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Graduate School

COMPARATIVE IN VITRO STUDIES ON BOND
STRENGTH AND SEALANT POLYMERIZATION

by

Gerald E. Sievers

A Manuscript Submitted by Gerald E. Sievers in
Partial Fulfillment of the Requirements for the Degree
Master of Science in Orthodontics

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Each person whose signature appears below certifies that this manuscript in his opinion is adequate, in scope and quality, in lieu of a thesis for the degree Master of Science.

, Chairman

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ABSTRACT

COMPARATIVE IN VITRO STUDIES ON BOND STRENGTH AND SEALANT POLYMERIZATION

by

Gerald E. Sievers

A comparison of two orthodontic sealants and two adhesives was made. An experimental adhesive was tested (Unitek*) also an experimental sealant (Unitek**) was evaluated for its ability to polymerize into a thin film. An established adhesive and sealant system (Concise***) was compared with the above products.

The factors which were considered important to the study were: the strength of the bond formed; the working efficiency; the ability for these materials to bond to the lingual of teeth; and the value of using contralateral teeth when testing bond strength.

An Instron testing machine was used to record the shear strength of the bond between bracket and tooth. Because the acid etch pattern and depth varies from tooth to tooth, pairs

* Unitek Experimental Adhesives, Unitek Corp., Monrovia, CA

** Unitek Experimental Sealant, Unitek Corp., Monrovia, CA

*** Concise Enamel Bond-Composite System, 3M Co. St. Paul, MI

of contralateral premolars were bonded in main part of this study. Sixty other human teeth were also tested.

The results showed no statistically significant differences in bond strength between the two adhesive-sealant systems, but that using contralateral teeth is very important in a bond strength study. The data indicated that brackets bonded to the lingual of the premolars formed a weaker bond, but the difference was of borderline statistical significance only. Also, the brackets were designed for buccal surfaces, and thus not fitting perfectly on the lingual surfaces.

Unitek corporation developed eight different groups of sealants. These sealants were tested for working time, esthetics, and ability to polymerize. Unitek continued these studies and the experimental sealant they developed was used in the bonding study evaluated in this paper.

INTRODUCTION

There is much uncertainty in the field of orthodontics regarding the use of bonding adhesives and sealants.^{4,13} Progress has been made in the past few years. Using different combinations of filled and unfilled composite resins there has been a steady increase in retentive bond strength.^{11,14,20} Early reports showed varying depths of adhesive resin penetration into etched enamel,^{5,10} but later studies^{9,14} have questioned if viscosity has any significant effect on the penetration of a resin into etched enamel. Probably, individual differences in enamel properties are quite important in determining a tooth's response to etching.⁸

The difficulties with regard to sealants are mainly dealing with problems in polymerization of such materials.²⁴ This is due to the inhibiting effect of atmospheric oxygen, which varying with different sealants may prevent polymerization completely or result in only patches of protective sealant remaining on an etched tooth.²⁴ Another controversy is whether or not a sealant will increase or decrease the bond strength.^{11,12,15} It is agreed that sealants enhance the ability to wet and adjust to etched enamel, but some workers have indicated that the resulting bond may be weaker.¹²

Recently, Jorgensen⁸ showed that the etch pattern of

human enamel varies greatly in depth and design from tooth to tooth. However in the same study, contralateral teeth were found to show a high degree of etch pattern resemblance.⁸ Because of these findings it was felt that in the present study, it would be optimal to use pairs of contralateral premolars, which had been removed for orthodontic reasons. Both the lingual and buccal surfaces were used in evaluating different materials. An additional in vitro evaluation using sixty random human teeth was done. This involved the same method of testing as with the contralateral premolars, but only the buccal surface was bonded.

The purpose of this study was to examine and compare the bond strength and overall efficiency of a new experimental direct-bond paste-paste system* with a widely used adhesive.** Experiments were also made to compare an experimental sealant (Unitek***) with the Concise sealant.** The setting time, workability, esthetics, strength, and specifically the effectiveness of the sealant to polymerize and leave a thin film were factors for evaluation.

