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Qualification Report Supplement for Raychem Nuclear Grade Adhesive – S1119

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Qualification Report Supplement for Raychem Nuclear Grade Adhesive – S1119	**Rev D**

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Tested by:		
Sheila Roberts	Sheila Roberts Signature:	11/15/2002 Date:
Howard Evans	Howard E Evans Signature:	11/15/2002 Date:
Prepared by:		
Bridget Gilchrist	Bridget Gilchrist Signature:	11/15/2002 Date:
Approved by:		
Dennis Lynch for Product Management	Dennis Lynch Signature:	11/15/02 Date:
Approved by:		
Kathy Maher for Technical	Kathryn M. Maher Signature:	11/15/02 Date:

Energy Products
8000 Purfoy Road
Fuquay-Varina, NC 27501

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REF	REV	PAGE	PARA	DESCRIPTION
1	A	9		Added Table 5
2	A	1	2	Change in zinc resinate tackifier
3	A	8	3-4	PPS 3012/19 testing
4	A	9	1	Inserted Table 5
5	A	11	2	Changed Table 5 to Table 6
6	A	12	1	Relabeled Table 5 to Table 6
7	A	13	6	Performance Statement
1	B	ii	7	Clerical Added Page 15
2	B	3	3	Clerical - changed Table 6 to Table 7
3	B	15	1	Clerical - inserted table and labeled as Table 7
1	C	10	Note 5	Listed Wyle No's. Added as of 4/23/80 to *
2	C	10	Note 5	Listed Wyle No's. Added as of 4/23/80 to *
1	D	i	1	Added to Section 5 PPS 3012/19 Rev. C
2	D	ii	1	Added Table 6, Removed Table 7, Relabeled Table 6 as 7.
3	D	1	3	Added Para 3 Change in resin
4	D	2	4	Remove reference to Table 7
5	D	6	1	Added Sec. 3.1.7
6	D	8	1	Added Table 6
7	D	9	2	Added reference (6)
8	D	9	2	Removed *Not Yet Issued
9	D	9	4	Clerical-Change Table 6 to 7
10	D	10	1	Relabeled table 6 as Table 7
11	D	9	1	Added EDR 5336 to Table 7 & removed * Not Yet Issued
12	D	11	6	Performance Statement
13	D	13	1	Removed Label "Table 7

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TABLES

1.0 SUMMARY

An evaluation of the effects of a modification to Raychem's nuclear grade adhesive has been conducted to determine its effect on the previous qualification of Raychem cable accessories. Material and functional product tests have been conducted to evaluate the new adhesive. The results and subsequent analysis of the test data indicate that the new adhesive is equivalent or superior to the original adhesive as a wire splicing sealant for nuclear applications. The evaluation of the adhesives concludes that results of environmental type tests, done in accordance with the guidelines of IEEE Standards 383 and 323, on Raychem product configurations obtained using S1024 adhesive apply to S1119 without further requalification.

In 1997, a zinc resinate tackifier used in S1119 was obsoleted by its supplier. A suitable replacement for this raw material was identified and qualification of a new S1119 formulation using the new raw material (WCSF-500 tubing lot # N15738) was successfully performed in accordance with PPS 3012/19. The replacement of the zinc resinate tackifier was considered a “minor” change to the material.

In 2002, one of the resins used in S1119 was obsoleted by its supplier. A suitable replacement for this raw material was identified and qualification of a new S1119 formulation using the new raw material (T446, M17109-49-2 PCN 279885-000, Batch 131869-1and WCSF 500 38/13-S, EC20051-17) was successfully performed in accordance with PPS 3012/19 Rev. C. PPS 3012/19 was modified to specify tests that are more relevant to actual product applications. All PPS 3012/19 Rev. C tests were performed for both the “new” adhesive (molded plaques, 131869-1 or products WCSF 500, tubing lot # EC20051-17) and the “old “ adhesive (molded plaques, 93B-4898 or products WCSF-500 tubing lot # N15738 and # N16085). Both the “new” and the “old” S1119 formulations passed the material qualification tests. Results indicate that the resin change has not adversely affected the performance of the adhesive and that the form, fit, and function of the product has not been compromised.

2.0 BASIS

In the past, the standard Raychem nuclear grade adhesive was characterized as S1024. This was alternately designated as /83 or -N in the product descriptions. Raychem, in anticipation of the unavailability of one of the raw materials in this adhesive, took occasion to modify the nuclear adhesive incorporating the latest technology and processing considerations. The result is an adhesive characterized as S1119, which is designated /144 in the product description for molded parts and -N for tubing.

