# ORTHODONTIC CEMENTS MIXED BY THE FROZEN SLAB TECHNIQUE

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# INTRODUCTION

Probably no dental practitioner is more dependent upon the performance of dental cement than is the orthodontist. With today's pre-formed bands, dental cement is utilized mainly as a long term filling and sealing agent. The dental cements available and most commonly used for the cementation of orthodontic bands have two properties which are disadvantageous in their clinical use: a short working time not permitting the cementation of more than a few bands at one time, and a setting time in the oral environment requiring a wait of several minutes before the cement sets, creating more chance for the deleterious effects of saliva contamination on the unset cement and requiring a wait of several minutes until the excess cement can be removed. The dental profession is continuously searching for a cement or a mixing technique that will allow a longer working time and a shorter setting time without compromising the necessary filling and sealing properties of the cement. At the present time most orthodontists are mixing zinc phosphate cement on a cooled, chilled or frozen slab in order to extend the working time and decrease the setting time.

The purpose of this in vitro study is to determine if silicophosphate and polycarboxylate cements in addition to zinc phosphate can be mixed on a frozen slab in order to extend their working time and decrease their setting time without adversely affecting their physical properties.

# LITERATURE REVIEW

Paffenbarger et al[1] reported mixing $ZnPO_4$ on glass slabs immersed in water and chipped ice. The temperature was below the dew point and the slabs sweated; however, this was found to have no harmful effect on the cement. The cool slab produced an extended working time, but as soon as the mix was transferred to the mouth, setting was completed uniformly.

Henschel[2] measured working and setting times of zinc phosphate cement mixed at temperatures ranging from 40 to 100° F. Henschel found that cements mixed between 40-60° F showed increased working times as compared with those mixed at room temperature or higher.

Jendresen[3] substantiated these results by demonstrating that the working time of zinc phosphate cement could be doubled and the setting time could be decreased 30-50% without significantly affecting the strength and solubility of the cement by chilling the mixing slab to 45° F.

Tuenge[4] mixed zinc phosphate cement on a frozen slab at

-23° C or -9° F and found working time could be increased four times and the setting time of the frozen slab mix decreased to one-half the room temperature mix.

Kendzior et al[5] mixed zinc phosphate at 23° C, 6° C, and -10° C and found that mixing the cement substantially below the dew point was an acceptable practice provided the powder/liquid ratio is modified. The working time of the cement was increased three times for the 6° C mix and seven times for the -10° C mix. The setting time in an oral environment was decreased one to three minutes for both the 6° C and -10° C mixes.

Friend[6] suggested the use of a cooled slab when mixing polycarboxylate cement on a warm day. McLean[7] used a chilled slab and refrigerated the powder when mixing polycarboxylate cement to extend the working time. Traces of moisture condensation on the slab were found to have no adverse effect on the properties of the cement. Kafalias et al[8] mixed polycarboxylate cement on a cool glass slab.

Garman and Morris[9] recommend mixing Durelon according to the manufacturer's instruction for basing on a "frozen" glass slab. Garman and Morris report that the manufacturer's powder to liquid ratio can be doubled, the working time extended to fifteen minutes, and the setting time reduced when Durelon is mixed on a frozen glass slab.

Wilson and Batchelor[10] suggest that an increased powder to liquid ratio of slicate cement can be achieved by use of a cool slab; however, the slab should not be cooled below 21° C because of the possibility of moisture contamination.

Cameron et al[11] state that the best orthodontic cementation mix combines the maximum amount of powder consistent with the time requirement; however, Cameron found that cements with a higher powder to liquid ratio did not necessarily have better physical properties. Paffenbarger[1] found that extremely heavy mixes of putty-like consistency were not appreciably stronger than more plastic mixes; however, thicker mixes were stronger than thinner.

In the American Dental Association's "Guide to Dental Materials"[13] are found the specifications for zinc phosphate and silicophosphate cements: specifications number 8, 9, and 21. Polycarboxylate cements have no ADA specification, so ADA Spec. #8 is used.

Volland, Paffenbarger, and Sweeny, 1935[12], established A.D.A. Spec. #8 as the average consistency of 165 mixes from seventeen groups of practicing dentists. A disc diameter of 30 ± 1 mm in the slump test was established from the average mix these dentists were using for cementation purposes. Cameron et al[11], in 1963, modified A.D.A. Spec. #8 because the 30 ± 1 mm disc diameter establishing the cement consistency was designed for the cementation of crowns and inlays not orthodontic bands. Cameron surveyed thirteen orthodontic practitioners and established an average powder to liquid ratio from the mixes the dentists were utilizing clinically. The A.D.A. consistency was run on this mean mix and a disc diameter of 26.5 ± 1 mm was established as the standard orthodontic mix for both zinc phosphate and silicophosphate cements. Miller and Houston[14] used a consistency of cement considerably thicker than the Federation Dental Internationale Spec. #6 in their solubility and compressive strength tests on orthodontic cements. The consistency was close to the one selected by Cameron et al.

Jendersen[3] established a powder to liquid ratio for a room temperature control mix as the average powder to liquid ratio from mixes of thirteen practitioners. Two operators using the average P/L ratio of the 13 practitioners mixed a control and then a series of four cold slab mixes. The cold slab mix nearest the control mix by consensus was used as the powder to liquid ratio in the subsequent cold slab testing. Kendzior et al[5] used a powder to liquid ratio in their cold temperature mixing to produce a consistency of 26.5 ± 1 mm as described by Cameron.

The common way for dentists to test consistency of a clinical cement mix is the string test. The viscosity of the cement is considered correct when a string of cement 3/4-1 inch long can be pulled from the mix by the spatula. Moon and Modjeski[15] report there are a number of variables that cause a variation in the string test: the amount of cement in the area from which the string is pulled, the flow of excess cement from the spatula as the string is pulled, and changes in the powder to liquid ratio. As the powder to liquid ratio increases, the string length increases to two inches, then as more powder is added the string length decreases back to 3/4 to one inch. Moon and Modjeski[15] suggest a simple test for measuring consistency and they call it the flow test; in which consistency is correlated to flow. Two minutes after starting a mix, a specified amount of cement is wiped on the edge of a vertical slab at 13° C and allowed to flow for one minute. The slab is then turned horizontal and flow length measured. Flow equals flow length divided by flow mass.

