THE DURABILITY OF PENETRANT-CLASS SEALER-COATED AIR-ENTRAINED CONCRETE SUBJECTED TO FREEZE/THAW

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RHODE ISLAND DEPARTMENT OF TRANSPORTATION

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EXECUTIVE SUMMARY

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ABSTRACT

Previous tests conducted at the Rhode Island Department of Transportation (RIDOT) comparing the effectiveness of various coating treatments for concrete had indicated a possible detrimental effect of penetrant sealers on concrete freeze/thaw durability. A test was specifically designed to study the effects of penetrant sealers on the freeze/thaw (F/T) durability of air-entrained concrete. Three sealer-coated specimen groups were tested in the study and gave performance comparable to that of the uncoated controls, with only a slight relative change in the resilient modulus as a result of exposure to freeze/thaw. The specimens that were coated when completely dried seemed to suffer a slightly greater loss of paste, which would reduce the effectiveness of the chloride intrusion protection. An attempt was also made to develop a procedure to test for depth of penetration of the sealers. With the equipment used, it was not possible to determine the presence of the sealer in the concrete samples in order to determine penetration depth; this may have been due to a limited presence of the sealer components.

INTRODUCTION

The Rhode Island Department of Transportation has long considered ways to reduce the penetration of chloride ions into structural concrete to protect the reinforcing steel. Two previous FHWA-sponsored RIDOT research studies[1, 2] indicated that while penetrant sealers (silanes and their variants) were very effective at mitigating the intrusion of chlorides, they appeared to have a detrimental effect on the durability of the concrete when subjected to freeze/thaw cycling (per ASTM C-666). However, typically these tests were run using non-air-entrained mixes to accelerate the test time. This was considered to be a reasonable condition, as all specimens (including the uncoated controls) were made using the same mix design.

As the sealers penetrate the pore structure of the concrete matrix, the effects of changing the pore structure through air-entrainment was thought to have a possible effect on freeze/thaw durability. After consideration of the nature of penetrant sealers, it was decided to examine the effects of air-entrainment on the resistance to freeze/thaw deterioration of coated specimens relative to uncoated specimens. As many penetrant sealers are sensitive to the moisture content of the concrete surface, it was also decided to compare specimens coated saturated-surface dry (all the sealers tested either require or recommend a dry substrate prior to sealer) to specimens that were dried to constant weight at the time of sealer application. Finally, as the penetration depth of the sealers also would have an effect on the chloride intrusion protection provided, and possibly the degree of vapor barrier created, tests were conducted to determine the depth to which the sealers were absorbed into the concrete using Fourier Transform Infrared analysis.

To compare the effectiveness of the penetrant sealers after freeze/thaw testing, samples from the test were used in a modified version of AASHTO T-259 “Resistance of Concrete to Chloride Ion Penetration”. These results of this ponding test are intended to be used only for informational purposes and not for the sake of a *critical* evaluation of the sealers.

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[1] Fruggiero, R. L., and Fera, J. D., “Chloride Inhibitor for Concrete Barriers”, FHWA-RI-RD-83-1, February 1983

[2] Fera, J. D., “Laboratory Evaluation of Concrete Sealers For Vertical Highway Structures”, FHWA-RI-90-1, January 1991

In an attempt to determine the depth of penetration of the sealers into concrete, powdered samples taken from coated specimens at fixed depths were analyzed using a Fourier Transform Infrared (FTIR) spectrometer. The spectra developed were then compared to neat samples of the sealers to develop a method of measuring the maximum depth at which the sealers were still present.

SELECTION OF TEST SEALERS

The sealers were selected to provide different compositions to compare the effects of these differences on the freeze/thaw durability of the coated concrete specimens. The sealers were of the following types:

Sealer Number One - Alkyltrialkoxysilane: a light, clear fluid
Sealer Number Two - Alkylalkoxysilane: a milky fluid
Sealer Number Three - Isobutyltrialkoxysilane: a clear fluid

SAMPLE PREPARATION

The concrete mix design [Class XX (AE)] was selected that was typical of the bridge structure mixes used by the State of Rhode Island in highway construction projects. The specimen molds used were 7.5 × 10 × 40 cm, to accommodate the freeze/thaw machine (see Appendix A for details on the machine). The specimens were made in three batches for the first run and two batches for the second run (Appendix B gives the mix design and batch test results). The specimen numbers given indicate the batch number (first number) and the number within the batch (second number), e.g. 3-4 would be specimen number four from the third batch. The specimens were all cured in a hydrated lime water bath for fourteen days[3]. They were then lightly sandblasted to remove any surface contamination.

- For the first freeze/thaw test run, the specimens were dried to a constant weight and then immersed in a bath of tap water for one week to achieve saturation. After being removed, the specimens were allowed to attain a surface dry condition and were immediately coated with the sealers. The specimens were placed in a vertical orientation (40 cm high) for sealer application. Four specimens were selected for each group. The sides and top of the specimens were coated per manufacturer's recommendations first and as soon as the specimens had dried, the bottoms were coated. Sealer number one was readily absorbed

[3] Per ASTM C 192, "Laboratory Practice for Fabrication of Concrete Test Specimens", as referenced in ASTM C 666.

into the concrete and one application was sufficient to meet the manufacturer's recommended coverage rate. The other two sealers required two coats and still did not meet the recommended coverage rate[4]. It was decided not to apply a third coat, as this would be unreasonable in a field application; also, the manufacturers had noted that coverage rates might be lower for a vertical application.

- For the second freeze/thaw test run, the specimens were dried to a constant weight, lightly sandblasted and coated with the sealers. Application of the sealer was as above.

- For the chloride intrusion test, two specimens from each sample group from the first freeze/thaw run were placed side by side with the troweled face facing down, such that the eight specimens formed a slab. The joints at the top between the specimens were sealed with a silicone sealant and plastic strips were secured in place around the perimeter of the slab with the silicone, forming a dike. The solution was a three percent sodium chloride solution, maintained at a depth of about 1.25 cm.

- To test the penetration of the sealers, 2.5 centimeter thick disks cut from the top of concrete cylinders (10 × 20 centimeter) were coated. The sealer was applied only to the tops of the disks and in as many coats as was applied to the prism specimens. An additional disk for each sealer was ponded with approximately one-half centimeter of the sealer for approximately one day.

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[4]The coverage rate was based on the sides of the specimens; the tops and bottoms were excluded because they were oriented horizontally when the coating was applied. The amount used was measured by filling a beaker with a set volume of the sealer and noting the amount remaining after application and estimating the difference. The rate was calculated by taking the amount of materials used and dividing by the total area of the sides.

TEST PROCEDURE

Freeze/Thaw

The specimens were placed in the freeze/thaw machine, which was designed to function in accordance with ASTM C666[5]. After each series of cycles, inspections were performed. The specimens were then replaced into the freeze/thaw machine according to a randomization chart, to ensure that each specimen was subjected to the same conditions. At 300 cycles, the specimens were removed from the machine and the test run was concluded.

The specimens were visually inspected, weighed and tested for resonant frequency (per ASTM C215 - transverse method)[6]. This was done prior to placement in the freeze/thaw machine to establish a baseline and after each series of cycles. Note that for the second run, another E-Meter, was used to measure the resonant frequency[7]. However, the specimens from the first run were re-tested with the new equipment and the results were compared to those obtained with the original equipment for the final inspection and were found to be nearly equivalent. The effect on the results between the first and second runs are therefore considered negligible.

For the resilent modulus (based on the resonant frequency), which was the main criterion for judging the conditions of the concrete, the specimens were oriented with the 40 × 10 cm faces parallel to the ground. All significant[8] peaks were recorded, including those due to harmonics. This allowed comparisons of the peaks between cycles so that any patterns could be detected. It also allowed a check in the event that peaks were in relatively close proximity. Subsequent inspections would then establish which peak occurred at the resonant frequency. The values would remain relatively close throughout the inspections.

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5 The control system of the machine was modified in-house with a computerized automation system. An excerpt of the manual written for the machine, with the modifications, is provided in Appendix A

6 The placement of the transducer was modified in the current version of C215. However, use of the older method of placement was stated to be acceptable by Mr. Karim Naser, chairperson of ASTM Committee C-8, the governing committee for the test.

7 The input signal transducer was also replaced. The test platform and the output transducer remained the same throughout the project

8 A peak was considered to be significant if its amplitude was at least thirty percent of the amplitude of the largest peak.

When performing visual inspections, certain distress characteristics are expected when deterioration begins. These are, in order from least severe to most: light paste loss, heavy paste loss, coarse aggregate exposed, light cracking of the surface mortar, spalling, severe cracking of the concrete and fracture of the specimens. These characteristics, if present, were recorded, with specific comments as necessary. This portion of the inspection is somewhat subjective and is only included to describe the condition of the surface, which has a significant impact on the effectiveness of the protection provided by the sealer.

Weighing consisted of recording the weight in grams of each specimen, surface dry, and tracking the percentage weight change relative to the initial weight.

Sealer Penetration

To measure the penetration of the sealers, a masonry bit was used to remove material to several controlled depths[9] and the powder was analyzed using an FTIR spectrometer (see "Sealer Penetration Determination" for the procedure). The spectra for the sealer was matched against the spectra for the removed material and a comparison was made to determine the presence of the sealers at a given depth.

Chloride Intrusion of Specimens Exposed to Freeze/Thaw

As mentioned in the introduction, an informational chloride intrusion test was performed. A three percent sodium chloride ponding solution was poured onto the slab created from the specimens from the first freeze/thaw run, to a depth of approximately one and one-quarter centimeters.

During the second freeze/thaw run, a relay controlling the heating system failed at 175 cycles. For four cycles, the specimens thawed by transfer of heat from the environment surrounding the machine. Thawing for these cycles took sixteen hours. As ASTM C666 does not place a maximum limit on the length of the thawing period and only requires that it not be less than 25%

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[9] The depths used were 1.5, 3, 4.5 and 6 mm.

of the freezing phase, no corrective action was taken, other than repairing the relay. All cycling that took place afterwards proceeded normally.

TEST RESULTS

Specimen	Sealer	Freq. @ 0 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 36 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 72 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 100 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 136 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 172 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 200 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 236 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 272 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 300 Cycles, Hz / Rel. Dynamic Modulus, %
3-1	Control	2163	2138	2127	2130	2128	2116	2127	2127	2114	2124
		-	97.7	96.7	97.0	96.8	95.7	96.7	96.7	95.5	96.4
3-2		2128	2096	2106	2098	2103	2094	2099	2104	2102	2107
		-	97.0	97.9	97.2	97.7	96.8	97.3	97.8	97.6	98.0
3-3		2137	2093	2099	2097	2100	2096	2103	2108	2117	2111
		-	95.9	96.5	96.3	96.6	96.2	96.8	97.3	98.1	97.6
3-4		2141	2104	2105	2100	2105	2098	2117	2111	2107	2108
		-	96.6	96.7	96.2	96.7	96.0	97.8	97.2	96.8	96.9
1-3	1	2125	2094	2087	2089	2083	2079	2082	2084	2077	2083
		-	97.1	96.5	96.6	96.1	95.7	96.0	96.2	95.5	96.1
1-4		2147	2110	2111	2100	2099	2099	2105	2101	2094	2107
		-	96.6	96.7	95.7	95.6	95.6	96.1	95.8	95.1	96.3
2-5		2105	2073	2069	2063	2061	2050	2063	2071	2062	2069
		-	97.0	96.6	96.0	95.9	94.8	96.0	96.8	96.0	96.6
2-6		2096	2070	2063	2059	2059	2053	2060	2065	2056	2063
		-	97.5	96.9	96.5	96.5	95.9	96.6	97.1	96.2	96.9
1-5	2	2117	2100	2094	2089	2084	2076	2081	2074	2072	2069
		-	98.4	97.8	97.4	96.9	96.2	96.6	96.0	95.8	95.5
1-6		2130	2108	2107	2094	2095	2087	2080	2085	2083	2074
		-	97.9	97.9	96.6	96.7	96.0	95.4	95.8	95.6	94.8
2-1		2122	2100	2097	2088	2089	2080	2081	2080	2063	2074
		-	97.9	97.7	96.8	96.9	96.1	96.2	96.1	94.5	95.5
2-3		2086	2070	2066	2056	2052	2043	2047	2046	2034	2028
		-	98.5	98.1	97.1	96.8	95.9	96.3	96.2	95.1	94.5
1-1	3	2129	2110	2105	2092	2091	2074	2087	2089	2085	2086
		-	98.2	97.8	96.6	96.5	94.9	96.1	96.3	95.9	96.0

Table 1 - Resonant Frequency Test Results, Run One: Specimens Coated Saturated Surface Dry

Specimen	Sealer	Freq. @ 0 Cycles, Hz	Freq. @ 36 Cycles, Hz	Freq. @ 72 Cycles, Hz	Freq. @ 100 Cycles, Hz	Freq. @ 136 Cycles, Hz	Freq. @ 172 Cycles, Hz	Freq. @ 200 Cycles, Hz	Freq. @ 236 Cycles, Hz	Freq. @ 272 Cycles, Hz	Freq. @ 300 Cycles, Hz
		Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %
1-2	3	2114	2097	2092	2083	2080	2070	2069	2075	2065	2072
		-	98.4	97.9	97.1	96.8	95.9	95.8	96.3	95.4	96.1
2-2		2103	2083	2078	2067	2066	2054	2060	2057	2053	2059
		-	98.1	97.6	96.6	96.5	95.4	96.0	95.7	95.3	95.9
2-4		2131	2107	2100	2090	2090	2084	2089	2085	2085	2074
		-	97.8	97.1	96.2	96.2	95.6	96.1	95.7	95.7	94.7

Table 1 - Resonant Frequency Test Results, Run One: Specimens Coated Saturated Surface Dry (continued)

Note: The dynamic modulus is calculated by the formula $E=C_m \rho n^2$,

where:

ρ=mass of the specimen, kg

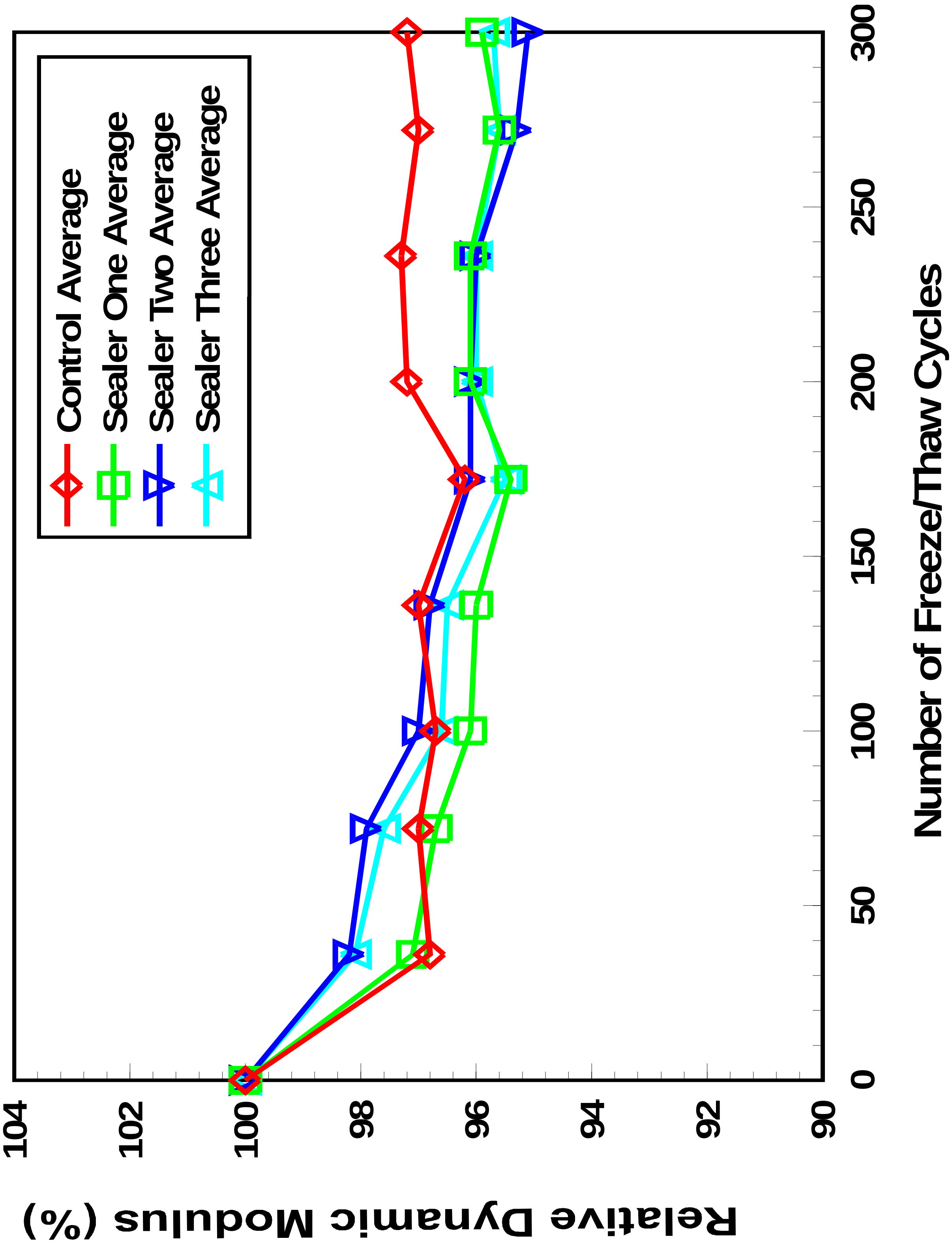

Figure 1 - Relative Dynamic Modulus, Run One Test Group Averages

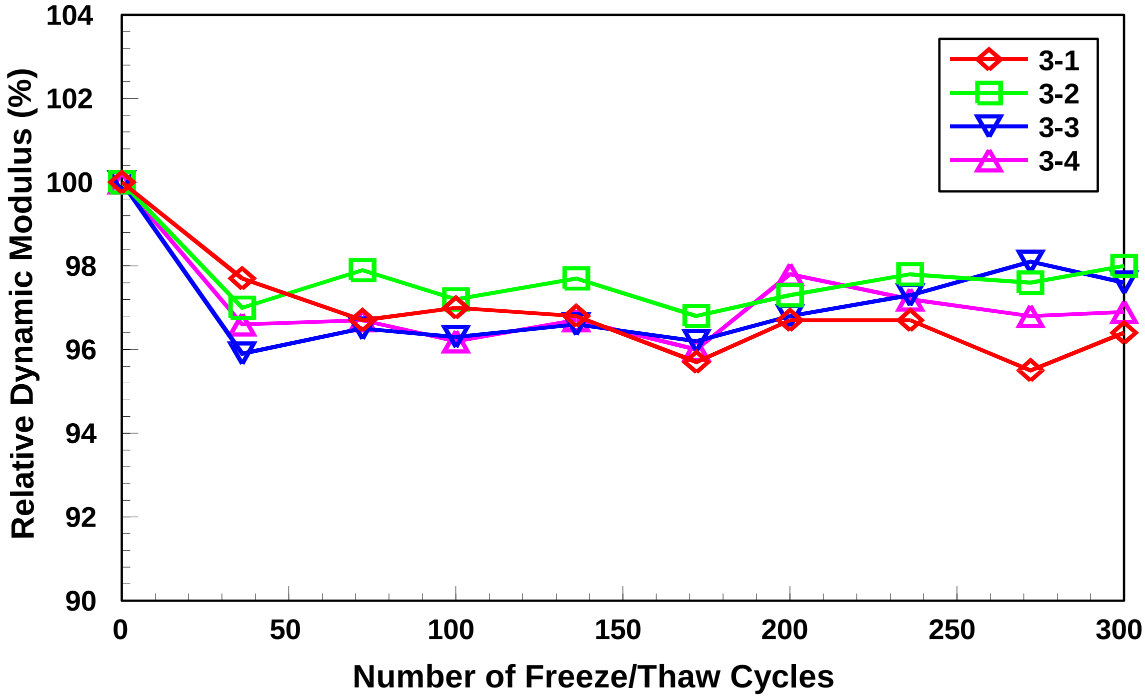

Figure 2 - Relative Dynamic Modulus, Run One Control Group

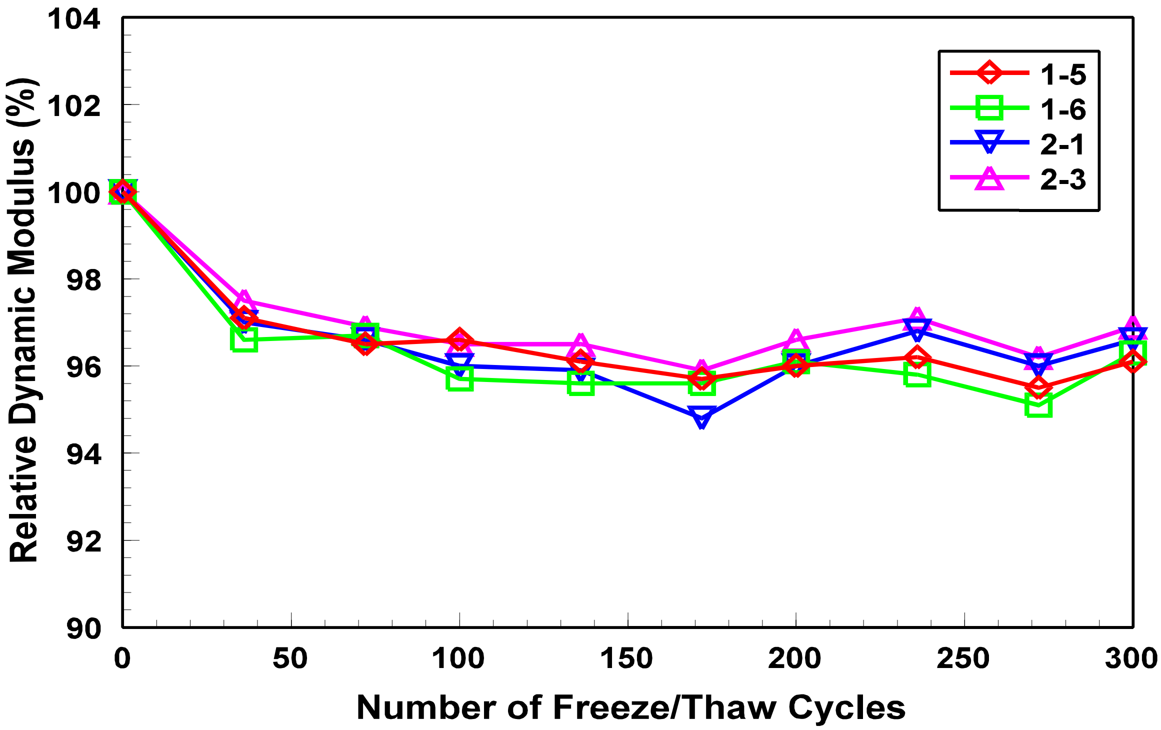

Figure 3 - Relative Dynamic Modulus, Run One Sealer Number One Test Group

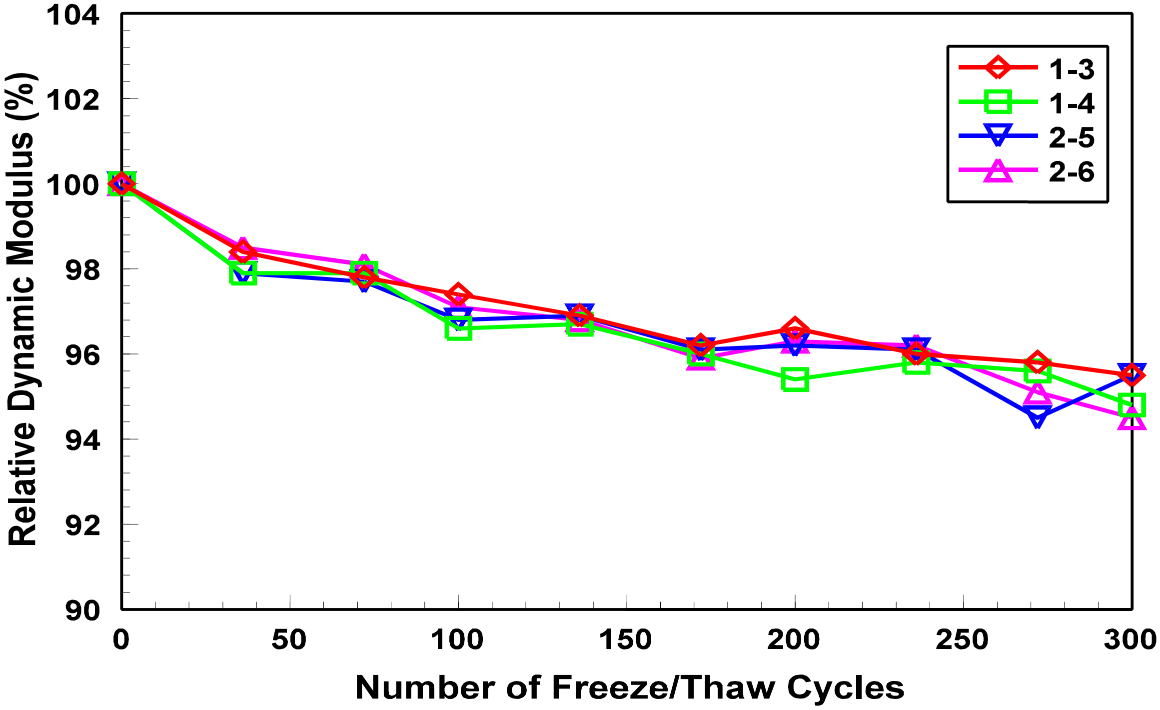

Figure 4 - Relative Dynamic Modulus, Run One Sealer Number Two Test Group

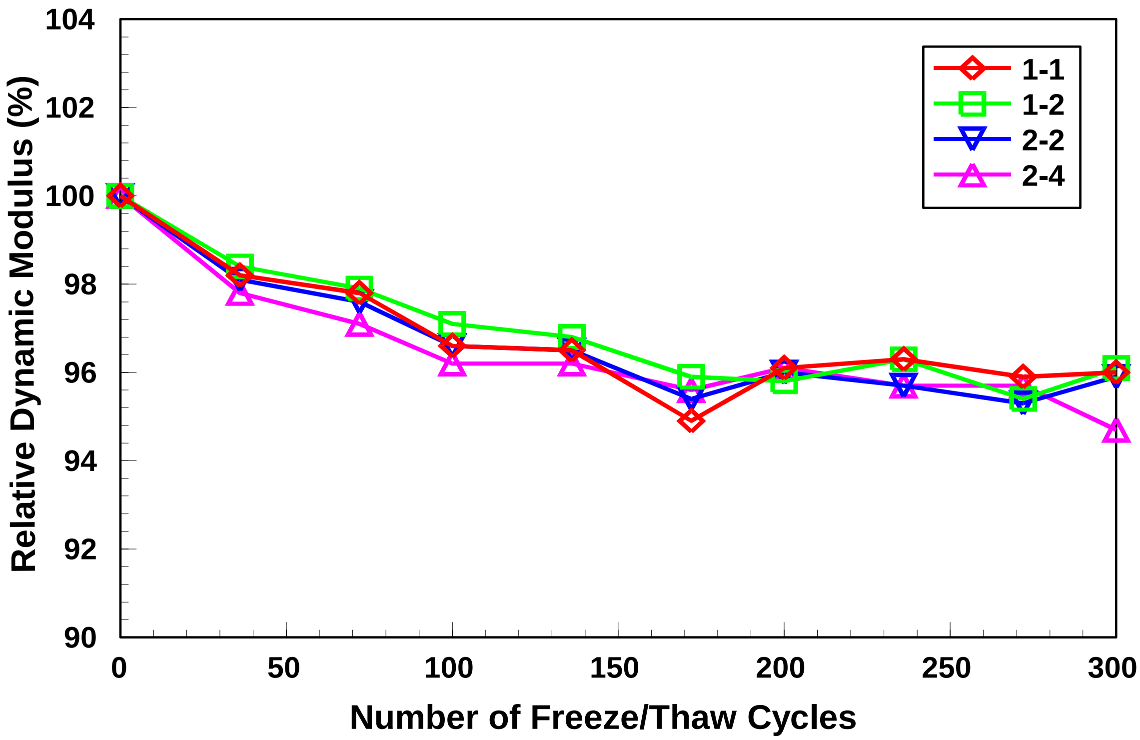

Figure 5 - Relative Dynamic Modulus, Run One Sealer Number Three Test Group

Specimen	Sealer	Weight @ 0 Cycles (g)	% Change @ 36 Cycles	% Change @ 72 Cycles	% Change @ 100 Cycles	% Change @ 136 Cycles	% Change @ 172 Cycles	% Change @ 200 Cycles	% Change @ 236 Cycles	% Change @ 272 Cycles	% Change @ 300 Cycles
3-1	Control	7605	0.93	1.09	1.09	1.12	1.17	1.22	1.18	1.18	1.25
3-2		7448	0.91	1.13	1.13	1.3	1.3	1.34	1.33	1.29	1.33
3-3		7517	0.92	1.13	1.16	1.3	1.26	1.33	1.3	1.38	1.45
3-4		7475	0.91	1.14	1.2	1.23	1.24	1.34	1.34	1.36	1.34
1-3	1	7512	0.32	0.76	0.87	0.99	1.14	1.21	1.26	1.24	1.24
1-4		7521	0.17	0.56	0.45	0.89	0.89	0.96	0.98	1	1.05
2-5		7395	0.16	0.55	0.68	0.88	0.99	1.11	1.15	1.18	1.28
2-6		7433	0.23	0.63	0.74	0.93	1.02	1.14	1.1	1.26	1.25
1-5	2	7562	0.07	0.17	0.24	0.34	0.42	0.5	0.57	0.75	0.85
1-6		7479	0	0.15	0.19	0.21	0.27	0.37	0.52	0.68	0.78
2-1		7448	0	0.17	0.13	0.7	0.28	0.36	0.44	0.63	0.68
2-3		7400	0.04	0.15	0.14	0.23	0.3	0.36	0.43	0.55	0.62
1-1	3	7448	0.3	0.7	0.75	0.89	0.97	0.97	0.98	0.95	0.94
1-2		7410	0.19	0.54	0.62	0.81	0.85	0.86	1.01	1.01	1.15
2-2		7424	0.31	0.61	0.67	0.77	0.84	0.96	1.02	1.14	1.12
2-4		7469	0.11	0.4	0.52	0.68	0.83	0.94	1.07	1.2	1.2

