A Comparison of the Accuracy and Dimensional Stability of Four Interocclusal Registration Materials

Yvonne Balthazar Hart
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A COMPARISON OF THE ACCURACY AND DIMENSIONAL STABILITY
OF FOUR INTEROCCLUSAL REGISTRATION MATERIALS

by

Yvonne Balthazar Hart, D.D.S.

A Thesis Submitted to the Faculty of the Graduate School
of Loyola University in Partial Fulfillment of
the Requirements for the Degree of
Master of Science
April
1979
DEDICATION

In loving memory of my mother, Dorothy S. Balthazar.
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Also deserving of recognition are the author's husband, Dr. Timothy Hart, for his guidance in data analysis, the author's mother-in-law, Dr. Annette Hart, for her timely assistance, and the author's father, Dr. Eugene Balthazar, for his moral support.
VITA

Yvonne Balthazar was born October 12, 1947 in Aurora, Illinois to Eugene R. and Dorothy Balthazar. She was the third of four children; having two sisters, Renee and Dorothy, and one brother, Eugene.

In 1965 she graduated from West Aurora High School. She began her formal education at Manhattanville College in Purchase, New York. She graduated in 1969 with a B.A. degree in Art. She received a B.S. degree in dental hygiene from Marquette University, Milwaukee, Wisconsin in 1971. She began her formal dental studies at the Loyola University School of Dentistry, Chicago College of Dental Surgery in 1973 and graduated in 1977 with a Doctor of Dental Surgery degree.

Her graduate studies began in the Department of Oral Biology of Loyola University School of Dentistry, Chicago College of Dental Surgery in 1977. Specialty training was in the Department of Fixed Prosthodontics under the Director of Graduate Fixed Prosthodontics, Dr. William F. Malone.
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CHAPTER I

INTRODUCTION

With the development of the articulator and lost wax technique of fabricating restorations, it has become increasingly inconvenient and impractical to work exclusively in the presence of the patient. Access and vision in the oral cavity are limited. Fabrication of restorations is time consuming.

Articulators have been developed which approximate the patient's maxillomandibular relation and movements. This enables the dentist and technician to study the dentition in function and constructive restorative appliances in the patient's absence (Harcourt, 1973).

However, if the interarch registration is inexact, the mounted casts will not reflect the patient's true maxillomandibular relationship. This error will reflect itself in some dimension of the diagnosis and resultant treatment.

Berman (1960) stated the material used to record this maxillomandibular relation was largely responsible for the accuracy of the record. Also, the stability of the recording material was of growing importance. Models cannot always be poured and mounted on the articulator the same day the
impressions are taken. Often impressions and bite registration are mailed to the technician. The impressions are then poured at his convenience and models articulated.

The dentist and technician often hand articulate models, guided by wear facets. This has proven to be accurate (Strohaver, 1972). However, if (1) the existing occlusion is not within physiologic limits, (2) posterior occlusal stops in the partially edentulous patient are absent, or, (3) the patient is edentulous, hand articulation is not possible. Some recording medium is necessary.

Classically, impression materials have served to record the maxillomandibular relation. Wax, impression plaster of Paris, impression compound and zinc oxide-eugenol impression pastes have been adapted to this need. None have proved to be ideal.

Waxes are inadequate registration materials. Millstein, Clark and Leff (1975), found interocclusal wax records were not repeatable. Also, an inordinate amount of closing force was necessary in establishing the record. This affected the registration of a centric relation record because it set up a resistance reaction by the patient causing him to occlude forward of the position desired (Boos, 1960). Also, Berman (1960), Shanahan (1960) and Strohaver (1972) found wax to be inaccurate and unstable.

Plaster of Paris and impression compound have proven
accurate but inconvenient. Because they were rigid at mouth temperature, any undercut area in the mouth caused breakage of the record upon removal (Berman, 1960).

Berman and others felt zinc oxide-eugenol paste to be the material of choice. The material, (1) mixed to a true fluidity, (2) offered no resistance to closure, (3) adhered to its carrier, (4) set to a hard, non-compressible consistency, and (5) was sharp and easily read. Articulation could be accomplished accurately without fear of distortion or of unequal compression of the record in the vertical dimension.

However, others found the setting time objectionable for an accurate centric relation record. The extended setting time resulted in muscle fatigue and the introduction of registration error. Also, the material was brittle and had a tendency to fracture (Craig, 1975). Patients objected to the odor and eugenol-related tissue reactions (Molnar, 1974).

A non-eugenol zinc oxide paste registration material has been introduced. Because it contains no eugenol, the objectionable odor and tissue reactions have been eliminated. More recently, two elastomeric impression materials, introduced in the early 1970's have been modified and marketed as registration materials. These are the silicone and polyether systems.
The purpose of this investigation is to examine the dimensional stability of these newer materials in a controlled laboratory environment. Four materials, five specimens of each, were tested at five time intervals, immediately, at one hour, twenty-four hours, forty-eight hours, seventy-two hours, and one hundred sixty-eight hours. The results of the investigation were subjected to statistical evaluation. Unrelated "T" tests were used. From the results, it is hoped to draw clinically relevant conclusions and recommendations.
CHAPTER II

REVIEW OF LITERATURE

Interocclusal records are utilized to record centric relation and centric occlusion. It is necessary to briefly discuss these terms.

Centric relation or centric jaw relation is concerned with the relation of the mandible to the maxilla. It was defined as the most retruded relation of the mandible to the maxilla from which the patient could make lateral jaw movements (Sears, 1960). It was a border position and considered by many to be reproducible (Gehl, 1960). Centric relation is purported to be a non-functional position.

The use of centric jaw relation records is predicated on the fact the maxillary cast has been properly oriented to the articulator so the maxillary cast has the same relation to the temporomandibular articulation as it does in the mouth. This is accomplished through location of the hinge axis. The hinge axis was reported to be the center of rotation for mandibular opening and closing (Brotman, 1960). The maxilla is related to the hinge axis by taking a face bow measurement. This is transferred to the articulator.
As well as orienting the maxilla to the temporomandibular joint, the hinge axis is important in taking a centric relation record. If the condyle is in its rearmost position, the mandible, upon opening, arcs with a rotational movement, the hinge axis being the center of rotation. This rotation is followed by a translatory movement as the opening is increased. If the centric relation record was taken as the mandible arced in the rotation phase of opening, the record was thought to be reproducible and unaffected by changes in vertical dimension (Harcourt, 1973).

Brotman (1960) stated in finding centric relation, the mandible was guided posteriorly until the temporomandibular joint ligaments were taut. These tightened ligaments caused the condyle to swing upward into contact with the anterior slopes of their respective fossae, with the menisci interposed. Since the temporomandibular ligaments could not be stretched under normal conditions and since bony structures of the fossae and the condyles did not seem to change readily, centric relation was repeatable.

