TEST METHODS AND PROTOCOL FOR EVALUATING SPLICING CEMENT PERFORMANCE IN EPDM ROOFING MEMBRANE SYSTEMS

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The climate in the United States varies more than any other country in the world—from the hot and dry summers in the Southwest to very cold winters in the North Central states to the hot and humid summers of the Southeast. EPDM roofing systems are subject to those extremes in temperature, moisture and wind on both a daily and seasonal basis. Splicing cements as well as the membrane must resist these stresses induced by the climate cycles of the weather.

Ponds of water on flat roofs with improper drainage are more common than most roofing experts would like to admit and can remain in place for many weeks or months. Moisture is also available to attack seams from underneath the membrane; especially if the old deteriorated moisture-laden roof is left in place. Stresses are applied to the splice from building movement, thermally induced by large temperature swings, and by the wind. The splicing cement must resist these various aging and stress factors to offer long-term performance.

Adhesive building tack or "green" strength is very important when assembling a cured membrane splice on an uneven insulation board or when forming an inside or outside corner with flashing. The results of insufficient adhesive green strength, especially when the membrane is hot, are "fishmouths" and open corners. Splicing cements must have good green strength.

This paper will examine the test methods and protocol used to evaluate the performance of splicing cements used in EPDM roofing membrane systems. These splice cement tests are designed to simulate the stresses and the environmental conditions to which the splice system is subjected in actual practice. The adhesive screening tests, adhesive performance characterization tests and adhesive field tests are described.

KEYWORDS

Adhesive green strength, adhesive test methods, butyl adhesive, creep-rupture, dead load, EPDM adhesion, peel strength, shear strength, single-ply roofing, splice aging tests, splicing cement, test methods, wind uplift.

INTRODUCTION

Thousands of gallons of adhesives are used each month to splice together and adhesively attach EPDM membrane in various adhered, ballasted and mechanically-fastened roofing systems. Adhesives are a key factor to the success of EPDM single-ply roofing systems' long-term service life under variable environmental conditions. There are elements of the environment that try to age the adhesive bondline throughout the roof system's life. There are also elements that try, during the splice assembly, to affect the quality of the splice from the time the splice is made.

This paper will describe the test methods and protocol used to simulate those elements in the laboratory to evaluate the performance of a splicing cement. Over the years, new test methods and protocol have continued to be developed as information has been gained from on-the-roof usage. With this increase came the subtle, but definite formation of three different levels or tiers of tests. These consist of adhesive screening tests, adhesive performance characterization tests and adhesive field testing. A particular splicing cement must excel at each level or tier before it passes on to the next tier. This paper will concentrate on the laboratory portions of the testing, *adhesive screening* and *performance characterization tests.*

ADHESIVE SCREENING TESTS

These tests are intended to be a basic adhesive evaluation designed to "weed out" cements that obviously should not be considered for use as field splicing cements. The tests are geared toward contact-type cements, as this has been by far the most successful method of forming field splices, but, the tests can be modified for other types of cements and adhesive systems. All are laboratory tests. Where testing involves bonding to rubber membrane, the membrane is always 1.52mm (.060 in.) thick, cured and non-reinforced, unless stated otherwise. All tests are performed against or with a control. (An adhesive control is the adhesive that is the standard of performance at that current period of time.)

Cement Application Tests

- Qualitative Bonding Tack—The qualitative bonding tack test is a simple "touch" type of test that determines an adhesive's ability to form a bond after a given drying time (usually two hours after application for contact adhesives). After being applied to the EPDM membrane and allowing it to dry for the given drying time, a particular adhesive is graded on a five point scale (poor, fair, good, very good and excellent) on its ability to form a tight bond quickly using only light hand contact pressure. An adhesive must have "good" bonding tack to receive serious consideration (in most cases, a "good" rating would be equal to the control).