Table 2 - Specimen Weight Gain, Run One: Specimens Coated Saturated Surface Dry

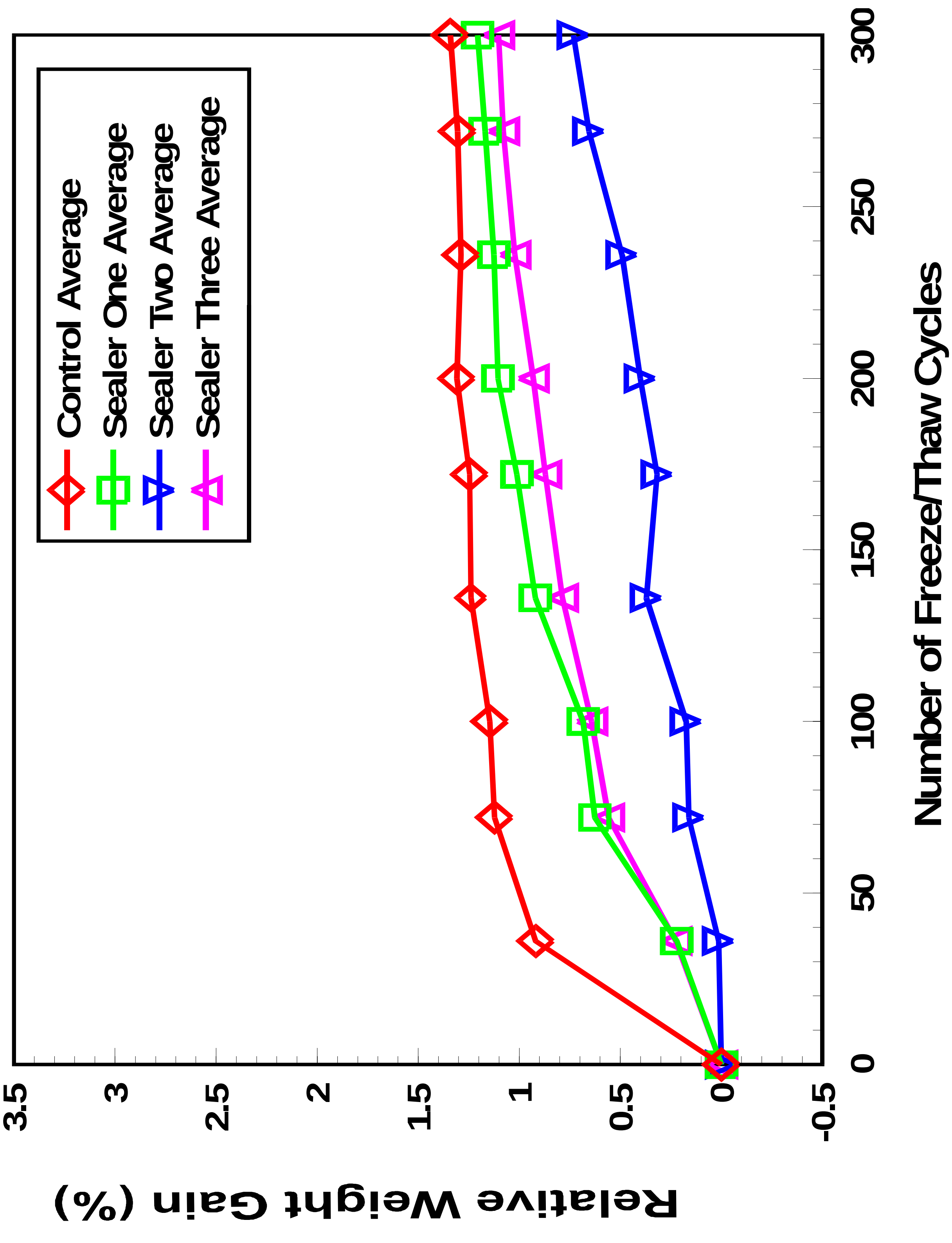

Figure 6 - Weight Gain, Run One Test Group Averages

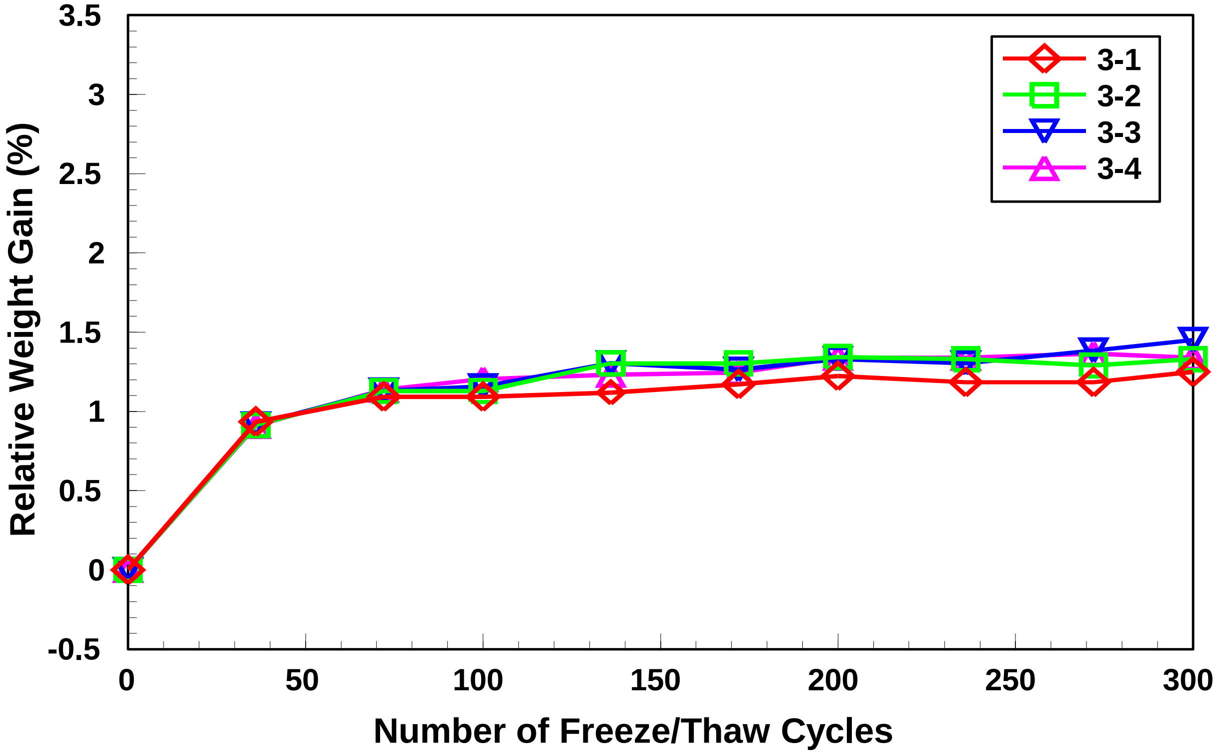

Figure 7 - Weight Gain, Run One Control Group

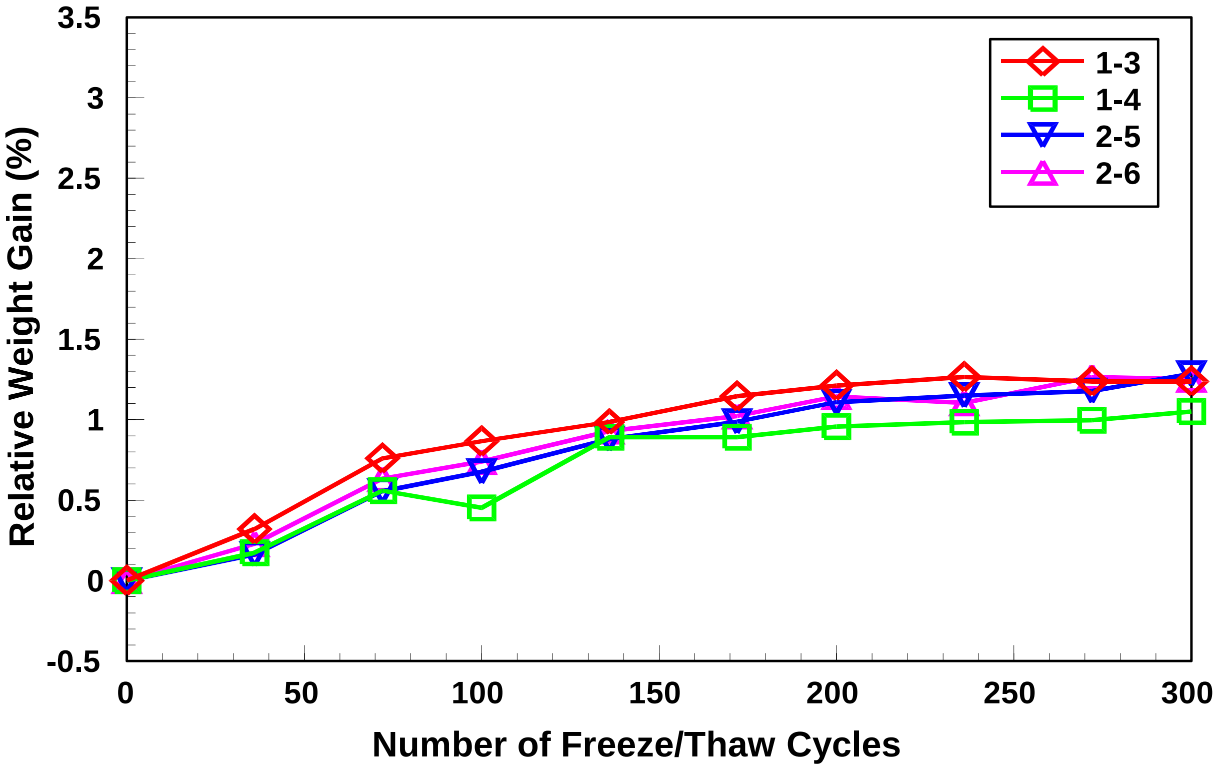

Figure 8 - Weight Gain, Run One Sealer Number One Test Group

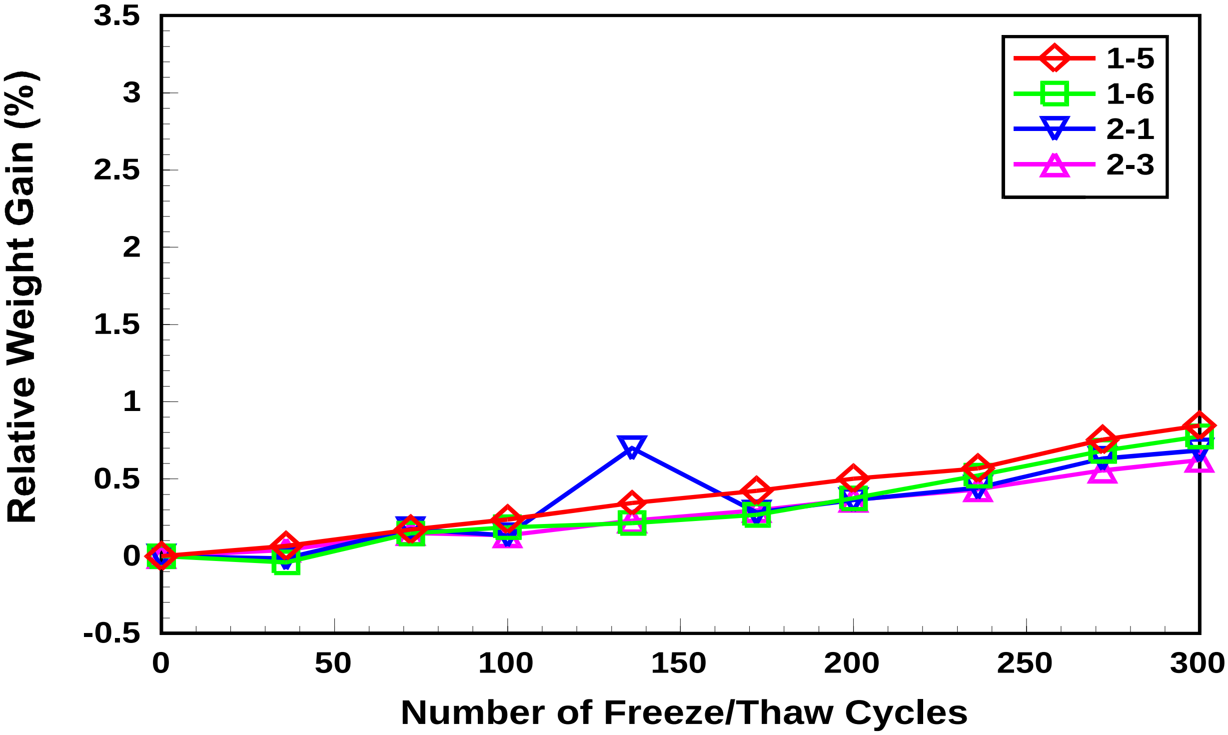

Figure 9 - Weight Gain, Run One Sealer Number Two Test Group

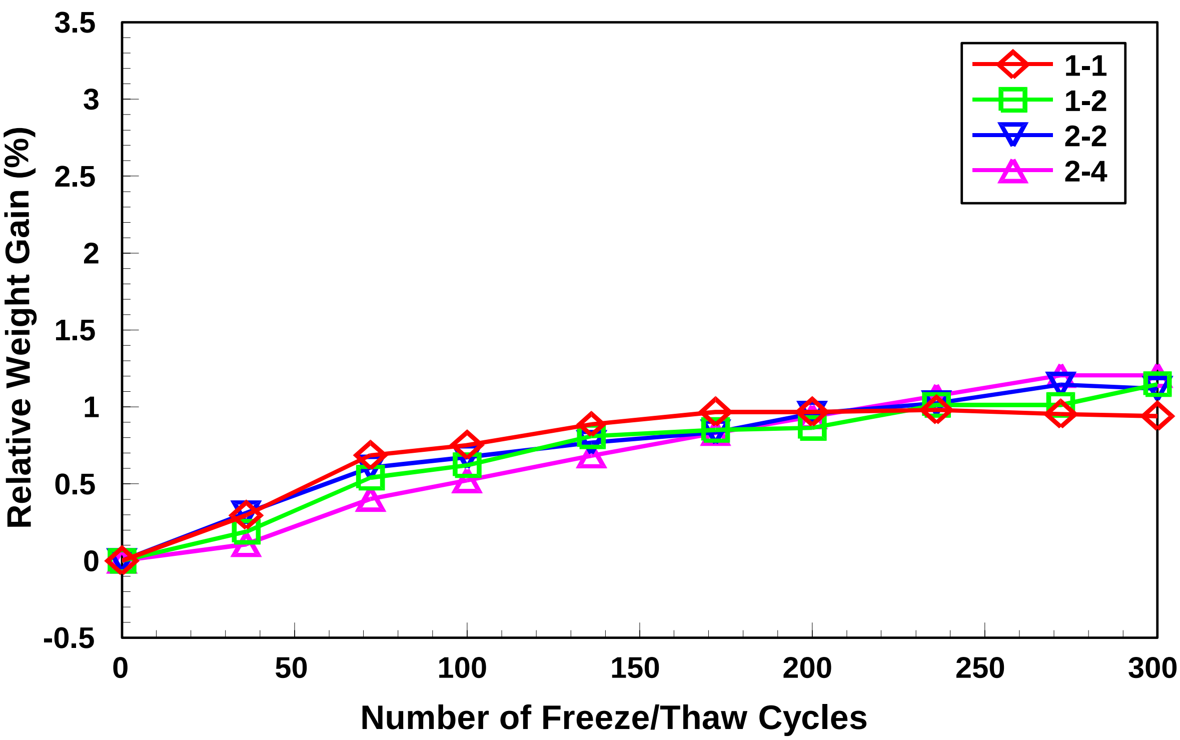

Figure 10 - Weight Gain, Run One Sealer Number Three Test Group

Specimen	Sealer	Freq. @ 0 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 36 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 72 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 108 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 144 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 179 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 215 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 251 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 269 Cycles, Hz / Rel. Dynamic Modulus, %	Freq. @ 300 Cycles, Hz / Rel. Dynamic Modulus, %
1-5	-	2040	1997	2020	1981	2023	2038	2051	2046	2056	2041
		-	95.8	98.0	94.3	98.3	99.8	101.1	100.6	101.6	100.1
1-6		2089	2023	2079	2027	2030	2091	2098	2108	2107	2096
		-	93.8	99.0	94.2	94.4	100.2	100.9	101.8	101.7	100.7
2-3		2051	2003	2049	2004	2034	2055	2058	2067	2072	2055
		-	95.4	99.8	95.5	98.3	100.4	100.7	101.6	102.1	100.4
2-6		2091	2035	2064	2055	2045	2063	2066	2077	2089	2072
		-	94.7	97.4	96.6	95.6	97.3	97.6	98.7	99.8	98.2
1-1	1	2080	2055	2058	2044	2025	2035	2026	2032	2036	2031
		-	97.6	97.9	96.6	94.8	95.7	94.9	95.4	95.8	95.3
1-8		2112	2076	2080	2064	2053	2055	2053	2061	2057	2048
		-	96.6	97.0	95.5	94.5	94.7	94.5	95.2	94.9	94.0
2-1		2103	2082	2086	2077	2061	2059	2055	2059	2057	2062
		-	98.0	98.4	97.5	96.0	95.9	95.5	95.9	95.7	96.1
2-8		2092	2068	2073	2160	2034	2045	2034	2041	2045	2035
		-	97.7	98.2	106.6	94.5	95.6	94.5	95.2	95.6	94.6
1-2	2	2094	2013	2064	2055	2044	2047	2034	2042	2042	2030
		-	92.4	97.2	96.3	95.3	95.6	94.4	95.1	95.1	94.0
1-7		2104	2078	2084	2069	2042	2046	2022	2011	2027	2001
		-	97.5	98.1	96.7	94.2	94.6	92.4	91.4	92.8	90.4
2-2		2044	2009	2015	1997	1995	1637	1957	1970	1963	1965
		-	96.6	97.2	95.5	95.3	64.1	91.7	92.9	92.2	92.4
2-5		2086	2057	2059	2042	2000	1996	1989	1994	2002	1980
		-	97.2	97.4	95.8	91.9	91.6	90.9	91.4	92.1	90.1
1-3	3	2080	2044	2065	2055	2041	2025	2018	2013	2017	1996
		-	96.6	98.6	97.6	96.3	94.8	94.1	93.7	94.0	92.1

Table 3 - Resonant Frequency Test Results, Run Two: Specimens Coated Dry

Specimen	Sealer	Freq. @ 0 Cycles, Hz	Freq. @ 36 Cycles, Hz	Freq. @ 72 Cycles, Hz	Freq. @ 108 Cycles, Hz	Freq. @ 144 Cycles, Hz	Freq. @ 179 Cycles, Hz	Freq. @ 215 Cycles, Hz	Freq. @ 251 Cycles, Hz	Freq. @ 269 Cycles, Hz	Freq. @ 300 Cycles, Hz
		Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %	Rel. Dynamic Modulus, %
1-4	3	2080	2048	2054	2041	2035	2035	2004	2017	2016	1982
		-	96.9	97.5	96.3	95.7	95.7	92.8	94.0	93.9	90.8
2-4		2060	2030	2034	2018	1997	1995	1974	1974	1980	1965
		-	97.1	97.5	96.0	94.0	93.8	91.8	91.8	92.4	91.0
2-7		2108	2071	2073	2061	2049	2048	2028	2023	2129	2013
		-	96.5	96.7	95.6	94.5	94.4	92.6	92.1	102.0	91.2

Table 3 - Resonant Frequency Test Results, Run Two: Specimens Coated Dry (continued)