Whether or not the hinge axis was the proper maxillomandibular position was not known (Harcourt, 1973). However, it was considered a physiologically acceptable maxillomandibular relationship (Brotman, 1960).
Centric occlusion is reported to be a functional relationship of teeth (Wood, 1968). Centric occlusion was the relationship of the opposing teeth involving maximum tooth contact (Gehl, 1960).

In studying Australian Aborigones, Beyron (1960) found most subjects had an anterior-posterior distance between the retruded contact position, centric relation, and the intercuspal position, centric occlusion.

Boos (1962) stated 95% of dentistry was practiced with the presenting or conforming tooth occlusion as the basic criterion of interocclusal relationship. Brecker (1959) stated it was unwise to disturb a physiologically acceptable occlusion. However, certain conditions such as abrasion, periodontal involvement, and temporomandibular dysfunction, made it necessary to look beyond the presented occlusion (Boos, 1962). For diagnosis and treatment of extensive problems, a reproducible record was necessary.

Specifically, interocclusal records were utilized in the following situations:

1.) In the registration of centric relation, an interarch relation, (A) for diagnosis and treatment of natural teeth by means of articulated casts, and (B) in the diagnosis and treatment of edentulous or partially edentulous patients by means of occlusal rims mounted on
an articulator (Wirth, 1971).

2.) In the registration of centric occlusion, an interocclusal record. This was an important objective in the fabrication of single crowns or fixed partial dentures in which the occlusal position and tooth form existing in the patient were considered to be within physiologic limits and were to be maintained (Braly, 1972).

The most important aspect of transferring a patient's characteristics from the clinical environment to a laboratory situation is, then, proper duplication of the relationship between the maxillary and mandibular arches.

Brotman (1960) stated an error was introduced at the occlusal surfaces of the teeth when the relationship of both casts to each other and to the mechanical hinge of the articulator did not coincide with the relation of both jaws to each other and to the anatomic hinge. If this relationship was recorded inaccurately, the articulated movements of the casts would be incorrect. This resulted in (1) faulty diagnosis and treatment planning, and (2) incorrectly fabricated restorations (Kingery, 1959).

Kingery (1959) stated there were two sources of error in bite registration: the first was technical, and the second of patient origin. The potential for both these types of error can be reduced by choosing an adequate registration material. According to Berman (1960) the
material used had much to do with the resultant accuracy of the record. Authorities agreed the ideal registration material should possess certain properties.

1. It should offer no resistance to closure during registration (Berman, 1960, Silverman, 1960, Boos, 1972, Granger, 1963, Skurnik, 1969). Richter (1972) stated the material should have no viscosity that would cause the mandible to be displaced.

2. It should be easy to handle (Richter, 1972, Wirth, 1971).

3. The ideal material should have reproducible accuracy (Skurnik, 1969).

4. Berman (1960) stated the material should be rigid when set. However, Douglas (1975) suggested the ideal material should be resilient when set.

5. The material should set in less than two minutes to avoid muscle fatigue (Berman, 1960).

6. The ideal registration should be fairly hard when set to eliminate warpage in trimming the registration and in storage (Richter, 1972).

7. Berman (1960), Wirth (1971), and Skurnik (1969) believed sharp detail must be recorded. However, Richter (1972) suggested the material not be so accurate in recording occlusal detail that stone casts would be prevented from fully seating.
8. It should be dimensionally stable for storage purposes (Richter, 1972). Wirth (1971) suggested adequate rigidity or plasticity was necessary for making, removing, handling, and storing the bite registration.

9. The material must be non-toxic; it can not adversely affect the tissues involved in the recording (Wirth, 1971).

Materials historically utilized for bite registration have been: Impression plaster of Paris, impression compound, a variety of waxes, and zinc oxide-eugenol paste. They have been used with and without a carrier system depending on the type of registration desired. None have proved to be the ideal material. Recently, a non-eugenol zinc oxide paste, silicone and polyether systems have been developed as impression materials and are now being marketed as registration materials. A review of the literature of the physical properties and handling characteristics of these materials follows.

Impression Plaster of Paris

Historically, impression plaster had been used as a bite registration material. Its chief constituent was calcined calcium sulfate hemi-hydrate. On mixing with water, this reacted to form a rigid mass of calcium sulfate dihydrate (Combe, 1975).

Berman (1960) found plaster (1) flows readily,
(2) it broke easily because it was mixed thin for the registration, and, (3) lacked adherence.

Skurnik (1969) noted working with plaster was complicated and not conducive to a neat and clean field of operation. Further, if undercuts were present, plaster would fracture upon removal from the mouth. Craig (1975) noted the popularity of dental impression plaster had waned with the introduction of the elastic materials.

**Waxes**

Millstein (1971), Kapur (1957), Gysi (1960), and Schyler (1959) found waxes to be inadequate because the repetitive accuracy or precision was unsatisfactory.

The coefficient of thermal expansion of waxes was higher than any other dental material, and therefore, was significant potential source of error in dental procedures. The wax was shrunk appreciably on cooling from its solidification temperature to room temperature.

Shannahan (1960) found baseplate wax and impression wax to be inaccurate. These waxes demanded excessive occlusal force in recording the bite registration, resulting in inaccurately related arches. Berman (1960), in finding there was 102 to 357 grams of resistance to penetration depending on the type of wax, concurred with Shannahan.

Yet another source of error in wax related interocclusal records dealt with the patient's voluntary and involuntary exertion of nonphysiologic forces during the
procedure. Boos (1962) stated when interference by the resistance of the material or by the operator even adjusting the lips, there was a resistance reaction by the patient. He incised forward of the desired position. The registrations were affected. Gysi (1960), experimenting with various checkbite techniques of recording centric relation, found no two wax registrations alike.

Millstein and Kronman (1971) studied the accuracy of two types of baseplate wax. They studied closing pressures, storage environments, time intervals, and seating pressures. They found: (1) Complete closure under pressure comparable to a clinical setting, 172 p.s.i., was not achieved when wax was present. (2) Storage of the wax registration resulted in distortion. Storage of the record in cold water showed the greatest dimensional change; air cooling produced the least. Also, cooling from mouth temperature to room temperature caused distortion of the record. (3) There was considerable vertical and slight horizontal change when the model was gently placed into a previously formed wax registration. (4) Exact reproduction of the original wax recording was never achieved.

Tylman (1978) stated waxed did not accurately reproduce the incisal and occlusal forms of the teeth. It tended to spread laterally as the teeth closed into the material, not correctly registering the incisal or occlusal form.
Further, he stated waxes had considerable dimensional change caused by any fluctuation in temperature. He felt the texture and nature of wax materials tended to cause the patient to close in an undesirable pattern with the further possibility of its moving the teeth into abnormal positions.

