Optical Adhesive Property Study

Kansas City Division

P. D. Sundvold

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Topical Report

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OPTICAL ADHESIVE PROPERTY STUDY

P. D. Sundvold

Published January 1996

Topical Report

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Abstract

Tests were performed to characterize the mechanical and thermal properties of selected optical adhesives to identify the most likely candidate which could survive the operating environment of the Direct Optical Initiation (DOI) program. The DOI system consists of a high power laser and an optical module used to split the beam into a number of channels to initiate the system. The DOI requirements are for a high shock environment which current military optical systems do not operate. Five candidate adhesives were selected and evaluated using standardized test methods to determine the adhesives' physical properties. EC2216, manufactured by 3M, was selected as the baseline candidate adhesive based on the test results of the physical properties.

Summary

Tests were performed to characterize the mechanical and thermal properties of selected optical adhesives to identify the most likely candidate which could survive the operating environment of the Direct Optical Initiation (DOI) program. The DOI system consists of a high power laser and an optical module used to split the beam into a number of channels to initiate the system. The DOI system is designed for a high shock and extreme thermal environment which existing military optical packages currently do not operate. To survive the environment, the optical elements making up the system will need to be bonded into mounts. Mechanical mounting could induce localized high stress areas, causing the optical element to fracture.

Five candidate adhesives were selected and evaluated using standardized test methods to determine the adhesives' physical properties. EC2216, manufactured by 3M, was selected as the baseline adhesive based on the test results of the physical properties. It had good tensile properties, compression, and lap shear strength; and the coefficient of thermal expansion matches well with Invar mounts and fused silica optics.

DISCUSSION

SCOPE AND PURPOSE

This work was performed to characterize and identify a probable optical adhesive to be used on the Direct Optical Initiation (DOI) firing system. The DOI firing system uses a laser to generate a high energy beam which is then split into numerous channels, focused into fibers, and then used to launch optical slapper detonators. This study was to characterize adhesives based on standard test methods and use the results as a basis for selecting the most likely candidate for the DOI optical adhesive. This will also help identify tests which may be most pertinent for future quality acceptance criteria.

Three adhesives (one with three different mix ratios) were chosen as likely candidates. These adhesives were subjected to 11 different tests measuring 18 parameters. The tests encompassed both mechanical and thermal property measurements.

ACTIVITY

Background

The DOI firing system is being designed to withstand current and proposed weapon environments and, as such, must withstand thermal environments of -55°C to 75°C and mechanical shock environments of 3000 g's. The laser and other optical systems will use adhesives to bond the optical elements to ruggedized mounts to maintain the required alignments. Current military applications do not cover all the environmental conditions or have the lifetime requirements necessary for nuclear weapon applications.

Three adhesives were chosen as probable candidates based on previous use in military optical systems. These were A12, manufactured by Armstrong Products Company; EC2216 (gray), manufactured by 3M Industrial Tape and Specialties Division; and EA9309.3, manufactured by Aerospace Adhesives and Structural Materials. The A12 was tested in three different mix ratios since it had been used that way in previous military optical systems. The other two were mixed in accordance with manufacturer's recommendations.

Ten different tests were identified to characterize the adhesives. Table 1 lists the test method and the properties determined from that test and identifies an Appendix which contains information for that particular test. Upon completion of a test, a report was generated and distributed. These reports contain description of the purpose of the test, background, cure schedule, test procedure, and results along with copies of the ASTM or other standard. These reports are included as Appendices.

Table 1. Summary of Testing Methods

<u>Test Method</u>	<u>Properties Determined</u>	<u>Appendix</u>
1) ASTM D 651	Tensile Strength @ Temp Modulus of Elasticity @ Temp Percent Elongation @ Temp	B
2) ASTM D 695	Compressive Strength @ Temp Compressive Moduli @ Temp	C
3) ASTM D1002	Lap Shear on AI @ Temp	D
4) Rheometric Mechanical Spectrometry (RMS)	Shear Moduli Glass Transition Temp	F
5) Dynamic Mechanical Analysis (DMA)	Tensile Moduli Glass Transition Temp	G
6) Thermogravimetric Analysis (TGA)	Thermal Stability Weight Loss	H
7) Differential Scanning Calorimeter (DSC)	Kinetics of Cure Specific Heat Vs Temp	I
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9) ASTM E 1225	Thermal Conductivity	J
10) ASTM D 792	Specific Gravity	K

Once the testing was completed, the data was analyzed to determine the optimal adhesive which would provide the best properties for the thermal and mechanical environments identified for DOI.

Additional testing to try to determine Poisson ratio at room temperature was later performed on the EC2216 to generate data for finite analysis of the mounted optics.

Results

After review of the data, it was determined that EC2216 had the best properties to survive the environments for DOI without affecting the optical system. Table 2 shows a summary of the results of the testing for the EC2216.

Table 2. Properties of EC2216

<u>Property</u>	<u>-65°F</u>	<u>Room Temperature</u>	<u>165°F</u>
Tensile Strength (psi)	7,340	2,690	610
Tensile Moduli (psi)	838,000	10,100	5,140
Elongation (%)	1.04	35	11.9
Comp. Prop. Limit (psi)	9,450	286	397
Ult. Comp. Strain (psi)	16,900	1,960	625
Ult. Comp. Stress (%)	4.61	10	10
Comp. Moduli (psi)	905,000	53,900	6,060
Lap Shear (psi)	2,480	3,520	865

Poisson Ratio (room temperature): 0.592

Coefficient of Thermal Expansion: Below Tg: 39.4×10^{-6} in/in°F

(Tg = 84.9°F)

Above Tg: 105.6×10^{-6} in/in°F

RMS Glass Transition Temperature: 102.2°F

DMA Glass Transition Temperature: 69.4°F

Thermal Decomposition Temperature: 651.9°F

Residue (percent filler) 30.3

Heat of Reaction: 164.6 J/gm

Activation Energy: 51.1 Kj/mole

Thermal conductivity (at 20°C): 0.2468 BTU/hr-ft-°F

Linear Shrinkage: 1.40%

Volume Shrinkage: 4.06%

Density: 0.0493 lb/in³

Specific Gravity: 1.366

ACCOMPLISHMENTS

Five candidate optical adhesives were tested in accordance with standard test methods to determine which adhesive had the properties required to hold mounted optics to survive DOI environments. These tests concluded that EC2216, manufactured by 3M, is the leading choice for the DOI application. It has good strength and expansion properties through the temperature extremes. The properties of EC2216 will enable properly designed bonded, mounted optics to survive 3000-g shock environments and temperature extremes of -55°C to 75°C. Test method to ensure compliant material could be the lap shear test since it is not dependent upon forming

parts made of the adhesive and then testing. The lap shear test looks at both the bonding to a substrate and the properties of the material.

FUTURE WORK

The results of these tests indicate that optical systems can be designed to withstand weapon environments using EC2216. The effects of a thirty-year shelf life are unknown. Existing military systems using adhesives to bond optics do not have the long life requirements, and, thus, there is no data to the effect of long-term aging. The next phase will be an accelerated aging study over two years. Both EC2216 and the 2:3 mix ratio A12 will be tested due to the length of the aging study. This will provide a backup adhesive if the EC2216 fails to perform adequately in the other tests.

Appendix A

Mix Ratio and Cure Schedules

Mix Ratio and Cure Schedule

The optical adhesive test samples were mixed and cured per Table A-1 for all tests. The Kinetics of Cure test samples were cured using different schedules. The cure schedule for those tests are defined in Appendix I,

Table A-1. Mix Ratio and Cure Schedule

Adhesive	Mix Ratio*		Cure Schedule
	A Comp.	B Comp.	
A12 1:1	1	1	24 hrs @ room temp., 2 hrs @ 160°F
A12 1:2	1	2	24 hrs @ room temp., 2 hrs @ 160°F
A12 2:3	2	3	24 hrs @ room temp., 2 hrs @ 160°F
EC2216 (Gray)	1.4	1	16 hrs @ room temp., 16 hrs @ 160°F
EA9309.3NA	10	2.2	7 days at room temperature

* Material was mixed based on weight, i.e., 10:2.2 would be mixed using 10 grams of A component mixed with 2.2 grams of B component or some factor thereof.

Appendix B

Tensile Strength

Tensile Strength

Test Purpose:

The purpose of this test is to determine the tensile strength, modulus of elasticity, and percent elongation over temperature of the optical adhesives .

Background:

The testing is per ASTM D 651 and is included in Appendix L.

Sample Preparation:

Fifteen samples of each material were mixed and cured per Table A-1 in Appendix A. After curing, the sample's length and width were measured and then the top and bottom surfaces were machined to 0.125 inch final thickness.

Test Procedure:

Testing is per ASTM D 651 and is included in Appendix L.

Test Results:

The following tables lists the average and standard deviation (s) for the tensile strength, tensile moduli, and percent elongation. Table B-1 shows the results at -65°F, Table B-2 is at room temperature, and Table B-3 is at 165°F. Five samples of each material were tested at each temperature.

Table B-1: Tensile Results at -65°F

Material	Tensile Strength		Tensile Moduli		Elongation	
	(psi)	s	(psi)	s	(%)	s
A12 1:1	8,540	1280	549,000	50,900	1.56	0.50
A12 2:3	8,490	2190	576,000	69,000	1.68	0.73
A12 1:2	7,840	1870	500,000	80,000	2.00	0.69
EC 2216 (gray)	7,370	1090	838,000	119,000	1.04	0.18
EA 9309.3 NA	7,410	1010	390,000	51,500	2.17	0.39

Table B-2: Tensile Results at Room Temperature

Material	Tensile Strength		Tensile Moduli		Elongation	
	(psi)	s	(psi)	s	(%)	s
A12 1:1	6,390	437	411,000	52,000	2.57	0.72
A12 2:3	3,250	419	191,000	31,000	13.1	7.3
A12 1:2	1,190	85	2,390	344	87	7.6
EC 2216 (gray)	2,690	356	10,100	3,630	35	5.0
EA 9309.3 NA	5,620	167	363,000	150,000	2.8	0.67

Table B-3: Tensile Results at 165°F

Material	Tensile Strength		Tensile Moduli		Elongation	
	(psi)	s	(psi)	s	(%)	s
A12 1:1	893	134	7,320	1,980	16.0	2.0
A12 2:3	263	29	1,500	23	17.8	2.1
A12 1:2	135	25	844	74	16.7	3.6
EC 2216 (gray)	610	66	5,140	112	11.9	1.1
EA 9309.3 NA	1,280	184	89,500	46,600	21.7	4.7

Appendix C

Compressive Strength

Compressive Strength

Test Purpose:

The purpose of this test is to determine the compressive strength and compressive moduli over temperature of the optical adhesives.

Background:

The testing is per ASTM D 695 and is included in Appendix L.

Sample Preparation:

Fifteen samples, 0.5" x 0.5" x 1.0" long, were mixed and cured per Table A-1 in Appendix A. After curing, the samples were machined flat.

Test Procedure:

The test procedure was per ASTM D 695 and is included in Appendix L.

Test Results:

The following tables list the average and standard deviation (s) for the proportional limit, ultimate compressive stress and strain, and the compressive moduli. Table C-1 shows the results at -65°F, Table C-2 is at room temperature, and Table C-3 is at 165°F.

Table C-1: Compressive Results at -65°F

Material	Proportional Limit		Ultimate Compressive				Compressive Moduli	
	(psi)	s	Stress		Strain		(psi)	s
			(psi)	s	(%)	s		
A12 1:1	11,040	783	19,100	352	6.17	.21	673,000	18,400
A12 2:3	9,600	196	16,600	195	5.60	.32	616,000	6,630
A12 1:2	8,000	450	13,700	311	5.24	.26	520,000	15,700
EC 2216 (gray)	9,450	286	16,900	207	4.61	.35	905,000	16,700
EA 9309.3 NA	7,540	309	16,900	523	8.92	.64	439,000	11,900

Table C-2: Compressive Results at Room Temperature

Material	Proportional Limit		Ultimate Compressive				Compressive Moduli	
	(psi)	s	Stress (psi)		Strain (%)		(psi)	s
A12 1:1	5,150	106	9,140	221	3.63	.01	446,000	16,200
A12 2:3	2,740	200	4,120	197	4.36	.22	180,000	15,900
A12 1:2	236	50	461	25	10.0		10,500	999
EC 2216 (gray)	286	26	1,960	65	10.0		53,900	8510
EA 9309.3 NA	4,650	383	7,710	153	4.24	.24	338,000	6460

Table C-3: Compressive Results at 165°F.

Material	Proportional Limit		Ultimate Compressive				Compressive Moduli	
	(psi)	s	Stress (psi)		Strain (%)		(psi)	s
A12 1:1	470	44	751	16	10.0		7,220	161
A12 2:3	112	7.3	183	4.1	10.0		1,770	46.6
A12 1:2	54	3.5	97.3	.80	10.0		948	11.7
EC 2216 (gray)	397	12	625	13	10.0		6,060	138
EA 9309.3 NA	390	59	1,230	67	10.0		44,800	7270

Appendix D

Lap Shear

Lap Shear

Test Purpose:

The purpose of this test is to determine the lap shear strength of the optical adhesives on aluminum test samples .

Background:

The testing is per ASTM D 1002 and is included in Appendix L.

Sample Preparation:

Fifteen samples of each adhesive were mixed, applied, and cured per Table A-1 in Appendix A.

The surfaces of the aluminum plates were etched per 9916023 (included in Appendix L) prior to the application of the adhesive. This process is as follows:

- a) Vapor degrease in 1,1,1-Trichloroethylene per 9906006 (included in Appendix L)
- b) Dry
- c) Etch (in Sodium dichromate, sulfuric acid and water solution at 150°F for 10 minutes per 9906010 - included in Appendix L)
- d) Rinse in deionized (DI) water
- e) Dry

Test Procedure:

Five samples of each adhesive were prepared and tested in accordance with ASTM D 1002 and are included in Appendix L.

Test Results:

Table D-1 shows the average lap shear strength and the standard deviation (s) for -65°F, room temperature, and 165°F.

Table D-1: Lap Shear Results

Material	Lap Shear Strength					
	-65°F (-54°C)		R.T.		165°F (74°C)	
	(psi)	s	(psi)	s	(psi)	s
A12 1:1	2,710	310	3,460	382	1,400	72.2
A12 2:3	3,760	449	3,140	82.5	334	9.5
A12 1:2	2,880	389	1,720	22.8	220	9.1
EC 2216 (gray)	2,480	305	3,520	83.7	865	233
EA 9309.3 NA	6,390	795	4,530	192	2,100	10.1

Appendix E

Coefficient of Thermal Expansion

Coefficient of Thermal Expansion

Test Purpose:

This test will determine the glass transition temperature (T_g) and coefficient of thermal expansion (CTE) above and below the T_g .

Background:

Thermal mechanical analysis (TMA) is a technique in which deformation of a substance is measured under a static load or a function of temperature as in this case. TMA is used to determine glass transition temperature (T_g) and coefficient of thermal expansion (CTE) above and below the T_g .

The setup uses a DuPont 9900 controller with a 943 TMA which uses a linear variable differential transformer (LVDT) to detect changes in sample dimensions during heating. The quartz probe, which contains the core of the LVDT, is placed in contact with the sample. As probe displacement occurs, due to changes in the sample dimension, a movement of the LVDT core results in a linear voltage output proportional to probe displacement.

Sample Preparation:

One sample approximately 0.250" diameter x 0.3" long was made and tested from each adhesive. The samples were mixed and cured per Table A-1 in Appendix A.

Test Procedure:

The samples were tested with a TA instrument TMA using an expansion probe with 1 gram tray weight in air from -60°C to 80°C at 5°C per minute then back to room temperature and up to 80°C at 5°C per minute.

Test Results:

Figures E-1 thru E-5 show the dimensional change as a function of temperature. The coefficient of thermal expansion (representative by α) is determined by the slope of the curve. There are two distinct slopes and intersection of the two lines gives the glass transition temperature (a more accurate way to determine glass transition temperature is Rheometric Mechanical Spectrometry and is discussed in Appendix F). Table E-1 shows the glass transition temperature (T_g) and the coefficient of thermal expansion (CTE) below and above the T_g .

Figures E-6 thru E-10 are also plots of the dimensional change as a function of temperature. In this case the material is allowed to cool to room temperature and then reheated. As can be seen from the curves, there is a definite shift between the heating and cooling phases although the slope of the curves remain similar. There is a fair amount of stress relaxation in the EA9309.3 NA material as evident in Figure E-10.

Table E-1: Coefficient of Thermal Expansion (CTE)

Material	Glass Transition Temperature		CTE below T _g		CTE above T _g	
	(°F)	(°C)	A	B	A	B
A12 1:1	119.7	48.7	37.8	68	86.1	155
A12 1:2	104.4	40.2	50.0	90	109.4	197
A12 2:3	111.4	44.1	43.9	79	120.6	217
EC 2216 (gray)	84.9	29.4	39.4	71	105.6	190
EA 9309.3 NA	102.2	39.0	45.6	82	74.4	134

A units: in/in/°F x 10⁻⁶

B units: μm/m/°C

Sample: A12 adhesive 1:1 ratio
Size: 7.4950 mm
Method: DOI
Comment: expansion probe, 1 gm tray weight

TMA

File: A: DOI.66
Operator: D.Brown
Run Date: 02/13/91 11:27

Figure E-1. Dimensional Change as a Function of Temperature for A12 1:1 Ratio

Sample: A12 adhesive 1:2 ratio
Size: 7.8420 mm
Method: DOI
Comment: expansion probe, 1 gm tray weight

TMA

File: A: DOI.68
Operator: D.Brown
Run Date: 02/13/91 14:55

Figure E-2. Dimensional Change as a Function of Temperature for A12 1:2 Ratio

Sample: A12 adhesive 2:3 ratio
Size: 7.4930 mm
Method: DOI
Comment: expansion probe, 1 gm tray weight

TMA

File: A: DOI.67
Operator: D.Brown
Run Date: 02/13/91 13:05

Figure E-3. Dimensional Change as a Function of Temperature for A12 2:3 Ratio

Sample: EC 2216 GRAY

Size: 7.8310 mm

Method: DOI

Comment: expansion probe, 1 gm tray weight

TMA

File: A: DOI.70

Operator: B. PARKER

Run Date: 02/14/92 08:40

Figure E-4. Dimensional Change as a Function of Temperature for EC2216

Sample: EA 9309.3 NA

Size: 8.0120 mm

Method: DOI

Comment: expansion probe, 1 gm tray weight

TMA

File: A:DOI.69

Operator: D.Brown

Run Date: 02/14/91 05:11

Figure E-5. Dimensional Change as a Function of Temperature for EA9309.3 NA

Sample: A12 adhesive 1:1 ratio
Size: 7.4950 mm
Method: DOI
Comment: expansion probe, 1 gm tray weight

TMA

File: DOI.66
Operator: D.Brown
Run Date: 02/13/91 11:27

Figure E-6. Dimensional Change During Temperature Cycle for A12 1:1 Ratio

Sample: A12 adhesive 1:2 ratio
Size: 7.8420 mm
Method: D01
Comment: expansion probe, 1 gm tray weight

TMA
TMA

File: D01.68
Operator: D.Brown
Run Date: 02/13/91 14:55

Figure E-7. Dimensional Change During Temperature Cycle for A12 1:2 Ratio

Sample: A12 adhesive 2:3 ratio
Size: 7.4930 mm
Method: DOI
Comment: expansion probe, 1 gm tray weight

TMA

File: DOI.67
Operator: D.Brown
Run Date: 02/13/91 13:05

Figure E-8. Dimensional Change During Temperature Cycle for A12 2:3 Ratio

Sample: EC 2216 GRAY

Size: 7.8310 mm

Method: DOI

Comment: expansion probe. 1 gm tray weight

TMA

File: DOI.70

Operator: B. PARKER

Run Date: 02/14/92 08:40

Figure E-9. Dimensional Change During Temperature Cycle for EC2216

Sample: EA 9309.3 NA

Size: 8.0120 mm

Method: DDI

Comment: expansion probe, 1 gm tray weight

TMA

File: DDI.69

Operator: D.Brown

Run Date: 02/14/91 05:11

Figure E-10. Dimensional Change During Temperature Cycle for EA9309.3 NA

Appendix F

RMS Glass Transition Temperature

RMS Glass Transition Temperature

Test Purpose:

The purpose of this test is to determine the shear modulus and glass transition temperature of the optical adhesives using Rheometric Mechanical Spectrometry.

