusivity* $cm^2\ s^{-1}$	Conductivity** $w\ cm^{-1}C^{-1}$
IE3	20.9-21.2	22	0.0074	0.0207
		52	0.0075	0.0209
		100	0.0074	0.0207
		202	0.0067	0.0187
		308	0.0064	0.0179
		414	0.0060	0.0168
		503	0.0058	0.0162
IE3	21.2-21.5	24	0.0071	0.0198
		50	0.0072	0.0201
		103	0.0069	0.0193
		207	0.0066	0.0184
		307	0.0061	0.0170
		405	0.0059	0.0165
		498	0.0055	0.0154
IE6	1.5-1.9	21	0.0070	0.0193
		54	0.0071	0.0196
		105	0.0071	0.0196
		201	0.0063	0.0174
		302	0.0061	0.0169
		406	0.0060	0.0166
		502	0.0056	0.0155
IE6	11.3-11.6	21	0.0076	0.0212
		49	0.0077	0.0215
		103	0.0075	0.0210
		203	0.0071	0.0199
		301	0.0066	0.0185
		404	0.0063	0.0176
		506	0.0061	0.0171

Table 26, cont'd

IE6	16.8-17.0	21	0.0075	0.0211
		51	0.0074	0.0208
		105	0.0072	0.0202
		207	0.0067	0.0188
		304	0.0062	0.0174
		405	0.0061	0.0171
		509	0.0058	0.0163
IE6	21.0-21.5	22	0.0078	0.0218
		50	0.0075	0.0210
		106	0.0074	0.0207
		208	0.0067	0.0188
		304	0.0067	0.0188
		403	0.0064	0.0179
		500	0.0063	0.0176
IE6	24.7-25.0	21	0.0077	0.0216
		52	0.0077	0.0215
		104	0.0074	0.0207
		203	0.0069	0.0193
		302	0.0067	0.0188
		405	0.0062	0.0174
		503	0.0060	0.0168
IE7	6.6-7.0	22	0.0078	0.0215
		53	0.0076	0.0210
		107	0.0073	0.0202
		212	0.0066	0.0182
		304	0.0067	0.0185
		408	0.0061	0.0169
		501	0.0060	0.0166

Table 26, cont'd

IE20	10.7-11.1	22	0.0076	0.0211
		54	0.0075	0.0208
		100	0.0073	0.0203
		199	0.0068	0.0189
		302	0.0066	0.0183
		402	0.0061	0.0170
		506	0.0059	0.0164
IE20	17.7-18.2	22	0.0078	0.0215
		52	0.0076	0.0210
		105	0.0073	0.0201
		202	0.0070	0.0193
		303	0.0067	0.0185
		402	0.0062	0.0171
		503	0.0061	0.0168
IE20	26.8-27.0	22	0.0077	0.0215
		51	0.0074	0.0206
		104	0.0073	0.0204
		204	0.0068	0.0190
		306	0.0063	0.0176
		403	0.0060	0.0167
		501	0.0058	0.0162

*measured

**calculated from diffusivity, specific heat and density

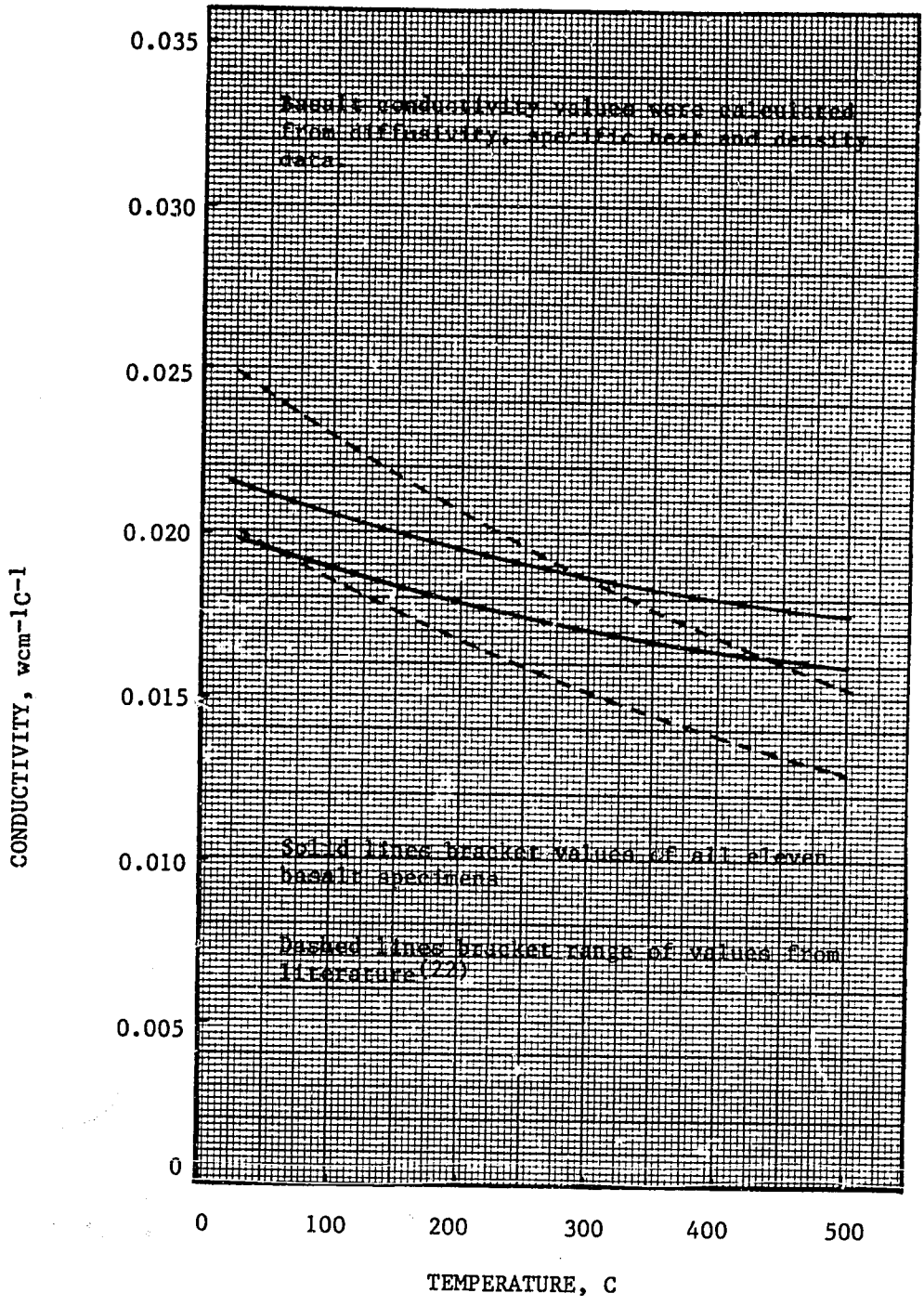


FIGURE 17. Axial Thermal Conductivity of Specimens from Pomona Member Basalt, Washington

TABLE 27. Enthalpy and Specific Heat Data for Specimens from Pomona Basalt, Washington

Specimen Location	Identification Depth, Ft	Temperature C	Enthalpy cal g^{-1}	Specific Heat, cal $g^{-1}C^{-1}$
IE3	20.9 - 21.2	0	0	0.235
		53	9.913	
		132	25.615	
		286	60.462	
		546	127.589	
	A = -3.045 B = 0.2348 Corr. Coeff = 0.9984			
IE3	21.2 - 21.5	0	0	0.234
		53	10.237	
		131	26.000	
		285	60.350	
		555	129.093	
	A = -2.6759 B = 0.2335 Corr. Coeff. = 0.9987			
IE6	1.5 - 1.9	0	0	0.234
		53	10.045	
		131	25.478	
		285	60.172	
		565	131.668	
	A = -2.9696 B = 0.2342 Corr. Coeff. = 0.9986			
IE6	11.3 - 11.6	0	0	0.235
		53	9.626	
		130	25.354	
		285	60.552	
		565	131.585	
	A = -3.0302 B = 0.2345 Corr. Coeff. 0.9987			
IE6	16.8 - 17.0	0	0	0.235
		53	9.897	
		130	25.676	
		285	60.105	
		568	132.779	
	A = -2.9523 B = 0.2349 Corr. Coeff. = 0.9986			

Table 27, cont'd

IE6	21.0 - 21.5	0	0	0.233
		94	17.456	
		194	39.546	
		360	78.535	
		564	130.506	
	A = -3.2870	B = 0.2331	Corr. Coeff = 0.9987	
IE6	24.7 - 25.0	0	0	0.232
		94	17.452	
		191	38.827	
		363	78.535	
		556	127.979	
	A = -3.1832	B = 0.2315	Corr. Coeff. = 0.9987	
IE7	6.6 - 7.0	0	0	0.232
		94	17.476	
		190	38.195	
		364	79.575	
		541	124.362	
	A = -3.1726	B = 0.2317	Corr. Coeff. = 0.9987	
IE20	10.7 - 11.1	0	0	0.232
		94	17.496	
		187	38.034	
		363	79.876	
		537	123.689	
	A = -3.0520	B = 0.2323	Corr. Coeff. = 0.9989	
IE20	17.7 - 18.2	0	0	0.231
		94	17.437	
		185	37.586	
		363	79.603	
		535	122.782	
	A = -2.9999	B = 0.2314	Corr. Coeff. = 0.9989	

Table 27, cont'd

IE20	26.8 - 27.0	0	0	0.232
		93	17.412	
		185	37.325	
		362	79.635	
		534	122.526	
	A = -2.9767	B = 0.2315	Corr. Coeff. = 0.9989	

Combined enthalpy data for all specimens

A = -3.0319 Specific Heat = 0.233 cal $g^{-1}C^{-1}$

B = 0.2329 Correlation Coefficient = 0.9987

TABLE 28. Room Temperature Density Data for Specimen from Pomona Member Basalt, Washington

Specimen Identification		Density	
Location	Depth, Ft	g cm^{-3}	Material
IE3	20.9 - 21.2	2.85	All basalt rocks
IE3	21.2 - 21.5	2.87	
IE6	1.5 - 1.9	2.83	
IE6	11.3 - 11.6	2.86	
IE6	16.8 - 17.0	2.87	
IE6	21.0 - 21.5	2.88	
IE6	24.7 - 25.0	2.90	
IE7	6.6 - 7.0	2.86	
IE20	10.7 - 11.1	2.87	
IE20	17.7 - 18.2	2.86	
IE20	26.8 - 27.0	2.89	

two runs of Specimen S-15-3 (3045 Ft depth), one with the apparatus before modification, the other, after. As indicated in Figure 5, results from both runs are similar enough to be represented by one curve (Curve No. 5, 7).

Another specimen, from the Salt Valley Dome, was also utilized for repeat measurements in the modified set-up. Smoothed curves through plots of the Table 17 data for Specimen 3-77 (1952 ft. depth) again illustrate similarity of results from the two runs. (See curves 2 and 5 of Figure 11)

This brief study confirms that confidence in conductivity data generated early in the program is justified. Although the two measurement methods are similar in principle, they are dissimilar enough in application to have exposed potential systematic errors by one or the other. Yet, results are similar enough to enhance confidence in all data presented in this report.

3.1.2 Conductivity of Reference Standard Pyroceram 9606

The accuracy of conductivity data generated by any comparative method is dependent on how well the conductivity of the reference standard is known. For these studies, Pyroceram 9606* was selected as the standard for several reasons. It's conductivity is close to that of the program materials, it is stable in the temperature range of interest (RT to 500C), it has been evaluated in this range by Rudkin[1] and Flynn[2] and a table of recommended values has been published in the TPRC Data Series[3]

About midway through this program, attention was drawn to the fact that the values being used for the conductivity of Pyroceram 9606 were lower than those being used by Morgan in referee measurements at the Oak Ridge National Laboratory. With Morgan's cooperation in supplying some of his reference material, an extensive study was

*Brand name of glass ceramic manufactured by Corning Glass Works, Corning, NY

carried out to investigate this apparent discrepancy. A complete description of this work, and results, are presented in a Lagedrost-to-Morgan letter dated February 20, 1981.(A copy of this document is included as Appendix F to this report.) The net result is that considerable confidence was established in the values for Pyroceram 9606 utilized in the present study, and therefore, in the conductivity values being reported for the program materials.

Final verification of accuracy of the measurement technique was established through its use to measure conductivity values of another potential reference standard, clear, optical quality, fused silica. Considerable data have been reported on fused silica; the TPRC Data Series[4] presents a recommended curve of conductivity versus temperature (Appendix G). The cited literature shows a nearly linear relationship in the range RT-350C; the measurements of this investigation indicate a similar relationship. An absolute comparison was achieved by linear regression analysis. The maximum difference between the literature and the present measurements was 7.8 percent at the highest measured temperature (approximately 300C) and less than 1.0 percent at 100C and below. By extrapolation of the curve to 500C, the difference is less than 4 percent.

This comparison adds to the evidence that conductivity values on the geologic materials, as measured in this program, are well within the accuracy objective of ±15 percent. Further, they are considered to be adequate to make valid comparisons among materials and sites, and with results from similar studies by other workers.

3.2 COMPARISON OF DATA ON PROGRAM MATERIALS

In this section, comments on comparative performance of the various program materials for the measured properties are presented.

3.2.1 Thermal Expansion

3.2.1.1 Salts

A general characteristic of the relationship between linear thermal expansion and temperature of rock salt, as well as of most rocks, is a gradually increasing expansion rate (slope) with increasing temperature. Figures 4, 6, 8, 10, 12 and 14 show the linear expansion curves of all salt specimens which were measured from the six salt deposits in this study. Each of the figures shows this general relationship.

The actual salts measured varied in color, crystal size and degree of adherence of one crystal to another. It is assumed from this and from the variation in other properties that these salts vary in purity and are not necessarily composed only of halite. One should expect some variation in expansion of the different specimens if this is true. In each figure, it can be seen that the salts from the different depths vary in expansion rates.

An interesting and significant fact is that one salt from each source has the same expansion-temperature relationship as the consensus or recommended relationship as reported in NBS Monograph 167 on salt properties(5). Values ranged from 2.2 to 2.8% at 500C.

There is no consistent trend among the several salt sources of relative expansion rate versus depth from which the specimens were taken

3.2.1.2 Basalts

Figure 16 shows the expansion as a function of temperature of eleven basalt specimens. All of the curves fall together such that the narrow wedge depicted represents all of the materials measured. This expansion-temperature relationship is approximately linear from room temperature to 500C, and closely coincides with the Cindas curve for Tholeiitic Basalt(6). Values at 500C were from 0.6 to 1.6%.