- Viscosity, % Solids, Rheology—These properties, viscosity, % solids and rheology, are for liquid adhesives and are grouped together since they tend to be interrelated. Generally, none are a point of contention unless they are significantly different than the control. The important point here is that one must be able to apply the cement with simple hand tools, either a brush or a roller. If the viscosity, % solids or rheology makes this difficult, then the cement probably will not receive further consideration. Viscosity is typically determined by using a Brookfield rotating disc/spindle viscometer. Solids (%) is determined from the change in weight of a sample after heating it in an oven. Rheology is determined by how smoothly and evenly the adhesive applies to a substrate, and how well it levels out after application.
- Drying Time—To determine drying time, a liquid contact cement is expected to dry to the "touch" (does not attach to or string from the substrate when touched with a dry finger) in about 30 minutes, indoors. Drying times significantly longer or shorter than this are generally avoided.
- Green Strength Lap Shear Adhesion, Room and Elevated Temperature—The green strength lap shear test is based on ASTM D 816–Method B, a constant rate adhesion test used to evaluate the initial or green strength of a cement. The cleaned substrates are coated with the cements, then are allowed to dry and are bonded to form a 2.54cm (1 in.) overlap. Lap shear specimens, 2.54cm (1 in.) wide, are then cut from the bonded sample. Elevated temperature green strength specimens are conditioned for 15 minutes at room temperature, then 15 minutes at the elevated temperature before testing.* Room temperature green strength specimens are conditioned for one hour at room temperature before testing. Testing is performed at a rate of 5.08cm (2 in.)/min. (see Tables 1 and 2).

Short-Term Aging Performance Tests

Initial and Short-Term Aged Room and Elevated Temperature Adhesion Tests—Initial and short-term aged tests consist of performing the standard type of peel (according to ASTM D 1876) and lap shear (ASTM D 816–Method B) adhesion tests. The tests are done at room and elevated temperature at a constant rate of separation (see Figure 1) after a few basic aging conditions. The cleaned substrates are coated with the cements, then are allowed to dry and are bonded. The bonding process involves smoothing the substrates together by hand and then rolling them in two directions with a 5.08cm (2 in.) wide steel hand roller. One set of samples are bonded with a 2.54cm (1 in.) overlap and another set with a minimum 15.24cm (6 in.) overlap. It is desirable to make these samples as large as is physically possible as it reduces the variability in the test results that are obtained from smaller samples. Peel and lap shear adhesion specimens that are 2.54cm (1 in.) wide are then cut from the bonded samples. All specimens are allowed to age for seven days at room temperature. One set of peel and lap shear adhesion specimens are then tested at room and elevated temperature at a rate of 5.08cm (2 in.)/min. The other aging conditions that are generally used are:

- 30 days at room temperature, 22°C (72°F).
- Seven and 30 days in a 70°C (158°F) forced air oven.
- Seven and 30 days in a 70°C (158°F) circulating water bath.

These conditions can vary slightly, but the aging temperatures should not be increased to the point where unnatural degradation reactions begin to occur. (Example: aging Neoprene adhesives at temperatures exceeding 100°C (212°F) where dehydrohalogenation begins to take place.)

Once all the test results are obtained, the results are compared with the control(s) and with that adhesive's initial (unaged) results (see Table 3). The adhesive should not only compare well with the control(s), but the aged results should not be significantly lower than the initial results.

ADHESIVE PERFORMANCE CHARACTERIZATION TESTS

Adhesive performance characterization testing is designed to take a promising cement candidate and completely characterize it. This level of laboratory testing has been developed over the years to simulate various conditions observed in the field. Because of this, these test methods tend to be very specific and many have yet to become standard test methods, although most of the tests have borrowed parts from existing standards. Also, because its source is based on field observations, this tier tends to be constantly evolving. These tests have been successful in screening out undesirable adhesive candidates, increasing the success ratio of the final field testing stage.