Background:

Rheometric analysis is used to determine the shear modulus, damping coefficient (tan delta), and the glass transition temperature. The RMS was used in a forced torsional mode. In this mode, a rectangular sample is clamped between two grips. One grip is held fixed, and the other grip subjects the sample to a torsional oscillation of fixed amplitude. The amount of energy necessary to oscillate the sample in this manner is monitored as a function of temperature, as is the restoring force supplied by the sample. These energy measurements are converted to modulus and damping data.

Sample Preparation:

Samples, approximately 0.5" x 0.125" x 1.5", were mixed and cured per Table A-1 in Appendix A.

Test Procedure:

Rheometrics mechanical spectrometer testing was performed at 3°C per minute from -65°C to 125°C in the dynamic mode. All samples were run at a frequency of 1 hertz. The strain was automatically adjusted from a minimum of 0.05% to a maximum of 2%, and a measurement was recorded every minute.

Test Results:

Table F-1 shows the glass transition temperatures which are the temperatures at maximum tan delta. The curves showing the Shear Modulus (G'), G'' and Tan Delta as a function of temperature are shown in Figures F-1 through F-5. Tables F-2 through F-6 show the recorded data at each interval.

Table F-1: Glass Transition Temperature by RMS:

Material	Glass Transition Temperature	
	(°C)	(°F)
A12 1:1	75	167.0
A12 2:3	48	118.4
A12 1:2	40	104.0
EC 2216 (gray)	39	102.2
EA 9309.3 NA	87	188.6

Figure F-1. Shear Modulus as a Function of Temperature for A12 1:1 Ratio

Figure F-2. Shear Modulus as a Function of Temperature for A12 1:2 Ratio

A-12 EPOXY (2:3) -85C TO 125C AT 3 DEG.C/MIN. 2/26/92

Figure F-3. Shear Modulus as a Function of Temperature for A12 2:3 Ratio

EC2216 EPOXY -85C TO 125C AT 3 DEG. C/MIN. 2--28-92

Figure F-4. Shear Modulus as a Function of Temperature for EC2216

Figure F-5. Shear Modulus as a Function of Temperature for EA9309.3 NA

Table F-2. RMS Recorded Data for A12 (1:1)

Rheometrics		RECAP II
Experiment No. :	7	Sample No. : 1
Title:		
A-12 EPOXY (1:1) -65C TO 125C AT 3 DEG.C/MIN. 2-29-92		
Operator : Karen		
Date and Time : Sat 05-Jan-80 - 9:58:19pm		
Operating Mode : DYNAMIC		
Sweep Type : 7 Temperature Ramp		
Geometry : Torsion Rectangular		
THICK	:	2.660
WIDTH	:	12.510
LENGTH	:	21.890
Notes :		
Run from -65C to 125C at 3 deg.C/min. with a min. strain of .05% and max. of 2% with a measurement interval of 1. This sample was run at 1 hertz.		

A-12 EPOXY (1:1) -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
1	1.785e+10	0.000e+00	0.000e+00	4.861e-02	0.0	-65.0
2	1.727e+10	0.000e+00	0.000e+00	4.864e-02	1.0	-62.0
3	1.679e+10	0.000e+00	0.000e+00	4.865e-02	2.0	-59.0
4	1.598e+10	0.000e+00	0.000e+00	4.873e-02	3.0	-56.0
5	1.500e+10	0.000e+00	0.000e+00	4.882e-02	4.0	-52.0
6	1.455e+10	0.000e+00	0.000e+00	5.876e-02	5.0	-50.0
7	1.376e+10	0.000e+00	0.000e+00	5.883e-02	6.0	-46.0
8	1.309e+10	0.000e+00	0.000e+00	5.887e-02	7.0	-44.0
9	1.266e+10	0.000e+00	0.000e+00	5.894e-02	8.0	-41.0
10	1.247e+10	0.000e+00	0.000e+00	7.044e-02	9.0	-38.0
11	1.228e+10	0.000e+00	0.000e+00	7.045e-02	10.0	-34.0
12	1.220e+10	0.000e+00	0.000e+00	7.045e-02	11.0	-31.0
13	1.212e+10	0.000e+00	0.000e+00	7.046e-02	12.0	-29.0
14	1.214e+10	0.000e+00	0.000e+00	7.046e-02	13.0	-24.0
15	1.225e+10	0.000e+00	0.000e+00	7.044e-02	14.0	-21.0
16	1.254e+10	0.000e+00	0.000e+00	7.041e-02	15.0	-18.0
17	1.313e+10	0.000e+00	0.000e+00	7.036e-02	16.0	-15.0
18	1.449e+10	0.000e+00	0.000e+00	7.021e-02	17.0	-12.0
19	1.613e+10	4.766e+08	2.955e-02	7.000e-02	18.0	-9.0
20	1.597e+10	4.748e+08	2.973e-02	7.000e-02	19.0	-7.0
21	1.581e+10	4.825e+08	3.053e-02	7.003e-02	20.0	-5.0
22	1.569e+10	4.733e+08	3.018e-02	7.005e-02	21.0	-3.0
23	1.553e+10	4.594e+08	2.959e-02	7.006e-02	22.0	1.0
24	1.537e+10	4.458e+08	2.899e-02	7.010e-02	23.0	5.0
25	1.522e+10	4.498e+08	2.955e-02	7.013e-02	24.0	8.0
26	1.508e+10	4.556e+08	3.021e-02	7.013e-02	25.0	11.0
27	1.488e+10	4.447e+08	2.989e-02	7.015e-02	26.0	15.0
28	1.469e+10	4.536e+08	3.088e-02	7.018e-02	27.0	17.0
29	1.451e+10	4.562e+08	3.144e-02	7.020e-02	28.0	20.0
30	1.434e+10	4.684e+08	3.267e-02	7.021e-02	29.0	24.0
31	1.414e+10	4.593e+08	3.248e-02	7.026e-02	30.0	26.0
32	1.393e+10	4.781e+08	3.432e-02	7.029e-02	31.0	29.0
33	1.369e+10	4.865e+08	3.554e-02	7.030e-02	32.0	34.0
34	1.342e+10	5.115e+08	3.810e-02	7.032e-02	33.0	37.0
35	1.313e+10	5.300e+08	4.037e-02	7.038e-02	34.0	39.0
36	1.278e+10	5.542e+08	4.336e-02	7.042e-02	35.0	42.0
37	1.232e+10	5.965e+08	4.844e-02	7.048e-02	36.0	45.0
38	1.164e+10	6.722e+08	5.777e-02	7.058e-02	37.0	48.0
39	1.042e+10	8.530e+08	8.186e-02	7.073e-02	38.0	52.0
40	8.761e+09	1.085e+09	1.239e-01	8.528e-02	39.0	55.0
41	6.986e+09	1.295e+09	1.854e-01	1.019e-01	40.0	58.0
42	5.395e+09	1.379e+09	2.555e-01	1.225e-01	41.0	61.0
43	3.892e+09	1.349e+09	3.467e-01	1.487e-01	42.0	64.0
44	2.539e+09	1.185e+09	4.667e-01	1.780e-01	43.0	66.0
45	1.499e+09	8.972e+08	5.985e-01	2.137e-01	44.0	69.0
46	7.707e+08	5.582e+08	7.243e-01	2.575e-01	45.0	71.0
47	4.246e+08	3.266e+08	7.693e-01	3.091e-01	46.0	75.0
48	2.571e+08	1.839e+08	7.152e-01	3.716e-01	47.0	78.0
49	1.762e+08	1.031e+08	5.850e-01	4.459e-01	48.0	81.0
50	1.356e+08	5.970e+07	4.403e-01	5.351e-01	49.0	84.0

4-12 EPOXY (1:1) -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
51	1.144e+08	3.650e+07	3.192e-01	6.420e-01	50.0	87.0
52	9.888e+07	2.029e+07	2.052e-01	7.704e-01	51.0	92.0
53	9.202e+07	1.274e+07	1.384e-01	9.251e-01	52.0	95.0
54	9.106e+07	8.223e+06	9.030e-02	1.110e+00	53.0	98.0
55	9.048e+07	5.520e+06	6.100e-02	1.332e+00	54.0	100.0
56	9.085e+07	3.770e+06	4.150e-02	1.599e+00	55.0	102.0
57	9.142e+07	2.510e+06	2.746e-02	1.917e+00	56.0	105.0
58	9.086e+07	1.736e+06	1.911e-02	2.001e+00	57.0	109.0
59	9.049e+07	1.185e+06	1.309e-02	2.001e+00	58.0	112.0
60	9.396e+07	5.721e+05	6.088e-03	2.001e+00	59.0	115.0
61	9.408e+07	5.397e+05	5.737e-03	2.001e+00	60.0	119.0
62	9.523e+07	4.185e+05	4.395e-03	2.001e+00	61.0	122.0
63	9.374e+07	3.318e+05	3.540e-03	2.001e+00	62.0	124.0
64	9.591e+07	3.161e+05	3.296e-03	2.001e+00	63.0	124.0

Table F-3. RMS Recorded Data for A12 (1:2)

Rheometrics		RECAP II
Experiment No. :	45	Sample No. : 1
Title:		
A-12 EPOXY (1:2) -65C TO 125C AT 3 DEG.C/MIN. 2-25-92		
Operator :Karen		
Date and Time : Thu 03-Jan-80 - 10:58:05pm		
Operating Mode : DYNAMIC		
Sweep Type : 7 Temperature Ramp		
Geometry : Torsion Rectangular		
THICK :	3.200	
WIDTH :	12.460	
LENGTH :	23.370	

Notes :

Ran sample from -60C to 125C at 3 deg.C/min. with a minimum strain of .05% and max. of 2% with a measurement interval of 1. This sample was run at 1 hertz.

1-1E EPOXY (1:2) -65C TO 125C AT 3 DEG.C/MIN. 2-25-92

NO.	G' dyn/cm.sq.	G'' dyn/cm.sq.	TAN_DELTA	%STRAIN	TIME min.	TEMP degrees c
1	1.261e+10	7.777e+08	6.166e-02	4.653e-02	0.0	-65.0
2	1.261e+10	7.438e+08	5.897e-02	4.652e-02	1.0	-62.0
3	1.254e+10	7.385e+08	5.891e-02	4.652e-02	2.0	-59.0
4	1.245e+10	7.271e+08	5.842e-02	4.653e-02	3.0	-56.0
5	1.234e+10	7.012e+08	5.682e-02	4.656e-02	4.0	-53.0
6	1.225e+10	6.810e+08	5.560e-02	4.657e-02	5.0	-49.0
7	1.216e+10	6.611e+08	5.437e-02	4.660e-02	6.0	-47.0
8	1.205e+10	6.428e+08	5.336e-02	4.662e-02	7.0	-43.0
9	1.192e+10	6.221e+08	5.217e-02	4.664e-02	8.0	-40.0
10	1.181e+10	6.180e+08	5.231e-02	4.663e-02	9.0	-37.0
11	1.168e+10	6.056e+08	5.187e-02	4.666e-02	10.0	-34.0
12	1.150e+10	5.627e+08	4.893e-02	5.788e-02	11.0	-31.0
13	1.136e+10	5.639e+08	4.963e-02	5.788e-02	12.0	-28.0
14	1.121e+10	5.661e+08	5.052e-02	5.791e-02	13.0	-23.0
15	1.106e+10	5.525e+08	4.994e-02	5.792e-02	14.0	-21.0
16	1.093e+10	5.575e+08	5.079e-02	5.794e-02	15.0	-18.0
17	1.089e+10	5.312e+08	4.879e-02	5.797e-02	16.0	-15.0
18	1.073e+10	5.494e+08	5.122e-02	5.798e-02	17.0	-12.0
19	1.054e+10	5.640e+08	5.350e-02	5.803e-02	18.0	-9.0
20	1.031e+10	5.821e+08	5.646e-02	5.807e-02	19.0	-7.0
21	1.007e+10	6.040e+08	5.998e-02	5.811e-02	20.0	-4.0
22	9.867e+09	6.388e+08	6.474e-02	5.813e-02	21.0	-3.0
23	9.584e+09	6.856e+08	7.153e-02	5.818e-02	22.0	1.0
24	9.228e+09	7.292e+08	7.902e-02	5.824e-02	23.0	5.0
25	8.752e+09	7.745e+08	8.849e-02	7.110e-02	24.0	7.0
26	8.193e+09	8.731e+08	1.066e-01	7.124e-02	25.0	11.0
27	7.373e+09	1.018e+09	1.380e-01	7.141e-02	26.0	15.0
28	6.401e+09	1.146e+09	1.791e-01	8.461e-02	27.0	17.0
29	5.150e+09	1.267e+09	2.442e-01	1.026e-01	28.0	21.0
30	3.806e+09	1.281e+09	3.367e-01	1.231e-01	29.0	24.0
31	2.462e+09	1.163e+09	4.684e-01	1.480e-01	30.0	26.0
32	1.396e+09	9.094e+08	6.513e-01	1.794e-01	31.0	29.0
33	6.394e+08	5.635e+08	8.813e-01	2.146e-01	32.0	33.0
34	2.985e+08	3.186e+08	1.067e+00	2.576e-01	33.0	37.0
35	1.777e+08	1.667e+08	1.130e+00	3.101e-01	34.0	40.0
36	9.541e+07	9.007e+07	1.054e+00	3.713e-01	35.0	42.0
37	5.521e+07	1.505e+07	8.810e-01	4.451e-01	36.0	45.0
38	3.031e+07	8.817e+07	6.977e-01	5.358e-01	37.0	47.0
39	1.601e+07	1.531e+07	4.947e-01	6.422e-01	38.0	50.0
40	8.211e+07	1.031e+07	3.681e-01	7.377e-01	39.0	53.0
41	4.539e+07	6.575e+06	2.557e-01	8.255e-01	40.0	55.0
42	2.411e+07	4.772e+06	1.975e-01	1.109e+00	41.0	58.0
43	2.211e+07	3.138e+06	1.417e-01	1.330e+00	42.0	61.0
44	2.246e+07	2.443e+06	1.088e-01	1.597e+00	43.0	63.0
45	2.292e+07	1.730e+06	7.546e-02	1.919e+00	44.0	65.0
46	2.065e+07	1.205e+06	5.839e-02	2.001e+00	45.0	71.0
47	2.111e+07	9.804e+05	4.645e-02	2.001e+00	46.0	75.0
48	2.137e+07	8.077e+05	3.780e-02	2.001e+00	47.0	77.0
49	2.140e+07	6.262e+05	2.973e-02	2.001e+00	48.0	80.0
50	2.137e+07	5.204e+05	2.436e-02	2.001e+00	49.0	84.0

-12 EPOXY (1:2) -65C TO 125C AT 3 DEG.C/MIN. 2-25-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
51	2.049e+07	3.671e+05	1.792e-02	2.001e+00	50.0	87.0
52	2.117e+07	2.716e+05	1.282e-02	2.001e+00	51.0	92.0
53	2.151e+07	2.809e+05	1.306e-02	2.001e+00	52.0	95.0
54	2.145e+07	2.749e+05	1.283e-02	2.001e+00	53.0	98.0
55	2.164e+07	1.928e+05	8.911e-03	2.001e+00	54.0	100.0
56	2.178e+07	1.595e+05	7.324e-03	2.001e+00	55.0	103.0
57	2.179e+07	1.516e+05	6.958e-03	2.001e+00	56.0	105.0
58	2.069e+07	1.415e+05	6.836e-03	2.001e+00	57.0	109.0
59	2.106e+07	1.465e+05	6.958e-03	2.001e+00	58.0	112.0
60	2.122e+07	1.347e+05	6.348e-03	2.001e+00	59.0	116.0
61	2.116e+07	2.208e+05	1.044e-02	2.001e+00	60.0	119.0
62	2.114e+07	1.748e+05	8.270e-03	2.001e+00	61.0	122.0
63	2.116e+07	1.892e+05	8.942e-03	2.001e+00	62.0	124.0
64	2.127e+07	1.804e+05	8.484e-03	2.001e+00	63.0	124.0

Table F-4. RMS Recorded Data for A12 (2:3)

Rheometrics		RECAP II
Experiment No. :	46	Sample No. : 1
Title:		
A-12 EPOXY (2:3) -65C TO 125C AT 3 DEG.C/MIN. 2/26/92		
Operator :Karen		
Date and Time : Fri 04-Jan-80 - 10:10:32pm		
Operating Mode : DYNAMIC		
Sweep Type : 7 Temperature Ramp		
Geometry : Torsion Rectangular		
THICK	:	3.240
WIDTH	:	12.520
LENGTH	:	24.000

Notes :

This sample was run from -65C to 125C at 3 deg.C/min. with a min. strain of .05% and max. of 2% with a measurement interval of 1. This sample was run at 1 hertz.