Vermilyea et al[16] state that the viscous changes during the ten minute test period are not included in Specification #8. A Brookfield rotational viscometer was used to evaluate the consistencies of the test cements as a function of time, temperature, and shear rate. Paffenbarger in 1935 established A.D.A. Specification #8 for the mixing of zinc phosphate cement. The cement was mixed for 1½ minutes using at least one half the polished glass slab surface with incorporation of 1/16, 1/16, 1/8, 1/4, 1/4, and 1/4 increments of the powder respectively for 10, 10, 15, 15, and 30 seconds. Henschel[2] averaged a mixing time of two to three minutes. Jendersen[3] used a 30 second cold slab mixing technique for zinc phosphate cement with immediate incorporation of all powder. He felt this technique allowed a better standardization of the mix. Tuenge[4] used an incremental mixing time ranging from 30 seconds to one minute as the powder to liquid ratio increased over the range of 1.4g to 2.8g/0.5 ml.

A.D.A. Specification #21 specifies that zinc silicophosphate cement be mixed for one minute with 1/2 the powder incorporated for 15 seconds, then 1/4 of the powder for 15 seconds, then the remaining 1/4 of the powder incorporated for 15 seconds, then the whole mass spatulated for 15 seconds.

A.D.A. Guide to Dental Materials[13] suggests that polycarboxylate cements be mixed very quickly since the mixing technique only requires the wetting of all particles. Kafalias et al[8] mixed polycarboxylate cements by incorporating 4/5 of the powder at once then the rest over a 30 second total time of mix.

Cameron et al[11] in establishing their orthodontic mix used a powder to liquid ratio of 1.8 g to 0.5 ml for Tenacin zinc phosphate cement. Williams et al[17] mixed Tenacin 1.25 g to 0.5 ml. Norman et al[18] used 1.4 g Tenacin powder to 0.5 ml liquid. Savignac et al[19] determined a mean powder to liquid ratio of 1.13 g/0.5 ml for Stratford-Cookson cement from three mixes from each of 31 participating dentists. The conclusion of Savignac was that the powder to liquid ratio should not be less than 1.3 g/0.5 ml.

Henschel[2] and Jendresen[3] were able to increase their powder to liquid ratio to approximately 2 g/0.5 ml by mixing on cold slab, an increase in powder of 30-40%. The Henschel, Jendresen slab temperatures were approximately 40° F. Kendzior[5] was able to incorporate 49-70% more powder when the cement was mixed at 6° C, and at -10° C, 63-91% more powder was incorporated into the mixes. Kendzior found the amount of powder necessary to maintain a constant viscosity increased almost linearly as the slab temperature decreased.

Vermilyea et al[16] found Tenacin's initial viscosity dropped 54% with a 7° C decrease in mixing slab temperature.

Phillips et al[20] mixed silicophosphate cement with a powder to liquid ratio of 1.2 g/0.4 ml. Cameron in his test establishing a 26.5 ± 1 mm disc diameter for orthodontic cement mixed Kryptex with a powder to liquid ratio of 1.4 g to 0.4 ml. Williams et al[17] mixed Kryptex with a P/L ratio of 1.32/0.4.

Rich et al[21] in an orthodontic cement retention study mixed Durelon cement 0.49 g/0.5 ml. McLean[7] recommends a Durelon powder to liquid ratio for a lining cement of 2 g/1 g,for a luting cement 1.5 g/1 g. Garman and Morris[9] report doubling the manufacturers' recommended powder to liquid ratio for Durelon when mixed to a basing consistency on a frozen slab. Vermilyea[16] et al found Durelon's initial viscosity dropped 8% with a 7° C decrease in slab temperature.

To date all studies utilizing a cool, cold or frozen slab have used glass slabs. Henschel[2] did not allow any condensation in the mixing surface. Jendresen[3] removed the cold slabs from the refrigerator and allowed them to sit 20 seconds before mixing with no attempt to wipe off moisture condensation. Kendzior et al[5] sealed the glass slabs in plastic bags, but did not attempt to eliminate moisture on the slabs after removing them from the bags prior to mixing. Tuenge[4] also sealed the slabs in plastic bags; however, the slabs were scraped with a plastic blade and wiped dry with a paper towel prior to mixing.

Norman et al[22] found cement liquids should be protected from water contamination during mixing. An increased water content increased the setting time, markedly increased the solubility, and decreased the compressive strength of zinc phosphate and silicophosphate cements. As reported earlier, McLean[7] felt traces of moisture condensation on the slab when mixing polycarboxylate cement had no effect on the properties of the cement.

Jendresen[3] estimated the working time by clinical judgement of five dentists loading orthodontic bands at 15 second intervals from three control mixes until the mixes were too thick to use. Tuenge[4] estimated working time two ways: the cement was left on the slab until unfit to use, and a full arch set of bands were filled and the filled bands left on the slab until unfit to use. Kendzior[5] had two orthodontists and a dental assistant determine on a subjective basis when the cement was no longer usable. He found the working time continued to increase as slab temperature decreased.

There is no standard at the present time for estimating working time of cementing materials. Plant et al[23] utilized an oscillating rheometer to estimate the working and setting times of cements. The oscillating rheometer apparatus was designed to produce a chart trace. The working time of the cements were determined as the time when the trace width dropped to 95% of its initial width. The setting time was the time when the rheometer no longer oscillated in the cement.

Kent et al[24] report the resistance of a freshly set cement to aqueous attack increases with its speed of set, which signifies clinically that the risk of moisture contamination is reduced. Wilson and Batchelor[10] found a close correlation between decreased setting time and decreased phosphate eluted in the first 24 hours. The close correlation between phosphate eluted and setting time for a dental silicate cement indicates that initial erosion is largely dependent on the rate of reaction in the cement. Durability may be dependent on setting time. Phillips et al[25] and Rich et al[21] report no significant difference in the setting times of zinc phosphate and carboxylate cements.

Mitchem[26] concluded that the differences demonstrated in the laboratory tests of compressive strength and solubility for dental silicate cements did not predict clinical behavior. With Mitchem's findings in mind a discussion of the physical properties of the cements will begin.

Jendresen[3] and Kendzior et al[5] report no difference in compressive strength and solubility between cold slab mixes and room temperature mixes of zinc phosphate cement. Tuenge[4] reported that frozen slab mixes had a lower compressive strength and greater solubility than room temperature mixes. Tuenge found an increase in compressive strength as the P/L ratio increased on the frozen slab.