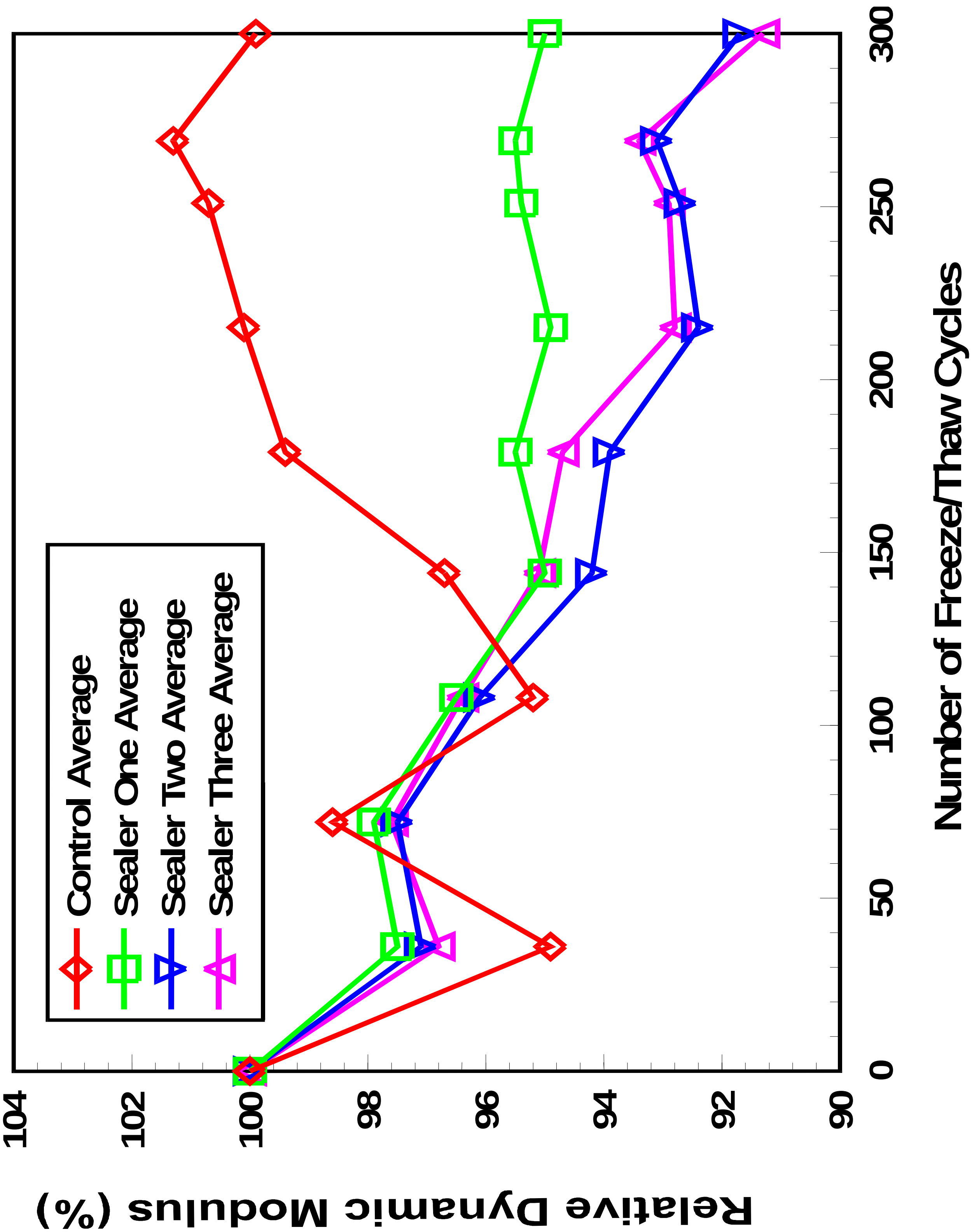

Figure 11 - Relative Dynamic Modulus, Run Two Test Group Averages

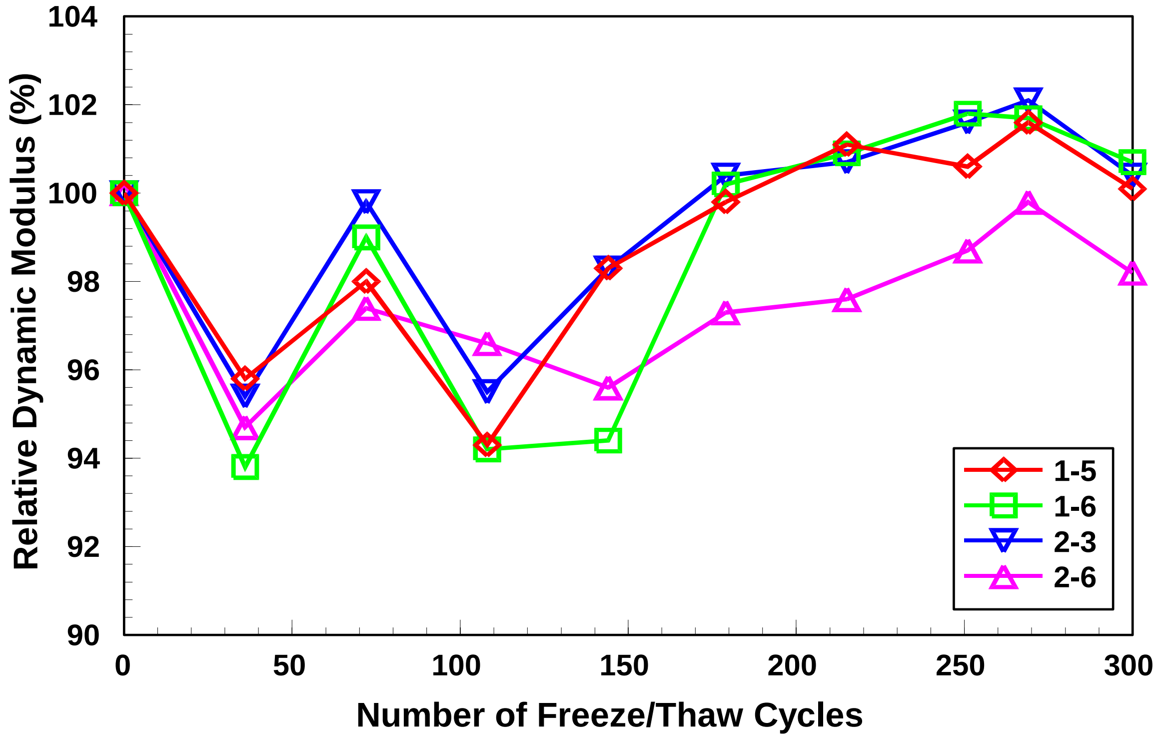

Figure 12 - Relative Dynamic Modulus, Run Two Control Group

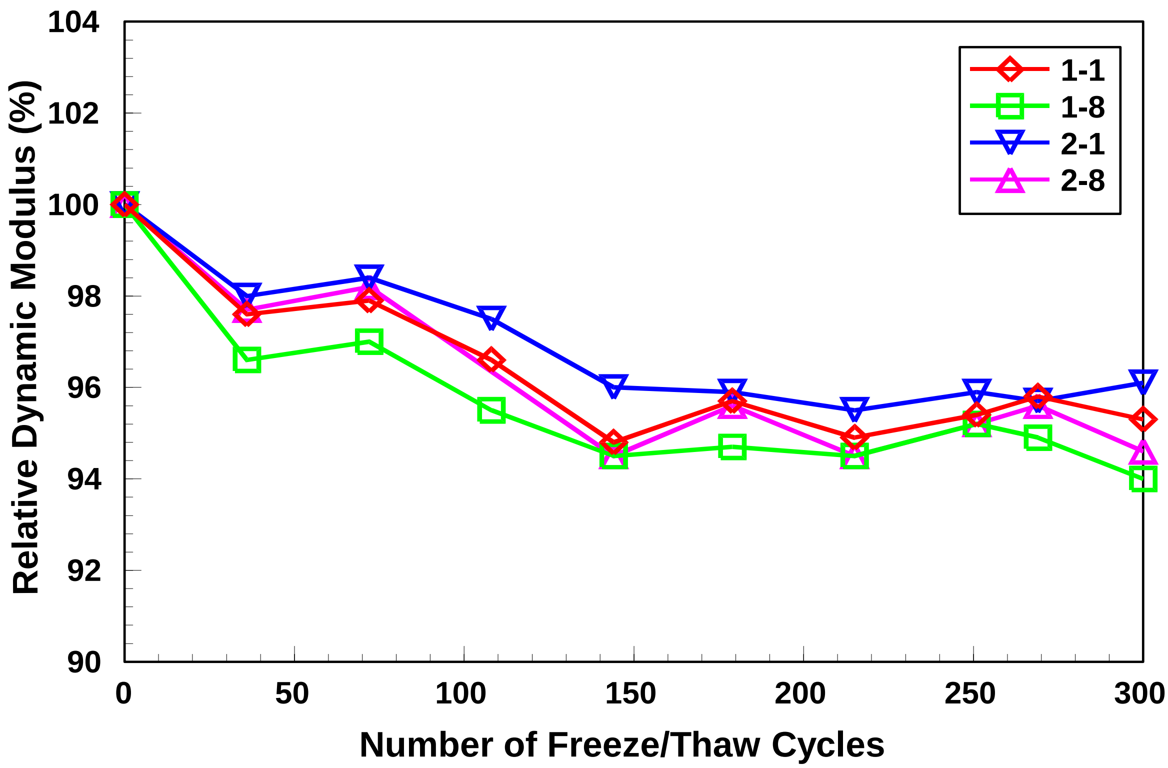

Figure 13 - Relative Dynamic Modulus, Run Two Sealer Number One Test Group

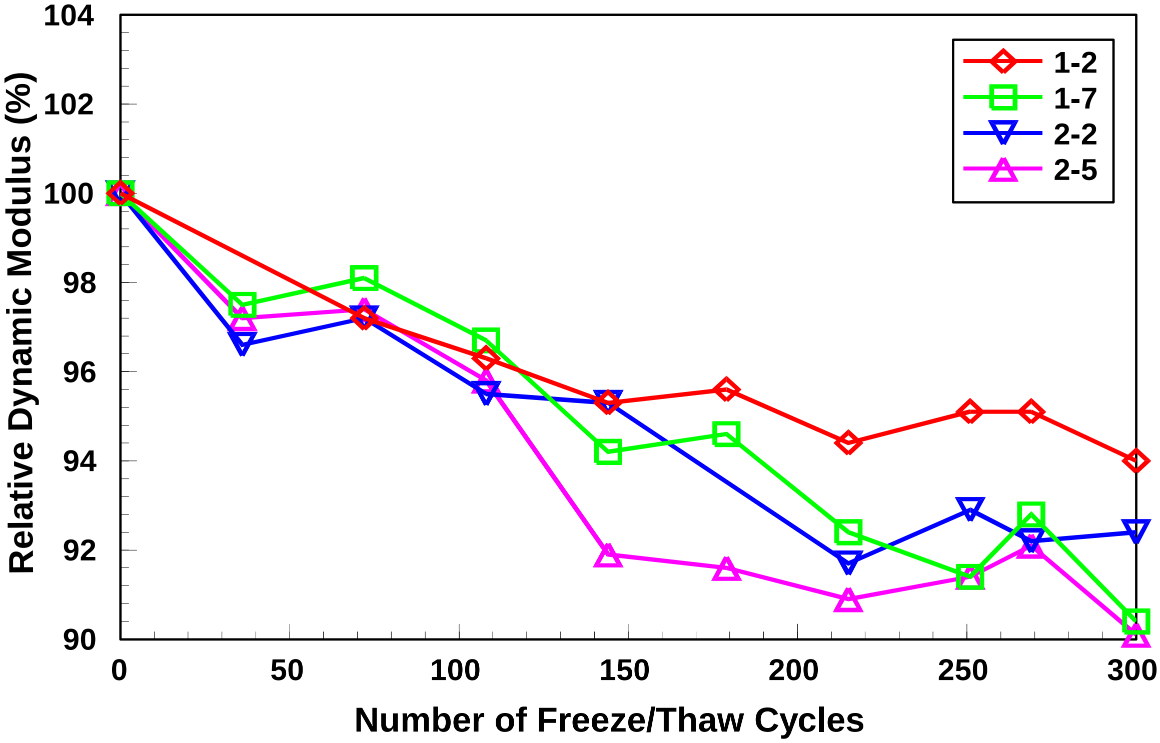

Figure 14 - Relative Dynamic Modulus, Run Two Sealer Number Two Test Group

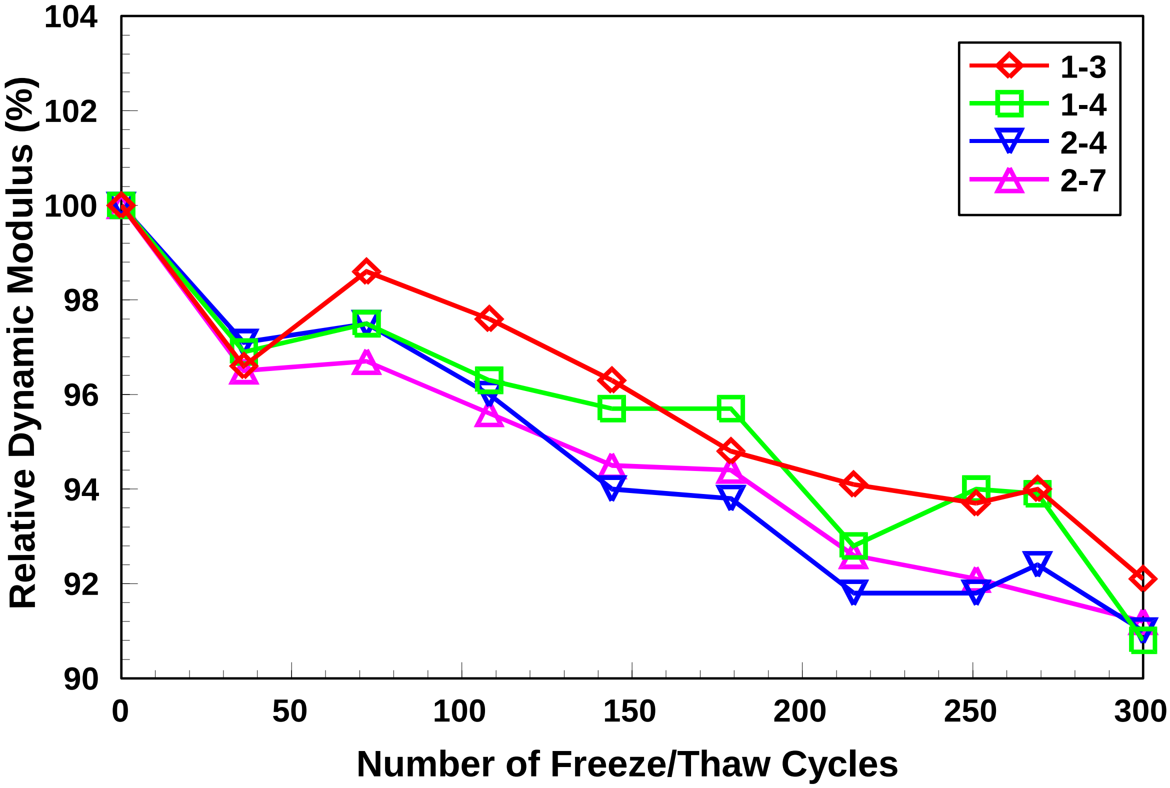

Figure 15 - Relative Dynamic Modulus, Run Two Sealer Number Three Test Group

Specimen	Sealer	Weight @ 0 Cycles (g)	% Change @ 36 Cycles	% Change @ 72 Cycles	% Change @ 108 Cycles	% Change @ 144 Cycles	% Change @ 179 Cycles	% Change @ 215 Cycles	% Change @ 251 Cycles	% Change @ 269 Cycles	% Change @ 300 Cycles	% Change After 300 Cycles (Dried) [1]
1-5	-	7264	2.74	2.92	2.96	2.90	2.96	2.93	2.97	2.92	2.93	-0.73
1-6		7419	2.36	2.66	2.68	2.72	2.71	2.72	2.86	2.72	2.72	-0.13
2-3		7337	2.81	3.05	3.08	3.08	3.18	3.16	3.24	3.26	3.22	-0.75
2-6		7399	2.37	2.58	2.61	2.59	2.64	2.69	2.69	2.66	2.65	-0.26
1-1	1	7361	-0.14	0.62	0.76	1.22	1.51	2.01	2.13	2.16	2.20	-0.05
1-8		7500	0.44	0.76	1.00	1.52	1.80	2.28	2.33	2.31	2.28	-0.27
2-1		7372	0.37	0.53	0.65	0.94	1.26	1.86	2.03	2.06	2.13	-0.34
2-8		7509	0.31	0.49	0.59	1.01	1.27	1.81	1.94	1.93	2.00	-0.09
1-2	2	7351	0.29	0.35	0.33	0.44	0.45	0.54	0.52	0.50	0.46	0.45
1-7		7351	0.29	0.33	0.37	0.71	0.83	1.20	1.41	1.41	1.46	0.24
2-2		7225	0.60	0.83	0.89	1.15	1.33	1.74	1.85	1.85	1.77	-0.06
2-5		7434	0.38	0.46	0.51	1.36	1.71	2.13	2.14	2.07	2.02	0.55
1-3	3	7401	0.39	0.38	0.43	0.85	1.01	1.51	1.61	1.62	1.63	-0.27
1-4		7450	0.42	0.48	0.52	0.70	0.85	1.17	1.28	1.29	1.28	0.27
2-4		7425	0.38	0.58	0.66	0.75	0.84	1.10	1.13	1.04	0.84	0.81
2-7		7402	0.26	0.38	0.42	0.51	0.66	1.00	1.04	1.00	0.93	0.51

Note 1: These values represent the actual loss [-] or gain [+] of material from the specimens