Skurnik (1969) believed wax to be the most versatile recording medium. Properly handled, he felt waxes were valuable recording agents.

**Zinc Oxide-Eugenol**

The basic composition of many zinc oxide-eugenol pastes was the same: Zinc oxide, eugenol and rosin. Plasticizers, fillers, accelerators, and other additives were incorporated to provide the properties desired for the different products.

The principal reaction of zinc oxide-eugenol paste is unknown. However, long sheath-like crystals of zinc eugenolate were identified in the final product. They made up about two percent of the set product (Phillips, 1973).

Wirth (1971) classified interocclusal record setting reactions as (1) chemical and (2) thermoplastic. Chemical reactions occur in plaster of Paris, zinc oxide-eugenol pastes, silicone and polyether materials. Thermoplastic reactions are limited to the various wax materials. It was felt there were difficulties inherent with registration
materials having a chemical setting reaction. (1) These materials were irreversible upon set, therefore, the registration could not be modified. (2) The long setting time permitted registration errors to be introduced. (3) These materials had a tendency to fracture and stick to the teeth. (4) The details of the teeth on the record produced, surpassed the detail of many casts, thereby hindering mounting procedures.

Zinc oxide-eugenol was considered to be dimensionally stable. Craig (1975) found slight shrinkage of .1% or less at the end of thirty minutes after mixing. No significant change occurred at the end of twenty-four hours.

Berman (1960) stated the material of choice for interocclusal records to be zinc oxide-eugenol. (1) The material mixed to a true fluid consistency, (2) offered no resistance to closure, and (3) adhered to its carrier. (4) It set to a hard, noncompressible consistency. (5) It was sharp and easily read. It was felt articulation of the casts could be accomplished accurately, without fear of distortion or compressing the record unevenly in the vertical dimension.

Skurnik (1969) stated zinc oxide eugenol paste was simple to use and posed no storage problem. However, both hands of the dentist were required in the manipulation of the ZOE record. Thus, the dentist could not guide the
mandible into centric relation.

Tylman (1978) stated zinc oxide-eugenol washes were the closest in meeting the requirements for an ideal interocclusal registration material. (1) It remained rigid with little or no dimensional change after setting. (2) It accurately reproduced the incisal and occlusal form. (3) The record could be easily reassembled if broken or damaged.

Strohaver (1972) compared the accuracy of articulator mountings utilizing various interocclusal records. ZOE with the Lucia Jig was found to be the most accurate.

**Non-Eugenol Zinc Oxide Paste**

ZOE pastes and the non-eugenol zinc oxide paste systems seemed to have similar properties. Little has been written about the non-eugenol zinc oxide paste.

Molnar (1974) discussed the virtues of Nogenol, a zinc oxide-eugenol like material, containing no eugenol. Nogenol did not sting, elicited no burning sensation, and exhibited no unpleasant taste or odor. Another positive aspect of Nogenol was, it was non-allergic. Also, Nogenol shared many of the advantages of ZOE paste. It was fluid and offered no resistance to closure. Nogenol was dimensionally stable and could be trimmed with a sharp knife.

**Silicones**

Silicones are synthetic polymers. The chain is composed of silicon and oxygen linked as follows to form a
The length of the siloxane chain determined the molecular weight and thus affected the character of the silicone. As the siloxane chains increased in length, the material became more viscous. Also, cross-linking affected polymer properties, as seen in silicone resins and rubbers (Craig, 1975). A small degree of cross-linking prevented the polymer chains from sliding over one another when the material was stressed. Thus, elasticity was increased (Combe, 1973). Extreme cross-linking affected hardness and chemical reactivity.

Dimensional changes occurred during the setting. Polymerization affected shrinkage as well as the loss of alcohol as the setting reaction proceeded (Philips, 1973). According to the A.D.A. Specification number 19, for elastomeric impression materials, curing contraction should not exceed 0.6% at the end of twenty-four hours. The highly filled putty-like silicones were thought to have less polymerization contraction than the less viscous silicone materials.

Millstein (1975) studied the relationship of the
differential accuracy of silicone-body interocclusal records and weight loss due to volatiles. He found a direct correlation at the $p=.001$ level, between dimensional change and percent of weight loss. The materials tested displayed weight losses over forty-eight hours that were generally proportional to their respective dimensional changes. However, the dimensional change for Citricon was not statistically significant for the 24-48 hour period.

Hertford (1973) found viscosity characteristics were linked to filler content. Silicone putty has a high percentage of filler, up to 75%. The "snap set" of the silicone materials was expressed in the steep rise of its viscosity versus time data. He found polyethers exhibited a similar rise.

Craig (1975) stated silicone putty had a lower strain in compression and may have had lower values for permanent set and dimensional change in twenty-four hours than less viscous silicones. Permanent set was in the range of 1 to 2% and strain values were between 2% and 3%. The dimensional changes in twenty-four hours were as low as $-0.1\%$ or as high as $-0.3\%$. Additional shrinkage occurred up to two or more weeks (Phillips, 1973). Silicones took much longer to reach a maximum contraction than the polyether materials (Chong, 1969). Combe (1975) stated the thermal conductivity of silicone rubber was twice that of ordinary rubber. The
thermal coefficient of silicone is approximately $200 \times 10^6$ per degree Centigrade. Polyether is $220 \times 10^6$ per degree Centigrade. Contraction occurs in cooling the impression from mouth temperature to room temperature.

**Polyether**

Polyether, also an elastomer, was patented in 1969. The elastomeric compound is made from polyethers terminated with amino groups which are cross-linked with strong acids such as aromatic sulfonic acid esters. The cross-linked rubber was reported to have high dimensional accuracy after polymerization and storage (Craig, J.A.D.A., 1977).

Rohan (1970) reported the unrestrained dimensional stability of Impergum to be superior to the silicone impression materials.

Docking (1970) stated polyether materials possessed the highest recovery after deformation. It had the least dimensional change following removal from the mouth.

Wilson (1971) noted heavy bodied elastomers such as silicone putty material were about twice as stable as light-bodied materials in the unstrained state. However, polyether was more stable than the silicone putty.

Causton and Braden (1971-2) found polyether to be highly water absorbent. Phillips (1973) stated silicone was water repellent. However, the polyether polymer absorbed water, a process complicated further by the simultaneous
extraction of the water soluble plasticizer. The result was slight expansion of the material.

Finally, each of the materials studied and their respective chemistry will be discussed. The formulas given are representations of the basic natures of the actual, more involved, patented, commercial formulations.

**Zinc Oxide-Eugenol**

Zinc oxide-eugenol bite registration paste is a paste-paste system. The base paste contains zinc oxide plus a plasticizer, such as olive oil, light mineral oil, or linseed oil. Hydrogenated rosin can also be present. It quickens the set and makes the paste more cohesive. Substitutions are carnauba wax, kauri gum, or coumarone resins.

