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## Physical Properties of Dental Base Materials Containing Calcium Hydroxide

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PHYSICAL PROPERTIES OF DENTAL BASE  
MATERIALS CONTAINING CALCIUM HYDROXIDE

by

THOMAS | SARNA

Library -- Loyola University Medical Center

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

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## VITA

Thomas Sarna, the son of Stanley J. Sarna and Genevieve M. (Jamroz) Sarna, was born October 11, 1953, in Evergreen Park, Illinois.

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## INTRODUCTION

Since its inception, dentistry has searched for a restorative material which would provide a painless and permanent replacement for decayed or damaged tooth structure. It is hoped that not only would the material establish a permanent replacement for lost tooth structure, but that it would also protect the remaining vital tooth structure and provide an environment which was non-irritating and palliative in which vital tissue could repair itself. Over the years, a variety of restorative substances have been used, including metals such as tin, gold and copper and other materials such as gutta-percha, porcelain and acrylic resins. To some extent these materials have fulfilled the requirements of replacing lost tooth structure; but, by themselves, they have failed to produce a restoration which establishes an environment that is non-irritating and palliative to the remaining vital tooth.

In order to protect and aid in the recovery process of the traumatized pulpal tissue, various therapeutic substances have been placed under the material used for the permanent replacement of the lost tooth structure. These substances have been grouped

into two basic categories, liners and bases.

A liner is a suspension of a material, usually zinc oxide or calcium hydroxide, in an aqueous or volatile liquid. It is applied to the cavity surface in order to leave a thin film of material.<sup>1</sup> Liners were developed primarily to be used in shallow cavities to neutralize free acid from zinc phosphate and silicate cements. By themselves, they do not have sufficient strength and must be covered by a base material.

A base is usually a layer of cement or a cement-like material which is placed beneath a permanent restoration and is able to withstand the compressive forces which are generated during the placement of the restoration, as well as the compressive forces generated during mastication after the placement of the restoration. The most commonly used bases consist of zinc phosphate cement or zinc oxide-eugenol or a mixture containing calcium hydroxide. The purpose of a base material is two-fold. First, it protects the pulp against additional external insults after or during the placement of the restoration. These insults are thermal shock, mechanical trauma, irritation, galvanic current, microleakage and toxic substances such as acid.<sup>1-4</sup> The second purpose of

a base material is to function as a therapeutic agent, encouraging the repair of injured pulpal tissue and eliminating or preventing postoperative discomfort after placement of the final restoration.<sup>5</sup>

Of the various materials used as bases under permanent restoration, the most commonly used are the hard setting calcium hydroxide products. These calcium hydroxide bases are thought to have several beneficial qualities. They protect the vital pulp from chemical, thermal and bacterial irritation.<sup>6</sup> They stimulate pulpal repair of injured dentin, promote the deposition of additional dentin thereby serving as therapeutic agents and have a positive effect on the clinical success of the permanent restoration.<sup>4</sup>

Calcium hydroxide bases are presently being scrutinized because of their physical properties. Questions have arisen concerning the long term efficacy of calcium hydroxide and its ability to protect vital dentin during acid-etch restorative procedures.<sup>7,8</sup> There is a question as to whether the hard-setting calcium hydroxide containing bases possess sufficient strength to resist masticatory forces under a permanent restoration. To answer these questions, this work will study those

physical properties of calcium hydroxide which would influence the longevity and protective ability of calcium hydroxide. Specifically, these properties are compressive strength, water solubility and acid solubility.



## LITERATURE REVIEW

The use of calcium hydroxide in dentistry was first advocated by Hermann in 1930.<sup>9</sup> He used a commercial brand of calcium hydroxide named calxyl<sup>10</sup> which was mixed with distilled water and used as the therapeutic agent for a pulpotomy procedure performed on deciduous and young permanent teeth.<sup>11</sup>

Zander, in 1939, found that both calxyl and pure calcium hydroxide mixed with distilled water produced the same results when placed on an exposed dental pulp. The result of their placement on vital pulpal tissue was the formation of an amorphous calcified layer on the exposed pulpal surface which then formed a matrix for regular dentin formation and resulted in a bridge of hard material covering the exposed pulp.<sup>12</sup> Zander believed that the calcium ions from the therapeutic agent diffused into the pulp and entered into the formation of the new dentin roof.

Marmase, in 1946, used calcium hydroxide mixed with distilled water for pulp capping procedures on exposed permanent teeth. His study reported that when a mixture of calcium hydroxide and water was applied to human pulpal tissue, a success rate of 85% was achieved.<sup>13</sup>

In 1949, Glass and Zander reported a true healing

of vital exposures treated with calcium hydroxide and distilled water. This was shown histologically by a calcific barrier formation after capping a pulp exposure.<sup>14</sup> Cohen also found a clinical success rate of 79% in 239 cases using calcium hydroxide mixed with anesthetic solution as a direct pulp capping agent in 1951.<sup>15</sup>

The indirect pulp cap technique was first introduced by Hess in 1950.<sup>16</sup> He described a technique where a therapeutic material was placed in deep cavities in which the removal of all decay would expose the dental pulp. Various therapeutic materials were studied which included calcium hydroxide and water, zinc oxide-eugenol paste, serum derived calcium and water and dentinoid (a mixture of 60 parts calcium phosphate, five parts thymol, three parts thymol iodide and 10 parts of resin for a binder mixed with eugenol to make a paste).<sup>10</sup> Of these, the most successful results were achieved by using calcium hydroxide and water.<sup>16</sup>

Calcium hydroxide's pulpal protective properties were investigated by Berk in 1950. Using an aqueous solution of calcium hydroxide and methylcellulose, he demonstrated that this therapeutic agent when placed under a silicate restoration protected the dental pulp.<sup>17</sup> He

concluded that a calcium hydroxide containing base placed in a deep cavity before inserting a silicate restoration would protect the pulp from chemical irritation.

Massler also reported a method of management of deep caries which was similar to that of Hess. His method of managing deep carious lesions was to leave in place a small amount of tooth decay which lay over the dental pulp. In cases of actual or suspected pulp exposure, a mixture of calcium hydroxide and sterile water was first placed and then covered by a mixture of zinc oxide-eugenol. At a later date, the layers of calcium hydroxide and zinc oxide-eugenol were removed, and it was found that the area of the exposure or near exposure had been sealed with secondary dentin.<sup>18</sup>

Nyborg, in 1955, compared calcium hydroxide bases to various inert materials and found that a calcium hydroxide base was more successful in pulp healing.<sup>19</sup>

Again, in 1958, Nyborg used a calcium hydroxide base as a pulp capping material in an in vivo test and had a success rate of 86% in 222 trials.<sup>20</sup>

Sowden, in 1956, demonstrated a technique in which only a portion of a deep carious lesion was excavated and then based with a mixture of calcium hydroxide and sterile water. This mixture was placed over the remaining decay

in order to arrest the existing caries and stimulate favorable dentin changes. In fact, he showed via radiographs that soft carious dentin will remineralize when covered with calcium hydroxide.<sup>21</sup>

In 1958, Berk used calcium hydroxide thickened with methylcellulose as a therapeutic agent under deep restorations. He placed calcium hydroxide in the deepest area of the cavity preparation and then placed a permanent restoration. In recall examinations he found a high percentage of cases with increased radiopacity beneath the base which was believed to be new dentin formation below the base. He was credited for introducing calcium hydroxide as a thermal insulating base.<sup>17</sup>

The osteogenic potential of calcium hydroxide was also tested for the first time in 1958. Pellets of calcium hydroxide in methylcellulose paste along with pellets of other test materials were implanted into the backs of rats. The formation of a heterotropic ossification or calcification was only found consistently around the implanted pellets of calcium hydroxide methylcellulose paste, and in some instances this calcified material closely resembled bone. The only other material which gave any evidence of calcification around it were pellets of Plaster of Paris, and then calcification occurred in

less than one half of the specimens.<sup>22</sup>

Klein reported an increase in radiopacity in the dentin of teeth which had previously been restored with amalgam and a base of calcium hydroxide and methyl-cellulose. He reported this increased radiopacity indicated an increase in mineralization of dentin between the calcium hydroxide and the pulp by using a densitometric evaluation.<sup>23</sup>

The term "reconstructed dentin" was coined by Dalmale,<sup>24</sup> who used a paste of calcium hydroxide and sterile water as an indirect pulp cap. Two months after placing a calcium hydroxide containing base and then a permanent restoration on a vital tooth, "reconstructed dentin" had formed under the calcium hydroxide containing base. At a six month check-up examination, radiographic evidence demonstrated additional dentin formation under the calcium hydroxide containing base in 90% of the cases. He reported a 100% clinical success rate at the six month period with this technique.<sup>24</sup>

Law and Lewis studied the effect of calcium hydroxide in deep carious lesions "where pulpal exposures were highly probable but not actually visible on the roentgenogram".<sup>25</sup> The carious lesions were excavated without exposing the pulp and a layer of calcium hydroxide

and sterile water were placed over the remaining caries followed by placement of an amalgam restoration. Within a seven day period, they found radiopaque areas on the pulpal side of the caries. After six months, they removed the amalgam, calcium hydroxide and remaining caries and proceeded to restore the teeth. Frequently, they found a hard homogeneous semitransparent textured layer below the residual caries. After a two-year period, they reported 76% of the teeth had no pulp exposures.<sup>25</sup>

Mehlum selected 26 permanent molars from young patients with large carious lesions. After partial excavation of the decay, he placed a calcium hydroxide and sterile water paste over the remaining decay followed by a temporary restoration of zinc oxide-eugenol cement. After a three-month post-operative period the teeth were re-excavated removing the zinc oxide-eugenol, the calcium hydroxide and the caries. In 80% of the cases additional dentin had formed and no pulp exposure was evident.<sup>26</sup>

Klein, in 1961, studied deciduous teeth restored with and without the use of a calcium hydroxide containing base under an amalgam restoration. In those teeth restored utilizing a calcium hydroxide containing base, it was demonstrated radiographically that additional dentin was deposited in 93% of the cases. In the teeth restored

without using a calcium hydroxide containing base, only one per cent of the cases demonstrated additional dentin deposition.<sup>27</sup>

Sawusch, using a commercial calcium hydroxide base as a direct pulp capping agent experienced an 85% success rate in 142 cases.<sup>28</sup>

Delaney used a commercial hard-setting calcium hydroxide material (Hydrex) as sole base. Using Hydrex as both a base and a direct pulp capping agent he reported an 88% clinical success rate in 354 cases which had pulp exposures.<sup>29</sup>

Mohammed examined histologic sections of the pulps of dogs one week after the insertion of a base into a deep cavity preparation. The bases used in his study were commercial preparations of zinc phosphate cement, zinc oxide-eugenol cement and a calcium hydroxide containing base. In those cases which did not present with a pulp exposure there was no pulpal injury demonstrated after one week with any of the bases. In those teeth where pulpal exposures were present, calcium hydroxide base proved to be the least irritating of the materials tested. Zinc oxide-eugenol cement caused tissue destruction 80% of the time, while zinc phosphate cement consistently caused pulp necrosis. It was Mohammed's recommendation that all

deep cavities be based with a calcium hydroxide material to protect against the occurrence of an undetected exposure.<sup>30</sup>

Mjor, in 1961, used non-carious human teeth to determine the effect of a calcium hydroxide base and amalgam on dentin. He prepared non-carious teeth of children and young adults and then placed amalgam with no base or amalgam with a calcium hydroxide base. It was demonstrated that the dentin under the cavity preparations covered with a calcium hydroxide base and amalgam showed a marked increase in microhardness, whereas the dentin covered only with amalgam showed no change. Mjor termed this increase in microhardness "secondary intradentinal mineralization". This increase in mineralization to a large extent is accounted for by additional mineral deposition in the walls of the dentinal tubules or intratubular mineralization.<sup>31</sup>

The effect of zinc oxide-eugenol, calcium hydroxide, and amalgam on human dentin was demonstrated by Mjor in 1963. He found that dentin covered with a calcium hydroxide base and amalgam had an increase in mineralization, but there was no increase in the predentin layer. In the zinc oxide-eugenol and amalgam covered dentin there was no increase in mineralization, but there was an



increase in the predentin layer. In the amalgam covered dentin with no base there was no increase in either mineralization or thickness of the predentin layer, but severe pulp changes were noted.<sup>32</sup>

Again in 1968, Mjor and Furseth demonstrated that when human dentin is covered with a calcium hydroxide base there is an increased mineralization inside of the dentinal tubules. This was an intratubular calcification with no change in the intratubular or peritubular dentin and frequently resulted in an obstruction of the tubule by mineral crystals.<sup>33</sup>

Brannstrom verified this occlusion of the dentinal tubules by using a scanning electron microscope. On human teeth previously based with a calcium hydroxide containing material, the scanning electron microscope revealed various degrees of constriction of dentinal tubules directly under the area covered with the calcium hydroxide containing base.<sup>34</sup>

The source of calcium for repair and formation of new dentin-like hard tissue was originally postulated by Zander to be derived directly from the calcium hydroxide base.<sup>12</sup> Sciaky and Pisanti, using radioactive calcium chloride, were the first to discover that the calcium ions necessary for the calcification of new dentin did not come

from the base material, but were derived from the pulpal blood supply.<sup>35</sup>

In 1964, Pisanti and Sciaky, using autoradiography, showed that the calcium ions which were incorporated into new dentin formations came directly from the blood serum.<sup>36</sup> The postulate that calcium utilized in the formation of new dentin does not originate from the calcium hydroxide base was further verified by Attalla and Noryain in 1969.<sup>37</sup>

Using autoradiography, they confirmed that the calcium ions incorporated in the formation of new dentin does not come from the calcium hydroxide containing base. They did note, however, that the calcium ions from the base may diffuse through the dentinal tubules.<sup>37</sup>

Holland also demonstrated that calcium ions incorporated in calcium hydroxide base material could reach the pulp from the base. He used a mixture of calcium hydroxide and distilled water as a direct pulp capping agent in dogs. The calcium hydroxide and water used as a direct pulp capping agent produced a superficial necrotic area followed by a large layer of granulation tissue. Amongst this granulation tissue were tiny granules which in part were made up from the calcium which originated from the calcium hydroxide base which was utilized as a pulp capping material.<sup>38</sup>

Schroder and Granath also studied the histologic effect of a paste of calcium hydroxide and water on human pulps. They applied calcium hydroxide to amputated pulpal tissue and found that the calcium hydroxide with its pH of 12 caused a multilayer necrosis. These layers consisted of three distinct zones. Zone I, the most superficial, consisted of compressed tissue due to pressure and edema from Zone II. Zone II showed edema and liquefaction necrosis due to the severe chemical injury from the high pH. Zone III, the deepest layer, consisted entirely of coagulation necrosis. These multilayers of necrosis were due to a mechanico-chemical effect. It was felt that the calcifying and matrix forming effect of calcium hydroxide was not due to any unique property of calcium hydroxide.<sup>39</sup> Instead, any mild irritation such as that produced by Zone III, as long as it was devoid of infection, would stimulate the underlying tissue of the pulp to produce new border tissue which assumes the appearance of dentin.