Table 4 - Specimen Weight Gain, Run Two: Specimens Coated Dry

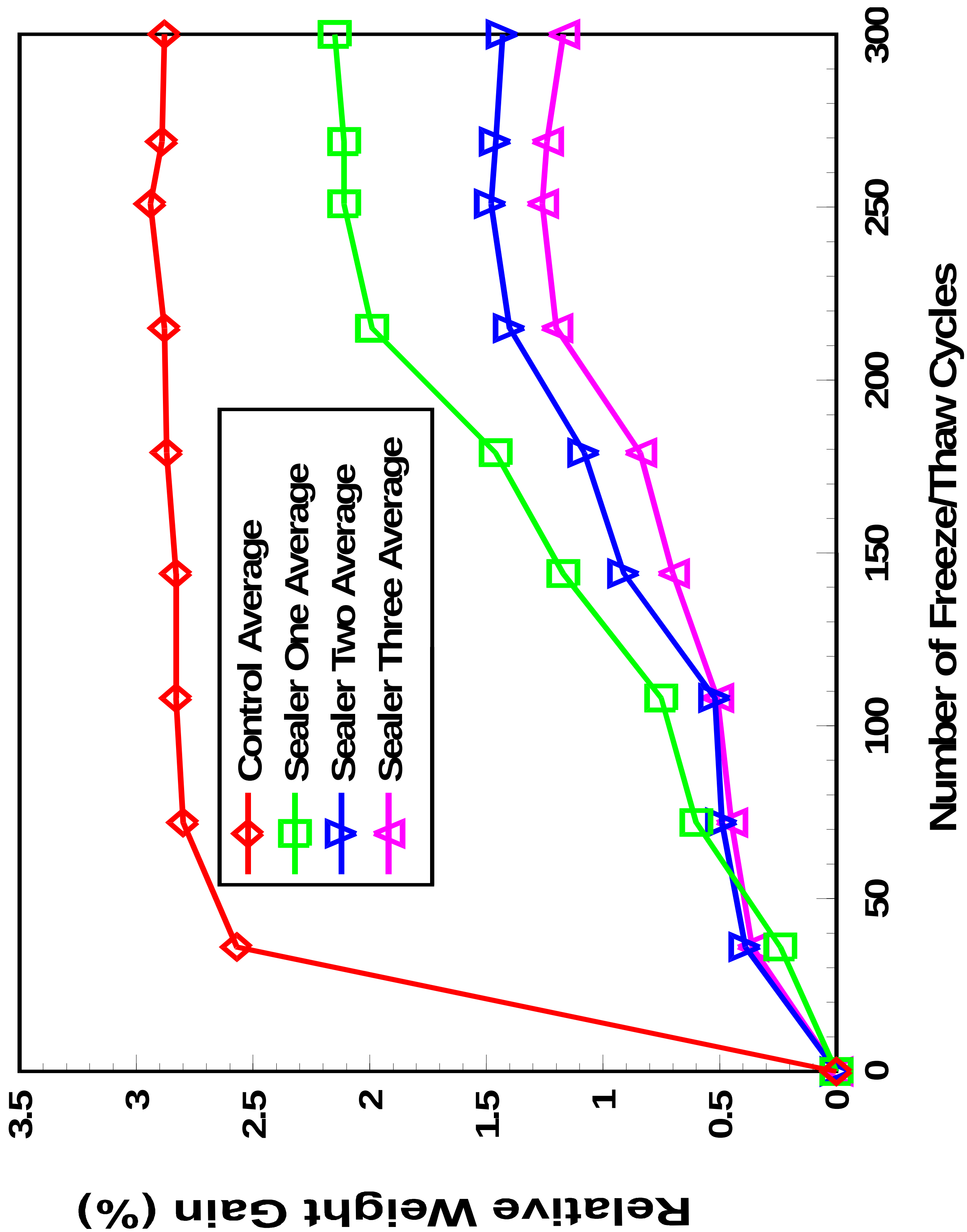

Figure 16 - Weight Gain, Run Two Test Group Averages

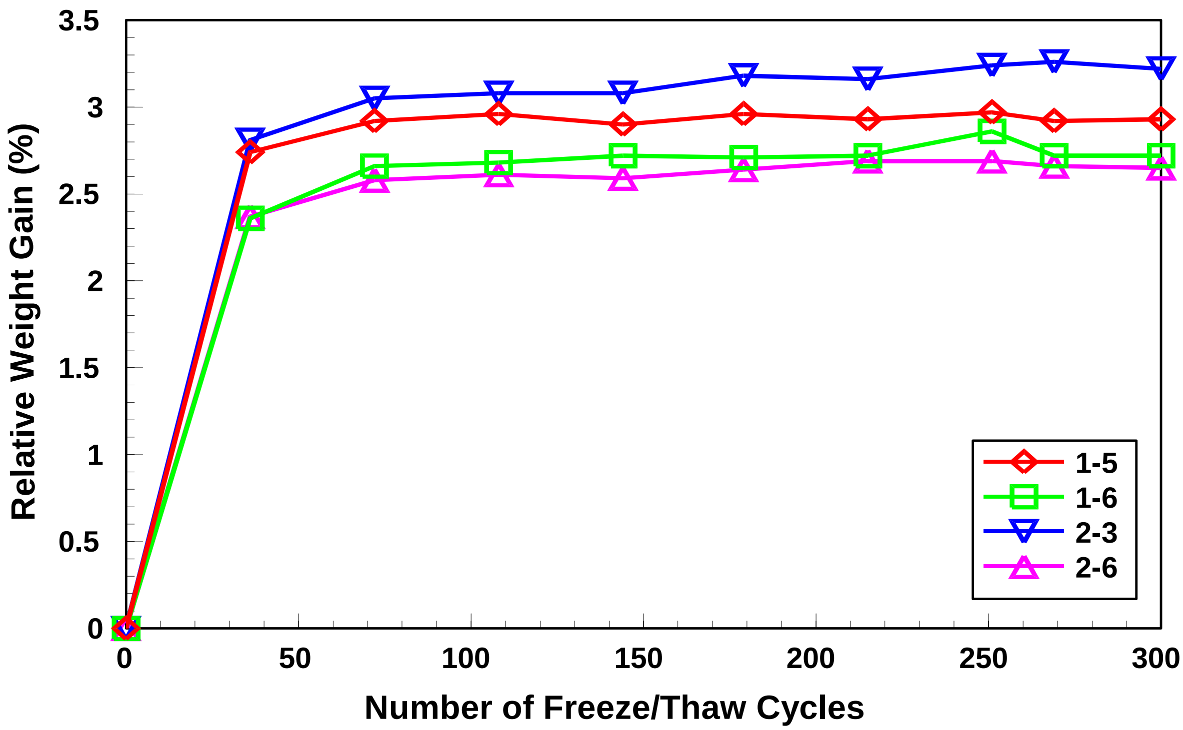

Figure 17 - Weight Gain, Run Two Control Group

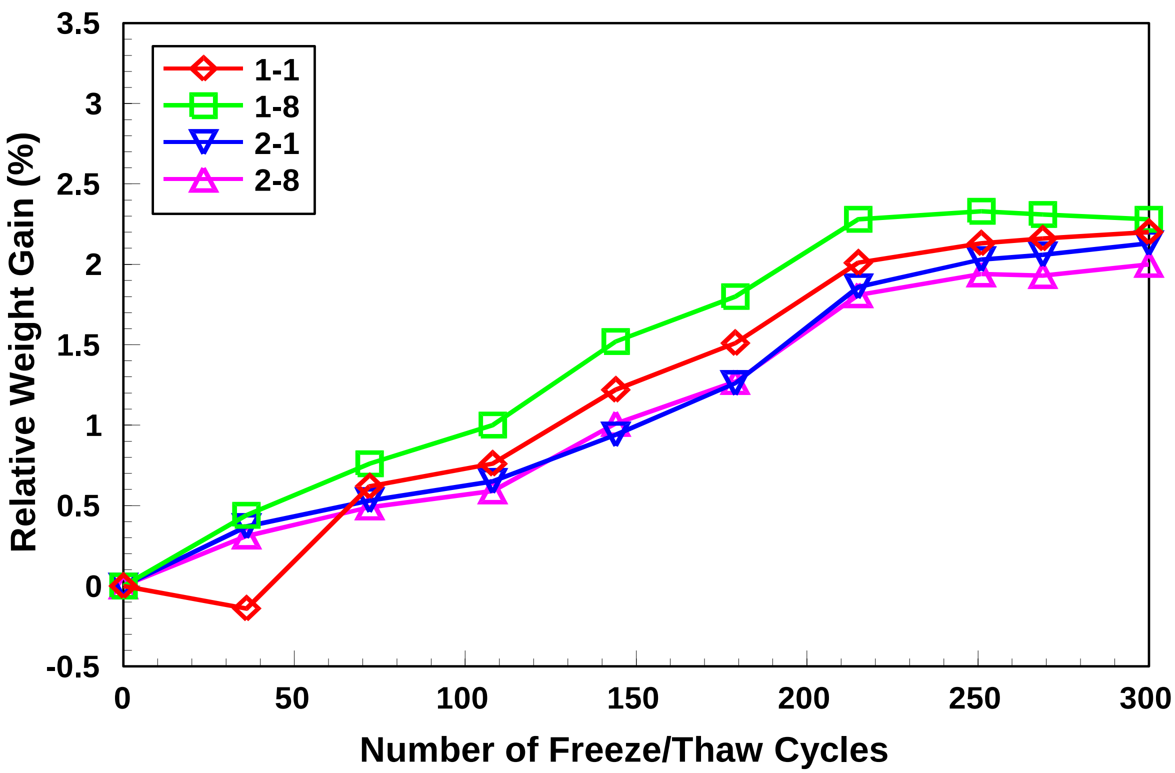

Figure 18 - Weight Gain, Run Two Sealer Number One Test Group

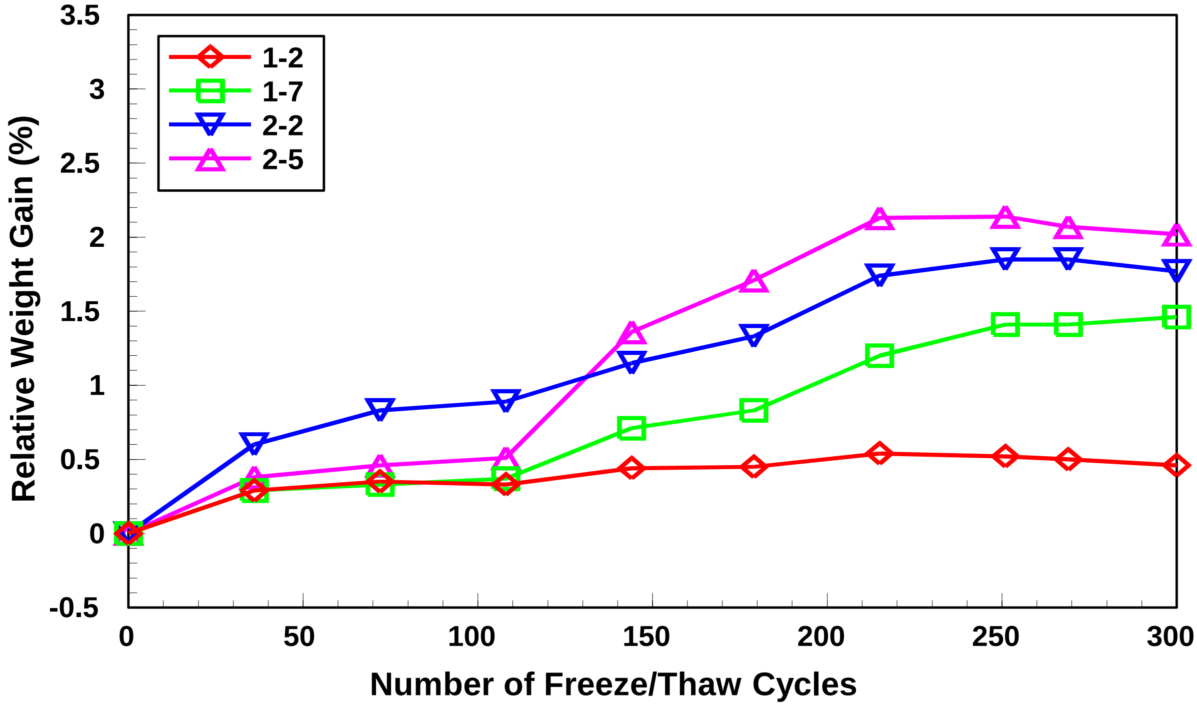

Figure 19 - Weight Gain, Run Two Sealer Number Two Test Group

Specimen Number	Sealer Number	Drilling Depth (cm)	kg Cl^-/m³ Concrete	Average of kg Cl^-/m³ Concrete - Same Sealer/Same Depth
3-2	Control	1.9	2.2	3.1
3-4	Control	1.9	4.0	
1-4	1	1.9	0.7	0.7
2-6	1	1.9	0.7	
2-1	2	1.9	3.1	2.8
2-3	2	1.9	2.5	
1-1	3	1.9	4.4	4.1
1-2	3	1.9	3.8	
3-2	Control	3.8	0.4	0.6
3-4	Control	3.8	0.7	
1-4	1	3.8	0.5	0.4
2-6	1	3.8	0.3	
2-1	2	3.8	0.5	0.5
2-3	2	3.8	0.5	
1-1	3	3.8	0.8	0.7
1-2	3	3.8	0.6	

Table 5 - Informational Chloride Intrusion Test

The visual inspection showed little apparent significant deterioration on any of the specimens in either of the runs. The specimens in the second run appeared to have lost more surface paste, although the difference when compared to the first was slight.

When looking at the larger weight gains for the second run specimens, it should be kept in mind that the specimens were dry when the freeze/thaw test was started. This is readily apparent when the weight gains for the control specimens during the first 36 cycles are examined. This weight gain also masks any changes due to the loss of paste. However, the last weight taken for the specimens after the conclusion of the test and after the specimens had dried to constant weight accurately displays the material lost.

SEALER PENETRATION DETERMINATION

The three sealers were applied one each to three concrete samples (cut from cylinder tops) and allowed to cure. After curing, four powdered portions were taken at four different depths[10] from each concrete sample coated with one of the three sealers. In addition, four powdered portions were taken at the same depths from an *untreated* concrete sample. These portions were to serve as concrete blanks or original background samples over the concrete depth range. Consequently, there were a total of sixteen samples for IR analyses, that is, three coated samples at four depths, plus one uncoated sample at four depths. Plots for the FTIR analysis are provided for sealer number one, as its results are indicative of those for all three sealers, except in the case of the Section 1C, as shown below.

1. FTIR Analyses:

A) As Pellets - The four powdered depth portions for each of the three sealers were run as pellets, whereby a weighed amount of powdered sample is mixed with a weighed amount of potassium bromide salt {**KBr**}. The ratio used is 1:10, sample to KBr. The powdered mixture is compressed into a hard pellet disc and run as such in the FTIR spectrometer (see Figure 21). This is the *standard method* for analyzing solids. The uncoated concrete background samples were also run in the same fashion.

Results were that the coated and uncoated blank samples were indistinguishable, i.e., gave the same FTIR spectra. The presence of any of the three sealers was *not* found at any of the four depths for any sample. At this point, it could not be ascertained whether *no* sealers were present *or* present in such small amounts that the FTIR instrument could not detect them.

B) As Powders - Neat via Diffuse Reflectance FTIR -The next attempt was to analyze the samples as powders without any Kbr addition and without compressing into a pellet disc. This technique is used for solid or powdered sample containing small amounts of chemicals which cannot be removed or extracted. It was hoped that this method would concentrate the amounts of the sealers and their presence would be detectable.

However, results were the same as with the pellet samples. The coated samples and uncoated blanks were indistinguishable (same spectra). Even though the coated samples should have been more concentrated, none of the three sealers were found at any of the four depths (see

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10 1.5, 3.0, 4.5, and 6.0 mm; these were selected based on the manufacturers' literature, which gave 6.0 mm as the maximum depth of penetration.

Figure 22). Again, it was still unknown whether any coatings were actually in the samples *or* their amounts were too small for detection.

C) <u>Analyses of Sealers - Neat</u> - FTIR spectra for each of the three sealers was acquired to gain insight as to where the major adsorption bands should be and to be used as standards, for sample comparisons. These spectra were very clear and definitive for each sealer (see Figure 23). However it was observed that the coatings contained *very* volatile components, which resulted in spectral bands decreasing and disappearing. Spectral-time study runs were made in each sealer. These studies showed that sealers number one and three samples gave little or no spectra after 45 minutes, while number two gave substantial spectra for four hours. Number two was considerably less volatile.

D) <u>Ponded Samples -</u> More concentrated coated samples were prepared by ponding concrete samples with the respective sealers. Powdered samples were taken at each of the four depths (1.5, 3.0, 4.5 and 6.0 mm). These samples should have had sufficient amounts of sealer at least near the concrete surface (1.5 and 3.0 mm) to be detected by the FTIR technique.

Each of the three ponded sealers produced FTIR spectra at depths of 1.5, 3.0, 4.5 and 6.0 mm, indicating their presence (see Figure 24). These results looked promising in regard to tracking the sealers at *sufficient* concentrations. Another factor which enabled the sealers spectra to be isolated and distinguished was the technique of subtracting the blank concrete background spectra from the coated sample. Previously, only a *visual* comparison of the blank and the samples were made, whereby the subtle differences could be missed. Because the subtractive spectra showed some potential, it was decided to rerun the initial (powdered) samples.

E) <u>Rerun of Original Coated Samples -</u> The three coated samples and their appropriate concrete blank backgrounds were rerun via the FTIR and the subtractive spectra technique applied (see Figure 25). However, *no* distinguishable or definitive spectra were obtained for any of the sealers. Again, as with earlier FTIR results, it was not certain whether the sealers were present in such small amounts that the FTIR system could not detect them.

2. Volatile and Non-volatile Components:

Ten grams of each sealer was weighed into aluminum dishes and allowed to stand uncovered at ambient temperature (20 - 22ºC). The samples and dishes were reweighed periodically (every 1 $^{1}/_{2}$ hours) for six hours. They were then weighed after standing a total of twenty-four hours. The weight loss and weights remaining were determined and plotted against time in hours.

Sealer number one was the most volatile, with 36.6% loss during the first 1.5 hours. Sealer number two lost 8.4% and number three 1.6% during this time. After twenty-four hours, number one had lost 80.3%, number two 59.1% and number three 20.5%.