The catalyst paste contains eugenol with talc or kaolin as a filler to form a paste. Either or both pastes may contain accelerators such as zinc acetate or nitric acid.

The reaction is not known. However, electron photomicrographs did show the presence of zinc oxide surrounded by a matrix of the reaction compound, unreacted eugenol and zinc eugenolate crystals (Phillips, 1973).

Two properties were necessary for the reaction to set: (1) The presence of the orthomethoxy group was essential and (2) if the zinc oxide was completely dehydrated, the mixture did not set. Water (5.0%) in the zinc oxide allowed
for a fifteen minute setting time. It is thought the first reaction in the setting reaction of ZOE is the hydrolysis of zinc oxide to its hydroxide.

Zinc Oxide + Eugenol $\rightarrow$ Zinc Eugenolate

\[
\text{ZnO} + \text{OCH}_3 \xrightarrow{\text{OH}} \text{H}_2\text{C} = \text{HC} - \text{OH}_2
\]

A chelate

Non-Eugenol Zinc Oxide Paste

This reaction produces a product similar to that of the ZOE reaction product. It is formed by a saponification reaction, producing an insoluble soap. The zinc oxide is reacted with a carboxylic acid.

\[
\text{ZnO} + 2 \text{RCOOH} \rightarrow (\text{RCOO})_2\text{Zn} + \text{H}_2\text{O} \quad \text{(Phillips, 1973)}
\]

Silicone Putty

Silicones can be either a paste-paste system or a paste-liquid system. Citricon putty is the latter.

The base paste contains a silicone polymer with terminal hydroxy groups and a filler.

\[
\text{HO} - \left[ \begin{array}{c} \text{CH}_3 \\ \text{Si} - \text{O} \\ \text{CH}_3 \end{array} \right] \quad \text{Si} - \text{OH} \\
\text{CH}_3 \\
n \text{CH}_3
\]

Silicone Polymer
The reaction liquid contains a cross-linking agent. It can be an alkoxy ortho-silicate, a polymer of it, or an organohydrogen siloxane with an activator, an organotin compound.

\[
\begin{align*}
\text{OR} & \quad \text{CH}_3 \begin{bmatrix} \text{CH}_3 \\ \text{Si} & - \text{O} \\ \text{OR} \end{bmatrix} \quad \text{CH}_3 \\
\text{RO} & \quad \text{Si} & - \text{OR} \\
\text{OR} & \quad \text{Si} & - \text{CH}_3
\end{align*}
\]

Alkoxy ortho-silicate  Organohydrogen siloxane

Cross-linking occurs when the setting reaction takes place. Two reactions are possible:

\[
\begin{align*}
-\text{OH} + \text{RO} \quad \text{Si} & \quad \text{OR} + \text{OH} \quad \xrightarrow{\text{CH}_3} \quad \text{Si} & \quad \text{OR} + 3\text{ROH} \\
\text{cross-linking} & \quad \text{agent} & \quad \text{alcohol linked} & \quad \text{polymer} \\
\text{of silicone} & \quad \text{of silicone} & \quad \text{together}
\end{align*}
\]

\[
\begin{align*}
\text{H}_2 \quad \text{H} & \quad \text{Si} & \quad \text{CH}_3 \\
\text{OH} & \quad \xrightarrow{\text{0}} & \quad \text{0} \\
\text{H}_2 \quad \text{H} & \quad \text{Si} & \quad \text{CH}_3 \\
\text{OH} & \quad \xrightarrow{\text{0}} & \quad \text{0}
\end{align*}
\]

2 chains of silicone polymer + organohydrogen siloxane

Alcohol or hydrogen gas is the byproduct of the silicone cross-linking setting reaction.

Polyether

The polyether system is also a paste-paste system. The base paste contains an unsaturated polyether with imine
and groups, a plasticizer and a filler.

The reactor paste contains an aromatic sulphonate, a plasticizer and an inorganic filler. When the two components are combined, a cationic polymerization reaction occurs involving cross-linking of imine groups.

\[
\text{CH}_3 - \text{CH}_2 - \text{CH} - \text{O} - \text{C} - \text{CH}_2 - \text{O} - \text{C}_3\text{NCH}_2\text{CH}_2
\]

\[
\text{CH}_3 \text{C} - \text{CH} - \text{CH}_2 - \text{CH}_3
\]

\[
\text{CH}_3 \text{NCH}_2\text{CH}_2
\]
# TABLE 1

## SUMMARY OF REVIEW OF LITERATURE

<table>
<thead>
<tr>
<th>AUTHOR</th>
<th>MATERIAL</th>
<th>RESULTS</th>
</tr>
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| Berman, 1960       | Impression plaster of Paris | 1. Flowed readily  
|                    |                   | 2. Broke easily because it was mixed thin.  
|                    |                   | 3. Lacked adherence.                                                     |
| Shannahan, 1960    | Wax               | Excessive occlusal force found in recording the bite registration.       |
| Berman, 1960       | Wax               | 102-357 grams of resistance to penetration depending on the type of wax. |
| Gysi, 1960         | Wax               | No two wax registrations were found to be the same.                     |
| Millstein, 1971    | Wax               | 1. Complete closure under pressure comparable to a clinical setting (172 p.s.i.) was not achieved.  
<p>|                    |                   | 2. Wax distorted when stored.                                            |
| Skurnik, 1969      | Wax               | Wax was found to be versatile in that it was a reversible medium.         |
| Berman, 1960       | ZOE               | The material of choice because (1) it mixed to a true fluidity; (2) offered no resistance to closure; (3) adhered to carrier; (4) set to a hard non-compressible consistency; and (5) offered detail. |</p>
<table>
<thead>
<tr>
<th>AUTHOR</th>
<th>MATERIAL</th>
<th>RESULTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strohaver, 1975</td>
<td>ZOE</td>
<td>ZOE with Lucia jig was found to be most accurate method of recording centric relations.</td>
</tr>
<tr>
<td>Millstein, 1975</td>
<td>Silicone</td>
<td>There was a direct correlation between dimensional change and percent of weight loss.</td>
</tr>
<tr>
<td>Hertford, 1973</td>
<td>Silicone</td>
<td>Viscosity was linked to filler content.</td>
</tr>
<tr>
<td>Rohan, 1970</td>
<td>Polyether</td>
<td>Unrestrained dimensional stability of polyether was superior to silicone.</td>
</tr>
<tr>
<td>Docking, 1970</td>
<td>Polyether</td>
<td>Polyether possessed highest recovery after deformation.</td>
</tr>
<tr>
<td>Causton, 1971</td>
<td>Polyether</td>
<td>Polyether was found to be highly water absorbent.</td>
</tr>
<tr>
<td>Stepensky, 1978</td>
<td>Polyether</td>
<td>Absorption of water by polyether caused expansion.</td>
</tr>
</tbody>
</table>
CHAPTER III

METHODS AND MATERIALS

Four materials of different compositions were studied: a zinc oxide-eugenol paste, a non-eugenol zinc oxide paste, a silicone putty and a polyether registration material. Each material was a two component, base-catalyst, system.