Only at 2.8 g/0.5 ml, the highest frozen slab P/L ratio, did the compressive strength of the frozen mix equal the compressive strength of the room temperature mix. Kendzior et al[5] report if a constant viscosity is maintained then the compressive strength and solubility values remain constant as the temperature of the mixing slab decreases. Savignac et al[19] found the compressive strength of zinc phosphate cement increased with an increase in the powder to liquid ratio; the range was 8,900 psi to 17,000 psi. Mixing zinc phosphate substantially below the dew point is acceptable provided the P/L ratio is increased. As reported earlier, Norman et al[18] found an increased solubility and a decreased compressive strength in zinc phosphate and silicophosphate cements when the water content of the liquids was increased, as can occur in a frozen slab mix with moisture condensation on the mixing surface. However, unlike Jendresen and Kendzior, Norman did not maintain a constant viscosity by increasing his P/L ratio as the liquid was diluted by water contamination.

Anderson and Paffenbarger[27] report that silicophosphate cements are stronger than zinc phosphate cements in compressive strength. Williams et al[17] found the compressive strength of silicophosphate cement to be twice that of zinc phosphate.

Smith[28] reports that polycarboxylate cements have compressive strength comparable though somewhat lower than zinc phosphate cement. Rich et al[21] and Grieve[29] found that carboxylate cement was clearly inferior to zinc phosphate in compressive strength. Phillips et al[25] found zinc phosphate cement to be twice as strong as carboxylate cement. Mortimer and Tranter[30] report that the compressive strengths of polycarboxylate cements do not meet the minimum requirement set by A.D.A. Specification #8. Increasing the amount of powder in carboxylate cement mix 50% resulted in an improvement in the compressive strength. Kafalias et al[8] also found an increase in the compressive strength of polycarboxylate cement when the amount of powder is increased; however, as compared to zinc phosphate the increase is modest.

Wilson and Batchelor[31] state that the only reason for having the 24 hour solubility disintegration test is to determine if the value for solubility and disintegration for a cement is unusually high. If the value is too high then the cement absorbs excessive water, swells and disintegrates. Wilson et al[32] and Norman et al[33] felt the solubility-disintegration test is a useful criteria for comparing the same type of cement, but it has no value in comparing the relative merits of different types of cement. Wilson et al[34] concluded that the solubility and disintegration test period of 24 hours is too short to be clinically predictive. Clinically the test gives a measure of a cement's resistance to early moisture attack. Miller[35] found that extending the solubility test over two to three days to a steady state basis did not rank the test cement specimens in the same order as the 24 hour test. Miller submitted that such steady rates would give a better basis of assesssment of probable durability of cements. Norman et al[18] report the belief by some investigators that the rate of disintegration in dilute organic acids common to the oral cavity could provide data which are more meaningful clinically than does the solubility in distilled water. Never has it been established that the solubility of cements in either water or organic acids is an index of clinical disintegration of cements. Richter and Ueno[36] found it questionable to infer that distilled water and the fluids of the oral environment have a similar effect on cements.

Richter and Ueno[36] in vitro A.D.A. solubility tests ranked cements from best to worst: polycarboxylate, zinc phosphate, and silicophosphate; however, in vivo tests ranked the cements silicophosphate least degradation, then zinc phosphate, and last polycarboxylate. Cameron et al[11] and Eichner et al[37] found zinc phosphate cements to be much less soluble in A.D.A. specification tests than silicate cements. Anderson and Paffenbarger[27] found the solubility of zinc phosphate cement in distilled water 1/10 that of silicophosphate cement. Sadowsky and Retief[38] found the solubility of zinc phosphate to be 0.1% by weight and silicophosphate to be 0.8% in the 24 hour test. The general clinical opinion is that silicophosphate cement is less soluble in the mouth than zinc phosphate cement.

Wilson[39] explained that dental silicate cements are unique in containing sodium ions which have a high propensity to form highly soluble salts whose solubility and disintegration are higher when compared to other dental cements. This does not mean the dental silicate cement matrix is more susceptable to aqueous attack since soluble sodium salts do not form part of the binding matrix. For this reason values obtained for dental silicate cements using the A.D.A. test cannot be used to compare them with other types of dental cement.

Phillips et al[25] and Rich et al[21] found the solubility of polycarboxylate cement to be equal to zinc phosphate in the 24 hour A.D.A. test. Mortimer and Tranter[30] found an increased powder content of polycarboxylate cement did not affect the solubility of the set cement. Moser et al[40] tested polycarboxylate cement in water over 16 days and found a prolonged high solubility rate.

Phillips et al[20] found enamel surfaces in apposition to silicate or silicophosphate cement showed a higher fluoride content and a decrease in calcium acid solubility. The effect of silicophosphate cement on enamel was considerably less than produced by silicate cement. Clark et al[41] observed in a 14 month clinical test no decalcified enamel under loose bands when the band was cemented with silicophosphate cement; however, decalcification was occasionally evident when zinc phosphate was used.

# MATERIALS AND METHODS

The three cements used in this study were zinc phosphate (Tenacin), silicophosphate (Fluoro-thin), and polycarboxylate (Durelon). All three cement powders were removed from the small bottles as provided by the manufacturer and incrementally mixed in one large bottle with a tumbling action to insure a uniform powder consistency throughout the experiment. The Tenacin and Fluoro-thin liquids were aspirated from the bottles as packaged by the manufacturer into a 1.00 cc calibrated hypodermic syringe and then the correct liquid volume dispensed for each mix. The Durelon liquid was dispensed from the syringe provided by the manufacturer.

The mixing surfaces used in this study were room temperature glass slabs, frozen glass slabs, Caruso cold slabs (Unitek extended time cold slab), and the pad provided by the manufacturer of Durelon. Table 1 lists the various mixing surfaces used for each cement. Table 2 gives the mixing slab temperature for the various mixing surfaces when mixing of the test cement specimens was initiated. Figure 1 is a graph showing the approximate rise in temperature vs. time for the Caruso and glass slabs.

The frozen glass slabs were used immediately after removal from the freezer with no attempt to eliminate the frost or moisture condensation. The Caruso slabs were allowed to sit for ten minutes and then were wiped with an alcohol soaked sponge and then dried with a paper towel. This procedure was followed in order to eliminate as much moisture condensation as possible prior to mixing. The frozen slabs were used only once in a 12 hour period. The cement powders were weighed on Mettler P163 balance. Table 3 gives the powder to liquid ratios of the room temperature mixes.

Room temperature mixes of Tenacin and Fluoro-thin were mixed according to the manufacturer's recommended powder to liquid ratio of 1.3 g/0.5 ml and 1.1 g/0.4 ml respectively. The manufacturers of Durelon did not recommend a powder to liquid ratio for orthodontic cementation, so a median powder to liquid ratio between that for basing and cementation was chosen as the powder to liquid ratio for this experiment. For cementation the manufacturer recommends three scoops from the measuring stick to three full calibrations from the liquid dispensing syringe. For basing, the recommendation is three scoops to two full calibrations from the syringe. The mean ratio would be provided by mixing three full scoops from the measuring stick with 2½ full calibrations from the syringe. Three full scoops from the measuring stick averaged 0.6 g and 2½ calibrations from the syringe was .55 ml or 0.51 g. The cements were mixed in an atmosphere of 50% humidity and 22° C. The mixing time for each cement test condition is found in Table 4.

