15 July 2010

Committee C24 on Building Seals and Sealants
Subcommittee C24.40 on Weathering

Research Report: C24-1057

Interlaboratory Study to Establish Precision Statements for ASTM C1519-10, Standard Practice for Evaluating Durability of Building Construction Sealants by Laboratory Accelerated Weathering Procedures

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1. Introduction:

Interlaboratory Study 112 was conducted to establish a precision statement for C1519, Standard Practice for Evaluating Durability of Building Construction Sealants by Laboratory Accelerated Weathering Procedures.

2. Test Method:

The Test Method used for this ILS is C1519-10. To obtain a copy of C1519, go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

3. Participating Laboratories:

The following laboratories participated in this interlaboratory study

1. Dow Corning
2200 W Salzburg Rd
DC2 Building
Midland, MI 48686
US
Ken Yarosh
KEN.YAROSH@DOWCORNING.COM

2. Momentive
260 Hudson River
Waterford, NY
12188
US
David French
david.french@momentive.com

3. Schnee-Morehead
111 N Nursery Rd
Irving, TX
75060
US
Lauri Marino
lorim@itwsm.com

4. Description of Samples:

There were 4 samples of varying targeted results used for this study. Each sample was prepared and distributed by Steve Evans. Below is a list of the samples with the corresponding supplier:

1. Acrylic Waterbourne
   Provided by DAP Inc

2. MS Sealent
   Provided by BASF Construction Chemicals

3. Polyurethane
   Provided by BASF Construction Chemicals

4. Silicone
   Provided by Dow Corning Corp.

5. Interlaboratory Study Instructions

Laboratory participants were emailed the test program instructions. For a copy of the instructions, please see Annex A.

6. Description of Equipment/Apparatus[1]:

For information on the equipment/apparatus used by each laboratory, please see Annex B.

7. Data Report Forms:

Each laboratory was provided with a data report form for the collection of data. A copy of the data is provided in Annex C.

Please note: The laboratories have been randomly coded and cannot be identified herein.

8. Statistical Data Summary:

A summary of the statistics calculated from the data returned by the participating laboratories is provided in Annex D.

9. Precision and Bias Statement:

9.1 The precision of this test method is based on an interlaboratory study of ASTM C1519 - 04 Standard Practice for Evaluating Durability of Building Construction Sealants by Laboratory Accelerated Weathering Procedures, conducted in 2009. A total of three laboratories participated in this study, recording observations of the total loss of bond in adhesion and cohesion, cracking, crazing, chalking, and discoloration. Observations were recorded on a scale from 0 – 5, with 0 indicating no observed change, and 5 indicating excessive change. Observations on triplicate specimens of four different sealant materials (were made regularly every 4 weeks beginning with the "4 week weathering / movement cycle," continuing through the "48 week weathering / movement cycle," and then less frequently afterwards. One of the three participating laboratories stopped reporting all observations after Week 28, and another laboratory stopped reporting data after Week 32. Details of every rating reported in this study can be found in ASTM Research Report No. C24-1057.[i]

9.1.1 *Repeatability limit (r)* - Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the

[1] The equipment listed was used to develop a precision statement for C1519-10. This listing is not an endorsement or certification by ASTM International.

same operator using the same equipment on the same day in the same laboratory.

9.1.1.1 Due to a lack of significant and consistent observable change in most of the selected analyses, little repeatability data is available for the time periods from 4 – 24 weeks.

9.1.1.2 Repeatability limits for ratings reported at 28 weeks are listed in Tables 1 - 5 below.

9.1.1.2.1 Repeatability limits for ratings reported by a single laboratory after Week 28 are listed in Tables 1a – 5a for comparison.

9.1.2 *Reproducibility limit (R)* - Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.1.2.1 Due to a lack of significant and consistent observable change in most of the selected analyses, little reproducibility data is available for the time periods from 4 – 24 weeks.

9.1.2.2 Reproducibility limits for ratings reported at 28 weeks are listed in Tables 1 - 5 below.

9.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E 177.

9.1.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would normally have an approximate 95% probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95% probability limit would imply. Consider the repeatability limits and the reproducibility limits as general guides, and the associated probability of 95% as only a rough indicator of what can be expected.

Table 1. Chalking at 28 weeks
(3 laboratories)

Sample	Average [ii] $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Waterborne Acrylic	0	0	0	0	0
Silicone	0	0	0	0	0
MS Sealant	0	0	0	0	0
Polyurethane	0	0	0	0	0

Table 1a. Chalking (> 28 weeks)
(1 laboratory)

Sample	Average $\bar{x}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Waterborne Acrylic @ 69 weeks	0	0	0
Silicone @ 69 weeks	0	0	0
MS Sealant @ 69 weeks	0	0	0
Polyurethane @ 48 weeks	0	0	0

Table 2. Cracking at 28 weeks
(3 laboratories)

Sample	Average [ii] $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Waterborne Acrylic	0.6	0.9	1.2	2.5	3.4
Silicone	0.7	0	1.2	0	3.2
MS Sealant	0	0	0	0	0
Polyurethane	1.7	0.4	2.4	1.1	5.0 [iii]

Table 2a. Cracking (> 28 weeks)
(1 laboratory)