* Unitek Experimental Adhesives, Unitek Corp., Monrovia, CA
** Concise Enamel Bond-Composite System, 3M Co., St. Paul, MI
*** Unitek Experimental Sealant, Unitek Corp., Monrovia, CA

MATERIALS AND METHODS

I. Bond strength - contralateral pairs of premolars

Two sealants and two adhesives were tested on twenty-four premolars: the experimental paste-paste bonding system and the Concise system, where a resin-paste combination²³ was selected. The number of buccal and lingual surfaces sealed and bonded is shown in Table I.

All premolars had been recently extracted as part of orthodontic treatment and were placed in water and a small amount of lysol disinfectant. In order to facilitate bonding to both the buccal and lingual surfaces, the teeth were cut into halves with a rotating disk. Each half was then fixed in a circular mold of cold cure acrylic.*

The resulting molds were cleaned and trimmed on a rotating wheel, and the teeth polished with pumice and a bristle brush. They were then rinsed, air-dried, and etched with 37 percent phosphoric acid** for 60 seconds. They were rinsed again, thoroughly air-dried, and the characteristic dull white surface was noted.

Two drops of monomer and catalyst were mixed for approximately two seconds. The sealant was then carefully painted

* Unitek Cold Cure Acrylic, Unitek Corp., Monrovia, CA

** Unitek Etching Acid, Unitek Corp., Monrovia, CA

on the etched tooth in a thin film with mini-brushes.* A meticulous attempt was made to regulate the thickness of this layer so that it was uniform for each surface.

Equal amounts of experimental adhesive pastes were mixed with a spatula and placed on the bracket to be bonded. Using a scaler**, the bracket was then firmly pressed onto the tooth and the excess removed.²⁵ With Concise adhesive the same process took place except a resin-paste combination was used to speed the setting.²³ The teeth were then placed in water at a temperature of 24 C for 24 hours.

The brackets used in this study*** had a small pad especially adapted for the buccal premolar surface. This placed a bias on the lingual bond strengths values, but because of their small size many of the pads seemed to visually fit the lingual quite well.

An Instron mechanical testing machine**** was used to exert the force and record the shear loading strength. The crosshead speed was 0.1 inches per minute. An 0.018 inch round wire in the form of a loop was applied the pull to the bracket (Fig. 1). Thus a shear stress was employed to break

- * Unitek Mini-brushes, Unitek Corp., Monrovia, CA
- ** RM 349 Rocky Mountain Orthodontics, Denver, Colorado
- *** Unitek Minipad Brackets, (bracket-001275, base-019-415)
Unitek Corp., Monrovia, CA
- **** Instron, model 1123, Canton, Mass.

the tooth to bracket bond. A diagram of the appliance is shown on page 19.

II. Bond strength - different teeth

Sixty extracted human molars, bicuspids, cuspids, and incisors were utilized in an identical study to the one described above. The teeth had been extracted and placed in water at room temperature. These teeth had been stored for an essentially unknown length of time. Only the buccal surface was bonded, and therefore 15 surfaces, were evaluated with each sealant-adhesive combination.

III. Sealant polymerization

The evaluation of the combination of a catalyst and accelerator to produce a quality sealant involved eight groups of sealants. Within each, those factors influencing (1) the thickness of the film; (2) the working time; (3) esthetics; and (4) storage life; were varied (Tables VIII-XV).

The method used to test sealants involved placing two drops of catalyst and two drops of accelerator resin into a dappen dish and mixing with a small bristle brush.* A layer was painted on a mixing pad** within a standardized oval

* Unitek Mini-brushes (605-001), Unitek Corp., Monrovia, CA

** Unitek Mixing Pads, (#704-030), Unitek Corp., Monrovia, CA

marked with a pencil. The size was 2.5 inches in length and 1.5 inches in width.

The working time was recorded as the time until the sealant became "stringy" in the dappen dish. The final set was recorded when the sealant was solid. Following final set the sealant was wiped with gauze, thereby removing any unpolymerized material. The characteristics of the sealants were then compared and evaluated on the basis of film thickness, quality, and working time.