Ramussen and Mjor also concluded that the mineralizing effect of calcium hydroxide was not a unique effect due to the material. By placing calcium hydroxide pellets alone and in various diffusion chambers subcutaneously in rats, they found the mineralization

which occurred around the implant was not due to the calcium hydroxide, the calcium ions, the hydroxide ions or the high pH (12.5). Instead, the effect of hard tissue formation would have occurred in any case due to mechanical or other stimuli, and the role of calcium hydroxide was to create a favorable milieu for hard tissue formation.<sup>40</sup>

Stanley, studying pulpotomies performed with calcium hydroxide containing bases found that different results occurred when various commercial materials were used.<sup>41</sup> Using Dycal (LD Caulk Co.), a chemical cautery occurred at the site of placement. This tissue was mummified and then resorbed. A calcified bridge was then formed directly against the Dycal. When Pulpdent (52.5% calcium hydroxide in aqueous methylcellulose) was used, tissue mummification also occurred, but the calcified bridge occurred at the junction of the mummified tissue and the vital pulp. It was also noted that unlike other materials, Dycal caused no delay in the formation of a calcified bridge, and that calcium hydroxide may penetrate to various levels of pulpal tissue, thus introducing mineralized bridge formation in stalactite form, thus jeopardizing the coronal pulp vitality.<sup>41</sup>

Stanley showed that the type, location and degree of pulpal calcification due to calcium hydroxide may be

due to the composition of various commercial preparations.<sup>41</sup> Shubich showed that commercially prepared calcium hydroxide bases have widely varying percentages of calcium hydroxide. This does not mean that all the calcium hydroxide contained in a material is available as a therapeutic agent. Depending upon the ingredients of the base, fillers, binders and catalyst, the amount of available calcium and hydroxide ions may vary greatly from one material to another.<sup>42</sup>

Stanley<sup>41</sup> and also Pereira<sup>43</sup> noted that mineralized bridge formation suggested by radiographic exams are not always confirmed by histologic exams. This inconsistency between radiographic and histologic evidence may be due to a phenomenon known as Fresnell Diffraction, which causes an increase in radiographic density at the edges of the preparation.<sup>44</sup> Thus, an increase in radiographic density nearest the cavity preparation may not in fact be an increase in mineralization, but may instead be a radiographic artifact which mimics an increase in density.

Torneck and Wagner<sup>45</sup> observed that calcium hydroxide, when used as a cavity base, stimulated pulpal repair and promoted cell mitosis. The stimulating effect may be associated with local tissue pH changes and the

presence of calcium ions. The mitogenic effect is thought to be produced by low concentrations of calcium ions which stimulate DNA synthesis. Thus, calcium hydroxide's therapeutic effect may promote a greater number of cells to participate in the healing process.

In addition to its mitogenic effect, calcium hydroxide bases have also been shown to be bacteriostatic and bacteriocidal when placed in deep cavity preparations.<sup>46-48</sup>

Trowbridge reported the effects of calcium hydroxide and zinc oxide-eugenol bases on intradental nerve activity. They observed that eugenol is capable of blocking action potentials because it causes the release of potassium ions from the nerve membrane. Calcium hydroxide bases had little effect when tested on sensory nerve conductivity due to the low amount of free calcium ions available. If, however, calcium chloride was added to the calcium hydroxide base, more free calcium ions were available and an inhibition of neuronal impulses frequency occurred.<sup>49</sup> Therefore, if a base possesses an abundance of free calcium ions, sensory nerve transmission may be partially or completely inhibited depending on the amount of free calcium ions.

Via, in 1955, studied 103 cases of pulpotomies performed on deciduous molars using calcium hydroxide as the therapeutic agent. He concluded that over 68 per cent of the treatments performed were failures. Of the teeth that were judged to be failures, over one half exhibited internal resorption with or without periapical involvement, and in one instance, resorption was noted on the unerupted permanent teeth beneath the deciduous tooth on which the calcium hydroxide pulpotomy had been performed.<sup>103</sup>

Weine has stated that calcium hydroxide has the greatest success rate as a pulp-capping agent; however, if this type of therapy fails, it may make routine endodontics difficult or impossible.<sup>104</sup> Calcium hydroxide pulp-caps have been shown to produce heavy depositions of new dentin which may occlude the pulp chamber and produce extensive dentin bridging which makes nonsurgical endodontic treatment impossible.<sup>104</sup>

In addition to being physiologically tolerated by the dental pulp, bases must be able to withstand the compressive forces generated during the placement of the restoration and mastication. They should be relatively insoluble in both the oral environment and in acids used in restorative procedures. Bases should

be easy to mix and set rapidly and still provide a physical barrier for pulpal protection.

Paffenbarger, in 1934, tested various brands of zinc phosphate cement for consistency of mix, adhesive and hydraulic properties, setting time, compressive strength and solubility. In the process of the work, the American Dental Association Specification for Dental Cementing Medium (A.D.A. Specification #8) was established. It is this specification which gives dental cements and bases definite requirements for constancy of mix, time of setting, ultimate compressive strength, disintegration and film thickness.<sup>50</sup>

Harvey, in 1944, studied the acidity of freshly prepared dental cements. His study was unique in that he did not use diluents to obtain and thus influence the initial pH. From his study he found the initial pH of cements to be: zinc phosphate cement 1.71 to 1.75; silicate cement 1.50 to 1.64 and zinc oxide-eugenol cement 8.4 to 9.4. Harvey stated that because some cements had a low initial pH, the dentist had a responsibility for protecting the pulp from this high initial acidity.<sup>51</sup>

Swartz, in 1966, studied the effect of zinc phosphate cement and silicate cement on the dental pulp. She recommended a base of Dycal or Cavitec be placed on



the exposed dentin to prevent the penetration of the acid constituents of the two cements. It was found that both the calcium hydroxide base (Dycal) and the zinc oxide-eugenol base (Cavitec) served as effective barriers against the acid contained in the cements.<sup>52</sup>

The penetration of acid from silicate cements was studied in vivo by Swartz in 1968. Findings show that components of silicate cement may penetrate a considerable thickness of dentin and that by using a protective zinc oxide-eugenol or calcium hydroxide base it was possible to prevent pulpal injury. It was concluded that the most reliable and secure method to protect the pulp from injury from the restorative material is to use an intermediary base of zinc oxide-eugenol or calcium hydroxide that is at least 0.1 mm thick.<sup>53</sup>

Erikson studied the permeability of various materials for the purpose of protecting the dental pulp from the injurious properties of the dental restorative materials. It was found that both Dycal and Copalite were impermeable to acids, while the permeability of Dropsin was significant. There was, however, an increase in permeability after treatment with water in samples of Dycal and Copalite indicating the protective capacity of the lining material can be reduced with time

and contact with moisture.<sup>54</sup>

Plant studied the acidity produced by freshly placed base materials. Of the base materials he studied, he observed that both Dropsin and zinc phosphate cement had a low initial pH after placement. This early acidity was shown to cause severe pulpal changes which could lead to total pulp necrosis.<sup>55</sup>

According to Stanley, composite restorative materials were very irritating to pulpal tissue when placed on exposed vital dentin. The injurious component contained in the composite material has been shown to be methacrylic acid. In order to protect the vital pulp against chemical injury from methacrylic acid, Stanley suggested that no composite material be placed against vital dentin, except in areas where there is sufficient sclerotic dentin blocking the dentinal tubules. These areas are typically found beneath old restorations and in areas of severe abrasion. In restorative areas where sclerotic dentin is absent and less than 1.0 mm. of dentin remains between the restoration and the pulp, a calcium hydroxide containing base should be used. In addition, Stanley stated that if a cleaner or an acid treatment were to be used prior to the placement of the composite material in order to improve retention, these cleaners and acids

must be restricted to the enamel and a calcium hydroxide containing base be placed on all exposed dentinal surfaces.<sup>56</sup> This placement of the base is to be done in order to prevent pulpal injury from either the cleaner or the acid treatment.

The objects of an acid treatment (usually 37% phosphoric acid) of a tooth preparation prior to placement of a composite restoration are to clean chemically the enamel, improve the wettability and capillary attraction of the enamel to the polymer, increase the surface area available to contact the polymer and create micropores into which polymer tags can form.<sup>57</sup> This acid treatment, if allowed to contact dentin, has been shown to have marked effects. It causes the dentinal tubules to widen and become funnel shaped. The acid treatment removes plugs in openings of tubules and widens the peritubular dentin.<sup>58</sup> It allows a bacterial infiltration into almost all tubules below the acid treated area. Hauwers found more bacteria in dentin exposed to acid than in dentin not exposed to acid, and exposure of dentin to acid could cause necrosis of the vital pulpal tissue beneath it.<sup>59</sup>

Presently there is a consensus of how to prevent pulpal injury from acid treatment prior to the placement of a composite restoration. This involves the placement

of a calcium hydroxide base on all dentin surfaces prior to the acid treatment. The calcium hydroxide base neutralizes any excess acid and thereby protects the pulp.<sup>57-59</sup> This technique, however, is not without problems. Dennison noted that a calcium hydroxide base may decrease the etchability of enamel, particularly in areas adjacent to the enamel.<sup>57</sup>

Since acid used to etch enamel can permeate dentin,<sup>60</sup> contact between acid and dentin must be minimized. Retief showed conclusively that commonly used acid etchants (phosphoric acid) caused great pulpal irritation and possible pulp necrosis.<sup>61</sup>

To overcome the pulpal irritating properties of using acid to etch a preparation, the most common means of protection is the placement of a commercial "hard setting" calcium hydroxide product.<sup>62</sup> This calcium hydroxide base serves as a chemical barrier to prevent or reduce irritation from both the acid etchant and the restorative material.

Nachtsheim found that under simulated clinical situations, Procal, a hard setting calcium hydroxide product, does not allow acid penetration and remains intact as a base after exposure to 37% phosphoric acid for 2 minutes. Also tested were the products Procap,

Dycal, Rheocap and Pulp-Cap, and for all these products there was a complete resistance to acid at base thickness of 0.25 mm.<sup>63</sup>

Phillips studied the actual acid solubility of commercial hard setting calcium hydroxide bases in 37% phosphoric acid and found them to vary in degree of solubility. This solubility from greatest to least was found to be Dycal 5.6%; Procal 5.4%; Renew 3.5%; Realit 0.3%; and Life 0.1%. It was also noted that varying the base catalyst ratio also influenced the acid solubility.<sup>62</sup>

Norman in 1959 studied the clinical solubility of various bases and cements. He concluded that the total solubility of a material is markedly influenced by the type and pH of the acid to which they are exposed in the oral cavity. He stated that solubility should not only be tested in distilled water, but also in diluted organic acids such as acetic, citric and lactic acid.<sup>64</sup>

Norman in 1963 studied various cement bases in regards to solubility and their ability to prevent leakage.<sup>65</sup> He found that without an additional restorative material, no cement base tested prevented leakage in a cavity preparation. With regards to solubility, zinc phosphate cement was the most soluble

in acetic acid when compared to zinc oxide-eugenol or silicate cement. An in vivo study was done in 1969 by Norman testing zinc phosphate, silicate cement and zinc oxide-eugenol cement and showed that it never has been established that cement solubility should be done in water or dilute acid to simulate the clinical environment. Laboratory tests are done in distilled water while in the oral environment dilute acids which may increase solubility abound. It was stated that abrasion is also an important factor for clinical solubility and that the amount of cement lost via solubility would vary greatly from patient to patient but would remain constant for the individual.<sup>66</sup>

Wilson and Batchelor showed that eugenol is continuously leached from zinc oxide-eugenol cement. A weak chelate, zinc eugenolate, (which forms the matrix of these materials) decomposes causing the cement to disintegrate. This continued loss of eugenol leaching from the matrix causes the chelate to lose stability. The equilibrium between the chelate, eugenol and zinc oxide is altered. Consequently, the matrix progressively hydrolyzes to eugenol and zinc hydroxide leading to a loss of mechanical strength and disintegration.<sup>67</sup>

Richter's studies of clinical solubility of cement

bases showed that abrasion produced by brushing and mastication are significant factors in cement base degradation if the cement base is exposed to the oral environment. It was demonstrated that if any cement base is exposed to the oral environment, it will eventually be lost.<sup>68</sup>

Mitchem<sup>69</sup> and Osborne<sup>70</sup> examined cement solubility testing procedures in vivo. They concluded that there was little correlation between laboratory and clinical solubility test results. Also there was a poor correlation between the solubility of the same cements in different patients with some patients experiencing more solubility with the same cement than others.

The ability of a base material to withstand compressive force has also been studied. Chong in 1967 stated that the minimum compressive strength a base must have to support amalgam condensation ranged between 100 and 170 psi. It was noted that those bases displaced by heavy condensation forces were also displaced by light force.<sup>71</sup>

Studies on the force an operator exerts upon an amalgam condensing instrument were done by various authors. Taylor in 1930 reported the average operator used 3-4 lbs. of hand pressure to condense amalgam.<sup>72</sup>

Phillips in 1944 stated that the average force was around 4 pounds.<sup>73</sup> Basker in 1968 studied the forces of condensing amalgam into cavity preparations in the mouth. His values for condensation forces were between 0.2 and 11.0 pounds with the mean value being 2.6 pounds. Basker also noted that as the cavity preparation was filled with amalgam there was a consistent decrease in the amount of pressure exerted by the operator.<sup>74</sup> Therefore, the initial increments of amalgam were condensed under greater pressure than those increments which were more superficial.

Plant in 1970 felt the early strength of base materials was most important. He stated that in the selection of a cavity base, it is important to remember the degree of set attained by the material at the time of placement of the restoration and select a base accordingly.<sup>75</sup>

Plant stated that the greatest forces applied to a base material occurred during the placement of the final restoration. The greatest forces applied to a base during the placement of an amalgam occurs during the first four minutes of the amalgam condensation. The amount of pressure transmitted to a base material is directly related to the amount of condensation



pressure used by the operator, the size of the face of the amalgam condensing instrument, the position of the amalgam condensing instrument and the quality, quantity and type of amalgam being placed. If the base is only partially set during amalgam placement, it is then quite possible to displace the base thereby negating its purpose.<sup>76</sup>

Several authors have stated that all bases should possess sufficient strength to withstand the condensation forces of not only dental amalgam and gold foil, but they must also resist the forces of mastication.<sup>76-80</sup>

Vieria studied the different types and sizes of cavity preparations and whether or not the line angles created in the cavity preparation influenced the physical requirements of the base material. The only consistent result which he demonstrated from this study was that amalgam restorations with multiple bases have a reduced fracture strength when compared to those utilizing a single base.<sup>77</sup>

Farah used a computerized dental model which was used to study forces induced in a Class I amalgam restoration when supported by various bases.<sup>80</sup> He demonstrated that the nature of the base influences the fracture of the overlaying amalgam and that amalgams

with multiple bases are more prone to fracture than those with a single base. Farah, however, did state that the base alone was not the only factor related to amalgam fracture, insufficient amalgam depth, sharp anatomical contours in the amalgam surface and premature<sup>80</sup> occlusal contacts were also contributing factors.