-12 EPOXY (2:3) -65C TO 125C AT 3 DEG.C/MIN. 2/26/92

NO.	G' dyn/cm.sq.	G'' dyn/cm sq.	TAN_DELTA	%STRAIN	TIME min.	TEMP degrees c
1	1.661e+10	8.543e+08	5.144e-02	4.694e-02	0.0	-65.0
2	1.645e+10	8.685e+08	5.278e-02	4.696e-02	1.0	-61.0
3	1.626e+10	8.464e+08	5.205e-02	4.700e-02	2.0	-59.0
4	1.602e+10	8.105e+08	5.058e-02	4.705e-02	3.0	-55.0
5	1.587e+10	7.934e+08	5.015e-02	4.707e-02	4.0	-52.0
6	1.562e+10	7.627e+08	4.884e-02	4.709e-02	5.0	-49.0
7	1.541e+10	7.480e+08	4.853e-02	4.711e-02	6.0	-47.0
8	1.519e+10	7.297e+08	4.804e-02	4.713e-02	7.0	-43.0
9	1.498e+10	6.784e+08	4.529e-02	4.717e-02	8.0	-40.0
10	1.480e+10	6.730e+08	4.549e-02	4.719e-02	9.0	-37.0
11	1.462e+10	6.507e+08	4.451e-02	4.721e-02	10.0	-34.0
12	1.445e+10	6.432e+08	4.451e-02	4.723e-02	11.0	-31.0
13	1.428e+10	6.363e+08	4.456e-02	4.727e-02	12.0	-28.0
14	1.407e+10	6.429e+08	4.568e-02	4.728e-02	13.0	-24.0
15	1.380e+10	6.348e+08	4.572e-02	4.731e-02	14.0	-21.0
16	1.372e+10	6.227e+08	4.539e-02	4.732e-02	15.0	-18.0
17	1.353e+10	6.244e+08	4.615e-02	4.735e-02	16.0	-15.0
18	1.333e+10	6.174e+08	4.631e-02	4.739e-02	17.0	-12.0
19	1.311e+10	6.195e+08	4.725e-02	4.743e-02	18.0	-8.0
20	1.286e+10	6.165e+08	4.795e-02	4.746e-02	19.0	-7.0
21	1.262e+10	6.499e+08	5.150e-02	4.749e-02	20.0	-4.0
22	1.243e+10	6.624e+08	5.330e-02	4.751e-02	21.0	-2.0
23	1.220e+10	6.841e+08	5.609e-02	4.754e-02	22.0	1.0
24	1.191e+10	6.928e+08	5.817e-02	4.759e-02	23.0	5.0
25	1.159e+10	7.441e+08	6.421e-02	4.764e-02	24.0	8.0
26	1.121e+10	7.813e+08	6.969e-02	4.768e-02	25.0	11.0
27	1.069e+10	8.432e+08	7.887e-02	4.774e-02	26.0	15.0
28	1.010e+10	8.545e+08	8.464e-02	5.887e-02	27.0	17.0
29	9.389e+09	9.707e+08	1.034e-01	5.899e-02	28.0	21.0
30	8.470e+09	1.097e+09	1.295e-01	5.915e-02	29.0	23.0
31	7.299e+09	1.212e+09	1.660e-01	7.046e-02	30.0	26.0
32	5.894e+09	1.318e+09	2.236e-01	8.518e-02	31.0	29.0
33	4.240e+09	1.315e+09	3.100e-01	1.015e-01	32.0	34.0
34	2.830e+09	1.183e+09	4.181e-01	1.233e-01	33.0	37.0
35	1.689e+09	9.267e+08	5.486e-01	1.479e-01	34.0	40.0
36	9.064e+08	6.454e+08	7.121e-01	1.788e-01	35.0	42.0
37	4.244e+08	3.858e+08	9.091e-01	2.150e-01	36.0	45.0
38	1.968e+08	2.028e+08	1.030e+00	2.573e-01	37.0	48.0
39	1.034e+08	9.917e+07	9.589e-01	3.091e-01	38.0	52.0
40	6.898e+07	5.376e+07	7.794e-01	3.727e-01	39.0	55.0
41	5.216e+07	3.030e+07	5.810e-01	4.454e-01	40.0	58.0
42	4.376e+07	1.806e+07	4.127e-01	5.349e-01	41.0	61.0
43	3.909e+07	1.121e+07	2.869e-01	6.421e-01	42.0	64.0
44	3.749e+07	7.321e+06	1.953e-01	7.703e-01	43.0	67.0
45	3.657e+07	4.869e+06	1.331e-01	9.255e-01	44.0	69.0
46	3.533e+07	3.009e+06	8.518e-02	1.111e+00	45.0	71.0
47	3.467e+07	2.064e+06	5.952e-02	1.331e+00	46.0	74.0
48	3.492e+07	1.455e+06	4.167e-02	1.598e+00	47.0	78.0
49	3.555e+07	1.192e+06	3.352e-02	1.918e+00	48.0	81.0
50	3.504e+07	8.739e+05	2.494e-02	2.000e+00	49.0	84.0

1-12 EPOXY (2:3) -65C TO 125C AT 3 DEG.C/MIN. 2/26/92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
51	3.512e+07	7.086e+05	2.017e-02	2.000e+00	50.0	87.0
52	3.522e+07	5.503e+05	1.563e-02	2.000e+00	51.0	92.0
53	3.470e+07	4.575e+05	1.318e-02	2.000e+00	52.0	95.0
54	3.459e+07	4.139e+05	1.196e-02	2.000e+00	53.0	98.0
55	3.435e+07	3.313e+05	9.644e-03	2.000e+00	54.0	100.0
56	3.452e+07	3.203e+05	9.278e-03	2.000e+00	55.0	103.0
57	3.468e+07	2.794e+05	8.057e-03	2.000e+00	56.0	105.0
58	3.494e+07	3.221e+05	9.217e-03	2.000e+00	57.0	109.0
59	3.521e+07	3.030e+05	8.606e-03	2.001e+00	58.0	112.0
60	3.531e+07	2.286e+05	6.470e-03	2.001e+00	59.0	116.0
61	3.542e+07	2.465e+05	6.958e-03	2.000e+00	60.0	119.0
62	3.568e+07	2.657e+05	7.446e-03	2.000e+00	61.0	122.0
63	3.563e+07	2.058e+05	5.768e-03	2.001e+00	62.0	124.0
64	3.537e+07	2.202e+05	6.226e-03	2.000e+00	63.0	124.0

Table F-5. RMS Recorded Data for EC2216

Rheometrics RECAP II

Experiment No. : 8 Sample No. : 1

Title:
EC2216 EPOXY -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

Operator :Karen

Date and Time : Sat 05-Jan-80 - 11:55:46pm

Operating Mode : DYNAMIC

Sweep Type : 7 Temperature Ramp

Geometry : Torsion Rectangular

THICK : 2.570
WIDTH : 12.490
LENGTH : 24.130

Notes :
Ran sample from -65C to 125C at 3 deg.C/min. with a min. strain of
.05% and max. of 2% with a measurement interval of 1. This sample
(was run at 1 hertz.

EC2216 EPOXY -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
1	2.519e+10	1.014e+09	4.027e-02	4.858e-02	0.0	-65.0
2	2.498e+10	9.741e+08	3.899e-02	4.860e-02	1.0	-62.0
3	2.475e+10	9.506e+08	3.841e-02	4.862e-02	2.0	-59.0
4	2.451e+10	8.859e+08	3.615e-02	4.864e-02	3.0	-55.0
5	2.426e+10	8.161e+08	3.364e-02	4.868e-02	4.0	-52.0
6	2.407e+10	7.788e+08	3.236e-02	4.869e-02	5.0	-50.0
7	2.386e+10	7.402e+08	3.102e-02	4.870e-02	6.0	-47.0
8	2.364e+10	7.390e+08	3.126e-02	4.871e-02	7.0	-43.0
9	2.339e+10	7.386e+08	3.158e-02	4.872e-02	8.0	-40.0
10	2.319e+10	7.432e+08	3.205e-02	4.874e-02	9.0	-37.0
11	2.294e+10	7.051e+08	3.074e-02	4.877e-02	10.0	-34.0
12	2.265e+10	6.890e+08	3.042e-02	4.879e-02	11.0	-31.0
13	2.241e+10	7.039e+08	3.141e-02	4.881e-02	12.0	-28.0
14	2.214e+10	7.070e+08	3.193e-02	4.882e-02	13.0	-24.0
15	2.185e+10	7.510e+08	3.432e-02	4.884e-02	14.0	-21.0
16	2.157e+10	7.671e+08	3.557e-02	4.886e-02	15.0	-18.0
17	2.124e+10	7.815e+08	3.679e-02	4.888e-02	16.0	-15.0
18	2.088e+10	8.422e+08	4.034e-02	4.890e-02	17.0	-12.0
19	2.047e+10	8.852e+08	4.324e-02	4.893e-02	18.0	-8.0
20	1.994e+10	9.342e+08	4.685e-02	4.898e-02	19.0	-7.0
21	1.938e+10	1.034e+09	5.333e-02	4.901e-02	20.0	-4.0
22	1.889e+10	1.113e+09	5.891e-02	4.905e-02	21.0	-3.0
23	1.820e+10	1.210e+09	6.649e-02	4.909e-02	22.0	1.0
24	1.731e+10	1.334e+09	7.719e-02	4.917e-02	23.0	5.0
25	1.622e+10	1.483e+09	9.146e-02	4.925e-02	24.0	8.0
26	1.489e+10	1.629e+09	1.094e-01	5.930e-02	25.0	11.0
27	1.315e+10	1.803e+09	1.376e-01	5.945e-02	26.0	14.0
28	1.127e+10	1.930e+09	1.712e-01	7.103e-02	27.0	17.0
29	9.231e+09	1.985e+09	2.150e-01	8.544e-02	28.0	20.0
30	7.121e+09	1.966e+09	2.760e-01	1.021e-01	29.0	23.0
31	5.078e+09	1.794e+09	3.533e-01	1.227e-01	30.0	26.0
32	3.338e+09	1.489e+09	4.460e-01	1.485e-01	31.0	29.0
33	1.954e+09	1.062e+09	5.436e-01	1.783e-01	32.0	34.0
34	1.171e+09	7.061e+08	6.023e-01	2.155e-01	33.0	37.0
35	7.154e+08	4.445e+08	6.090e-01	2.581e-01	34.0	39.0
36	4.157e+08	3.733e+08	5.643e-01	3.103e-01	35.0	42.0
37	2.157e+08	1.673e+08	4.892e-01	3.704e-01	36.0	45.0
38	1.054e+08	1.054e+08	4.005e-01	4.449e-01	37.0	48.0
39	2.111e+08	6.544e+07	3.038e-01	5.345e-01	38.0	52.0
40	1.235e+08	4.427e+07	2.302e-01	6.420e-01	39.0	55.0
41	1.776e+08	3.027e+07	1.705e-01	7.700e-01	40.0	58.0
42	1.693e+08	2.144e+07	1.268e-01	9.252e-01	41.0	61.0
43	1.637e+08	1.566e+07	9.581e-02	1.109e+00	42.0	64.0
44	1.605e+08	1.184e+07	7.374e-02	1.332e+00	43.0	67.0
45	1.589e+08	9.315e+06	5.864e-02	1.597e+00	44.0	70.0
46	1.584e+08	7.427e+06	4.690e-02	1.917e+00	45.0	71.0
47	1.550e+08	6.304e+06	4.067e-02	2.000e+00	46.0	74.0
48	1.511e+08	5.685e+06	3.762e-02	2.000e+00	47.0	78.0
49	1.485e+08	5.080e+06	3.413e-02	2.000e+00	48.0	81.0
50	1.481e+08	4.703e+06	3.175e-02	2.000e+00	49.0	84.0

EC221-5 EPOXY -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm ² .sq.	dyn/cm ² .sq.			min.	degrees c
51	1.485e+08	4.565e+06	3.071e-02	2.000e+00	50.0	87.0
52	1.506e+08	4.754e+06	3.157e-02	2.000e+00	51.0	92.0
53	1.505e+08	4.774e+06	3.172e-02	2.000e+00	52.0	96.0
54	1.518e+08	4.823e+06	3.178e-02	2.000e+00	53.0	98.0
55	1.540e+08	4.664e+06	3.028e-02	2.000e+00	54.0	100.0
56	1.529e+08	4.617e+06	3.019e-02	2.000e+00	55.0	103.0
57	1.468e+08	4.368e+06	2.975e-02	2.000e+00	56.0	105.0
58	1.459e+08	4.321e+06	2.961e-02	2.000e+00	57.0	109.0
59	1.470e+08	4.389e+06	2.946e-02	2.000e+00	58.0	113.0
60	1.502e+08	4.329e+06	2.882e-02	2.000e+00	59.0	115.0
61	1.511e+08	4.279e+06	2.833e-02	2.000e+00	60.0	119.0
62	1.489e+08	4.254e+06	2.857e-02	2.000e+00	61.0	123.0
63	1.466e+08	4.083e+06	2.784e-02	2.000e+00	62.0	125.0
64	1.473e+08	4.048e+06	2.747e-02	2.000e+00	63.0	124.0

Table F-6. RMS Recorded Data for EA9309

----- Rheometrics RECAP II -----
Experiment No. : 6 Sample No. : 1

Title:

EA9309.3MA EPOXY -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

Operator : Karen

Date And Time : Sat 05-Jan-80 - 7:46:42pm

Operating Mode : DYNAMIC

Sweep Type : 7 Temperature Ramp

Geometry : Torsion Rectangular

THICK : 2.900
WIDTH : 12.480
LENGTH : 24.050

Notes :

Run from -65C to 125C at 3 deg.C/min. with a min. strain of .05% and
max. of 2% with a measurement interval of 1. This sample was run at
1 hertz.

72030 DINA EPOXY -45C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	D'	D''	TAN_DELTA	STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
1	1.429e+10	1.043e+09	7.301e-02	4.831e-02	0.0	-65.0
2	1.406e+10	1.044e+09	7.411e-02	4.831e-02	1.0	-62.0
3	1.369e+10	1.046e+09	7.644e-02	4.835e-02	2.0	-59.0
4	1.323e+10	1.055e+09	7.976e-02	4.839e-02	3.0	-55.0
5	1.270e+10	9.757e+08	7.684e-02	5.827e-02	4.0	-52.0
6	1.229e+10	9.158e+08	7.457e-02	5.832e-02	5.0	-50.0
7	1.192e+10	8.423e+08	7.064e-02	5.837e-02	6.0	-47.0
8	1.156e+10	7.950e+08	6.877e-02	5.842e-02	7.0	-43.0
9	1.129e+10	7.321e+08	6.486e-02	5.843e-02	8.0	-40.0
10	1.101e+10	6.839e+08	6.211e-02	5.849e-02	9.0	-37.0
11	1.074e+10	6.462e+08	6.018e-02	6.994e-02	10.0	-34.0
12	1.052e+10	6.195e+08	5.891e-02	6.995e-02	11.0	-31.0
13	1.028e+10	5.871e+08	5.713e-02	6.999e-02	12.0	-28.0
14	1.005e+10	5.588e+08	5.561e-02	7.004e-02	13.0	-24.0
15	9.858e+09	5.192e+08	5.266e-02	7.006e-02	14.0	-21.0
16	9.671e+09	4.935e+08	5.102e-02	7.010e-02	15.0	-18.0
17	9.514e+09	4.681e+08	4.920e-02	7.011e-02	16.0	-15.0
18	9.363e+09	4.412e+08	4.712e-02	7.012e-02	17.0	-12.0
19	9.232e+09	4.047e+08	4.384e-02	7.014e-02	18.0	-9.0
20	9.072e+09	3.533e+08	3.894e-02	8.437e-02	19.0	-7.0
21	8.959e+09	3.307e+08	3.691e-02	8.440e-02	20.0	-3.0
22	8.863e+09	3.126e+08	3.519e-02	8.440e-02	21.0	-2.0
23	8.802e+09	2.929e+08	3.328e-02	8.441e-02	22.0	2.0
24	8.717e+09	2.810e+08	3.224e-02	8.443e-02	23.0	5.0
25	8.634e+09	2.657e+08	3.077e-02	8.443e-02	24.0	7.0
26	8.537e+09	2.552e+08	2.982e-02	8.444e-02	25.0	11.0
27	8.457e+09	2.432e+08	2.876e-02	8.449e-02	26.0	14.0
28	8.374e+09	2.293e+08	2.738e-02	8.450e-02	27.0	18.0
29	8.294e+09	2.222e+08	2.679e-02	8.451e-02	28.0	21.0
30	8.217e+09	2.200e+08	2.677e-02	8.452e-02	29.0	23.0
31	8.142e+09	2.244e+08	2.756e-02	8.450e-02	30.0	26.0
32	8.054e+09	2.218e+08	2.753e-02	8.452e-02	31.0	29.0
33	7.957e+09	2.191e+08	2.753e-02	8.456e-02	32.0	34.0
34	7.873e+09	2.162e+08	2.746e-02	8.455e-02	33.0	37.0
35	7.775e+09	2.123e+08	2.730e-02	8.460e-02	34.0	39.0
36	7.669e+09	2.160e+08	2.816e-02	8.464e-02	35.0	42.0
37	7.570e+09	2.116e+08	2.795e-02	1.022e-01	36.0	45.0
38	7.447e+09	2.162e+08	2.903e-02	1.022e-01	37.0	48.0
39	7.297e+09	2.234e+08	3.062e-02	1.023e-01	38.0	52.0
40	7.149e+09	2.366e+08	3.309e-02	1.023e-01	39.0	55.0
41	6.963e+09	2.560e+08	3.677e-02	1.023e-01	40.0	57.0
42	6.727e+09	2.843e+08	4.226e-02	1.024e-01	41.0	60.0
43	6.438e+09	3.345e+08	5.196e-02	1.025e-01	42.0	64.0
44	6.081e+09	4.153e+08	6.886e-02	1.026e-01	43.0	67.0
45	5.436e+09	5.549e+06	1.022e-01	1.227e-01	44.0	69.0
46	4.319e+09	7.673e+08	1.776e-01	1.230e-01	45.0	71.0
47	2.989e+09	8.357e+08	2.796e-01	1.492e-01	46.0	75.0
48	1.513e+09	6.864e+08	3.787e-01	1.783e-01	47.0	78.0
49	1.005e+09	4.600e+08	4.579e-01	2.131e-01	48.0	80.0
50	4.866e+08	2.682e+08	5.513e-01	2.586e-01	49.0	84.0

TA9309.3NA EPOXY -65C TO 125C AT 3 DEG.C/MIN. 2-28-92

NO.	G'	G''	TAN_DELTA	%STRAIN	TIME	TEMP
	dyn/cm.sq.	dyn/cm.sq.			min.	degrees c
51	2.363e+08	1.410e+08	5.967e-01	3.097e-01	50.0	87.0
52	1.454e+08	6.489e+07	4.462e-01	3.716e-01	51.0	92.0
53	1.121e+08	3.257e+07	2.905e-01	4.455e-01	52.0	95.0
54	1.001e+08	1.991e+07	1.988e-01	5.354e-01	53.0	98.0
55	9.471e+07	1.402e+07	1.480e-01	6.428e-01	54.0	101.0
56	8.862e+07	1.093e+07	1.233e-01	7.702e-01	55.0	103.0
57	8.866e+07	9.440e+06	1.065e-01	9.253e-01	56.0	105.0
58	8.442e+07	7.335e+06	8.689e-02	1.110e+00	57.0	109.0
59	8.243e+07	5.598e+06	6.791e-02	1.331e+00	58.0	113.0
60	8.107e+07	4.279e+06	5.278e-02	1.598e+00	59.0	116.0
61	8.147e+07	3.339e+06	4.098e-02	1.917e+00	60.0	119.0
62	8.049e+07	2.587e+06	3.215e-02	2.000e+00	61.0	123.0
63	7.964e+07	2.083e+06	2.616e-02	2.000e+00	62.0	124.0
64	7.937e+07	1.982e+06	2.497e-02	2.000e+00	63.0	124.0

Appendix G

DMA Glass Transition Temperature

DMA Glass Transition Temperature

Test Purpose:

The purpose of this test is to look at the viscoelastic properties of optical adhesives and determine the glass transition temperature using Dynamic Mechanical Analysis (DMA).

Background:

The technique of Dynamic Mechanical Analysis is one of the most sensitive single techniques available for characterizing and interpreting the mechanical behavior of materials. DMA is based on observing the viscoelastic response of materials subjected to a small oscillatory strain. The DuPont 983 DMA uses a flexural bending deformation mode of strain, but also has the capability for other deformations such as shear.

The DMA technique separates the viscoelasticity of a material into the two components of Modulus (E): a real part, which is the elastic modulus (E'); and an imaginary part, which is the damping or viscous component (E''). The standard complex variable notation is: $E = E' + iE''$. This separation of measurements into the two components describing two independent processes within the materials' elasticity (energy storage) and viscosity (energy dissipation) is the fundamental characteristic of dynamic mechanical analysis that separates it from other mechanical testing techniques.

The high sensitivity of DMA is particularly advantageous in the measurement and interpretation of transitions that are studied by the measurement of tan delta (the ratio of lost to stored energy). In the Resonant Mode, the 983 DMA operates on the mechanical principle of forced resonant vibratory motion at a fixed amplitude (strain), which is selected by the operator. The arms and sample are displaced by the electromagnetic driver, which subjects the sample to a fixed deformation and sets the system in resonant oscillation. The amplitude and frequency of oscillation are measured by the LVDT. That measurement, together with the amount of energy sent to the driver, is used to calculate the desired viscoelastic properties.

Sample Preparation:

Samples, approximately 0.5" x 0.125" x 1.5" were made and tested from each adhesive. The samples were mixed and cured per Table A-1 in Appendix A.

Test Procedure:

The DMA tests were performed using a TA instruments model 983 dynamic mechanical analyzer. The DMA was run in the resonant mode at a heat rate of 3°C per minute from -65°C to 80°C.

Test Results:

Table G-1 shows the glass transition temperature for the different optical adhesives. Figures G-1 through G-5 show the frequency response versus temperature.