Cement Application Tests

- Qualitative Bonding Tack—The qualitative bonding tack method is basically the same as the one that is used in the screening tests, except it is done after specific times at specific temperatures. The cement is applied to the substrate and then placed into the temperature condition being evaluated. The cement is then allowed to dry to the touch, when the time period being evaluated begins. At the completion of the time period, cement-coated substrates are mated with light hand pressure and the bond formed is rated on the five point scale stated previously. The times and temperatures generally used are:
  - 15 minutes at 22°C (72°F–room temperature).
  - 30 minutes at 22°C.
  - 60 minutes at 22°C.
  - 90 minutes at 22°C.
  - 15 minutes at 70°C (158°F).
  - 15 minutes at 4°C (39°F).
- Viscosity Stability/Shelf Life—The viscosity stability/shelf life test is an evaluation of how much the viscosity (see Viscosity, % Solids, Rheology) of the cement changes with time after the adhesive is manufactured to the point that it is no longer usable. The method involves taking a full-container sample of the cement and storing it at a controlled temperature. Every month, the viscosity is meas-

**The elevated temperature test temperature varies for color of adhesive and membrane combination being tested. White adhesive/membrane is tested at 50°C (122°F) and black adhesive/membrane is tested at 70°C (158°F).*

ured and compared to the initial viscosity. Periodically, adhesion samples will be prepared to ensure the product is still capable of preparing a field splice of good performance. A cement is expected to have a minimum shelf life of nine months.

- Outdoor Field Splice Preparation—The outdoor field splice preparation test is the actual preparation of field splices outdoors on top of insulation boards under varying conditions, always including the extremes of a cold January-like day and a hot July-like day. The splices prepared are typically 1.5m-3.0m (5-10 ft.) long. The cement is evaluated for its ease of application; its drying time and bonding tack under actual conditions. Specimens are cut and brought into the laboratory for accelerated aging, and are then tested for peel and lap shear strength (see Table 4). The remaining splice is left outdoors on a rack with no slope from which specimens are cut periodically, brought into the laboratory, and tested for peel and lap shear adhesion.

Long-Term Performance Tests

- Dead Load Shear Adhesion–Room and Elevated Temperature—In the dead load shear adhesion test, a lap shear specimen (prepared in the same manner as the lap shear adhesion test) is exposed to a constant load instead of a constant rate (see Figure 2). The test method is a modification of ASTM D 3654 which was developed for creep failure of pressure sensitive tapes.

  After bonding, specimens are tested after aging for one hour at RT (room temperature), one day at RT, seven days at RT, and 30 days at 70°C. Generally, the test load at RT is 1000g (2.2 lbs.) and 500g (1.1 lbs.) at elevated temperature. The time it takes for the overlap to separate completely, and the weight to drop is recorded. If no failure occurs after seven days, the test is stopped (see Table 5), the weight is increased, and the test is repeated until a failure is obtained.

- Dead Load Peel Adhesion or Creep Rupture Test–Room and Elevated Temperature—Similar in concept to the dead load shear adhesion test (see Figure 2), the dead load peel adhesion test uses a peel adhesion specimen. The time and temperature the specimen is conditioned before testing is the same as for the dead load shear test. Generally, the test load used is 350g (0.77 lbs.) at RT and 100g (0.22 lbs.) at elevated temperature for the one hour and one day aged samples at RT. And 750g (1.65 lbs.) at RT and 225g (0.50 lbs.) at elevated temperature for the seven days at RT and 30 days at 70°C (158°F) aged samples (see Table 6). Like the dead load shear test, weights are changed to generate peel separation.

- Long-Term Aged Adhesion Tests–Low, Room and Elevated Temperature—The tests and test methods employed for long-term aged adhesion tests are the same as those used in the constant rate peel and lap shear adhesion tests performed at the screening test level. The only difference is that adhesion testing at −20°C (−4°F) is included to see if the cement begins to stiffen or become brittle at low temperature. Also, aging times are increased significantly:
  - Seven, 30, 60, 90, 120 and 365 days at RT.
  - Seven, 30, 60, 90, 120 and 365 days in a 70°C forced air oven.
  - Seven, 30, 60, 90, 120 and 365 days in a 70°C circulating water bath.