Powers also found that the modulus of elasticity of the cement base supporting the restoration determined in part the amount of deflection that may occur at the cement base - restoration interface. If a cement base has a low value of the modulus of elasticity this will result in a greater deflection at the cement base - restoration interface which in turn may lead to failure of the cement base or the restoration.<sup>81</sup>

Farah in 1981 determined that the supporting ability of a base depends on its modulus of elasticity. Therefore, by knowing the value of modulus of elasticity one may be able to determine the failure load as a function of thickness. An increase in the modulus of elasticity translates to an increase in the strength of a base. Therefore, Farah stated a base with a low modulus of elasticity should not exceed a thickness of 0.5 mm, and only materials with a higher modulus of elasticity may be used as thicker bases.<sup>82</sup>

Hormati stated that amalgam restorations are stronger over a base with a high modulus of elasticity indicating that the modulus of elasticity of a material is as important as its compressive strength. Hormati stated that the optimum thickness for thermal insulation of a base is 0.75 mm and at this thickness there is no advantage of using zinc phosphate cement over a hard setting calcium hydroxide cement such as Dycal.<sup>83</sup>

Bryant studied the displacement of various bases during amalgam condensation may result in contact between amalgam and dentin thus negating the insulating properties of the base resulting in hypersensitivity and eventual failure of the restoration if the base is extended to the restoration's margins. It was stated that the fracture of a base after a placement of a restoration is unlikely to effect the restoration.<sup>84</sup>

Lloyd studied the compressive strength of Dycal and found that the material's strength increased with an increase in peripheral constraints. Therefore, materials with low compressive strengths may be used with confidence when external support and confinement exists. By totally constraining a material, free expansion is suppressed and the strength of the material is greatly increased thus the success or failure of a

base material under compression is related to its position and placement in the preparation.<sup>85</sup> Lloyd recommended that cements that were previously regarded as too weak may indeed possess sufficient strength to serve adequately as a strong and resistant base provided there is a measure of constant constraint.

Parameters also indicated as having an effect upon the ultimate strength of a base are its base-catalyst ratio, the presence of moisture during placement, the number of layers of base placed and the temperature at which the base is placed.<sup>84-88</sup>

Akester in 1979 reported that in clinical conditions commercial hard setting calcium hydroxide bases had disappeared under a correctly placed amalgam leaving a void which was indistinguishable from recurrent caries.<sup>7</sup> This clinical phenomenon termed "disappearing Dycal"<sup>7</sup> was also noted by Barnes and Kidd. They felt that the loss of the calcium hydroxide containing base (Dycal) beneath a properly sealed restoration may not affect the prognosis of the tooth or the restoration but it would lead to the dilemma of whether or not to remove an otherwise sound restoration to distinguish between the loss of the base and the recurrence of caries.<sup>8</sup>

In response to the phenomenon of "disappearing

Dycal" several studies have been undertaken to test the physical properties of the commercial hard setting calcium hydroxide containing bases. McComb evaluated five commercial calcium hydroxide bases (Dycal, Procal, Life, Renew and Reolit) for their setting time, compressive strength, water solubility, acid (37% phosphoric acid) solubility and radiopacity of the set cement.<sup>89</sup> She demonstrated that all of the materials tested set within a seven minute period with Dycal and Reolit having the fastest setting time. Dycal had the highest early (ten minutes) compressive strength, but Life had the highest compressive strength after 24 hours. All the materials, except Reolit, showed distinct dissolution when placed in distilled water for 24 hours, with Dycal and Procal being the most soluble. Life and Reolit showed the most resistance to 37% phosphoric acid and Procal demonstrated the least resistance to acid. In respect to radiopacity all of the materials were similar to human dentin.<sup>89</sup>

Hwas in 1984 tested the water solubility, the acid solubility and the compressive strength of four hard setting calcium hydroxide containing bases (Dycal, Life, Procal and Renew).<sup>90</sup> After 24 hours of being submersed in distilled water, it was found that Dycal

was the least soluble material and Renew was the most water soluble material. The water solubility values of Life and Procal fell in between. The acid (37% phosphoric acid) solubility test demonstrated that Life was the least soluble, followed by Dycal and Renew, with Procal being the most soluble in acid. All the materials possessed sufficient compressive strength to withstand the condensation of amalgam, with Life having a 24-hour compressive strength of almost twice that of all the other materials.<sup>90</sup>

Draheim tested the compressive strength of two commercial calcium hydroxide bases (Dycal and Life) in 1985. It was demonstrated that Life had a significantly higher compressive strength than Dycal, even though both had a similar chemical composition. Both Dycal and Life, however, have sufficient compressive strength (greater than 170 psi) to withstand the condensation of amalgam.<sup>91</sup>

## MATERIALS AND METHODS

The materials used in this study are listed below in Table 1. All except Dropsin consisted of a two paste system in collapsible tubes which when mixed together form a hard final product. Dropsin is a powder and liquid system.

TABLE 1  
CALCIUM HYDROXIDE BASE MATERIALS

| <u>MATERIAL</u>          | <u>Batch #</u>     | <u>Base/Catalyst<br/>Ratio</u> | <u>Manufacturer</u>           |
|--------------------------|--------------------|--------------------------------|-------------------------------|
| Life<br>Regular          | 1 1236<br>1309     | 1.0gm./1.0gm.                  | Sybron/Kerr<br>Romulus, Mich. |
| Life<br>Fast Set         | 4 1181<br>4 1100   | 1.0gm./1.0gm.                  | Sybron/Kerr<br>Romulus, Mich. |
| Nu Cap                   | 050583A<br>072883A | 1.0gm./1.2gm.                  | Coe<br>Chicago, Ill.          |
| Dycal Adv.<br>Formula II | 12 2082<br>12 2082 | 1.0gm./1.17gm.                 | L. D. Caulk<br>Milford, Del.  |
| Dropsin                  | 18 8210<br>18 8210 | 0.6gm./0.2ml.                  | Svedia<br>Enkaping, Sweden    |

All products were manipulated according to manufacturer's instructions and were proportioned by weight (0.0001 gm.) or volume (1 ml.).

Modifications of ANSI/ADA specifications Nos. 8 and 9 were used to study the 24 hour water solubility of the hard setting calcium hydroxide base materials<sup>92-93</sup>. All products were proportioned using the ratios in Table 1. Enough material was mixed to slightly over fill a stainless steel ring shown in Figure 1.

To form the specimen, the stainless steel rings were very lightly coated with high vacuum silicone grease (Dow Corning) to facilitate in the removal of the specimen. The specimen ring was then placed on a thin polyethylene sheet backed by a glass plate (50 x 50 x 5 mm.). The material was then mixed and placed into the specimen ring. Another sheet of polyethylene followed by a glass plate were placed directly on top of the specimen ring thus forcing the materials to conform to the inside of the ring and eliminate any voids. Two binder clips were used to hold the apparatus together (Fig. 2).

The apparatus containing the specimens was then transferred to a constant temperature/humidity chamber (Fig. 3) and held at 37° C. and 100% relative humidity for one hour.



Figure 1.

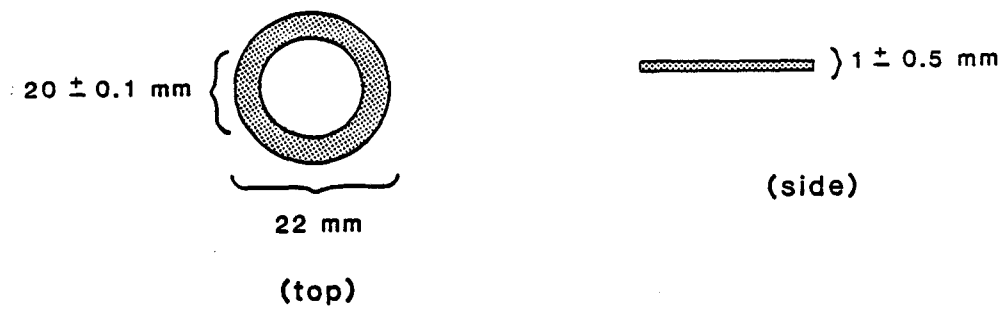


Fig. 1. Stainless Steel Ring

Figure 2

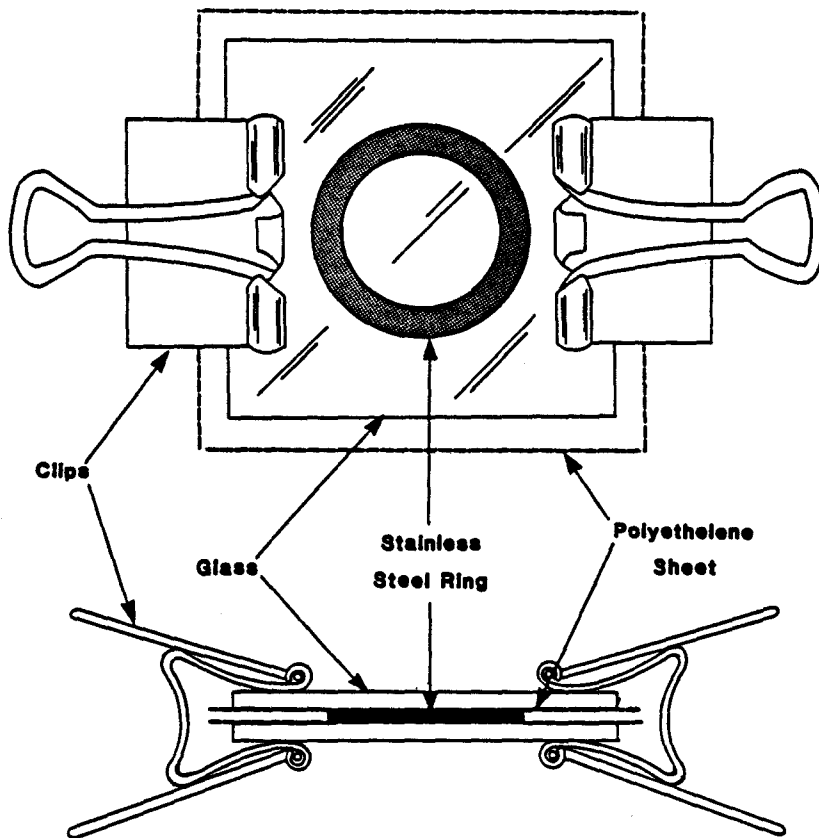


Fig. 2. Speciman Formation Apparatus

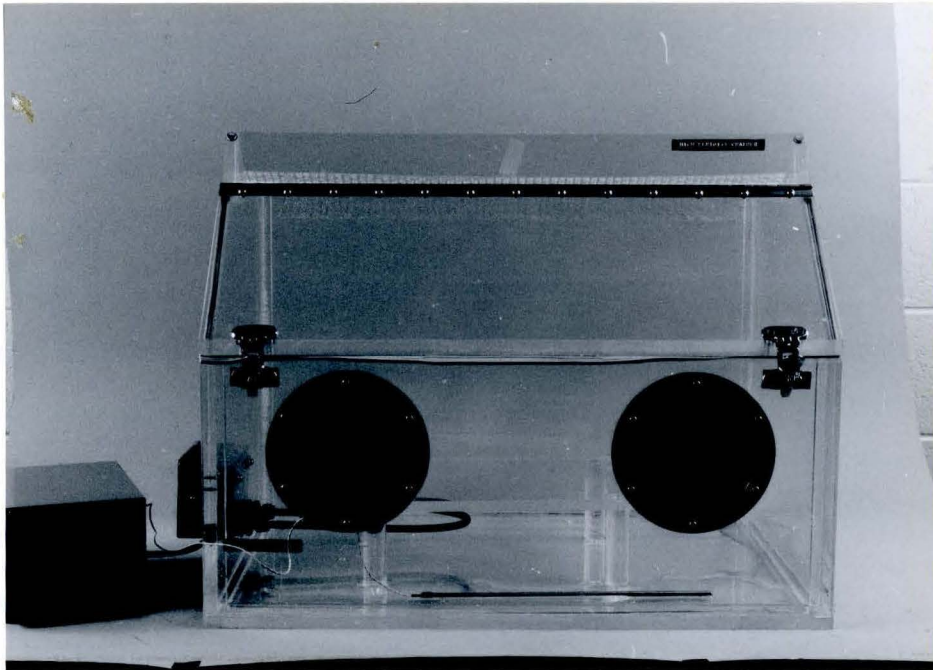


Fig. 3. Constant Temperature/Humidity Chamber

After the one hour period the apparatus was removed from the chamber and the specimens separated from the apparatus. Because of the fragility of one particular product (Dropsin) an additional step was performed to facilitate the removal of the specimen from the stainless steel ring without fracture. This additional step for Dropsin only consisted of placing the material and stainless steel ring in a dessicator for 24 hours before attempting to remove the specimen. The specimens were lightly brushed to remove any adherent particles and were suspended in weighing bottles by 0.030 inch in diameter orthodontic stainless steel wire and weighed to the nearest 0.0001 gm. The weight of the specimen was calculated by:

$$((W_b + W_w) + W_s) - (W_b + W_w) = W_s \quad (\text{Equation 1})$$

or

$$W_t - (W_b + W_w) = W_s$$

Where  $W_t$  = The Combined weight of the Bottle ( $W_b$ ) and the Wire Hanger ( $W_w$ ) and the weight of the Sample ( $W_s$ ).

The samples were then submerged by adding 50 ml. + 1 ml. of de-ionized water to the weighing bottles. The

bottles containing the specimens were then placed back in the constant temperature/humidity chamber and held there for an additional 23 hours.

Following this time period, the samples were removed from the bottles, patted dry with paper towels and stored in coin envelopes.

Several methods were used to evaporate the remaining water and obtain the weight of the residue which had dissolved from the specimens. Initially, evaporation at 110° C. and drying at 150° C. described by Hwas in 1984<sup>90</sup> was used. Constant weight was not achieved due to the continuous thermal break down of the residue at the drying temperature. A similar method using 98° C. to dry the specimens did not achieve constant weight. A third method using a vacuum chamber held at 50° C. and 650 mm. Hg failed to achieve constant weight. At this point it was felt that the samples were being degraded at elevated temperatures.

A fourth trial involved twenty additional samples. After the 24 hour period, 10 samples were placed in a dessicator at room temperature, and 10 samples were placed in a vacuum chamber (Fig. 4) maintained at  $1.5 \times 10^{-2}$  mm. Hg at 37° C. The samples

were maintained in these conditions for fourteen days and weighed at regular intervals. It was found that the samples continued to lose weight throughout the period and failed to achieve a constant weight. Those samples placed in the vacuum lost weight more rapidly than those in the dessicator.

Next, ten samples were made and not submerged in water. Five were placed in a dessicator and five were placed in the vacuum chamber (Fig. 4) set at  $1.5 \times 10^{-2}$  mm. Hg and  $37^{\circ}$  C. It was found that over a two week period these samples continued to lose weight and did not reach a constant weight.

The final technique involved placing the weighing bottle with water and residue in a vacuum chamber at  $37^{\circ}$  C. and 650 mm. Hg. The bottle remained at these conditions until the water was evaporated. Following evaporation, the pressure in the chamber was reduced to  $1.5 \times 10^{-2}$  mm. Hg for a 48 hour period. The weighing bottles were placed in a dessicator and removed periodically until constant weight was achieved.

