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Use of a Portland Cement Accelerator with Mineral Trioxide Aggregate

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USE OF A PORTLAND CEMENT ACCELERATOR WITH MINERAL TRIOXIDE AGGREGATE.

A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at Virginia Commonwealth University.

by

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Abstract

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The use of Mineral Trioxide Aggregate (MTA) is gaining popularity among clinicians. Despite the many ideal qualities it possesses, it is often difficult to manipulate and often requires a second appointment for placement of a restoration to allow for setting. If the time to set of MTA can be accelerated to a single appointment time frame without significantly altering its properties, then MTA may gain even wider acceptance. The purpose of this study is to identify the percentage of a Portland Cement Accelerator (PCA), that when added to MTA, will decrease the time to set of MTA towards a single appointment time frame. Ten Teflon sample molds were prepared to hold 20 standardized

chambers in each. Three sample molds were prepared with a 5.0% (by weight of MTA) accelerator, 3 with 10.0% accelerator and 3 with 15.0% accelerator mixed with MTA and water. Another sample mold contained a mixture of MTA and water only and acted as the control. Samples were tested using a dial indicator microgauge apparatus that measured the depth of needle penetration starting at 2 minutes and then every minute up to 15 minutes. Samples were also tested at 3, 4, 24, 48 and 72 hours. A mixed-model repeated measures ANOVA showed the four accelerator groups were significantly different and there was a significant time trend. The 5.0% accelerator group set significantly faster compared to the 15.0% and the control at 15 minutes or less ($p<0.05$). In conclusion, it appears that 5.0% PCA when added to MTA can accelerate the setting reaction.

Introduction

Since its introduction in 1993, MTA (mineral trioxide aggregate), has been gaining popularity among clinicians based on its excellent clinical properties. MTA fulfills many of the requirements of an ideal root-end filling and repair material (18). However, its four hour initial setting time necessitates a second appointment for a final restoration since the material is easily washed out during procedures that require irrigation or rinsing (11, 22). MTA's initial fluidity also makes final irrigation of the surgical site difficult to impossible in many cases. The slow setting time also makes handling difficult during placement (22). Any ability to gain initial setting times within the time frame of a single dental appointment would enhance the clinical usefulness of MTA.

One study showed that MTA was capable of setting from the moisture it received from a simulated PDL material and that a moist cotton pellet was not necessary for full setting of the material (1). This would be important if MTA were immediately covered with a permanent restoration. In addition to being primarily a repair material, it is also showing better clinical and histological results as a pulp-capping and pulpotomy agent (16). MTA is difficult to use after pulp-capping or a pulpotomy when a resin bonded permanent restoration is desired directly in contact with the MTA.

Recently, several studies have been undertaken to compare various aspects of MTA with those of Portland cement (PC) including composition, healing ability, antimicrobial activity, connective tissue reactivity, sealing ability, and bone reactivity (2, 4, 5-7, 15, 19).

In 2000, Estrela et al., and again in 2003, Funteas et al. showed that MTA and PC share the same chemical elements except that MTA also contains bismuth (2, 23). Holland et al., demonstrated similar results between MTA and PC when used as a pulp capping agent in dogs. Both MTA and PC had nearly complete tubular hard tissue bridge formation in almost all specimens demonstrating similar healing ability. Saidon et al., used cell and histological studies to compare MTA to PC. They found that both materials were well tolerated and exhibited healing with minimal inflammation. Given the similarity between these two materials, the question is raised whether Portland cement additives are interchangeable with MTA.

Use of an accelerated Portland cement (APC) has been evaluated recently to determine its cytotoxicity and healing ability. Both APC variants tested were non-toxic and showed potential for bone healing (3). To date, no studies have been published using a set accelerator with MTA.

Given the popularity of MTA in clinical usage, it is the aim of this study to determine the appropriate percentage of a PCA that, when added to MTA, will accelerate the setting reaction towards a single appointment time frame.

Material and Methods

Ten Teflon molds were used to standardize the testing of these materials. Each round mold had 20 identical 3 mm deep by 3 mm wide chambers. The molds were cleaned ultrasonically and rinsed with double deionized water prior to use. Four main groups were used to compare setting times. Experimental groups 1, 2 and 3 were prepared as MTA/accelerator/distilled water (12) with accelerator amounts (by weight of MTA) at 5.0%, 10.0% and 15.0% respectively. Group 4 was the negative control with MTA and distilled water only. Each group was repeated three times except for the control group. Accelerator amounts were based on a pilot study showing the approximate amounts needed to gain initial set within the required working times. In the experimental groups, 1.5 g of MTA was mixed with 0.3 ml of distilled water and the corresponding percentage by weight of accelerator. The same amount of water was used on all samples to standardize the viscosity of the mixture and all samples were mixed according to the manufacturer's directions.

After thorough mixing, sample material was transferred to Centrix Separate clear tubes with plugs to prevent dehydration and tubes were loaded in a C-R e/z syringe (Centrix, Shelton, CT). Samples were immediately transferred to the Teflon sample molds using the C-R e/z syringe to minimize voids. Once placed, the surface of the material was planed flush with the surface of the sample holders using a # 11 surgical blade (20). Total mixing and handling time was 5 minutes from the time mixing was

started until the samples were loaded in the testing apparatus. At that point, the clock was reset to zero and the first measurement commenced at 2 minutes. Samples were stored, when not being tested, under a sponge moistened with distilled water at 37º C. The experiment was performed in a blinded manner with the author performing measurements unaware of which sample was being tested.

Testing of the samples was performed with a dial indicator microgauge (Mitutoyo, MTI Corp., Aurora, Il) starting at minute 2 and every minute thereafter for 13 additional minutes. The dial indicator applies an internal spring loaded force equivalent to approximately 98 g. Samples were then tested at 3 h, 4 h, 24 h, 48 h and 72 h. Before testing, the microgauge needle, 1mm in diameter, was set to the bottom of a well, then raised against the resistance of its internal spring. The needle then rested on a 1 mm thick sloped, plastic ramp over the well to be tested. Prior to each sampling, an initial "start" reading was made. Then at the appropriate time interval, the ramp was slowly pulled to one side allowing the needle to make a gentle and consistent contact with the surface of the material. The needle was allowed to penetrate for 5 seconds (12, 14) at which time a second "end" reading was made. When needle penetration was made, initial set was not recorded for that time. Once a material resisted complete circular indentation by the needle, it was considered at the initial set stage.

Results

It was observed that penetration was initially stable, and then as the material began to set, the penetration progressively decreased. After a certain point, the material had "set" and penetration stabilized to "zero" penetration which is measured at 1mm to account for the thickness of the plastic ramp. Thus the relationship between penetration and time followed a sigmoid curve, which may be described by the following equation:

$$\text{penetration} = \frac{(\text{Max} - \text{Min})}{\left(1 + \left(\frac{\text{Time}}{\text{Median}}\right)^{\text{Slope}}\right)} + \text{Min}$$

Where "Max" is the maximum penetration, "Min" is the minimum penetration, "Time" is measured in minutes, "Median" is the time that results in 50% penetration, and "Slope" is the slope-like parameter for the sigmoidal curve.