  At this level, the adhesion of rubber to other substrates like galvanized and stainless steel is also evaluated. The compiled test results are compared with the control(s) and that adhesive's initial (unaged) results. The aged results should not be significantly lower than the initial results (see Table 7).

- RMA Test Method RP-10—Adhesion samples are prepared according to this RMA (Rubber Manufacturers Association) test method and are aged under a simulated weathering cycle that consists of the following conditions:
  - 24 hours in a 80°C (176°F) forced air oven.
  - 72 hours in a 80° C circulating water bath.
  - Eight hours in a −18°C (0°F) freezer.
  - 64 hours in a 80°C circulating water bath.

  The test method requires that the bonded samples be exposed to four of the above cycles, although eight or more cycles are probably necessary to weed out most undesirable splicing cements. The cycled samples are then tested in a peel configuration and are compared to bonded samples that have been aged at RT over the same period. No significant reduction in the peel strength of the cycled sample when compared to the RT aged sample is allowed, which is a more stringent variation of the RMA method (see Table 8).

- Wind Uplift Test—Due to the increasing popularity of mechanically-attached systems, this has become a key test. A 1.52m (5 ft.) x 2.74m (9 ft.) uplift table is prepared according to standard FM (Factory Mutual) methods (see Figure 3). This is the only test in either the screening or performance characterization levels where 1.14mm (0.45 in.) thick, cured reinforced membrane is used. The assembled table is allowed to age for a minimum of seven days before it is tested. The test is performed at room temperature. During testing, the pressure under the table is increased at 66.7 Newton (15 lbs.) increments, held for one minute at the new level. The cycle is repeated until the system fails (see Table 9).

FIELD TESTING

After the laboratory testing is completed, field testing is the final evaluation a splicing cement candidate has to complete before it can be approved as a qualified product. As extensive as laboratory testing is, field testing is still a must. This is because laboratory testing tends to be single element testing, whereas natural aging combines elements that can create synergistic accelerated aging. This testing alone requires a minimum time period of two years to complete. When added to the time period required to complete the previous levels, it becomes obvious why the splicing cement approval process can require three or more years to complete.

This testing is composed of two basic levels. In the initial level, the cement is evaluated on various small projects, taking into account typical roof environments, substrates and application conditions. The application characteristics in the hands of a roofer are also closely observed. When the project is complete, long-term performance is evaluated by obtaining sample cuts from the projects, usually at six month or one year intervals. In the second level, the cement is introduced to the field in a controlled manner into various test markets. If the performance of the adhesive system continues to be satisfactory after the test market period, the product becomes fully qualified. Research and development will continue to monitor the quality control test results and the field performance of the product.

CONCLUSION

In this paper, the laboratory test methods and protocol of the adhesive screening tests and adhesive performance characterization tests, designed to simulate the application and service environments, were described. Finally, the culmination of all the laboratory and outdoor testing is reached in the adhesive field testing protocol. This protocol puts the splicing cement into the actual environments in which it must perform. Successful completion of all the tests, which requires years to complete, insures the long-term field performance of the splicing cement.

	Adhesive Type		
Adhesion Test	**Butyl Contact Adhesive A Control**	**Butyl Contact Adhesive B**	**Butyl Contact Adhesive C**
RT Lap Shear Adhesion—kPa (psi)	64.8 (9.4)	95.2 (13.8)	58.6 (8.5)
70°C Lap Shear Adhesion—kPa (psi)	26.9 (3.9)	53.1 (7.7)	26.2 (3.8)
Status	N/A	Continue work on this adhesive.	Comparable to control, probably continue to work with it.
Instron Jaw Speed = 5.08cm (2 in.)/min.			