The materials within each group of sealant varied in following manner and are shown in Tables VIII through XV. Group one (Table VIII) evaluated a low viscosity resin used with varying percentages of amine and peroxide in the accelerator and catalyst, respectively. Group two (Table IX) was similar except a medium viscosity resin was used. Group three (Table X) used a high viscosity resin. Group four (Table XI) used high viscosity resin and percentages of 2.5 to 3.0 and 2.5 to 2.9, with increasing increments of a tenth of one percent of amine and peroxide, respectively.

In group five (Table XII) acetone was incorporated into the catalysts. With Group six (Table XIII) methyl methacrylate was incorporated in the accelerators. The concentrations of peroxide and amines which produced the most favorable sealants were then tested with methyl methacrylate included

(Table XIV). This represented group seven. In group eight the diagonal sealants in group seven were reevaluated (Table XV). These sealants were then tested for shelf life.

As the tests generated better results, a ceramic tile* was painted with a blue dye** and covered with sealant. Following setting time of each sealant combination the film was wiped with a piece of gauze. The polymerized sealant prevented the removal of the dye and therefore, indicating how well it had coated the tile; it was thus possible to find a sealant which had an ideal working time and overcame the factor of oxygen inhibition of polymerization.

* White Smooth Tile, Dal-tile, Monterrey, Mexico

** Blue dye, FDC-Blue Dye No. 1

RESULTS

I. Bond Strength - contralateral premolars

When the brackets were pulled off, there was no clear enamel-to-composite or bracket-to-composite break. Generally, what did take place was an irregular fracture, that is, there was remaining peripheral composite on the bracket as well as on the enamel. Table II displays the data representing the force at which the fracture took place for each tooth. There was a remarkably wide range of force, varying from 17 pounds to 77 pounds.

Using a general linear hypothesis computer program,* it was shown that there was no significant difference in bond strengths between the different sealant-adhesive combinations when bonding contralateral premolars (Table III). The large P values indicate the high degree of etching similarity that opposing teeth display ($p = 0.576$). The data are given in Table III.

The buccal versus lingual bonding tests revealed a trend towards difference in bonding strength (Table III), just above borderline significance ($p = .064$). In Table IV means and standard deviations of the adhesive strengths are tabulated. Neither of the differences are statistically signi-

* Refer to Table III

ficant when considering 12 trials.

II. Bond Strength - different teeth

The bond strengths of the sixty human teeth revealed no appreciable statistical difference between either the adhesives or sealants. The mean and standard deviation values can be found in Table VII. The actual individual values are displayed in Table V and the p-values (adhesive $p = 0.083$, sealant $p = 0.552$) are in Table VI. Again, the fracture resistance varied greatly, ranging from 4.0 pounds to 55.0 pounds of force.

The sealants had very little effect on the bond strength (Tables IV, VII), and this is represented by a very high p-value also ($p = 0.550$). The adhesive strengths reveal no statistically significant difference ($p = 0.222$).

III. Sealant Polymerization

Acetone proved very effective in preventing oxygen inhibition, and acetone-containing sealants polymerized into a very thin film. Also, methyl methacrylate aided in preventing the inhibition of polymerization effect of oxygen. Addition of this chemical and high viscosity allowed an ideal thickness of sealant, resulting in a fine quality experimental sealant. Optimum percentages of amine and peroxide were determined empirically.

The results of the sealant tests are given in Tables VIII through XIII. The first value in these tables is the working time; the second figure represents the time to final set. The optimal time for working with the sealant was approximately 1 minute.

Group one, the lowest viscosity sealants, resulted generally in non-polymerized patchy films. Group two, because of its medium viscosity, produced slightly better sealants. The high viscosity resins in group three produced almost completely polymerized films which improved in quality as the percentage of both amine and peroxide increased.

Group four displays the tenth of a percent increases of amine and peroxide from 2.5 percent to 3.0 percent. The most complete film is the one with 2.9 percent peroxide and 3.0 percent amine. This film did have several of 1-2 millimeter spots which did not polymerize.

Acetone was incorporated into the catalyst of group five and resulted in several near perfect films. With methyl methacrylate included in the accelerator, group six resulted in several ideal films also. With both group five and six the shorter working times produced the ideal sealants.