The estimated parameters for each of the samples and for the combined samples is shown in Table 1. The summary figure is shown in Figure 11. As may be seen, the 15.0% accelerator samples seem indistinguishable from the control sample. At ten minutes, the 10.0% accelerator had not yet fully set. Surprisingly, all three 5.0% accelerator samples had lower penetrations early on.

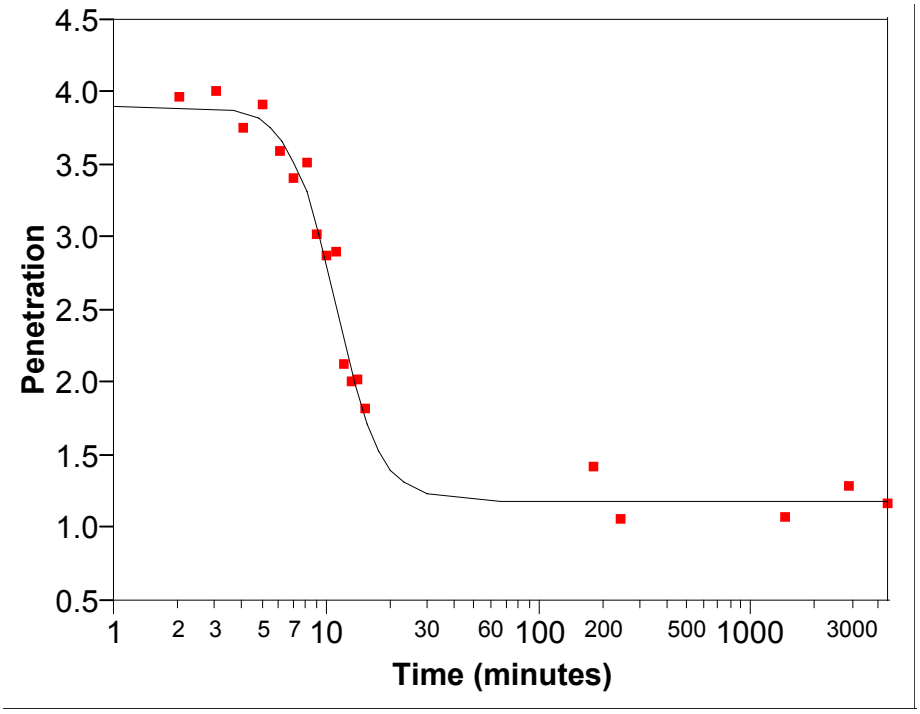
A mixed-model repeated-measures ANOVA of the clinically relevant time periods (15 minutes or less), showed the following results. The four accelerator groups were significantly different ($F(3,6) = 6.48$, $p = 0.0260$) and there was a significant time trend (F

(13,78) = 4.60, p < .0001). Additionally, the test of interaction showed that there was no evidence that the differences between the four accelerator groups varied across time (F (39, 78) = 0.85, p = 0.7025). The LS Mean penetration for each accelerator group are shown in Table 1and Figure12. Tukey's HSD multiple comparison procedure indicated that the 5.0% accelerator was significantly different than the 15.0% accelerator (at alpha = 5%). Also, the uncorrected p-value indicated that the 5.0% accelerator group was significantly different than the control at all times up to 15 minutes (p = 0.0472).

Figure 1

Penetration vs Time with 0% Accelerator

(Sample 4)



Control: Sample 4 was the 0% accelerator, control case. The results are shown in Figure 1. The estimated Max = 3.91 (SE = 0.088), Slope = 4.1 (SE = 0.55), Median = 11.11 (SE = 0.371), Min = 1.19 (SE = 0.07).

Figure 2

Penetration vs Time with 5% Accelerator

(Sample 5)

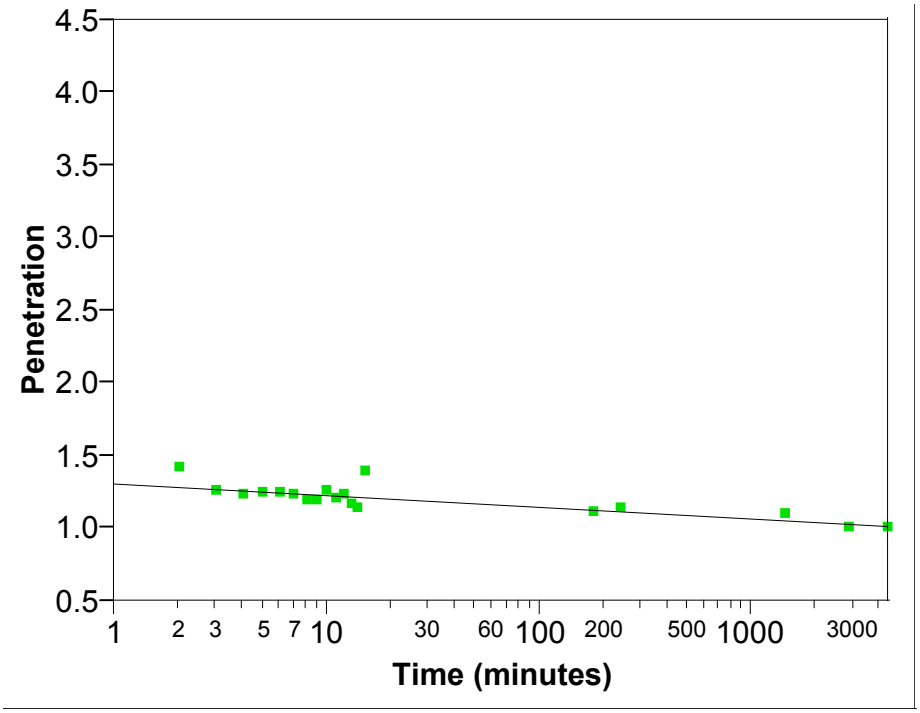


Figure 3

Penetration vs Time with 5% Accelerator

(Sample 6)