Sample	Average $\bar{x}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Waterborne Acrylic @ 69 weeks	2.3	0.6	1.6
Silicone @ 69 weeks	2.0	0	0
MS Sealant @ 69 weeks	1.0	0	0
Polyurethane @ 48 weeks	4.0	0	0

Table 3. Crazing at 28 weeks
(3 laboratories)

Sample	Average [ii] $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Waterborne Acrylic	1.7	0	1.5	0	4.3
Silicone	0	0	0	0	0
MS Sealant	0	0	0	0	0
Polyurethane	0.5	0	0.7	0	2.0

Table 3a. Crazing (> 28 weeks)
(1 laboratory)

Sample	Average $\bar{x}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Waterborne Acrylic @ 69 weeks	3.0	0	0
Silicone @ 69 weeks	0	0	0
MS Sealant @ 69 weeks	0	0	0
Polyurethane @ 48 weeks	2.0	0	0

Table 4. Discoloration at 28 weeks
(3 laboratories)

Sample	Average [ii] $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Waterborne Acrylic	0.3	0	0.6	0	1.6
Silicone	0	0	0	0	0
MS Sealant	0	0	0	0	0
Polyurethane	3.5	0	0.7	0	2.0

Table 4a. Discoloration (> 28 weeks)
(1 laboratory)

Sample	Average $\bar{x}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Waterborne Acrylic @ 69 weeks	1.0	0	0
Silicone @ 69 weeks	0	0	0
MS Sealant @ 69 weeks	0	0	0
Polyurethane @ 48 weeks	4.0	0	0

Table 5. Total Loss of Bond at 28 weeks
(3 laboratories)

Sample	Average [ii] $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Waterborne Acrylic	0.2	0.7	0.7	1.9	1.9
Silicone	0.3	0	0.6	0	1.6
MS Sealant	0	0	0	0	0
Polyurethane	1.2	1.1	1.9	3.0	5.0 [iii]

Table 5a. Total Loss of Bond (> 28 weeks)
(1 laboratory)

Sample	Average $\bar{X}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Waterborne Acrylic @ 69 weeks	2.3	0.6	1.6
Silicone @ 69 weeks	2.5	0.7	2.0
MS Sealant @ 69 weeks	2.0	0	0
Polyurethane @ 48 weeks	4.0	0	0

9.2 Bias— At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

Annex A:

Thanks to all of the participants in ILS #0112.

As we discussed in our January meeting, during this month's weathering cycle, please summarize your data so far and forward your results to John Gallagher at ASTM. He will use this information to develop a P&B statement for our June meeting. Please continue your weathering cycles. We will decide in our June meeting whether to continue or to stop the ILS.

Here is the form again:

I hope to see you in Vancouver.

Regards,

Ken

Thanks to everyone for participating in ILS #0112.

For our upcoming meeting in Ft. Lauderdale, you do not need to bring samples or test results. We will continue accelerated weather exposure cycles and plan on reviewing final data in our June 2009 meeting in Vancouver. For our meeting next week, we can discuss any problems, issues, questions or learnings from the ILS so far.

See you in sunny Ft. Lauderdale.

Hopefully, everyone is well under way with the round robin testing for ILS#0112.

For clarification, after every 4 week accelerated weather exposure cycle, samples are to be subjected to 6 movement cycles at +/-25% at room temperature in accordance with ASTM C1519. After this exposure, samples are evaluated for total bond loss and other changes. With help from John Gallagher of ASTM, we have developed this Excel form which can be used to record test results.

<< File: ILS# 112 Excel Data Report Form.xls >>
Thanks to everyone for their help.

RR: **C24-1057**

ILS# 0112
Subcommittee: C24.40
Technical Contact: Kenneth Yarosh
Staff Manager: Steve Mawn
Standard: C1519

Labs:

1. Rohm & Haas – Vicki Demerast (QUV & Xenon Arc)
2. Momentive – David French (QUV only)
3. BASF – Bill McCann (QUV only)
4. Degussa – Rodney Conn (QUV only)
5. Dow Chemical – Hermann Wentz (QUV & Xenon Arc)
6. Klosowski Scientific – Jerry Klosowski (QUV only)
7. Gardner Gibson – Morton Jones (QUV only)
8. Schnee-Morehead - Lauri Marino (QUV only)
9. Dow Corning – Ken Yarosh (QUV only)
10. DAP – Tom Hairston (QUV & Xenon Arc)

Materials:

Tensile Adhesion Teflon Jigs (1 per test sample)
1" x 3" Anodized Aluminum (2 per test sample)–
Sealants: 4 sealant types (1 component):

1) Acrylic Waterborne
2) Polyurethane
3) MS Sealant
4) Silicone

All sealants rated at +/-25% movement, white in color. MSDS available through Test Block Company, Steve Evans (989-797-3700)

Distributor: Test Block Company, Steve Evans (989-737-3700)

Replicates: 3 test samples of each sealant type

Analyses:

Record the following as stated in the proposed revision of C1519 Practice

1) After each 4 week weathering cycle, extend the sealant to the prescribed extension (25%) and measure and record to the nearest millimeter the total loss of bond in adhesion or cohesion among the three specimen
2) After each 4 week weathering cycle, extend the sealant to the prescribed extension (25%) and make other observations such as cracks, crazing, chalking, discoloration or other.
3) Record other information such as indicated in the Report section of the standard test method.