Group seven combined the varying of amine and peroxide percentages with methyl methacrylate. The highest quality films were correlated with increasing percentages of amine

and peroxide, and thus the shortest working time. Group eight was a diagonal of group seven and again 4 percent peroxide and 10 percent amine with methyl methacrylate resulted in a very near ideal film.

The experimental sealant used in the bonding study was a sealant which produced a polymerized film and optimal working time. It was the result of further studies by Unitek* and did not include methyl methacrylate or acetone.

* Unitek Experimental Sealant, Unitek Corp., Monrovia, CA

DISCUSSION

Acid etching with phosphoric acid and bonding has become a competitive alternative to banding in the past few years. Because of micro-undercuts in the prepared tooth surface, it is possible to have a bracket adhere firmly to a tooth. The amount of retention that etching will produce is highly variable from tooth to tooth and can differ markedly with specific areas of the enamel surface.^{8,19}

The main reasons for the failure of a bracket bonded to a tooth are as follows:^{23,25} 1) moisture contamination, 2) poor adaptation of the bonding pad to the curvature of the tooth, 3) disturbed setting of the adhesive, 4) excessive forces, and 5) individual variation (differences in enamel composition). These factors were controlled as much as possible in this study. The use of a very dry in vitro field; small mini-pad brackets to minimize adaptation variation due to enamel surface curvature; slow controlled dislocating forces which were accurately recorded; and contralateral premolars to minimize individual mineralization differences; each aided in optimal evaluation of the strength of the bonding systems being tested.

A problem which relates to the variability of the lingual radius of premolars must be recognized. Although the

brackets used in this study were small, the unique shape of the premolar lingual cusp sometimes resulted in an inadequate adaptation. This is likely to explain the tendency to differences in bond strength between buccal and lingual surfaces (Tables I-III). A special lingual bracket pad would seem to be advantageous in the clinical situation.

The bond strengths of the sixty random human teeth showed similar statistical results as the contralateral premolars. The overall bond strength values were slightly lower on these teeth. This could have been due to differences in enamel quality and the surface curvature variations. The brackets used were specifically manufactured for premolars, and even though the pads were small the adaptation was not as complete with molars, cuspids, and incisors.

In the attempts to find a favorable sealant, eight separate groups of sealants were tested. Within each of these groups the viscosity as well as the ingredients varied. The primary variable in the catalyst was the percentage of peroxide present. With the accelerator it was the content of amine.

Because atmospheric oxygen inhibits the polymerization of a sealant, an attempt was made to use acetone as an agent to displace the O_2 during the setting of the sealant. This was accomplished by placing acetone in the catalyst. But

because of its tendency to yellow with aging, the storage of acetone resulted in an esthetic problem. This factor as well as the consideration of its toxicity caused it to be dismissed as a possibility. It should be mentioned, however, that good clinical results have been experienced in Scandinavia with acetone-containing sealants.²⁴

Another agent which was incorporated into the accelerator was methyl methacrylate. This functioned as a volatile substance which also displaces O_2 during polymerization.

Viscosity was considered an important factor in finding a suitable sealant. This is due to the concept that as the viscosity is increased, the effect of oxygen inhibition of polymerization is decreased.²⁴ There was a balance to be considered between the maximum amount of inhibition control and a proper thickness of sealant.

An important variable when considering a sealant for clinical use is the working time, therefore the working and setting time was evaluated for every combination of catalyst and accelerator. The ideal working time appeared to be approximately 1 minute. This should allow sufficient time to paint an entire dentition to be bonded.

In this study, the different sealants were not a significant factor when differences in bond strength were analyzed. However, with the best combinations the experimental sealant

apparently polymerized into a smooth protective film over the complete surface of each tooth painted with it. The Concise sealant displayed some problems of flowing into thick islands and elsewhere, leaving areas unpolymerized and therefore unprotected. This confirms previous findings by others.²⁴ A bond-strength study without sealants would be a helpful control to observe if the sealant affects the fracture pattern and strength. In such a study on pairs of contralateral teeth, Raadal has shown that at least with the Concise system, the sealant makes no statistically significant difference in bond strength.¹⁵

An interesting concept that must be considered in evaluating future sealants is that of its use as a fracture medium. As adhesives increase in strength the sealant could control the strength as well as where the bracket-to-tooth break will take place. This would aid substantially in efficient debonding and prevent potential enamel damage by filers particles entering into the etched tooth surface.