Table G-1: Glass Transition Temperature by DMA:

Material	Glass Transition Temperature	
	(°F)	(°C)
A12 1:1	130.5	54.7
A12 1:2	74.5	23.6
A12 2:3	85.8	29.9
EC 2216 (gray)	69.4	20.8
EA 9309.3 NA	119.0	66.1

Sample : A12 1:1
Size : 34.73 x 12.40 x 2.80 mm
Method : LNCA SUBAMBIENT FILLED
Comment: DMA RESONANT 3 DEGREES C/MINUTE

DMA

File : C: RES.03
Operator: DAVID A. SPIEKER
Run Date: 12-May-92 08:32

Figure G-1. Frequency Response as a Function of Temperature for A12 1:1 Ratio

Sample : A12 1:2
Size : 34.73 x 12.40 x 2.80 mm
Method : LNCA SUBAMBIENT FILLED
Comment: DMA RESONANT 3 DEGREES C/MINUTE

DMA

File : C: RES.02
Operator: DAVID A. SPIEKER
Run Date: 11-May-92 13:57

Figure G-2. Frequency Response as a Function of Temperature for A12 1:2 Ratio

Sample : A12 2:3
Size : 34.73 x 12.40 x 2.80 mm
Method : LNCA SUBAMBIENT FILLED
Comment: DMA RESONANT 3 DEGREES C/MINUTE

DMA

File : C:RES.04
Operator: DAVID A. SPIEKER
Run Date: 12-May-92 09:59

Figure G-3. Frequency Response as a Function of Temperature for A12 2:3 Ratio

Sample : EC2216
Size : 34.73 x 12.40 x 2.80 mm
Method : LNCA SUBAMBIENT FILLED
Comment: DMA RESONANT 3 DEGREES C/MINUTE

DMA

File : C: RES.05
Operator: DAVID A. SPIEKER
Run Date: 12-May-92 11:31

Figure G-4. Frequency Response as a Function of Temperature for EC2216

Sample : EA 9309.3NA
Size : 34.73 x 12.40 x 2.80 mm
Method : LNCA SUBAMBIENT FILLED
Comment: DMA RESONANT 3 DEGREES C/MINUTE

DMA

File : C:RES.01
Operator: DAVID A. SPIEKER
Run Date: 11-May-92 11:18

Figure G-5. Frequency Response as a Function of Temperature for EA9309.3 NA

Appendix H

Thermal Stability

Thermal Stability

Test Purpose:

This test will determine the thermal stability of the optical adhesives using Thermogravimetric Analysis.

Background:

Thermogravimetric Analysis (TGA) is an analytical technique which measures weight loss of a material versus temperature at a programmed heating rate. TGA analysis can provide information on organic materials such as thermal stability, decomposition temperature, decomposition kinetics, filler contents and oxidation degradation studies.

The TGA apparatus is a Du Pont 9900 controller with 951 thermogravimetric analyzer which is essentially a very sensitive thermal balance. The sample pan serves as the weight tray and is surrounded with a quartz tube and inserted into a furnace chamber. The furnace heats the sample up to 1000°C.

Sample Preparation:

One sample (of approximately 20 mg) was made and tested from each adhesive per Table A-1 in Appendix A.

Test Procedure:

The samples were tested in air at 10 degrees per minute from room temperature to 700°C. The weight was monitored and recorded. The temperature was noted when the material significantly started outgassing and losing weight. The residue was weighed at the end of the test.

Test Results:

Table H-1 shows the thermal decomposition temperature and the residue (percent filler). The test data are included as Figures H-1 through H-5. The figures note the sample material, initial weight, and rate the test is performed and then plots the percent weight versus the temperature. The thermal decomposition temperature is based on the intersection of lines extended from the 100% line and a line representative of the slope during decomposition. As can be seen from the figures, the material actually begins decomposing at a lower temperature.

Table H-1: Thermal Decomposition Temperature and Residue:

Material	Thermal Decomposition Temperature		Residue (Percent filler)
	(°F)	(°C)	
A12 1:1	698.2	370.1	29.8
A12 1:2	683.6	362.0	30.6
A12 2:3	677.3	358.5	34.5
EC 2216 (gray)	651.9	344.4	30.3
EA 9309.3 NA	604.4	318.0	7.6

Sample: A12 1/1
Size: 11.01 mg
Rate: 10 DEG. C/MIN. IN AIR
Program: TGA Analysis V2.0

TGA

Date: 2-Jan-00 Time: 19:08:17
File: A121-1.01
Operator: KLC
Plotted: 5-Jan-00 19:45:39

Figure H-1. Percent Weight as a Function of Temperature for A12 1:1 Ratio

DuPont 109

Sample: A12 1/2
Size: 11.20 mg
Rate: 10 DEG. C/MIN. IN AIR
Program: TGA Analysis V2.0

TGA

Date: 2-Jan-00 Time: 23:06:26
File: A121-2.01
Operator: KLC
Plotted: 5-Jan-00 19:41:01

Figure H-2. Percent Weight as a Function of Temperature for A12 1:2 Ratio

DuPont 109

Sample: A12 2/3
Size: 15.09 mg
Rate: 10 DEG. C/MIN. IN AIR
Program: TGA Analysis V2.0

TGA

BBB: T7me: 19:47:11
File: A122-3.01
Operator: KLC
Plotted: 5-Jan-00 19:52:52

Figure H-3. Percent Weight as a Function of Temperature for A12 2:3 Ratio

Sample: EA9309.3NA
 Size: 5.60 mg
 Rate: 10 DEG.C/MIN. IN AIR
 Program: TGA Analysis V2.0
 Date: 3-Jan-00
 File: EA9309.01
 Operator: KLC
 Plotted: 3-Jan-00 22:32:16

TGA

Figure H-4. Percent Weight as a Function of Temperature for EC2216

Sample: EC2216
Size: 10.61 mg
Rate: 10 DEG. C/MIN. IN AIR
Program: TGA Analysis V2.0

TGA

Date: 3-Jan-00 Time: 22:37:59
File: EC2216.01
Operator: KLC
Plotted: 5-Jan-00 19:35:47

Figure H-5. Percent Weight as a Function of Temperature for EA9309.3 NA

Appendix I
Specific Heat

Specific Heat

Test Purpose:

The purpose of this test is to look at the specific heat as a function of temperature and also give the kinetics of cure using Differential Scanning Calorimetry (DSC).

Background:

The technique of Differential Scanning Calorimetry (DSC) measures heat flow into (endothermic reaction) or out of (exothermic reaction) a sample as the sample undergoes a change of state, chemical reaction, decomposition or various second order transitions. The temperature at which their changes occur can be accurately measured as well as a quantitative determination of the energy associated with any of their transitions. The DSC can be used to determine specific heat of materials, heat of fusion, heat of reaction, reaction kinetics, and activation energy (reaction) of organic materials.

The DSC apparatus is a DuPont 9900 controller with 912 DSC dual cell and cell base. The DSC accurately measures the temperature at which chemical reactions or transitions occur as well as being able to quantitate the energy associated with reactions or transitions. Samples and an inert reference material are placed on raised sample and reference platforms of a constantan dish, which transfers heat to both sample and reference. Sample temperature is monitored by a chromel-alumel junction on the sample disk. Differential temperature between sample and reference is monitored by chromel-constantan junction formed by the constantan disk and chromel wafer that is in contact with the underside of each raised platform. The calorimetric sensitivity and temperature accuracy are calibrated using metals of known heat of fusion to a sensitivity on 0.01 mW/cm.

Sample Preparation:

Samples were mixed as indicated in Table I-1 and then cured as indicated in the next section.

Table I-1: Adhesive Mix Ratios

Adhesive	Mix Ratio*	
	A Comp.	B Comp.
A12 1:1	1	1
A12 1:2	1	2
A12 2:3	2	3
EC2216 (Gray)	1.4	1
EA9309.3NA	10	2.2

* Material was mixed based on weight, i.e., 10:2.2 would be mixed using 10 grams of A component mixed with 2.2 grams of B component or some factor thereof.

Test Procedure:

Kinetics tests were performed using a TA instruments differential scanning calorimeter. The ASTM E 698 analysis program was used for the runs at the five different heat rates. The average room temperature was 22°C. The samples were cured as follows:

A12:

Five samples, one at each heat rate was cured at 2, 4, 6, 8, and 10 deg.C/min. for each mix ratio.

One sample was tested after setting at room temperature for 24 hours.

Two samples were cured for two hours at 160°F after the 24 hours at room temperature.

One sample was tested after curing for 7 days at room temperature.

EC2216:

Five samples, one at each heat rate, were cured at 2, 4, 6, 8, and 10 deg.C/min.

One sample was tested after setting at room temperature for 16 hours.

Two samples were cured for two hours at 160°F after the 16 hours at room temperature.

One sample was tested after curing for 7 days at room temperature.

EA9309.3NA:

Five samples, one at each heat rate, were cured at 2, 4, 6, 8, and 10 deg.C/min.

One sample was tested after setting at room temperature for 24 hours.

Three samples were cured for 7 days at room temperature.

Test Results:

Figure I-1 shows a set of curves for all the optical adhesives of specific heat as a function of temperature from -55°C to 75°C.

DSC plots at the various heat rates (2,4,6,8, and 10°C) are shown for the EC 2216 in Figures I-2 through I-6. The area under the curve is the Heat of Reaction. The temperature noted at the top of the curve is the temperature at which fifty percent of the reaction has occurred. Figure I-7 shows a plot of the Log of the Heat rate for the EC 2216 versus reciprocal of the temperature for the five different heat rates. This is essentially a data verification check to assure no first order errors. The slope of the curve is the activation energy and the Y intercept is the Frequency Factor (Z). The frequency factor gives the frequency of reactions as a function of temperature. Figure I-8 shows the predicted cure rate at 71°C and room temperature (22°C) for the EC 2216. Eact is the Activation Energy, Log(Z) is the Frequency Factor and Enthalpy is the Heat of Reaction based on the average of five runs. The next curve in Figure I-9 shows the predicted cure rate at room temperature and the actual measured value after 24 hours for the EC 2216. The other adhesives had similar shaped curves. Table I-2 shows a summary of the Heat of Reaction, Activation Energy, and Frequency Factor for all the different optical adhesives

Table I-2: Heat of Reaction, Activation Energy, Frequency Factor

Material	Heat of Reaction (J/gm.)	Activation Energy (KJ/mole)	Frequency Factor (1/min.)
A12 1:1	204.8	57.4	7.372
A12 2:3	140.9	55.9	7.251
A12 1:2	127.9	51.5	6.569
EC 2216 (gray)	164.6	51.1	6.617
EA 9309.3 NA	265.7	48.6	6.346

Figure I-1. Specific Heat as a Function of Temperature for the Optical Adhesives

Sample: EC 2216 Gray
Size: 22.4000 mg
Method: R.T. to 300C at 2C/min.

DSC

File: DOI1.27
Operator: B. Parker
Run Date: 02/17/92 14:36

Figure I-2. Heat Flow as a Function of Temperature at 2°C/min for EC2216

Sample: EC-2216 Gray
Size: 26.7200 mg
Method: R.T. to 300C at 4C/min.

DSC

File: DOI1.23
Operator: B. Parker
Run Date: 02/17/92 12:15

Figure I-3. Heat Flow as a Function of Temperature at 4°C/min for EC2216

Sample: EC 2216 Gray
Size: 24.3200 mg
Method: R.T. to 300C at 6C/min.

DSC

File: DOI1.22
Operator: B. Parker
Run Date: 02/17/92 10:20

Figure I-4. Heat Flow as a Function of Temperature at 6°C/min for EC2216

Sample: EC 2216 Gray
Size: 26.4300 mg
Method: A.T. to 300C at 8C/min.

DSC

File: DOI1.29
Operator: B. Parker
Run Date: 02/18/92 06:13

Figure I-5. Heat Flow as a Function of Temperature at 8°C/min for EC2216

Sample: EC 2216 Gray
Size: 26.7200 mg
Method: R.T. to 300C at 10C/min.

DSC

File: DOI1.25
Operator: B. Parker
Run Date: 02/17/92 13:34

Figure I-6. Heat Flow as a Function of Temperature at 10°C/min for EC2216

Sample : EC 2216 Gray

DSC

Operator: B. Parker

Run Date: 02/17/92

Comment:

Figure I-7. Log of the Heat Rate Versus the Reciprocal of the Temperature for EC2216

Sample : EC 2216 Gray

DSC

Operator: B. Parker

Run Date: 02/17/92

Comment:

Figure I-8. Predicted Cure Rate at 71°C and 22°C for EC2216

Sample : EC 2216 Gray

DSC

Operator: B. Parker

Run Date: 02/17/92

Comment:

Figure I-9. Predicted Cure Rate at 22°C and Measured Value for EC2216

Appendix J

Thermal Conductivity

Thermal Conductivity

Test Purpose:

The purpose of this test is to determine the thermal conductivity of a material using the guarded-comparative-longitudinal heat flow technique on the optical adhesives .

Background:

The testing is per ASTM E 1225 and is included in Appendix L. This test method describes a steady state technique for the determination of the thermal conductivity, X , of homogeneous-opaque solids. This test method is for materials with effective conductivities in the approximate range of $.12 < X < 116$ BTU/hr-ft-°F over the approximate temperature range between -300 and 1550°F which is appropriate for the optical adhesives.

Sample Preparation:

Samples were molded into a two-inch diameter by one-inch tall cylinders in RTV molds and cured per Table A-1 in Appendix A. After curing, a 0.020 inch by 0.020 inch groove was machined on each flat side parallel to each other. A thermal couple was RTV'ed in place in both grooves.

Test Procedure:

The samples were prepared and tested in accordance with ASTM E 1225. The thermal conductivity tests were performed using a Dynatech Model TCFCM-N20 comparative thermal conductivity instrument. A test specimen is inserted under load between two similar specimens of a material of known thermal properties. A temperature gradient is established in the test stack and heat losses minimized by use of a longitudinal guard having approximately the same temperature gradient conditions. At equilibrium conditions, the thermal conductivity is derived in terms of the temperature gradients in the respective specimens and the thermal conductivity of the reference materials.

Test Results:

Table J-1 shows the thermal conductivity at 20°C. Figures J-1 through J-6 show the thermal conductivity as a function of temperature (divide the values by 12 to get the units in BTU/hr-ft-°F). Figure J-6 shows the test data for a pyocerm 9606 reference standard. The deviation between actual and measured thermal conductivity ranged from 0.2 to 2.4 percent high for the measured data.

Table J-1: Thermal Conductivity at 20°C

Material	Thermal Conductivity (at 20°C) BTU/hr-ft-°F
A12 1:1	0.1978
A12 2:3	0.2262
A12 1:2	0.1918
EC 2216 (gray)	0.2468
EA 9309.3 NA	0.1474

Figure J-1. Thermal Conductivity as a Function of Temperature for A12 1:1

Figure J-2. Thermal Conductivity as a Function of Temperature for A12 1:2

Figure J-3. Thermal Conductivity as a Function of Temperature for A12 2:3

Figure J-4. Thermal Conductivity as a Function of Temperature for EC2216

Figure J-5. Thermal Conductivity as a Function of Temperature for EA9309.3 NA

Figure J-6. Pyrocerm 9606 Reference Standard

Appendix K

Shrinkage and Density

Shrinkage and Density

Test Purpose:

The purpose of this test is to determine the shrinkage after curing and the density of the optical adhesives.

Background:

To determine the shrinkage of adhesives after curing one must use caution to get the correct results. Shrinkage determination is made by measuring the mold in which the material will be cured and the dimensions of the final cured specimen. Adhesives, being what they are, have a tendency to bond to the mold, giving erroneous results. Typically, this is corrected by lining the mold with polyethylene film so that the adhesives do not bond to the mold. The film is removed after curing, and the specimens are then measured (the thickness of the film is accounted for in the baseline measurements of the mold). In this determination, an ASTM mold was not available, and RTV silicon tensile molds were used. The RTV silicon mold was flexible and would move with the adhesive while it cured.

The density measurements were performed per ASTM D 792 and is included in Appendix L.

Sample Preparation:

Samples were mixed and cured per Table A-1 in Appendix A.

Test Procedure:

Shrinkage data was obtained using 15 molded tensile test specimens for each formulation. The specimens were approximately 7.018 inches long and 0.752 inch wide and were machined to a final thickness of 0.125 inch. Measurements were made using a digital caliper for length and a digital micrometer for width. The linear shrinkage percent is calculated by dividing the length of the cured material by the length of the mold. Since the final thickness of the tensile specimens were machined to 0.125 inch, the volume shrinkage was calculated by dividing the volume of the sample ($\text{length} \times \text{width}^2$) and dividing by the volume of the mold ($\text{length} \times \text{width}^2$).

Density (or Specific Gravity) measurements were performed per ASTM D 792 test method A. This test method involves weighing a one-piece specimen of one to 50 grams in air and in water (using a sinker with plastics that are lighter than water).

This test method is suitable for plastics that are wet by, but not affected by water. Two samples of each formulation were measured.

Test Results:

Table K-1 shows the results of the shrinkage and density for the cured optical adhesives.

Table K-1: Linear Shrinkage, Volume Shrinkage, Density, and Specific Gravity

Material	Linear shrinkage (percent)	Volume Shrinkage (percent)	Density (lbs./in³)	Specific Gravity
A12 1:1	1.32	4.20	0.0497	1.375
A12 2:3	1.27	4.35	0.0491	1.360
A12 1:2	1.26	3.83	0.0484	1.340
EC 2216 (gray)	1.40	4.06	0.0493	1.366
EA 9309.3 NA	1.12	4.30	0.0423	1.170

Appendix L
ASTM Standards

Standard Test Method for Tensile Strength of Molded Electrical Insulating Materials¹

This standard is issued under the fixed designation D 651; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

The committee responsible for this standard has voted its withdrawal. In the absence of substantial reasons that it should be continued, the Society will approve withdrawal from publication in June 1991.

1. Scope

1.1 This test method covers the general procedure for determining the tensile strength of various molded electrical insulating materials. However, modifications in the procedure may be desirable for different types of such materials, and existing ASTM methods covering the requirements for testing a given material should be consulted for special details that may apply.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing²

D 638 Test Method for Tensile Properties of Plastics²

D 647 Practice for Design of Molds for Test Specimens of Plastic Molding Materials²

D 731 Test Method for Molding Index of Thermosetting Molding Powder²

D 1896 Practice for Transfer Molding Specimens of Thermosetting Compounds³

D 3123 Test Method for Spiral Flow of Low-Pressure Thermosetting Molding Compounds⁴

3. Apparatus

3.1 Any universal testing machine may be used, provided it is accurate to 1% of the lowest load to be applied. Special specimen holders made of steel, as shown in Fig. 1, shall be used for such test specimens as are of a shape to fit them. The specimen holders shall be attached to the heads of the testing machine by links held so that the pull is central, to avoid any

transverse strain. Equally suitable holders, suitably mounted, shall be used for specimens of other shapes.

4. Test Specimen

4.1 *Compression Molded*—Unless otherwise required, the test specimen for hot-molded and plastic cold-molded materials shall be molded to the form and dimensions shown in Fig. 2, the use of either the 1/8-in. (3.2-mm) or the 1/4-in. (6.3-mm) specimen being permissible. Values for tensile strength obtained with these two specimens may be unequal, and in such cases the specimen which is considered most representative of the material and its intended use shall be tested.

4.2 *Transfer or Injection Molded*—Soft flow materials (see Test Methods D 731 and D 3123) shall be molded to the dimensions of Fig. 1 in Test Method D 638.

5. Mold

5.1 *Compression Molded*—The specimens shall be molded in molds conforming to Fig. 3 of Practice D 647. The pressure and other molding conditions used in molding test specimens shall be agreed upon by the seller and the purchaser.