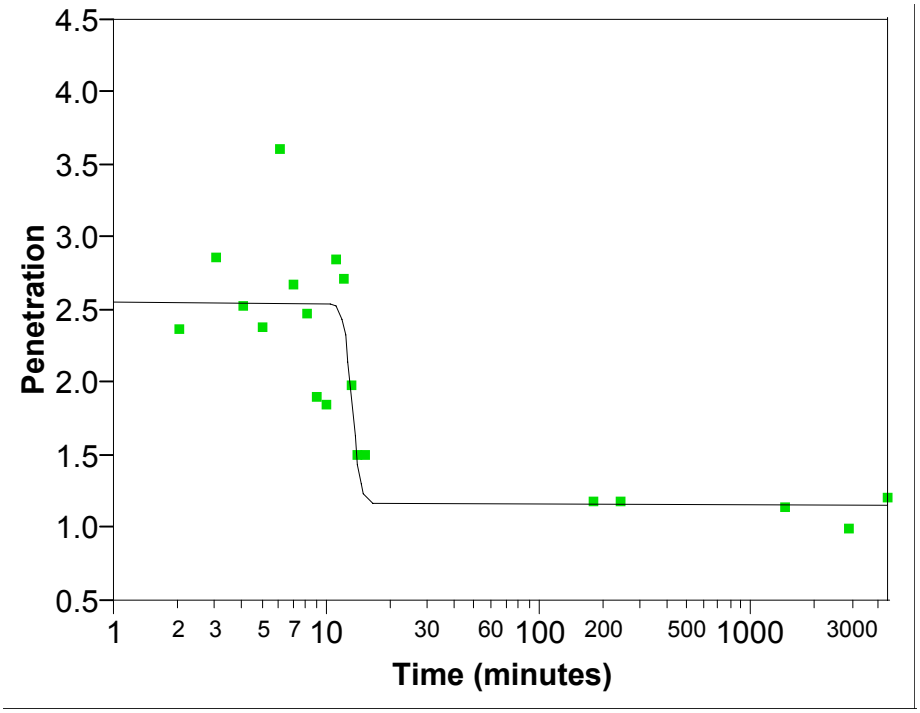
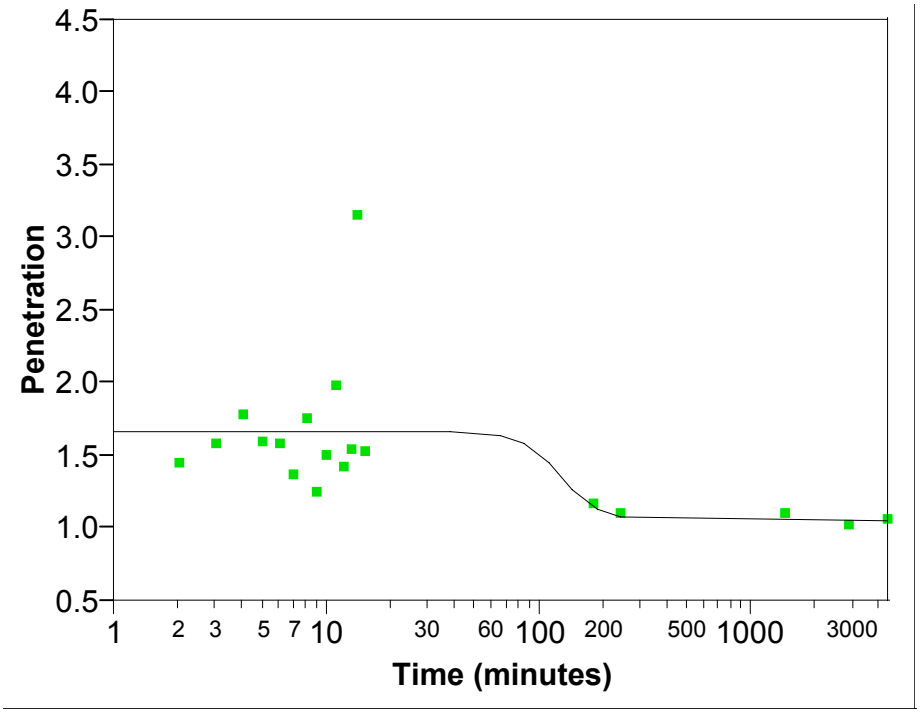


Figure 4

Penetration vs Time with 5% Accelerator

(Sample 9)



Accelerator = 5.0%: Samples 5, 6, and 9 used an accelerator proportion of 0.05. The results are shown in Figure 2, 3 and 4. For sample 5, the estimated Max = 1.4 (SE = 0.339), Slope = 0.34 (SE = 0.26), Median = 50.67 (SE = 184.505), Min = 0.93 (SE = 0.241).For sample 6, the estimated Max = 2.56 (SE = 0.128), Slope = 23.28 (SE = 20.731), Median = 13.39 (SE = 0.601), Min = 1.17 (SE = 0.176). For sample 9, the estimated Max = 1.05 (SE = 0.249), Slope = -4.65 (SE = 54.855), Median = 128.02 (SE = 577.841), Min = 1.67 (SE = 0.115).

Figure 5

Penetration vs Time with 10% Accelerator

(Sample 1)

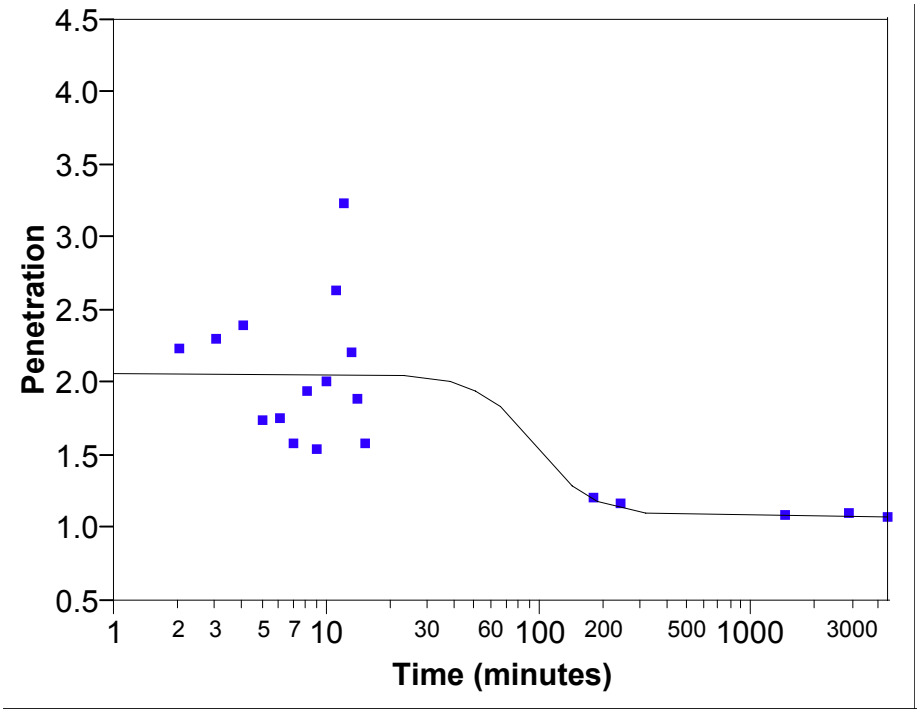


Figure 6

Penetration vs Time with 10% Accelerator

(Sample 4)