The base and catalyst for each material was measured by weight according to the manufacturer's given weight ratio for the two components (Table 2). A centrogram triple beam balance, model 311 (± 0.05 gms.) was used (Figure 1). First, the mixing pad was weighed. An unknown amount of base was squeezed onto the pad, then weighed. The original paper weight was subtracted from this combined paper-base weight to determine the base weight. This base weight was used to determine the amount of catalyst according to the manufacturer's ratio.

After the weight of the base and catalyst had been calculated, the two components of each material were combined according to the manufacturer's specifications. Each material was mixed with a tapered spatula in a back and forth direction, over a wide area of a paper mixing pad. The pad was turned ninety degrees and the mixing motion repeated until an homogenous mixture resulted, within
### TABLE 2

**NAME, BATCH NUMBER, AND MANUFACTURER OF EACH MATERIAL**

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>MANUFACTURER</th>
<th>BATCH NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZOE Bite Registration Paste</td>
<td>Kerr Romulus, Michigan</td>
<td>Base: 81618 Catalyst: 813220A</td>
</tr>
<tr>
<td>Nogenol</td>
<td>Coe Laboratories Chicago, Illinois</td>
<td>Base: 100378 Catalyst: 0638</td>
</tr>
<tr>
<td>Citricoin</td>
<td>Kerr Chicago, Illinois</td>
<td>Base: 3604-09491 Catalyst: 8-04023</td>
</tr>
</tbody>
</table>
FIGURE 1

CENTROGRAM TRIPLE BEAM BALANCE
the manufacturer's specified mixing time.

An undetermined amount of material was carried from the mixing pad to the die (Figure 2). The die produced a sample disc measuring 0.3 cm. in depth by 3.0 cm. in diameter. The material was slowly scraped from the spatula onto the die, within the circular rim. The die and rim were then inverted onto a 2" by 2" square plate glass slab covered with a thin plastic sheet, and hand pressure applied to initially squeeze out the excess material for approximately five seconds. A vice was applied to run through the central axis of the die, plastic and glass slab. The vice was tightened until no more material exuded from the seam between the die rim and plastic covered glass slab (Figure 3).

The die and glass slab were submerged in a Blue M water bath* heated to a constant 32 degrees Centigrade. This was in accordance with A.D.A. specification #19, to simulate mouth conditions. The amount of time the apparatus was left in the bath varied among the products tested. The time for each was determined by the mixing time plus the setting time plus three additional minutes as suggested in A.D.A.specifications #19 (Table 3).

An interval timer was used to determine the time each material was to stay in the bath. These time periods are listed in Table II.

*Blue M Electric Co., Blue Island, Illinois
FIGURE 2

TEFLON DIE
FIGURE 3

DIE AND 2x2 PLASTIC COVERED GLASS SLAB HELD TOGETHER WITH "C" CLAMP
# TABLE 3

**MIXING AND SETTING TIMES, WEIGHT RATIOS OF MATERIALS**

<table>
<thead>
<tr>
<th></th>
<th>Polyether</th>
<th>Silicone</th>
<th>Non-zinc Oxide Paste</th>
<th>Zinc Oxide Eugenol Paste</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mixing time</strong></td>
<td>20-30 sec.</td>
<td>30 sec.</td>
<td>30 sec.</td>
<td>30 sec.</td>
</tr>
<tr>
<td><strong>Working time</strong></td>
<td>2 min.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Initial Set</strong></td>
<td>5 min.</td>
<td>1 min.</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Final Set</strong></td>
<td>5 min.</td>
<td>3 min.</td>
<td>&quot;until hard&quot;</td>
<td>3 min.</td>
</tr>
<tr>
<td><strong>ADA Spec.</strong></td>
<td>3 min.</td>
<td>3 min.</td>
<td>3 min.</td>
<td>3 min.</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>8 min.</td>
<td>6 min.</td>
<td>18 min.</td>
<td>6 min.</td>
</tr>
</tbody>
</table>

**Weight Ratio of Base to Catalyst**

<p>| | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1:0.4</td>
<td>8:1</td>
<td>5:3</td>
</tr>
</tbody>
</table>
When setting was complete, the assembly was removed from the water bath, dried with paper toweling, and the glass slab gently pried from the die. The die rim was then separated from the body of the die and the material gently removed from either the rim or the body, to which it had adhered.

The circular sample was placed into the corresponding recessed area on the microscope table. The distance was measured immediately between two parallel ridges formed from the like negative indentations in the die surface. Also, measurements were taken at one hour, 24 hours, 48 hours, 72 hours and 168 hour intervals. The Gaetner microscope, graduated in 0.01 mm. increments was the measuring device employed* (Figure 4).

Five samples of each of the four materials were made. The distance between the two parallel lines was measured five times per sample for each time interval. This was accomplished by recording the placement of the beginning of the first ridge on the die under the microscope and the placement of the beginning of the second ridge. Measurements were always taken at the beginning of the ridge. The second was subtracted from the first to find the intermediate distance. The distance according to manufacturer's specifications was 2.490 cm. Verification procedures

*Gaetner Scientific Corp., Chicago, Illinois
FIGURE 4
GAETNER TRAVELING MICROSCOPE
proved this value to be improper for the purpose of this experiment. (The die was measured on the same apparatus as the samples.) The determined values, as opposed to the manufacturer's values, was used for all computations.

Temperature and humidity were recorded each day throughout the experiment. The samples were stored separately, on a piece of paper toweling, at room temperature.
CHAPTER IV

RESULTS

The distance between the left and right ridges of each disc was measured five times. Care was taken to repeat the measurement procedures at the center of each disc. This was done to avoid introduction of any improper shrinkage values that may have been brought about by geometrical influences of the sample's non-linear shape.

Only the actual position of the ridges on the discs were recorded at the time of the experiment. Afterwards, the raw data computations were performed. The initial task was the determination of the distance between the two parallel ridges.

Both a simple mean and standard deviation of the five measurements made on each disc at six time intervals, were calculated. The mean of each was considered to represent one "raw" data point for all subsequent analyses.

The standard deviation of each set of five measurements was used to expose any particularly inconsistent measurements. If the standard deviation was, indeed, unusual (arbitrarily, when $S.D. = .0100 \text{ cm.}$) the five readings were analyzed. If a single reading had an unexplained discrepancy in the 4th or 5th (from the right)
significant digit, and such an error did not repeat itself in the other readings at that time interval of that disc, the single reading was discarded. The four remaining measurements were employed for the determination.