Expressed another way: After twenty-four hours, number one had 1.98 grams remaining, number two had 4.10 grams remaining and number three had 7.95 grams from the ten grams initially weighed out. All three sealers were still liquid, although all were clear and colorless by this point. Sealer number two, which was milk white initially, had only some patchy residue on the bottom of the aluminum dish. No odor was detected from what remained of any of the three sealers.

3. Comments

A) Two parts of the work support each other in regard to volativity of the three sealers. The appreciable volatility was first observed while trying to obtain FTIR spectra of the neat sealers. Later, the volatility was measured by carrying out weight loss and drydown determinations. It should be noted that in each case volatility was observed at ambient temperature, i.e. at 20 - 22ºC. No *heat* was applied as usually done in drydown determinations, e.g., 100ºC. Consequently, the volatility of sealers is high.

Volatility may be due to solvents involved, and/or to volatile *active* components. In either case, the amount of active component actually remaining in a concrete surface after a certain time could be considerably less than that in the amount of sealer initially applied.

B) It is not known whether the FTIR method failed to detect the coatings from the powdered samples at the recommended application rates because there was no sealer present, too little sealer present or whether the *remaining* residue after the volatilitization process did *not* adsorb infrared light. Regardless, the FTIR technique was unable to determine the penetration depth.

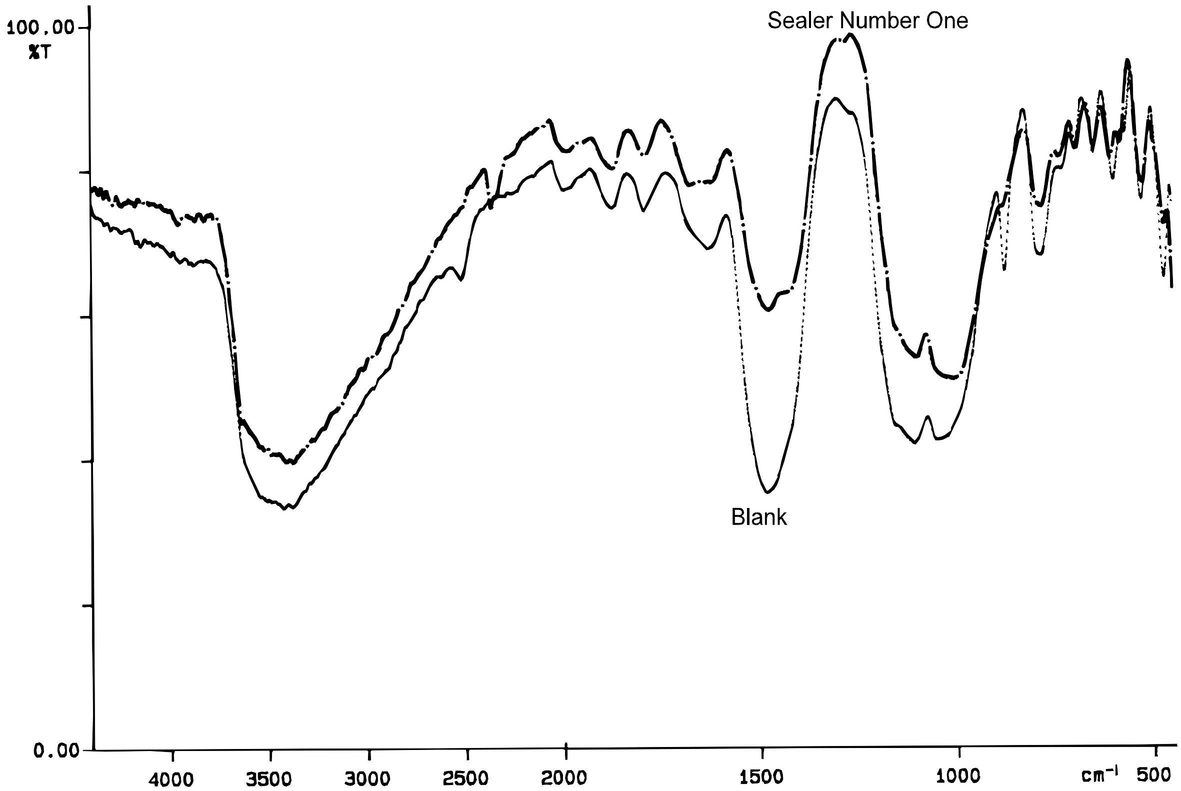

Figure 21 - FTIR Spectra: Sealer Number One (3.0 mm) and Blank, Pellet Sample

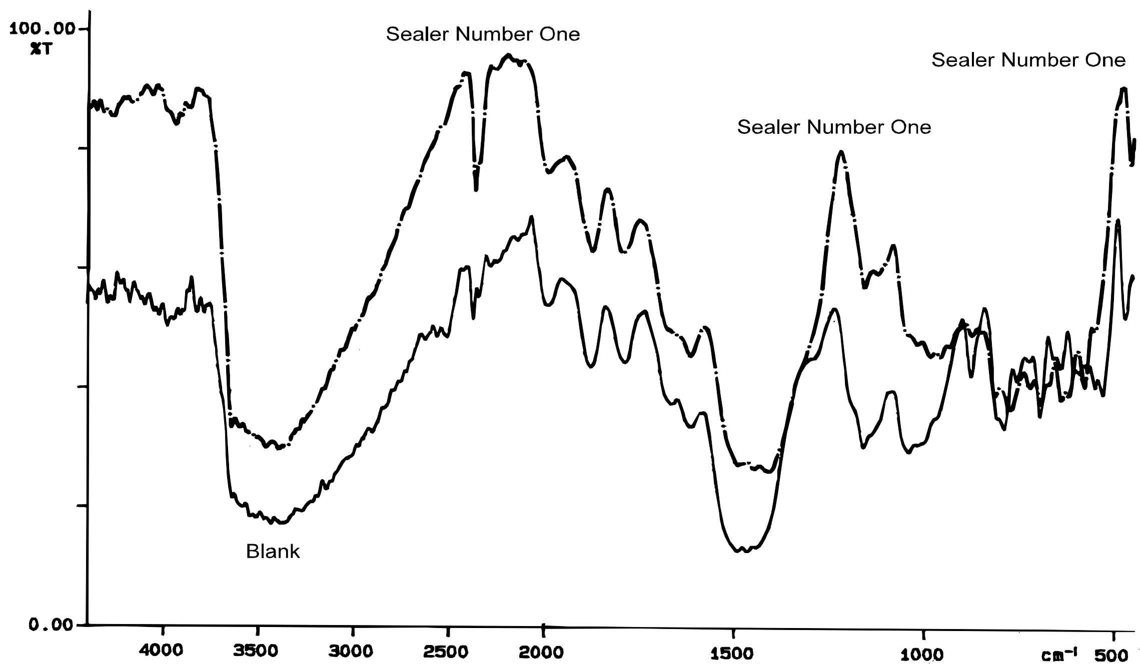

Figure 22 - FTIR Spectra: Sealer Number One (3.0 mm) and Blank, Powdered Sample

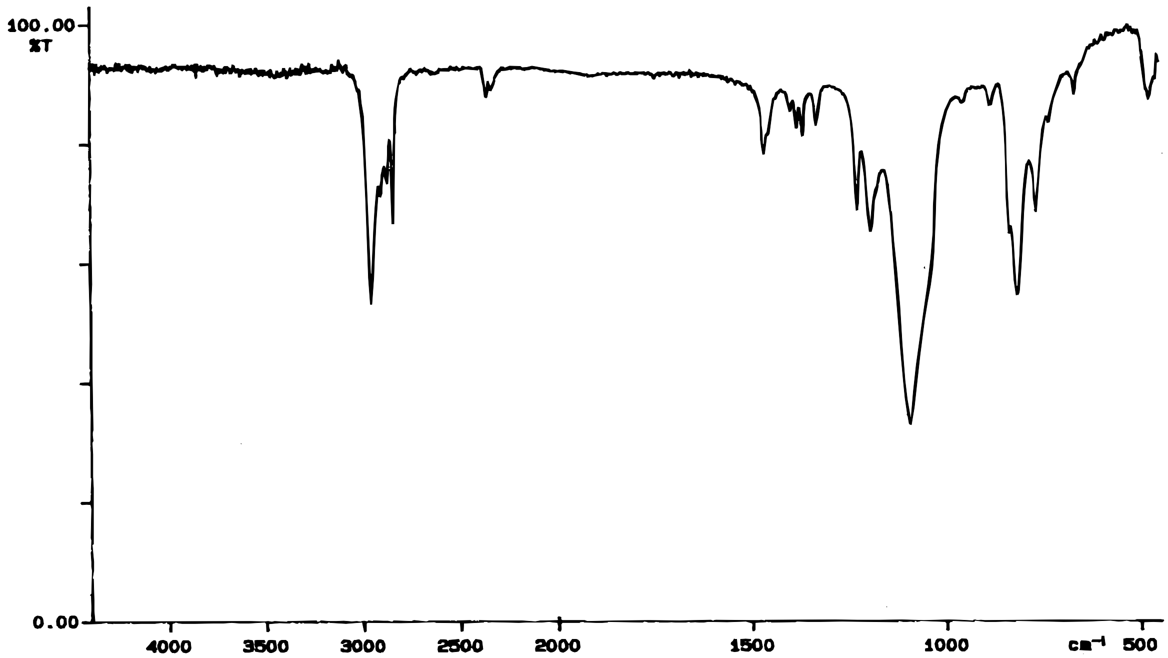

Figure 23 - FTIR Spectra: Sealer Number One, Neat

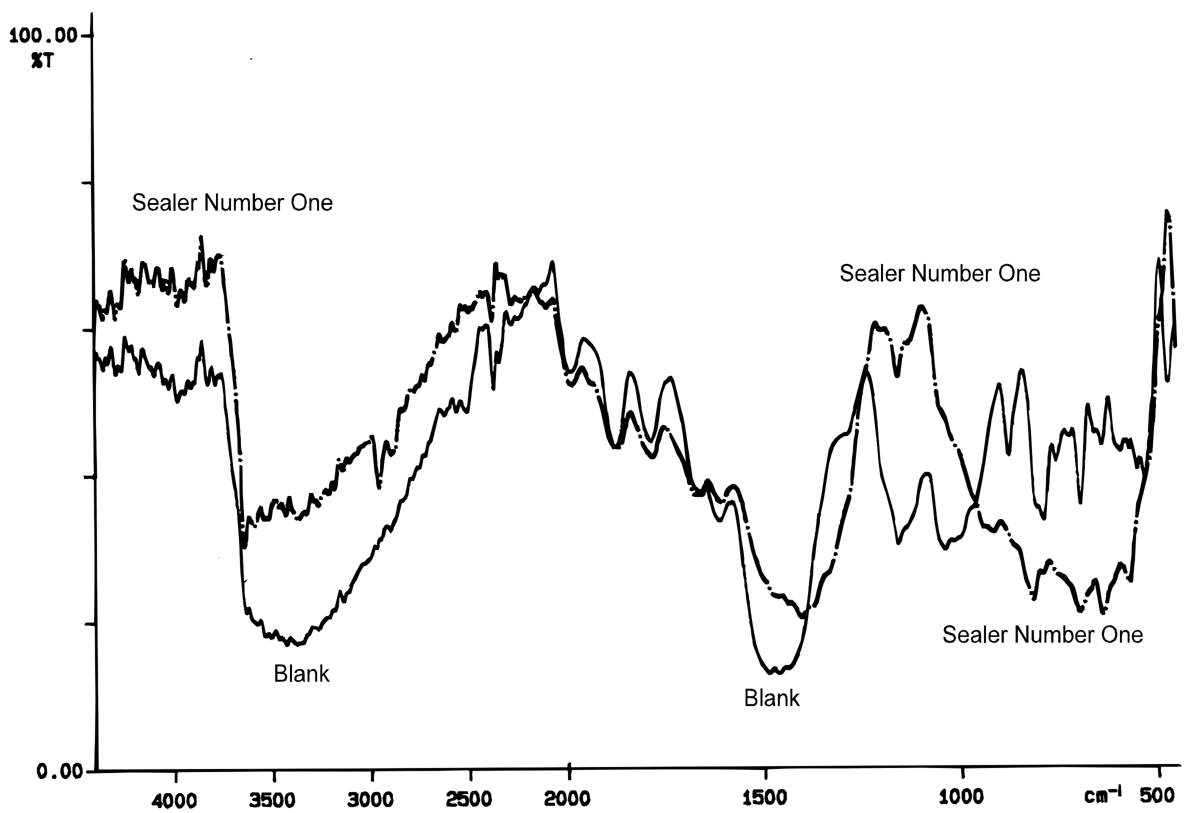

Figure 24 - FTIR Spectra: Sealer Number One (3.0 mm) and Blank, Ponded Sample

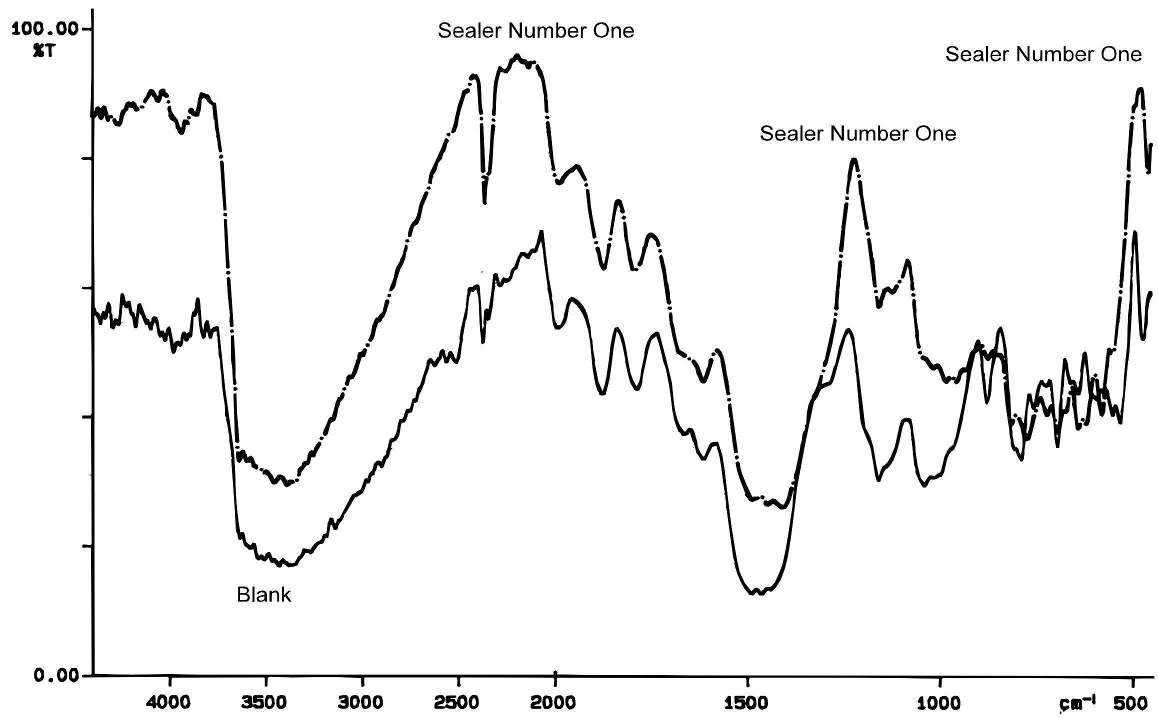

Figure 25 - FTIR Spectra: Sealer Number One (3.0 mm) and Blank, Rerun of Powdered Sample Using Subtraction Method

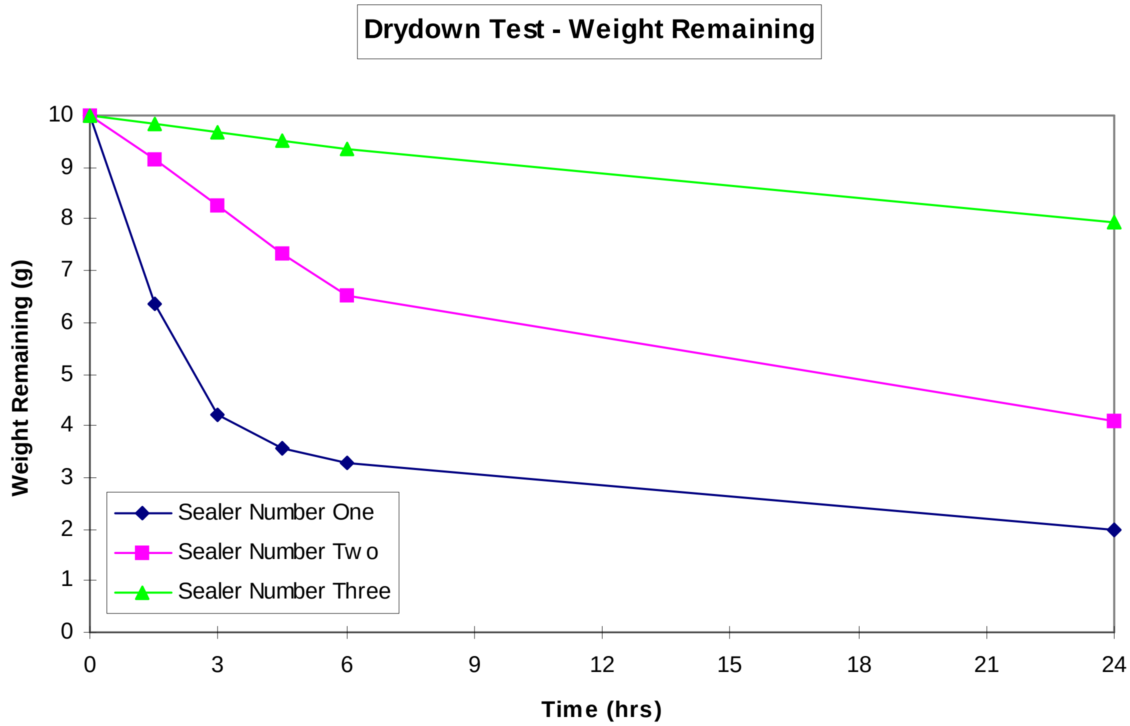

Figure 26 - Sealer Drydown Test, Weight Remaining

ANALYSIS

Statistical analyses were performed on the test data for both freeze/thaw runs, based on the relative dynamic modulus measurements made on the specimens at the end of each run (see Appendix C for analysis results). These values were taken to encompass the cumulative effect of freeze/thaw-induced deterioration on the concrete durability. Analysis of Variance was performed on the specimen groups within each test and t-Tests were performed to compare the effect of the two runs on each sealer and the differences between the sealers and the control for each run.