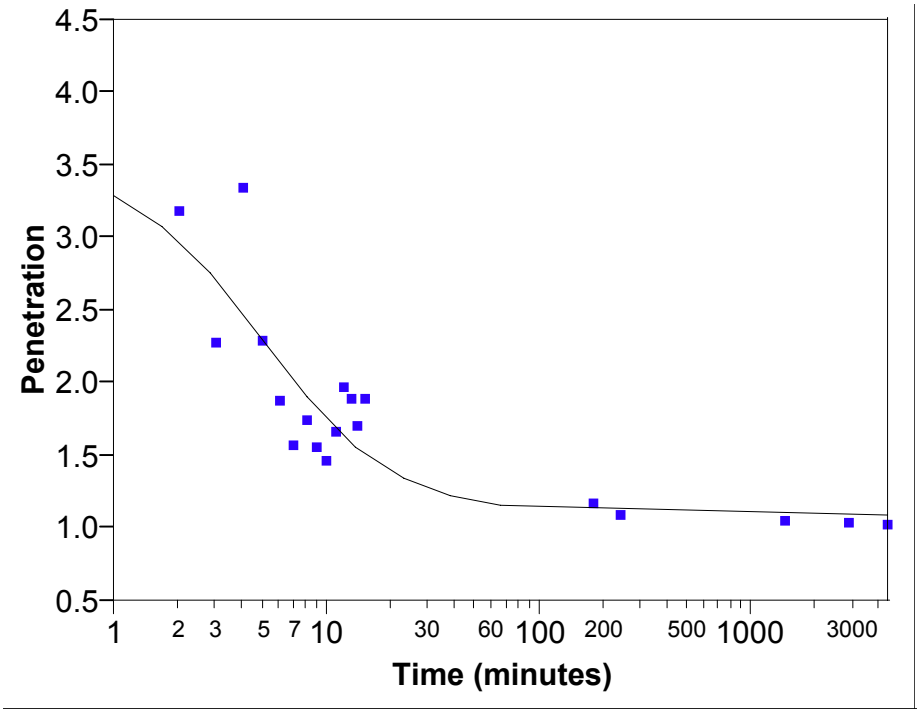
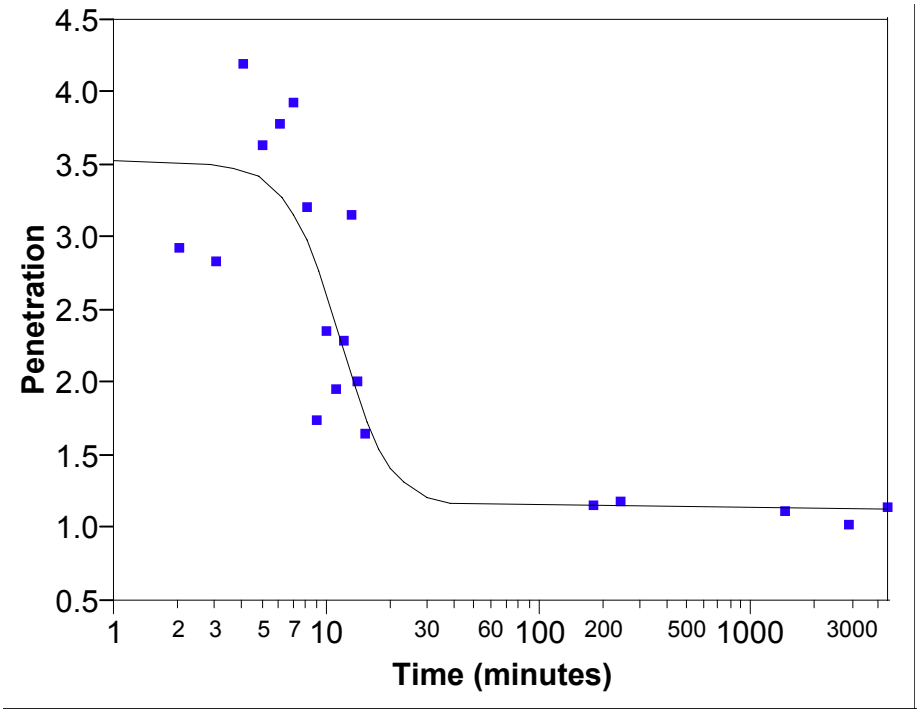


Figure 7

Penetration vs Time with 10% Accelerator

(Sample 8)



Accelerator = 10.0%: Samples 1, 4, and 8 used an accelerator proportion of 0.10. The results are shown in Figure 5, 6 and 7. For sample 1, the estimated Max = 2.07 (SE = 0.136), Slope = 3.13 (SE = 27.531), Median = 97.53 (SE = 578.029), Min = 1.08 (SE = 0.253). For sample 4, the estimated Max = 3.53 (SE = 0.203), Slope = 1.39 (SE = 0.376), Median = 4.9 (SE = 0.893), Min = 1.1 (SE = 0.153). For sample 9, the estimated Max = 1.05 (SE = 0.249), Slope = -4.65 (SE = 54.855), Median = 128.02 (SE = 577.841), Min = 1.67 (SE = 0.115).

Figure 8

Penetration vs Time with 15% Accelerator

(Sample 2)

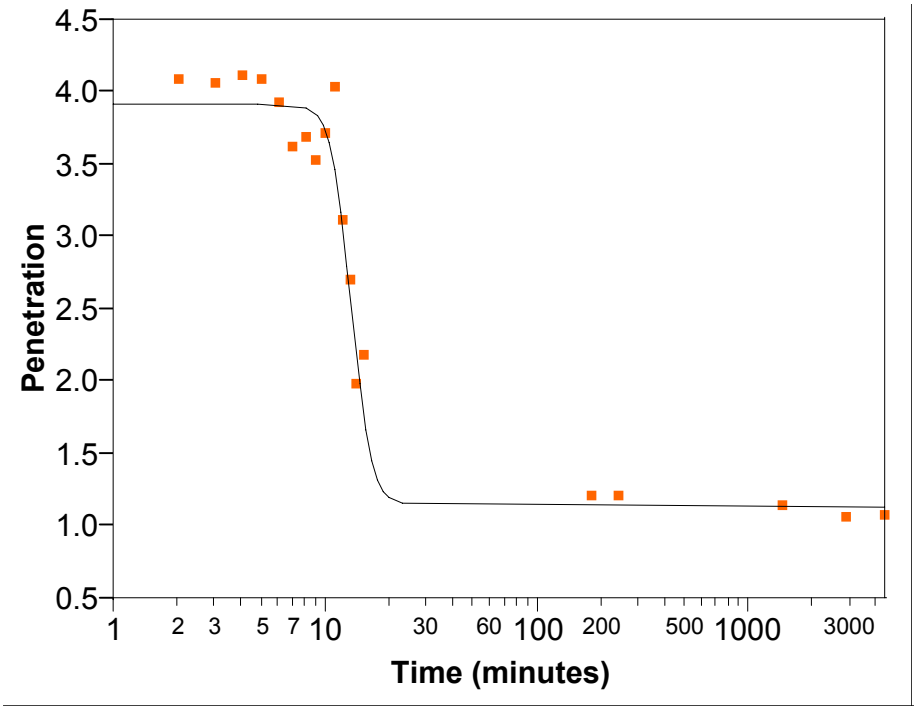


Figure 9

Penetration vs Time with 15% Accelerator

(Sample 7)