3.2.2 Specific Heat

3.2.2.1 Salts

Specific heat values were determined from the relationships between the enthalpy and temperature values as measured in the Bunsen Ice Calorimeter. The enthalpy can be described with a linear function over short temperature intervals. However, over a wide interval, the linearity does not persist. For use in this study, the interval from room temperature to 350C in the earlier measurements and room temperature to 500C in later measurements was considered to lie within the linear region of the test materials. On this assumption, a linear regression analysis was performed to determine the slope of the curve of enthalpy versus temperature. This was assumed to be equal to the specific heat for each specimen over the range of measurements. The linear equation is given in Table 9. The constants A and B (y-intercept and slope) are given for each specimen in Tables 9, 12, 15, 18, 21, 24 and 27 along with the enthalpy-temperature data.

Assuming linearity based on visual plots of the enthalpy data, the derived specific heat then is the average specific heat over the temperature range of the enthalpy data.

The values of specific heat for the salt specimens range from 0.213 to 0.223 cal $g^{-1}C^{-1}$. The average value for all of the salts from each source is listed at the end of the table for that source.

The specific heat for rock salt (pure NaCl) reported in the Cindas compilation[7] ranges from 0.205 to 0.239 cal $g^{-1}C^{-1}$ over the range from room temperature to 500C. The average is 0.222 cal $g^{-1}C^{-1}$.

Only one group, that from Gibson Dome, has an average value equal to the Cindas reference. The average Richton value (for salts only) is 0.220, Salt Valley 0.217, Palo Duro 0.215, Cypress Creek 0.215, and Vacherie 0.217. This is a variation among sources of 3%.

3.2.2.2 Basalts

Table 27 lists the enthalpies and specific heats for the eleven basalt specimens from the Pomona Member Basalt, Saddle Mountain Formation, Washington. The specific heat average for the eleven specimens is 0.233 cal $g^{-1}C^{-1}$ over the range room temperature to 500C.

A graph representing specific heat values for basalts is found in Cindas compilation(8). Here, the average value over this same temperature range is 0.239 cal $g^{-1}C^{-1}$. There is a difference of less than 3% between these averages.

3.2.3 Thermal Conductivity

The conductivity of forty-three of the fifty-nine materials studied was measured in the steady-state comparative apparatus. Due to the friable nature of the salt specimens, and of some of the rock specimens, it was not possible to machine satisfactory specimens from six materials. The basalt samples were received as 1-3/4 inch drill cores and were not large enough for steady-state measurements. Instead, they were measured in the pulsed-laser, diffusivity apparatus. One rock material from the Gibson Dome was also measured in this apparatus.

The detailed conductivity-temperature data for all of the materials except the basalts appear in Tables 8, 11, 14, 17, 20 and 23.

The conductivity-temperature graphical representation appears in Figures 5, 7, 9, 11, 13, and 15 for the six groups of materials from salt domes.

3.2.3.1 Salts

Examination of Figures 5 through 15 shows that the conductivity values of the salts cover a wide range. At 50C, they vary from 0.022 to 0.046 $wcm^{-1}C^{-1}$, and at 500C, they vary from 0.012 to 0.027 $wcm^{-1}C^{-1}$

A noticeable feature of these curves is that salts from some sources show a minimum conductivity below 500C, whereas others do not. These minima occur between 300 and 400C.

A comparison of the data from the several sources shows that no source would appear to be advantageous over the others from a thermal conductivity point of view.

Figure 18 shows conductivity vs. temperature curves for other rock salts. The data are from listed published sources and provide a basis for comparison of original data of this report. The information presented in this figure shows a very wide range of conductivity values for any given temperature. This can be explained in part by the following discussion.

The measurement techniques vary considerably. Steady-state comparison using Pyroceram meters was used by several of the referenced authors, as well as by the authors of this report.

Some of the authors used an infinite-line source, steady-state technique, and some used a transient heat-flow diffusivity technique. Some of the measurements were on pure sodium chloride in single crystal form. Others were made on polycrystalline rock salt of varying degrees of purity, crystal size and inter-crystal bonding strengths. Some materials were tightly bonded and machined easily,

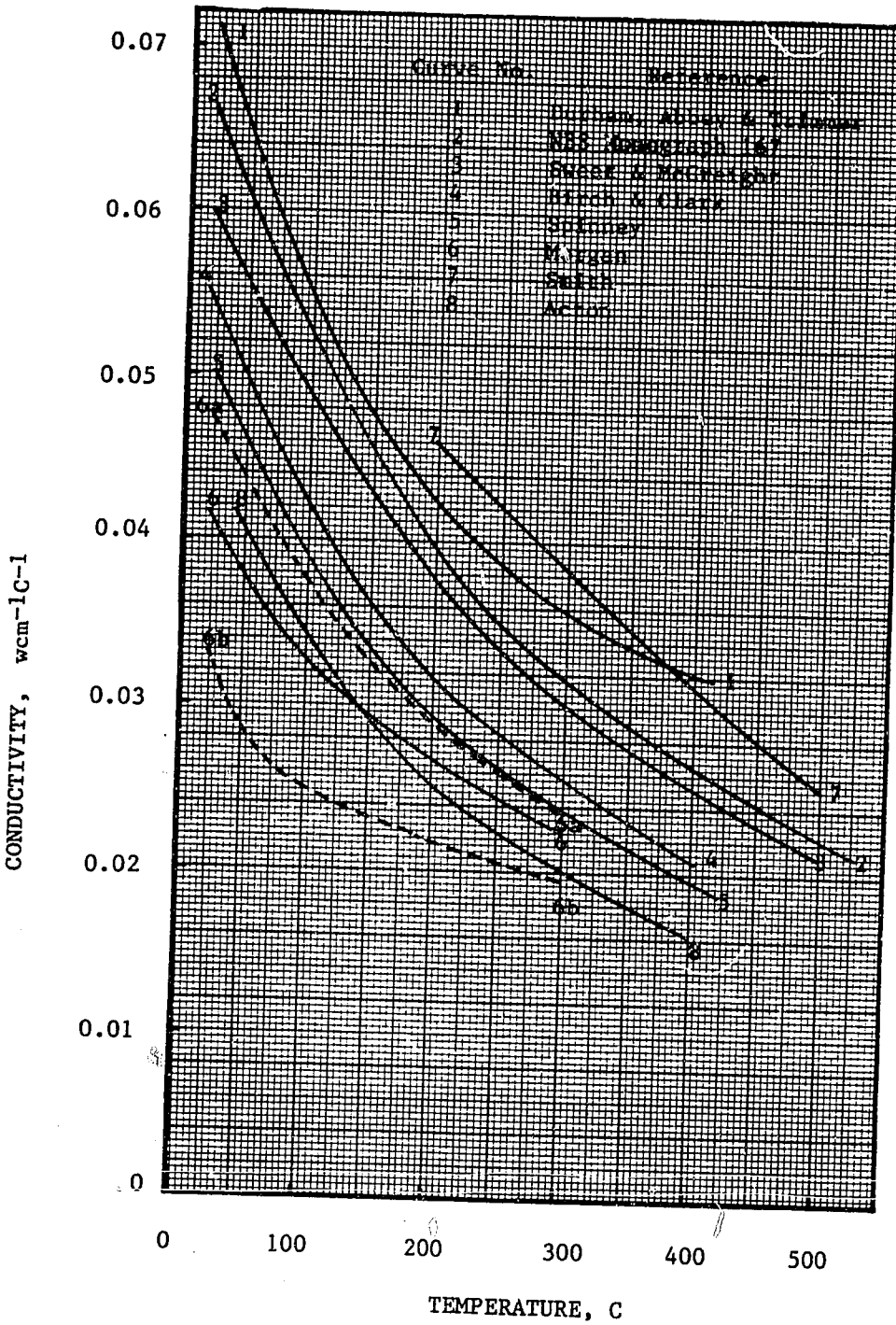


FIGURE 18. Thermal Conductivity of Crystalline Rock Salt. Data taken from Literature

whereas some were so friable that it was difficult or impossible to obtain satisfactory test pieces. All of these factors affect the conductivity values. The higher the purity and the higher the bonding strength or the absence of boundaries (single crystals), the greater the expected conductivity.

Figure 18 (Curve No. 2) is the same for the NBS recommended curve[9] as for the Yang curve[10] This was obtained on high-purity, single crystal NaCl under pressure. This is an idealized situation. The rock salts from the salt dome drill cores are impure, varying greatly in color, crystal size, amount of included material and amount of associated materials at grain boundaries.

The data of Morgan[11], shown in Figure 18 (Curve No. 6), most closely match the data of this report. The similarity would be immediately apparent if Figure 18 were superimposed on each of the figures representing conductivity of salts from the six sources of this study (Figures 5, 7, 9, 11, 13 and 15). The dotted curves in Figure 18 (Nos. 6a and 6b) represent the upper and lower limits of values for all of Morgan's specimens, and the solid curve, No. 6, represents his average. With few exceptions, all of the salt data of the present study fall within these boundaries.

Morgan used the steady-state technique employing Pyroceram 9606 heat-flow meters, and two-inch diameter specimens from poly-crystalline core drill samples. In other words, he followed the same method as used in the work presented in this report. Spinney[12] used a similar technique and similar materials. His values are close to Morgan's, but above them. The highest values obtained at moderate temperatures were by Smith[13] (Curve 7 in Figure 18).

He used the diffusivity technique on single crystal material. Acton's data[14] had the lowest value at 500C. He also used the diffusivity method. However, he studied polycrystalline material. Birch and Clark[15] also studied polycrystalline materials. Durham, Abey and Trimmer[16] used polycrystalline materials from Avery Island salt dome with an infinite line source technique. Sweet and McCreight[17] used a steady-state linear heat flow method on polycrystalline materials.

Because of the extreme variability among the salt materials studied by the various authors, it is not possible to generalize about the relative merits of the different methods used. However, because the type of materials (core drill specimens from salt domes), as well as the measurement methods used by Morgan, were the same as for this work, it is gratifying to note the agreement for a relatively large group of materials.

3.2.3.2 Basalts

Figure 17 shows the conductivity-temperature relationships of the basalts. The values range from 0.020 to 0.022 $wcm^{-1}C^{-1}$ at 100C, and from 0.016 to 0.018 $wcm^{-1}C^{-1}$ at 500C. The conductivities were calculated from the measured values of diffusivity, density and specific heat. The figure also shows the range of values cited in the Cindas volume[18]. The slope of the "measured" generalized Basalt curve is less than the slope of the generalized referenced curve over the same temperature range. There is considerable overlap of the two curves indicating that the values obtained by the diffusivity techniques are compatible with the values in the reference.

2 OF 2

3.2.4 Density

3.2.4.1 Salts

The Handbook of Chemistry and Physics (Chem. Rubber Co.)[19] lists sodium chloride as having a density of 2.165 g cm^{-3}. The NBS Monograph on Physical Properties Data for Rock Salt [20] lists the density of halite as 2.163 gcm^{-3} at 20C. The density of rock salt depends considerably on its purity. Anhydrite, the main impurity associated with rock salt, has a density of 2.96 g cm^{-3}, and if present, would increase the density of the material.

The densities of the salt materials measured for this report vary from 2.14 to 2.29 g cm^{-3}. The lowest density materials coming from Vacherie, Cypress Creek and Palo Duro materials, and the highest density coming from the Gibson Dome.

3.2.4.2 Basalt

The Cindas compilation [21] gives a range of densities for basalts from 2.20 to 2.85 g cm^{-3}, with a mean of 2.59.

The materials from the Pomona Member Basalts exceeded this range and measured from 2.83 to 2.90 g cm^{-3}.

Basalts are a rather broad class of rocks that are volcanic in origin and consist of varying proportions of feldspars, olivine, hornblend, biotite and other minerals, and do not have a closely defined composition. Physical properties vary accordingly.

4 CONCLUSIONS AND RECOMMENDATIONS

Of the several thermal properties dealt with in this study, thermal conductivity is considered to be the most important in attempting to evaluate prospective sites for nuclear waste isolation.

A study of the literature reveals a bewildering variation in cited conductivity values for rock salt, the principal material of this report. Several reasons for this variation exist. Halite, the mineral name for rock salt, shows a large temperature dependence of conductivity, especially at ambient temperatures. Although there is an abundance of literature on the subject, the direct comparison of data from different sources is difficult due to the varying mineral content associated with the rock salt, varying crystal development from granular, loosely held crystals, to single crystals several centimeters across, varying methods of measurement, and varying temperature ranges investigated. There is more than a two-fold difference between values reported. However, when results are compared between workers using the same measuring technique on polycrystalline salt specimens, there is a remarkably good agreement.

The present study is shown to be reliable; the accuracy of the methods used is well documented. It remains, however, to be determined whether laboratory conductivity measurements, or in-situ measurements, are of greater value to the application.

The laboratory measurements are under greater control and therefore, are better for comparisons of in-hand materials. The question remains open as to whether or not the in-hand materials are representative of the surroundings from which they came.

A second question is whether or not the core drilling process alters the material through mechanical abuse (loosening of crystalline bonding). The laboratory measurements on core-drill samples make it possible to sample great depths where in-situ measurements present considerable problems at great depths.

Steady-state results are probably more useful in heat flow modelling for waste isolation than transient methods, especially concerning heterogeneous materials.

The reader is cautioned against extrapolation of the conductivity data presented in this study down to room temperature or below, if extreme accuracy is desired. The large dependence of conductivity on temperature in this region makes extrapolation rather risky.

Specific heat, thermal expansion, and density values all fall well within the ranges cited in the literature for the rock salts. The methods are reliable and the variations with temperature are less than with conductivity.

Future work should include greater emphasis on careful conductivity evaluation of materials in or from sites being considered for waste isolation. Conductivity appears to be the most critical parameter of those studied with respect to site selection, and is therefore deserving of thorough understanding. Conductivity is the most variable of the properties reported here and most dependent on temperature, condition of specimens, and method used for measurement. Effective heat-dissipation modelling can only occur if conditions surrounding data are fully understood.

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21. See Reference 6, Table 3.2, p. 34

APPENDICES

A. Thermal Expansion Measurement Techniques

B. Specific Heat Measurement Technique

C. Thermal Conductivity Measurement Technique

D. Thermal Diffusivity Measurement Technique

E. Density Measurement Technique

F. Studies of Pyroceram 9606 Diffusivity and Conductivity

G. Comparison of Measured with "Recommended" Conductivity Data for Fused Silica

APPENDIX A

Thermal Expansion Measurement Method

Specifications

Test Procedure No. EMTL-TP-LE-101

THERMAL EXPANSION MEASUREMENT

BY

RECORDING QUARTZ DILATOMETER

Approved by:

Manager, EMTL

Manager, Thermal Properties Laboratory

Manager, QA/QC

October 25, 1979

THE ENERGY MATERIALS TESTING LABORATORY

A Division of

Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, Maine 04005

THERMAL EXPANSION MEASUREMENT

BY

RECORDING QUARTZ DILATOMETER

Foreword

This procedure is in essential conformance with ASTM Specification E-228, Standard Test Method for Linear Thermal Expansion of Rigid Solids with a Vitreous Silica Dilatometer. It includes a number of refinements to the basic specification to allow for protective atmospheres, when appropriate, and adaptation to sub-size or unique specimen requirements.