It was found that similar results could be achieved by eliminating the 48 hour period at  $1.5 \times 10^{-2}$  mm. Hg vacuum. The dry bottles were placed directly into a dessicator at room temperature and periodically

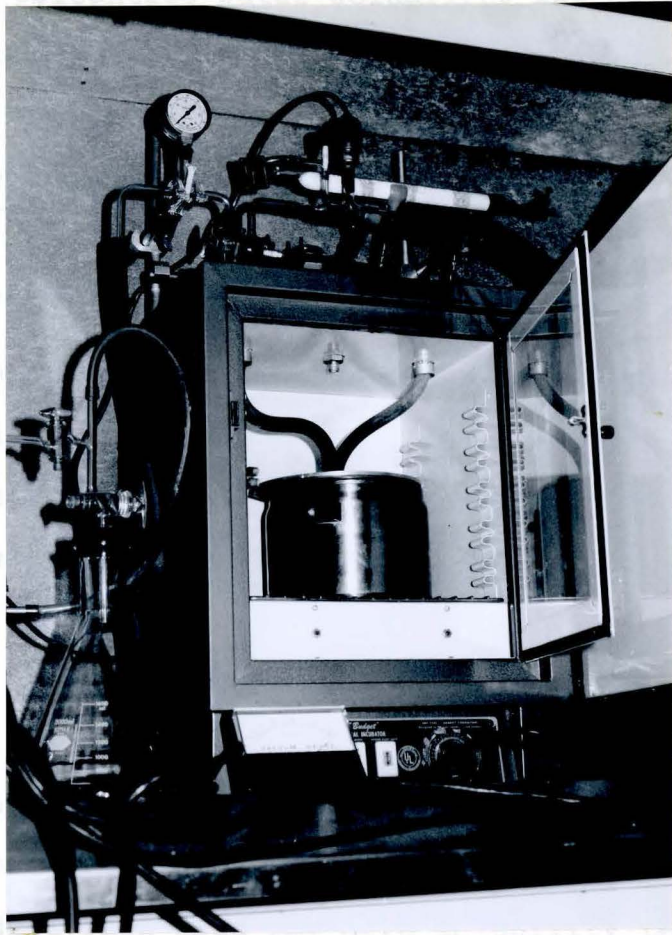


Fig. 4. Vacuum Chamber

removed until a constant weight was achieved.

The percentage water solubility was calculated using Equation 2.

$$\frac{(W_b + W_r) - W_b}{W_s} \times 100 + S_w \quad (\text{Equation 2})$$

Where  $W_r$  = weight of the residue,  $W_b$  = Weight of the Bottle

$W_s$  = weight of the sample and  $S_w$  = percentage water solubility

Ten samples were used for each product and the average water solubility was recorded.

#### ACID SOLUBILITY

Twenty samples of each product were prepared as described for the water solubility test. Ten samples of each product were used for acid solubility testing and ten samples were used for controls. Each sample was weighed to  $\pm 0.0001$  gm.

Control specimens were immersed in 50 ml. de-ionized water for 60 seconds, whereas experimental specimens were immersed in 50 ml. 37% phosphoric acid ( $H_3PO_4$ ) for 60 seconds. On removal from the beaker the specimens were rinsed in a stream of room



temperature tap water for 60 seconds. The samples were then immediately weighed according to a modification of A.D.A. Specification 27.<sup>94</sup>

The acid solubility of each product was determined by the following:

$$\frac{W_0 - W_1}{W_0} - \frac{W'_0 - W'_1}{W'_0} - 100 = S_A \quad (\text{Equation 3})$$

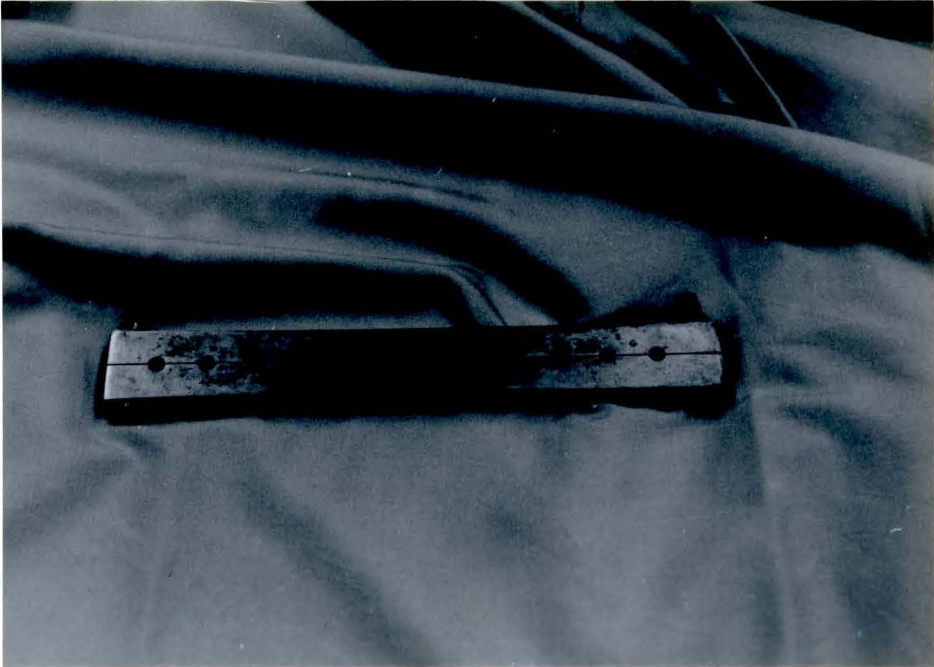
Where  $W$  = original sample weight,  $W_1$  = Sample weight after acid immersion,  $W'_0$  = original control sample weight,  $W'_1$  = control sample weight after immersion and  $S_A$  + Percentage acid solubility.

#### COMPRESSIVE STRENGTH

Specimens were prepared in a split-brass mold resulting in specimens 6 mm. in diameter and 12 mm. high (Fig. 5).

The mold was sprayed with a releasing agent (Miller-Stephenson Release Agent) and placed on a glass mixing slab covered by a thin polyethylene sheet. A mixture of the product to be tested was prepared and then used to slightly overfill the mold. The mold was

was covered by a coated polyethylene sheet and glass  
sliding door and held together by two C clamps. Three  
minutes after the start of the mix the apparatus was  
transferred to a constant temperature/humidity chamber



required to crush the specimens was recorded on a  
Mettler Super Series 1000 series chart recorder. The  
10 x 10 x 10 minute compressive strength an average of  
1000 psi were specimens were prepared.

Specimens prepared for 28 day compressive strength  
were stored in a desiccator for 24 hours. They were  
then removed from the desiccator and placed in the constant  
temperature/humidity chamber (70°F, 50% RH) for a total of 28 days before

Fig. 5. Split-Brass Mold

then covered by a second polyethylene sheet and glass mixing slab and held together by two C clamps. Three minutes after the start of the mix the apparatus was transferred to a constant temperature/humidity chamber (Fig. 3) set at 37° C. and 100% relative humidity.

Removal of the samples was done in two groups. For those samples to be tested for seven minute compressive strength, the mold was removed from the temperature/humidity chamber six minutes after mix and the ends of the specimen cylinders were ground flat under running water with 240 grit SiC metallographic paper. The specimens were then immediately removed from the mold and loaded into an Instron Universal Testing Machine using a compression strain rate of 0.5 in./minute and a 1000 lb. load cell. Force required to crush the specimens was recorded on a Houston Super Scribe 4900 strip chart recorder. For the seven minute compressive strength an average of eight or more specimens were recorded.

Samples tested for 24 hour compressive strength were allowed to remain in the temperature/humidity chamber for one hour after the initial mix. They were then surfaced flat and placed in the temperature/humidity chamber (Fig. 3) for a total of 24 hours before

being loaded and crushed on the Instron Universal Testing Machine. The 24 hour compressive strength was reported as the average of fourteen or more samples.

## RESULTS

WATER SOLUBILITY      The results for the water solubility and the disintegration of the hard setting calcium hydroxide materials; Life Regular Set, Life Fast Set, Nu Cap; Dycal Advanced Formula II and Dropsin are shown in Table 2 through 6. These tables show the specimen, the weight loss of the specimen (gm.) after 23 hours of submersion in water, the percentage of solubility of the specimen in water and a summary showing the mean and the standard deviation (s.d.) of the samples. The results were statistically analyzed using the Neuman-Keuls' multiple range test whose computer program and results are found in Appendix I. The results of this analysis is shown graphically in Figure 6. At  $p < 0.05$  Life Regular Set was statistically different from all other samples. Life Fast Set and Nu Cap were not statistically different. Dycal Advanced Formula II and Dropsin were also not statistically different ( $p < 0.05$ ).

The statistical results of these tests are shown in Tables 27 and 28, the mean and the standard deviation for the percentage of solubility after submersion in water for 23 hours was as follows: Life

Regular Set =  $4.81 \pm 0.66$ ; Life Fast Set =  $3.35 \pm 0.83$ ;  
Nu Cap =  $3.40 \pm 0.66$ ; Dycal Advanced Formula II =  
 $0.97 \pm 0.53$ ; and Dropsin =  $0.87 \pm 0.13$ . Dropsin had the  
lowest solubility while Life Fast Set, Nu Cap and  
Dycal Advanced Formula II fell in between. These  
results are demonstrated in the histogram (Fig. 6.)

ACID SOLUBILITY

The results of the control acid solubility tests and the acid solubility tests on specimens of the hard setting calcium hydroxide materials, Life Regular Set, Life Fast Set, Nu Cap, Dycal Advanced Formula II and Dropsin are presented in Tables 7 through 11.

The control of the material tested demonstrated the weight change of the material when submersed in deionized water for one minute. For all materials, except Dropsin, this represented a loss in weight. Dropsin's control specimen demonstrated a weight gain of 13.38%. In order to calculate the normalized acid solubility the mean value of the control was subtracted from the acid solubility.

The means and standard deviation of the normalized acid solubility specimens are as follows:  
Life Regular Set =  $0.37 \pm 0.10$ ; Life Fast Set =  $0.05 \pm 0.06$ ;  
Nu Cap =  $6.64 \pm 0.20$ ; Dycal Advanced Formula II =  $2.58 \pm 0.25$ ; and Dropsin =  $11.54 \pm 1.39$ . Life Fast Set had the lowest amount of orthophosphoric acid solubility followed by Life Regular Set, Dycal Advanced Formula II and Nu Cap, while Dropsin had the most solubility in acid (Table 12-16).

The data was analyzed by using the Newman-Keuls'

multiple range test (Appendix I). This test demonstrated that Life Regular Set and Life Fast Set were not statistically different ( $p < 0.05$ ) (Table 28) and all other samples were statistically different in the histogram in Figure 7.



EARLY COMPRESSIVE STRENGTH

The results for

the early compressive strength of the hard setting calcium hydroxide materials; Life Regular Set, Life Fast Set, Nu Cap, Dycal Advanced Formula II and Dropsin are presented in Tables 17 through 21. The mean and standard deviation for these materials are as follows: Life Regular Set (15 minutes) = 2577±499 psi; Life Fast Set (7 minutes) = 2092±558 psi; Nu Cap (8 minutes) = 1088±336 psi; Dycal Advanced Formula II (7 minutes) = 1541±376 psi; and Dropsin (7 minutes) = 242±72 psi. Table 27 shows the mean and the standard deviation of the early compressive strength. The data was analyzed using the Newman-Keuls' multiple range tests. (Table 28). All materials were significantly different at  $p \leq 0.05$ .

24-HOURS COMPRESSIVE STRENGTH

The results

for the 24-hour compressive strength of the hard setting calcium hydroxide materials: Life Regular Set, Life Fast Set, Nu Cap, Dycal Advanced Formula II and Dropsin are shown in Tables 22 through 25. These tables show the specimen number, the load at failure, the compressive strength and the mean and standard deviation. The mean and the standard deviation in psi for the materials are as follows: Life Regular Set =  $4372 \pm 1032$ ; Life Fast Set =  $3291 \pm 1651$ ; Nu Cap =  $1357 \pm 200$ ; Dycal Advanced Formula II =  $2330 \pm 680$ ; and Dropsin =  $951 \pm 141$ . Dropsin had the lowest 24-hour compressive strength and Life Regular Set had the highest, while Life Fast Set, Nu Cap and Dropsin fell in between. The data was analyzed using the Newman-Keuls' multiple range test. Life Regular Set and Life Fast Set were not significantly different ( $p < 0.05$ ). Nu Cap and Dropsin were not significantly different ( $p < 0.05$ ), whereas, Dycal Advanced Formula II was significantly different from all the other materials at the 95% level. The results are demonstrated in a histogram (Figure 8).

TABLE 2

Water Solubility of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Life Regular Set |                       |                   |                |                            |
|-------------------------|-----------------------|-------------------|----------------|----------------------------|
| Specimen No.            | Original Weight (gm.) | Weight Loss (gm.) | Solubility (%) | Summary $\bar{x} \pm$ s.d. |
| 1                       | 0.5832                | 0.0282            | 4.84           |                            |
| 2                       | 0.5547                | 0.0329            | 5.93           |                            |
| 3                       | 0.5806                | 0.0236            | 4.06           |                            |
| 4                       | 0.5739                | 0.0300            | 5.23           |                            |
| 5                       | 0.5789                | 0.0277            | 4.79           | 4.81                       |
| 6                       | 0.5632                | 0.0237            | 4.21           | $\pm$                      |
| 7                       | 0.5651                | 0.0305            | 5.40           | 0.66                       |
| 8                       | 0.5679                | 0.0294            | 5.18           |                            |
| 9                       | 0.5722                | 0.0267            | 4.67           |                            |
| 10                      | 0.5639                | 0.0212            | 3.76           |                            |

TABLE 3

Water Solubility of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Life Fast Set |                       |                   |                |                             |
|----------------------|-----------------------|-------------------|----------------|-----------------------------|
| Specimen No.         | Original Weight (gm.) | Weight Loss (gm.) | Solubility (%) | Summary<br>x %<br>±<br>s.d. |
| 1                    | 1.1163                | 0.0492            | 4.41           |                             |
| 2                    | 1.0941                | 0.0419            | 3.83           |                             |
| 3                    | 1.5445                | 0.0343            | 2.22           |                             |
| 4                    | 1.1079                | 0.0509            | 4.59           |                             |
| 5                    | 1.1478                | 0.0441            | 3.84           | 3.35                        |
| 6                    | 1.4291                | 0.0422            | 2.95           | ±<br>0.83                   |
| 7                    | 1.0986                | 0.0368            | 3.35           |                             |
| 8                    | 1.5907                | 0.0412            | 2.59           |                             |
| 9                    | 1.6436                | 0.0382            | 2.32           |                             |
| 10                   | 1.4662                | 0.0505            | 3.44           |                             |

TABLE 4

Water Solubility of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Nu Cap |                       |                   |                |                                      |
|---------------|-----------------------|-------------------|----------------|--------------------------------------|
| Specimen No.  | Original Weight (gm.) | Weight Loss (gm.) | Solubility (%) | Summary $\bar{x}\%$<br>$\pm$<br>s.d. |
| 1             | 1.4491                | 0.0481            | 3.32           |                                      |
| 2             | 1.4597                | 0.0430            | 2.94           |                                      |
| 3             | 1.1326                | -----             | -----          |                                      |
| 4             | 1.5470                | 0.0371            | 2.40           |                                      |
| 5             | 1.1516                | 0.0457            | 3.97           | 3.40                                 |
| 6             | 1.1269                | 0.0454            | 4.03           | $\pm$<br>0.66                        |
| 7             | 1.5591                | 0.0426            | 2.73           |                                      |
| 8             | 1.1277                | 0.0464            | 4.11           |                                      |
| 9             | 1.6605                | 0.0501            | 3.02           |                                      |
| 10.           | 1.0260                | 0.0418            | 4.07           |                                      |