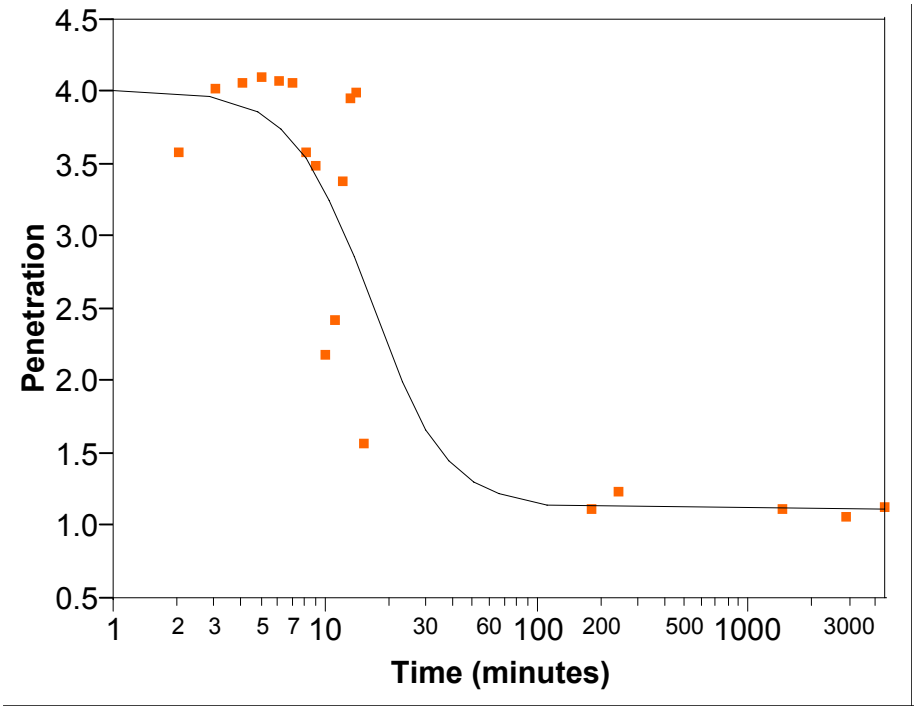
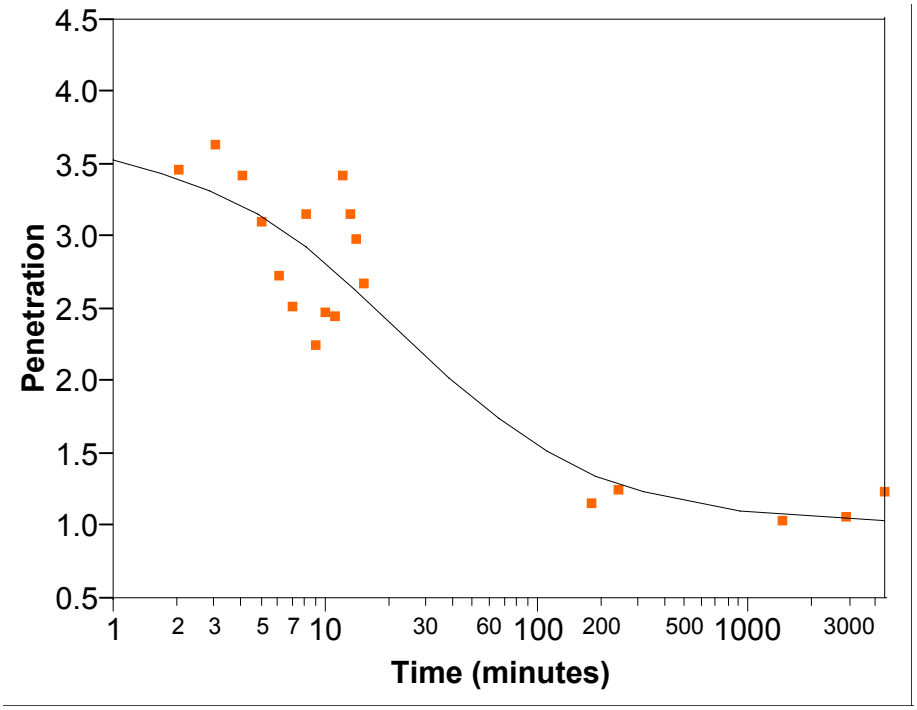


Figure 10

Penetration vs Time with 15% Accelerator

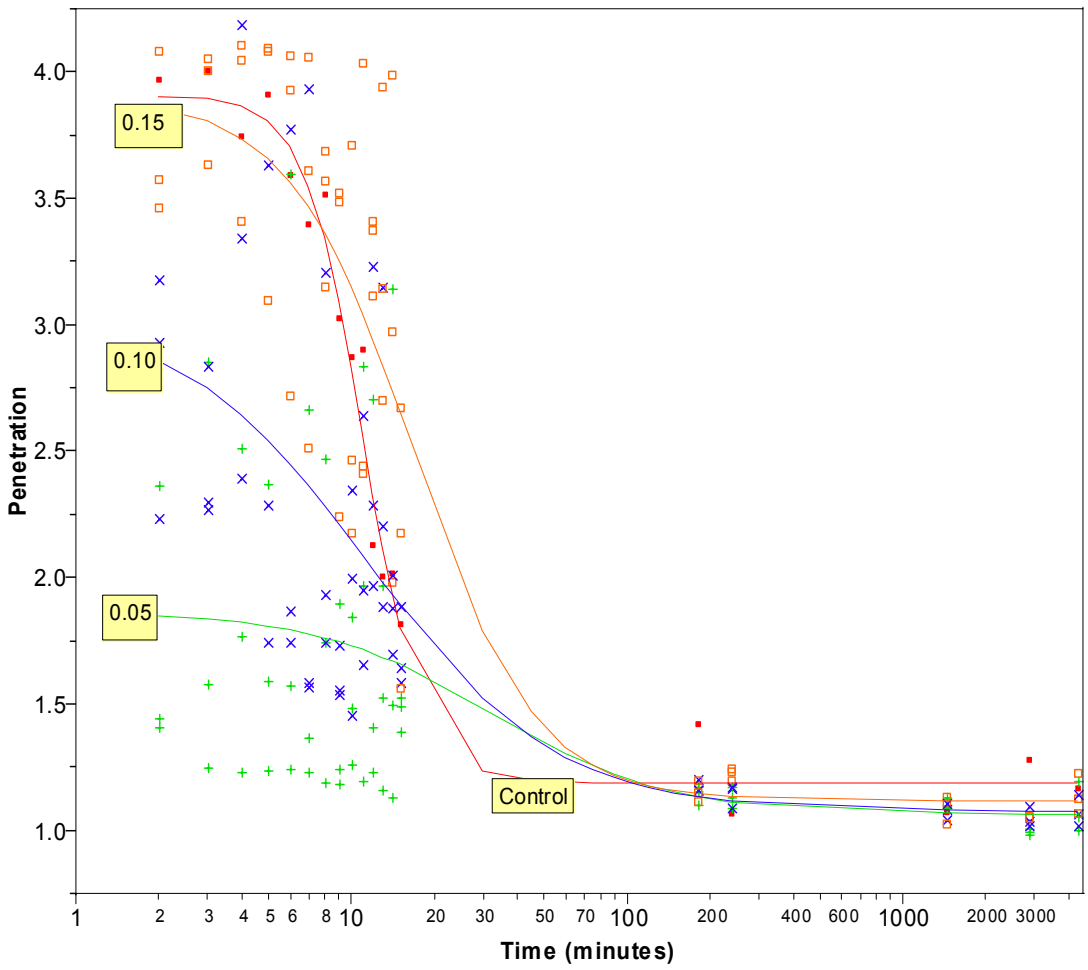
(Sample 10)



Accelerator = 15.0%: Samples 2, 7, and 10 used an accelerator proportion of 0.15. The results are shown in Figures 8, 9 and 10. For sample 2, the estimated Max = 3.92 (SE = 0.084), Slope = 9.3 (SE = 2.055), Median = 13.45 (SE = 0.311), Min = 1.14 (SE = 0.103). For sample 7, the estimated Max = 4.02 (SE = 0.495), Slope = 2.36 (SE = 2.376), Median = 16.33 (SE = 4.617), Min = 1.12 (SE = 0.29). For sample 10, the estimated Max = 3.67 (SE = 0.662), Slope = 0.91 (SE = 0.569), Median = 22.87 (SE = 14.35), Min = 1.02 (SE = 0.281).