5.2 *Transfer or Injection Molded*—The specimens shall be molded in molds conforming to the tension cavity shown in Fig. 7 of Practice D 647. The pressure and other molding conditions used in molding test specimens shall be agreed upon by the seller and the purchaser.

6. Conditioning

6.1 Unless otherwise specified, all specimens prior to testing shall be conditioned in accordance with Methods D 618, the procedure used to be agreed upon between the supplier and purchaser.

7. Test Conditions

7.1 Tensile strength tests for materials conditioned (Section 6) by Procedure A shall be performed at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$) and $50 \pm 2\%$ relative humidity. For materials conditioned by Procedure B, these tests shall be performed at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$) and the humidity prevailing in the laboratory. If, because of lack of equipment, materials conditioned by Procedures A and B cannot be tested under controlled humidity, the effect of exposure between conditioning the testing shall be established and reported.

¹ This test method is under the jurisdiction of ASTM Committee D-9 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.07 on Flexible and Rigid Insulating Materials.

Current edition approved Dec. 28, 1984. Published February 1985. Originally published as D 651 - 41 T. Last previous edition D 651 - 80.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 08.03.

FIG. 1 Specimen Holder

NOTE—To permit the use of one mold for all molding compounds, the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds	±5 %
For cold-molded compounds	±10 %

FIG. 2 Tension Test Specimen

8. Procedure

8.1 Five conditioned specimens shall be tested for each type of molded material, each specimen being properly aligned in the holders and broken. The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. (1.27 mm)/min with the machine running idle.

9. Report

9.1 The report shall include the following:

9.1.1 The breaking load of each specimen in pounds or kilograms.

9.1.2 The thickness and width of each specimen in inches or millimetres, as measured by a micrometer at the center of the specimen, that is, the point of minimum section.

9.1.3 The ultimate tensile strength in pounds-force per square inch or in mega-pascals of each specimen, calculated from the minimum area measured at the center of the test specimen before the load is applied.

9.1.4 The character of the material tested, with a description of the fracture and its location with respect to the middle point of the specimen.

9.1.5 The speed in inches or millimetres per minute at which the head of the testing machine traveled during the test.

9.1.6 The elongation in inches or millimetres if determined.

9.1.7 Conditioning procedure prior to testing, and

9.1.8 In the case of tests performed after conditioning but not in a controlled atmosphere, reference shall be made to the availability of data showing the effects of exposure between conditioning and testing, and the test conditions observed.

10. Precision and Bias

10.1 The precision of this method has not been determined. A statement of bias is unavailable in view of the lack of a standard reference material for this property.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Compressive Properties of Rigid Plastics¹

This standard is issued under the fixed designation D 695; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 1021. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the mechanical properties of unreinforced and reinforced rigid plastics, including high-modulus composites, when loaded in compression at relatively low uniform rates of straining or loading. Test specimens of standard shape are employed.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

NOTE 1—A complete metric companion to Test Method D 695 has been developed—D 695M.

NOTE 2—For compressive properties of resin-matrix composites reinforced with oriented continuous, discontinuous, or cross-ply reinforcements, tests may be made in accordance with Test Method D 3410.

1.3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing²

D 638 Test Method for Tensile Properties of Plastics²

D 695M Test Method for Compressive Properties of Rigid Plastics [Metric]²

D 3410 Test Method for Compressive Properties of Unidirectional or Crossply Fiber-Resin Composites³

D 4066 Specification for Nylon Injection and Extrusion Materials⁴

E 4 Practices for Load Verification of Testing Machines⁵

E 83 Practice for Verification and Classification of Extensometers⁵

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of Test Method⁶

3. Terminology

3.1 Definitions:

3.1.1 *compressive deformation*—the decrease in length

produced in the gage length of the test specimen by a compressive load. It is expressed in units of length.

3.1.2 *compressive strain*—the ratio of compressive deformation to the gage length of the test specimen, that is, the change in length per unit of original length along the longitudinal axis. It is expressed as a dimensionless ratio.

3.1.3 *compressive strength*—the maximum compressive stress (nominal) carried by a test specimen during a compression test. It may or may not be the compressive stress (nominal) carried by the specimen at the moment of rupture.

3.1.4 *compressive strength at failure (nominal)*—the compressive stress (nominal) sustained at the moment of failure of the test specimen if shattering occurs.

3.1.5 *compressive stress (nominal)*—the compressive load per unit area of minimum original cross section within the gage boundaries, carried by the test specimen at any given moment. It is expressed in force per unit area.

NOTE 3—The expression of compressive properties in terms of the minimum original cross section is almost universally used. Under some circumstances the compressive properties have been expressed per unit of prevailing cross section. These properties are called "true" compressive properties.

3.1.6 *compressive stress-strain diagram*—a diagram in which values of compressive stress are plotted as ordinates against corresponding values of compressive strain as abscissas.

3.1.7 *compressive yield point*—the first point on the stress-strain diagram at which an increase in strain occurs without an increase in stress.

3.1.8 *compressive yield strength*—normally the stress at the yield point (see also 3.1.1).

3.1.9 *crushing load*—the maximum compressive force applied to the specimen, under the conditions of test, that produces a designated degree of failure.

3.1.10 *modulus of elasticity*—the ratio of stress (nominal) to corresponding strain below the proportional limit of a material. It is expressed in force per unit area based on the average initial cross-sectional area.

3.1.11 *offset compressive yield strength*—the stress at which the stress-strain curve departs from linearity by a specified percentage of deformation (offset).

3.1.12 *percentage compressive strain*—the compressive deformation of a test specimen expressed as a percentage of the original gage length.

3.1.13 *proportional limit*—the greatest stress that a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law). It is expressed in force per unit area.

3.1.14 *slenderness ratio*—the ratio of the length of a

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 15.03.

⁴ Annual Book of ASTM Standards, Vol 08.03.

⁵ Annual Book of ASTM Standards, Vol 03.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

column of uniform cross section to its least radius of gyration. For specimens of uniform rectangular cross section, the radius of gyration is 0.289 times the smaller cross-sectional dimension. For specimens of uniform circular cross section, the radius of gyration is 0.250 times the diameter.

4. Significance and Use

4.1 Compression tests provide information about the compressive properties of plastics when employed under conditions approximating those under which the tests are made.

4.2 Compressive properties include modulus of elasticity, yield stress, deformation beyond yield point, and compressive strength (unless the material merely flattens but does not fracture). Materials possessing a low order of ductility may not exhibit a yield point. In the case of a material that fails in compression by a shattering fracture, the compressive strength has a very definite value. In the case of a material that does not fail in compression by a shattering fracture, the compressive strength is an arbitrary one depending upon the degree of distortion that is regarded as indicating complete failure of the material. Many plastic materials will continue to deform in compression until a flat disk is produced, the compressive stress (nominal) rising steadily in the process, without any well-defined fracture occurring. Compressive strength can have no real meaning in such cases.

4.3 Compression tests provide a standard method of obtaining data for research and development, quality control, acceptance or rejection under specifications, and special purposes. The tests cannot be considered significant for engineering design in applications differing widely from the load-time scale of the standard test. Such applications require additional tests such as impact, creep, and fatigue.

5. Apparatus

5.1 *Testing Machine*—Any suitable testing machine capable of control of constant-rate-of-crosshead movement and comprising essentially the following:

5.1.1 *Drive Mechanism*—A drive mechanism for imparting to the cross-head movable member, a uniform, controlled velocity with respect to the base (fixed member), this velocity to be regulated as specified in Section 9.

5.1.2 *Load Indicator*—A load-indicating mechanism capable of showing the total compressive load carried by the test specimen. The mechanism shall be essentially free from inertia-lag at the specified rate of testing and shall indicate the load with an accuracy of $\pm 1\%$ of the maximum indicated value of the test (load). The accuracy of the testing machine shall be verified at least once a year in accordance with Practices E 4.

5.2 *Compressometer*—A suitable instrument for determining the distance between two fixed points on the test specimen at any time during the test. It is desirable that this instrument automatically record this distance (or any change in it) as a function of the load on the test specimen. The instrument shall be essentially free of inertia-lag at the specified rate of loading and shall conform to the requirements for a Class B-2 extensometer as defined in Practice E 83.

NOTE 4—The requirements for extensometers cited herein apply to compressometers as well.

NOTE—Devices similar to the one illustrated have been successfully used in a number of different laboratories. Details of the device developed at the National Bureau of Standards are given in the paper by Aitchinson, C. S., and Miller, J. A., "A Subpress for Compressive Tests," Natl. Advisory Committee for Aeronautics, Technical Note No. 912, 1943.

FIG. 1 Subpress for Compression Tests

FIG. 2 Compression Tool

5.3 *Compression Tool*—A compression tool for applying the load to the test specimen. This tool shall be so constructed that loading is axial within 1:1000 and applied through surfaces that are flat within 0.025 mm (0.001 in.) and parallel to each other in a plane normal to the vertical loading axis. Examples of suitable compression tools are shown in Figs. 1 and 2.

5.4 *Supporting Jig*—A supporting jig for thin specimens is shown in Figs. 3 and 4.

5.5 *Micrometers*—Suitable micrometers, reading to 0.01

FIG. 3 Support Jig for This Specimen

mm or 0.001 in. for measuring the width, thickness, and length of the specimens.

6. Test Specimens

6.1 Unless otherwise specified in the materials specifications, the specimens described in 6.2 and 6.7 shall be used. These specimens may be prepared by machining operations from materials in sheet, plate, rod, tube, or similar form, or they may be prepared by compression or injection molding of the material to be tested. All machining operations shall be done carefully so that smooth surfaces result. Great care shall be taken in machining the ends so that smooth, flat parallel surfaces and sharp, clean edges to within 0.025 mm (0.001 in.) perpendicular to the long axis of the specimen, result.

6.2 The standard test specimen, except as indicated in 6.3 to 6.7, shall be in the form of a right cylinder or prism whose length is twice its principal width or diameter. Preferred specimen sizes are 12.7 by 12.7 by 25.4 mm (0.50 by 0.50 by 1 in.) (prism), or 12.7 mm (0.50 in.) in diameter by 25.4 mm (1 in.) (cylinder). Where elastic modulus and offset yield-

stress data are desired, the test specimen shall be of such dimensions that the slenderness ratio is in the range from 11 to 16:1. In this case, preferred specimen sizes are 12.7 by 12.7 by 50.8 mm (0.50 by 0.50 by 2 in.) (prism), or 12.7 mm (0.50 in.) in diameter by 50.8 mm (2 in.) (cylinder).

6.3 For rod material, the test specimen shall have a diameter equal to the diameter of the rod and a sufficient length to allow a specimen slenderness ratio in the range from 11 to 16:1.

6.4 When testing tubes, the test specimen shall have a diameter equal to the diameter of the tube and a length of 25.4 mm (1 in.) (Note 5). For crushing-load determinations (at right angles to the longitudinal axis), the specimen size shall be the same, with the diameter becoming the height.

NOTE 5—This specimen can be used for tubes with a wall thickness of 1 mm (0.039 in.) or over, to inside diameters of 6.4 mm (0.25 in.) or over, and to outside diameters of 50.8 mm (2.0 in.) or less.

6.5 Where it is desired to test conventional high-pressure laminates in the form of sheets, the thickness of which is less than 25.4 mm (1 in.), a pile-up of sheets 25.4 mm (1 in.) square, with a sufficient number of layers to produce a height of at least 25.4 mm (1 in.), may be used.

6.6 When testing material that may be suspected of anisotropy, duplicate sets of test specimens shall be prepared having their long axis respectively parallel with and normal to the suspected direction of anisotropy.

6.7 *Reinforced Plastics, Including High-Strength Composites and High-Strength Composites and Highly Orthotropic Laminates*—The following specimens shall be used for reinforced materials, or for other materials when necessary to comply with the slenderness ratio requirements or to permit attachment of a deformation-measuring device.

6.7.1 For materials 3.2 mm (1/8 in.) and over in thickness, a specimen shall consist of a prism having a cross section of 12.7 mm (1/2 in.) by the thickness of the material and a length such that the slenderness ratio is in the range from 11 to 16:1 (Note 6).

NOTE 1—Cold rolled steel.

NOTE 2—Furnished four steel machine screws and nuts, round head, slotted, length 31.75 mm (1 1/4 in.).

NOTE 3—Grind surfaces denoted "Gr."

FIG. 4 Support Jig, Details

FIG. 5 Compression Test Specimen for Materials Less than 3.2 mm Thick

6.7.2 For materials under 3.2 mm ($\frac{1}{8}$ in.) thick, or where elastic modulus testing is required and the slenderness ratio does not provide for enough length for attachment of a compressometer or similar device, a specimen conforming to that shown in Fig. 6 shall be used. The supporting jig shown in Figs. 3 and 4 shall be used to support the specimen during test (Note 7).

NOTE 6—If failure for materials in the thickness range of 3.2 mm ($\frac{1}{8}$ in.) is by delamination rather than by the desirable shear plane fracture, the material may be tested in accordance with 6.7.2.

NOTE 7—Round-robin tests have established that relatively satisfactory measurements of modulus of elasticity may be obtained by applying a compressometer to the edges of the jig-supported specimen.

6.8 When testing syntactic foam, the standard test specimen shall be in the form of a right cylinder 25.4 mm (1 in.) in diameter by 50.8 mm (2 in.) in length.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for not less than 40 h prior to test in accordance with Procedure A of Methods D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and $\pm 2\%$ relative humidity.

7.1.1 Note that for some hygroscopic materials, such as nylons, the material specifications (for example, Specification D 4066) call for testing “dry as-molded specimens.” Such requirements take precedence over the above routine preconditioning to 50 % RH and require sealing the specimens in water vapor-impermeable containers as soon as molded and not removing them until ready for testing.

7.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, unless otherwise specified in the test methods. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and $\pm 2\%$ relative humidity.

8. Number of Test Specimens

8.1 At least five specimens shall be tested for each sample in the case of isotropic materials.

8.2 Ten specimens, five normal to and five parallel with the principal axis of anisotropy, shall be tested for each sample in the case of anisotropic materials.

8.3 Specimens that break at some obvious fortuitous flow

shall be discarded and retests made, unless such flaws constitute a variable, the effect of which it is desired to study.

9. Speed of Testing

9.1 Speed of testing shall be the relative rate of motion of the grips or test fixtures during the test. Rate of motion of the driven grip or fixture when the machine is running idle may be used if it can be shown that the resulting speed of testing is within the limits of variation allowed.

9.2 The standard speed of testing shall be 1.3 ± 0.3 mm (0.050 ± 0.010 in.)/min, except as noted in 10.5.4.

10. Procedure

10.1 Measure the width and thickness of the specimen to the nearest 0.01 mm (0.001 in.) at several points along its length. Calculate and record the minimum value of the cross-sectional area. Measure the length of the specimen and record the value.

10.2 Place the test specimen between the surfaces of the compression tool, taking care to align the center line of its long axis with the center line of the plunger and to ensure that the ends of the specimen are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the compression tool plunger.

NOTE 8—The compression tool may not be necessary for testing of lower modulus (for example, 100 000 to 500 000 psi) material if the loading surfaces are maintained smooth, flat, and parallel to the extent that buckling is not incurred.

10.3 Place thin specimens in the jig (Figs. 3 and 4) so that they are flush with the base and centered (Note 9). The nuts or screws on the jig shall be finger tight (Note 10). Place the assembly in the compression tool as described in 5.3.

NOTE 9—A round-robin test, designed to assess the influence of specimen positioning in the supporting jig (that is, flush versus centered mounting), showed no significant effect on compressive strength due to this variable. However, flush mounting of the specimen with the base of the jig is specified for convenience and ease of mounting. Substantiating data are filed at ASTM Headquarters (RR:D20-1061).

NOTE 10—A round-robin test on the effect of lateral pressure at the supporting jig has established that reproducible data can be obtained with the tightness of the jig controlled as indicated.

10.4 If only compressive strength or compressive yield strength, or both, are desired, proceed as follows:

10.4.1 Set the speed control at 1.3 mm/min (0.050 in./min) and start the machine.

10.4.2 Record the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture).

10.5 If stress-strain data are desired, proceed as follows:

10.5.1 Attach compressometer.

10.5.2 Set the speed control at 1.3 mm/min (0.050 in./min) and start the machine.

10.5.3 Record loads and corresponding compressive strain at appropriate intervals of strain or, if the test machine is equipped with an automatic recording device, record the complete load-deformation curve.

10.5.4 After the yield point has been reached, it may be desirable to increase the speed from 5 to 6 mm/min (0.20 to 0.25 in./min) and allow the machine to run at this speed until the specimen breaks. This may be done only with relatively ductile materials and on a machine with a weighing system with response rapid enough to produce accurate results.

11. Calculation

11.1 *Compressive Strength*—Calculate the compressive strength by dividing the maximum compressive load carried by the specimen during the test by the original minimum cross-sectional area of the specimen. Express the result in megapascals or pounds-force per square inch and report to three significant figures.

11.2 *Compressive Yield Strength*—Calculate the compressive yield strength by dividing the load carried by the specimen at the yield point by the original minimum cross-sectional area of the specimen. Express the result in megapascals or pounds-force per square inch and report to three significant figures.

11.3 *Offset Yield Strength*—Calculate the offset yield strength by the method referred to in 4.10.

11.4 *Modulus of Elasticity*—Calculate the modulus of elasticity by drawing a tangent to the initial linear portion of the load deformation curve, selecting any point on this straight line portion, and dividing the compressive stress represented by this point by the corresponding strain, measure from the point where the extended tangent line intersects the strain-axis. Express the result in gigapascals or pounds-force per square inch and report to three significant figures (See Annex A1).

11.5 For each series of tests, calculate to three significant figures the arithmetic mean of all values obtained and report as the "average value" for the particular property in question.

11.6 Calculate the standard deviation (estimated) as follows and report to two significant figures:

TABLE 1 Precision, Compressive Strength
(Values are in Units of Megapascals)

Material	Average	S_r^A	S_R^B	r^C	R^D
Acetal	100	1.1	2.1	3.1	5.9
Polystyrene	106	1.4	3.5	3.9	9.8
Linen Filled Phenolic	158	3.7	7.5	10.4	21.0

^A S_r is the within-laboratory standard deviation of the average.

^B S_R is the between-laboratory standard deviation of the average.

^C $r = 2.8 S_r$

^D $R = 2.8 S_R$

TABLE 2 Precision, Compressive Modulus
(Values are in Units of Megapascals)

Material	Average	S_r^A	S_R^B	r^C	R^D
Acetal	3.28	0.14	0.25	0.39	0.70
Polystyrene	3.88	0.07	0.74	0.20	2.07
Linen Filled Phenolic	6.82	0.23	0.90	0.64	2.52

^A S_r is the within-laboratory standard deviation of the average.

^B S_R is the between-laboratory standard deviation of the average.

^C $r = 2.8 S_r$

^D $R = 2.8 S_R$

$$s = \sqrt{(\sum X^2 - n\bar{X}^2)/(n-1)}$$

where:

s = estimated standard deviation,

X = value of single observation,

n = number of observations, and

$\bar{X}$ = arithmetic mean of the set of observations.

NOTE 11—The method for determining the offset compressive yield strength is similar to that described in the Annex of Test Method D 638.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, etc.,

12.1.2 Method of preparing test specimens,

12.1.3 Type of test specimen and dimensions,

12.1.4 Conditioning procedure used,

12.1.5 Atmospheric conditions in test room,

12.1.6 Number of specimens tested,

12.1.7 Speed of testing,

12.1.8 Compressive strength, average value, and standard deviation,

12.1.9 Compressive yield strength and offset yield strength average value, and standard deviation, when of interest,

12.1.10 Modulus of elasticity in compression (if required), average value, standard deviation, and

12.1.11 Date of test.

13. Precision and Bias⁷

13.1 Tables 1 and 2 are based on a round robin conducted in 1987 per Practice E 691, involving three materials tested by six laboratories for D 695M. Since the test parameters overlap within tolerances and the test values are normalized, the same data is used for both methods. For each material, all of the samples were prepared at one source. Each test result was the average of five individual determinations. Each lab obtained two test results for each material.