1.0 Background

When heat is added to or removed from a body, so that there is a change in its temperature, there is a corresponding change in its volume. Exceptions occur, however, in some specially prepared alloys or composite materials in which the various components have dissimilar or unique expansion characteristics. Frequently, thermal expansion is related to temperature through a coefficient such that as a body is heated from T_1 to T_2, its volume change, V_1 to V_2, is expressed as

$$\alpha = \frac{(V_2 - V_1)}{V_1 (T_2 - T_1)} \qquad (1)$$

More generally, the change in length or volume of a body as heat is added or removed is expressed as a percent expansion for a specific temperature change.

Many methods have been developed for measuring thermal expansion; variations exist to respond to the various requirements including level of accuracy, temperature range, compatibility problems, specimen availability, etc. The methods may be grouped as either relative, in which expansion of the material is investigated relative to the expansion of a reference standard, or absolute, in which expansion of the material is measured directly. The method described here is in the relative category. It is one of a group referred to as push-rod dilatometers. Variations of these are described in References 1-6.

2.0 Scope

2.1 This method covers the determination by a vitreous silica dilatometer of the linear thermal expansion, from -195 to +1000 C, for rigid solids including metals, ceramics and refractories, glasses, rocks and minerals, plastics, wood, and inorganic cements, pastes, and mortars.

2.2 For this purpose, a rigid solid is defined as a material which, at test temperatures and under the stresses imposed by specimen-supporting members in the thermal expansion apparatus, has a negligible creep or elastic strain rate, or both, insofar as they would significantly affect the precision of thermal length change measurements.

2.3 It is recognized that many rigid solids require detailed preconditioning and specific thermal test schedules for correct evaluation of linear thermal expansion behavior for certain materials applications. Since a general method of test cannot cover all specific requirements, details of this nature are discussed in particular material specifications.

3.0 Description of Terms

3.1 Linear Thermal Expansion is the change in length per unit length resulting from a change in temperature of the material. Symbolically represented by $\Delta L/L_0$, where ΔL is the observed change in length and L_0 is the length of the specimen at reference temperature T_0, linear thermal expansion has the units of inches per inch, or centimeters per centimeter, often expressed as percentage or parts per million.

3.2 Mean Coefficient of Linear Thermal Expansion, αm, between temperatures T_1 and T_2, is defined as:

$$\alpha m = (L_2 - L_1)/L_0(T_2 - T_1) = \Delta L/(L_0 \cdot \Delta T) \qquad (2)$$

where L_1 and L_2 = specimen lengths at temperatures T_1 and T_2, respectively. αm is therefore obtained by dividing the linear thermal expansion ($\Delta L/L_0$) by the change of temperature (ΔT). Units are inches per inch, or centimeters per centimeter per degree change in temperature, often expressed in parts per million per degree.

3.3 Instantaneous Coefficient of Linear Thermal Expansion, α_T, at temperature T, is defined by the following expression:

$$\alpha_T = T_1 - T_2(L_2 - L_1)/L_0(T_2 - T_1) = dL/(dT \cdot L_0) \quad (3)$$

α_T has the same units as α_m.

4.0 Measurement Apparatus and Method

4.1 The Orton Recording Quartz Dilatometer, or equivalent, will be used for this measurement. This equipment utilizes a linear variable differential transformer as the transducer to sense dilation, and a thermocouple to sense temperature. The LVDT must provide a linear output over a length of at least 0.050 in. (1.27mm), with readout capability to 0.0001 in. (0.003mm), and estimates to 0.00002 in. (0.0005mm). Potential errors should not exceed ± 0.00005 in. (0.0013mm) for any length change. These values may be confirmed using a specially mounted micrometer for which accuracy may be confirmed and traceable to standards acquired from the National Bureau of Standards.

4.2 In this procedure, the specimen is supported between members of a quartz frame and push-rod assembly. The assembly is inserted into a furnace capable of uniformly heating the specimen zone. As the specimen temperature is changed, changes in its length dimension result in relative displacement of the quartz push-rod and frame assembly. The amount of displacement is sensed by the LVDT and recorded on one scale of an X-Y plotter. Specimen temperature, sensed by the thermocouple, is recorded on the other scale. Thus, a continuous record of dilation versus temperature is produced.

4.3 Micrometer Calipers, with an index permitting direct reading of 0.001 in. (0.025mm) for measuring the initial specimen length. A high-grade screw micrometer customarily used in machine shop practice is satisfactory.

4.4 Electric Furnace, capable of maintaining the difference between the maximum and minimum temperatures of the specimen within 2 C.

4.5 Liquid Baths may be used when expansion data below 100 C are required. The bath shall be arranged so that a uniform temperature throughout the specimen is maintained. Means to control the desired temperature to within ±0.2 C shall be provided.

4.6 Temperature-Measuring Instruments - A calibrated thermocouple shall be provided for determining the temperature of the test specimen. Although a Type K thermocouple is recommended, Types T, E, S, or J may be used. Types T and E can be calibrated to indicate temperatures accurate to ±0.2 C and ±0.5 C, respectively, in the range -190 to 350 C. A Type S thermocouple can be calibrated to indicate temperatures accurate to ±0.5 C in the range 0 to 1000 C and is especially recommended for use in the range 350 to 1000 C. A Type S thermocouple should not be used for subzero temperatures. A Type K thermocouple can be calibrated to indicate temperatures accurate to ±0.5 C in the range -190 to 350 C. A Type J thermocouple can be calibrated to indicate temperatures accurate to ±0.5 C in the range 0 to 350 C. The thermocouple may be referenced to 0 C by means of an ice-water bath, if appropriate compensation for reference junction temperatures is not available in the recording instrument. Thermocouple calibration and recording accuracy should be such that potential errors can be limited to ±1 percent of the full-scale range being evaluated.

5.0 Apparatus Calibration

5.1 Temperature Calibration - A potentiometer capable of accurate voltage inputs to within ±1 X 10^{-6} volts shall be used to verify that the recorder converts the thermocouple input signal correctly to temperature. The procedures specified by the recorder manufacturer shall be followed.

5.2 Dilation Calibration - The LVDT and its signal conditioning equipment shall be calibrated using a micrometer for which accuracy is traceable to the NBS. Procedures specified by the manufacturer of the dilatometer apparatus shall be followed.

5.3 Calibration Checkout - After the procedures of 5.1 and 5.2 have been concluded, the apparatus accuracy shall be confirmed by thermal expansion measurement of an NBS standard reference material. The standard having expansion characteristics closest to those anticipated for the unknown should be used. Available standards include the following:

SRM-736	Copper
SRM-737	Tungsten
SRM-731	Borosilicate glass
SRM-739	Fused silica

6.0 Safety Precautions

6.1 The use of a vitreous silica dilatometer above 800 C may be accompanied by viscous flow and a time-dependent change of thermal expansion in the vitreous silica. The magnitude of these effects above 800 C will depend on the particular type of vitreous silica used to fabricate the dilatometer. To minimize errors caused by these effects, frequent calibration of the dilatometer is recommended when expansion measurements above 800 C are made.

7.0 Test Specimen Design

7.1 In the fabrication of test specimens several design considerations shall be followed:

7.2 Specimen length should be between 2 and 5 in. (51 and 127mm). Generally, specimens shorter than 2 in. result in a loss of sensitivity while specimens longer than 5 in. are subject to axial temperature differences in excess of the specified 2 C because of furnace gradients.

7.3 The minimum diameter or thickness of the specimen shall be 3/16 in. (4.8mm) or one sixteenth of the specimen length, whichever is smaller. Smaller sections may be subject to stresses large enough to produce significant creep or elastic strain rates, or both. The maximum diameter or thickness is determined by the inside diameter of the tube-type dilatometer and the distance between fixed and transmission rods in the rod-type dilatometer.

7.4 The shapes of specimen ends and the vitreous silica contact surface shall be designed so that the specimen remains laterally fixed during the test. Ideally, the specimen is a 2-inch-long rod nominally ¼ inch by ¼ inch. However, many other sizes and configurations can be accommodated by appropriate selection of control and boundary conditions.

7.5 Conditioning of specimens is generally necessary before reproducible expansion data can be obtained. For example, heat treatments are frequently necessary to eliminate certain effects (strain, moisture, etc.) which may introduce length changes not associated with thermal expansion.

8.0 Measurement Procedure

8.1 Clean the specimen and install it in the dilatometer after making certain that the end surfaces, as well as the contact surfaces of the dilatometer, are free of foreign particles. Take care to assure good seating of the specimen in the dilatometer. Place the thermocouple junction at the midpoint of the specimen. Either embed the junction in the specimen or place it on or close enough to its surface to insure representative temperature measurement. Mount the extensometer to provide good stable contact with the transmission rod and set it to a convenient reading. Insert the dilatometer assembly into the furnace or bath and then heat or cool by the furnace or baths, or both, to desired thermal schedules.

8.2 For the Orton Automatic Recording Dilatometer as used in this procedure, follow the instructions as furnished by the manufacturer, and summarized here:

a) Set the operating controls as follows:

Main Power	Off
Recorder	Manual
Furnace	Front or rear (Select)
Drive	Off
Program Timer	15 %
Program Power	10 %
Data Module	x1
Expansion	Zero
Correction Factor	Out
Temperature	Zero

b) Apply power to the instrument - turn on main power.

c) Turn on the recorder by operating the recorder power switch.

d) Load a sheet of graph paper. Ascertain that the lines on the graph paper are parallel to the pen bars.

e) Set the X axis to 0°C.

f) Set the Y axis zero upscale about 2 inches.

g) Switch the "Expansion" switch to x.01.

h) Adjust the LVDT or Data Module zero control until the reading corresponds with the point set in (f).

i) Move the "Expansion" switch between x.01 and x.05. Deflection should be less than one inch. Movement of less than .05 inch is desirable if ranges are to be changed during the run.

j) Load the sample into the furnace, visually checking for proper centering.

k) Place the thermocouple so that it rests on top of or very near the sample.

l) Set the temperature limit at the desired program shutoff.

m) Check operation of the temperature limit and shrink limit.

n) Set the Temperature Switch to the desired temperature range.

o) Adjust the Y axis recorder zero control for the desired starting point on the Y axis. The Expansion range switch should be in the desired plotting range.

p) Push the Start pushbutton. The white light should remain on.

q) Select front or rear furnace.

r) Turn the Drive switch on.

s) Move the Furnace switch to ON.

t) Lower the recorder pen.

9.0 Data Processing and Reporting

9.1 The data as recorded provide a direct indication of percent expansion versus temperature for the measured specimen. This plot, or a copy, may be used directly in a report.

9.2 In the event that measurement was made on an abnormal-size specimen, and the recording options did not permit direct recording in integral increments of the expansion, the recorded data should be corrected as necessary before reporting.

9.3 When the final, room-temperature dimensions of the specimen are different from the initial dimensions, both sets should be recorded and reported. The important dimensions are weight and length.

9.4 Specific conditions of the measurement procedure which are pertinent to results should also be reported. These include heating rate, atmosphere, etc.

10.0 General Comments

10.1 It is to be emphasized that the procedures and information outlined in this specification are merely a guide to measurement of the indicated property, and as such should not be presumed to include all possible contingencies which might occur.

10.2 It is understood that the expansion measurement will be set up and carried out by someone who has an adequate background in thermal physics and related sciences, and is experienced in all phases of the procedures relating to this measurement.

10.3 This specification includes, by inference, the utilization of advanced technology and quality workmanship in all phases of the measurement. Employment of these procedures by untrained or inexperienced personnel might void the terms and spirit of this specification.

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APPENDIX B

Specific Heat Measurement Method Specifications

Test Procedure No. EMTL-TP-SH-301

SPECIFIC HEAT MEASUREMENT

BY

DROP (ICE) CALORIMETER

Approved by:

Tom Daley
Manager, EMTL

John F. Ragedorst
Manager, Thermal Properties Laboratory

Ray Flaherty
Manager, QA/QC

October 25, 1979

THE ENERGY MATERIALS TESTING LABORATORY

A Division of

Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, Maine 04005

SPECIFIC HEAT MEASUREMENT

BY

DROP (ICE) CALORIMETER

Foreword

This procedure is in essential conformance with ASTM Specification D-2766, Standard Method of Test for Specific Heat of Liquids and Solids. It includes consideration of a number of modifications and refinements to the basic specification, to permit measurements over extended temperature ranges and under potentially hostile environments.

1.0 Background

The temperature of a system can be changed by the addition to or removal of heat. The amount of heat which must be exchanged per unit mass and per unit temperature change at any given temperature and pressure is defined as the specific heat of the system. The defining equation is:

$$c = \frac{Q}{m\ \Delta T}\ , \qquad (1)$$

where:

c = specific heat

Q = quantity of heat

m = mass of material

ΔT = change in material temperature

If the heat content, or enthalpy, is represented by the symbol H, then the heat capacity at constant pressure of a unit mass is given by:

$$C_p = \left(\frac{dH}{dT}\right)_p \qquad (2)$$

A similar equation can be used to express specific heat at constant volume.

References 1 and 2 are suggested for studies of the theory of specific heat.

With regard to techniques for the measurement of specific heat in solids, the primary methods are the adiabatic method, the comparative method, the pulse-heating method, the method of mixtures (drop method), and variations of these. The technique described here is a variation of the drop method, and is sometimes referred to as the ice calorimeter.

2.0 Description of the Method

In this method, the heat given up by a pre-heated specimen in cooling to equilibrium in the calorimeter, is used to melt a portion of the ice in a sealed ice-water bath. The resulting net change in volume of the bath is determined through use of a transfer agent, usually mercury. Thus, through calibration, it is possible to relate heat quantity to mercury volume (or weight) exchange, for a given calorimeter. All heat transfer occurs at the ice point.

A single drop of a specimen of known weight from a known temperature provides an enthalpy value for that temperature. A series of drops from various temperatures thus provides data for a curve describing enthalpy versus temperature. The slope or derivative of this curve is the specific heat of the specimen.

3.0 Apparatus Calibration

3.1 An electric heater for which the power can be accurately measured is lowered into the calorimeter.

3.2 The amount of mercury moved as a function of power input is recorded to establish the constant for the apparatus.

3.3 Performance of the calorimeter is then checked through enthalpy measurements of Reference Standard Al_2O_3 (SRM-720, Sapphire) acquired from the National Bureau of Standards. Values of individual enthalpy points must be within ± 1 percent of the NBS value.