This evaluation reports the results of extensive testing done to confirm the adequacy of the S1119 adhesive and makes a determination regarding the necessity for requalification of type tests performed with S1024. It must be noted in comparing the two adhesives that although S1119 demonstrates better material properties, it does not detract from the capabilities of S1024. The improvements made in S1119 represent an additional margin of safety and future capability in the adhesive.

3.0 TECHNICAL EVALUATION

Raychem supplies cable accessory products to the nuclear industry to meet a variety of termination and splicing needs. The products feature insulating, heat shrinkable parts coated with a nuclear grade adhesive. The function of the nuclear adhesive is to provide an environmental seal capable of protecting the encapsulated substrate from the environment throughout the life of the plant. The nuclear adhesive must be resistant to the deleterious effects of thermal aging and radiation exposure and be capable of functioning throughout a postulated LOCA/SLB event. In accordance with the guidelines of IEEE Standards 383 and 323, sections 1.5 and 6.8 respectively, the following evaluation is presented to analyze the effect of a modification of Raychem's nuclear grade adhesive on previous type testing.

The properties of the adhesive material that can be related to its function as a sealant for electrical applications are:

1 - Elongation
2 - Adhesion
3 - Water Absorption
4 - Electrical Properties
5 - Flow (as related to installation)

In order to evaluate the relative performance qualifications of S1024 and S1119 as nuclear grade sealants for Raychem products, relevant material and functional testing will be reviewed comparing the properties listed above. The effect of thermal aging, radiation, and LOCA environment on these properties will be evaluated. S1024 has undergone extensive product testing to the nuclear environment successfully. This evaluation, as stated in the Basis, is predicated on the previous qualification type testing of products using S1024 adhesive. The evaluation that follows presents test data in section 3.1 to demonstrate that S1119 has better material properties than S1024. The functional testing performed is summarized in section 3.2 and further substantiates the performance capabilities of the S1119 adhesive. Raychem's specification requirements (RT-1050/4 and /15) as well as a side-by-side specification comparison for the two adhesives are included in section 5.0. The RT-1050 specifications represent the basic material performance characteristics and requirements. It is evident that the material specification requirements for S1119 have been made more inclusive of requirements related to nuclear products.

3 1 MATERIALS TESTING

3.1.1 Elongation

Elongation is a useful index for evaluating the relative effects of thermal and radiation conditioning on polymeric materials. Elongation is considered here to demonstrate the relative stability of the S1119 material to S1024, which has repeatedly demonstrated satisfactory performance in functional LOCA testing. Table 1 presents comparative data on the elongation property of S1024 and S1119 after irradiation to 50, 100, and 150 Mrads. Measurements were made at room temperature, 170°C and 200°C. Table 2 presents elongation data for slab samples of the adhesives thermally aged for 1500 hours at 150°C and then irradiated to 200 Mrads. This would typically represent worst case LOCA Pre-conditioning. S1119 demonstrates better retention of elongation than S1024. Embrittlement of the adhesive, precipitated by a total loss of elongation, must be precluded to prevent cracking and maintain the function of the sealant. The data demonstrates, by comparison, that the S1119 is more resistant to embrittlement under the required service extremes. S1024 has demonstrated functional performance, S1119 has better elongation properties to contribute to its performance.

TABLE 1
Physical Properties After
Irradiation to 50, 100, and 150 Mrads
(Average of Four Measurements)

	S1024			S1119		
50 Mrads	23°C	170°C	200°C	23°C	170°C	200°C
Elongation (%)	288	96	93	360	318	250
100 Mrads						
Elongation (%)	194	80	60	448	312	220
150 Mrads						
Elongation (%)	128	60	55	396	220	155

TABLE 2
Physical Properties After 1500 Hours
at 150°C and 200 Mrads
(Average of Five Measurements)

	S1024			S1119		
	R.T.	170°C	200°C	R.T.	170°C	200°C
Elongation (%)	20	11	9	33	16	17

3.1.2 Adhesion

Although there is no quantitative correlation between adhesion and seal performance, it is clear that good adhesion promotes good sealing. The requirements for adhesion have been written into the material specifications for both S1024 and S1119 (ref. section 5.0). Table 3 gives a direct comparison of peel strengths for the two adhesives using a rolling drum peel strength test. Table 4 presents the same type comparative data after irradiation to 50 and 100 Mrads. The bond strength for S1119 is better than for S1024.