Figure 11

Penetration vs. Time Summary



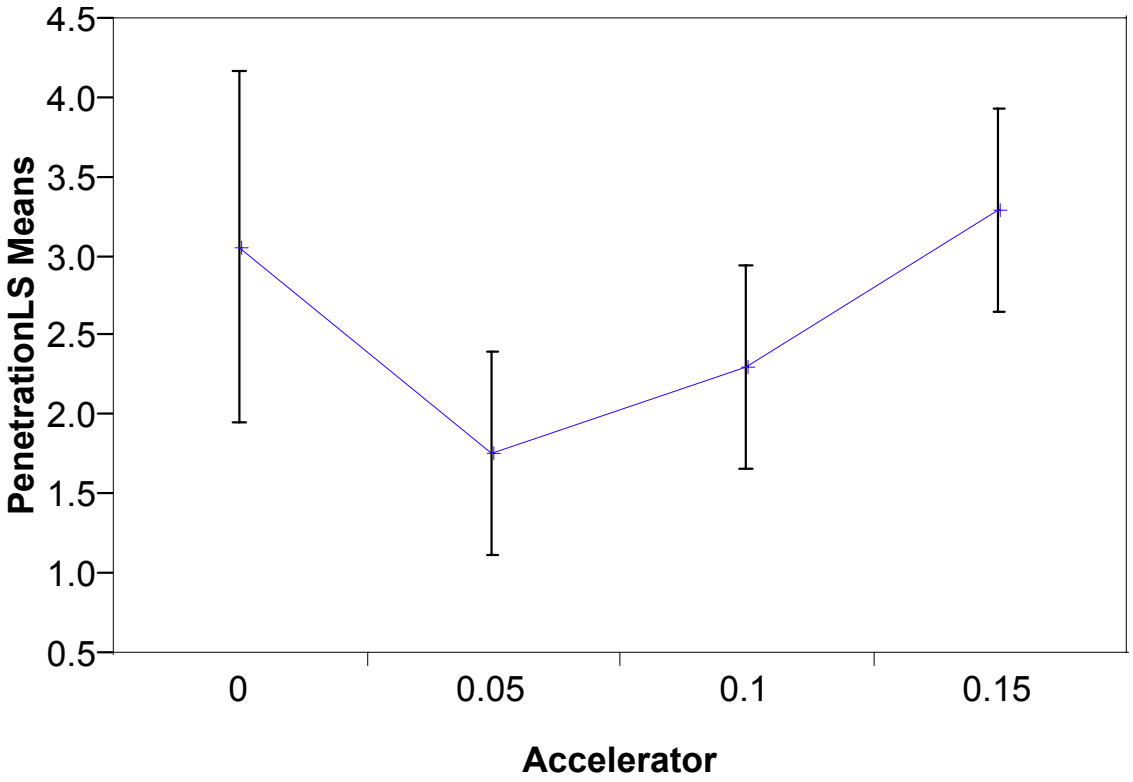
Accelerator

- **0**
- **0.05**
- **0.1**
- **0.15**

Figure 12

Repeated-Measures ANOVA Results: Comparing the Four Groups

(LS Means and 95% CI)



For completeness, the LS Means for each time point and each accelerator are shown in Table 3.

Table 1

Summary of Results

		Max		Slope		Median		Min	
Accelerator	Sample	Est	SE	Est	SE	Est	SE	Est	SE
0.00	**3**	**3.910**	**0.088**	**4.096**	**0.550**	**11.11**	**0.37**	**1.191**	**0.070**
0.05	5	1.401	0.339	0.339	0.260	50.67	184.51	0.931	0.241
0.05	6	2.558	0.128	23.281	20.731	13.39	0.60	1.166	0.176
0.05	9	1.052	0.249	-4.653	54.855	128.02	577.84	1.670	0.115
0.05	**all**	**1.872**	**0.253**	**1.359**	**1.985**	**31.93**	**43.25**	**1.066**	**0.188**
0.10	1	2.067	0.136	3.134	27.531	97.53	578.03	1.081	0.253
0.10	4	3.533	0.203	1.394	0.376	4.90	0.89	1.100	0.153
0.10	8	3.527	0.341	3.566	2.126	11.55	1.79	1.139	0.254
0.10	**all**	**3.053**	**0.686**	**1.280**	**1.054**	**11.48**	**6.25**	**1.079**	**0.157**
0.15	2	3.925	0.084	9.301	2.055	13.45	0.31	1.138	0.103
0.15	7	4.023	0.495	2.363	2.376	16.33	4.62	1.123	0.290
0.15	10	3.671	0.662	0.914	0.569	22.87	14.35	1.016	0.281
0.15	**all**	**3.902**	**0.294**	**1.955**	**1.070**	**16.71**	**2.77**	**1.121**	**0.142**

Table 2

Repeated-Measures ANOVA Results: Comparing the Four Groups

Accelerator	LS Mean	SE	95% CI	
none	3.058	0.453	1.950	4.166
0.05	1.757	0.261	1.117	2.396
0.10	2.304	0.261	1.664	2.943
0.15	3.296	0.261	2.657	3.936

Table 3

Repeated-Measures ANOVA Predicted Penetration

	Accelerator = 0 (control)		
Minutes	**LS Mean**	**95% CI**	
2	3.964	2.581	5.347
3	4.001	2.618	5.384
4	3.742	2.359	5.125
5	3.903	2.520	5.286
6	3.585	2.202	4.968
7	3.394	2.011	4.777
8	3.511	2.128	4.894
9	3.017	1.634	4.400
10	2.863	1.480	4.246
11	2.897	1.514	4.280
12	2.121	0.738	3.504
13	1.996	0.613	3.379
14	2.009	0.626	3.392
15	1.811	0.428	3.194
	Accelerator = 0.05		
2	1.739	0.940	2.537
3	1.893	1.095	2.692
4	1.838	1.039	2.637
5	1.733	0.935	2.532
6	2.139	1.340	2.938
7	1.753	0.955	2.552
8	1.802	1.003	2.601
9	1.441	0.643	2.240
10	1.530	0.731	2.328
11	2.000	1.202	2.799
12	1.782	0.983	2.580
13	1.553	0.754	2.351
14	1.923	1.125	2.722
15	1.469	0.670	2.267