The analyses indicated that there was an effect on the concrete caused by the sealers for both runs. It also indicated that, with the exception of sealer number one, the controls performed better than the treated specimens; the performance of number one was comparable to that of the control. The specimens from run one also performed better than those in run two[11]; however, the difference was fairly small and could be at least partly attributable to additional curing that took place in the run one specimens while soaking in preparation for application of the sealer.

Examining the graphs of the relative dynamic modulus results indicates some unexpected fluctuations in the values, notably at 36 and 172 cycles on run one and 36 and 108 cycles on run two. While specimens may under some circumstances undergo substantial deterioration between two inspections, normally the specimens continue to deteriorate in subsequent inspections. In these cases, the specimens' modulus either leveled off in the following inspection or actually increased. For a single sample, this is not unusual. It can be caused by a partial delamination of surface material, affecting the way the signal passes through the concrete. When full delamination occurs, the sound material underneath would give results more representative of the condition of the concrete. However, for this behavior to be displayed by several specimens indicates a systemic effect, possibly some difficulty with the test equipment. As the overall pattern for the control specimens was as expected and the overall pattern for the test specimens is reasonable, the final measurements are taken to be accurate. It should also be noted that while the fluctuations in the values are significant in terms of reproducibility when compared to past data, the actual variances are generally relatively small, on the order of one to two percent.

Although the durability of the concrete was greater overall when the concrete was not treated with a sealer, the changes in the modulus were relatively small (less than ten percent). It should also be emphasized that none of the moduli of the specimens even approached the sixty percent

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[11] Sealer number one showed no significant difference between the two runs when the confidence limit was taken as 99%, as was done for the results reported here, but a lower confidence limit showed some sign that the run one specimens performed better.

value of the relative dynamic modulus that is considered a sign of failure by ASTM C215, or even the eighty percent value that past experience has shown to precede the start of significant visible deterioration.

CONCLUSION AND RECOMMENDATIONS

From the results, it is readily apparent that little significant deterioration occurred in any of the specimens. While the coated test specimens showed more distress than the controls, it was not enough to consider the effect of the coating to be of any real detriment to the concrete.

Of more concern are the results in the attempt to detect the penetration depth of the sealers through the use of the FTIR spectrometer. Using the normal coating method, no trace of the sealers could be detected. The question is whether this indicates that there is limited penetration of the sealers into the pore structure of the concrete or only that the FTIR equipment is not capable of detecting the sealers components in the quantities involved.

While only provided for information, the chloride ponding tests showed limited protection against chloride intrusion was provided by sealers one and two after freeze/thaw testing. Sealer number one provided substantially better protection near the surface, but near the depth where the top rebar mat would typically be, both it and sealer number two were only slightly better than the control. Number three actually averaged slightly worse at the lower depth and was significantly worse at the shallower depth.

Since previous testing[12] indicates that this generic class of sealer typically provides very good protection against chloride intrusion, these results coupled with the results for the FTIR testing may indicate that the coatings penetrate only slightly into the concrete and that the loss of paste due to freeze/thaw deterioration is sufficient to remove that protection. This seems to be the case even when the deterioration is relatively slight, as in the case of the specimens in this test.

It should again be noted that ASTM C666 is considered by many to be a harsh test and representative only of either severe or prolonged exposure in a freeze/thaw environment. Therefore, the test may be the equivalent of several years field exposure. The coating may then be considered to provide protection for a length of time sufficient to begin to allow the pore structure of the concrete to close, creating an internal barrier against chloride ion intrusion. However, after some period of time, perhaps three to seven years[13], an additional coat of the sealer might be required to restore the protection. This might be necessary for the life of the structure, although it is possible (although not recommended) that after twenty to twenty-five years the pore system in the concrete may have closed enough that no further coatings are required.

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[12] Fera, J. D., "Laboratory Evaluation of Concrete Sealers For Vertical Highway Structures", FHWA-RI-90-1, January 1991

[13] A *very* rough estimate of the equivalent time of field exposure compared to 300 cycles freeze/thaw testing

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C. Spencer

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C. Spencer
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Sealer Penetration Determination and Report Section of the Same Title **...........** Dr. J. Walsh [R]

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Key: I - RIDOT Summer Intern M - Materials Section, R - Research and Technology Development

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3. Triolo, Mario, F, Elementary Statistics, The Benjamin/Cummings Publishing Company, California, 1989

4. ASTM C-666-92, "Test Method for the Resistance of Concrete to Rapid Freezing and Thawing", Annual Book of ASTM Standards - 1994, Volume 4.02, American Society for Testing and Materials, Philadelphia, 1994

5. ASTM C-215-91, "Test Method for Fundamental Transverse, Longitudinal and Torsional Frequencies of Concrete Specimens", Annual Book of ASTM Standards - 1994, Volume 4.02, American Society for Testing and Materials, Philadelphia, 1994

APPENDIX A

OPERATION OF MODIFIED LOGAN FREEZE/THAW MACHINE

Introduction

The second RIDOT freeze/thaw machine is a modified Logan Freeze/Thaw Cabinet, which had been designed to conform to ASTM C 666 (the modifications to the system do not affect its conformance); Appendix C contains a graph showing the temperature recorded through typical cycles to confirm that the machine stays within the requirements of C 666 (a variation of no more than 3.3˚C throughout the specimen chamber). The machine was purchased in 1994 and was provided with no temperature control or data recording system. At our request, the manufacturer installed a relay to allow the machine to be switched from the cooling phase to the heating phase by computer (it was later decided to add relays to control the main power, heating system and circulation fan). An Advantech PCL-711B analog/digital input/output ISA bus PC expansion board is installed in a standard Intel/DOS machine. Once the program is started and the cycle length is entered, operation of the machine is automatic. The refrigeration and heating systems remain unmodified.

Theory of Operation

The equipment is intended to simulate the freezing and thawing environment that concrete would be exposed to in certain climates. When concrete, in the normal course of events, absorbs water and then is subjected to temperatures that alternate both above and below freezing for extended periods of time, the expansion of the water as it turns to ice can cause internal stresses in the concrete, causing microcracking. Ultimately, this can result in the failure of the concrete as the microcracks network and form larger cracks. When the concrete in question is structural, the results can be catastrophic.

Modern concrete mixes routinely incorporate an admixture which causes the concrete to entrain air. The void structure that is formed when the concrete cures acts to relieve stress by allowing cavities for the moisture to expand into when it freezes (note: these cavities are normally microscopic). The higher the air content (entrainment), the better the resistance to freeze/thaw deterioration. The tradeoff comes in the concrete strength; as the air content increases, the strength of the cured concrete decreases. Determining the freeze/thaw durability of a given mix at a given air content therefore becomes very important to allow a satisfactory balance. Other factors can also affect performance, especially sealers (type, thickness, application) and additives.

The machine subjects concrete specimens to freeze/thaw cycling while they are maintained in a saturated condition. The cycling is intended to simulate the normal conditions of a moderate to severe cold weather climate. The specimens are removed from the machine in a thawed condition once every thirty-six cycles or less (per ASTM C 666) for testing. This allows the deterioration of the specimens to be charted.

ASTM C666 is considered to be a particularly harsh test by many concrete product experts and some test organizations. That the specimens are kept in a saturated condition, with very little clearance for the water around the sides (causing high surface loads as the water freezes), with short cycling times (possibly generating increased stresses due to thermal shock) and relatively small specimens (whereas field concrete tends to be fairly massive and less likely to freeze throughout), this opinion seems somewhat justified. Considering the New England environment, however, (especially near the coast), the severe test conditions are necessary to insure confidence in the survivability of the concrete being tested. Note that the physical layout of the machine places the specimens long dimension horizontally. Further, the nature of freezing water causes the top of the specimen to freeze first and also to thaw last (each specimen has one heating element, placed near the bottom of the specimen). This means that part of the specimen near the top is being frozen very rapidly, in effect "shock" freezing the concrete. This tends to accelerate the deterioration of inferior (low quality aggregate, low air entrainment, etc.)

concrete in that part of the specimen. Based on past experience, however, sound concrete can easily withstand these conditions.

Machine Layout

The machine holds up to sixteen prism specimens, seven and one-half by ten by forty centimeters. The specimens are placed lengthwise in a stainless steel compartment, forty-one and one-half centimeters long and with an interior dimension that allows approximately three millimeters on each side of the specimen when a spacer is added (C666 *requires* a maximum of three millimeters of water around the specimen). The compartments sit flat on a water-saturated felt cloth over a refrigeration platform, with heating elements between each compartment and on each end. The compartments are held in place by stainless steel clips; these clips also secure the position of the heating elements. The top of each compartment is open. When the prisms are placed in the compartment, that space is filled with water (the top of the concrete is also covered with a maximum of six millimeters of water).

Heat transfer between the compartments and heating and refrigeration systems is direct. The fourth and fifteenth compartments each contain a temperature probe placed in a concrete specimen (see Figure A1 for probe construction and Table A1 for calibration data). The outputs of these probes are averaged to determine the temperature of the specimens. The changeover points for the heating and cooling phases are at -17.8 and 4.4˚C. A thermal limit relay (provided by the manufacturer) is built into the heating system to prevent the temperature in the cabinet from rising too high in the event of a malfunction. It can be set to a desired temperature limit and is normally set to 30˚C.

The refrigeration system sits on a platform underneath the cabinet. There is a sight glass mounted in one of the pressure lines. The lines run from the system through the bottom of the cabinet to connect to the coils inside the specimen platform. The heating system consists of a thermal limit relay and nineteen heating elements. Each element has its own plug, which is plugged into a line outlet mounted on the machine. The relays, refrigeration system cutoff switch and circuit breakers are mounted on the side of the cabinet near the refrigeration system. A diagram (see Figure A2) of the machine exterior controls and interior layout follows. A plot

MEASURED TEMP (°C)	MEASURED VOLTAGE, PROBE #1 (V)	CALCULATED TEMP, PROBE #1 (°C)	MEASURED VOLTAGE, PROBE #2 (V)	CALCULATED TEMP, PROBE #2 (°C)
-20.6	4.32	-20.4	4.26	-20.6
-20.0	4.27	-19.9	4.20	-20.1
-19.4	4.22	-19.4	4.13	-19.4
-18.9	4.18	-19.0	4.08	-19.0
-18.3	4.12	-18.4	4.04	-18.5
-17.8	4.08	-18.0	3.97	-17.9
-17.2	4.00	-17.2	3.87	-16.9
-16.7	3.95	-16.6	3.81	-16.4
-16.1	3.91	-16.3	3.79	-16.2
-15.6	3.84	-15.6	3.72	-15.6
-15.0	3.78	-14.9	3.69	-15.2
-14.4	3.74	-14.5	3.61	-14.5
-13.9	3.70	-14.1	3.57	-14.1
-13.3	3.65	-13.6	3.51	-13.5
-12.8	3.58	-12.8	3.44	-12.9
-12.2	3.51	-12.1	3.37	-12.2
-11.7	3.46	-11.6	3.31	-11.6
-11.1	3.39	-11.0	3.24	-10.9
-10.6	3.34	-10.5	3.18	-10.4
-10.0	3.29	-9.9	3.13	-9.9
-9.4	3.24	-9.4	3.08	-9.4
-8.9	3.19	-9.0	3.02	-8.9
-8.3	3.14	-8.4	2.96	-8.3
-7.8	3.09	-7.9	2.91	-7.9
-7.2	3.05	-7.4	2.86	-7.4
-6.7	2.95	-6.5	2.76	-6.4
-6.9	2.98	-6.8	2.79	-6.8
-5.7	2.85	-5.5	2.66	-5.5
-5.0	2.78	-4.7	2.59	-4.8
-4.2	2.71	-4.0	2.51	-4.0
-3.7	2.65	-3.4	2.46	-3.5
-2.7	2.56	-2.5	2.35	-2.5
-2.0	2.50	-1.9	2.28	-1.9
-1.1	2.41	-1.0	2.19	-1.0
-0.6	2.36	-0.5	2.13	-0.5
0.1	2.31	0.1	2.07	0.1
0.8	2.24	0.8	2.00	0.8
1.4	2.17	1.5	1.93	1.4
1.9	2.13	1.9	1.91	1.6
2.7	2.05	2.7	1.80	2.7
3.3	2.00	3.2	1.75	3.2
4.0	1.93	3.9	1.67	3.9
4.8	1.85	4.7	1.58	4.7
5.4	1.80	5.3	1.52	5.3
6.0	1.74	5.8	1.46	5.9
6.6	1.68	6.5	1.41	6.4
7.0	1.65	6.8	1.35	6.9

Note: Probe calibration equations - [PROBE #1 TEMP = -10.2 * (PROBE #1 VOLTAGE) + 23.57], [PROBE #2 TEMP = -9.5 * (PROBE #2 VOLTAGE) + 19.73]. Measured temperature by NIST traceable device, with device probe and probes #1 and #2 in ethylene glycol solution warming to ambient temperature from approximately -20 º C.

Table A1 - Temperature Probe Calibration Chart two probe specimens.

(see Figure A3) of the control temperatures during cycles is also included; this confirms that the machine meets ASTM C666 specifications. The temperatures shown are for the center of the C666 requires that the temperature not vary by more than 3.3˚C on any point on the surface of any specimens. Since the temperature at the center of the specimens lag behind that on the surface, it can be accepted that the surface temperatures varies less than the interior temperatures. C666 also makes provisions for greater temperature variations during changeover (no value is specified); as the greatest variation (<3˚C) is near the low changeover point, this indicates that the machine is well within the requirements.