Tenacin was mixed at room temperature by incremental mixing of 1/16, 1/16, 1/8, 1/4, 1/4, 1/4 of the powder respectively for 10, 10, 10, 15, 15, and 30 seconds according to A.D.A. Specification #8. Fluoro-thin at room temperature and all frozen mixes of Fluoro-thin and Tenacin were mixed by incorporating 1/2 the powder for 15 sec., then 1/4 the powder for 15 sec., then the remaining 1/4 the powder for 15 sec. and then spatulation of the whole mass for 15 sec. This is the mixing technique for dental silicate cements in A.D.A.

Specification #9. All mixes of Durelon were mixed for 30 sec. with immediate incorporation of all the powder.

A modified A.D.A. Specification #8 standard consistency test procedure was used. The modifications were the following:

1. All frozen slab mixes were slump tested on a frozen glass slab with the 20 g plate and 100 g weight cooled on the glass slab during the mixing and preparation of the frozen slab test mixes.
2. Frozen glass was used as the slump test surface because;
    a. The test surface would be approximately the same temperature as the mixing surface, therefore, minimizing the possibility of altering the viscosity of the frozen mix during the test.
    b. Polished glass is specified as the test surface in A.D.A. Spec. #8.
3. The weight and plate were placed to initiate the test for Fluoro-thin 2½ minutes after initiation of spatulation because the mixing time was one minute instead of the

specified 1½ minutes. The weight and plate were removed ending the test after 9½ minutes for a total test time of seven minutes. With Durelon the test was initiated after two minutes and ended after nine minutes. Even though the frozen mixes of the Tenacin were mixed for only one minute instead of 1½ minutes for the room temperature mixes, the initiation of the test (three minutes) and the ending (10 minutes) remained the same as specified in Specification #8.

Once the consistency of the three test cements was determined at room temperature, by trial and error using different powder to liquid ratios, the same disc diameter was determined for each frozen mix. See Table 5.

The Specification #8 consistency test was run with Tenacin mixed on the Caruso slab and slumped on a room temperature slab to see if the different temperatures of frozen mixing surface and the room temperature testing surface had any effect on the consistency test. The same test was run by mixing on a room temperature glass slab and slump testing on a frozen glass slab to see if the condensation on the frozen glass test slab had an effect on the cement during the consistency test.

Working time was defined as the time from the initiation of mix until the mix was unusable. An estimate of the working time of the three test cements mixed at room temperature and on the frozen slabs was made by filling orthodontic bands at one minute intervals until the cement mix was judged unusable. The three cements were then slump tested as outlined previously with one exception, the 20 g glass plate and the 100 gm weight were placed to initiate the test at the estimated working time. By trial and error working time was established as the time when initiation of the slump test resulted in a disc diameter 90% of the standard consistency. This test for working time estimation will be called the Mitchem test, because of its suggested use by Dr. J. C. Mitchem, Department of Dental Materials, University of Oregon Health Sciences Center.

The setting time of cements was determined as specified in

A.D.A. Specifications #8 and #9, with the exception that the setting time was reported to the nearest 30 sec.

The compressive strength and solubility and disintegration tests were conducted as described in A.D.A. Specifications #8 and #9, with the following exceptions: Fluoro-thin was placed in 37° C, 100% humidity oven 2½ minutes after initiation of spatulation. Durelon was placed in the oven two minutes after initiation of spatulation.

## RESULTS AND DISCUSSION

The consistency test diameters for room temperature mixes of the three test cements are shown in Table 6. The disc diameter of 28.80 mm for zinc phosphate in this test is higher than the 26.5 ± 1 mm diameter established by the Cameron et al[11], but less than the 30.0 ± 1 mm diameter in A.D.A. Spec. #8 test. Cameron used a Tenacin powder to liquid ratio of 1.8 g/0.5 ml whereas in this study only 1.3 g/0.5 ml were used. The mean disc diameter for silicophosphate of 22.85 mm for this test is less than the 25 ± 1 mm disc diameter specified in A.D.A. Spec. #21. Cameron mixed Kyptex at a P/L ratio of 1.4 g/0.4 ml to obtain a disc of 26.5 ± 1 mm in diameter, whereas in this test only 1.1 g/0.4 ml of Fluoro-thin was used to obtain the 22.85 mm disc. Durelon was mixed at the P/L ratio of 0.6 g/0.55 ml. Rich et al[21] in their study of cement retention as related to orthodontics mixed Durelon at 0.40 g/0.5 ml. The mean disc diameter of Durelon mixed at the powder to liquid ratio employed in this test was 32.47 mm.

The standard consistencies for the cements mixed on the various test surfaces are shown in Table 7. The F value and t values in Table 7 indicate there is no significant difference in the disc diameters of each individual cement mixed at room temperature and under frozen conditions. There was found to be a greater variance for Tenacin mixed on the frozen glass slab. This result could be due to varying amounts of moisture condensation being incorporated into the frozen glass test mixes.

Table 8 lists the P/L ratios of the test mixes. By mixing Tenacin on a frozen glass slab the amount of powder incorporated into the mix could be increased 71% over the room temperature mix. The amount of powder in the mix could be increased 48% when the Caruso slab was used as the mixing surface. Henschel[2] and Jendresen[3] were able to increase the amount of powder when mixing zinc phosphate cement on a $40^{\circ}$ F slab 30-40%. Kendzior et al[5] was able to incorporate 49-70% more powder when mixing at $6^{\circ}$ C 63-91% more powder when the mixing surface temperature was $-10^{\circ}$ C. The increases in the powder to liquid ratios for frozen mixes in this test are similar to those reported by other experimenters. Fluoro-thin mixed on the Caruso slab allowed the incorporation of 36% more powder. Garman and Morris[9] report doubling the amount of powder in a frozen mix of Durelon mixed to a basing consistency.

An attempt was made to mix Fluoro-thin on a frozen glass slab. It was found that the powder to liquid ratio can only be increased to about 1.7 g/0.4 ml and still allow the Fluoro-thin powder particles to be wetted. The disc diameter in the consistency test for a 1.7 g/0.4 ml powder to liquid mix of Fluoro-thin, mixed on a frozen glass slab, was much greater than the 22.85 mm disc diameter of Fluoro-thin mixed at room temperature. The conclusion was made that Fluoro-thin could not be mixed on a frozen glass slab as utilized in this experiment and attain the same consistency as the manufacturer's recommended room temperature mix. The mixing of Durelon was also attempted on the frozen glass slab. The Durelon liquid froze on the slab. The conclusion was made that Durelon cement could not be mixed on a frozen glass slab with frost on the surface.