NOTE 12—Caution: The following explanations of r and R (13.2 through 13.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 1 and 2 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory, and materials or between specific laboratories. The principles of 13.2 through 13.2.3 would then be valid for such data.

13.2 *Concept of r and R* —If $S(r)$ and $S(R)$ have been

⁷ Supporting data are available from ASTM Headquarters. Request RA D-20-1150.

calculated from a large enough body of data, and for test results that were averages from testing five specimens:

13.2.1 *Repeatability, r* —Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day. The two test results should be judged not equivalent if they differ by more than the r value for that material.

13.2.2 *Reproducibility, R* —Comparing two results for the

same material, obtained by different operators using different equipment on different days. The two test results should be judged not equivalent if they differ by more than R value for that material.

13.2.3 Any judgement per 13.2.1 and 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 There are no recognized standards by which to estimate bias of this test method.

ANNEX

(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, AC , that does not represent a property of the material. It is an artifact caused by a takeup of slack, and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

A1.2 In the case of a material exhibiting a region of Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (CD) region of the curve is constructed through the zero-stress axis. This intersection (B) is the corrected zero-strain point from which all extensions or strains must be measured, including the yield offset (BE), if applicable. The elastic modulus can be determined by dividing the stress at

any point along the line CD (or its extension) by the strain at the same point (measured from point B , defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (H'). This is extended to intersect the strain axis at point B' , the corrected zero-strain point. Using point B' as zero strain, the stress at any point (G') on the curve can be divided by the strain at that point to obtain a secant modulus (slope of line $B'G'$). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.

NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region

NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement¹

This standard is issued under the fixed designation D 792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace method 5011 and 5012 of Federal Test Method Standard 406 and 14011 and 14021 of FTMS 601, and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These test methods describe the determination of the specific gravity (relative density) and density of solid plastics in forms such as sheets, rods, tubes, or molded items.

1.2 Two test methods are described:

1.2.1 *Test Method A*—For testing solid plastics in water, and

1.2.2 *Test Method B*—For testing solid plastics in liquids other than water.

NOTE 1—Alternatively, Test Method D 1505 may be applied to many such forms, as well as to films and sheeting.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing²

D 891 Test Methods for Specific Gravity of Liquid Industrial Chemicals³

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique⁴

D 1622 Test Method for Apparent Density of Rigid Cellular Plastics⁵

D 1898 Practice for Sampling of Plastics⁶

E 1 Specification for ASTM Thermometers⁶

E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases⁷

¹ These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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² *Annual Book of ASTM Standards*, Vols 08.01, 10.01, and 10.02.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ *Annual Book of ASTM Standards*, Vol 08.01.

⁵ *Annual Book of ASTM Standards*, Vol 08.02.

⁶ *Annual Book of ASTM Standards*, Vols 05.03 and 14.03.

⁷ *Annual Book of ASTM Standards*, Vols 04.02 and 15.05.

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods⁸

3. Definitions

3.1 *specific gravity (relative density)*—the ratio of the weight in air of a unit volume of the impermeable portion of the material at 23°C (73.4°F) to the weight in air of equal density of an equal volume of gas-free distilled water at the same temperature. The form of expression shall be:

$$\text{Specific gravity (relative density) } 23/23^{\circ}\text{C} \\ (\text{or sp gr } 23/23^{\circ}\text{C})$$

NOTE 2—This definition is essentially equivalent to the definition for apparent specific gravity and apparent density in Definitions E 12, because the small percentage difference introduced by not correcting for the buoyancy of air is insignificant for most purposes.

3.2 *density*—the weight in air in milligrams per cubic metre of impermeable portion of the material at 23°C. The form of expression shall be:

$$D^{23}, \text{ mg/m}^3 \text{ (Notes 2, 3, 4)}$$

NOTE 3—Densities in other units may be obtained, when desired, by multiplying by appropriate factors.

NOTE 4—Specific gravity 23/23°C can be converted to density 23°C, mg/m³, by use of the following equation:

$$D^{23} \text{ } ^{\circ}\text{C, mg/m}^3 = \text{sp gr } 23/23^{\circ}\text{C} \times 0.9975$$

4. Summary of Test Methods

4.1 A specimen of the solid plastic is weighed in air. It is then immersed in a liquid, its loss in weight upon immersion is determined, and its specific gravity (relative density) calculated.

5. Significance and Use

5.1 The specific gravity or density of a solid is a property that can be measured conveniently to identify a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or specimens, or to indicate the average density of a large item.

5.2 Changes in density of a single specimen may be due to changes in crystallinity, loss of plasticizer, absorption of solvent, or to other causes. Portions of a sample may differ in density because of difference in crystallinity, thermal history, porosity, and composition (types or proportions of resin, plasticizer, pigment, or filler).

NOTE 5—Reference is made to Test Method D 1622.

⁸ *Annual Book of ASTM Standards*, Vols 06.03, 08.03, and 14.02.

5.3 Density is useful for calculating strength-weight and cost-weight ratios.

6. Sampling

6.1 The sampling units used for the determination of specific gravity (relative density) shall be representative of the quantity of product for which the data are required, in accordance with Practice D 1898.

6.1.1 If it is known or suspected that the sample consists of two or more layers or sections having different specific gravities, either complete finished parts or complete cross sections of the parts or shapes shall be used as the specimens, or separate specimens shall be taken and tested from each layer. The specific gravity (relative density) of the total part cannot be obtained by adding the specific gravity of the layers, unless relative percentages of the layers are taken into account.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for not less than 40 h prior to test in accordance with Procedure A of Methods D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and $\pm 2\%$ relative humidity.

7.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and $\pm 2\%$ relative humidity.

TEST METHOD A FOR TESTING SOLID PLASTICS IN WATER (SPECIMENS 1 TO 50 g)

8. Scope

8.1 This test method involves weighing a one-piece specimen of 1 to 50 g in water, using a sinker with plastics that are lighter than water. This test method is suitable for plastics that are wet by, but otherwise not affected by water.

9. Apparatus

9.1 *Analytical Balance*—A balance with a precision within 0.1 mg, accuracy within 0.05 % relative (that is, 0.05 % of the weight of the specimen in air), and equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

NOTE 6—Assurance that the balance meets the performance requirements should be provided by frequent checks on adjustments of zero point and sensitivity and by periodic calibration for absolute accuracy, using standard weights.

9.2 *Wire*—A corrosion-resistant wire for suspending the specimen.

9.3 *Sinker*—A sinker for use with specimens of plastics that have specific gravities less than 1.000. The sinker shall: (1) be corrosion-resistant; (2) have a specific gravity of not less than 7.0; (3) have smooth surfaces and a regular shape; and (4) be slightly heavier than necessary to sink the specimen. The sinker should have an opening to facilitate attachment to the specimen and wire.

9.4 *Immersion Vessel*—A beaker or other wide-mouthed vessel for holding the water and immersed specimen.

9.5 *Thermometer*—A thermometer with an accuracy of $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$) is required if the test is not performed in the Standard Laboratory Atmosphere of Methods D 618, (compare 16.4).

10. Materials

10.1 *Water*—The water shall be substantially air-free, distilled, or demineralized water.

NOTE 7—Water may be rendered substantially air-free by boiling and cooling or by shaking under vacuum in a heavy-walled vacuum flask. (Precaution: Use gloves and shielding.) If the water does not wet the specimen, a few drops of a wetting agent shall be added. If this solution does not wet the specimen, Method B shall be used.

11. Test Specimens

11.1 The test specimen shall be a single piece of the material under test of any size and shape that can conveniently be prepared and tested, provided that its volume shall be not less than 1 cm^3 (0.06 in.^3), and its surface and edges shall be made smooth. The thickness of the specimen should be at least 1 mm (0.04 in.) for each 1 g of weight. A specimen weighing 1 to 5 g usually will be found convenient, but specimens up to approximately 50 g may be used (Note 8). Care should be taken in cutting specimens to avoid changes in density resulting from compressive stresses or frictional heating.

NOTE 8—Specifications for certain plastics require a particular method of specimen preparation and should be consulted if applicable.

11.2 The specimen shall be free from oil, grease, and other foreign matter.

12. Procedure

12.1 Weigh the specimen in air to the nearest 0.1 mg for specimens weighing 1 to 10 g or to the nearest mg for specimens weighing more than 10 to 50 g.

12.2 Attach to the balance a piece of fine wire sufficiently long to reach from the hook above the pan to the support for the immersion vessel. Attach the specimen to the wire such that it is suspended about 2.5 cm (1 in.) above the vessel support.

NOTE 9—The specimen may be weighed in air after hanging from the wire. In this case, record the weight of the specimen, $a = (\text{weight of specimen} + \text{wire, in air}) - (\text{weight of wire in air})$.

12.3 Mount the immersion vessel on the support, and completely immerse the suspended specimen (and sinkers, if used) in water (10.1) at a temperature of $23 \pm 2^\circ\text{C}$. The vessel must not touch wire or specimen. Remove any bubbles adhering to the specimen, wire, or sinker, paying particular attention to holes in the specimen and sinker. Usually these bubbles can be removed by rubbing them with another wire. If the bubbles cannot be removed by this method or if bubbles are continuously formed (as from dissolved gases), the use of vacuum is recommended (Note 11). Weigh the suspended specimen to the required precision (12.1) (Note 10). Record this weight as b (the weight of the specimen, sinker, if used, and the partially immersed wire in liquid). Unless otherwise specified, weigh rapidly in order to minimize absorption of water by the specimen.

NOTE 10—It may be necessary to change the sensitivity adjustment of the balance to overcome the damping effect of the immersed specimen.

NOTE 11—Some specimens may contain absorbed or dissolved gases, or irregularities which tend to trap air bubbles; any of these may affect the density values obtained. In such cases, the immersed specimen may be subjected to vacuum in a separate vessel until evolution of bubbles has substantially ceased before weighing (see Test Method B). It must also be demonstrated that the use of this technique leads to results of the required degree of precision.

12.4 Weigh the wire (and sinker, if used) in water with immersion to the same depth as used in the previous step (Notes 12 and 13). Record this height as w (weight of the wire in liquid).

NOTE 12—It is convenient to mark the level of immersion by means of a shallow notch filed in the wire. The finer the wire, the greater the tolerance which may be permitted in adjusting the level of immersion between weighings. With wire Awg No. 36 or finer, disregard its degrees of immersion and, if no sinker is used, use the weight of the wire in air as w .

NOTE 13—If the wire is left attached to the balance arm during a series of determinations, the weight a may be determined either with the aid of a tare on the other arm of the balance or as in Note 11. In such cases, care must be taken that the change of weight of the wire (for example, from visible water) between readings does not exceed the desired precision.

12.5 Repeat the procedure for the required number of specimens. Two specimens per sample are recommended. Determine acceptability of number of replicate test specimens by comparing results with precision data given in Tables 1 and 2 of Section 23. Additional specimens may be required to give the desired precision.

13. Calculation

13.1 Calculate the specific gravity of the plastic as follows:

$$Sp\ gr\ 23/23^{\circ}C = a/(a + w - b)$$

where:

a = apparent weight of specimen, without wire or sinker, in air,

b = apparent weight of specimen (and of sinker, if used) completely immersed and of the wire partially immersed in liquid, and

w = apparent weight of totally immersed sinker (if used) and of partially immersed wire.

13.2 Calculate the density of the plastic as follows:

$$D^{23^{\circ}C},\ mg/m^3 = sp\ gr\ 23/23^{\circ}C \times 0.9975$$

14. Report

14.1 The report shall include the following:

14.1.1 Complete identification of the material or product tested, including method of specimen preparation and conditioning.

14.1.2 Average specific gravity (relative density) for all specimens from a sampling unit, reported as $sp\ gr\ 23/23^{\circ}C = \text{---}$, or average density reported as $D^{23^{\circ}C} = \text{---}\ g/cm^3$,

14.1.3 A measure of the degree of variation of specific gravity or density within the sampling unit such as the

standard deviation and number of determinations on a homogeneous material or the averages plus these measures of dispersion on different layers or areas of a nonhomogeneous product,

14.1.4 Any evidence of porosity of the material or specimen,

14.1.5 The method of test (Method A of Methods D 792), and

14.1.6 Date of test.

15. Precision and Bias

15.1 See Section 23.

TEST METHOD B FOR TESTING SOLID PLASTICS IN LIQUIDS OTHER THAN WATER (SPECIMENS 1 TO 50 g)

16. Scope

16.1 Test Method B uses a liquid other than water for testing one-piece specimens, 1 to 50 g, of plastics that are affected by water or which are lighter than water.

17. Apparatus

17.1 The apparatus shall include the balance, wire, and immersion vessel of Section 8, and, optionally, the following:

17.2 *Pycnometer with Thermometer*—A 25-mL specific gravity bottle with thermometer, or

17.3 *Pycnometer*—A pycnometer of the Weld type, preferably with a capacity of about 25 mL and an external cap over the stopper.

17.4 *Thermometer*—A thermometer having not fewer than four divisions per $^{\circ}C$ or two divisions per $^{\circ}F$ over a temperature range of not less than $5^{\circ}C$ or $10^{\circ}F$ above and below the standard temperature, and having an ice point for calibration. A thermometer short enough to be handled inside the balance case will be found convenient. ASTM Thermometer 23C (see Specification E 1) and Anschütz-type thermometers have been found satisfactory for this purpose.

17.5 *Constant-Temperature Bath*—An appropriate constant-temperature bath adjusted to maintain a temperature of $23 \pm 0.1^{\circ}C$.

18. Materials

18.1 *Immersion Liquid*—The liquid used shall not dissolve, swell, or otherwise affect the specimen, but should wet it and should have a specific gravity less than that of the specimen. In addition, the immersion liquid should be nonhygroscopic, have a low vapor pressure, a low viscosity, and a high flash point, and should leave little or no waxy or tarry residue on evaporation. A narrow cut distilled from kerosine meets these requirements for many plastics. The specific gravity $23/23^{\circ}C$ of the immersion liquid shall be determined shortly before and after each use in this method to a precision of at least 0.1 % relative, unless it has been

TABLE 1 Test Method A Tested in Water

Material	Mean	S_r	S_N	I_r	I_N
Polypropylene	0.9007	0.00196	0.00297	0.00555	0.00841
Cellulose Acetate Butyrate	1.1973	0.00232	0.00304	0.00657	0.00860
Polyphenylene Sulfide	1.1708	0.00540	0.00738	0.01528	0.02089
Thermoset	1.3136	0.00271	0.00313	0.00767	0.02171
Polyvinyl Chloride	1.3396	0.00243	0.00615	0.00688	0.01947

TABLE 2 Test Method B Tested in Liquids Other Than Water

Material	Mean	S_p	S_R	I_p	I_R
Polypropylene	0.9023	0.00139	0.00239	0.00393	0.00678
LDPE	0.9215	0.00109	0.00195	0.00308	0.00552
HDPE	0.9678	0.00128	0.00189	0.00356	0.01007
Thermoset	1.3130	0.00160	0.00217	0.00453	0.01282

established experimentally in the particular application that a lesser frequency of determination can be used to assure the desired precision.

NOTE 14—For the determination of the specific gravity of the liquid, the use of a standard plummet of known volume (Note 15) or of Method A, C, or D of Test Methods D 891, using the modifications required to give specific gravity 23/23°C instead of specific gravity 60/60°F, is recommended. One suggested procedure is the following:

If a constant-temperature water bath is not available, determine the weight of the clean, dry pycnometer with thermometer to the nearest 0.1 mg on an analytical balance. Fill the pycnometer with water (10.1) cooler than 23°C. Insert the thermometer-stopper, causing excess water to be expelled through the side arm. Permit the filled bottle to warm in air until the thermometer reads 23.0°C. Remove the drop of water at the tip of the side arm with a bit of filter paper, taking care not to draw any liquid from within the capillary, place the cap over the side arm, wipe the outside carefully, and weigh the filled bottle again to the nearest 0.2 mg. Empty the pycnometer, dry, and fill and weigh with the other liquid in the same manner as was done with the water. Calculate the specific gravity 23/23°C of the liquid, d , as follows:

$$d = (b - e)/(w - e)$$

where:

e = apparent weight of empty pycnometer,

w = apparent weight of pycnometer filled with water at 23.0°C, and

b = apparent weight of pycnometer filled with liquid at 23.0°C.

If a constant-temperature water bath is available, a pycnometer without a thermometer may be used (compare 30.2).

NOTE 15—One standard object which has been found satisfactory for this purpose is the Reimann Thermometer Plummets. These are normally supplied calibrated for measurements at temperatures other than 23/23°C, so that recalibration is necessary for the purposes of these methods.

19. Test Specimens

19.1 See Section 11.

20. Procedure

20.1 The procedure shall be similar to Section 12, except for the choice of immersion liquid, and the temperature during the immersed weighing (12.3) shall be $23 \pm 0.5^\circ\text{C}$ ($73.4 \pm 1.0^\circ\text{F}$).

21. Calculation

21.1 The calculations shall be similar to Section 13, except that d , the specific gravity 23/23°C of the liquid, shall

be placed in the numerator:

$$Sp\ gr\ 23/23^\circ\text{C} = (a \times d)/(a + w - b)$$

22. Report

22.1 See Section 14.

23. Precision and Bias⁹

23.1 Tables 1 and 2 are based on round robins conducted in 1985 involving five materials tested by six laboratories for Test Method A. Four materials were tested with Test Method B by six laboratories. Each test result was based on two individual determinations. Each laboratory obtained four test results for each material.

23.2 In Tables 1 and 2, for the materials indicated, and for mean values that are derived from testing two specimens:

23.2.1 S_p is the within-laboratory standard deviation of the mean and $I_p = 2.83 S_p$. (See 23.2.3 for application of I_p .)

23.2.2 S_R is the between-laboratory standard deviation of the mean, and $I_R = 2.83 S_R$. (See 23.2.4 for application of I_R .)

23.2.3 **Repeatability**—In comparing two mean values for the same material obtained by the same operator using the same equipment on the same day, the means should be judged not equivalent if they differ by more than the I_p value for that material and condition.

23.2.4 **Reproducibility**—In comparing two mean values for the same material obtained by different operators using different equipment on different days, the means should be judged not equivalent if they differ by more than the I_R value for that material and condition. (This applies between different laboratories or between different equipment within the same laboratory.)

23.2.5 The judgements per 23.2.3 and 23.2.4 will have an approximate 95 % (0.95) probability of being correct.

23.2.6 Other materials may give somewhat different results.

23.3 For further information on the methodology used in this section, see Practice E 691.

⁹ Supporting data are available from ASTM Headquarters. Request RR:D 20 - 1133.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)¹

This standard is issued under the fixed designation D 1002; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace methods 1032 and 1032.1T of Federal Test Method Standard No. 173a and for listing in the DoD Index of Specifications and Standards.

The accuracy of the results of strength tests of adhesive bonds will depend on the conditions under which the bonding process is carried out. Unless otherwise agreed upon by the manufacturer and the purchaser, the bonding conditions shall be prescribed by the manufacturer of the adhesive. In order to ensure that complete information is available to the individual conducting the tests, the manufacturer of the adhesive shall furnish numerical values and other specific information for each of the following variables:

- (1) Procedure for preparation of surfaces prior to application of the adhesive, the cleaning and drying of metal surfaces, and special surface treatments such as sanding that are not specifically limited by the pertinent test method.
- (2) Complete mixing directions for the adhesive.
- (3) Conditions for application of the adhesive, including the rate of spread or thickness of film, number of coats to be applied, whether to be applied to one or both surfaces, and the conditions of drying where more than one coat is required.
- (4) Assembly conditions before application of pressure, including the room temperature, relative humidity, length of time, and whether open or closed assembly is to be used.
- (5) Curing conditions, including the amount of pressure to be applied, the length of time under pressure, method of applying pressure (pressure bag, press platens, etc.), heat-up rate, and the temperature of the assembly when under pressure. It should be stated whether this temperature is that of the bondline or of the atmosphere at which the assembly is to be maintained.
- (6) Conditioning procedure before testing, unless a standard procedure is specified, including the length of time, temperature, and relative humidity.