The mean and standard deviation for all five sample discs of each material at each time interval were then calculated. Also the distance between the two parallel scribes on the die itself were measured fifteen times. The mean of these measurements was 2.4909 cm. with a standard deviation of 1.86. Therefore, in all subsequent calculating the value for the die was considered to be 2.4909 cm.

The dimensional change of the disc from the die was also calculated as the difference between the die and mean of the five samples for each material (Table 5).

The non-ZOE paste was analyzed at first without using a separating medium between the sample and the die. This resulted in continual problems in cracking and chipping of the samples as they were separated from the die. Since this created an undesirable experimental situation, the determinations of non-ZOE paste were repeated utilizing a releasing agent.* The results of both procedures are reported in Table 4. However, further analysis in this study will omit consideration of the data for ZOE paste without the die release agent.

*Jelenko Pri-Die Mold Release
## Table 4

Mean Batch Data for All Samples
(all measurements expressed in cm.)

<table>
<thead>
<tr>
<th></th>
<th>0 HR.</th>
<th>1 HR.</th>
<th>24 HR.</th>
<th>48 HR.</th>
<th>72 HR.</th>
<th>1 WK.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyether</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M</td>
<td>2.48821</td>
<td>2.48725</td>
<td>2.48337</td>
<td>2.48282</td>
<td>2.48282</td>
<td>2.48114</td>
</tr>
<tr>
<td>SD</td>
<td>.00122</td>
<td>.00120</td>
<td>.00098</td>
<td>.00132</td>
<td>.00132</td>
<td>.00190</td>
</tr>
<tr>
<td>% Shrinkage</td>
<td>.1082</td>
<td>.1464</td>
<td>.3021</td>
<td>.3343</td>
<td>.3243</td>
<td>.3917</td>
</tr>
<tr>
<td>Silicone</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M</td>
<td>2.48606</td>
<td>2.48284</td>
<td>2.47261</td>
<td>2.47181</td>
<td>2.47102</td>
<td>2.46705</td>
</tr>
<tr>
<td>SD</td>
<td>.00286</td>
<td>.00181</td>
<td>.00223</td>
<td>.00163</td>
<td>.00200</td>
<td>.00318</td>
</tr>
<tr>
<td>% Shrinkage</td>
<td>.1442</td>
<td>.3237</td>
<td>.7343</td>
<td>.7664</td>
<td>.7982</td>
<td>.9373</td>
</tr>
<tr>
<td>ZOE</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M</td>
<td>2.47976</td>
<td>2.48123</td>
<td>2.47708</td>
<td>2.47836</td>
<td>2.47226</td>
<td>2.46840</td>
</tr>
<tr>
<td>SD</td>
<td>.00281</td>
<td>.00260</td>
<td>.00205</td>
<td>.00271</td>
<td>.00300</td>
<td>.00224</td>
</tr>
<tr>
<td>% Shrinkage</td>
<td>.4474</td>
<td>.3883</td>
<td>.5547</td>
<td>.5034</td>
<td>.7482</td>
<td>.9033</td>
</tr>
</tbody>
</table>
TABLE 4 (continued)

<table>
<thead>
<tr>
<th></th>
<th>0 HR.</th>
<th>1 HR</th>
<th>24 HR.</th>
<th>48 HR.</th>
<th>72 HR.</th>
<th>1 WK.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-Eugenol</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ZOE with no</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Separator</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M=</td>
<td>2.48311</td>
<td>2.48385</td>
<td>2.48365</td>
<td>2.48591</td>
<td>2.48393</td>
<td>2.48428</td>
</tr>
<tr>
<td>SD=</td>
<td>.0055</td>
<td>.0068</td>
<td>.0062</td>
<td>.0014</td>
<td>.0021</td>
<td>.0014</td>
</tr>
<tr>
<td>Non-Eugenol</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ZOE with</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Separator</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M=</td>
<td>2.48824</td>
<td>2.48791</td>
<td>2.48693</td>
<td>2.48680</td>
<td>2.48687</td>
<td>2.48590</td>
</tr>
<tr>
<td>SD=</td>
<td>.0058</td>
<td>.0047</td>
<td>.0041</td>
<td>.0041</td>
<td>.0047</td>
<td>.0041</td>
</tr>
<tr>
<td>% Shrinkage</td>
<td>.1068</td>
<td>.1201</td>
<td>.1595</td>
<td>.1644</td>
<td>.1619</td>
<td>.2009</td>
</tr>
</tbody>
</table>
**TABLE 5**

COMPARISON OF MATERIALS BATCH MEANS WITH DIE BASED ON "UNRELATED T-TEST", df=5

<table>
<thead>
<tr>
<th>Material</th>
<th>0 HR.</th>
<th>1 HR.</th>
<th>24 HR.</th>
<th>48 HR.</th>
<th>72 HR</th>
<th>1 WK.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Polyether</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T= 3.68589</td>
<td>5.047</td>
<td>12.804</td>
<td>12.156</td>
<td>10.212</td>
<td>8.538</td>
<td></td>
</tr>
<tr>
<td><strong>Silicone</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>ZOE</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Non-Eugenol Zinc Oxide</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>No Sep.</td>
<td>2.370</td>
<td>1.731</td>
<td>1.956</td>
<td>5.794</td>
<td>3.866</td>
<td>7.507</td>
</tr>
<tr>
<td><strong>Non-Eugenol Z.O.-Sep.</strong></td>
<td>.764</td>
<td>1.060</td>
<td>1.600</td>
<td>1.661</td>
<td>1.416</td>
<td>2.025</td>
</tr>
</tbody>
</table>
The means and standard deviations for each material are presented in graphical form in Figures 5 through 8. In order to aid in direct comparison, all four materials are graphically presented in simplified form in Figure 9.

The final determination made from these raw data dealt with the rate of dimensional change. The rate of dimensional change was expressed in terms of average percent change per hour in the sample from the beginning to the end of an experimental time period. The results were tabulated and are presented in Table 9. It is important to note that these figures represent the mean rate of dimensional change for a given time interval. The rate of dimensional change is a convenient parameter in visualizing the level of the shrinkage activity for a material at any given time after mixing. A bar graph representation of these determinations is presented in Figure 10.

In order to facilitate direct comparisons between individual materials at given time intervals, the statistical analysis was performed using multiple "T"-tests. First, a two-tailed "T"-test was employed to compare the samples to the die itself. The results (Table 5) reveal, with the exception of the non-ZOE paste, the values for all the materials were significantly different at a 0.05 level of significance from the die at all time intervals.