3.4 For additional information, consult Reference 3.

4.0 Specimen Preparation

4.1 Since specific heat is a mass function, specimen dimensions need not be defined. The only dimension of concern is weight.

4.2 In this procedure, the specimen will be contained in a compatible capsule. Sealing of the capsule is necessary if there are atmosphere control problems, or if reversible transformations incorporating heat effects are involved.

4.3 The specimen must be clean, and must be representative in composition of the material being evaluated.

5.0 Measurement Procedure

5.1 Following appropriate preparation and cleaning of all calorimeter components, freeze an integral ice mantel onto the outer surface of the calorimeter well (inner surface of the ice-water sealed chamber).

5.2 Assure that the ice-water bath temperature is at the freezing point of pure water at the prevailing atmospheric pressure.

5.3 Provide a reasonable quantity of mercury in an external accounting system, which is connected by tube to the reservoir of mercury inside the calorimeter. The arrangement should be such that the vertical level of mercury in the external reservoir is the same as that inside the system.

5.4 Load an encapsulated specimen into a furnace above the calorimeter well, and heat to a temperature within the range to be examined.

5.5 While the specimen is heating, monitor on 10-minute intervals any change in weight of the mercury in the external accounting system. This provides baseline data of trends due to changes in atmospheric pressure, which will facilitate refinement of the measured enthalpy data.

5.6 After at least four such monitorings, and assuming the specimen has reached the desired temperature, drop the encapsulated specimen into the calorimeter well, having opened the connecting gate for a minimum time to keep the calorimeter well thermally isolated as much as possible.

5.7 Continue to monitor exchange of mercury on 10-minute intervals, until further changes are negligible.

5.8 Convert the total volume of mercury exchange to calories. This provides an enthalpy value for the temperature to which the specimen was preheated.

5.9 A separate similar drop of an empty capsule provides, by difference, the enthalpy contribution by the specimen alone, at the drop temperature.

5.10 The drop process, Paragraphs 5.6 through 5.9, is repeated at a sufficient number of temperatures in the range to be studied, to provide a reasonably well-defined curve of enthalpy versus temperature.

6.0 Calculation of Specific Heat

6.1 Plot all enthalpy values versus temperature.

6.2 Establish a best-fit curve to the enthalpy data.

6.3 Calculate specific heat from the Paragraph 6.2 curve, either by differentiating an equation which describes the curve, or by graphically determining successive slopes.

7.0 Reporting

7.1 Tabulate the enthalpy data.

7.2 Tabulate calculated specific heat values. (Optional)

7.3 Plot specific heat versus temperature. (Optional)

7.4 Describe how specimen was prepared and encapsulated

8.0 General Comments

8.1 It is to be emphasized that the procedures and information outlined in this specification are merely a guide to measurement of the indicated property, and as such should not be presumed to include all possible contingencies which might occur.

8.2 It is understood that the expansion measurement will be set up and carried out by someone who has an adequate background in thermal physics and related sciences, and is experienced in all phases of the procedures relating to this measurement.

8.3 This specification includes, by inference, the utilization of advanced technology and quality workmanship in all phases of the measurement. Employment of these procedures by untrained or inexperienced personnel might void the terms and spirit of this specification.

9.0 References

9.1 Debye, P., "The Theory of Specific Heat", Ann. Physik, Vol. 39 (4), pp. 789-839 (1912).

9.2 Einstein, A., "The Planck's Theory of Radiation and the Theory of Specific Heat", Ann. Physik, Vol 22 (4), pp. 180-190 (1907).

9.3 Ginnings, D.C., and Corruccini, R.J., "An Improved Ice Calorimeter - The Determination of its Calibration Factor and the Density of Ice at O.C.", Journal of Research, National Bureau of Standards, Vol. 38, pp. 583-591 (1947).

APPENDIX C

Thermal Conductivity Measurement Method

Specifications

Test Procedure No. EMTL-TP-TC-201

THERMAL CONDUCTIVITY MEASUREMENT BY STEADY-STATE COMPARATIVE METHODS

Approved by:

Manager, EMTL

Manager, Thermal Properties Laboratory

Manager, QA/QC

October 25, 1979

THE ENERGY MATERIALS TESTING LABORATORY

A Division of

Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, Maine 04005

THERMAL CONDUCTIVITY MEASUREMENT

BY

STEADY-STATE COMPARATIVE METHODS

Foreword

This procedure is in essential conformance with ASTM Specification C-518, Standard Method of Test for Thermal Conductivity of Materials by Means of the Heat Flow Meter. Although ASTM C-518 was written primarily to apply to homogeneous insulating, building, and other materials for which thermal conductivities do not exceed approximately 2 Btu hr^{-1} ft^{-2} F^{-1} (1.13 mw cm^{-2} c^{-1}), the basic principles can be applied to a wide variety of methods, materials, and conductivity ranges. The primary requirement is that the material used as a heat-flow-meter be certifiable with regard to the accuracy of its thermal conductivity.

1.0 Background

The thermal conductivity of a material relates the heat flow to the temperature gradient under steady-state conditions. This is in contrast to thermal diffusivity, which is a property of interest where transient conditions prevail.

The process by which heat is transferred is diffusional even though the detailed mechanisms are not always the same in different materials. Gases tend to have the lowest thermal conductivity since the thermal energy is transported by the atoms or molecules themselves which interact by collisions. Liquids possess a mobility more characteristic of a gas than of a solid, yet have short-range order not unlike the solid phase. Thus, in most cases, the liquid phase has a conductivity which is intermediate between the particular material's gas and solid phases. Heat transfer in dielectric solids is mainly by elastic waves or lattice vibrations - the quantized energy of which is sometimes referred to as a phonon. In highly ordered solids, such as single crystals, these phonons are able to travel relatively far at low temperatures, and hence, high conductivities are observed. Greater disorder from impurities, vacancies, crystallite boundaries, anharmonic coupling, etc., lead to more scattering of phonons and this lowers thermal conductivity. Electrical conductors possess free electrons which offer yet another mode for the transport of thermal energy; however, the general considerations of disorder and scattering still apply. In some metals the bulk of the thermal energy is transported by free electrons, which leads to a relationship between thermal conductivity and electrical resistivity known as the Wiedemann-Franz law.

Materials that are not opaque to electromagnetic radiation in the visible and infrared are capable of heat transport by photons in addition to phonons. Other heat-transport mechanisms and complications, such as anisotropy, quite often require special attention to measure and interpret correctly thermal-conductivity data. A number of procedures are available for evaluating heat-flow mechanisms and conductivity in specific materials, and these studies are an interesting part of thermal-conductivity research.

Many methods and techniques exist for the experimental determination of the thermal conductivity of solids. Selection of a method is based upon the temperature range to be examined, the range of thermal conductivity values anticipated, the physical nature of the material, geometry of available samples, required accuracy, economics, etc. Broadly, the methods are identified in two principal categories, steady-state and transient. Within the steady-state category are two basic methods, absolute and comparative. The apparatus described here is steady-state, comparative, and is intended for solids having moderate-to-low conductivities.

2.0 Scope

2.1 This method covers the determination of thermal conductivity of various classes of solids by use of a heat-flow meter.

2.2 Definition of thermal conductivity by this method implies that conductivity of the meter must be known through measurement by some absolute technique, or through traceability to work performed by or recognized by the National Bureau of Standards.

2.3 For practical reasons, the meter should be selected on the basis of its conductivity being in the same general range anticipated for that of the unknown.

2.4 The suggested limiting temperatures for this method are -40 C and 1000 C (-40 F and 1832 F).

3.0 Description of Terms

3.1 Thermal conductivity may be expressed in a variety of units, as identified in Paragraph 9.0 Reports.

3.2 The quantities to be measured include temperatures, rate of one dimension heat-flow, dimensions relative to thermocouple locations, and pertinent dimensions of the specimen and heat-flow meter.

4.0 Measurement Apparatus and Method

4.1 The significant components of a steady-state comparative thermal conductivity apparatus consist of a heat source, a heat-flow meter, a specimen to be evaluated, and a heat sink. Ancillary equipment includes power supplies, coolants, thermocouples, vacuum chambers, recorders, etc., as need to satisfy specific conditions.

4.2 Depending on the type and conductivity range of the specimen, the heat source can be in the form of a flat plate or a cartridge; whatever is suitable to impart a uniform heat load to the receiving surface of the specimen. The source is electrically heated by means compatible with the materials and temperature ranges involved. Facilities should be available to monitor the voltage and current to the heater, although this is not mandatory in all cases.

4.3 The heat-flow meter is a material of known thermal conductivity, through which flows the same quantity of heat which flows through the specimen; the components are arranged in series. Thermocouples are positioned in the meter such that, with its known conductivity, calculation of the heat flow rate through it is possible.

4.4 The specimen should be in the form of a right cylinder, the dimensions of which are dictated by its anticipated conductivity. If the conductivity is expected to be low, i.e., in the range of insulators, the specimen should be a disk in which the diameter is large compared with the thickness. Conversely, if the conductivity is high, as in a metal, the specimen should be a rod, in which the diameter is small compared with the length.

4.5 The sink can be any flat surface which can be maintained in good physical contact with the specimen or meter, and which can be cooled by a medium appropriate from the standpoints of temperature range and compatibility.

5.0 Apparatus for Thermal Conductivity Measurement of Geologic Media

5.1 Materials in this category include salt, granite, tuff, basalt, shale, caprock, or combinations of these.

5.2 The specimen shall be a disk, nominally three inches in diameter by one inch thick. Grooves nominally 0.07 inch wide and 0.07 inch deep shall be machined across the diameter of each face; these are for positioning of thermocouples.

5.3 The heat-flow meter shall be of Pyroceram 9606, as investigated and characterized by the National Bureau of Standards (See reference 1). Its diameter is nominally three inches, and length, nominally two inches.

5.4 The heat sink is a water-cooled copper plate on which the heat-flow meter rests.

5.5 The meter is fitted with Type K thermocouples along its axis, and at corresponding radial locations. The axial thermocouples provide information to determine heat flow through the specimen and the meter; the radially-located thermocouples evaluate the quality of one-dimension heat flow.

5.6 The heat source is a three-inch-diameter metal bar, preferably nickel or a similar high-conductivity metal, into which are fitted a sufficient number of cartridge electric heaters to provide at least 100 watts of power.

5.7 The entire assembly shall be mounted inside an evacuable chamber, for the purpose of minimizing atmospheric contamination, or of effecting a significant change in the moisture content of the sample. The measurement actually takes place in static air, to maintain, as much as possible, in-situ conditions.

6.0 Apparatus Calibration

6.1 Statements here apply only for the apparatus as described in Paragraph 5.0.

6.2 Calibration of the heat-flow meter is unnecessary since it was acquired from and has been characterized by the National Bureau of Standards.

6.3 Certification that thermocouple wire falls within the ANSI limits of error specified in ANSI-C 96.1 is obtained from the supplier. Confirmation of this accuracy can be achieved through measurements against a secondary standard procured from the Nation Bureau of Standards. However, for the materials class indicated in Paragraph 5.0, conformance to the ANSI specification is adequate.

6.4 Calibration of length-measuring instruments, principally micrometers in this case, must fall within local requirements schedules. Here, calibration checks must be performed on a six-month schedule.

6.5 Calibration of quantities relative to the heat source and the heat sink are not necessary.

7.0 Measurement Procedure

7.1 Clean the specimen, using a material in which it is not soluble.

7.2 Locate an insulated (2-hole ceramic) thermocouple in each groove in the specimen, with the bead on or near the axis. To achieve good thermal bonding, cement the bead into the groove, using a conductive, high-temperature cement.

7.3 Position the specimen on the heat-flow meter (in the chamber), seperated from it by a thin, conductive cloth. Graphite cloth is satisfactory.

7.4 Place a second cloth on top of the specimen.

7.5 Position the heat source on top of this cloth. The entire assembly of heat source, specimen, and meter should be aligned.

7.6 Insulate the assembly by surrounding and covering it with a powder or granular insulation. Close the chamber.

7.7 Flow coolant water through the heat sink.

7.8 Introduce power to the heater.

7.9 After thermal equilibrium is achieved throughout the specimen - meter assembly, record all temperatures.

7.10 Proceed to another thermal equilibrium by increasing the power setting to the heater, and again record all temperatures.

7.11 Record data at each of a number of equilibria through the range to be examined.

8.0 Calculations

8.1 From the above temperature data, used in combination with dimension data on the specimen, calculate thermal conductivity from the relation.

$$\lambda = \frac{q}{A} \cdot \frac{x}{\Delta T} ,$$

where λ = thermal conductivity
q/A = heat flow per unit cross-section area
x = thickness of specimen between its thermocouples.
ΔT = temperature difference across distance x.

8.2 This value of conductivity applies for the average temperature through the specimen thickness.

8.3 Calculate a conductivity value for each thermal equilibrium.

9.0 Data Reporting

9.1 Identify the specimen, its pertinent dimensions, and conditions.

9.2 Tabulate conductivity data versus temperature.

9.3 Plot data; and fit curve, if appropriate.

9.4 Present data in either of the following units:

$$w\ cm^{-1}\ C^{-1}$$
$$Btu\ in\ hr^{-1}\ ft^{-2}\ F^{-1}$$

10.0 General Comments

10.1 It is to be emphasized that the procedures and information outlined in this specification are merely a guide to measurement of the indicated property, and as such should not be presumed to include all possible contingencies which might occur.

10.2 It is understood that the conductivity measurement will be set up and carried out by someone who has an adequate background in thermal physics and related sciences, and is experienced in all phases of the procedures relating to this measurement.

10.3 This specification includes, by inference, the utilization of advanced technology and quality workmanship in all phases of the measurement. Employment of these procedures by untrained or inexperienced personnel might void the terms and spirit of this specification.

REFERENCE 1:

Robinson, H.E., and Flynn, D.R., "The Current Status of Thermal Conductivity Reference Standards at the National Bureau of Standards," Proceedings of the Third Conference on Thermal Conductivity, Oak Ridge National Laboratory, Tennessee (October 16-18, 1963).

APPENDIX D

Thermal Diffusivity Measurement Method

Specifications

Test Procedure No. EMTL-TP-TD-501

THERMAL DIFFUSIVITY MEASUREMENT

BY

LASER FLASH TECHNIQUE

Approved by:

Tom Daley
Manager, EMTL

John F. Lagedrost
Manager, Thermal Properties Laboratory

Ray Carpentier
Manager, QA/QC

November 20, 1979

THE ENERGY MATERIALS TESTING LABORATORY

A Division of

Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, ME 04005

THERMAL DIFFUSIVITY

Measurement Method and Procedure

1.0 Background

Thermal diffusivity is a quantity which enters into equations relating to heat flow under nonsteady-state conditions. Because of its relationship to thermal conductivity, diffusivity is of particular interest in studying steady-state as well as nonsteady-state heat flow situations.