TABLE 3
Comparative Adhesive Peel Measurements
for S1024 and S1119

Peel Strength *
(Average of Four Measurements)

Adhesive Peel	S1024	S1119
WCSF to Polyethylene	18	32
WCSF to Neoprene	9	17
WCSF to Aluminum	11	28
WCSF to Copper	11	29
WCSF to Lead	10	29
WCSF to PVC	15	20
WCSF to Steel	12	27

*lbs. per linear inch

TABLE 4
Comparative Peel Strengths After Irradiation

Peel Strength (pli)
(Average of Five Measurements)

	S1024			S1119		
	0MR	50MR	100MR	0MR	50MR	100MR
WCSF to WCSF	92	51	19	90	81	71

The specification developed for S1119 also requires a minimum of 120 psi for lap shear strength after heat aging for 200 hours at 175°C. The lap shear strength is another method of measuring the bond strength of materials.

The improved adhesion properties of S1119 combined with excellent elongation properties create an ideal combination of properties for sealing applications.

3.1.3 Water Absorption

Resistance to water absorption is an important factor in seal performance to assure that insulation resistance values remain high enough to prevent excessive leakage currents.

The RT specification (see section 5.0) for S1119 has a more stringent water absorption requirement than the specification for S1024
(ref. RT 1050/15 vs. RT1050/4: 0.5% vs. 1.0%).

3.1.4 Electrical Properties

Both S1024 and S1119 have electrical insulating properties well in excess of the functional requirements. The RT specifications list minimum requirements. Both materials typically have volume resistivities greater than 10^{13} ohm-cm and dielectric strengths greater than 500 volts/mil (average of 5 readings on a 6 x 6 x .075 inch sheet specimen - see RT-1050, paragraph 4.3.3.2). Since the sealing distance is typically much greater than the insulation thickness of either the wire or the splice coverings, the electrical properties of the adhesives are very conservative.

3.1.5 Flow

Flow of the adhesives during application is an important property to assure complete sealing during application. Both adhesives have undergone extensive evaluation in product configurations by the Applied Technology Group to assure adequate flow and sealing during recovery (shrinking) of the various product configurations. Evaluations were conducted by visual observation of flow from the ends of the coated parts and by cross-sectional investigations. As expected, because of the similarity of base polymers and compounding materials, both the S1024 and S1119 exhibit adequate flow during installation.

3.1.6 PPS 3012/19 Testing(lab ref. #15206-20)

Testing in accordance with PPS 3012/19 was successfully performed on WCSF-500, lot N15738. The WCSF was coextruded with the new S1119 formulation. The results of the PPS tests are listed in Table 5. The adhesive peel testing was performed on substrates which had been abraded(as noted) prior to installing the WCSF tubing. The WCSF was shrunk onto the substrates by placing the prepared assembly into a 150^{o}C oven for 20 minutes. The adhesive peel testing was performed as described in PPS 3012.

The fungus resistance test on PPS 3012/19 was not performed since it had been determined that this test should be removed from the PPS.

3.1.7 PPS 3012/19 Rev. C

Qualification of a new S1119 adhesive formulation, in which a resin was replaced with a comparable alternative, was successfully performed in accordance with PPS 3012/19 Rev. C. See Table 6 for PPS test results comparing the “old” and the “new” S1119 formulations (raw data can be found in laboratory notebooks # 17521, 17192, 15206, and 18495).
During the course of this qualification effort, product performance requirements were examined. As a result, modifications which were determined to be more applicable to product function were made to PPS 3012/19. The “Aged” and “Aged and Irradiated” S1119 adhesive peel strength requirements were modified to WCSF/WCSF. In addition, dielectric constant, shear strength, and fungus tests were removed from the PPS.