TABLE 5

Water Solubility of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Advanced Formula Dycal II |                       |                   |                |   |
|----------------------------------|-----------------------|-------------------|----------------|---|
| Specimen No.                     | Original Weight (gm.) | Weight Loss (gm.) | Solubility (%) | Summary<br>$\bar{x}$ %<br>$\pm$<br>s.d. |
| 1                                | 1.5819                | 0.0119            | 0.75           |   |
| 2                                | 1.1654                | 0.0124            | 1.06           |   |
| 3                                | 1.1355                | 0.0094            | 0.83           |   |
| 4                                | 1.1299                | 0.0108            | 0.96           |   |
| 5                                | 1.6296                | 0.0393            | 2.41           | 0.97                                    |
| 6                                | 1.4812                | 0.0107            | 0.72           | $\pm$<br>0.53                           |
| 7                                | 1.4642                | 0.0121            | 0.83           |   |
| 8                                | 1.1353                | 0.0099            | 0.87           |   |
| 9                                | 1.1175                | 0.0081            | 0.72           |   |
| 10                               | 1.5470                | 0.0080            | 0.52           |   |

TABLE 6

Water Solubility of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Dropsin |                       |                   |                |   |
|----------------|-----------------------|-------------------|----------------|---|
| Specimen No.   | Original Weight (gm.) | Weight Loss (gm.) | Solubility (%) | Summary<br>$\bar{x}$ %<br>$\pm$<br>s.d. |
| 1              | 1.3357                | 0.0115            | 0.86           |   |
| 2              | 1.3521                | 0.0130            | 0.96           |   |
| 3              | 1.7444                | 0.0120            | 0.69           |   |
| 4              | 1.3218                | 0.0130            | 0.98           |   |
| 5              | 1.2929                | 0.0128            | 0.99           | 0.87                                    |
| 6              | 1.3190                | 0.0121            | 0.92           | $\pm$<br>0.13                           |
| 7              | 1.3080                | 0.0022            | 0.99           |   |
| 8              | 1.6891                | 0.0130            | 0.77           |   |
| 9              | 1.7664                | 0.0113            | 0.64           |   |
| 10             | 1.3062                | 0.0113            | 0.86           |   |

TABLE 7

Acid Solubility Control\* of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Life Regular Set |                       |                     |                 |   |
|-------------------------|-----------------------|---------------------|-----------------|---|
| Specimen No.            | Original Weight (gm.) | Weight Change (gm.) | Weight Change % | Summary<br>$\bar{x}$ %<br>$\pm$<br>s.d. |
| 1                       | 0.5781                | -0.0016             | 0.28            |   |
| 2                       | 0.5657                | -0.0009             | 0.16            |   |
| 3                       | 0.5550                | -0.0002             | 0.04            |   |
| 4                       | 0.5841                | -0.0012             | 0.21            |   |
| 5                       | 0.5708                | -0.0006             | 0.11            | 0.13                                    |
| 6                       | 0.5734                | -0.0008             | 0.14            | $\pm$                                   |
| 7                       | 0.5645                | -0.0011             | 0.19            | 0.09                                    |
| 8                       | 0.5878                | -0.0011             | 0.19            |   |
| 9                       | 0.5660                | -0.0000             | 0.00            |   |
| 10                      | 0.5805                | -0.0000             | 0.00            |   |

\*Weight change due to 1 minute submersion in water.



TABLE 8

Acid Solubility Control\* of Hard Setting Ca(OH)<sub>2</sub> Bases

| Brand: Life Fast Set |                       |                     |                 |                             |
|----------------------|-----------------------|---------------------|-----------------|-----------------------------|
| Specimen No.         | Original Weight (gm.) | Weight Change (gm.) | Weight Change % | Summary<br>x %<br>±<br>s.d. |
| 1                    | 0.5732                | -0.0017             | 0.30            |                             |
| 2                    | 0.5641                | -0.0021             | 0.37            |                             |
| 3                    | 0.5581                | -0.0022             | 0.39            |                             |
| 4                    | 0.5715                | -0.0016             | 0.28            |                             |
| 5                    | 0.5644                | -0.0020             | 0.35            | 0.32                        |
| 6                    | 0.5616                | -0.0017             | 0.30            | ±                           |
| 7                    | 0.5752                | -0.0027             | 0.47            | 0.08                        |
| 8                    | 0.5671                | -0.0016             | 0.28            |                             |
| 9                    | 0.5783                | -0.0011             | 0.19            |                             |
| 10                   | 0.5579                | -0.0013             | 0.23            |                             |

\*Weight change due to 1 minute submersion in water.

TABLE 9

Acid Solubility Control\* of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Nu Cap |                       |                     |                 |                             |
|---------------|-----------------------|---------------------|-----------------|-----------------------------|
| Specimen No.  | Original Weight (gm.) | Weight Change (gm.) | Weight Change % | Summary<br>x %<br>±<br>s.d. |
| 1             | 0.5803                | -0.0008             | 0.14            |                             |
| 2             | 0.5771                | -0.0002             | 0.03            |                             |
| 3             | 0.5929                | -0.0032             | 0.54            |                             |
| 4             | 0.5816                | -0.0004             | 0.07            |                             |
| 5             | 0.5860                | -0.0009             | 0.15            | 0.20                        |
| 6             | 0.5986                | -0.0019             | 0.32            | ±                           |
| 7             | 0.6043                | -0.0012             | 0.20            | 0.14                        |
| 8             | 0.5814                | -0.0011             | 0.19            |                             |
| 9             | 0.5859                | -0.0011             | 0.19            |                             |
| 10            | 0.5801                | -0.0009             | 0.15            |                             |

\*Weight change due to 1 minute submersion in water.

TABLE 10

Acid Solubility Control\* of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Dycal Advanced Formula II |                       |                     |                 |                             |
|----------------------------------|-----------------------|---------------------|-----------------|-----------------------------|
| Specimen No.                     | Original Weight (gm.) | Weight Change (gm.) | Weight Change % | Summary<br>x %<br>±<br>s.d. |
| 1                                | 0.6315                | -0.0008             | 0.12            |                             |
| 2                                | 0.6416                | -0.0019             | 0.30            |                             |
| 3                                | 0.6360                | -0.0012             | 0.19            |                             |
| 4                                | 0.6062                | -0.0010             | 0.16            |                             |
| 5                                | 0.6411                | -0.0011             | 0.17            | 0.20                        |
| 6                                | 0.6388                | -0.0011             | 0.17            | ±<br>0.05                   |
| 7                                | 0.6995                | -0.0018             | 0.26            |                             |
| 8                                | 1.0543                | -0.0521             | 4.94            |                             |
| 9                                | 0.7318                | -0.0016             | 0.22            |                             |
| 10                               | 0.6260                | -0.0013             | 0.21            |                             |

\*Weight change due to 1 minute submersion in water.

TABLE 11

Acid Solubility Control\* of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Dropsin |                       |                     |                 |                                  |
|----------------|-----------------------|---------------------|-----------------|----------------------------------|
| Specimen No.   | Original Weight (gm.) | Weight Change (gm.) | Weight Change % | Summary $\bar{x}$ %<br>±<br>s.d. |
| 1              | 0.8355                | +0.1052             | +12.59          |                                  |
| 2              | 0.7409                | +0.1182             | +15.95          |                                  |
| 3              | 0.7768                | +0.1104             | +14.21          |                                  |
| 4              | 0.7747                | +0.1043             | +13.46          |                                  |
| 5              | 0.7725                | +0.1105             | +14.30          | +13.38                           |
| 6              | 0.8231                | +0.1007             | +12.23          | ±<br>1.58                        |
| 7              | 0.8185                | +0.1082             | +13.22          |                                  |
| 8              | 0.7656                | +0.1135             | +14.82          |                                  |
| 9              | 0.8098                | +0.1032             | +12.76          |                                  |
| 10             | 0.8105                | +0.0830             | +10.24          |                                  |

\*Weight change due to 1 minute submersion in water.

TABLE 12

Normalized\* Acid Solubility ( $\text{H}_3\text{PO}_4$ , 37%) of the Hard  
Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Life Regular Set |                             |                           |                 |   |                               |                             |
|-------------------------|-----------------------------|---------------------------|-----------------|---|-------------------------------|-----------------------------|
| Speci-<br>men<br>No.    | Original<br>Weight<br>(gm.) | Weight<br>Change<br>(gm.) | Solubility<br>% | Normalized<br>Weight<br>Change<br>(gm.) | Normalized<br>Solubility<br>% | Summary<br>- %<br>±<br>s.d. |
| 1                       | 0.5709                      | -0.0030                   | 0.53            | -0.0022                                 | 0.40                          |                             |
| 2                       | 0.5677                      | -0.0037                   | 0.65            | -0.0029                                 | 0.52                          |                             |
| 3                       | 0.5812                      | -0.0026                   | 0.45            | -0.0018                                 | 0.32                          |                             |
| 4                       | 0.5756                      | -0.0029                   | 0.50            | -0.0029                                 | 0.37                          |                             |
| 5                       | 0.5748                      | -0.0026                   | 0.45            | -0.0026                                 | 0.33                          | 0.37                        |
| 6                       | 0.5557                      | -0.0032                   | 0.58            | -0.0032                                 | 0.45                          | ±<br>0.10                   |
| 7                       | 0.5620                      | -0.0032                   | 0.57            | -0.0024                                 | 0.44                          |                             |
| 8                       | 0.5631                      | -0.0016                   | 0.28            | -0.0008                                 | 0.15                          |                             |
| 9                       | 0.5664                      | -0.0031                   | 0.55            | -0.0023                                 | 0.42                          |                             |
| 10                      | 0.5549                      | -0.0022                   | 0.40            | -0.0014                                 | 0.27                          |                             |

\*Weight change due to solubility in  $\text{H}_3\text{PO}_4$  and  
1 minute in water (Table 7).

TABLE 13

Normalized\* Acid Solubility ( $H_3PO_4$ , 37%) of Hard  
Setting  $Ca(OH)_2$  Bases

| Brand: Life Fast Set |                             |                           |                 |   |                               |                                  |
|----------------------|-----------------------------|---------------------------|-----------------|---|-------------------------------|----------------------------------|
| Speci-<br>men<br>No. | Original<br>Weight<br>(gm.) | Weight<br>Change<br>(gm.) | Solubility<br>% | Normalized<br>Weight<br>Change<br>(gm.) | Normalized<br>Solubility<br>% | Summary<br>-<br>x %<br>±<br>s.d. |
| 1                    | 0.5733                      | -0.0020                   | -0.35           | -0.0002                                 | -0.03                         |                                  |
| 2                    | 0.5640                      | -0.0018                   | -0.32           | -0.0000                                 | 0.00                          |                                  |
| 3                    | 0.5792                      | -0.0016                   | -0.28           | +0.0002                                 | +0.05                         |                                  |
| 4                    | 0.5680                      | -0.0023                   | -0.40           | -0.0005                                 | -0.08                         |                                  |
| 5                    | 0.5648                      | -0.0023                   | -0.40           | -0.0005                                 | -0.08                         | -0.05                            |
| 6                    | 0.5807                      | -0.0025                   | -0.43           | -0.0007                                 | -0.11                         | ±<br>0.06                        |
| 7                    | 0.5654                      | -0.0025                   | -0.44           | -0.0007                                 | -0.12                         |                                  |
| 8                    | 0.5623                      | -0.0024                   | -0.43           | -0.0006                                 | -0.11                         |                                  |
| 9                    | 0.5817                      | -0.0016                   | -0.28           | +0.0002                                 | +0.05                         |                                  |
| 10                   | 0.5656                      | -0.0020                   | -0.35           | -0.0002                                 | -0.03                         |                                  |

\*Weight change due to solubility in  $H_3PO_4$  and  
1 minute in water (Table 8).

TABLE 14

Normalized\* Acid Solubility ( $H_3PO_4$ , 37%) of Hard  
Setting  $Ca(OH)_2$  Bases

| Brand: Nu Cap |                       |                     |              |                                |                         |                                   |
|---------------|-----------------------|---------------------|--------------|--------------------------------|-------------------------|-----------------------------------|
| Specimen No.  | Original Weight (gm.) | Weight Change (gm.) | Solubility % | Normalized Weight Change (gm.) | Normalized Solubility % | Summary $\bar{x}$ %<br>$\pm$ s.d. |
| 1             | 0.5950                | -0.0397             | 6.67         | -0.0385                        | 6.47                    |                                   |
| 2             | 0.5859                | -0.0397             | 6.68         | -0.0385                        | 6.48                    |                                   |
| 3             | 0.5756                | -0.0394             | 6.84         | -0.0382                        | 6.64                    |                                   |
| 4             | 0.5891                | -0.0412             | 6.99         | -0.0400                        | 6.79                    |                                   |
| 5             | 0.5755                | -0.0399             | 6.93         | -0.0387                        | 6.73                    | 6.64                              |
| 6             | 0.5856                | -0.0392             | 6.69         | -0.0380                        | 6.49                    | $\pm$<br>0.20                     |
| 7             | 0.5778                | -0.0406             | 7.03         | -0.0394                        | 6.83                    |                                   |
| 8             | 0.5914                | -0.0415             | 7.02         | -0.0403                        | 6.82                    |                                   |
| 9             | 0.6138                | -0.0432             | 7.04         | -0.0420                        | 6.84                    |                                   |
| 10            | 0.6150                | -0.0398             | 6.47         | -0.0386                        | 6.27                    |                                   |

\*Weight change due to solubility in  $H_3PO_4$  and  
1 minute in water (Table 9).

TABLE 15

Normalized\* Acid Solubility ( $H_3PO_4$ , 37%) of Hard  
Setting  $Ca(OH)_2$  Bases

| Brand: Advanced Formula Dycal II |                       |                     |              |                                |                         |                                   |
|----------------------------------|-----------------------|---------------------|--------------|--------------------------------|-------------------------|-----------------------------------|
| Specimen No.                     | Original Weight (gm.) | Weight Change (gm.) | Solubility % | Normalized Weight Change (gm.) | Normalized Solubility % | Summary $\bar{x}$ %<br>$\pm$ s.d. |
| 1                                | 0.6145                | -0.0166             | 2.70         | -0.0102                        | 2.50                    |                                   |
| 2                                | 0.6275                | -0.0151             | 2.41         | -0.0087                        | 2.21                    |                                   |
| 3                                | 0.6327                | -0.0177             | 2.80         | -0.0113                        | 2.60                    |                                   |
| 4                                | 0.6290                | -0.0165             | 2.62         | -0.0101                        | 2.42                    |                                   |
| 5                                | 0.5867                | -0.0180             | 3.07         | -0.0116                        | 2.87                    | 2.58                              |
| 6                                | 0.6239                | -0.0195             | 3.13         | -0.0131                        | 2.93                    | $\pm$<br>0.25                     |
| 7                                | 0.6383                | -0.0175             | 2.73         | -0.0111                        | 2.53                    |                                   |
| 8                                | 0.6003                | -0.0187             | 3.12         | -0.0123                        | 3.92                    |                                   |
| 9                                | 0.6512                | -0.0163             | 2.50         | -0.0099                        | 2.30                    |                                   |
| 10                               | 0.6318                | -0.0174             | 2.75         | -0.0110                        | 2.55                    |                                   |

\*Weight change due to solubility in  $H_3PO_4$  and  
1 minute in water (Table 10).