Thermal conductivity is usually defined as the quantity of heat transmitted in a direction normal to a surface of unit area, due to unit temperature gradient in unit time under steady-state conditions. This was expressed by Fourier([1]) as:

$$\frac{Q}{A} = \lambda \frac{dT}{dx} , \qquad (1)$$

where:

Q/A = quantity of heat flow through area A

λ = thermal conductivity

$\frac{dT}{dx}$ = temperature gradient through thickness x

In cases where the thermal conductivity may be considered independent of temperature, that is, over a fairly short temperature range, but where temperature varies with time, Equation (1) becomes:

$$\rho C_p \frac{dT}{dt} = \lambda \frac{d^2T}{dx^2} , \qquad (2)$$

where:

ρ = material density

C_p = specific heat at constant pressure

(1) References at end

The quantity $\frac{\lambda}{\rho C_p}$ was defined by Thomson[2] as thermal diffusivity, so Equation (2) may be expressed:

$$\frac{dT}{dt} = \alpha \frac{d^2T}{dx^2} , \qquad (3)$$

where α is thermal diffusivity. More specific to this discussion, the relationship of thermal conductivity and thermal diffusivity is expressed as:

$$\lambda = \alpha \rho C_p \qquad (4)$$

Detailed treatments of this derivation are presented by several authors, including those cited in References 2-4.

The above outline explains briefly the prominence and importance of thermal diffusivity measurements in studies involving thermal conductivity in particular, and thermal transport in general. Thermal diffusivity measurements bypass most of the difficult problems associated with accurate, steady-state (conductivity) measurements, and can be made with far less specimen inventory and at considerably lower cost. The development of advanced measurement techniques has encouraged use of the diffusivity approach to evaluate conductivity on many classes of solids.

2.0 Measurement Method

In the procedure described here, thermal diffusivity is measured by a flash method. A short-duration pulse of thermal energy is absorbed on one face of a slab specimen, and allowed to propagate through the thickness of the specimen. The thermal response of the opposite face is monitored as a function of time, and recorded with an oscilloscope. Thermal diffusivity is then calculated as a relation of this time function and the specimen thickness. This measurement method conforms generally to Specification ASTM C-714-72, with modifications to permit accurate measurements to elevated temperatures.

Parker et al [5] are usually credited with development of initial work using flash methods to measure thermal diffusivity over a wide range of temperatures. Subsequently, other researchers (References 6-11) have developed procedures and analyses which make possible the application of the flash method to non-ideal specimen materials through use of corrections for heat losses, finite dimensions, finite pulse times, anisotropic structures, etc.

The simplified relationship which has been derived to relate diffusivity with specimen thickness and heat pulse traverse time is basically:

$$\alpha = \frac{\omega L^2}{t_{\frac{1}{2}}}, \qquad (5)$$

where:

L = specimen thickness

$t_{\frac{1}{2}}$ = time for back-face temperature to reach one-half its maximum

ω = parameter which is a function of heat loss from the specimen. For the ideal case of zero heat loss, the value of this parameter is 0.139.

Heat loss corrections have been identified by Taylor[12], Cowan[8], Cape and Lehman[7], and Larson and Koyama[13].

3.0 Measurement Apparatus

The essential features of the flash thermal diffusivity measurement apparatus are illustrated schematically in Figure A-1. The specimen, usually in the form of a disk, is positioned in an isothermal zone of a furnace inside an evacuable chamber. The pulsed heat source and the thermal response detector device, along with associated instrumentation, are located external to this chamber.

The specific features of this apparatus include the following:

3.1 Specimen Design

The specimen is a disk nominally 0.500 inch (1.27cm) in diameter, and with a thickness such that the half-time required for the heat pulse to traverse this thickness is of the order of 100 times the duration of the flash source. Longer or shorter half-times, resulting from specimen thicknesses outside this range, will require additional analysis. (See References 10,12,13) The specimen faces must be flat and parallel.

3.2 Furnace Design

The furnace element is of tubular design, with power supply adequate to achieve and maintain a given specimen temperature to

within 0.1 degree K during the measurement. The element is enclosed in an evacuable chamber, which includes provision for maintenance and control of inert gas pressure.

3.3 Temperature Measurement

The specimen ambient temperature is measured by thermocouple, while the temperature excursion caused by the thermal pulse is monitored by an infrared detector which views the back face of the specimen remotely. This detector is selected to give appropriate sensitivity for the temperature range being examined.

3.4 Flash Source

Our apparatus uses a pulsed laser to provide the heat source for this measurement. The laser rod is of neodymium glass which is triggered by a xenon flash lamp, and has a pulse duration of approximately 1 ms.

3.5 Heat Pulse Recording

The signal from the IR detector is displayed on an oscilloscope which is capable of digitizing the recorded quantities, as well as providing an analog recording. Figure A-2 illustrates an example of an oscilloscope trace, showing the parameters used to calculate thermal diffusivity.

3.6 Temperature Range of Operation

Our present apparatus is capable of measuring thermal diffusivity through the range from room temperature to 2760 C (5000 F).

3.7 Measurement Accuracy

Since this is an absolute measurement method, no calibration of the apparatus is required. However, it is essential that the measurements be carried out with care, and that the operator must have a comprehensive understanding of the many basic principles of physics that are involved. Obviously, it is also necessary that the various ancillary components, especially the oscilloscope, be properly calibrated.

The accuracy of diffusivity data derived by this method may be certified by measurements on an acceptable standard reference material. In our laboratory, we use Armco iron as the reference standard in the temperature range RT-800 C, and ATJ graphite in the range above 800 C. Both materials have been extensively characterized by many laboratories, and the consensus of these data are used as the reference standard curves.

Figures A-3 and A-4 are plots of the thermal diffusivities of these two reference materials, along with data recorded for them in our apparatus. To insure valid performance of the apparatus, such measurements are carried out as part of each program involving a given set of materials. Generally, the curves indicate that conformance is within ± 5 percent of absolute values.

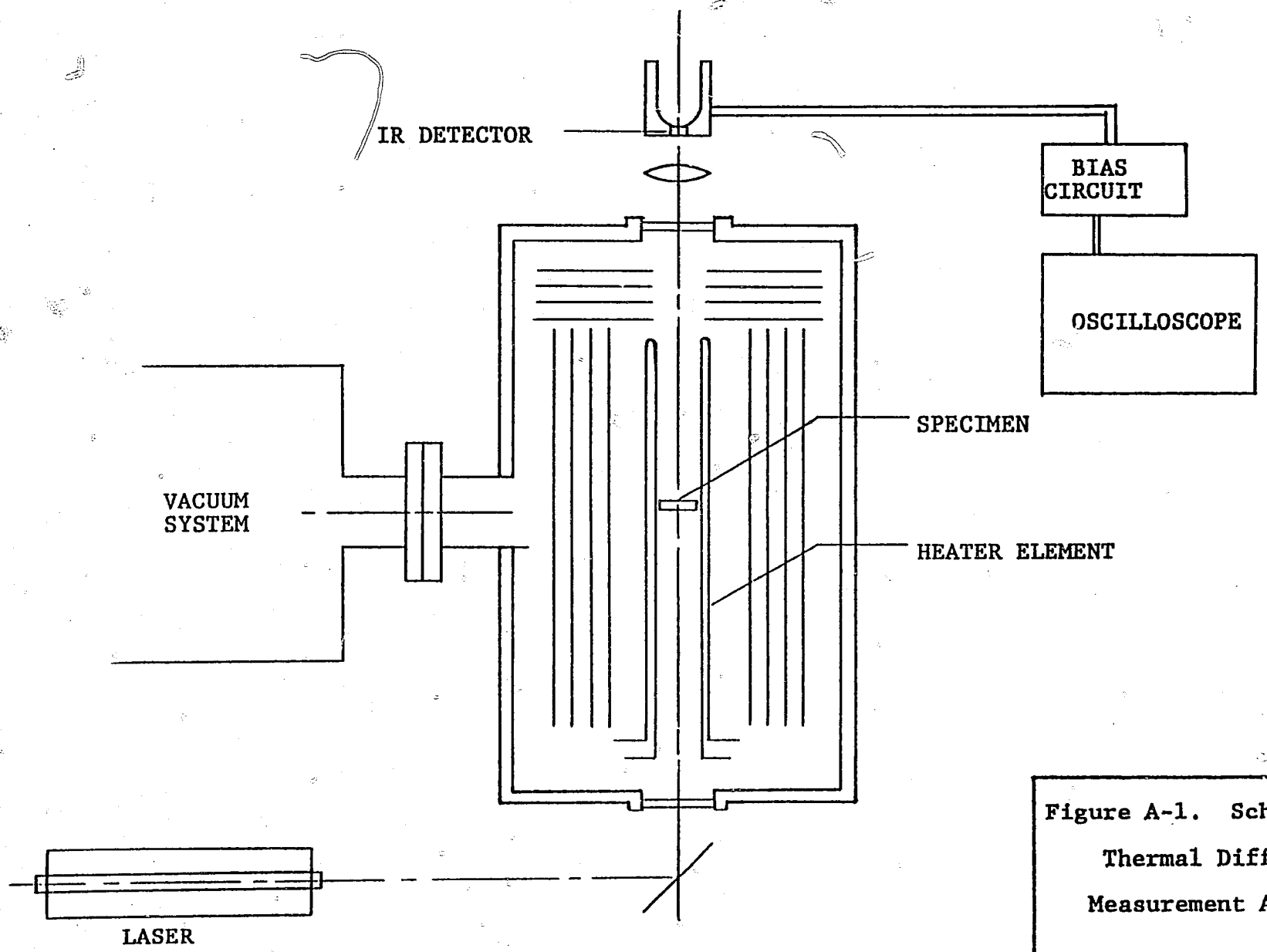

Figure A-1. Schematic of Thermal Diffusivity Measurement Apparatus

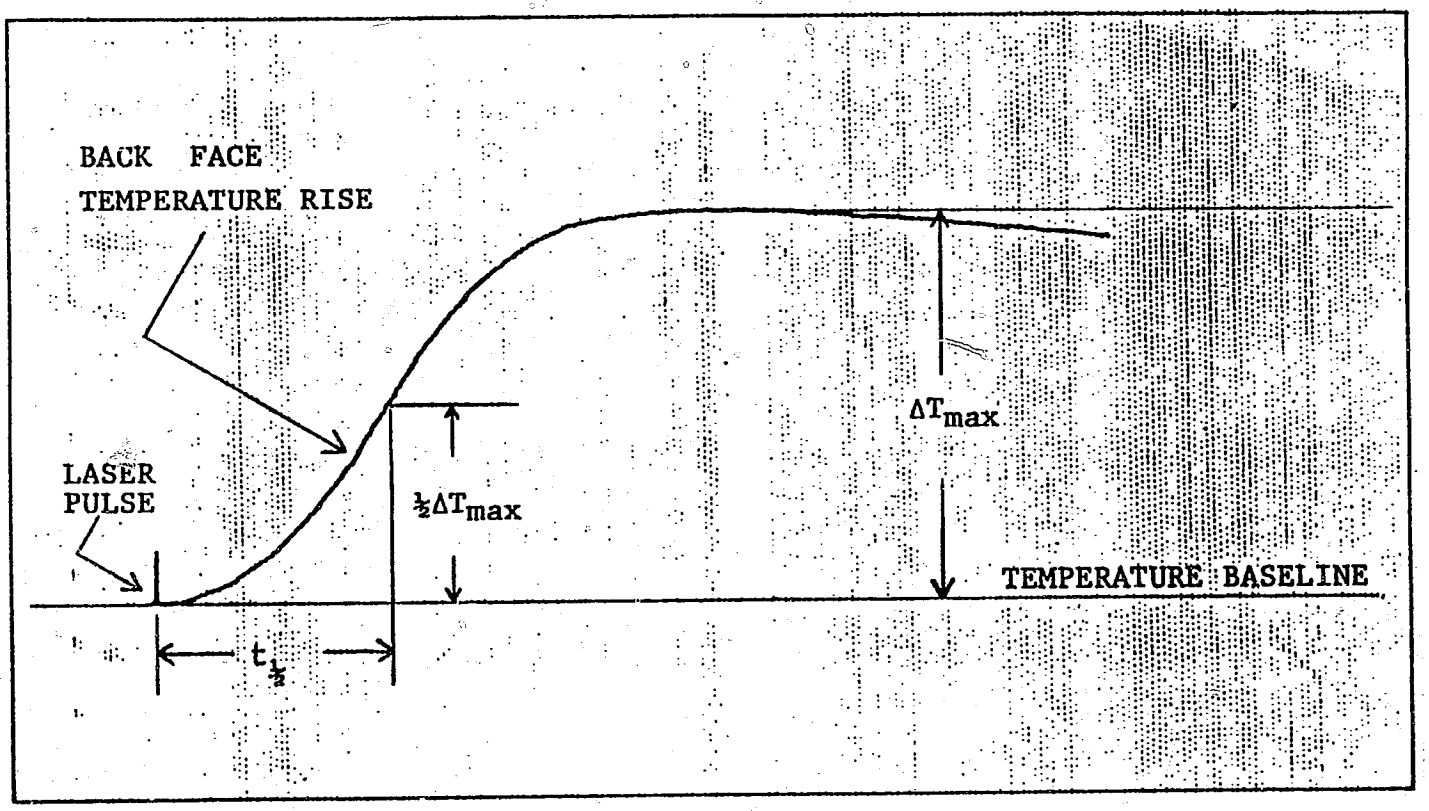

Figure A-2. Example of Oscilloscope Trace, Showing Basic Parameters Used to Calculate Thermal Diffusivity.