Table 5. PPS 3012/19

Test	***PPS Requirement***	***WCSF-500, N15738 data (M14275-07, 93B-4898)***
Density	*0.87-0.97*	*0.9509*
Low Temperature Flexibility (4 hrs. @ -40°C)	*No Cracking*	*No Cracking*
Adhesive Peel Strength		
WCSF/Cu(abraded)	*10 pli, min.*	*31 pli*
WCSF/Neoprene(unabraded)	*10 pli, min.*	*17 pli*
WCSF/PE(abraded)	*8 pli, min.*	*44 pli*
WCSF/Pb(abraded)	*8 pli, min.*	*13 pli*
WCSF/Al(abraded)	*8 pli, min.*	*19 pli*
Shear Strength (20 min. @ 150°C)	*120 psi, min.*	*343 psi*
Dielectric Strength(0.075")	*400 v/mil, min.*	*515 v/mil*
Volume Resistivity	*$1x10^{12}$ ohm-cm, min.*	*3.8 x 10^{13} ohm-cm*
Dielectric Constant	*3.5 max.*	*2.0*
Water Absorption (24 hr. @ 23°C)	*0.5%, max.*	*0.2%*
Resistance to Liquids (24 hrs. @ 23°C)		
% weight change		
Detergent Solution	*1%, max.*	*0.1%*
Sunoco Cable Oil #147 (unavail.-used EPPA-016)	*10%, max.*	*2%*
Corrosive Effect (16 hrs. @ 121°C)	*No Corrosion*	*No Corrosion*
Blocking	*Free to First Degree*	*Free to First Degree*
Shear Softening Temperature	*70°C, min.*	*155°C*
Accelerated Aging		
Adhesive Peel Strength		
WCSF/Cu(abraded)	*10 pli, min.*	*35 pli*
Shear Strength		
Cu/Cu	*120 psi, min.*	*1100 psi*
Heat Aging & Irradiation (200 hrs. @ 175°C followed by 50 +2, -0 MRad)		
Adhesive Peel Strength		
WCSF/Cu(abraded)	*10 pli, min.*	*11 pli*
Shear Strength		
Cu/Cu	*115 psi, min.*	*658 psi*

Table 6. PPS 3012/19 Rev. C

Test	***Requirement Imperial Units***	***S1119 M14275-07 "Old"***	***S1119 M17109-49 "New"***
Density	*(0.87-0.97) g/cm^3*	*0.951 g/cm^3*	*0.958 g/cm^3*
Low Temperature Flexibility 4 hrs. at –(40±3)°C	*No Cracks*	*No Cracks*	*No Cracks*
Adhesive Peel Strength Substrates			
WCSF/WCSF	*10 lb./inch min.*	*57 lb./inch*	*76 lb./inch*
WCSF/ Cu	*10 lb./inch min.*	*31 lb./inch*	*31 lb./inch*
WCSF/Neoprene	*10 lb./inch min.*	*17 lb./inch*	*40 lb./inch*
WCSF/PE	*8 lb./inch min.*	*44 lb./inch*	*16 lb./inch*
WCSF/Pb	*8 lb./inch min.*	*13 lb./inch*	*35 lb./inch*
WCSF/Al	*6 lb./inch min.*	*19 lb./inch*	*22 lb./inch*
Dielectric Strength (0.075±5) inch wall	*400 V/mil min.*	*515 V/mil*	*600 V/mil*
Volume Resistivity	*1X10^{12} Ohm-cm min.*	*3.8 x 10^{13} Ohm-cm*	*7.77 x 10^{12} Ohm-cm*
Water Absorption 24 hrs. at (23±2)°C	*0.5% max.*	*0.2%*	*0.175%*
Resistance to Liquids 24 hrs. at (23±2)°C Weight Change			
(a) Detergent Solution	*1.0% max.*	*0.1%*	*0.15%*
(b) Cable Oil	*10.0% max.*	*2%*	*1.86%*
Corrosive Effect 16 hrs. at (135±2)°C	*No Corrosion*	*No Corrosion*	*No Corrosion*
Blocking (50±2)°C (Adhesive)	*Free to First Degree*	*Free to First Degree*	*Free to First Degree*
Softening Temperature	*60°C min.*	*68.5 °C*	*68.2°C*
Accelerated Aging 200 hrs. at (175±5)°C Adhesive Peel Strength, WCSF/WCSF	*10 lb./inch min.*	*30 lb./inch*	*53 lb./inch*
Heat Aging and Radiation 200 hrs. at (175±5)°C followed by (50+2, -0) Mrads Radiation Adhesive Peel Strength, WCSF/WCSF	*No Adhesive Failure*	*No Adhesive Failure*	*No Adhesive Failure*