A range may be prescribed for any variable by the manufacturer of the adhesive if it can be assumed by the test operator that any arbitrarily chosen value within such a range or any combination of such values for several variables will be acceptable to both the manufacturer and the purchaser of the adhesive.

1. Scope

1.1 This test method covers the determination of the comparative shear strengths of adhesives for bonding metals when tested on a standard specimen and under specified conditions of preparation and test.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Apparatus

2.1 The testing machine shall conform to the requirements of ASTM Methods E4, Verification of Testing

Machines.² The testing machine shall be so selected that the breaking load of the specimens falls between 15 and 85 percent of the full-scale capacity. The machine shall be capable of maintaining a rate of loading of 80 to 100 kg/cm² (1200 to 1400 psi)/min, or, if the rate is dependent on crosshead motion, the machine should be set to approach this rate of loading. It shall be provided with a suitable pair of self-aligning grips to hold the specimen. It is recommended that the jaws of these grips shall engage the outer 25 mm (1 in.) of each end of the test specimen firmly (Note 1). The grips and attachments shall be so constructed that they will move into alignment with the test specimen as soon as the load is applied, so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly.

NOTE 1—The length of overlap of the specimen may be varied where necessary. The length of the specimen in the jaws, however, must not be varied. The distance from the end of the lap to the end of the jaws should be 63 mm (2½ in.) in all tests.

¹ This test method is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.83 on Metal Bonding Adhesives.

Current edition approved July 28, 1972. Published October 1972. Originally published as D 1002 - 49 T. Last previous edition D 1002 - 64 (1970).

² Annual Book of ASTM Standards, Vol 03.01.

FIG. 1 Form and Dimensions of Test Specimen

3. Test Specimens

3.1 Test specimens shall conform to the form and dimensions shown in Fig. 1. These shall be cut from test panels prepared as prescribed in Section 4. The recommended thickness of the sheets is 1.62 ± 0.125 mm (0.064 ± 0.005 in.). The recommended length of overlap for most metals of 1.62 mm (0.064 in.) in thickness is 12.7 ± 0.25 mm (0.5 ± 0.01 in.).

NOTE 2—Since it is undesirable to exceed the yield point of the metal in tension during test, the permissible length of overlap in the specimen will vary with the thickness and type of metal, and on the general level of strength of the adhesive being investigated. The maximum permissible length may be computed from the following relationship:

$$L = Fty / t\tau$$

where:

L = length of overlap, in.,
 t = thickness of metal, in.,
 Fty = yield point of metal (or the stress at proportional limit), psi, and
 τ = 150 percent of the estimated average shear strength in adhesive bond, psi.

NOTE 3—A variation in thickness of the metal, and the length of overlap, will likely influence the test values and make direct comparison of data questionable. For this reason, in comparative or specification tests, the thickness should preferably be 1.62 ± 0.125 mm (0.064 ± 0.005 in.) and the length of overlap should preferably be 12.7 ± 0.25 mm (0.5 ± 0.01 in.), or not in excess of the value computed in Note 2. For development tests values could be different, but should then be constant.

3.2 The following grades of metal are recommended for the test specimens:

Metal	ASTM Designation ⁴
Brass	B 36, Alloy 8
Copper	B 152, Type A
Aluminum	B 209, Alloy 2024, T3 temper
Steel	A 109, Grade 2
Corrosion-resisting steel	A 167, Type 302
Titanium	B 265

⁴ These designations refer to the following specifications of the American Society for Testing and Materials:

- ³ B 36, for Brass Plate, Sheet, Strip, and Rolled Bar,³
- ³ B 152, for Copper Sheet, Strip, Plate, and Rolled Bar,³
- ³ B 209, for Aluminum and Aluminum-Alloy Sheet and Plate³
- ³ A 109, for Cold-Rolled Carbon Steel Strip,³
- ³ A 167, for Stainless and Heat-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip,³ and
- ³ B 265, for Titanium and Titanium Alloy Strip, Sheet, and Plate⁴

³ Annual Book of ASTM Standards, Vol 02.01.

⁴ Annual Book of ASTM Standards, Vol 02.02.

⁵ Annual Book of ASTM Standards, Vol 01.03.

⁶ Annual Book of ASTM Standards, Vol 02.04.

3.3 At least 30 specimens shall be tested, representing at least four different joints. However, if statistical analysis of data and variance is employed, it should be possible to reduce this number.

4. Preparation of Test Joints

4.1 It is recommended that test specimens be made up in multiples of at least five specimens, and then cut into individual test specimens (Note 4), Figs. 2 and 3. Cut sheets of the metals prescribed in 3.1 and 3.2 to suitable size. All edges of the metal panels and specimens which will be within (or which will bound) the lap joints shall be machined true (without burrs or bevels and at right angles to faces) and smooth (rms 160 max) before the panels are surface-treated and bonded. Clean and dry the sheets carefully, according to the procedure prescribed by the manufacturer of the adhesive, and assemble in pairs. Prepare and apply the adhesive according to the recommendations of the manufacturer of the adhesive. Apply the adhesive to a sufficient length in the area across the end of one or both metal sheets so that the adhesive will cover a space approximately 6 mm ($\frac{1}{4}$ in.) longer than the overlap as selected in Section 3. Assemble the sheets so that they will be held rigidly so that the length of the overlap will be controlled, as indicated in Section 3, within 0.25 mm (± 0.01 in.), and the adhesive allowed to cure as prescribed by the manufacturer of the adhesive.

NOTE 4—Bonding specimens in multiple panels is believed to give more representative specimens. However, individual specimens may be prepared if agreeable to the supplier or the purchaser of the adhesive.

5. Preparation of Test Specimens

5.1 Cut the test specimens, as shown in Fig. 1, from the panels, Figs. 2 and 3. Perform the cutting operation so as to avoid overheating or mechanical damage to the joints (Note 5). For final preparation trim panel area according to Fig. 2. Measure the width of the specimen and the length of the overlap to the nearest 0.25 mm (0.01 in.) to determine the shear area.

NOTE 5—A five-tooth, typesetter's circular saw has been found suitable for such purposes.

6. Procedure

6.1 Test the specimens, prepared as prescribed in Section 5, as soon after preparation as possible. The manufacturer of the adhesive may, however, prescribe a definite period of conditioning under specific conditions before testing.

6.2 Place the specimens in the grips of the testing machine so that the outer 25 mm (1 in.) of each end are in contact with the jaws (Note 1) and so that the long axis of the test specimen coincides with the direction of applied pull through the center line of the grip assembly. Apply the loading immediately to the specimen at the rate of 80 to 100 kg/cm² (1200 to 1400 psi) of the shear area per min. Continue the load to failure. This rate of loading will be approximated by a free crosshead speed of 1.3 mm (0.05 in.)/min.

7. Calculations

7.1 Record the load at failure and the nature and amount of this failure (cohesion in adhesive or metal, or adhesion) for each specimen. Express all failing loads in kilograms per

FIG. 2 Standard Test Panel

square centimeter (pounds per square inch) of shear area, calculated to the nearest 0.06 cm^2 (0.01 in.^2).

8. Report

8.1 The report shall include the following information:

8.1.1 Complete identification of the adhesive tested, including type, source, date manufactured, manufacturers' code numbers, form, etc.,

8.1.2 Complete identification of the metal used, its thickness, and the method of cleaning and preparing its surface prior to bonding,

8.1.3 Application and bonding conditions used in preparing specimens,

8.1.4 Average thickness of adhesive layer after formation of the joint within 0.001 in. The method of obtaining the thickness of the adhesive layer shall be described including procedure, location of measurements, and range of measurements.

8.1.5 Length of overlap used,

8.1.6 Conditioning procedure used for specimens prior to testing,

8.1.7 Number of specimens tested,

8.1.8 Number of joints represented and type of joint if other than single overlap,

8.1.9 Maximum, minimum, and average values for the failing load, and

8.1.10 The nature of the failure, including the average estimated percentages of failure in the cohesion of the adhesive, contact failure, and adhesion to the metal.

FIG. 3 Optional Panel for Acceptance Tests Only

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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Standard Test Method for Thermal Conductivity of Solids by Means of the Guarded- Comparative-Longitudinal Heat Flow Technique¹

This standard is issued under the fixed designation E 1225; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Editorial corrections were made throughout in March 1990.

1. Scope

1.1 This test method describes a steady state technique for the determination of the thermal conductivity, λ , of homogeneous-opaque solids (see Notes 1 and 2). This test method is for materials with effective conductivities in the approximate range $0.2 < \lambda < 200 \text{ W/m}\cdot\text{K}$ over the approximate temperature range between 90 and 1300 K. It can be used outside these ranges with decreased accuracy.

NOTE 1—For purposes of this technique, a system is homogeneous if the apparent thermal conductivity of the specimen, λ_A , does not vary with changes of thickness or cross-sectional area by more than $\pm 5\%$. For composites or heterogeneous systems consisting of slabs or plates bonded together, the specimen should be more than 20 units wide and 20 units thick, respectively, where a unit is the thickness of the thickest slab or plate, so that diameter or length changes of one-half unit will affect the apparent λ_A by less than $\pm 5\%$. For systems that are non-opaque or partially transparent in the infrared, the combined error due to inhomogeneity and photon transmission should be less than $\pm 5\%$. Measurements on highly transparent solids must be accompanied with infrared absorption coefficient information or the results must be reported as apparent λ_A .

NOTE 2—This test method may also be used to evaluate the thermal conductance/resistance of materials in contact.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- C 177 Test Method for Steady State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus²
- C 408 Test Method for Thermal Conductivity of White-ware Ceramics³
- C 1045 Practice for Calculating Thermal Transmission Properties from Steady State Heat Flux Measurements²

¹ This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

Current edition approved Dec. 31, 1987. Published February 1988.

² Annual Book of ASTM Standards, Vol 04.06.

³ Annual Book of ASTM Standards, Vol 15.02.

D 4351 Test Method for Measuring the Thermal Conductivity of Plastics by the Evaporation-Caloric Method⁴

E 230 Temperature-Electromotive Force (EMF) Test for Standardized Thermocouples⁵

E 220 Method for Calibration of Thermocouple Comparison Techniques⁵

F 433 Practice for Evaluating Thermal Conductivity of Gasket Materials⁶

3. Descriptions of Terms and Symbols Specific to Standard

3.1 Terms:

3.1.1 *Thermal Conductivity, λ* —In an optically thick with constant properties, the quantity of heat flow per area at steady state, q' , is given by $q' = \lambda \nabla T$, where λ is thermal conductivity. For one-dimensional heat flow

in the Z direction, this reduces to $q' = \lambda \frac{dT}{dZ}$, where dT/dZ is

the temperature gradient. When only two sensors are used, the differential must be replaced with $\Delta T/\Delta Z$, the temperature difference between the two sensors.

3.1.2 *Apparent Thermal Conductivity*—In some cases the quantity of heat flow will be a function of additional system parameters such as the emitting and bounding surfaces and the specimen size. In these cases the comparator determines only an apparent thermal conductivity, λ_A , defined as $\lambda_A = -q' \frac{\Delta Z}{\Delta T}$.

3.2 Symbols:

- $\lambda_M(T)$ = thermal conductivity of meter bars (reference materials) as a function of temperature ($\text{W/m}\cdot\text{K}$),
- λ_M^1 = thermal conductivity of top meter bar ($\text{W/m}\cdot\text{K}$),
- λ_M^2 = thermal conductivity of bottom meter bar ($\text{W/m}\cdot\text{K}$),
- $\lambda_S(T)$ = thermal conductivity of specimen corrected for heat exchange where necessary, ($\text{W/m}\cdot\text{K}$),
- $\lambda'_S(T)$ = thermal conductivity of specimen calculated by ignoring heat exchange correction ($\text{W/m}\cdot\text{K}$),

⁴ Annual Book of ASTM Standards, Vol 08.03.

⁵ Annual Book of ASTM Standards, Vol 14.03.

⁶ Annual Book of ASTM Standards, Vol 09.02.

FIG. 1(a) Schematic of a Comparative-Guarded-Longitudinal Heat Flow System Showing Possible Locations of Temperature Sensors

- $\lambda(T)$ = thermal conductivity of insulation as a function of temperature, $(W/m \cdot K)$.
 T = absolute temperature in degrees Kelvin (K).
 z = position as measured from the upper end of the column, (m).
 l = specimen length, (m).
 T_z = the temperature at z , (K).
 q' = heat flow per unit area, (W/m^2) .
 $\delta\lambda, \delta T, \text{ etc.}$ = uncertainty in $\lambda, T, \text{ etc.}$.
 r_1 = specimen radius, (m).
 r_2 = guard cylinder inner radius, (m).
 $T_g(z)$ = guard temperature as a function of position, z , (K), and
 z = distance from upper end of the column, (m).

4. Summary of Test Method

4.1 A test specimen is inserted under load between two similar specimens of a material of known thermal properties. A temperature gradient is established in the test stack and heat losses minimized by use of a longitudinal guard having approximately the same temperature gradient conditions. At equilibrium conditions, the thermal conductivity is derived in terms of the temperature gradients in the respective specimens and the thermal conductivity of the reference materials.

4.2 General Features of Test Method:

4.2.1 The general features of the guarded longitudinal

FIG. 1(b) Schematic of Typical Test Stack and Guard System Illustrating Matching of Temperature Gradients

heat flow technique are shown in Fig. 1. A specimen of unknown conductivity, λ_s , but having an estimated thermal conductance of λ_s/l_s , is mounted between two meter bars of known thermal conductivity, λ_M , of the same cross-section and similar thermal conductance, λ_M/l_M . A more complex but suitable arrangement is a column consisting of a disk heater with a specimen and a meter bar on each side between heater and heat sink. Approximately one-half of the power would then flow through each specimen. When the meter bars and specimen are right-circular cylinders of equal diameter the technique is described as the cut-bar method. When the cross-sectional dimensions are larger than the thickness it is described as the flat slab comparative. Essentially any shape can be used as long as the meter bars and specimen have the same conduction areas.

4.2.2 A force is applied to the column that is surrounded by an insulation material of thermal conductivity, λ_i . The insulation is enclosed in a guard shell with a radius, r_B , and the shell is held at temperature, $T_g(z)$. A temperature gradient is imposed on the column by maintaining the top at a temperature, T_T , and the bottom at temperature T_B . $T_g(z)$ is usually a linear temperature gradient situation with the gradient matched approximately to that established in the test stack. However, an isothermal guard with $T_g(z)$ equal to the average temperature of the specimen may also be used. An unguarded system is not recommended due to the potential very large errors particularly at elevated temperatures (1).⁷ At steady state, the temperature gradients along the sections are calculated from measured temperatures

⁷ The boldface numbers in parentheses refer to a list of references at the end of this test method.

NOTE—The material selected for the meter bars should have a thermal conductivity as near as possible to the thermal conductivity of the unknown.

FIG. 2 Approximate Values for the Thermal Conductivity of Several Possible Reference Materials for Meter Bars

along the two meter bars and the specimen. The value of λ_s (λ_s as uncorrected for heat shunting) can then be determined using the following where the notation is shown in Fig. 1:

$$\lambda_s = \frac{(Z_4 - Z_1) \lambda_M (T_2 - T_1)}{(T_4 - T_3) 2 (Z_2 - Z_1)} + \left[\frac{\lambda_M^2 (T_6 - T_3)}{2 (Z_6 - Z_3)} \right] \quad (1)$$

This is a highly idealized situation, however, since it assumes no heat exchange between the column and insulation at any position and uniform heat transfer at each meter bar-specimen interface. The errors caused by these two assumptions vary widely and are discussed in Section 9. Because of these two effects, restrictions must be placed on this test method if the desired accuracy is to be achieved.

5. Significance and Use

5.1 The comparative method of measurement of thermal conductivity is especially useful for engineering materials including ceramics, polymers, metals and alloys, refractories, carbons, and graphites including combinations and other composite forms of each. It is capable of generating relatively accurate data on specimens that are too small (or possibly have an unusual shape) to accept heater and heat sink designs required by absolute methods.

5.2 Proper design of a guarded-longitudinal system is difficult and it is not practical in a method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person

without technical knowledge concerning theory of heat temperature measurements, and general testing practice. Standardization of this test method is not intended to restrict in any way the future development by research workers of new or improved methods or improved procedures. However, new or improved techniques must be thoroughly tested and requirements for qualifying an apparatus are outlined in Section 10.

6. Requirements

6.1 Meter Bar Reference Materials:

6.1.1 Reference materials or transfer standards of known thermal conductivities must be used for the meter bars. Since the minimum measurement error of the method is the uncertainty in λ_M , it is preferable to use standards available from a national standards laboratory. Other reference materials are available because numerous measurements of λ have been made and general acceptance of values has been obtained. Table 1 lists the currently available recognized reference materials including those available from National Institute of Standards and Technology. Figure 2 shows the approximate variation of λ_M with temperature.

6.1.2 Table 1 is not exhaustive and other materials may be used as references. The reference material and the source λ_M values shall be stated in the report.

6.1.3 The requirements for any reference material include stability over the temperature range of operation, compatibility with other system components, reasonable cost, ease of thermocouple attachment, and an accurate known thermal conductivity. Since heat shunting errors for specific λ_s increase as λ_M/λ_s varies from unity, (1) the reference which has a λ_M nearest to λ_s should be used for the meter bars.

6.1.4 If a sample has a λ_s between two reference materials the reference with the higher λ_M should be used to reduce the total temperature drop along the column.

6.2 Insulation Materials:

6.2.1 A large variety of powder, particulate, and fiber materials exist for reducing both radial heat flow in the column-guard annulus and surrounds and heat shunting along the column. Several factors must be considered during selection of the most appropriate insulation. The insulation must be stable over the anticipated temperature range, have a low λ_i , and be easy to handle. In addition, the insulation should not contaminate system components such as the temperature sensors, it must have low toxicity, and it should not conduct electricity. In general, powders and particulates are used since they pack readily. However, low density fiber blankets can also be used.

6.2.2 Some candidate insulations are listed in Table 2.

6.3 Temperature Sensors:

6.3.1 There shall be a minimum of two temperature sensors on each meter bar and two on the specimen. Whenever possible, the meter bars and specimen should each contain three sensors. The extra sensors are useful in confirming linearity of temperature versus distance along the column or indicating an error due to a temperature sensor decalibration.

6.3.2 The type of temperature sensor depends on the system size, temperature range, and the system environment as controlled by the insulation, meter bars, specimen, and

TABLE 1 Reference Materials For Use as Meter Bars

Material	Temperature Range (K)	Percentage Uncertainty in λ ($\pm$ %)	λ_M (W/m·K)	Material Source
Electrolytic Iron SRM 734	To 1000	2	^a	NIST ^a
Tungsten SRM 730	4 to 300 300 to 2000 >2000	2 2 to 5 5 to 8	λ_M Dependent on T ^a	NIST ^a
Austenitic Stainless SRM 735	4 to 1200	<5 %	$\lambda_M = 1.22T^{0.432}$ T > 200K ^a	NIST ^a
Iron	80 to 1200	2	λ_M should be calculated from measured values ^{c,d}	...
Copper	90 to 1250	<2	$\lambda_M = 416.3 - 0.05904T + 7.087 \times 10^{-7}T^3$	manufacturer
Pyroceram Code 9606	90 to 1200	...	^{e,g}	ceramic glass
Fused Silica ^h	1300	<8	$\lambda_M = (84.7/T) + 1.484 + 4.94 \times 10^{-4}T + 9.6 \times 10^{-13}T^4$	glass
Pyrex 7740	90 to 600	6	^{e,g}	glass

^a National Institute of Standards and Technology, Washington, D.C. 20234. See Special Publications 260-52 and 260-46.