TABLE 16

Normalized\* Acid Solubility ( $H_3PO_4$ , 37%) of Hard  
Setting  $Ca(OH)_2$  Bases

| Brand: Dropsin |                       |                     |              |                                |                         |                                  |
|----------------|-----------------------|---------------------|--------------|--------------------------------|-------------------------|----------------------------------|
| Specimen No.   | Original Weight (gm.) | Weight Change (gm.) | Solubility % | Normalized Weight Change (gm.) | Normalized Solubility % | Summary<br>-<br>X %<br>±<br>s.d. |
| 1              | 0.7758                | +0.0199             | 2.57         | -0.0858                        | 10.81                   |                                  |
| 2              | 0.7735                | +0.0163             | 2.11         | -0.0894                        | 11.27                   |                                  |
| 3              | 0.8144                | +0.0078             | 0.96         | -0.0979                        | 12.42                   |                                  |
| 4              | 0.7456                | +0.0043             | 0.58         | -0.1014                        | 12.80                   |                                  |
| 5              | 0.7748                | +0.0080             | 1.03         | -0.0977                        | 12.35                   | 11.54                            |
| 6              | 0.7854                | +0.0049             | 0.62         | -0.1008                        | 12.76                   | ±<br>1.39                        |
| 7              | 0.7736                | +0.0013             | 0.17         | -0.1044                        | 13.21                   |                                  |
| 8              | 0.8191                | +0.0242             | 2.95         | -0.0815                        | 10.43                   |                                  |
| 9              | 0.8066                | +0.0367             | 4.55         | -0.0690                        | 8.83                    |                                  |
| 10             | 0.8454                | +0.0240             | 2.84         | -0.0817                        | 10.54                   |                                  |

\*Weight change due to solubility in  $H_3PO_4$  and  
1 minute in water (Table 11).

TABLE 17

Early Compressive Strength of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases  
(15 Minutes)

| Brand: Life Regular Set |                        |                      |         |                          |
|-------------------------|------------------------|----------------------|---------|--------------------------|
| Specimen No.            | Load at Failure (lbs.) | Compressive Strength |         | Summary                  |
|                         |                        | psi                  | (MPa)   | $\bar{x}$ %<br>±<br>s.d. |
| 1                       | 118.8                  | 2712                 | (18.70) |                          |
| 2                       | 128.0                  | 2922                 | (20.15) |                          |
| 3                       | 100.4                  | 2292                 | (15.80) | <u>lbs.</u>              |
| 4                       | 141.0                  | 3219                 | (22.20) | 112.8                    |
| 5                       | 81.6                   | 1863                 | (12.85) | ±                        |
| 6                       | 99.8                   | 2278                 | (15.71) | 21.9                     |
| 7                       | 78.6                   | 1794                 | (12.37) | <u>psi</u>               |
| 8                       | 87.1                   | 1989                 | (13.71) | 2577                     |
| 9                       | 106.4                  | 2429                 | (16.75) | ±                        |
| 10                      | 126.4                  | 2886                 | (19.90) | 499                      |
| 11                      | 140.4                  | 3205                 | (22.10) | <u>MPa</u>               |
| 12                      | 124.6                  | 2845                 | (16.62) | (17.77)                  |
| 13                      | 133.6                  | 3050                 | (21.03) | ±                        |
|                         |                        |                      |         | (3.44)                   |

TABLE 18

Early Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases  
(7 Minutes)

| Brand: Life Fast Set |                        |                      |         |                                     |
|----------------------|------------------------|----------------------|---------|-------------------------------------|
| Specimen No.         | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>$\bar{x}$ %<br>±<br>s.d. |
|                      |                        | psi                  | (MPa)   |                                     |
| 1                    | 99.4                   | 2269                 | (15.64) |                                     |
| 2                    | 142.0                  | 3242                 | (22.35) |                                     |
| 3                    | 132.2                  | 3018                 | (20.81) | <u>lbs.</u><br>91.7                 |
| 4                    | 83.8                   | 1913                 | (13.19) | ±<br>24.4                           |
| 5                    | 83.6                   | 1909                 | (13.16) |                                     |
| 6                    | 111.6                  | 2548                 | (17.57) |                                     |
| 7                    | 97.8                   | 2233                 | (15.40) | <u>psi</u><br>2092                  |
| 8                    | 83.6                   | 1909                 | (13.16) | ±<br>558                            |
| 9                    | 59.6                   | 1361                 | (9.38)  |                                     |
| 10                   | 75.4                   | 1721                 | (11.87) |                                     |
| 11                   | 72.0                   | 1644                 | (11.33) | <u>MPa</u><br>(14.42)               |
| 12                   | 68.8                   | 1571                 | (10.83) | ±<br>(3.85)                         |
| 13                   | 81.6                   | 1863                 | (12.85) |                                     |

TABLE 19

Early Compressive Strength of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases  
(8 Minutes)

| Brand: Nu Cap |                        |                      |         |                                     |
|---------------|------------------------|----------------------|---------|-------------------------------------|
| Specimen No.  | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>$\bar{x}$ %<br>±<br>s.d. |
|               |                        | psi                  | (MPa)   |                                     |
| 1             | 29.7                   | 678                  | ( 4.67) |                                     |
| 2             | 17.0                   | 388                  | ( 2.68) |                                     |
| 3             | 33.7                   | 769                  | ( 5.30) | <u>lbs.</u><br>44.1                 |
| 4             | 47.7                   | 1089                 | ( 7.51) | ±<br>14.8                           |
| 5             | 54.2                   | 1237                 | ( 8.53) |                                     |
| 6             | 22.6                   | 516                  | ( 3.56) |                                     |
| 7             | 50.4                   | 1151                 | ( 7.94) | <u>psi</u><br>1008                  |
| 8             | 46.1                   | 1052                 | ( 7.25) | ±<br>336                            |
| 9             | 44.6                   | 1018                 | ( 7.02) |                                     |
| 10            | 50.9                   | 1162                 | ( 8.01) |                                     |
| 11            | 51.0                   | 1164                 | ( 8.03) |                                     |
| 12            | 74.0                   | 1689                 | (11.65) | <u>MPa</u><br>(6.95)                |
| 13            | 63.0                   | 1438                 | ( 9.92) | ±<br>(2.32)                         |
| 14            | 37.9                   | 856                  | ( 5.96) |                                     |
| 15            | 39.3                   | 897                  | ( 6.18) |                                     |

TABLE 20

Early Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases  
(7 Minutes)

| Brand: Dycal Advanced Formula II |                        |                      |         |                                      |
|----------------------------------|------------------------|----------------------|---------|--------------------------------------|
| Specimen No.                     | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>x %<br>±<br>s.d.          |
|                                  |                        | psi                  | (MPa)   |                                      |
| 1                                | 56.2                   | 1283                 | ( 8.85) |                                      |
| 2                                | 45.1                   | 1030                 | ( 7.10) |                                      |
| 3                                | 77.8                   | 1776                 | (12.25) | <u>lbs.</u><br>67.5                  |
| 4                                | 56.5                   | 1290                 | ( 8.89) | ±<br>16.5                            |
| 5                                | 82.8                   | 1890                 | (13.03) |                                      |
| 6                                | 82.2                   | 1877                 | (12.94) |                                      |
| 7                                | 52.7                   | 1203                 | ( 8.29) | <u>psi</u><br>1541                   |
| 8                                | 86.8                   | 1982                 | (13.67) | ±<br>376                             |
|                                  |                        |                      |         | <u>MPa</u><br>(10.62)<br>±<br>(2.59) |

TABLE 21

Early Compressive Strength of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases  
(7 Minutes)

| Brand: Dropsin |                        |                      |        |                                     |
|----------------|------------------------|----------------------|--------|-------------------------------------|
| Specimen No.   | Load at Failure (lbs.) | Compressive Strength |        | Summary<br>$\bar{x}$ %<br>±<br>s.d. |
|                |                        | psi                  | (MPa)  |                                     |
| 1              | 9.2                    | 210                  | (1.45) |                                     |
| 2              | 9.0                    | 205                  | (1.41) |                                     |
| 3              | 14.1                   | 322                  | (2.22) | <u>lbs.</u><br>10.6                 |
| 4              | 13.7                   | 313                  | (2.16) | ±<br>3.1                            |
| 5              | 9.2                    | 210                  | (1.45) |                                     |
| 6              | 8.7                    | 199                  | (1.37) |                                     |
| 7              | 6.6                    | 151                  | (1.04) | <u>psi</u><br>242                   |
| 8              | 7.5                    | 172                  | (1.19) | ±<br>72                             |
| 9              | 12.3                   | 281                  | (1.94) |                                     |
| 10             | 15.9                   | 363                  | (2.50) | <u>MPa</u><br>(1.67)                |
|                |                        |                      |        | ±<br>(0.50)                         |

TABLE 22

24 Hour Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases

| Brand: Life Regular Set |                        |                      |         |                                     |
|-------------------------|------------------------|----------------------|---------|-------------------------------------|
| Specimen No.            | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>$\bar{x}$ %<br>±<br>s.d. |
|                         |                        | psi                  | (MPa)   |                                     |
| 1                       | 239.0                  | 5457                 | (37.63) |                                     |
| 2                       | 185.5                  | 4235                 | (29.20) |                                     |
| 3                       | 230.0                  | 5251                 | (36.21) |                                     |
| 4                       | 140.0                  | 3196                 | (22.04) | <u>lbs.</u>                         |
| 5                       | 156.5                  | 3574                 | (24.64) | 191.5                               |
| 6                       | 172.0                  | 3927                 | (27.08) | ±                                   |
| 7                       | 200.0                  | 4566                 | (31.48) | 45.2                                |
| 8                       | 204.5                  | 4669                 | (32.19) |                                     |
| 9                       | 151.5                  | 3459                 | (23.85) |                                     |
| 10                      | 127.0                  | 2900                 | (20.00) | <u>psi</u>                          |
| 11                      | 190.5                  | 4349                 | (29.99) | 4372                                |
| 12                      | 127.5                  | 2911                 | (20.07) | ±                                   |
| 13                      | 267.0                  | 6069                 | (42.03) | 1032                                |
| 14                      | 261.5                  | 5970                 | (41.16) |                                     |
| 15                      | 214.5                  | 4897                 | (33.76) |                                     |
| 16                      | 159.0                  | 3630                 | (25.03) |                                     |
| 17                      | 125.5                  | 2865                 | (19.75) | <u>Mpa</u>                          |
| 18                      | 209.0                  | 4772                 | (32.90) | (30.14)                             |
| 19                      | 240.5                  | 5491                 | (37.86) | ±                                   |
| 20                      | 229.0                  | 5228                 | (36.05) | (7.12)                              |

TABLE 23

24 Hour Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases

| Brand: Life Fast Set |                        |                      |         |                      |
|----------------------|------------------------|----------------------|---------|----------------------|
| Specimen No.         | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>x̄ ± s.d. |
|                      |                        | psi                  | (MPa)   |                      |
| 1                    | 211.5                  | 4829                 | (33.30) |                      |
| 2                    | 169.0                  | 3858                 | (26.60) |                      |
| 3                    | 218.5                  | 4989                 | (34.40) |                      |
| 4                    | 121.0                  | 2763                 | (19.05) | <u>lbs.</u>          |
| 5                    | 104.0                  | 2374                 | (16.37) | 144.1                |
| 6                    | 103.5                  | 2363                 | (16.29) | ±                    |
| 7                    | 197.5                  | 4509                 | (31.09) | 72.3                 |
| 8                    | 108.5                  | 2477                 | (17.08) |                      |
| 9                    | 241.0                  | 5502                 | (37.94) | <u>psi</u>           |
| 10                   | 140.5                  | 3208                 | (22.12) | 3291                 |
| 11                   | 211.5                  | 4829                 | (33.30) | ±                    |
| 12                   | 240.0                  | 5479                 | (37.78) | 1651                 |
| 13                   | 134.0                  | 3059                 | (21.09) |                      |
| 14                   | 179.0                  | 4087                 | (28.18) |                      |
|                      |                        |                      |         | <u>MPa</u>           |
|                      |                        |                      |         | (22.69)              |
|                      |                        |                      |         | ±                    |
|                      |                        |                      |         | (11.38)              |



TABLE 24

24 Hour Compressive Strength of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand: Nu Cap |                        |                      |         |                          |
|---------------|------------------------|----------------------|---------|--------------------------|
| Specimen No.  | Load at Failure (lbs.) | Compressive Strength |         | Summary                  |
|               |                        | psi                  | (MPa)   | $\bar{x}$ %<br>±<br>s.d. |
| 1             | 57.6                   | 1315                 | ( 9.07) |                          |
| 2             | 53.7                   | 1226                 | ( 8.45) |                          |
| 3             | 52.9                   | 1208                 | ( 8.33) |                          |
| 4             | 56.2                   | 1283                 | ( 8.85) | <u>lbs.</u>              |
| 5             | 63.5                   | 1450                 | (10.00) | 59.4                     |
| 6             | 65.7                   | 1500                 | (10.34) | ±                        |
| 7             | 63.0                   | 1438                 | ( 9.91) | 8.8                      |
| 8             | 67.1                   | 1532                 | (10.56) |                          |
| 9             | 37.8                   | 863                  | ( 5.95) | <u>psi</u>               |
| 10            | 63.0                   | 1438                 | ( 9.91) | 1357                     |
| 11            | 61.0                   | 1393                 | ( 9.60) | ±                        |
| 12            | 59.0                   | 1347                 | ( 9.29) | 200                      |
| 13            | 67.6                   | 1543                 | (10.64) |                          |
| 14            | 55.0                   | 1256                 | ( 8.66) |                          |
| 15            | 44.8                   | 1023                 | ( 7.05) |                          |
| 16            | 69.4                   | 1584                 | (10.92) | <u>MPa</u>               |
| 17            | 54.2                   | 1237                 | ( 8.53) | (9.36)                   |
| 18            | 55.3                   | 1263                 | ( 8.71) | ±                        |
| 19            | 65.8                   | 1502                 | (10.36) | (1.38)                   |
| 20            | 76.1                   | 1737                 | (11.98) |                          |

TABLE 25

24 Hour Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases

| Brand: Dycal Advanced Formula II |                        |                      |         |   |
|----------------------------------|------------------------|----------------------|---------|---|
| Specimen No.                     | Load at Failure (lbs.) | Compressive Strength |         | Summary<br>$\bar{x}$ ± s.d.   |
|                                  |                        | psi                  | (MPa)   |   |
| 1                                | 118.0                  | 2694                 | (18.57) | <u>lbs.</u><br>102.1<br>±<br>29.8<br><br><u>psi</u><br>2330<br>±<br>680<br><br><u>MPa</u><br>(16.06)<br>±<br>(4.69) |
| 2                                | 94.4                   | 2155                 | (14.86) |   |
| 3                                | 156.6                  | 3575                 | (24.65) |   |
| 4                                | 119.4                  | 2726                 | (18.80) |   |
| 5                                | 114.2                  | 2607                 | (17.98) |   |
| 6                                | 61.6                   | 1406                 | ( 9.69) |   |
| 7                                | 64.6                   | 1475                 | (10.17) |   |
| 8                                | 123.6                  | 2813                 | (19.40) |   |
| 9                                | 73.2                   | 1671                 | (11.52) |   |
| 10                               | 81.6                   | 1863                 | (12.84) |   |
| 11                               | 83.6                   | 1909                 | (13.16) |   |
| 12                               | 100.8                  | 2301                 | (15.86) |   |
| 13                               | 64.6                   | 1475                 | (10.17) |   |
| 14                               | 150.8                  | 3443                 | (23.74) |   |
| 15                               | 139.8                  | 3192                 | (22.00) |   |
| 16                               | 98.0                   | 2237                 | (15.42) |   |
| 17                               | 90.2                   | 2059                 | (14.20) |   |