3.2 FUNCTIONAL TESTING

Significant testing has been conducted to confirm the positive results of the material testing with functional product tests. Functional testing to the severe environmental extremes required for the product configurations verifies that the material properties of the adhesives are sufficient to perform their intended function under simulated installed conditions. The following functional tests have been conducted with the S1119 adhesive:

Reference	Report No.	Title/Description
(1)	EDR-5008	"Qualification of Adhesive Coated WCSF-N Heat Shrinkable Tubing to ANSI Cll9.1 1974"/Testing for sealed, insulated underground connector systems rated 600 volts.
(2)	EDR-5011	"Performance Test of Raychem WCSF-N Splices on EPR/Hypalon Wire"/LOCA test
(3)	EDR-5015	"Performance Test of Raychem Nuclear Motor Connection Kit (N-MCK) and Nuclear End Sealing Kit (N-ESK)"/LOCA test
(4)	EDR 5019	"Palo Verde I Test Report"/LOCA test
(5)	EDR-5032 EDR-5033 EDR-5034	"Environmental Test Report of Raychem Nuclear Cable Splice Assemblies"/LOCA test
(6)	EDR-5336	“Nuclear Products Requalification Testing”/LOCA test

Reference (1) describes testing done to ANSI Cll9.1, an insulated connector standard for underground systems. This test represents an extreme test of sealing integrity under very adverse operating conditions (excluding radiation). The S1119 adhesive as tested on WCSF-N splices passed all test requirements.

References (2), (3), (4), (5), and (6) describe simulated LOCA/MSLB (Loss of Coolant Accident/Main Steam Line Break) tests done on S1119 coated products. Reference (4), EDR-5019, also reports comparative tests with S1024. The LOCA/MSLB tests expose the adhesive to thermal aging and radiation exposure followed by high temperature, pressure, and steam while subjected to a conductive chemical spray. Successful completion of these tests verifies that the material properties of the S1119 adhesive are sufficient to maintain the electrical integrity of the splice sample during and subsequent to the environmental exposure. A summary of the basic test parameters for all these tests is given in Table 7.

TABLE 7
Functional Testing Summary

	Report No.	Description
(1)	EDR-5008	ANSI Cll9.1, WCSF-N, S1119 adhesive. Water Immersion; Flexing and Twisting after Heat and Cold Conditioning; Current Cycling/Water Immersion
(2)	EDR-5011	LOCA test; WCSF-N, S1119 adhesive. Aging - 168 hours at 150°C Radiation - 163.3 Mrads (+50 additional Mrads post LOCA) Temperature Profile - 340°F peak, 6 hours (2 peaks) Duration - 30 days with chemical spray
(3)	EDR-5015	LOCA test; NMCK and NESK, S1119 adhesive. Parameters same as EDR-5011.
(4)	EDR-5019	LOCA test; WCSF-N, S1119 and S1024 adhesive. Aging - Unaged, 168, 850, 1200, and 1500 hours at 150°C Radiation - 200 Mrads Temperature Profile - 400°F peak, 20 minutes >355°F, 6 hours Duration - 30 days; chemical spray 24 hours demineralized water spray to 30 days
(5)	(Wyle Test Report) 58442-1 58442-1 58442-3	LOCA/MSLB test; WCSF-N, NMCK, NPK, and NESK, S1119 adhesive. Aging - Unaged, 1000 and 1500 hours at 150°C 58442-2 Radiation - >200 Mrads Temperature Profile - 390°F peak, 4 minutes >314°F, 32.2 hours Duration - 30 days with chemical spray
(6)	EDR-5336	LOCA Test; Nuclear Products Requalification Testing Aging - Unaged, 878, and 1379 hours at 150°C Radiation - 196 MRads Temperature Profile - 425°F peak, 10 minutes, double peak, >360°F remaining 30 days Duration - 30 days with chemical spray

In the history of LOCA/MSLB testing performed by Raychem with either S1024 or S1119 no seal failures have ever been recorded.

4.0 CONCLUSION

IEEE Standards 323-1974 and 383-1974 require in sections 6.8 and 1.5 respectively that any modification to a product after type tests have been completed must be evaluated to determine the effect of the modification on the type tests. The concern and basis of this evaluation is to demonstrate, by evaluation of material and functional testing performed, that qualification type testing performed on Raychem products coated with S1024 adhesive are equally applicable to the same products coated with S1119.