Table 9 also shows the results of the consistency tests of

Tenacin mixed on a Caruso slab and consistency tested on a frozen glass slab compared to Tenacin mixed on a Caruso slab and tested on a room temperature slab. The t value indicates there was a significant difference in the two test diameters. Jendresen[3] and Kendzior et al[5] mixed their frozen mixes to the same consistency as a control room temperature mix. How the consistency was tested was not stated. Moon and Modjeski[15] propose that the string test is not an adequate way to test cement consistency. The results of this experimental work indicate that a frozen mix of zinc phosphate cement should not be consistency tested on a room temperature glass slab.

Table 9 also shows the results of the consistency tests of Tenacin mixed on a room temperature glass slab and consistency tested on a room temperature slab compared to Tenacin mixed at room temperature and tested on a frozen glass slab. The t value indicates there was no significant difference in the two test diameters; therefore, it appears that moisture condensation on a frozen glass test slab does not dilute the frozen mix of zinc phosphate cement causing a larger disc diameter in the consistency test.

Table 10 shows the working times of the test cements estimated by filling orthodontic bands until the cements were judged unusable. Table 11 shows the working times estimated by the Mitchem test. The estimate of the working time by filling orthodontic bands with room temperature mixes of Tenacin and Durelon test cements was the same as the working time estimated by the Mitchem test. The clinical estimate of the working times of Fluoro-thin was different by two minutes than that time determined by the Mitchem test. Plant et al[23] using the oscillating rheometer estimated the working time of zinc phosphate cement mixed at room temperature to be 7½ minutes. Kendzior et al[5] estimated the working time of Flecks $ZnPO_4$ cement mixed at room temperature to the Cameron orthodontic consistency to be two minutes. The working time determined in this test was about the median of these. Plant estimated the working time of a similar mix of Durelon to the one utilized in this test as being four minutes. This estimate is in close agreement to the three minute value estimated by both the band filling test and Mitchem test.

The working time for frozen slab mixes estimated by filling bands was significantly different than the time estimated by the Mitchem test. As can be seen in Tables 10 and 11 the difference was two minutes for Tenacin, eight minutes for Durelon and nine minutes for Fluoro-thin. The difference in the working time estimated by the two procedures might be attributed to the following:

1. Subjectivity of the band filling estimate.
2. Warming of the frozen glass consistency test slab to which the test cement was immediately transferred after mixing, as opposed to the maintenance of a cooler surface temperature of the Caruso slab on which the ban filling mix was maintained. Figure 1 shows the temperature vs. time curves for the two slabs.
3. Lack of correlation between the two test procedures.

Tables 10 and 11 show the percent increase in working time for cold slab mixes estimated by the two test procedures. The working time of Tenacin and Fluoro-thin was increased approximately 500%. Jendresen[3] found that the working time of zinc phosphate was doubled when mixed at 45° F. Tuenge[4] increased the working time 400%. Kendzior et al[5] increased the working time 300% at the 6° C slab temperature and 700% for -10° C slab temperature. Garman and Morris[9] report extending the working time of a basing consistency of Durelon to 15 minutes when mixed on a frozen slab. The working times estimated for Durelon in this procedure were 33 and 25 minutes for a mix somewhat thinner than the basing mix of Garman and Morris. No estimate of the working time of a frozen glass mix of Tenacin could be made by utilizing the Mitchem test because of the great amount of disc diameter variance disallowing any test diameter repeatability.

The one sample t values in Table 12 for the Mitchem test disc diameters compared to values of 90%, the standard consistency show no significant difference between the two diameters.

The setting times of the various test mixes in a simulated oral environment and the percent decreases in the setting times of frozen mixes compared to room temperature mixes are shown in Table 13.

The setting time of all the frozen mixes was decreased in the simulated oral environment. Jendresen[3], Tuenge[4], and Kendzior et al[5] all found that frozen mixes of zinc phosphate cement had a decreased setting time. Garman and Morris[9] found a decrease in the setting time for Durelon mixed on a frozen slab. Tenacin mixed on the frozen glass slab, with the largest powder to liquid increase of all the frozen mixes, had the greatest decrease in setting time. Jendresen[3] found the setting time of his frozen mixes to be decreased 30-50%. Tuenge[4] found a decreased setting time of 50%; Kendzior et al[5] found a setting time decrease of approximately 17-43% for the three cements tested. The setting time for the frozen mixes of zinc phosphate cement in this test was well within the range determined by previous experimenters. Garman and Morris found a reduction in the setting time of Durelon mixed on a frozen slab from 32-64%. The 15% reduction in setting time of Durelon in this test was less than the Garman and Morris time. The estimated setting time of 13 minutes in this test for the room temperature mix of Tenacin was more than Kendzior et al[5] and Plant et al[23]. Kendzior et al[5] estimated the setting time of zinc phosphate cement mixed at room temperature to range from approximately 5½-7½ minutes. Plant et al[23] using the oscillating rheometer estimated the setting time of $ZnPO_4$ cement mixed at room temperature to be 9 3/4 minutes. Kendzior et al used the Cameron consistency mix which incorporated a higher powder to liquid ratio than used in this experiment. The higher P/L ratio resulted in a shorter setting time. Plant et al used a powder to liquid ratio similar to that used in this experiment. Plant et al, using the same P/L ratio for Durelon as used for the room temperature mix in this experiment, estimated setting time to be 6½ minutes; whereas the estimate in this experiment was 10 minutes.

Table 14 lists the crushing force of all the cement test specimens in this experiment. Table 15 shows the sample size, mean, variance, standard deviation, and standard error of the measure of the crushing force of the cement mixes. Table 16 shows the crushing force converted to pounds per square inch and kilograms per square centimeters. The comparison $F^2_9 = 17.2652$, of the crushing force of the three types of test mixes of Tenacin indicates there is a significant difference in the crushing strength of the mixes. By inspection of the mean crushing strengths of the three mixes of Tenacin, it is clear that Tenacin mixed on a frozen glass slab has a crushing strength less than the other two mixes. Jendresen[3] and Kendzior et al[5] found no difference in the compressive strengths of zinc phosphate cement mixed at room temperature and on a frozen glass slab. Tuenge[4] reported a lower compressive strength for his frozen glass mixes of zinc phosphate; however, the compressive strength increased as P/L ratio increased on the frozen slab until Tuenge's greatest powder to liquid ratio of 2.8 g/0.5 ml had a compressive strength equal to the room temperature mix. Frozen glass was dropped from future testing of crushing strengths. The crushing strengths of the three cements mixed on both room temperature and Caruso slabs were compared. $F^2_{18} = 92.1791$, indicates there is a significant difference in the crushing strength of the three cements. By inspection of the mean crushing strength values, it can be seen that the compressive strength of Durelon is less than the other cements. The test, $F^{1}_{18} = .5371$, of crushing strengths obtained from frozen slab mixes compared to strengths of room temperature mixes, shows that the type of mixing surface does not alter the compressive strength.