Next, two-tailed "T"-tests (between independent means)
FIGURE 5

POLYETHER

MEAN INTRA-SCRIBE DISTANCE vs. TIME
FIGURE 6

SILICONE PUTTY

MEAN INTRA-SCRIBE DISTANCE vs. TIME

+ 1.0 s.d.
FIGURE 7

NON-EUGENOL ZINC OXIDE PASTE

INTRA-SCRIBE DISTANCE (cm.)

2.465

2.470

2.475

2.480

2.485

2.490

1 24 48 72 168

TIME (hours)

MEAN INTRA-SCRIBE
DISTANCE vs. TIME

± 1.0 s.d.
FIGURE 8
ZOE

MEAN INTRA-Scribe DISTANCE vs. TIME
FIGURE 9

ALL MATERIALS

MEAN INTRA-SCRIBE DISTANCE (cm.) vs. TIME (hours)

- Silicone
- ZOE
- Polyether
- Non-eugenol Zinc Oxide

MEAN INTRA-SCRIBE DISTANCE vs. TIME
FIGURE 10

COMPARISON OF ALL MATERIALS IN TERMS OF MEAN DIMENSIONAL CHANGE PER HOUR FOR EACH EXPERIMENTAL TIME INTERVAL

0-1 1-24 24-48 48-72 72-168
TIME INTERVAL (Hours)

P=Polyether
S=Silicone
Z=ZOE
N=Non-Eugenol ZO
### TABLE 9

**RATE OF DIMENSIONAL CHANGE (%/hour)**

<table>
<thead>
<tr>
<th></th>
<th>Immed-1hr.</th>
<th>1-24 hrs.</th>
<th>24-48 hrs.</th>
<th>48-72 hrs.</th>
<th>72-168 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 hour</td>
<td>23 hrs.</td>
<td>24 hrs.</td>
<td>24 hrs.</td>
<td>96 hrs.</td>
</tr>
<tr>
<td>Polyether</td>
<td>-.038</td>
<td>-.007</td>
<td>-.001</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Silicone</td>
<td>-.129</td>
<td>-.018</td>
<td>-.001</td>
<td>-.001</td>
<td>-.002</td>
</tr>
<tr>
<td>ZOE</td>
<td>.059</td>
<td>-.007</td>
<td>-.002</td>
<td>-.001</td>
<td>-.002</td>
</tr>
<tr>
<td>Non-Eugenol Zinc Oxide w/Die Sep.</td>
<td>-.0133</td>
<td>-.0027</td>
<td>.0002</td>
<td>-.0002</td>
<td>-.0004</td>
</tr>
</tbody>
</table>
for every possible combination of pairs of materials for each time interval were performed. The "T"-test values for each such comparison were assembled in chart form (Table 7).

The significance or non-significance of the difference between the means of each pair was determined at the 0.05 level of significance. These results are presented in Table 8.
<table>
<thead>
<tr>
<th>Number</th>
<th>Measurement in Cm.</th>
<th>Mean</th>
<th>S.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.4906</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>2.4904</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>2.4901</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>2.4908</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>2.4908</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>2.4909</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>2.4911</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>2.4910</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>2.4908</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>2.4911</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
**TABLE 7**

**COMPARISON OF ALL MATERIALS WITH EACH OTHER BASED ON "UNRELATED T-TEST"**

<table>
<thead>
<tr>
<th></th>
<th>Polyether df=8</th>
<th>Silicone df=8</th>
<th>ZOE df=8</th>
<th>Non-Eugenol Zinc Oxide df=8</th>
<th>Non-Eugenol Zinc Oxide df=8(Sep.)</th>
<th>Die df=5</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 hr.</td>
<td>1.145</td>
<td>4.599</td>
<td>1.514</td>
<td>.0096</td>
<td>3.689</td>
<td></td>
</tr>
<tr>
<td>1 hr.</td>
<td>3.386</td>
<td>3.509</td>
<td>.823</td>
<td>.224</td>
<td>5.047</td>
<td></td>
</tr>
<tr>
<td>24 hrs.</td>
<td>7.360</td>
<td>4.621</td>
<td>.074</td>
<td>1.394</td>
<td>12.804</td>
<td></td>
</tr>
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<td>2.388</td>
<td>3.035</td>
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<tr>
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<td>5.395</td>
<td>2.062</td>
<td>1.369</td>
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<td>7.131</td>
<td>2.178</td>
<td>1.746</td>
<td>8.538</td>
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<th>ZOE df=8</th>
<th>Non-Eugenol Zinc Oxide df=8</th>
<th>Non-Eugenol Zinc Oxide df=8(Sep.)</th>
<th>Die df=5</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 hrs.</td>
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<td>.797</td>
<td>.561</td>
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<td>.239</td>
<td>1.676</td>
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<tr>
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<td>2.462</td>
<td>2.803</td>
<td>5.077</td>
<td>13.654</td>
<td>12.156</td>
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<td>19.481</td>
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<td>16.507</td>
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<tr>
<td>1 wk.</td>
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<td>12.480</td>
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<th>ZOE df=8</th>
<th>Non-Eugenol Zinc Oxide df=8</th>
<th>Non-Eugenol Zinc Oxide df=8(Sep.)</th>
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<td>2.194</td>
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<td>6.201</td>
<td>11.249</td>
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<tr>
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<td>6.201</td>
<td>11.249</td>
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<td>3.555</td>
<td>11.249</td>
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<td>10.404</td>
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<td>10.404</td>
<td>10.404</td>
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<td>6.188</td>
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T A B L E 7 (continued)

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<th>ZOE df=8</th>
<th>Non-Eugenol Zinc Oxide df=8</th>
<th>Non-Eugenol Zinc Oxide df=8(Sep.)</th>
<th>Die df=5</th>
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<td></td>
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<tr>
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<td></td>
<td></td>
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<td></td>
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</tr>
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<td></td>
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<td></td>
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<td></td>
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<td>1 wk.</td>
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<td>2.025</td>
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</table>
# Table 8

## Comparison of All Materials Evaluated at All Time Intervals

<table>
<thead>
<tr>
<th></th>
<th>Die</th>
<th>Polyether</th>
<th>Silicone</th>
<th>ZOE</th>
<th>Non-Eugenol Zinc Oxide w/Separator</th>
<th>Non-Eugenol Zinc Oxide w/o Sep.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 hr.</td>
<td>X</td>
<td></td>
<td>0</td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1 hr.</td>
<td>X</td>
<td>X</td>
<td></td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>24 hrs.</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>48 hrs.</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>72 hrs.</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1 wk.</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

- Die
- Polyether
- Silicone
- ZOE
- Non-Eugenol Zinc Oxide w/Separator
- Non-Eugenol Zinc Oxide w/o Sep.
<table>
<thead>
<tr>
<th>Time (hrs)</th>
<th>Die</th>
<th>Polyether</th>
<th>Silicone</th>
<th>ZOE</th>
<th>Non-Eugenol</th>
<th>Non-Eugenol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>Zinc Oxide w/Separator</td>
<td>Zinc Oxide w/o Sep.</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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</tr>
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<td>1</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>0</td>
<td>0</td>
<td>X</td>
</tr>
<tr>
<td>24</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>48</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>72</td>
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<td>0</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>1 wk.</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
</tbody>
</table>

X = significant difference at .05 level
0 = no significant difference at .05 level

Based on t-tests between unrelated data pairs
Separate t-tests for each combination
CHAPTER V

DISCUSSION

Four materials, a zinc oxide-eugenol paste, a non-eugenol zinc oxide material, a silicone, and a polyether bite registration material were studied. Their accuracy and stability over a one week period was analyzed.