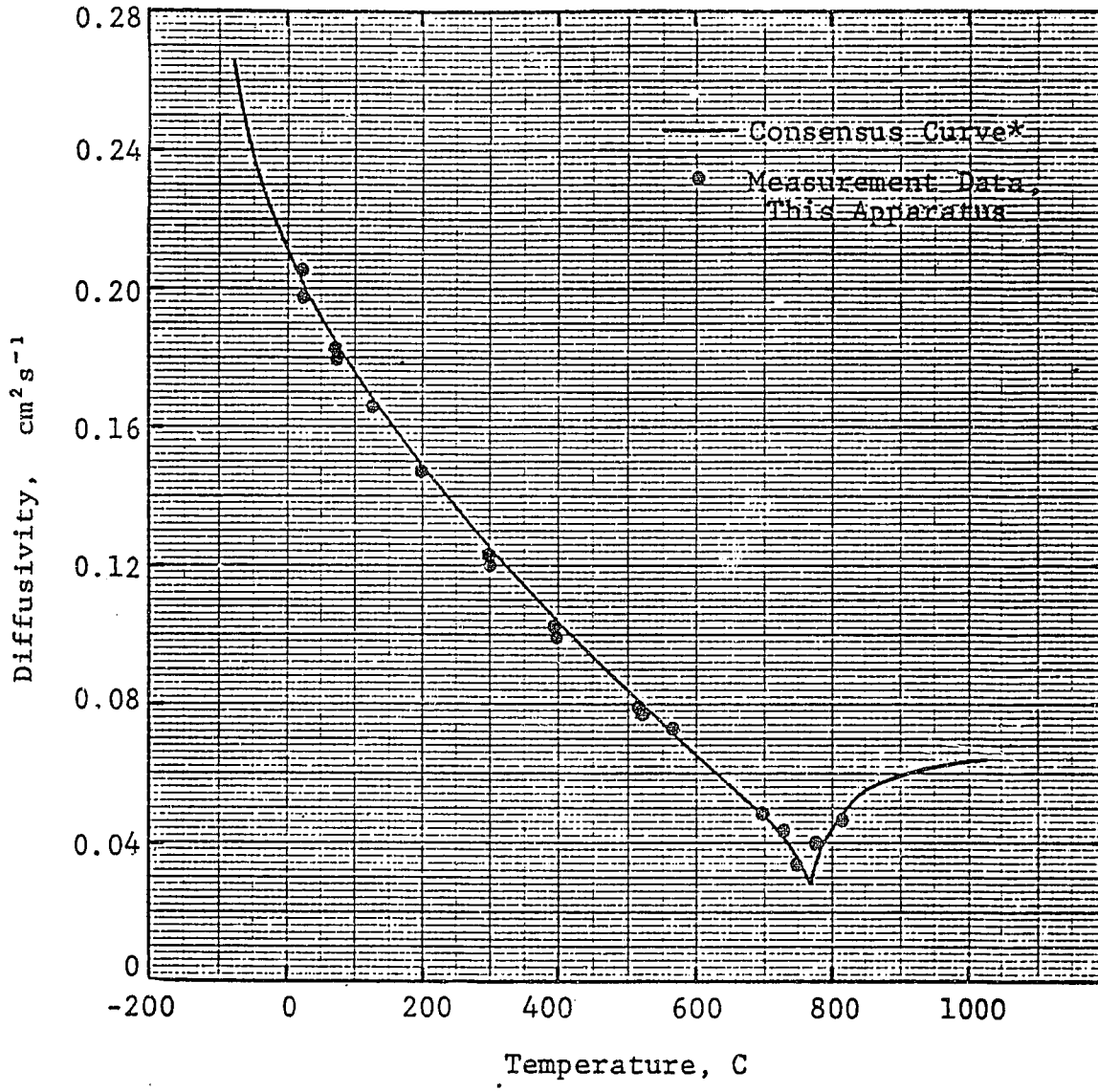

Figure A-3. Thermal Diffusivity of Reference Standard Armco Iron

*Based on a compilation by the Thermophysical Properties Research Center, Purdue University, of over 60 sets of data by various researchers, and published in "Thermophysical Properties of Matter Volume 10, IFI/Plenum (1973).

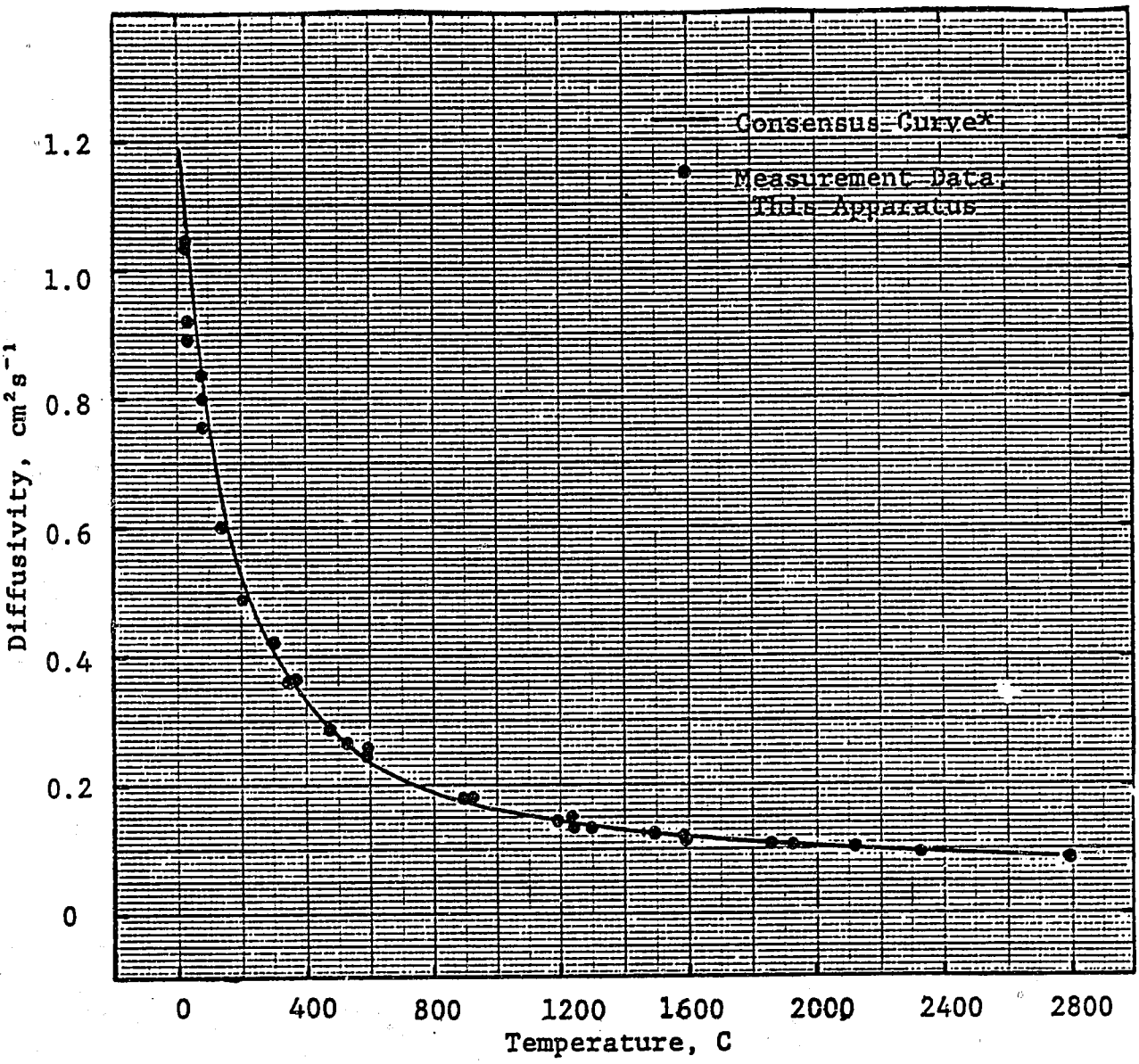

Figure A-4. Thermal Diffusivity of Reference Standard ATJ Graphite

*Based on a compilation of data by The Thermophysical Properties Research Center, Purdue University, and published in "Thermophysical Properties of Matter", Volume 10, IFI/Plenum (1973).

4. STEP BY STEP PROCEDURE

4.1 Changing Samples

1) Remove detector by loosening two set screws. The detector is delicate, thus, care in removing and storing is required.

2) Remove detector housing.

3) Loosen top of furnace by removing eight screws.

4) Remove bottom window by removing three screws and releasing vacuum. The window will drop out when vacuum is released.

5) Remove top of furnace.

6) Remove thermocouple.

7) Remove isothermal tube.

8) Remove ring and sample.

9) Lower the new sample using a ruler.

10) Insert isothermal tube.

11) Insert thermocouple.

12) Check thermocouple output.

13) Lay on top shield.

14) Check continuity of thermocouple using continuity light to insure that thermocouple is touching metal at the bottom.

15) Clean top window.

16) Lay on furnace top.

17) Blow off top window.

18) Clean bottom O-ring inside furnace.

19) Insert lower window into recess and pull vacuum.

20) Bolt on lower window cooling block.

21) Close top iris using drill bit as hole sizer.

22) Put on detector housing aligning magic marker lines.

23) Put on detector aligning scratches.

24) Tighten two set screws.

25) Detector alignment is attained by moving detector until maximum OS signal is obtained.

4.2 Laser Operation

1) Turn on ionized water circulating pump.

2) Check for water leaks around front and back rod O-rings and around black and blue box mating line.

4.3 Furnace Operation

1) Turn on water to furnace chamber and furnace electrodes.

2) Turn on furnace power with circuit breaker.

3) Pump down furnace three times and backfill to 15 inches vacuum of helium.

4) Adjust temperature of furnace using powerstat and temperature versus setting curve.

5) For a RT point, the furnace and cooling water are not operated.

4.4 Detector Operation

1) Add liquid nitrogen to InSb detector cryostat. This must be done twice a day.

2) The Si detector does not require LN_2.

3) The InSb detector is used to 1000 C with SM 32717 lens.

4) The Si detector is used above 1000 C with glass lens.

5) Above 1200 C the cooling adapter is used on the lens.

6) Above 1500 C, the 1-59 filter is used.

7) Above 1800 C, the 1-69 filter is used.

8) The small black box is used with the InSb detector and the large black box with the Si detector.

4.5 Firing Procedure

1) Turn off Vertical and Horizontal expansions and autocenter of OS.

2) Put time cursor at convenient value.

3) Turn laser power supply on, setting should be 4 KV.

4) Remove brass shields, one above mirror, one below furnace.

5) Turn on black box.

6) Record temperature.

7) Push Auto trigger.

8) Push Live storage.

9) Turn time base to 1 MS.

10) Zero digital mV signal using black box.

11) Autocenter line.

12) Push charge button.

13) Push Normal trigger.

14) Push Live storage button.

15) Push Hold Next storage.

16) Turn time base to convenient place.

17) Push FIRE button.

18) Check data for peak height and 5 X $T_{\frac{1}{2}}$.

19) Repeat as necessary.

20) Go to next channel.

21) Go to Step 6.

22) Turn channel selector to all to check similarity of data.

23) Turn off laser power.

24) Replace brass shields.

25) Turn off black box.

26) Record hard copy of data on chart recorder.

27) Turn furnace power up to next temperature.

4.6 Data Reduction

1) Record firing point by noting laser pip or by time cursor setting.

2) Set horizontal base line.

   a) Expand V and H expansions and move cursor to line before firing point.

   b) Find a point which represents horizontal base line before firing point.

   c) Turn off Hand autocenter.

   d) Adjust digital mV setting to zero using data move and inverter.

   e) Recenter data with autocenter.

3) Find peak.

   a) Turn V and autocenter on and move to peak.

   b) Turn off autocenter and turn on H.

   c) Find a point that represents average peak value and record mV value.

4) Find $T_{\frac{1}{2}}$.

   a) Run cursor to ½ Peak mV value.

   b) Find point that represents ½ peak value checking that the line runs through center.

c) Record time associated with ½ Peak Value and subtract Firing Point Time to determine T½. Record this value as T½.

5) Calculate ω factor.

a) Multiply T½ by 5 and add Firing Point time.

b) Run cursor to this time value.

c) Expand V and H and find mV point which represents the average mV value at 5 T½ + FP Time. Record this mV value.

d) Divide 5 T½ mV value by Peak Value X 2 and look this up on ω curve.

e) Record this value.

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13. Larson, K.B., and Koyama, K., "Correction for Finite-Pulse Time Effects in Very Thin Samples Using the Flash Method for Measuring Thermal Diffusivity", Journal of Applied Physics, Vol. 1967, Pg. 465 (1967).

APPENDIX E

Density Measurement Method
Specifications

Test Procedure No. EMTL-TP-D-401

DENSITY MEASUREMENT BY DISPLACEMENT

Approved by:

Manager, EMTL

Manager, Thermal Properties Laboratory

Manager, QA/QC

November 15, 1979

THE ENERGY MATERIALS TESTING LABORATORY

A Division of

Fiber Materials, Inc.
Biddeford Industrial Park
Biddeford, ME 04005

DENSITY MEASUREMENT BY DISPLACEMENT

Foreword

This specification is in essential conformance with ASTM Specification D-792, Standard Methods of Test for Specific Gravity and Density of Plastics by Displacement, as it applies to solids in tube, rod, or molded form.

1.0 Scope

This method of measuring the density of solids is especially useful in evaluating those materials which are of irregular shape, or which cannot be readily machined to a regular shape so that density can be calculated from dimensions. It is also useful in determining the porosity of porous materials. Generally, the measurement is made at room temperature.

2.0 Principle of Technique

Based on work by Archimedes, a relationship has been established to define density as a function of weight of an object in static gas, and its weight when immersed in a fluid of known density. Basically, the principle establishes a relationship between density and buoyant force on immersion. It states that a body immersed in a fluid is buoyed up by a force equal to the weight of the fluid displaced. Thus, the relationships can be expressed:

$$F = \rho g V \qquad (1)$$

where F = buoyant force
ρ = density of fluid
g = acceleration due to gravity
V = displaced volume

The quantities ρ and g are known, F is measured, and V, the displaced volume of fluid and also the volume of the immersed specimen, must be calculated.

Equation (1) may be rewritten:

$$V = \frac{F}{\rho g} \tag{2}$$

$$\frac{F}{g} = m \text{ (suspended mass)} \tag{3}$$

$$\therefore V = \frac{m}{\rho} \tag{4}$$

Where m is measured in weight units. Therefore, the volume of the specimen is known, and its density is calculated from

$$\rho_s = \frac{m_a}{V} \tag{5}$$

where ρ_s = specimen density
m_a = specimen mass in air

3.0 Specimen Preparation

3.1 A reasonable size and shape of specimen should be selected, based on equipment capabilities.

3.2 The selected specimen should be representative of the material being investigated.

3.3 The specimen should be clean.

4.0 Apparatus Components

4.1 An analytical balance capable of accuracy to within 0.1 percent of the specimen weight, and equipped with a suspension wire to support the specimen.

4.2 Immersion fluid in a suitable container. The fluid must be selected on the basis of its known density, and capability with the specimen material. For the geologic materials, toluene can generally be used.