The least significant difference (L.S.D.) between means equals 54.7224. A difference in mean crushing force values greater than 54.7224 lbs. indicates a significant difference in compressive strengths. A look at Table 15 shows Tenacin and Fluoro-thin room temperature and Caruso frozen mixes are clearly more than 54.7224 lbs. greater than the respective Durelon mixes; therefore making them significantly stronger than the Durelon mixes. Grieve[29] found carboxylate cements to be clearly inferior to zinc phosphate in compressive strength. Phillips et al[25] found zinc phosphate cements to be twice as strong as polycarboxylate cements. The inferior compressive strength of polycarboxylate was confirmed in this experiment. Using the L.S.D. between means it can be determined that Fluoro-thin mixed at room temperature is stronger than Tenacin mixed at both room temperature and on the Caruso slab; however, Fluoro-thin mixed on the frozen slab was not stronger in compressive strength than the Caruso mix of Tenacin. The frozen mix of Fluero-thin was stronger than the Tenacin room temperature mix. This strength difference is a very borderline situation and may in actuality not be of significance. Something that is of significance is the fact that the experimentally determined compressive strength for Fluoro-thin of approximately 850 Kg/cm$^2$ for both frozen and room temperature mixes listed in Table 16, does not satisfy the compressive strength requirement of 1400 Kg/cm$^2$ for silicophosphate in Specification #21. Anderson and Paffenbarger[27] report that silicophosphate cements are stronger than zinc phosphate cements. Williams et al[17] found the compressive strengths of silicophosphate cement to be twice that of zinc phosphate. These results were not duplicated by Fluoro-thin.

A test was run to determine if the difference in the crushing strengths of the three types of cements was altered by the mixing surface. The F value, $F^2_{18} = 0.6297$, indicates the differences between the compressive strength of three cements remained constant on both the room temperature and the Caruso slabs.

Table 17 lists the solubilities of the test cements as percent by weight. The F value in comparing the solubilities of the frozen and room temperature Tenacin mixes indicates there is no significant difference in their solubilities. Jendresen[3] and Kendzior et al[5] report no difference in solubility between cold slab mixes and room temperature mixes of zinc phosphate. Tuenge[4] reports that frozen slab mixes had a greater solubility than room temperature mixes of zinc phosphate.

The t value comparing frozen and room temperature mixes of Fluoro-thin indicates that there is no significant difference in the solubilities of the two mixes. The t value comparing Durelon frozen and room temperature mixes shows that there is a significant difference in the solubilities of the two mixes. This solubility difference could be due to the following:

1. Incomplete wetting of all the powder particles due to the

apparent increase in viscosity of the Durelon liquid when placed on the frozen slab, making mixing more difficult in the 30 second mixing period.

2. A frozen Durelon solubility test disc that appeared to have more porosity than the room temperature disc thus creating a greater surface area on which the distilled water could act during the 24 hour test.
3. Effect of moisture on the frozen mixing surface.

No attempt was made to compare one cement to another by means of the 24 hour solubility test because Wilson et al[32] and Norman et al[33] felt the solubility-disintegration test is a useful criteria for comparing the same type of cement, but has no value in comparing the relative merits of different types of cement.

Sadowsky and Retief[38] found the solubility of zinc phosphate mixed at room temperature to be 0.1% by weight. In this study the solubility was 0.06% by weight. They found the solubility of silicophosphate to be 0.8% by weight. The fluoro-thin solubility om the study was 0.6%. Anderson and Paffenbarger[27] found the solubility of zinc phosphate in distilled water 1/10th that of silicophosphate. The same result was obtained in this study. Phillips et al[25] and Rich et al[2] found the solubility of polycarboxylate cement mixed at room temperature to be equal to zinc phosphate. The same finding was made in this study. These comparisons are only made to substantiate the validity of the test results.

# SUMMARY AND CONCLUSIONS

1. Fluoro-thin can be mixed on a frozen slab without changing its compressive strength and solubility as determined by the A.D.A. Specification tests.
2. The compressive strength of Durelon is not changed when mixed on a frozen slab; however, the solubility is increased.
3. The compressive strength of Fluoro-thin as determined in these tests does not fulfill the requirements of A.D.A. Specification #21.
4. Fluoro-thin and Durelon should not be mixed on a frozen slab with frost present.
5. The powder to liquid ratio of Fluoro-thin and Durelon can be increased when mixed on a frozen slab; however, the percent increase is not as great as with zinc phosphate mixed on a frozen slab.
6. The temperature of the test surface in the A.D.A. Specification test for consistency should be the same as the surface

temperature of the mixing slab.

7. The working time of Fluoro-thin and Durelon can be greatly extended when the cements are mixed on a frozen slab.
8. The Mitchem Working Time Test could be a viable means of standardizing the working time testing of cement materials.
9. The setting time of Fluoro-thin and Durelon is decreased when the cements are mixed on a frozen slab and tested in a $37^{\circ}$ C and 100% humidity environment.