TABLE 26

24 Hour Compressive Strength of Hard Setting Ca(OH)<sub>2</sub> Bases

| Brand: Dropsin  |                              |                      |        |                                |
|-----------------|------------------------------|----------------------|--------|--------------------------------|
| Specimen<br>No. | Load at<br>Failure<br>(lbs.) | Compressive Strength |        | Summary<br>$\bar{x}$ ±<br>s.d. |
|                 |                              | psi                  | (MPa)  |                                |
| 1               | 41.7                         | 952                  | (6.56) |                                |
| 2               | 42.5                         | 970                  | (6.69) |                                |
| 3               | 42.8                         | 977                  | (6.74) |                                |
| 4               | 42.6                         | 973                  | (6.71) | <u>lbs.</u>                    |
| 5               | 50.6                         | 1155                 | (7.93) | 41.7                           |
| 6               | 42.3                         | 966                  | (6.66) | ±                              |
| 7               | 39.0                         | 890                  | (6.14) | 6.2                            |
| 8               | 43.8                         | 1000                 | (6.90) |                                |
| 9               | 44.8                         | 1022                 | (7.05) |                                |
| 10              | 42.4                         | 968                  | (6.67) | <u>psi</u>                     |
| 11              | 41.1                         | 938                  | (6.47) | 951                            |
| 12              | 42.9                         | 979                  | (6.75) | ±                              |
| 13              | 57.9                         | 1322                 | (9.12) | 141                            |
| 14              | 32.9                         | 751                  | (5.18) |                                |
| 15              | 35.3                         | 806                  | (5.56) |                                |
| 16              | 32.2                         | 735                  | (5.07) |                                |
| 17              | 45.7                         | 1043                 | (7.19) | <u>MPa</u>                     |
| 18              | 37.3                         | 852                  | (5.87) | (6.56)                         |
| 19              | 31.3                         | 715                  | (4.93) | ±                              |
| 20              | 44.4                         | 1014                 | (6.99) | (0.97)                         |

TABLE 27

Properties of Hard Setting  $\text{Ca}(\text{OH})_2$  Bases

| Brand                     | Water Solubility (%)<br>(N=10) | Normalized Acid Solubility (%)<br>(N=10) | Compressive Strength $\pm$ on -1 (N) |                               |
|---------------------------|--------------------------------|--|--------------------------------------|-------------------------------|
|                           |                                |  | Early                                | 24 Hour                       |
| Life Regular Set          | 4.81 $\pm$                     | 0.37 $\pm$                               | 2577 $\pm$ 4.99<br>(13)              | 4372 $\pm$ 1032<br>(20)       |
|                           | 0.66                           | 0.10                                     | (17.77) $\pm$ (3.44)<br>(13)         | (30.14) $\pm$ (7.12)<br>(23)  |
| Life Fast Set             | 3.35 $\pm$                     | 0.05 $\pm$                               | 2092 $\pm$ 558<br>(13)               | 3291 $\pm$ 1651<br>(13)       |
|                           | 0.83                           | 0.06                                     | (14.42) $\pm$ (3.85)<br>(13)         | (22.69) $\pm$ (11.38)<br>(13) |
| NuCap                     | 3.40 $\pm$                     | 6.64 $\pm$                               | 1008 $\pm$ 336<br>(15)               | 1357 $\pm$ 200<br>(20)        |
|                           | 0.66                           | 0.20                                     | (6.95) $\pm$ (2.32)<br>(15)          | (9.36) $\pm$ (1.38)<br>(20)   |
| Dycal Advanced Formula II | 0.97 $\pm$                     | 2.58 $\pm$                               | 1541 $\pm$ 376<br>(8)                | 2330 $\pm$ 680<br>(17)        |
|                           | 0.53                           | 0.25                                     | (10.62) $\pm$ (2.59)<br>(8)          | (16.06) $\pm$ (4.69)<br>(17)  |
| Dropain                   | 0.87 $\pm$                     | 11.54 $\pm$                              | 242 $\pm$ 72<br>(10)                 | 951 $\pm$ 141<br>(20)         |
|                           | 0.13                           | 1.39                                     | (1.67) $\pm$ (0.50)<br>(10)          | (6.56) $\pm$ (0.97)<br>(20)   |

TABLE 28

Newman - Keuls Multiple Range Test Results\*

| <u>Brand</u>              | <u>Water Solubility</u> | <u>Acid Solubility</u> | <u>Early Compressive Strength</u> | <u>24 Hour Compressive Strength</u> |
|---------------------------|-------------------------|------------------------|-----------------------------------|-------------------------------------|
| Life Regular Set          |                         |                        |                                   |                                     |
| Life Fast Set             |                         |                        |                                   |                                     |
| Nu Cap                    |                         |                        |                                   |                                     |
| Dropsin                   |                         |                        |                                   |                                     |
| Dycal Advanced Formula II |                         |                        |                                   |                                     |

\* Brands connected by vertical lines are not statistically different at  $p \leq 0.05$ .

## DISCUSSION

In 1979, Akester reported the radiographic appearance of voids under amalgam restorations which had been based with Dycal.<sup>7</sup> These voids occurred under intact restorations which had been placed two or three years previously. Upon removal of the amalgam restorations, a void was noted where the Dycal base had originally been placed. This phenomenon was also noted by Barnes and Kidd who described four cases in which they felt Dycal bases had disappeared.<sup>8</sup>

In response to these observations, studies have been undertaken to determine the physical characteristics of hard-setting calcium hydroxide products which may have been responsible for their disintegration under amalgam restorations. Researchers tested not only the physical properties of the calcium hydroxide materials but also the chemical make-up of the materials.<sup>59,62,89-91</sup>

In the current investigation the physical properties of Life Regular Set, Life Fast Set, Nu Cap, Dycal Advanced Formula II and Dropsin were evaluated.

Initially, the experimental procedure for water solubility was modeled after the technique used by Hwas

in 1984.<sup>90</sup> His technique involved making samples of the material to be tested and then submerging the sample in 50 ml. of de-ionized water at 37° C. and 100% humidity for 23 hours. After the 23-hour time period the specimen was removed and the 50 ml. of water containing any residue was heated to 98° C. to evaporate the water. After the water was evaporated the container containing the residue was heated to 150° C. to drive off any bound water. The containers were then placed in a dessicator and weighed until constant weight was achieved.<sup>90</sup>

During the initial water solubility trials using this procedure, it was noted that a very definite odor was given off by the residue when drying at 150° C. It was also noted that a constant weight of the residue could not be achieved using this procedure.

The final drying of the residue at 150° C. appeared to cause a thermal breakdown of the water soluble residue. It was initially felt that the high temperature (150° C.) used to drive off any bound water was the cause of the thermal breakdown and therefore the final drying temperature was lowered to 100° C. At this temperature during the drying of the residues an odor was still noted and the residue did not achieve a

constant weight.

At this point it was decided to determine if either the sample or the residue could be used to determine water solubility of the material. The same procedure was utilized but this time the actual samples were used, as well as their residue to attempt to calculate the water solubility.

Using this procedure with a final drying temperature of 150° C. both the samples and the residue were degraded by the high temperature and would give inaccurate measurements for the water solubility of the product.

At 100° C. the results were the same for both sample and residue. A thermal breakdown was still occurring in both the sample and residue and any measurements obtained from using either one would be erroneous.

It was now felt that even a temperature of 100° C. was too high to prevent a thermal degradation of both the sample or the residue, thus a new technique to remove the water had to be developed. This technique involved the use of a vacuum chamber (Fig. 4) at 650 mm. Hg vacuum and 50° C. Both the sample and residue were



tested with this technique and even with this low temperature constant weight could not be achieved for either the sample or the residue.

At this point the decision was made that the highest temperature that any sample or residue could be subjected to was body temperature ( $37^{\circ}$  C.) in order to eliminate any degradation which did not occur in the body. In order to eliminate any degradation of the sample by heat, evaporation and removal of any bound water was accomplished by a vacuum chamber. The water was first evaporated at 650 mm. Hg vacuum at  $37^{\circ}$  C. making sure that no boiling of the water occurred. After removal of all water, the vacuum was increased to  $1.5 \times 10^{-2}$  mm. Hg to remove any bound water. This final vacuum step was also carried out at body temperature ( $37^{\circ}$  C.).

The first attempt using this new procedure involved 20 samples of Life Regular Set. After 23 hours of submersion in 50 ml. of de-ionized water, ten samples were placed in a dessicator at  $37^{\circ}$  C. and ten samples were placed in a vacuum chamber which was maintained at  $1.5 \times 10^{-2}$  mm. Hg and  $37^{\circ}$  C. Both sets of samples were maintained at these conditions for 14 days. During the 14-day period, all samples

continued to lose weight and a constant weight was not achieved.

In order to eliminate the possible interaction between the sample and water during the 24-hour submersion period, ten additional samples of Life Regular Set were made. Five were placed in the vacuum chamber and five were placed in the dessicator without first being submerged for 23 hours. After a two week period, these samples also did not achieve a constant weight. It was therefore deduced that there was some substance which was being volitized even when the product was not submersed in water. This was visually demonstrated by a color change from white to bright yellow of the Life Regular Set samples when placed in the vacuum chamber.

At this point, it was decided that the specimens themselves were inherently unstable and this method was not suitable for water solubility measurements. It was therefore decided to concentrate on the residue left by the sample after the 23-hour submersion period. The technique for determining the water solubility involved the production of the sample and the submersion of the sample in a weighing jar containing 50 ml. of de-ionized water for 23 hours. For the 23-hour period

the sample and container were kept in a chamber at 37° C. and 100% humidity. After the 23-hour submersion period the sample was removed and the weighing jar containing the water and residue were placed in a vacuum chamber at 650 mm. Hg vacuum and 37° C. to evaporate all the water. Care was taken not to boil the water in order not to lose any of the residue if bumping occurred. After all the water was evaporated, the vacuum was increased to  $1.5 \times 10^{-2}$  mm. Hg for 48 hours. The bottles containing the now dry residue were then placed in a dessicator until constant weight was achieved. During the testing procedure the high vacuum pump failed. It was determined, however, that the 48-hour period at  $1.5 \times 10^{-2}$  mm. Hg vacuum was unnecessary to completely remove all water and the same results were obtained by using the low vacuum (650 mm. Hg) and then placing the weighing jars directly in the dessicator.

The problem of obtaining a constant weight of the sample and the residue during the water solubility procedure may be related to the make-up of the materials themselves.

The chemical composition of these materials has been shown to contain a high percentage of salicylate.<sup>95</sup>

It is because of the low vapor pressure of the salicylate that the continued loss of weight of the material occurs. The hard-setting calcium hydroxide product investigated in this study consist of a two-paste system, one being labeled as a base and the other as a catalyst. This terminology in fact is inaccurate with respect to the chemical make-up of the pastes. A better label for the pastes would be to term one the acid paste and the other the basic paste.<sup>95</sup> The acid paste would be that paste containing an ester of salicylic acid. The basic paste would be the paste containing the calcium hydroxide and a sulfonamide which is used as a "plasticizer". These materials set by a reaction between the calcium ions and the ester of salicylic acid.<sup>95</sup> The resultant hard material may be regarded as a chelation of calcium ions by a salicylate ions which forms a matrix in which lie an excess of unreacted calcium hydroxide.<sup>95</sup>

The original Dycal which was patented by Dougherty in 1962, serves as the chemical example of which all hard-setting calcium hydroxide bases are modeled. The original Dycal as proposed by Dougherty is a dental base where esters of salicylic acid and calcium ions react to form a hard, rigid mass of

calcium salicylate in which there is an excess of calcium hydroxide.<sup>96</sup>

The reaction of the phenol (in this case 1,3 butylene glycol disalicylate) and calcium ions takes place rapidly and forms a rigid yet water permeable substance. Calcium phenolates are capable of being formed from the combination of calcium ions and many different phenol compounds. Among the phenolic compounds capable of reacting are: phenol pyrogallol, eugenol and salicylic acid. Salicylic acid is the preferred phenolic compound for this reaction.<sup>96</sup> It is derived from phenol by means of the Kolbe Reaction<sup>97</sup> and then esterified to give the final product its proper characteristics. (Fig. 9).

To facilitate the reaction between the calcium ions and the phenol, the phenolic compound should be liquid, although it is possible for the reaction to occur with a solid phenolic compound. The consistency of the final reaction product, however, is more homogeneous when a liquid is utilized.<sup>41</sup> The reaction between calcium hydroxide and salicylate is very rapid and in order to decrease the reaction time the calcium hydroxide is diluted with an inert filler such as titanium dioxide and calcium tungstate (increases

radiopacity). These fillers along with the calcium hydroxide are mixed with ethyl toluenesulfonamide to provide a homogeneous paste to react with the salicylate.<sup>96</sup>

The set product is a chelated complex where calcium ions bridge salicylate units to form a solid structure (Fig. 10).<sup>98</sup> In the case of Dycal, the salicylate is a dimer (disalicylate) which chelates the calcium ions. The calcium ions may therefore be chelated by two dimer molecules forming a chain-like structure with calcium ions as the bridging units.<sup>98</sup>

This original formulation was modified by Jandourek in 1980 to provide a new base with improved physical properties.<sup>99</sup>

The new hard-setting calcium hydroxide base material was marketed under the trade name LIFE. This new formulation was similar to Dougherty's original formulation in that it also consisted of a two paste system, one being an acid paste and the other being a basic paste.<sup>96-100</sup> The significant difference in the formulation occurred in the acid paste.