SUMMARY

A standard direct bonding technique was performed on the buccal and lingual of 24 extracted premolars. Then the bonding strengths of two adhesive and sealant systems (Unitek experimental adhesive and sealant system, Concise Enamel-bond system) were compared with an Instron testing technique.

The results demonstrated that pairing of contralateral teeth is necessary to compare variables such as adhesives, mesh design, sealants, etching agents, and buccal versus lingual bonding. The etching pattern and depth varies too much from tooth to tooth, thus affecting the bond strength noticeably. Increasing the number of trials would also aid with problems.

A more detailed study is needed to compare the lingual etching qualities of teeth. This would require contralateral teeth and ideal fitting lingual brackets.

The Unitek experimental sealant polymerizes into an esthetic, thin, workable film, but detailed clinical studies are required to determine durability, long term esthetics, and the cleansing efficiency. This contrasted with the Concise sealant. When the procedure of painting a tooth was followed by washing and a blast of air patches of thick sealant were noted. Also the characteristic white frosted

appearance of etching was obvious in other areas.

As far as the adhesive bonding strengths are concerned, the difference between these values was not significant. This was true for both the test involving contralateral premolars as well as the sixty random in vitro human teeth. The statistical difference between these two adhesives could only be appreciated with another study which included a larger number of trials.

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TABLE I. Number of surfaces bonded.

Adhesive	Number of surfaces sealed with Concise Sealant	Number of surfaces sealed with Unitek Sealant
<u>Experimental</u> <u>Unitek</u>	12	12
Buccal	6	6
Lingual	6	6
<u>Concise</u>	12	12
Buccal	6	6
Lingual	6	6

TABLE II. Individual bond strength values.

Buccal

Sealant = EA Adhesive = ES	Sealant = C Adhesive = EA	Sealant = ES Adhesive = C	Sealant = C Adhesive = C
35.5	21.0	36.0	32.0
40.0	31.5	34.0	25.5
54.5	33.0	35.0	43.0
49.0	37.5	44.5	50.0
30.0	53.0	41.0	33.5
29.0	45.5	57.0	43.0
$\bar{x} = 36.0$	36.9	41.25	37.8

Lingual

29.5	21.0	30.0	28.0
20.0	29.0	26.0	35.0
24.5	51.0	21.0	30.0
29.5	36.5	35.0	31.0
36.5	17.0	33.5	33.5
34.0	29.5	77.0	26.0
$\bar{x} = 29.0$	30.6	37.1	25.5

C = Concise

EA = Experimental adhesive (Unitek)

ES = Experimental sealant (Unitek)

TABLE III. $Y = u + \text{Pair} + \text{Buccal/Lingual} + \text{Adhesive} + \text{sealant} + \text{error}$. Statistical evaluation of the significance of the variables.*

Source of variation	Sum of squares	Mean square	F	Significance of F
Pairs	1218.4	110.7	0.871	.576
Buccal/Lingual	468.7	468.7	3.685	.064
Adhesive	197.5	197.5	1.553	.222
Sealant	46.3	46.3	0.364	.550

* The above formula represents the general linear hypothesis computer program. This program evaluates the statistical significance of one variable as compared to one or more other variables. This process is repeated with each variable.

TABLE IV. Means and standard deviations for strength values.
Variable for entire population of adhesive.

	Experimental Adhesive	Concise Adhesive
Experi- mental Sealant	Mean = 33.91 S. D. = 10.12	Mean = 33.79 S. D. = 11.59
Concise Sealant	Mean = 39.16 S. D. = 14.99	Mean = 36.29 S. D. = 8.4
Combined Sealants	Mean = 33.85 S. D. = 10.64	Mean = 37.72 S. D. = 11.98

Table V. Individual bond strengths for random teeth.