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## TABLE 1

## CEMENT MIXING SURFACES

| | Room temp. glass | Frozen glass | Caruso | Durelon pad |
|---|---|---|---|---|
| Tenacin | X | X | X | |
| Fluoro-thin | X | | X | |
| Durelon | | | X | X |

## TABLE 2

## MIXING SLAB TEMPERATURES

| | Temperature |
|---|---|
| Room temp. glass and Durelon pad | 22° C |
| Frozen glass | ∼24° C |
| Caruso | 25° F or -4° C |

## TABLE 3

## POWDER TO LIQUID RATIO ROOM TEMPERATURE MIXES

| | |
|---|---|
| Tenacin | 1.3 g/0.5 ml |
| Fluoro-thin | 1.1 g/0.4 ml |
| Durelon | 0.6 g/0.55 ml or 0.51 g |

TABLE 4

MIXING TIME

| | Room temp. glass | Frozen glass | Caruso | Durelon pad |
|---|---|---|---|---|
| Tenacin | 90 sec. | 60 sec. | | |
| Fluoro-thin | 60 sec. | | 60 sec. | |
| Durelon | | | 30 sec. | 30 sec. |

## TABLE 5

## POWDER TO LIQUID RATIO FROZEN MIXES

| | |
|---|---|
| Tenacin | |
| Frozen glass | 2.225 g/0.5 ml |
| Caruso | 1.925 g/0.5 ml |
| Fluoro-thin | 1.5 g/0.4 ml |
| Durelon | 0.8 g/0.55 ml or 0.51 g |

TABLE 6

ROOM TEMPERATURE CONSISTENCY TEST (MILIMETERS)

| | P/L Ratio | Mean disc diam. | $S^2$ | S.D. | SEM | n |
|---|---|---|---|---|---|---|
| Tenacin | 1.3 g/0.5 ml | 28.80 | 0.2925 | 0.5408 | 0.3122 | 3 |
| Fluoro-thin | 1.1 g/0.4 ml | 22.85 | 0.0475 | 0.2179 | 0.1258 | 3 |
| Durelon | 0.6 g/0.55 ml<br>or 0.51 g | 32.47 | 0.1233 | 0.3511 | 0.2027 | 3 |

## TABLE 7

## STANDARD CONSISTENCY TEST (MILIMETERS)

|  | n | Mean | $S^2$ | S.D. | SEM |
|---|---|---|---|---|---|
| Tenacin |  |  |  |  |  |
| room temp. | 3 | 28.80 | 0.2929 | 0.5408 | 0.3122 |
| frozen glass | 7 | 28.73 | 8.2190 | 2.8669 | 1.0836 |
| Caruso | 4 | 29.38 | 0.6225 | 0.7890 | 0.3945 |
| Fluoro-thin |  |  |  |  |  |
| room temp. | 3 | 22.85 | 0.0475 | 0.2179 | 0.1258 |
| Caruso | 3 | 22.78 | 0.1408 | 0.3753 | 0.2167 |
| Durelon |  |  |  |  |  |
| room temp. | 3 | 32.47 | 0.1233 | 0.3511 | 0.2027 |
| Caruso | 3 | 33.13 | 0.9233 | 0.9609 | 0.5548 |

$F^{2}_{11}$ value P = 0.05 t value 1.1174 P = 0.05
0.1173 0.2794 P = 0.05

TABLE 8

POWDER TO LIQUID RATIOS (GRAMS PER MILILITER)

| | Room temp. | Frozen glass | Caruso | %powder increase-cold mixes |
|---|---|---|---|---|
| Tenacin | 1.3/0.5 | 2.225/0.5 | 1.925/0.5 | 71 and 48% |
| Fluoro-thin | 1.1/0.4 | | 1.5/0.4 | 36% |
| Durelon | 0.6/0.55 | | 0.8/0.55 | 33% |

## TABLE 9

## CONSISTENCY TEST

(testing surface at a different temperature than mixing surface, milimeters)

| | Mean disc diam. | $S^2$ | S.D. | S.E.M. | n | t value P = 0.05 |
|---|---|---|---|---|---|---|
| acin | | | | | | |
| mixed on Caruso slab, tested on frozen glass | 29,38 | 0.6225 | 0.7890 | 0.3945 | 4 | 8.4312 |
| mixed on Caruso, tested on room temp. glass slab | 23.85 | 0.9100 | 0.9539 | 0.5508 | 3 | |
| mixed on room temp. glass tested on room temp. glass | 28.80 | 0.2925 | 0.5408 | 0.3122 | 3 | 0.3160 |
| mixed on room temp. glass tested on frozen glass | 28.47 | 2.9208 | 1.7090 | 0.7643 | 5 | |

## TABLE 10

### WORKING TIME (BY FILLING BANDS)

| | Room Temp. | Caruso | % Increase |
|---|---|---|---|
| Tenacin | 4 minutes | 23 minutes | 575% |
| Fluoro-thin | 6 minutes | 30 minutes | 500% |
| Durelon | 3 minutes | 33 minutes | 1100% |

# TABLE 11

## WORKING TIME (MITCHEM TEST)

| | Room temp. | Caruso | % Increase |
|---|---|---|---|
| Tenacin | 4 minutes | 21 minutes | 525% |
| Fluoro-thin | 4 minutes | 21 minutes | 525% |
| Durelon | 3 minutes | 25 minutes | 833 1/3% |

TABLE 12

MITCHEM WORKING TIME TEST VS. STANDARD CONSISTENCY TEST

| | Disc Dia. mm. | 90% Stand. Consis. | Working Time Min. | u mm. | S | S.D. | SEM | t value p = 0.05 | n |
|---|---|---|---|---|---|---|---|---|---|
| in | | | | | | | | | |
| temp. | 28.80 | 25.92 | 4 | 25.95 | 0.4375 | 0.6614 | 0.3819 | .0786 | 3 |
| iso | 29.38 | 26.44 | 21 | 26.28 | 0.4258 | 0.6526 | 0.3768 | .4246 | 3 |
| o-thin | | | | | | | | | |
| temp. | 22.85 | 20.57 | 4 | 20.27 | 0.0658 | 0.2566 | 0.1481 | 2.0257 | 3 |
| iso | 22.78 | 20.50 | 21 | 19.87 | 0.6808 | 0.8251 | 0.4764 | 1.3224 | 3 |
| on | | | | | | | | | |
| temp. | 32.47 | 29.22 | 3 | 29.33 | 0.2808 | 0.5299 | 0.3060 | 0.3595 | 3 |
| iso | 33.13 | 29.82 | 25 | 30.07 | 0.1633 | 0.4041 | 0.2333 | 1.0716 | 3 |

TABLE 13

SETTING TIME (MINUTES)

| | Room temp. | Frozen glass | Caruso | Percent Decrease |
|---|---|---|---|---|
| Tenacin | 13 | 7½ | 8½ | 42 and 35% |
| Fluoro-thin | 10½ | | 6½ | 28% |
| Durelon | 10 | | 8½ | 15% |

## TABLE 14

### CRUSHING FORCE (POUNDS)

| | Room temperature | Frozen glass | Caruso |
|---|---|---|---|
| Tenacin | 532 | 330 | 495 |
| | 410 | 276 | 428 |
| | 478 | 332 | 444 |
| | 494 | 326 | 526 |
| Fluoro-thin | 472 | | 525 |
| | 572 | | 503 |
| | 527 | | 547 |
| | 544 | | 510 |
| Durelon | 239 | | 330 |
| | 268 | | 317 |
| | 300 | | 280 |
| | 271 | | 284 |