4.3 A thermometer with accuracy appropriate to determine the fluid temperature to within ±2C.

5.0 Procedure

5.1 Weigh the specimen in static air.

5.2 Weigh the specimen when totally immersed in the fluid, suspended on a fine wire.

5.3 Note and record the fluid temperature.

6.0 Calculations

6.1 Determine the specimen volume from Eq. (4)

6.2 Calculate the specimen density from Eq. (5)

6.3 Make appropriate corrections in the above for the part of the suspension wire which is immersed.

7.0 Report

7.1 Give brief description of specimen, how prepared, etc.

7.2 Present results in suitable units.

7.3 Where appropriate, calculate the relation of density verses temperature. This involves utilization of linear thermal expansion data for the material, and the assumption that the material expands isotropically.

8.0 General Comments

8.1 It is to be emphasized that the procedures and information outlined in this specification are merely a guide to measurement of the indicated property, and as such should not be presumed to include all possible contingencies which might occur.

8.2 It is understood that the density measurement will be set up and carried out by someone who has an adequate background in thermal physics and related sciences, and is experienced in all phases of the procedures relating to this measurement.

8.3 This specification includes, by inference, the utilization of advanced technology and quality workmanship in all phases of the measurement. Employment of these procedures by untrained or inexperienced personnel might void the terms and spirit of this specification.

APPENDIX F

Thermal Conductivity Evaluation
of Pyroceram 9606

Energy Materials
Testing Laboratory

February 20, 1981

Oak Ridge National Laboratory
Union Carbide Corporation
Post Office Box X
Oak Ridge, Tenn. 37830

Attention: Marvin T. Morgan
Waste Isolation Group
Chemical Technology Division

Subject: Thermal Conductivity Evaluation of Pyroceram 9606

Dear Marvin:

Since our last communication, we have continued our efforts to determine more exactly the thermal conductivity of the Pyroceram 9606 material used in our comparative thermal conductivity measurement apparatus. This is especially important in relation to our work in support of the Battelle ONWI Program. I regret the delay in getting results of this work to you, but since the work could not be funded, we had to fit it in whenever possible.

Our investigation consisted of several phases, as follows:

1) A review of existing literature;

2) A review and re-evaluation of conductivity data measured as early as 1969 on our supply of Pyroceram 9606, which was purchased from the U. S. National Bureau of Standards, by Battelle;

3) A review of Battelle and literature data on the thermal diffusivity of Pyroceram 9606;

4) New thermal diffusivity measurements and specific heat measurements on samples of our NBS Pyroceram as fabricated in 1969 and on new samples cut recently;

5) Thermal diffusivity measurements on three samples of Pyroceram 9606 furnished by ORNL;

6) Comparison of all thermal diffusivity data measured by us and as reported in the literature, and steady-state measured at Battelle;

7) Derivation of thermal conductivity data from all available sources, including those of Item (6) above;

8) Comparison of all thermal conductivity data of Item (7) above, among themselves, and with values being used by ORNL as supplied with their measurement instrument by its manufacturer, Dynatech Corporation;

9) Derivation of equations for each set of our thermal diffusivity data and thermal conductivity data;

10) Derivation of a recommended equation, or equations, for the thermal conductivity-temperature relation of Pyroceram 9606, to be used on the ONWI and other programs.

The attached tables and figures present all of the information derived in this informal study. Comments on each follow:

Table 1 gives results of thermal diffusivity measurements and calculated thermal conductivity values for the original diffusivity specimen cut from the original supply in 1969. It also gives equations for both diffusivity and conductivity curves versus temperatures, and correlation coefficients qualifying the data fit. Finally, it gives pertinent specimen details.

Tables 2 and 3 give corresponding data for two new specimens, recently fabricated from the original Battelle-NBS supply.

Tables 4, 5, and 6 give measured thermal diffusivity and derived thermal conductivity data for three samples furnished by ORNL. The ORNL #5 (Table 4) disk is from a ½" plate from which the ORNL reference meters were cut. According to your information, this plate was purchased from Corning Glass, Inc., and its conductivity averaged 5% higher than the original meters furnished with your Dynatech instrument. ORNL disks #2 and #3 were cut from a separate 1" disk, by ORNL. Note that the bulk density of ORNL #5 is 2.54 g cm^{-3}, while that of the other two ORNL disks, and the two recent FMI disks (Tables 2 and 3), is 2.59 g cm^{-3} in each case. Correspondingly, the derived conductivity of #5 is slightly lower than that of the others.

Table 7 is a compilation of the thermal diffusivity data of Tables 1-6, presented for comparative purposes at nominal temperatures. Each value was derived from the equation assigned to diffusivity data on the individual tables. In addition, this table includes data on Pyroceram 9606 by Rudkin and by Gibby, from the cited literature. For these latter two, we fitted equations to their data, to evaluate points at the indicated temperatures. Note that all of the values at a given temperature are very similar. Mean values are listed for each temperature ; the low values of standard deviations and variances among all the values at a given temperature attest to the close agreements. Finally, Table 7 gives bulk densities of all diffusivity specimens measured by us.

Figure 1 is a plot of actual data points of thermal diffusivity for all of the eight specimens identified on Table 7. Note that all points, including those by Rudkin and Gibby, are closely enough grouped to describe a curve of the property vs. temperature with high confidence. There appears to be no doubt that the thermal diffusivity values as measured are accurate, and that derivation of thermal conductivity from these data, using the definitive relationship

$$\alpha = \frac{\lambda}{\rho C_p}$$

(where λ is thermal conductivity, α is thermal diffusivity, ρ is density, and C_p is specific heat), is justified if

specific heat and density are well known. Density can be measured accurately without difficulty.

Figure 2 shows the specific heat curve for Pyroceram 9606 that we used in calculating thermal conductivity values from diffusivity data. This curve is taken from literature by Corning. We justify its use on the basis of our own specific heat measurements on a sample of our Pyroceram. These values, derived from enthalpy measurements in a Bunsen-type ice calorimeter, are illustrated as an X on Figure 2. Obviously, the agreement with the Corning curve justifies use of the latter for this study.

Table 8 is a set of steady-state data on the BMI-NBS Pyroceram, as measured at Battelle early in the 1970s. In this case, the measurements were made using a Type 347 Stainless Steel reference standard. Although this is not an ideal material to use as a standard when evaluating a ceramic, it was selected because its conductivity has been well established, and because of its stability and availability. Very few materials satisfy these requirements.

Table 9 presents a summation of thermal conductivity values from all our measurements, including the steady-state measurements of Table 8. Here again, the values are given at nominal temperatures, and were determined in each case from the equations which appear to best fit the tabulated data. Mean values for each temperature provide the basis for a recommended curve of conductivity vs. temperature, and the small standard deviations and variances demonstrate how closely all of the sets of data fit the mean.

Figure 3 is a plot of the seven sets of conductivity data presented in Tables 1-6 and 8. Again, these are actual data, as derived from individual thermal diffusivity measurements for the Tables 1-6 specimens. The similarity of all data sets tends to establish confidence in each.

A significant result of this entire study is that all of the seven sets of data generated by Battelle and the EMTL Division of Fiber Materials, and the two sets generated by Rudkin and by Gibby, involving at least five different sources or lots of material, provide results that are in good agreement with each other. Further, when compared

with conductivity values being used by ORNL, as furnished by Dynatech, these values are all significantly lower. As indicated in the final two columns of Table 9, the ORNL/Dynatech values of conductivity for Pyroceram 9606 are 5 to 10 percent higher than the consensus of all data generated and/or collected in this study. The obvious consequence is that conductivity values generated on unknown materials by comparison with this Pyroceram will be similarly affected.

Figure 4 compares the three thermal conductivity curves for Pyroceram 9606, involved in this study. The lower solid curve represents the consensus of all data generated at BMI and at FMI, and by Rudkin and Gibby, if we presume they used specific heat values close to those of Figure 2. The upper solid curve represents values being used by ORNL. The shorter, dashed curve represents values which we have used to date in evaluating the ONWI materials.

We have generated tentative equations for the recommended (lower) curve of Figure 4. To assure reasonably good fits to the data, the curve was segmented into three temperature ranges. The equations are:

1. $\lambda = A+B \ln T$

   where A = 0.0417476
   B = -0.0018029
   T = deg C, 0 to 300

2. $\lambda = Ae^{BT}$

   where A = o.o334745
   B = -0.00023175
   T = deg C, 300 to 700

3. $\lambda = Ae^{BT}$

   where A = 0.0325836
   B = -0.0001913
   T = deg C, 700 to ~1000

The net result of this study is that we have renewed confidence in our initial evaluations of Pyroceram 9606 thermal conductivity, and therefore that thermal conductivity values reported by us on the ONWI materials are reliable.

During our last discussion, you indicated that ORNL was measuring thermal diffusivity on some of the same Pyroceram we have investigated here. I will be grateful to know results of these measurements.

After you have had a chance to study this work, call and let me have your comments. I will send a copy of this to Gil Raines for his information.

Sincerely,

John

John F. Lagedrost
Manager
ThermoPhysics Lab

JFL/j

TABLE 1

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: Pyroceram 9606 (Original Standard, 1969)

Temperature C	Diffusivity $cm^2 s^{-1}$	Specific heat cal g^{-1} C^{-1}	Conductivity w cm^{-1} C^{-1}
19	0.0185	0.181	0.0362
113	0.0146	0.216	0.0342
55	0.0164	0.198	0.0351
149	0.0136	0.223	0.0327
198	0.0131	0.231	0.0327
290	0.0119	0.244	0.0314
406	0.0111	0.259	0.0311

$\alpha = 0.0036286 - 0.0024 \ln(Tc/10^4)$ $\lambda = 0.0255678 - 0.0017656 \ln(Tc/10^4)$

cc = 0.9913 cc = 0.947

Specimen details:

RT Thickness, in.	0.0374
RT Diameter	0.4998
Weight, g	0.3098
Density, g cm^{-3}	2.5765

TABLE 2

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: BMI Pyroceram 9606

Temperature C	Diffusivity, $cm^2 s^{-1}$	Specific heat, cal g^{-1} C^{-1}	Conductivity, w cm^{-1} C^{-1}
18	0.01800	0.181	0.03543
52	0.01656	0.196	0.03529
98	0.01525	0.211	0.03499
153	0.01393	0.223	0.03378
202	0.01312	0.232	0.03308
305	0.01198	0.247	0.03216
403	0.01095	0.258	0.03072
511	0.01016	0.269	0.02970
603	0.00961	0.277	0.02895
698	0.00924	0.284	0.02854

$\alpha = 0.00289 - 0.00253 \ln(Tc/10^4)$ $\lambda = 0.0357e^{-3.44\ (Tc/10^4)}$

cc = 0.974 cc = 0.988

Specimen details:

RT Thickness, in.	0.0402
Density, g cm^{-3}	2.5972

TABLE 3

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: BMI Pyroceram 9606

Temperature C	Diffusivity $cm^2\ s^{-1}$	Specific heat $cal\ g^{-1}\ C^{-1}$	Conductivity $w\ cm^{-1}\ C^{-1}$
28	0.01767	0.187	0.03589
46	0.01695	0.194	0.03571
101	0.01525	0.212	0.03512
146	0.01421	0.222	0.03425
208	0.01306	0.233	0.03303
302	0.01162	0.246	0.03105
411	0.01067	0.259	0.03000
504	0.01004	0.268	0.02923
618	0.00965	0.278	0.02913
720	0.00929	0.286	0.02885

$\alpha = 0.002095 - 0.00275 \ln(Tc/10^4)$ cc = 0.987

$\lambda = 0.03579 e^{-3.518(Tc/10^4)}$ cc = 0.928

Specimen details:

RT Thickness, in.	0.0402
Density, g cm^{-3}	2.5941

TABLE 4

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: ORNL Pyroceram 9606 #5

Temperature C	Diffusivity $cm^2 s^{-1}$	Specific heat, cal g^{-1} C^{-1}	Conductivity, w cm^{-1} C^{-1}
18	0.01830	0.181	0.03530
52	0.01660	0.196	0.03467
98	0.01503	0.211	0.03378
147	0.01373	0.222	0.03247
200	0.01253	0.232	0.03097
309	0.01136	0.247	0.02989
411	0.01043	0.259	0.02877
525	0.00997	0.270	0.02868
613	0.00955	0.278	0.02827
701	0.00914	0.284	0.02766

$\alpha = 0.002228 - 0.00265 \ln (Tc/10^4)$ $\quad$ $\lambda = 0.02194 - 0.00231 \ln (Tc/10^4)$

cc = 0.987 $\quad$ cc = 0.940

Specimen details:

RT Thickness, in.	0.0399
Density, g cm^{-3}	2.5452

TABLE 5

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: ORNL Pyroceram 9606 #2

Temperature C	Diffusivity cm s^{-1}	Specific heat, cal g^{-1} C^{-1}	Conductivity, w cm^{-1} C^{-1}
33	0.01716	0.188	0.03499
47	0.01643	0.197	0.03512
103	0.01451	0.212	0.03337
149	0.01350	0.222	0.03253
200	0.01275	0.232	0.03208
312	0.01161	0.248	0.03123
412	0.01072	0.259	0.03012
509	0.01004	0.269	0.02930
607	0.00951	0.277	0.02857
705	0.00918	0.284	0.02828

$\alpha = 0.00225 - 0.00265 \ln(Tc/10^4)$ $\quad$ $\lambda = 0.022646 - 0.00229 \ln(Tc/10^4)$

cc = 0.997 $\quad$ cc = 0.967

Specimen details:

RT thickness, in.	0.0390
Density, g cm^{-3}	2.5917

TABLE 6

THERMAL DIFFUSIVITY/CONDUCTIVITY

Program: Conductivity Standards
Billet No.: ORNL Pyroceram 9606 #3

Temperature C	Diffusivity $cm^2 s^{-1}$	Specific heat, cal g^{-1} C^{-1}	Conductivity, w $cm^{-1} C^{-1}$
18	0.01792	0.181	0.03520
24	0.01759	0.184	0.03513
47	0.01653	0.194	0.03480
97	0.01487	0.210	0.03388
149	0.01370	0.222	0.03300
196	0.01279	0.231	0.03205
304	0.01162	0.247	0.03115
409	0.01077	0.259	0.03028
506	0.01026	0.268	0.02982
612	0.00972	0.278	0.02933
713	0.00934	0.285	0.02890

$\alpha = 0.003139 - 0.002425 \ln(Tc/10^4)$ cc = 0.990

$\lambda = 0.0348e^{-2.9365(Tc/10^4)}$ cc = 0.947

Specimen details:

RT Thickness, in.	0.0327
Density, g cm^{-3}	2.5922

TABLE 7

THERMAL DIFFUSIVITY OF PYROCERAM 9606

Temperature C	Thermal Diffusivity, cm^2s^{-1}, $x10^{-3}$								Mean, $x10^{-3}$	Std. Dev., $x10^{-3}$	Var., $x10^{-6}$
	BMI 1969	EMTL 586	EMTL 587	ORNL #5 588	ORNL #2 589	ORNL #3 590	Rudkin (1)	Gibby (2)			
20	18.54	18.63	19.19	18.69	18.71	18.21	18.18	19.44	18.70	0.436	0.166
100	14.68	14.56	14.76	14.43	14.45	14.31	14.38	14.89	14.56	0.203	0.036
200	13.02	12.80	12.85	12.59	12.62	12.63	12.75	12.93	12.77	0.156	0.021
300	12.04	11.77	11.74	11.52	11.54	11.64	11.79	11.79	11.73	0.166	0.024
400	11.35	11.05	10.95	10.76	10.78	10.94	11.11	10.97	10.99	0.189	0.031
500	10.82	10.48	10.33	10.17	10.19	10.40	10.59	10.34	10.42	0.215	0.040
600	10.38	10.02	9.83	9.68	9.71	9.96	10.16	9.83	9.95	0.237	0.049
700	10.01	9.63	9.41	9.28	9.30	9.59	9.79	9.39	9.55	0.256	0.057
800	9.69	9.29	9.04	8.92	8.94	9.26	9.48	9.02	9.21	0.276	0.067
900	9.41	8.99	8.72	8.61	8.63	8.98	9.20	8.68	8.90	0.293	0.075
1000	9.15	8.72	8.43	8.33	8.35	8.72	8.95	8.39	8.63	0.306	0.082
Density, g cm^{-3}	2.577	2.597	2.594	2.545	2.592	2.592					

(1) Rudkin, R. L., "Thermal Diffusivity Measurements on Metals and Ceramics at High Temperatures," USAF Report No. ASD-TDR-62-24, Part II, 1-16 (1963).