Range = 04.0 to 55.0

	Sealant = U Adhesive = U	Sealant = U Adhesive = C	Sealant = C Adhesive = U	Sealant = C Adhesive = C
1	06.0	42.5	33.5	29.0
2	29.0	22.0	05.0	10.0
3	44.5	30.0	28.5	17.5
4	17.5	21.5	30.5	30.0
5	04.0	26.5	20.5	31.0
6	04.0	42.0	24.0	25.0
7	17.0	55.0	19.0	26.0
8	30.0	38.0	10.0	33.5
9	16.5	17.0	35.0	28.5
10	34.0	30.0	30.5	26.0
11	04.0	23.5	08.0	27.5
12	20.0	28.5	53.0	29.0
13	23.5	27.5	36.0	23.5
14	17.0	16.0	35.0	13.0
15	14.5	14.0	09.5	34.5

Table VI. $Y = U + \text{Adhesive} + \text{Sealant} + \text{error}$
 Statistical evaluation of the significance of
the variables.*

Source of Variation	Sum of Squares	F	Significance of F
Adhesive	390.15	3.10	0.083
Sealant	45.067	0.36	0.552

* Refer to Table III.

Table VII. Means and standard deviations for strength values.

	Experimental Adhesive	Concise Adhesive
Experimental Sealant	Mean = 18.76 S.D. = 11.94	Mean = 28.93 S.D. = 11.31
Concise Sealant	Mean = 25.56 S.D. = 13.48	Mean = 25.60 S.D. = 7.06
Combined Sealants	Mean = 22.16 S.D. = 12.98	Mean = 27.26 S.D. = 9.42

TABLE VIII. Low viscosity resin with varying percentages at amine and peroxides. (Working time/final set)

Reference No.	Group #1		
31/60-4 2% Peroxide	31/60-7 2% Amine	31/60-8 3% Amine	31/60-10 5% Amine
	7'10"/7'30"	3'40"/4'00"	3'15"/3'25"
31/60-5 3% Peroxide	4'55"/5'13"	2'45"/2'57"	1'55"/2'05"
31/60-6 4% Peroxide	4'10"/4'23"	2'35"/2'43"	1'43"/1'48"
			2'05"/2'15"
			1'30"/1'35"
			1'10"/1'15"

CATALYST

TABLE IX. Medium viscosity resin with varying percentages of amine and peroxide. (Working time/final set)

	Group #2	
Reference No.		
31/60-4	31/60-14	31/60-16
31/60-11	31/60-7	31/60-9
1% Peroxide	<u>2% Amine</u>	<u>4% Amine</u>
	9'47"/10'20"	1'30"/1'42"
	6'30"/6'55"	1'55"/2'10"
	4'40"/4'55"	1'15"/1'26"
	3'58"/4'15"	1'28"/1'38"
31/60-5	31/60-15	31/60-17
31/60-12	31/60-8	31/60-10
3% Peroxide	<u>3% Amine</u>	<u>5% Amine</u>
	3'20"/3'55"	50"/57"
	3'45"/4'05"	55"/1'05"
	1'12"/1'38"	1'10"/1'18"
	1'55"/2'05"	1'25"/1'30"
21/60-6	1'35"/1'45"	1'05"/1'20"
31/60-13	1'30"/1'42"	1'00"/1'10"
4% Peroxide	2'00"/2'23"	45"/50"
	2'20"/2'40"	50"/55"

CATALYST

TABLE X. High viscosity resin with varying groups of amine and peroxide.
(Working time/final set)

Reference No.	Group #3			
	<u>31/60-14</u> 2% Amine	<u>31/60-15</u> 3% Amine	<u>31/60-16</u> 4% Amine	<u>31/60-17</u> 5% Amine
31/60-11 2% Peroxide	2'20"/3'47"	1'25"/2'15"	1'00"/1'25"	45"/1'00"
31/60-12 3% Peroxide	1'25"/2'08"	55"/1'19"	43"/1'00"	32"/45"
31/60-13 4% Peroxide	52"/1'30"	40"/1'03"	25"/40"	20"/33"

CATALYST

TABLE XI. High viscosity resin with varying percentages of amine and peroxide in .1% increments. (Working time/final set) Group #4