The material testing performed demonstrates that in all properties related to the function of the adhesive as a wire splicing sealant for nuclear applications S1119 performs as well or better than S1024. The functional testing performed on S1119 includes four actual LOCA/MSLB performance tests with differing test parameters. The fact that functional LOCA/MSLB testing was performed eliminates the need to extrapolate material testing to cover functional tests. The functional testing includes both tubing products and molded parts coated with S1119.

The testing done by Raychem indicates that the modified adhesive S1119 is equivalent or superior in performance to S1024 as a wire splicing sealant for nuclear applications. It is reasonable to conclude, based on the data presented and the testing performed, that requalification of Raychem products previously type-tested and qualified for use within nuclear facilities using S1024 adhesive need not be required for the same products using S1119 adhesive.

Accordingly, products previously supplied with S1024 adhesive may be replaced with the same products coated with S1119 with no adverse effect on the products documented performance.

The PPS 3012/19 testing performed in 1997 indicates that the replacement of the zinc resinate tackifier has not had an adverse effect on the performance of S1119.

Qualification testing in accordance with PPS 3012/19 Rev. C was successfully performed for S1119 adhesive containing a new replacement resin. Results indicate that the resin change has not adversely affected the performance of the adhesive and that the form, fit, and function of the product has not been compromised.

5.0 ADHESIVE SPECIFICATIONS

RT-1050, /4, /15

COMPARISON OF SPECIFICATION REQUIREMENTS

PROPERTY	METHOD OF TEST Section	ASTM	UNIT	S1024 RT 1050/4 REQUIREMENT	S1119 RT 1050/15 REQUIREMENT	S1119 TYPICAL VALUES
Physical						
Visual	4.3.1.1	n/a		pass	pass	pass
Specific Gravity	4.3.1.2	D792		1.0 ± 0.05	0.92 ± .05	.92
Low Temp. Impact Brittleness	4.3.1.7	D746	°C	-40 max.	-40 max.	< -50
Heat Aging 200 hrs @ 175°C followed by test for adhesive peel to copper	4.3.1.9	D2671 Sect.49	lbs/in./width	-	10 min.	> 20
Blocking (adhesive @ 50°C)	4.3.1.8	D1146		pass	pass	pass
Adhesive Peel	4.3.1.9		lbs/in./width			
to polyethylene				10 min.	WCSF 8 min.	> 10
to neoprene				-	WCSF 10 min.	> 15
to PVC				15 min.	-	-
to steel				20 min.	-	-
viton to viton				25 min.	-	-
to aluminum				-	WCSF 6 min.	> 8
to copper				-	WCSF 10 min.	> 15
to lead				-	WCSF 8 min.	> 10
Shear Softening Temp.		D816	°C	-	70 min.	75
Chemical						
Water absorp. 24 hrs @ 25°C	4.3.2.1	D570	percent	1.0 max.	0.5 max.	< .3
Corrosive effect 16 hrs @ 121°C	4.3.2.2	*D2671		non-corr.	non-corr.	non-corr.
Fungus Resist.	4.3.2.5	G21		Rating of 1 or less	-	-
Solvent & Fluid						
Resistance	4.3.2.6	D543	percent			
Detergent Sol. (#12) 24 hrs. @ 25°C				1.0 max.	1.0 max.	< .8
Lube Oil (MIL-L-78-8) 1 week @ 23°C				10.0 max.	-	-
Transformer Oil (#49) 1 week @ 23°C				1.0 max.	-	-
Sunoco Cable Oil #147 24 hrs @ 25°C				-	10.0 max.	< 8
Lap Shear Strength		D1002	psi			
Copper/copper				-	120 min.	> 150
Copper/copper heat-aged 200 hrs @ 175°C				-	120 min.	> 150
Copper/copper heat-aged 200 hrs @ 175°C followed by 50 Mrad γ radiation (0.5-1.0 Mrad/hr)				-	115 min.	> 150
Electrical						
Volume Resistivity	4.3.3.1	D257	ohm-cm	10^{10} min.	10^{12} min.	> 10^{13}
Dielectric Strength	4.3.3.2	D149	volts/mil	500 min.	400 min.	> 500
Dielectric Constant		D150		-	3.5 max.	< 3.0

*Refer to D2671 Appendix A.1.5.2 for S1024 RT 1050/4 for Method of Test.
Refer to D2671 Sect. 79, Method A for S1119 RT 1050/15 for Method of Test.

DM:bt