^b Hust J. G., Cryogenics Division, NBS, Boulder, Colorado 80302.

^c Fulkerson W., et al., *Physics Review* 167, p. 765, (1968).

^d Luckas G. F., *Journal of Testing and Evaluation*, ASTM 1 (5), 422 (1973).

^e Moore, J. P., Graves, R. S. and McElroy, D. L., *Canadian Journal of Physics*, 45, 3849 (1967).

^f "Thermal Conductivity of Selected Materials," Report NSRDS-NBS 8, National Bureau of Standards, 1966.

^g L. C. Hulstrom, R. P. Tye, and S. E. Smith, *Thermal Conductivity* 19, Ed. D. W. Yarborough, Plenum Press, New York, In Course of Publication (see also High Temperature-High Pressures, 17, 707, 1985).

^h Above 700 K a large fraction of heat conduction in fused silica will be by radiation and the actual effective values may depend on the emittances of bounding surfaces and meter bar size.

ⁱ Recommended values from Table 3017 A-R-2 of the *Thermophysical Properties Research Center Data Book*, Vol. 3, "Nonmetallic Elements, Compounds, and Mixtures," Purdue University, Lafayette, Indiana.

TABLE 2 Suitable Thermal Insulation Materials

Material ^a	Typical Thermal Conductivity (W/m·K)		
	300K	800K	1300K
Poured Powders			
Diatomaceous Earth	0.053	0.10	0.154
Bubbled Alumina	0.21	0.37	0.41
Bubbled Zirconia	0.19	0.33	0.37
Vermiculite	0.07	0.16	...
Perlite	0.050	0.17	...
Blankets and Felts			
Aluminosilicate 60-120 kg/m ³	0.044	0.13	0.33
Zirconia 60-90 kg/m ³	0.039	0.09	0.25

^a All materials listed can be used up to the 1300 K limit of the comparative longitudinal except where noted.

gas within the system. Any sensor possessing adequate accuracy may be used for temperature measurement (2) and be used in large systems where heat flow perturbation by the temperature sensors would be negligible. Thermocouples are normally employed. Their small size and the ease of attaching them are distinct advantages.

6.3.3 When thermocouples are employed they should be fabricated from wires which are 0.1 mm diameter or less. A constant temperature reference shall always be provided for all cold junctions. This reference can be an ice-cold slurry (3), a constant temperature zone box, or an electronic ice point reference. All thermocouples shall be fabricated from either calibrated thermocouple wire (4) or from wire that has been certified by the supplier to conform with tables from E 230 to within the special limits of error given in Table 1 of E 230. E 230 should be consulted for information regarding thermocouple selection.

6.3.4 Thermocouple attachment is important to this technique in order to ensure that reliable temperature measure-

ments are made at specific points. The various techniques are illustrated in Fig. 3. Intrinsic junctions can be obtained with metals and alloys by welding individual thermo-elements to the surfaces (Fig. 3a). Butt or bead welded thermocouples junctions can be rigidly attached by peening, cementing, or welding in fine grooves or small holes (Figs. 3b to 3d).

6.3.5 In Fig. 3b, the thermocouple resides in a radial slot, and in Fig. 3c the thermocouple is pulled through a radial hole in the material. When a sheathed thermocouple or a thermocouple with both thermoelements in a two-hole electrical insulator is used, the thermocouple attachment shown in Fig. 3d can be used. In the latter three cases, the thermocouple should be thermally connected to the solid surface using a suitable glue or high temperature cement. All four of the procedures shown in Fig. 3 should include wire tempering on the surfaces, wire loops in isothermal zones, thermal wire grounds on the guard, or a combination of all three (5).

6.3.6 Since uncertainty in temperature sensor location leads to large errors, special care must be taken to determine the correct distance between sensors and to calculate the possible error resulting from any uncertainty.

6.4 Reduction of Contact Resistance:

6.4.1 This test method requires uniform heat transfer at the meter bar to specimen interfaces whenever the temperature sensors are within a distance equal to r_d from an interface (6). This requirement necessitates a uniform contact resistance across the adjoining areas of meter bars and specimens. This is normally attained by use of an applied axial load in conjunction with a conducting medium at the interfaces. Measurements in a vacuum environment are not recommended unless the vacuum is required for protection purposes.

FIG. 3 Thermocouple Attachments

6.4.2 For the relatively thin specimens normally used for materials having a low thermal conductivity the temperature sensors must be mounted close to the surface and in consequence the uniformity of contact resistance is critical. In such cases, a very thin layer of a compatible highly conductive fluid, paste, soft metal foil, or screen shall be introduced at the interfaces.

6.4.3 Means shall be provided for imposing a reproducible and constant load along the column with the primary purpose of minimizing interfacial resistances at meter bar-specimen interfaces. Since the force applied to the column usually affects the contact resistance, it is desirable that this force be variable to ensure that λ_S does not change with force variation. This force can be applied either pneumatically, hydraulically, by spring action, or by putting a dead weight on the column. The above load mechanisms have the advantage of remaining constant with change in column temperature. In some cases, the compressive strength of the specimen might be so low that the applied force must be limited to the dead weight of the upper meter bar. In this case, special care must be taken to limit error caused by poor contact by judicious positioning of temperature sensors away from any heat flow perturbation at the interfaces.

6.5 Guard Cylinder:

6.5.1 The specimen-meter bar column shall be enclosed with a guard tube or pipe normally of right circular symmetry. This guard cylinder can be either a metal or a ceramic but its inside radius should be such that the ratio r_B/r_A be between 2.0 and 3.5 (1). This guard cylinder shall contain at least one heater for controlling the temperature profile along the guard.

6.5.2 The guard shall be constructed and operated so that the temperature of the guard surface is either isothermal and equal to the approximate mean temperature of the specimen or preferably have an approximately linear profile with the top and bottom ends of the guard matched to corresponding positions along the column. In each case, at least three temperature sensors shall be attached to the guard at known positions to measure the temperature profile.

6.6 System Instrumentation:

6.6.1 The combination of temperature sensor and the instrument used for measuring the sensor output shall be adequate to ensure a temperature measurement precision of ± 0.04 K and an absolute error less than ± 0.5 %.

6.6.2 Instrumentation for this technique shall be adequate to maintain the required temperature control and measure all pertinent output voltages with accuracy commensurate with the system capability. Although control can be manual

a technique of this general description can be automated so that a computer carries out all the control functions, acquires all pertinent voltages, and calculates the thermal conductivity (7).

7. Sampling and Conditioning Test Specimens

7.1 Test Specimens—This test method is not restricted to a particular geometry. General practice is to use cylindrical or square cross-sections. The conduction area of the specimen and reference samples must be the same to within 1 % (see Note 3) and any difference in area shall be taken into account in the calculations of the result. For the cylindrical configuration, the radii of the specimen and meter bars must agree to within ± 1 % and the specimen radius, r_A , must be such that r_B/r_A is between 2.0 and 3.5. Each flat surface of the specimen and reference must be flat with a surface finish equal to or better than 32 $\sqrt$ and the normal to each end shall be parallel with the specimen axis to within ± 10 min.

NOTE 3—In some cases this requirement is not necessary. For example, some apparatus might consist of meter bars and specimen with high values of $\lambda_{1/2}$ and λ_5 so that thermal shunting errors would be small for long sections. These sections might be long enough to permit temperature sensor attachment far enough away from the interfaces that heat flow was uniform. The specimen length should be selected based on considerations of radius and thermal conductivity. When $\lambda_{1/2}$ is higher than the thermal conductivity of SRM 735 (stainless steel), long specimens with length/ $r_A \gg 1$ can be used. These long specimens permit the use of large distances between temperature sensors and this reduces the percentage error from uncertainty in sensor position. When $\lambda_{1/2}$ lower than the thermal conductivity of SRM 735, the length must be reduced because uncertainty from heat shunting becomes too large.

7.2 Sampling and Conditioning—Unless specifically required or prescribed, one representative specimen shall be prepared from the sample and no preconditioning undertaken.

8. Calibration and Verification

8.1 There are many situations that call for equipment tests before operations on unknown materials can be successfully accomplished. These include the following:

- 8.1.1** After initial equipment construction.
- 8.1.2** When the ratio of $\lambda_{1/2}$ to λ_5 is less than 0.3 or greater than 3 and it is not possible to match thermal conductance values.
- 8.1.3** When the specimen shape is complex or the specimen is inordinately small.
- 8.1.4** When changes have been made in the system geometry.
- 8.1.5** When meter bar or insulation material other than those listed in 5.1 and 5.2 are considered for use, and
- 8.1.6** When the apparatus has been to a high enough temperature to change the properties of a component such as thermocouples sensitivity.
- 8.2** These tests shall be run by comparing at least two reference materials in the following manner:
 - 8.2.1** A reference material which has the closest thermal conductivity to the estimated thermal conductivity of the unknown should be machined according to 6.1. and
 - 8.2.2** The λ of this specimen fabricated from a reference material shall then be measured as described in Section 9 using meter bars fabricated from another reference material which has the closest λ to that of the specimen. For example,

technique tests might be made by measuring on Pyroceram 9606 using meter bars fabricated from SRM 735 stainless steel. If the measured thermal conductivity of the specimen disagrees with the value from Table 1 after all corrections for heat exchange are applied, additional effort is required to find the error source(s).

9. Procedure

9.1 Where possible and practical, select the reference specimens (meter bars) such that the thermal conductance is of the same order as that expected for the test specimen. After instrumenting and installing the proper meter bars, the specimen should be instrumented similarly. It should then be inserted into the test stack such that it is aligned between the meter bars with at least 99 % of each specimen surface in contact with the adjacent meter bar and soft foil or other contacting medium, if any, used to reduce interfacial resistance. If the system must be protected from oxidation during the test or if operation requires a particular gas or gas pressure to control λ_p , the system should be pumped and purged, and the operating gas and pressure established. The predetermined force required for reducing the effects of non-uniform interfacial resistance should be applied to the load column.

9.2 Heaters at either end of the column should be energized (see Note 4) and adjusted until the T differences between positions Z_1 and Z_2 , Z_3 and Z_4 , and Z_5 and Z_6 are between 200 times the imprecision of the ΔT measurements and 30 K, and the specimen is at the average temperature desired for the measurement. Although the exact temperature profile along the guard is not important for $r_B/r_A \geq 3$, the power to the guard heaters should be adjusted until the temperature profile along the guard, $T_g(z)$, is constant with respect to time to within ± 0.1 K and either:

9.2.1 Approximately linear so that $T_g(z)$ coincides with the temperature along the sample column at a minimum of three places including the temperature at the top sensor on the top meter bar, the bottom sensor on the bottom bar, and the specimen midplane; or

9.2.2 Constant with respect to z to within ± 5 K and matched to the average temperature of the test specimen.

NOTE 4—These heaters can either be attached to the ends of the meter bars or attached to a structure adjacent to the meter bar. The heaters can be powered with A.C. or D.C. Several different heater configurations are acceptable. The power to these heaters shall be steady enough to maintain short term T fluctuations less than ± 0.03 K on the meter bar thermocouple nearest the heater. These two heaters, in conjunction with the guard shell heater and the system coolant shall maintain long term temperature drift less than ± 0.05 K/h.

9.3 After the system has reached steady state (T drift < 0.05 K/h), measure the output of all temperature sensors.

10. Calculation

10.1 Approximate Specimen Thermal Conductivity:

10.1.1 The outputs from the temperature sensors shall be converted to temperature and the apparent heat flow per unit area, q' , in the meter bars shall be calculated using the following:

$$q'_T = \lambda_M \frac{T_2 - T_1}{Z_2 - Z_1} \quad \text{top bar} \quad (2)$$

$$q'_B = \lambda_M \frac{T_6 - T_5}{Z_6 - Z_5} \quad \text{bottom bar} \quad (3)$$

In each of these equations, the λ_M value (see Note 5) to be inserted shall be obtained from the information of 6.1 for the average meter bar T . Although these two values, q'_T and q'_B , should agree with each other to within about $\pm 10\%$ when heat exchange with the insulation is small, good agreement is not a sufficient condition (nor always a necessary condition) for low heat shunting error.

10.1.2 A value for the specimen thermal conductivity at temperature $(T_3 + T_4)/2$, as uncorrected for heat exchange with the insulation can then be calculated using the following:

$$\lambda'_S = \frac{(q'_T + q'_B)(Z_4 - Z_3)}{2(T_4 - T_3)} \quad (4)$$

This would be at an average specimen T of $(T_4 + T_3)/2$. Calculate $1/r_A(T_4 - T_3/r_A)$, λ_M/λ_S , and apply a correction for extraneous heat flow as described in 10.2 to adjust λ' to λ . Report all results according to Section 11.

NOTE 5—This type calculation procedure actually requires only two T sensors on each column section. In this case, the third sensor on each section serves as a test for consistency of the other two. Some calculation procedures require more than the two sensors to obtain more knowledge about dT/dZ .

10.2 Corrections for Extraneous Heat Flow:

10.2.1 Calculation of the specimen thermal conductivity by a simple comparison of temperature gradients in the meter bars to that in the specimen is less valid when the specimen or meter bars, or both, have low thermal conductivities relative to that of the insulation. The apparatus should be designed to minimize these errors and information on this has been enclosed as an adjunct. The deviation from uniform heat flow has been expressed as (1) follows:

$$\tau = F_r F_\lambda \quad (5)$$

where F_r is a function of system dimensions, and F_λ is a function of λ_M/λ_I , λ_I , and λ_S (1). The F_r term has a value between 2 and 3 for the ratio of guard radius to column radius specified for this system. The F_λ term is shown in Fig. 4 as a function of λ_S/λ_I for various values of λ_M/λ_I for a linear guard. At high ratios of λ_M/λ_I and λ_S/λ_I , corrections would not be necessary since the departure from ideal heat flow would be small. For example, the product of F_r and F_λ would be less than 0.10 (10 %) for all measurements where λ_M/λ_I and λ_S/λ_I are greater than 30. If the value of F_r/F_λ is to be kept below 10 %, the ratios λ_M/λ_I and λ_S/λ_I are greater than 30. If the value of F_r/F_λ is to be kept below 10 %, the ratios λ_M/λ_I and λ_S/λ_I must be within the dotted boundaries on Fig. 4.

10.2.2 Measurements on materials where the ratios of λ_M/λ_I and λ_S/λ_I do not fall within these boundaries shall be accompanied with corrections for extraneous heat flow, but these corrections can be determined in the following three different ways:

10.2.2.1 Use of analytical techniques as described by Didion (1) and Flynn (8).

10.2.2.2 Using calculations from finite-difference or finite-element heat conduction codes, and

FIG. 4 Fractional Heat Exchange Between the Meter Specimen Column and Surrounding Insulation as a Function of λ_S/λ_I for Several Values of λ_M/λ_I

10.2.2.3 Determined experimentally by using several reference materials or transfer standards of different the conductance as specimens. The procedure must be cautiously since all such specimens should have the same as the specimen with an unknown thermal conductivity have the same surface finish.

11. Report

11.1 The report of the test results shall include following:

11.1.1 Complete specimen identification including shape and size,

11.1.2 Complete identification of insulation and source λ_I values, gas, and gas pressure,

11.1.3 Statements of thermocouple type, size, and attachment procedure,

11.1.4 Complete listing of the geometrical dimension: the system including r_A , r_B , specimen height, meter height, and distances between temperature sensors,

11.1.5 Column force,

11.1.6 Meter bar material and source of λ_M values if other than those listed in Table 1,

11.1.7 Reference to the use of this test method shall include a statement of the percentage variation of qualification results about the true value. For example: "thermal conductivity results on Pyroceram 9606 used: SRM 735 meter bars were within $\pm 4\%$ of the accepted values for Pyroceram over the temperature range from 2. to 900 K."

11.1.8 Variations, if any, from this test method. If results are to be reported as having been obtained by this method then all requirements prescribed by this method shall be met. Where such conditions are not met, the phrase, "All requirements of this method have been met with the exception of..." shall be added and a complete list of the exceptions included, and

11.1.9 Measured values of temperature and specimen thermal conductivity.

12. Precision and Bias

12.1 *Determinate Errors*—The determinate error of Eq. 1 is

$$\left| \frac{\lambda'_S}{\lambda_S} \right| = \left| \frac{\delta \lambda_M}{\lambda_M} \right| + \left| \frac{\delta(Z_4 - Z_3)}{Z_4 - Z_3} \right| + \left| \frac{\delta(T_4 - T_3)}{T_4 - T_3} \right| + \frac{\delta(T_2 - T_1)}{(Z_2 - Z_1)C} + \frac{(T_2 - T_1)\delta(Z_2 - Z_1)}{(Z_2 - Z_1)C} + \frac{\delta(T_6 - T_5)}{(Z_6 - Z_5)C} + \frac{\delta(T_6 - T_5)\delta(Z_6 - Z_5)}{(Z_6 - Z_5)^2 C} \quad (6)$$

where:

$$C = \frac{T_2 - T_1}{Z_2 - Z_1} + \frac{T_6 - T_5}{Z_6 - Z_5} \quad (7)$$

Usually the heat flows and temperature sensor spacings are essentially the same in the meter bars so that C approaches a numerical value of 2.

12.2 Example of Determinate Error:

12.2.1 Assumptions for a system where both meter bars and the specimen are of equal length is that the sensor spacings are all 13 mm and $\lambda_M \approx \lambda_S$:

$$\left| \frac{\delta \lambda_M}{\lambda_M} \right| = 0.003 \quad (8)$$

$$Z_2 - Z_1 = Z_4 - Z_3 = Z_6 - Z_5 = 13 \text{ mm};$$

$$T_2 - T_1 = T_4 - T_3 = T_6 - T_5 = 10 \text{ K};$$

$$\delta(Z_2 - Z_1) = \delta(Z_4 - Z_3) = \delta(Z_6 - Z_5) = 0.2 \text{ mm}; \text{ and}$$

$$\delta(T_2 - T_1) = \delta(T_4 - T_3) = \delta(T_6 - T_5) = 0.04 \text{ K}.$$

12.2.2 The maximum value of $\delta(Z_2 - Z_1)$ etc. was approximated by assuming an uncertainty of $\pm 1/2$ (sensor diameter) at each temperature measurement position. Therefore, if the diameter of each sensor is 0.2 mm, the uncertainty in the difference would be ± 0.2 mm. The number for $\delta(T_2 - T_1)$ etc. was calculated based on the sensor absolute accuracy.

12.2.3 With these values the fractional uncertainty in λ'_S , λ'_S/λ'_S , will be $|0.069|$ or $\pm 6.9\%$.

12.3 Indeterminate Errors:

12.3.1 There are at least three other errors that can contribute to total system error and these are (1) non-uniform interfacial resistance, (2) heat exchange between the column and guard, and (3) heat shunting through the insulation around the column. These three errors must be minimized or appropriate corrections applied to the data if the desired accuracy is to be obtained.

12.3.2 The contributions from the last two errors can be determined approximately using results from appropriate experiments carried out at different levels of guard temperature to specimen stack temperature out of balance.

12.4 *Overall*—A recent international, interlaboratory round robin study also involving absolute methods (9) has shown that a precision of $\pm 6.8\%$ can be attained over the temperature range 300 to 600 K. Although no definite bias could be established these are indications that the values were the order of 2 % lower than those obtained by absolute methods. This paper is on file at ASTM as a research report.

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