Reference No.	31/61-4 2.5% Amine	31/61-6 2.6% Amine	31/61-9 2.7% Amine	31/61-12 2.8% Amine	31/61-14 2.9% Amine	31/60-15 3.0% Amine
31/61-5 2.5% Peroxide	1'17"/2'00"	1'07"/1'34"	1'00"/1'21"	53"/1'16"	52"/1'17"	54"/1'20"
31/61-7 2.6% Peroxide	---	55"/1'27"	---	---	---	51"/1'06"
31/61-10 2.7% Peroxide	---	---	54"/1'23"	---	---	43"1'06"
31/61-11 2.8% Peroxide	---	---	---	47"/1'19"	---	41"/1'03"
31/61-13 2.9% Peroxide	---	---	---	---	48"/1'10"	42"/1'02"

TABLE XII. Acetone incorporated into the catalyst. (Working time)

<u>Acetone</u>	Group #5				
	31/61-5	31/61-7	31/61-10	31/61-11	31/61-13
31/70/1	7:10	6:30	6:40	6:05	5:30
31/70/2	3:20	3:15	3:07	2:55	2:55
31/70/3	3:05	2:35	2:25	2:25	2:10
31/70/4	2:15	1:55	2:00	1:40	1:40
31/70/5	1:45	1:40	1:30	1:35	1:25

TABLE XIII. Methyl methacrylate incorporated into the accelerator.
(Working time) Group #6

<u>In MMA</u>	31/61-5	31/61-7	31/61-10	31/61-11	31/61-13
31/71-1	7:40	7:15	6:40	6:15	6:20
31/71-2	6:15	6:05	6:00	6:00	6:00
31/71-3	4:05	3:10	3:05	3:00	2:45
31/71-4	4:05	3:35	3:00	2:40	2:30

TABLE XIV. Methyl methacrylate incorporated into the accelerator with varying percentages of amine and peroxide. (Working time/ final set)

Group #7

Ref. #	31/71-6 6% Amine	31/71-7 7% Amine	31/71-8 8% Amine	31/71-9 9% Amine	31/71-10 10% Amine
31/61-5	2'50"/3'10"	2'05"/2'20"	1'50"/2'00"	1'35"/1'45"	1'25"/1'35"
2.5% Peroxide					
31/61-7	2'25"/2'45"	2'10"/2'20"	1'50"/2'00"	1'20"/1'35"	1'15"/1'30"
2.6% Peroxide					
31/61-10	1'25"/2'30"	2'00"/1'10"	1'50"/2'05"	1'35"/1'45"	1'15"/1'25"
2.7% Peroxide					
31/61-11	2'15"/2'25"	1'50"/2'00"	140'/150"	1'30"/1'40"	1'20"/1'30"
2.8% Peroxide					
31/61-13	2'00"/2'10"	1'45"/1'55"	1'35"/145"	1'30"/1'40"	1'15"/1'30"
2.9% Peroxide					
31/60-6	2'20"/2'30"	2'05"/2'15"	1'45"/1'55"	1'30"/1'40"	1'25"/1'40"
4% Peroxide					
31/60-13	2'05"/2';5"	1'40"/1'50"	1'30"/1'40"	1'10"/1'20"	1'00"/1'10"
4% Peroxide					

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TABLE XV. A diagonal evaluation of sealants in Table XIV. (Working time/final set)

Group #8

Reference Number	31/71-6 6% Amine	31/71-7 7% Amine	31/71-8 8% Amine	31/71-9 9% Amine	31/71-10 10% Amine
31/61-5 2.5% Peroxide	2'50"/3'10"				
31/61-7 2.6% Peroxide		2'10"/2'20"			
31/61-10 2.7% Peroxide			1'50"/2'05"	1'35"/1'45"	
31/61-11 2.8% Peroxide				1'30"/1'40"	
31/61-13 2.9% Peroxide					1'15"/1'30"
31/61-6 4% Peroxide					1'25"/1'40"
31/60-13 4% Peroxide					1'00"/1'10"

LEGENDS

Fig. 1. Descriptive drawing of mold with embedded tooth and the Instron machine shearing a bonded bracket.

Fig. 1.