Table 1 *Room temperature and 70°C green strength lap shear adhesion test results of two butyl contact adhesives and a butyl contact adhesive control.*

	Adhesive Type			
Adhesion Test	**Neoprene Contact Adhesive**	**Butyl Adhesive Tape**	**Butyl Contact Adhesive A**	**Butyl Contact Adhesive B**
RT Lap Shear Adhesion—kPa (psi)	67.6 (9.8)	87.6 (12.7)	64.8 (9.4)	95.2 (13.8)
70°C Lap Shear Adhesion—kPa (psi)	29.0 (4.2)	30.3 (4.4)	26.9 (3.9)	53.1 (7.7)
Instron Jaw Speed = 5.08cm (2 in.)/min.				

Table 2 *Comparative results of four adhesive systems in green strength lap shear adhesion.*

	Adhesive Type					
	Butyl Contact Adhesive A Control		**Butyl Contact Adhesive B**		**Butyl Contact Adhesive C**	
Aging/Adhesion Test	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**
7 Days @RT Peel Adhesion—kN/m (lbs/in)	1.05(6.0)	0.16(0.9)	1.17(6.7)	0.35(2.0)	0.35(2.0)	0.03(0.2)
Lap Shear Adhesion—kPa (psi)	193.1(28.0)	73.1(10.6)	250.3(36.3)	83.4(12.1)	77.9(11.3)	38.6(5.6)
30 Days @70°C Peel Adhesion—kN/m (lbs/in)	1.45(8.3)	0.38(2.2)	1.61(9.2)	0.89(5.1)	1.05(6.0)	0.33(1.9)
Lap Shear Adhesion—kPa (psi)	250.3(36.3)	109.7(15.9)	335.2(48.6)	188.3(27.3)	220.7(32.0)	98.6(14.3)
Status	N/A	N/A	Continue work on this adhesive.	Continue work on this adhesive.	Eliminate this adhesive.	Eliminate this adhesive.
Instron Jaw Speed = 5.08cm (2 in.)/min.						

Table 3 Initial and short-term aged adhesion test results of two butyl contact adhesives and a butyl contact adhesive control.

	Adhesive Type					
	Neoprene Contact Adhesive Control		**Butyl Contact Adhesive A**		**Butyl Contact Adhesive B**	
Aging/Adhesion Test	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**
7 Days Outdoors (January) + 7 Days @RT Peel Adhesion—kN/m (lbs/in)	0.19(1.1)	0.31(1.8)	1.00(5.7)	0.17(1.0)	1.07(6.1)	0.35(2.0)
Lap Shear Adhesion—kPa (psi)	113.1(16.4)	93.8(13.6)	257.2(37.3)	70.3(10.2)	271.7(39.4)	97.2(14.1)
7 Days Outdoors (January) + 7 Days @70°C Peel Adhesion—kN/m (lbs/in)	0.23(1.3)	0.10(0.6)	0.96(5.5)	0.23(1.3)	1.01(5.8)	0.52(3.0)
Lap Shear Adhesion—kPa (psi)	119.3(17.3)	65.5(9.5)	271.0(39.3)	95.2(13.8)	320.7(46.5)	151.0(21.9)
Instron Jaw Speed = 5.08cm (2 in.)/min.						
Comments: The weather conditions at the time of sample preparation were −1°C (30°F), overcast and no breeze. While aging outdoors, the temperature ranged from −13°C (9°F) to 6°C (42°F).						

Table 4 Comparative results of three contact adhesives in peel adhesion and lap shear adhesion when samples were prepared outdoors on a January day.