The results indicated only one material proved to be an accurate registration material. The non-eugenol zinc oxide paste exhibited no significant difference between the die scribes and those of the sample. This initial accuracy proved to be dimensionally stable throughout the week. A slight shrinkage is seen but is not statistically significant.

The non-eugenol zinc oxide material's setting reaction was a saponification reaction, resulting in the production of an insoluble soap (Combe, 1975). This reaction attained a stable equilibrium in a comparatively short period of time. The product was unreactive and therefore stable.

This in part explained the stability. However, close inspection of that data revealed the lack of significant shrinkage was somewhat misleading. The non-eugenol zinc oxide paste data presented with a relatively large standard
deviation. Graphical inspection did illustrate, though, despite the large standard deviation, the data followed a consistently stable trend.

With the polyether, the silicone, and the ZOE, the measurements of the samples all proved to be significantly different when compared to the die. Polyether showed the least difference and ZOE, the greatest.

All materials reflected a pattern of shrinkage throughout the experiment with the exception of ZOE which expanded within the first hour. Thereafter, the ZOE also exhibited contraction deformation.

Polyether was the second most accurate material. Shrinkage was significant within the first twenty-four hours. Thereafter, the contraction curve of the polyether mimicked the non-eugenol zinc oxide product. The polyether curve reflected two properties of that material.

First, polyether had a high co-efficient of thermal expansion. Therefore, there was contraction of the material as it cooled from mouth to room temperature. This affected the immediate reading.

Secondly, the polyether setting reaction was a polymerization reaction. The setting time of the material as stated by the manufacturer and the completion of the reaction did not coincide. Distortion of the registration material resulted as the curing continued past the setting
time (Braden, 1972).

Of note, Craig (1975) stated there was 0.3% shrinkage of polyether at the end of twenty-four hours. This concurred with the present study.

It was of interest that polyether impression materials were hydrophilic. The material absorbed water and expanded. This was countered by the presence of water soluble plasticizers. However, the net result was initial expansion (Stepensky, 1978). There was no immediate expansion seen in the polyether registration material. The material exhibited contraction deformation suggesting the registration polyether was a modification of the impression material.

The greatest shrinkage rate for all materials appeared to be in the first twelve hours after the manufacturer's specified setting times. Only the silicone material reflected a significant shrinkage value after this time. The material continued to shrink at significant levels up to thirty hours. An insignificant rate of shrinkage continued throughout the week.

Simply, silicone was a condensation polymerization. Contraction resulted from both the shrinkage associated with the polymerization reaction and from the production of alcohol as a byproduct. Millstein (1975) in studying silicones found a statistical correlation between dimensional
change and percent weight loss.

Another factor in shrinkage is silicone's high coefficient of thermal expansion. As with polyether, the silicone shrunk as it cooled from mouth to room temperature (Craig, 1975). This distortion was reflected in the initial readings.

Also, Chong (1969) stated silicone had a longer polymerization period than polyether resulting in a sustained contraction period. Shrinkage continued over a two week period. This was born out in the results of this study.

Zinc oxide was the least accurate of the materials tested. It had an initial expansion at the end of the first hour. This was followed by contraction, which at the end of the one week period equalled that of silicone.

Set ZOE exhibited two exotherms in its thermogram. They appeared in the region of the exotherm for zinc eugenolate, indicating the formation of the amorphous zinc eugenolate. More importantly, it can explain the initial expansion of the ZOE material. Different ZOE materials tested by El-Tahawi and Craig (1971) showed the emission of heat from ten to sixty minutes. One peaked at ninty minutes after set.

These findings have clinical implications. Non-eugenol zinc oxide can be used effectively for a bite registration material and indefinitely stored.
Silicone putty is not recommended because of its initial inaccuracy. Its rate of contraction precludes storage of the registration. Registrations of this material could introduce error into the transferred physiologic maxillo-mandibular relation.

Polyether is very similar to the non-eugenol zinc oxide material in accuracy. However, its continued polymerization after set limits its stability and storage.

Limitations of this study should be discussed. A larger sample size might reduce the large standard deviations calculated. Also, there was a high human factor in the use of the microscope. Precise readings were difficult and required a prescribed time for the needed reproducibility.

The teflon disc caused some difficulty. The adhesion of the specimen tested and/or the flow of the material produced an unacceptable disc surface. The resultant accuracy determination was arduous. The ridges on the sample discs were at times indistinct and difficult to read. This problem and that of the microscope could be remedied by linear measurement based on physical contact with the sample as with a micrometer type instrument.
Chapter VI

Conclusion

Four materials: ZOE, non-eugenol zinc oxide paste, a silicone and a polyether bite registration material were studied to determine their accuracy and stability over a one week period. The results are as follows:

1. The non-eugenol paste was the only material in which there was no significant difference between the die and the sample. Non zinc oxide paste offered an accurate reproduction of the die scribes.

2. With polyether, silicone and zinc oxide-eugenol, there was a statistical difference between the die and their respective samples at the immediate reading and through the experiment. Polyether showed the least difference, ZOE the greatest.

3. All materials showed a pattern of shrinkage throughout except ZOE which expanded within the first hour followed, too, by a pattern of contraction.

4. The greatest rate of shrinkage for all materials appeared to be in the first twelve hours
after the set. Only the silicone material reflects a significant shrinkage value after this time. It continued to significantly shrink up to thirty hours with an insignificant rate of shrinkage continuing through the week.
BIBLIOGRAPHY


The thesis submitted by Yvonne Balthazar-Hart has been read and approved by the following committee:

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Professor, Fixed Prosthodontics, Loyola

The final copies have been examined by the director of the thesis and the signature which appears below verifies the fact that any necessary changes have been incorporated and that the thesis is now given final approval by the Committee with reference to content and form.

The thesis is, therefore, accepted in partial fulfillment of the requirements for the degree of Master of Science in Oral Biology.

April 23, 1979  
Date  

[Signature]  
Director's Signature