(2) Gibby, R. L., "The Thermal Diffusivity and Thermal Conductivity of Stoichiometric $(U_{0.8}Pu_{0.2})O_2$," Pacific Northwest Laboratory Report BNWL-704, 1-39 (1968).

TABLE 8

THERMAL CONDUCTIVITY

Program: Conductivity Standards
Billet No.: BMI Pyroceram 9606

Measurement by Steady-State Comparative,
Using Type 347 Stainless Steel

Temperature, C	Conductivity w cm^{-1} C^{-1}
213	0.0315
130	0.0338
284	0.0312
225	0.0319
168	0.0331
349	0.0301
274	0.0307
201	0.0318
413	0.0306
104	0.0332
86	0.0351
69	0.0346
359	0.0308
386	0.0298
220	0.0327

TABLE 9. THERMAL CONDUCTIVITY OF PYROCERAM 9606

Temp C	From S-S (BMI)	Conductivity, w $cm^{-1}C^{-1}$ From Thermal Diffusivity						Mean	Std. Dev., $x10^{-3}$	Var., $x10^{-6}$	ORNL/ Dynatech Values	Dev. from Mean Percent (+)
		BMI 1969	EMTL 586	EMTL 587	ORNL #5 588	ORNL #2 589	ORNL #3 590					
20	0.03850	0.03654	0.03547	0.03554	0.03630	0.03690	0.03460	0.03626	1.252	1.344	0.04014	10.70
100	0.03404	0.03370	0.03451	0.03455	0.03161	0.03319	0.03379	0.03363	1.008	0.871	0.03716	10.50
200	0.03212	0.03247	0.03334	0.03336	0.03016	0.03160	0.03282	0.03227	1.125	1.086	0.03495	8.30
300	0.03099	0.03176	0.03221	0.03220	0.02930	0.03068	0.03187	0.03129	1.053	0.951	0.03340	6.74
400	0.03019	0.03125	0.03112	0.03109	0.02870	0.03002	0.03094	0.03047	0.917	0.721	0.03219	5.64
500	0.02957	0.03086	0.03007	0.03002	0.02823	0.02951	0.03005	0.02976	0.806	0.557	0.03120	4.84
600	0.02907	0.03054	0.02905	0.02898	0.02785	0.02909	0.02918	0.02911	0.781	0.523	0.03045	4.60
700	0.02864	0.03026	0.02807	0.02798	0.02752	0.02874	0.02834	0.02851	0.877	0.659	0.02980	4.52
800	0.02827	0.03003	0.02712	0.02701	0.02724	0.02843	0.02752	0.02795	1.011	0.990	0.02921	4.51
900	0.02794	0.02982	0.02620	0.02608	0.02700	0.02816	0.02672	0.02742	1.325	1.504	0.0288	5.03
1000	0.02765	0.02963	0.02532	0.02517	0.02678	0.02792	0.02595	0.02692	1.605	2.209	0.0285	5.87

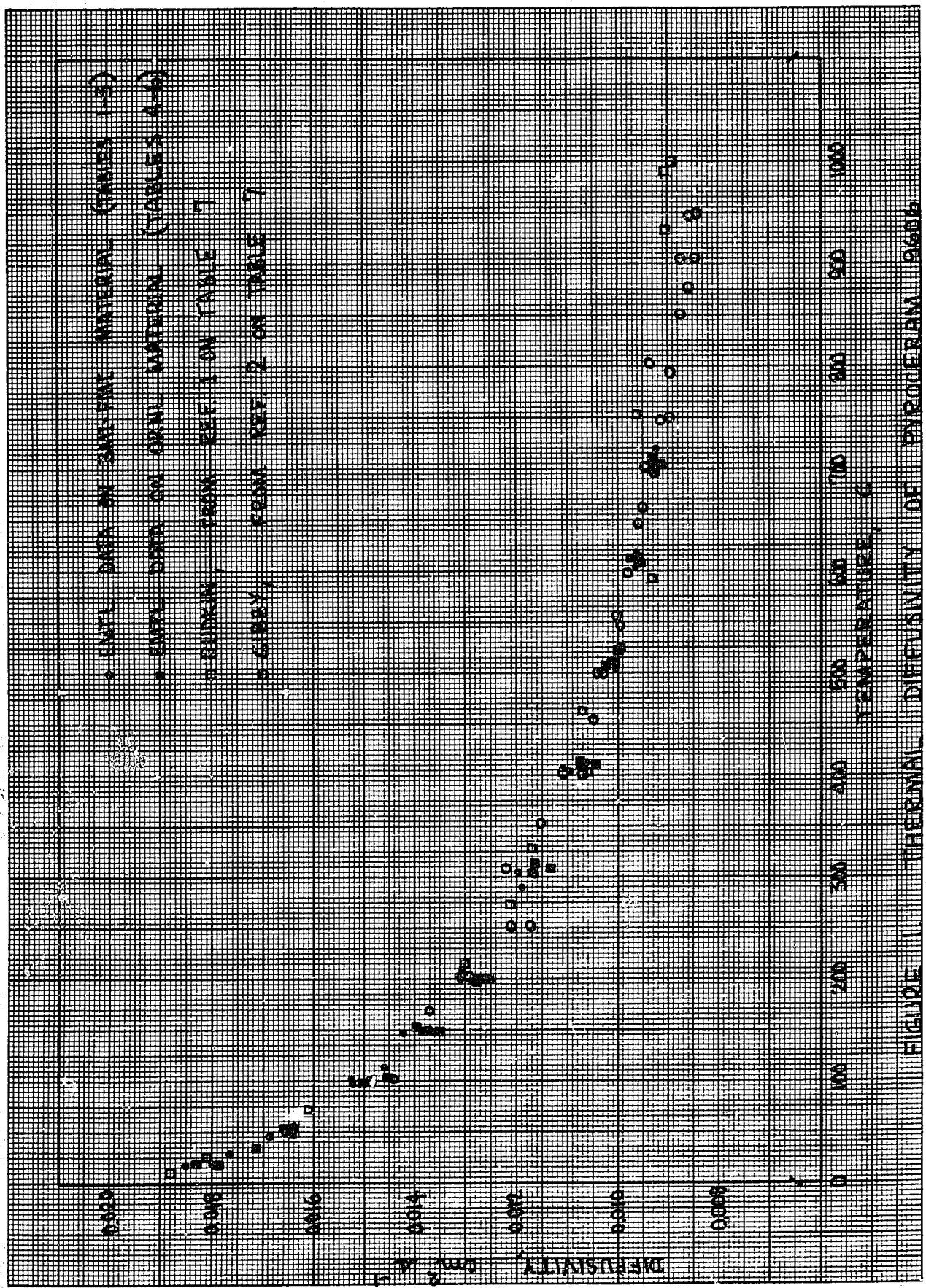
FIGURE II THERMAL DIFFUSIVITY OF PYROCERAM 9606
TEMPERATURE °C
DIFFUSIVITY
cm² s⁻¹
0.006
0.008
0.010
0.012
0.014
0.016
0.018
0.020
0.022
0.024
0.026
0.028
0.030
0.032
0.034
0.036
0.038
0.040
0.042
0.044
0.046
0.048
0.050
0.052
0.054
0.056
0.058
0.060
0.062
0.064
0.066
0.068
0.070
0.072
0.074
0.076
0.078
0.080
0
100
200
300
400
500
600
700
800
900
1000
EMRL DATA ON SAME MATERIAL (CARES 1963)
EMRL DATA ON OTHER GLASS CERAMIC MATERIALS (CARES 1963)
CORNING 9606 (CARES 1963)
CORNING 9606 (PRESENT WORK)

FIGURE 2. SPECIFIC HEAT OF PYROCERAM 9606

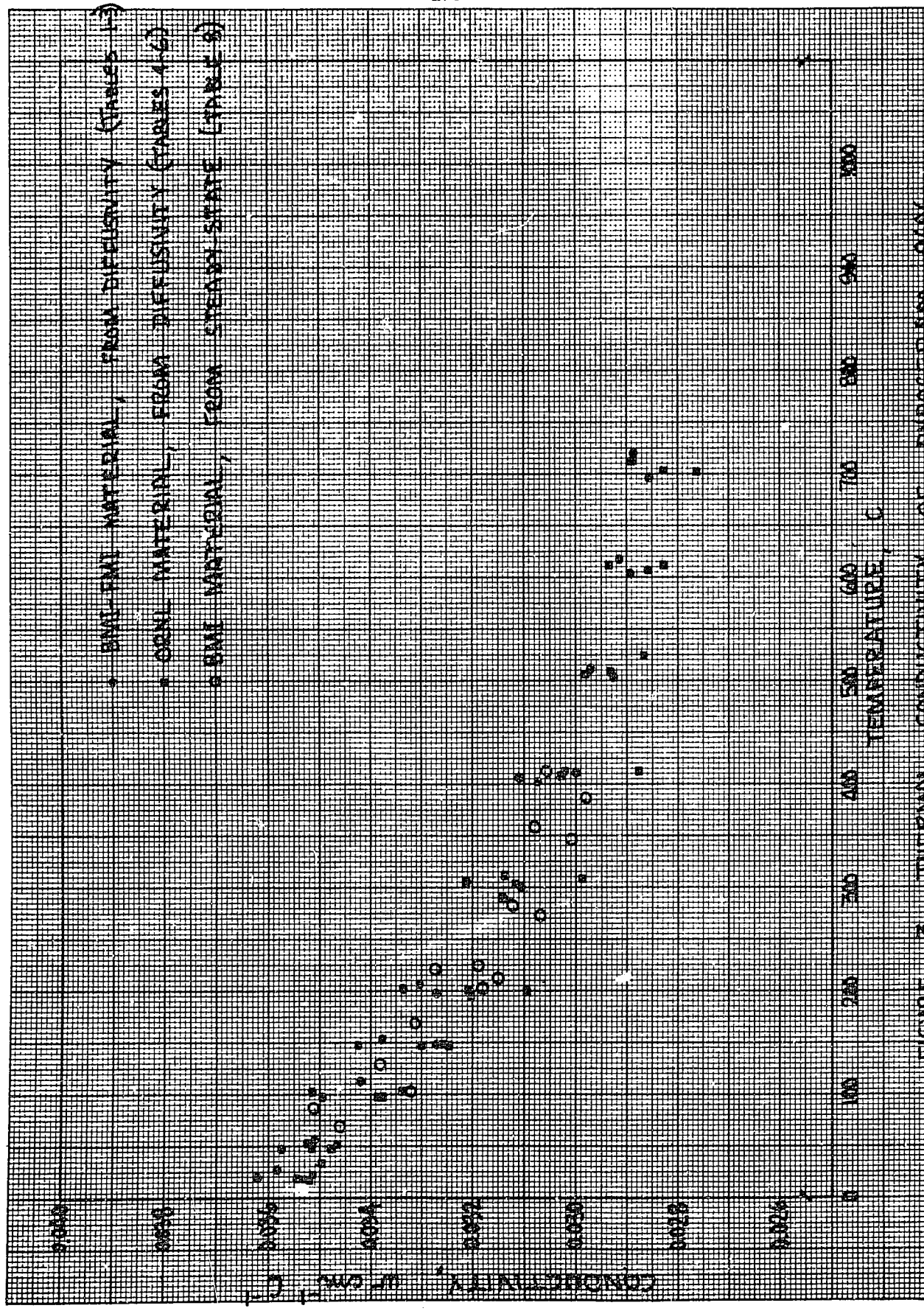
BAND GAP MATERIAL FROM DIFFUSIVITY (Table 5)
BAND GAP MATERIAL FROM RESISTIVITY (Table 4)
BAND GAP MATERIAL FROM THERMOPOWER (Table 3)
CONDUCTIVITY, Ω⁻¹ cm⁻¹
THERMAL CONDUCTIVITY, W cm⁻¹ °C⁻¹
TEMPERATURE, °C
THERMAL CONDUCTIVITY OF PYROCERAM 9606
FIGURE 5

FIGURE 4 THERMAL CONDUCTIVITY OF 2%DYCERAM 9606

APPENDIX G

Comparison of Laboratory Measured
Thermal Conductivity With
Recommended Values for Fused Silica

APPENDIX G. Comparison of Thermal Conductivity Data from Laboratory Measurements with "Recommended" Values from the Literature[1] For Fused Silica

Temperature K	Temperature C	Recommended Conductivity w cm-1C-1	Measured Conductivity[2] w cm-1C-1	Difference %
273	0	0.0133	0.0133	0
300	27	0.0138	0.0138	0
350	77	0.0145	0.0146	0.7
400	127	0.0151	0.0155	2.7
450	177	0.0157	0.0164	4.4
500	227	0.0162	0.0172	5.9
600	327	0.0175	0.0189	7.8
700	427	0.0192	0.0207	7.5
800	527	0.0217	0.0225	3.6

(1) See Reference 5.

(2) Measured values were interpolated to temperature using linear regression fit of laboratory data.

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