	Adhesive Type			
	Neoprene Contact Adhesive	**Butyl Adhesive Tape**	**Butyl Contact Adhesive A**	**Butyl Contact Adhesive B**
Test @RT—Time To Failure (min)				
1 hr @RT after bonding	218	109	5450	No Failure *
1 day @RT after bonding	4970	1134	No Failure *	No Failure *
1 week @RT after bonding	No Failure *	3285	No Failure *	No Failure *
* Test stopped at 1 week.				
Load = 1000g (2.2 lbs.)				

Table 5 Comparative results of four adhesive systems in dead load shear adhesion, tested at room temperature.

	Adhesive Type		
	Neoprene Contact Adhesive Control	Butyl Contact Adhesive A	Butyl Contact Adhesive B
Test @RT—Time to Failure (min)			
1 hour @RT after bonding (380g (0.77 lbs) load)	380	215	633
1 week @RT after bonding (750g (1.65 lbs) load)	83	3160	No Failure *
30 days @70°C after bonding (750g (1.65 lbs) load)	76	No Failure *	No Failure *
* Test stopped at 1 week.			

Table 6 Comparative results of three contact adhesives in dead load peel adhesion, (creep rupture), tested at room temperature.

	Adhesive Type							
	Neoprene Contact Adhesive		Butyl Adhesive Tape		Butyl Contact Adhesive A		Butyl Contact Adhesive B	
Aging/Adhesion Test	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**	**RT Test**	**70°C Test**
7 Days @RT Peel Adhesion—kN/m (lbs/in)	0.33(1.9)	0.30(1.7)	1.00(5.7)	0.10(0.6)	0.89(5.1)	0.14(0.8)	0.91(5.2)	0.26(1.5)
Lap Shear Adhesion—kPa (psi)	133.1(19.3)	87.6(12.7)	120.0(17.4)	35.2(5.1)	191.0(27.7)	80.0(11.6)	232.4(33.7)	84.1(12.2)
120 Days @RT Peel Adhesion—kN/m (lbs/in)	0.44(2.5)	0.40(2.3)	1.45(8.3)	0.26(1.5)	0.93(5.3)	0.26(1.5)	1.57(9.0)	0.47(2.7)
Lap Shear Adhesion—kPa (psi)	151.0(21.9)	84.1(12.2)	177.2(25.7)	62.8(9.1)	226.9(32.9)	91.7(13.3)	258.6(37.5)	93.1(13.5)
120 Days @70° Peel Adhesion—kN/m (lbs/in)	0.35(2.0)	0.05(0.3)	1.17(6.7)	0.33(1.9)	1.32(7.6)	0.49(2.8)	1.54(8.8)	1.07(6.1)
Lap Shear Adhesion—kPa (psi)	133.1(19.3)	59.3(8.6)	243.4(35.3)	125.5(18.2)	294.5(42.7)	156.6(22.0)	352.4(51.1)	211.7(30.7)
120 Days in 70°C Water Peel Adhesion—kN/m (lbs/in)	0.14(0.8)	0.03(0.2)	0.96(5.5)	0.40(2.3)	0.99(5.6)	0.65(3.7)	0.91(5.2)	0.77(4.4)
Lap Shear Adhesion—k/Pa (psi)	80.0(11.6)	43.4(6.3)	267.6(38.8)	142.8(20.7)	245.5(35.6)	128.3(18.6)	244.1(35.4)	162.1(23.5)
Instron Jaw Speed = 5.08cm (2 in.)/min.								

Table 7 Initial and long-term aged comparative adhesion results of four adhesive systems bonding cured EPDM rubber to itself.

	Adhesive Type		
	Neoprene Contact Adhesive	**Butyl Contact Adhesive A**	**Butyl Contact Adhesive B**
Aging/Adhesion Test			
28 Days @RT RT Peel Adhesion—kN/m (lbs/in)	0.24(1.4)	0.89(5.1)	0.91(5.2)
70°C Peel Adhesion—kN/m (lbs/n)	0.19(1.1)	0.19(1.1)	0.30(1.7)
4 RP-10 Cyles RT Peel Adhesion—kN/m (lbs/in)	0.14(0.8)	1.00(5.7)	1.08(6.2)
70°C Peel Adhesion—kN/m (lbs/in)	0.05(0.3)	0.28(1.6)	0.54(3.1)
Instron Jaw Speed = 5.08cm (2 in.)/min.			