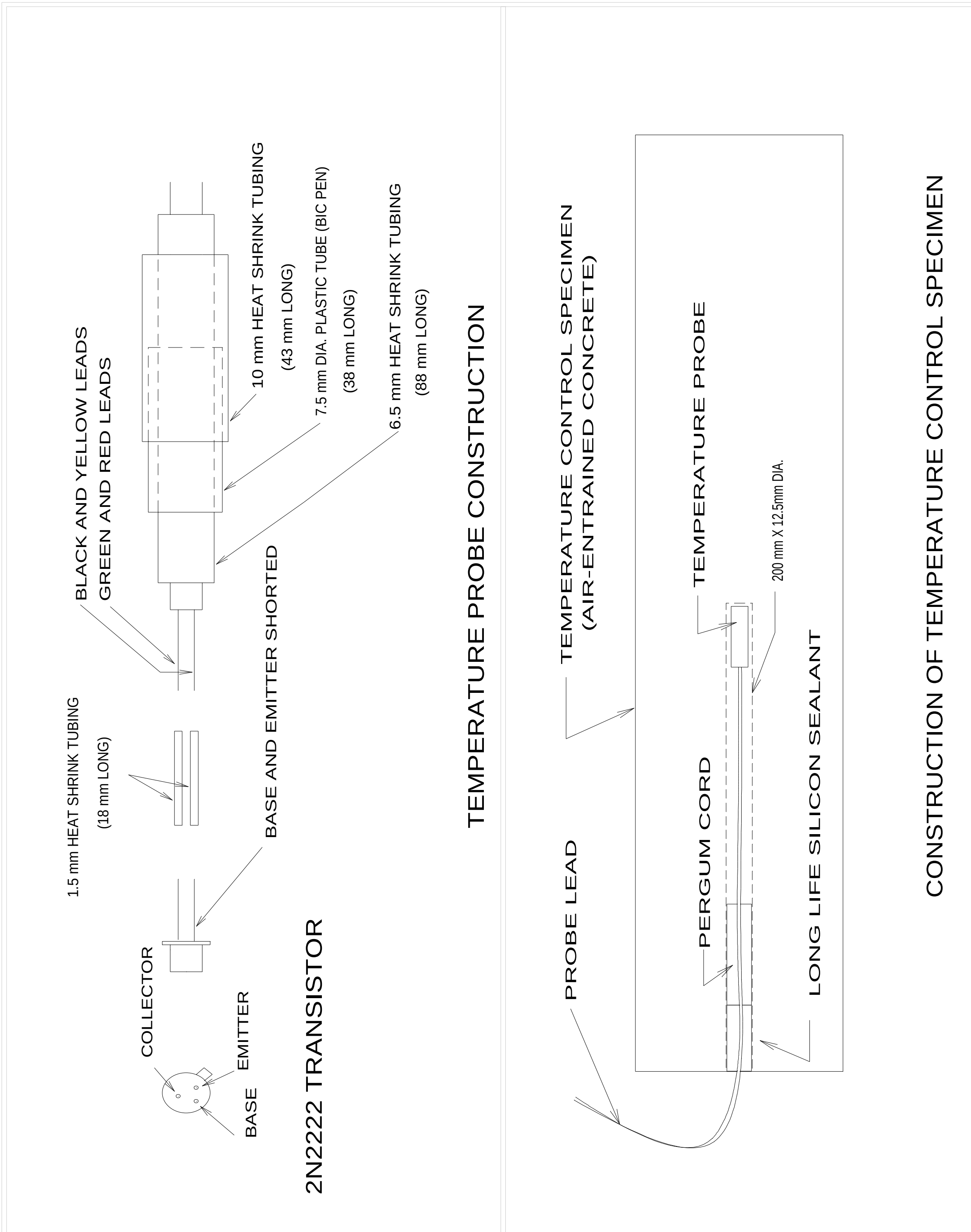

Figure A1 - Temperature Probe Construction and Placement in Control Specimens

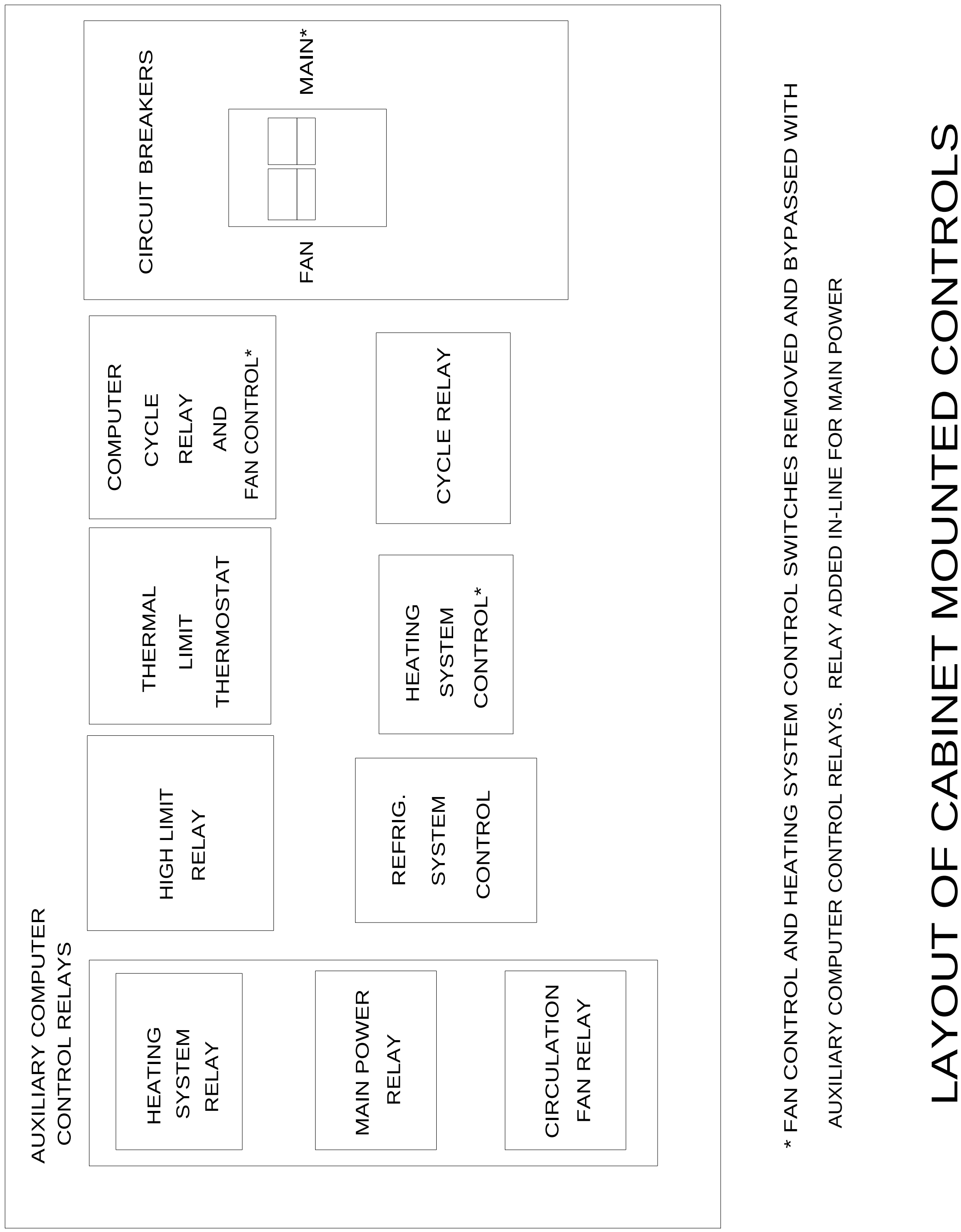

Figure A2 - Layout of Onboard Machine Controls

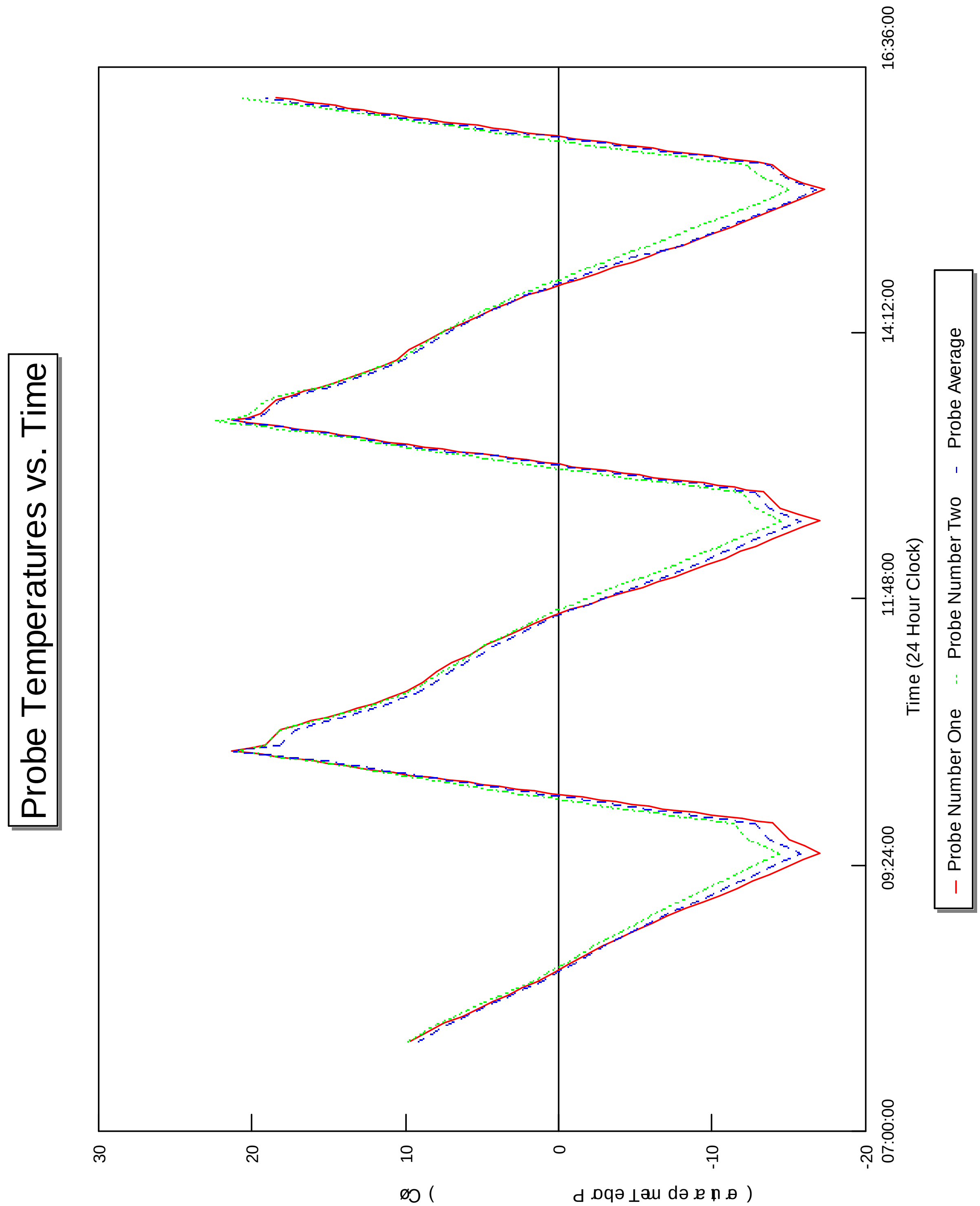

Figure A3 - Sample Plot of Control Probe Temperatures Over Three Cycles

Electronic Control System

The system consists of five major elements: the controlling computer, analog/digital I/O board plugged into the computer, the relay board used for digital output, the temperature probes and the circuit board that converts the probe output into voltages to feed to the analog inputs of the I/O board.

The computer can be any standard DOS/Intel-based computer with a standard ISA bus and 40 MB or larger hard drive. The CPU can be anything better than an 8088/86; the demands on it are not especially great.

The analog/digital I/O board is an Advantech PCL-711B and it plugs into a bus slot on the computer. It has eight analog inputs (two of which are used), one analog output (which is used to drive the strip chart recorder), sixteen digital inputs (none of which are used) and sixteen digital outputs (five of which are used). The analog inputs and outputs are connected to the I/O board using a PCL-715 terminal board. Advantech supplied driver programs to access the various I/O functions, which were then incorporated into a program to operate the machine. The manual for the board is available for study.

The PCLD-786 relay board is fed from the I/O board. It then controls the electric power fed to five relays, either directly on D/O channels eight through fifteen or through solid state relays (SSR) on D/O channels zero through seven :

1] The relay connected to channel zero controls power to the heating system.
2] The relay connected to channel one controls power to the strip chart recorder motor.
3] The relay connected to channel two controls power to the main line into the machine.
4] The relay connected to channel three controls power to the circulation fan.
5] The relay connected to channel eight controls the cycling relay on the machine (when the relay is actuated, the refrigeration system is powered; otherwise, the heating system is on).

The power for channels zero through three are fed from an external 120 volt line, jumpered from channel to channel. The power for channel eight requires only five volts and is supplied by the computer. The manual for the board is available for study.

The temperature probes are 2N2222 transistors, with the base and emitter. Their electrical characteristics vary with temperature, which is used to determine their temperature and hence, the temperature of their surroundings.

The probes are attached to a circuit board which converts the output to a voltage which can be read by the I/O board. A regulated twelve volt power supply feeds the circuit board. The outputs of the circuit are connected to a terminal board provided with the 711B and feed to A/D channels zero and one. The D/A channel feeds a signal to the strip chart recorder stylus movement from the same terminal board.

FREEZE/THAW MACHINE CONTROL PROGRAM

INPUTS:

1) Analog - Two (2) from temperature probes (transistors), one (1) in each of two (2) temperature control specimens, which are used to determine the cycling of machine. The two values are averaged to determine the point of changeover. Up to six more analog inputs are available and may be used in the future to add probes to various locations in the machine and on its exterior. Note: provisions are made for adding up to six (6) additional probes and storing the temperature data. All that is required is to remove the apostrophes remarking out the appropriate program lines.

2) Data inputs - The total elapsed cycles are inputted to the program from the disk at the start of each new set of cycles. The constants for the temperature probe calibration equations are also inputted to the program at this time.

OUTPUTS:

1) Digital - One (1) to the power input to the machine. One (1) to the heating system. One (1) to the circulation fan. One (1) to the strip chart recorder motor (see section 2). One (1) to the cycling relay that controls the heating and cooling phases.

2) Analog - One (1) to the strip chart recorder, which acts as a backup system to record the average temperatures of the thermal control specimens.

MANIPULATION:

1) Probe inputs - All will be read as voltages; equations (developed from calibration) will be used to convert the voltages to the temperatures of the probes. At the low changeover point (typically -17˚C), power will be sent to the main power, the heating system, the circulation fan and the strip chart recorder motor. This starts the heating phase. At the high changeover point (typically 2˚C), power will be sent to main power, the cycling relay, the circulation fan and the strip chart recorder motor.

2) Cycling - The system will count the number of cycles (based on the number of low changeover points encountered) and put the machine on standby at an operator inputted value for the cycles (generally thirty-six).

3) Data outputs - All inputs are saved to disk at intervals (to prevent the disk drive from operating constantly) for graphing and later analysis. The starting and ending times are recorded. The point of changeover is recorded to measure number of cycles.

APPENDIX B

CONCRETE MIX AND TEST DATA FOR SPECIMENS

Run one:

Nominal Aggregate Size (cm): 1.9
28 Day Design Strength (MPa): 28.0

	Mix 1	Mix 2	Mix 3
28 Day Strength (MPa)	40.1	34.7	38.0
Air Content (%)	5.3	5.9	5.3
Concrete Temp (˚C)	22	22	22
Air Temp (˚C)	22	22	22
Slump (cm)	10.0	8.8	10.0

Run two:

Nominal Aggregate Size (cm): 1.9
28 Day Design Strength (MPa): 28.0

	Mix 1	Mix 2
28 Day Strength (MPa)	35.8	35.4
Air Content (%)	5.5	5.7
Concrete Temp (˚C)	22	22
Air Temp (˚C)	22	22
Slump (cm)	12.0	11.5

APPENDIX C

STATISTICAL ANALYSIS RELATIVE DYNAMIC MODULUS DATA
[Output from Statmost for Windows]

t-Test Analysis Results - Run 1

Coating #1 vs. Control:
Descriptive Data

	Coating #1	Control	
Sample Size	4	4	-
Mean	96.475	97.225	Difference = -0.750000
Variance	0.1225	0.509167	Ratio = 0.240589

t-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-1.887328	0.108052	6	-	-
Unpaired	-1.887328	0.132158	4	-	-
Paired	-1.975281	0.142711	3	0.0275	0.379693

Coating #2 vs. Control:
Descriptive Data

	Coating #2	Control	
Sample Size	4	4	-
Mean	95.675	97.225	Difference = -1.550000
Variance	0.429167	0.509167	Ratio = 0.842881

t-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-3.200244	0.018594	6	-	-
Unpaired	-3.200244	0.018594	6	-	-
Paired	-3.905633	0.029809	3	0.154167	0.396863

Coating #3 vs. Control:
Descriptive Data

	Coating #3	Control	
Sample Size	4	4	-

	Coating #3	Control	
Mean	95.075	97.225	Difference = -2.150000
Variance	0.255833	0.509167	Ratio = 0.502455

t-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-4.916293	0.002667	6	-	-
Unpaired	-4.916293	0.004412	5	-	-
Paired	-4.507625	0.020397	3	-0.0725	0.47697

t-Test Analysis Results - Run 2

Coating #1 vs. Control:
Descriptive Data

	Coating #1	Control	
Sample Size	4	4	-
Mean	95	99.85	Difference = -4.850000
Variance	0.82	1.27	Ratio = 0.645669

t-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-6.70963	0.000532	6	-	-
Unpaired	-6.70963	0.000532	6	-	-
Paired	-4.947863	0.015841	3	-0.876667	0.980221

Coating #2 vs. Control:
Descriptive Data

	Coating #2	Control	
Sample Size	4	4	-
Mean	91.275	99.85	Difference = -8.575000
Variance	0.329167	1.27	Ratio = 0.259186

T-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-13.561798	0.00001	-	-	-

	t-Value	Probability	DF	Co-Variance	Std Deviation
Unpaired	-13.561798	0.000171	4	-	-
Paired	-14.621139	0.000694	3	0.111667	0.58648

Coating #3 vs. Control:
Descriptive Data

	Coating #3	Control	
Sample Size	4	4	
Mean	91.725	99.85	Difference = -8.125000
Variance	3.3425	1.27	Ratio = 2.631890

t-Test Results

	t-Value	Probability	DF	Co-Variance	Std Deviation
General	-7.566328	0.000277	6	-	-
Unpaired	-7.566328	0.00064	5	-	-
Paired	-6.432177	0.007618	3	-0.885	1.263181

ANOVA

Run 1
Descriptive Data

Name	Count	Mean	Std. Dev	Std. Err
Coating #1	4	96.475	0.35	0.175
Coating #2	4	95.075	0.5058	0.2529
Coating #3	4	95.675	0.6551	0.3276
Control	4	97.225	0.7136	0.3568

One-Way ANOVA Results

Source	DF	SS	MS	F	P
Between Groups	3	10.5475	3.5158	10.681	0.0011
Within Groups	12	3.95	0.3292	-	-
Total	15	14.4975	-	-	-

Run 2

Descriptive Data

Name	Count	Mean	Std.Dev.	Std.Err
Coating #1	4	95	0.9055	0.4528
Coating #2	4	91.725	1.8283	0.9141
Coating #3	4	91.275	0.5737	0.2869
Control	4	99.85	1.1269	0.5635

One-Way ANOVA Results

Source	DF	SS	MS	F	P
Between Groups	3	187.8725	62.6242	43.4764	0.000001
Within Groups	12	17.285	1.4404	-	-
Total	15	205.1575	-	-	-