Table 3 (continued)

Repeated-Measures ANOVA Predicted Penetration

	Accelerator = 0.10		
Minutes	**LS Mean**	**95% CI**	
2	2.776	1.978	3.575
3	2.461	1.662	3.259
4	3.302	2.503	4.101
5	2.548	1.750	3.347
6	2.459	1.660	3.258
7	2.355	1.556	3.154
8	2.290	1.491	3.088
9	1.605	0.806	2.403
10	1.928	1.129	2.726
11	2.077	1.279	2.876
12	2.490	1.691	3.288
13	2.407	1.608	3.205
14	1.858	1.059	2.657
15	1.699	0.901	2.498
	Accelerator = 0.15		
2	3.704	2.905	4.502
3	3.898	3.099	4.697
4	3.854	3.055	4.653
5	3.758	2.959	4.556
6	3.569	2.770	4.367
7	3.391	2.592	4.190
8	3.467	2.668	4.266
9	3.083	2.284	3.881
10	2.785	1.987	3.584
11	2.962	2.163	3.760
12	3.299	2.500	4.097
13	3.262	2.463	4.060
14	2.980	2.182	3.779
15	2.137	1.338	2.935

Discussion

Mineral Trioxide Aggregate has been in clinical use since 1998. It has been studied extensively and posseses many of the properties of an ideal root-end filling and repair material. One of the reported drawbacks to the material is the difficulty in handling and placement, as well as its slow setting time which often necesitates another treatment appointment for the final restoration (22). Any ability to accelerate the setting of the material to within a single appointment time frame, and the ability to manipulate and rinse around the MTA without the possibility of displacement would be greatly beneficial.

In this study, we attempted to use sample wells that more closely resembled the size of a root-end filling or repair encountered clinically. The original study by Torabinejad et al. of the physical properties of MTA used the ISO specification for root canal sealers (12). ISO specification 6876 dictates that a mold 10 mm in diameter and 1 mm in height should be used for testing setting times (14). Torabinejad et al., used molds 15 mm in diameter and 5 mm in height. The specification also states that materials that require moisture to set, as MTA does, should be tested in a plaster mold of said dimensions. In this study, the sample molds were made of Teflon as it was believed that components of set plaster could alter the setting characteristics of MTA. In addition, sample wells were kept small because of the considerable costs of MTA. Other

requirements such as a temperature of 37º C ± 1 and relative humidity not less than 95% were maintained while materials were not in the testing apparatus.

The dimensions of the indentor needle were decreased from the ISO 6876 standard of 2.0 ± 0.1 mm to 1.0 mm because of the smaller sample diameter. The internal spring and gear mechanism of the dial indicator delivered a force equivalent to a mass of approximately 98 g which fell within the standard mass of the indentor per ISO 6876 of 100 ± 0.5 g. The dial indicator microgauge was used to give more continuous data during the setting process rather than merely "set" or "unset." Since MTA is not considered a pure root canal sealer, these authors feel a new standard may need to be included that is appropriate for this unique material.

The PCA (Target Products Ltd., Burnaby, BC) used in this study is customarily used in percentages ranging from 1-5% by weight of the cement. Its primary use is for mining, tunneling or rock stabilization with shotcrete, however it can also be used with a conventional Portland cement for placing fence posts where rapid set or high early strength is required. The accelerator is a chloride-free, dry powder with a proprietary formula. Its MSDS states that it contains alkaline accelerators in an inert extender. The MSDS also states that the concentration of the material's active ingredient is below the published limits for individual declaration under the Workplace Hazardous Materials Information System (WHMIS) of Canada.

The fifteen minute timeframe, for early testing, was selected because it was felt that this represented the maximum amount of chair time that could be dedicated to the setting of MTA at a single appointment.

The results of this study were different than what was theorized. At the beginning of the study, we hypothesized that as the percentage of PCA increased, the setting time would always decrease proportionally. In a pilot study, we compared MTA with a 10.0% accelerator with MTA alone by simultaneously mixing them on a glass slab. We empirically observed their behavior by creating mounds of each. At 15 minutes, the accelerated sample was clearly more solid. It could be chipped off into discreet pieces with the blade of a mixing spatula. The regular MTA sample continued to have a mushy consistency for up to an hour and attempts to cut it into discreet pieces was like trying to cut mashed potatoes. In light of the results of the actual study, it is possible that the method chosen was not sensitive enough to effectively show these differences. Statistically, only the 5.0% group was different than the control up to 15 minutes.

As the percentage of PCA increased above 5.0%, the material began to behave more like the control as seen in figure 11. It is possible that as the amount of PCA increased above 5.0%, it began to interfere with the setting reaction of MTA. The setting curves shown in figures 1-10 show the somewhat erratic behavior of the material. There are reports in the engineering trade literature regarding "overdosing" Portland cement with additives, such as accelerators, causing setting retardation. This may explain the effects of the 10.0% and the 15.0% accelerators in this study.

Although previous studies have used an APC as a stand-alone material, it is our opinion that the addition of an accelerator separately to the mixture of MTA would be more useful. This would give clinicians the ability to determine the need for acceleration.

The addition of a PCA in the amount of 5.0% can accelerate the setting reaction of MTA significantly faster than MTA alone. Further studies to evaluate the biocompatibility and toxicity of PCA should be performed before its introduction into clinical use. Also, additional studies should be done to measure the effects of a PCA on MTA's physical and chemical properties.

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Literature Cited

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APPENDIX

SAMPLE 1
10% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.285	2.057	2.228
3 minutes	4.273	1.982	2.291
4 minutes	4.312	1.926	2.386
5 minutes	4.232	2.492	1.740
6 minutes	4.267	2.526	1.741
7 minutes	4.282	2.703	1.579
8 minutes	4.297	2.370	1.927
9 minutes	4.315	2.781	1.534
10 minutes	4.295	2.300	1.995
11 minutes	4.318	1.685	2.633
12 minutes	4.338	1.112	3.226
13 minutes	4.349	2.152	2.197
14 minutes	4.307	2.431	1.876
15 minutes	4.290	2.713	1.577
3 hours *	4.339	3.145	1.194
4 hours *	4.334	3.171	1.163
24 hours *	4.228	3.148	1.080
48 hours *	4.275	3.184	1.091
72 hours *	4.227	3.167	1.060

SAMPLE 2
15% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.368	0.289	4.079
3 minutes	4.365	0.310	4.055
4 minutes	4.391	0.286	4.105
5 minutes	4.444	0.364	4.080
6 minutes	4.375	0.449	3.926
7 minutes	4.325	0.717	3.608
8 minutes	4.384	0.700	3.684
9 minutes	4.373	0.849	3.524
10 minutes	4.295	0.583	3.712
11 minutes	4.393	0.361	4.032
12 minutes	4.335	1.224	3.111
13 minutes	4.387	1.687	2.700
14 minutes	4.388	2.410	1.978
15 minutes	4.394	2.217	2.177
3 hours *	4.380	3.185	1.195
4 hours *	4.372	3.178	1.194
24 hours *	4.367	3.235	1.132
48 hours *	4.345	3.293	1.052
72 hours *	4.307	3.242	1.065