Table 8 *Comparative results of three contact adhesives when tested according to RMA's Test Method RP-10.*

	Adhesive Type	
	Butyl Contact Adhesive A	**Butyl Adhesive Tape**
Air Pressure of Uplift Table at Failure:	667 Newtons (150 lbs)	467 Newtons (105 lbs)
Comments:	No seam failure. Failure is in membrane around fastener plate.	Seam failure.

Table 9 *Comparative results obtained with two adhesive systems on a wind uplift table test of a mechanically-attached roofing system.*

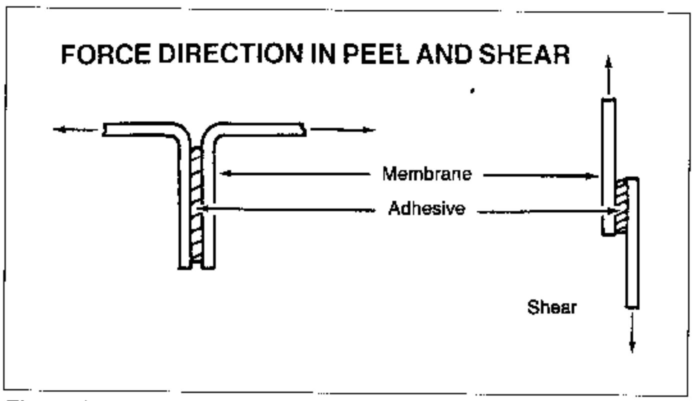

Figure 1 *Force direction in peel and shear.*

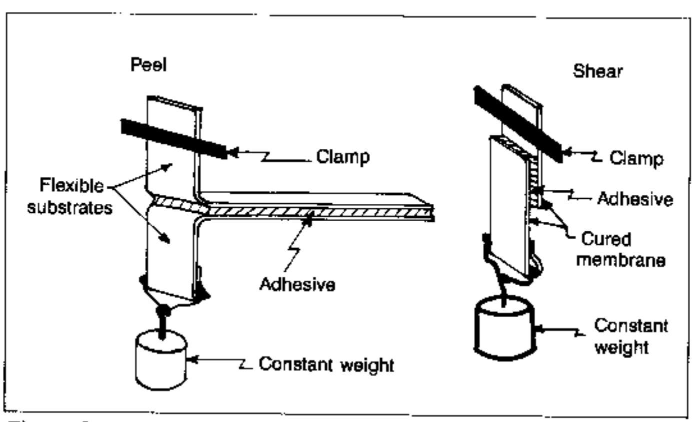

Figure 2 *Diagram of dead load adhesion samples.*

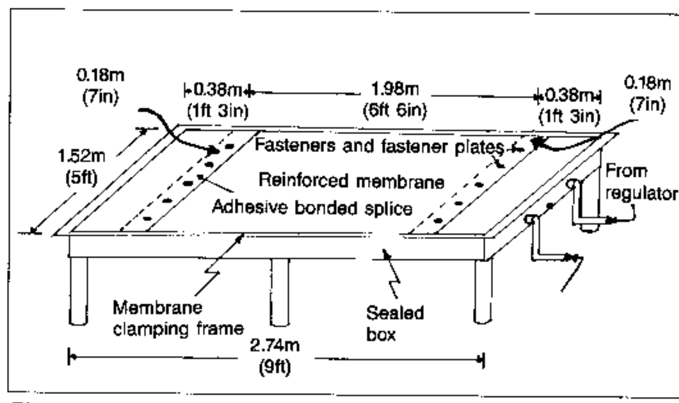

Figure 3 *Diagram of wind uplift table.*