## TABLE 15

### CRUSHING FORCE (POUNDS)

| | n | u | $S^2$ | S.D. | SEM |
|---|---|---|---|---|---|
| Tenacin | | | | | |
| Room temp. | 4 | 466.00 | 2,706.666 | 52.025 | 26.012 |
| Frozen glass | 4 | 316.00 | 717.333 | 26.783 | 13.391 |
| Caruso | 4 | 473.25 | 2,052.916 | 45.309 | 22.654 |
| Fluoro-thin | | | | | |
| Room temp. | 4 | 528.75 | 1,775.583 | 42.137 | 21.068 |
| Caruso | 4 | 521.25 | 378.916 | 19.465 | 9.732 |
| Durelon | | | | | |
| Room temp. | 4 | 269.50 | 621.666 | 24.933 | 12.466 |
| Caruso | 4 | 302.75 | 604.916 | 24.595 | 12.297 |

TABLE 16

COMPRESSIVE STRENGTH

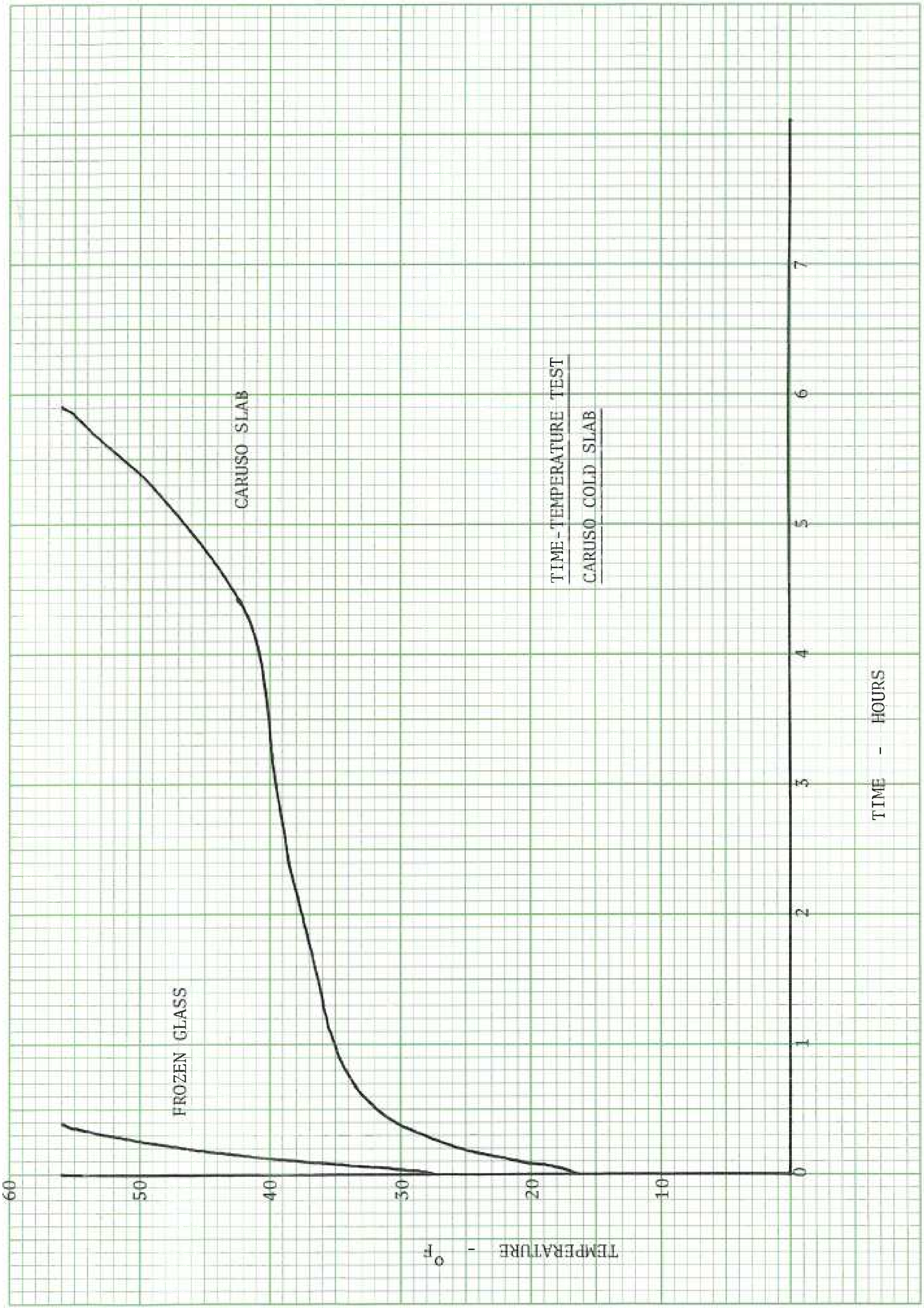
| | Psi | $Kg/cm^2$ |
|---|---|---|
| Tenacin | | |
| room temp. | 10,639 | 749 |
| frozen glass | 7,215 | 508 |
| Caruso | 10,805 | 760 |
| Silicophosphate | | |
| room temp. | 12,072 | 849 |
| Caruso | 11,901 | 837 |
| Durelon | | |
| room temp. | 6,153 | 433 |
| Caruso | 6,912 | 486 |

TABLE 17

24-HOUR SOLUBILITY IN DISTILLED WATER (PERCENT BY WEIGHT)

| | n | u | $S^2$ | S.D. | SEM | |
|---|---|---|---|---|---|---|
| in | | | | | | |
| m temp. | 2 | .0558 | .00070 | .02638 | .01865 | $F_3^2$ value p = 0.05<br>4.000 |
| zen glass | 2 | .0121 | .00002 | .00403 | .00285 | |
| uso | 2 | .0148 | .00002 | .00495 | .00350 | |
| o-thin | | | | | | |
| m temp. | 2 | .6364 | .00249 | .04992 | .03530 | t value p = 0.05<br>4.0197 |
| uso | 2 | .4394 | .00231 | .04808 | .03400 | |
| on | | | | | | |
| m temp. | 2 | .0601 | .00005 | .00226 | .00160 | 16.8025 |
| uso | 2 | .0937 | .00003 | .00170 | .00120 | |

FIGURE 1


TIME-TEMPERATURE TEST
CARUSO COLD SLAB
CARUSO SLAB
FROZEN GLASS
TIME - HOURS
0
1
2
3
4
5
6
7
TEMPERATURE - °F
60
50
40
30
20
10