SAMPLE 3
control - no accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.385	0.421	3.964
3 minutes	4.380	0.379	4.001
4 minutes	4.104	0.362	3.742
5 minutes	4.295	0.392	3.903
6 minutes	4.130	0.545	3.585
7 minutes	4.113	0.719	3.394
8 minutes	4.186	0.675	3.511
9 minutes	4.175	1.158	3.017
10 minutes	4.203	1.340	2.863
11 minutes	4.260	1.363	2.897
12 minutes	4.246	2.125	2.121
13 minutes	4.212	2.216	1.996
14 minutes	4.257	2.248	2.009
15 minutes	4.292	2.481	1.811
3 hours	4.259	2.845	1.414
4 hours * **	3.023	2.965	1.058
24 hours * **	2.945	2.880	1.065
48 hours *	4.245	2.971	1.274
72 hours * **	3.166	3.004	1.162

SAMPLE 4
10% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.337	1.162	3.175
3 minutes	4.264	2.002	2.262
4 minutes	4.312	0.976	3.336
5 minutes	4.309	2.030	2.279
6 minutes	4.264	2.399	1.865
7 minutes	4.293	2.733	1.560
8 minutes	4.319	2.581	1.738
9 minutes	4.337	2.786	1.551
10 minutes	4.275	2.827	1.448
11 minutes	4.309	2.657	1.652
12 minutes	4.251	2.287	1.964
13 minutes	4.260	2.379	1.881
14 minutes	4.258	2.564	1.694
15 minutes	4.272	2.391	1.881
3 hours *	4.354	3.199	1.155
4 hours *	4.313	3.227	1.086
24 hours *	4.242	3.205	1.037
48 hours *	4.214	3.181	1.033
72 hours *	4.189	3.176	1.013

SAMPLE 5
5 % Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.295	2.888	1.407
3 minutes	4.285	3.035	1.250
4 minutes	4.272	3.043	1.229
5 minutes	4.264	3.026	1.238
6 minutes	4.288	3.043	1.245
7 minutes	4.268	3.038	1.230
8 minutes	4.304	3.114	1.190
9 minutes	4.286	3.102	1.184
10 minutes	4.270	3.010	1.260
11 minutes	4.289	3.094	1.195
12 minutes	4.315	3.083	1.232
13 minutes	4.254	3.094	1.160
14 minutes	4.300	3.172	1.128
15 minutes	4.286	2.894	1.392
3 hours	4.326	3.222	1.104
4 hours	4.335	3.207	1.128
24 hours *	4.279	3.181	1.098
48 hours *	4.195	3.200	0.995
72 hours *	4.221	3.221	1.000

SAMPLE 6
5% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.333	1.968	2.365
3 minutes	4.314	1.462	2.852
4 minutes	4.254	1.740	2.514
5 minutes	4.289	1.919	2.370
6 minutes	4.257	0.658	3.599
7 minutes	4.272	1.606	2.666
8 minutes	4.279	1.807	2.472
9 minutes	4.248	2.352	1.896
10 minutes	4.268	2.426	1.842
11 minutes	4.251	1.416	2.835
12 minutes	4.272	1.568	2.704
13 minutes	4.303	2.333	1.970
14 minutes	4.259	2.762	1.497
15 minutes	4.241	2.751	1.490
3 hours *	4.311	3.139	1.172
4 hours *	4.309	3.136	1.173
24 hours *	4.323	3.190	1.133
48 hours *	4.180	3.195	0.985
72 hours *	4.290	3.093	1.197

SAMPLE 7
15% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.276	0.704	3.572
3 minutes	4.281	0.273	4.008
4 minutes	4.289	0.241	4.048
5 minutes	4.328	0.232	4.096
6 minutes	4.267	0.205	4.062
7 minutes	4.290	0.234	4.056
8 minutes	4.258	0.690	3.568
9 minutes	4.248	0.764	3.484
10 minutes	4.284	2.107	2.177
11 minutes	4.279	1.867	2.412
12 minutes	4.278	0.903	3.375
13 minutes	4.299	0.356	3.943
14 minutes	4.315	0.325	3.990
15 minutes	4.274	2.713	1.561
3 hours *	4.304	3.193	1.111
4 hours *	4.317	3.086	1.231
24 hours *	4.310	3.205	1.105
48 hours *	4.202	3.150	1.052
72 hours *	4.285	3.160	1.125

SAMPLE 8
10% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.339	1.413	2.926
3 minutes	4.355	1.526	2.829
4 minutes	4.330	0.146	4.184
5 minutes	4.301	0.675	3.626
6 minutes	4.274	0.503	3.771
7 minutes	4.331	0.405	3.926
8 minutes	4.308	1.104	3.204
9 minutes	4.334	2.605	1.729
10 minutes	4.291	1.951	2.340
11 minutes	4.313	2.366	1.947
12 minutes	4.321	2.042	2.279
13 minutes	4.277	1.135	3.142
14 minutes	4.247	2.243	2.004
15 minutes	4.295	2.655	1.640
3 hours *	4.330	3.177	1.153
4 hours *	4.349	3.182	1.167
24 hours *	4.302	3.201	1.101
48 hours *	4.194	3.182	1.012
72 hours *	4.347	3.212	1.135

SAMPLE 9
5% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.367	2.923	1.444
3 minutes	4.370	2.792	1.578
4 minutes	4.363	2.592	1.771
5 minutes	4.337	2.745	1.592
6 minutes	4.294	2.721	1.573
7 minutes	4.287	2.923	1.364
8 minutes	4.249	2.505	1.744
9 minutes	4.290	3.046	1.244
10 minutes	4.307	2.820	1.487
11 minutes	4.285	2.314	1.971
12 minutes	4.252	2.843	1.409
13 minutes	4.266	2.738	1.528
14 minutes	4.264	1.119	3.145
15 minutes	4.265	2.741	1.524
3 hours *	4.344	3.190	1.154
4 hours *	4.327	3.235	1.092
24 hours *	4.263	3.171	1.092
48 hours *	4.201	3.194	1.007
72 hours *	4.250	3.197	1.053

SAMPLE 10
15% Accelerator

Time	Starting #	Ending #	S-E #'s
2 minutes	4.132	0.672	3.460
3 minutes	4.144	0.513	3.631
4 minutes	4.172	0.763	3.409
5 minutes	4.130	1.033	3.097
6 minutes	4.141	1.423	2.718
7 minutes	4.167	1.658	2.509
8 minutes	4.180	1.031	3.149
9 minutes	4.171	1.931	2.240
10 minutes	4.151	1.684	2.467
11 minutes	4.180	1.739	2.441
12 minutes	4.159	0.749	3.410
13 minutes	4.164	1.022	3.142
14 minutes	4.188	1.215	2.973
15 minutes	4.159	1.487	2.672
3 hours *	4.156	3.008	1.148
4 hours *	4.151	2.908	1.243
24 hours *	4.053	3.027	1.026
48 hours *	4.149	3.098	1.051
72 hours *	4.186	2.960	1.226

* No complete indentation
** Needle slide not used, measurement started at surface of material

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