The acid paste while containing a derivative of salicylic acid (alkyl salicylate) also contained a phenol formaldehyde resin. This resin is a low

molecular weight polymer condensate (Fig. 11) which is derived by reacting a phenol with formaldehyde or a formaldehyde derivative. This resin is then blended with a specific alkyl salicylate to obtain the correct viscosity. To this blend is added inert water insoluble fillers (barium sulfate, titanium oxide and iron oxide) which modify reaction time, increase radiopacity and affect the final product color. The selection of the specific alkyl salicylate which is mixed with the resin is also very important. It has been found that by increasing the size of the alkyl group on the salicylate the reaction time will increase but the compressive strength of the final product will decrease. Thus, to increase the compressive strength of the final product a smaller chain alkyl salicylate is desirable; however, this in turn increases the reaction rate so modifiers must be added.<sup>99</sup> The basic paste of this product (Life) is essentially calcium oxide or calcium hydroxide extended with zinc oxide contained in an inert liquid which is used as a matrix for the basic paste. This inert liquid is ethyl toluenesulfonamide. The basic paste contains a stoichiometric excess of calcium hydroxide in order to provide enough calcium hydroxide after the reaction

to provide a therapeutic effect. The constituents of the two pastes (Life) are listed below:<sup>99</sup>

| Basic Paste   | Parts by Weight |
|---|-----------------|
| Resin condensate  | 38.0            |
| Methyl salicylate   | 12.0            |
| Barium Sulfate  | 37.9            |
| Titanium Dioxide  | 10.0            |
| Aerosil R972 silica<br>(a tradename of Degussa Corp.,<br>Teterboro, New Jersey)                                       | 2.0             |
| Mapico 3100 pigment<br>(a tradename for iron oxide<br>pigments sold by Citco Pigment<br>Division, New York, New York) | <u>0.1</u>      |
|   | 100.0           |

| Acid Paste               | Parts by Weight |
|--------------------------|-----------------|
| Calcium Hydroxide        | 51.3            |
| Zinc Oxide               | 13.75           |
| Zinc Stearate            | 0.25            |
| Ethyl toluenesulfonamide | 34.00           |
| Distilled water          | <u>0.7</u>      |
|                          | 100.0           |

When the two pastes are mixed a reaction takes place in which a complex calcium polysalicylate is formed. This calcium polysalicylate is thought to contain calcium ions which bridge the polysalicylate molecules together to



form a weak polymeric structure. Additional electrostatic bonds occur after the initial chelation of the calcium ions, forming a solid structure with great strength. These additional electrostatic bonds occur between the salicylate units of different polysalicylate molecules with water having the ability to accelerate the rate of formation of these bonds.<sup>98-101</sup> This newly formed calcium polysalicylate has the unique properties of being resistant to penetration or disintegration to acids used as etchants in dentistry and still allows excess calcium hydroxide to leech from the set material to allow for its therapeutic qualities.<sup>100</sup>

The reaction which forms the rigid calcium hydroxide containing base is between the calcium hydroxide and the salicylate. According to Prosser, the alkyl salicylate occurs in an ionized form (Fig. 12). This ionized form of the alkyl salicylate chelates calcium ions from the calcium oxide and the calcium hydroxide to bridge the salicylate units to form a chain structure. (Fig. 10).<sup>101</sup> Prosser stated that this bridging is not essential for cement formation and it is possible water plays a yet unknown structural role in the calcium hydroxide salicylate cement.<sup>101</sup>

Dropsin, the fifth material used in this study

is not a calcium hydroxide salicylate cement. It is instead a zinc phosphate cement which contains calcium hydroxide. The constituents of Dropsin are listed below.<sup>55</sup>

Liquid

|                    |     |
|--------------------|-----|
| phosphoric acid    | 25% |
| aluminum hydroxide | 8%  |
| distilled water    | 67% |

Powder

zinc oxide  
 calcium hydroxide  
 magnesium oxide  
 aluminum hydroxide

When the powder and liquid are mixed together a crystalline structure of zinc phosphate is formed. The reactions which take place are shown in Figure 13.<sup>102</sup> Calcium hydroxide is incorporated into the crystalline matrix and is available to provide a therapeutic effect. This addition of calcium hydroxide does not greatly effect the initial pH of freshly mixed Dropsin which has a pH of 2.2.<sup>55</sup>

Tables 2-6 show the actual amount of dissolved residue left after submerging the specimens in distilled

water for 23 hours. From the results of the statistical analysis (Table 28) it is shown there are three populations. Life Regular Set is the most soluble of all the materials tested with a water solubility of  $4.81\% \pm 0.66\%$ . This value falls in between the values found by Hwas<sup>90</sup> (5.3%) and McComb<sup>89</sup> (3.78%). The difference between the three results is most likely due to the method by which the material was tested rather than a variation in the formulation. The next most water soluble materials, Life Fast Set and Nu Cap were not tested by other investigators. The values for water solubility are Life Fast Set  $3.35 \pm 0.83\%$  and Nu Cap  $3.40 \pm 0.66\%$ . The least water soluble of materials tested were Dycal Advanced Formula II and Dropsin with values of  $0.97\% \pm 0.53$  and  $0.87\% \pm 0.13$ . Both these materials are less than 1% water soluble even though they both are of different chemical composition. One would expect these results from Dropsin since it is essentially zinc phosphate cement,<sup>55</sup> but, the insolubility of Dycal Advanced Formula II is remarkable. Its insolubility in water is an indication of the manufacturer's response to reports of "Disappearing Dycal".<sup>7,8</sup>

Photomicrographs of the surface of the specimens submerged in distilled water for 23 hours are shown in

Figure 14-18. All the materials demonstrated some surface changes with Dropsin (Fig. 18) and Dycal Advanced Formula II (Fig. 17) showing the least amount of surface change.

The data of the results of the acid solubility study are shown in Tables 7-16 and are graphically displayed in Figure 7. From the data presented here it is indicated that there is a significant statistical difference in the acid solubility of the five tested materials (Table 28). Dropsin, when exposed to 37% phosphoric acid for one minute had a normalized acid solubility of 11.54% and almost twice as much as Nu Cap which was the most acid soluble of the calcium hydroxide salicylate bases. This great solubility to acid is due to the fact that Dropsin is essentially a calcium hydroxide containing zinc phosphate cement. This finding of the increased solubility of zinc phosphate cement in acid has been confirmed by Norman and numerous other investigators.<sup>64-66,70</sup> It appears that the phosphoric acid breaks down the crystal matrix of set zinc phosphate cement degrading the rigid material.<sup>102</sup> Nu Cap, with an acid solubility of 6.64% was the second most soluble of the materials tested and the most soluble of the calcium hydroxide salicylate bases. Its acid solubility is

similar to the acid solubility of the original Dycal found by other investigators.<sup>69, 90</sup> It was therefore conjectured that its chemical composition may be similar to that of Dougherty's original Dycal.<sup>96</sup> Dycal Advanced Formula II proved to be the next least acid soluble of the materials tested. When compared to the results given for the acid solubility of the original Dycal, the Dycal Advanced Formula II is approximately half as soluble in acid as the original formula.<sup>69, 90</sup> Life Regular Set and Life Fast Set proved to be essentially insoluble when exposed to 37% phosphoric acid. This is of great significance when placing an acid etch composite restoration since these two materials will prevent any acid injury to tissue which they are covering. Stanley<sup>91</sup> has shown that an acid pretreatment on dentin causes an enhanced pulpal irritation due to an increase in the permeability of dentin.<sup>56</sup> Hauwers also showed that more bacteria are present in dentin tubules exposed to acid than in those not exposed.<sup>59</sup> It is therefore essential that all vital dentin be protected with a base before any acid treatment is performed.

The best suited material for this purpose, according to the results of this study, would be Life

Fast Set which is essentially insoluble in acid. All of the other calcium hydroxide salicylate bases tested here could also be used with the acid pretreatment. The only material tested which would not provide adequate protection from an acid treatment is Dropsin. Dropsin, which is essentially a zinc phosphate cement, breaks down rapidly when exposed to acid.<sup>55</sup> Dennison showed that dentin covered with a zinc phosphate cement base was still affected during an acid etch procedure.<sup>57</sup> Therefore, Dropsin cannot be recommended for use when an acid pretreatment is being performed.

Photomicrographs of the effect of one drop of 37% phosphoric acid are shown in Figures 19-23. Figure 20 is that of Life Fast Set which shows no discernible effect of the acid on the surface of the specimen. All other photomicrographs (Fig. 19-23) of the materials show some definite effect of the acid on the surface of the material with Dropsin (Fig. 23) having the greatest surface change of all the materials tested.

It should be noted that the acid solubility of the materials tested is reported as the Normalized Acid Solubility. This is due to a problem which occurred when preparing the Dropsin specimens. When preparing these particular specimens, it was impossible to remove

the sample without fracturing it. In order to facilitate the removal of the Dropsin specimens, the stainless steel ring along with the specimen were placed in a dessicator for 24 hours. After this time period, the specimen was easily removed from the ring. By placing the Dropsin specimen in a dessicator to facilitate removal without fracture of the material, the specimen lost water and therefore weight. If the specimen was used for the acid solubility test when it was removed from the dessicator, the specimen would show that it gained weight when it was submersed in acid as shown in Table 11. This is due to the absorption of water into the dessicated specimen. In order to obtain accurate values for the acid solubility of the materials a control was needed. This control value determination consisted of submersing 10 specimens of each material in distilled water for 1 minute and then rinsing them under room temperature tap water. Any weight gain or loss was then recorded and the mean was then calculated. This mean was calculated for all the materials of this experiment (Tables 7-11). Dropsin was the only material whose mean value of the control was a net gain in weight, indicating that the procedure used to facilitate removal of the specimen also removed water

from the specimen. The value for the acid solubility of materials was obtained by subtracting that material's control mean value from the acid solubility weight resulting in the materials' actual acid solubility which was termed the normalized acid solubility.

The early and 24-hour compressive strength tests of the five calcium hydroxide containing materials were carried out in a similar manner as described in the American Dental Association specification Nos. 8 and 9.<sup>92,93</sup> These tests were run on each specimen with no attempt to constrain the specimen to a specific shape. Lloyd raised the question to whether the compressive strength of a material, which is obtained by the testing of an unconstrained specimen, is directly applicable to a clinical situation where the material is almost totally constrained.<sup>85</sup> He found that materials that were previously regarded as too weak to withstand the forces of condensation may indeed possess sufficient strength to serve as an adequate base when areas of constraint are present.<sup>85</sup> This premise was supported by an earlier work by Chong.<sup>71</sup> His experimentation showed that the minimal compressive strength a base material must have to support the amalgam condensation forces is between 100 and 170 psi. Chong stated that if a base was to be



displaced or fractured it would be displaced or fractured by light as well as heavy condensation forces.<sup>71</sup> Thus, even weak temporary cements may be used as bases.

This is contrary to the old idea. That a base must be able to withstand the optimum pressure for amalgam condensation (8000 psi),<sup>84</sup> or at the very least it must be able to withstand the average amalgam condensation pressure of 3200 psi.<sup>74</sup> If this were the case then the only base material with enough unconstrained compressive strength to withstand such pressure would be zinc phosphate cement.<sup>102</sup> Clinical experience, Chong's investigation<sup>71</sup> and Lloyd's theory of constraint<sup>85</sup> tend to disprove these old ideas and thus allow the use of bases with lower unconstrained compressive strengths.

Tables 17-26 show the actual data obtained from the early and 24-hour compressive strength tests run on the five materials. This data is summarized in Table 26 and graphically depicted in Histogram #3. From the analysis using the Newman-Keuls' multiple range test (Table 28) it can be shown that all the early compressive strengths are significantly different from one another. Dropsin has the lowest compressive strength

of 242 psi  $\pm$  72 psi but still meets Chong's minimal value for the compressive strength of a base.<sup>71</sup> It is interesting to note that even though Dropsin is essentially a zinc phosphate cement, neither its early or 24-hour compressive strength is one tenth the normal value (14,500 psi) for zinc phosphate cement.<sup>102</sup> This low compressive strength of Dropsin may be due to the fact that the calcium hydroxide which is added for a therapeutic effect somehow interferes with the formation of the crystal matrix.

The highest early compressive strength was recorded from Life Regular Set. With the other three materials falling between the highest and lowest values recorded for the early compressive strength.

The statistical analysis of the 24-hour compressive strength (Table 28) demonstrates that there are three distinct populations. Life Regular Set and Life Fast Set are members of the population with the highest 24-hour compressive strength. Nu Cap and Dropsin are members of the population with the lowest 24-hour compressive strength, while Dycal Advanced Formula II makes up the population between the extremes.

The results of the compressive tests compare favorably with those products tested by other

investigators.<sup>90,91</sup>

It should be noted, however, that of all the products, Life Regular Set took the longest time to set (15 minutes). This length of time under clinical conditions may decrease but one should be aware that if the base material is not set and the permanent restoration placed a less than ideal result may occur.

From the data obtained from this investigation it appears that the manufacturer of Dycal has responded to earlier allegations of the disappearance of the product<sup>7,8</sup> with a new product Dycal Advanced Formula II which is essentially insoluble in water. Water solubility, however, would not seem to be a major clinical problem if a restoration was properly placed and its margins were intact. Perhaps, the situation where Akester,<sup>7</sup> Barnes and Kidd<sup>8</sup> described "Disappearing Dycal" involved a condition where the permanent restoration was defective. Considering the millions of restorations placed using the original formula of Dycal, one would expect to have more than six cases where the Dycal base disappeared. It may have been that the cases where calcium hydroxide bases "disappeared" occurred in conditions that were less than ideal. Since the articles which reported the disappearance of the bases were, in

fact, editorials there is no sound evidence which demonstrates that calcium hydroxide bases disappear. The dental research community, it seems, has been led into thinking that all calcium hydroxide bases are suspect on the basis of the opinion of two clinical editorials, rather than a sound scientific evidence, when in reality, there may have been no need to question the materials at all.

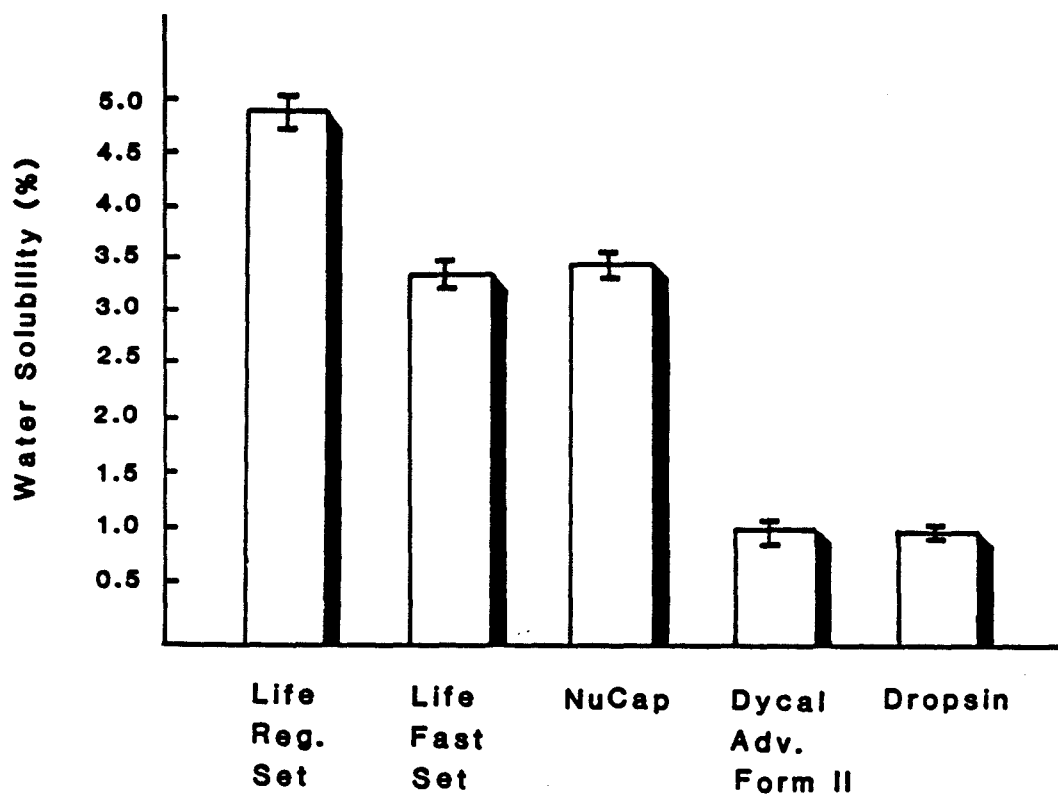


Fig. 6. Water Solubility of Hard Setting Calcium Hydroxide Base Materials.

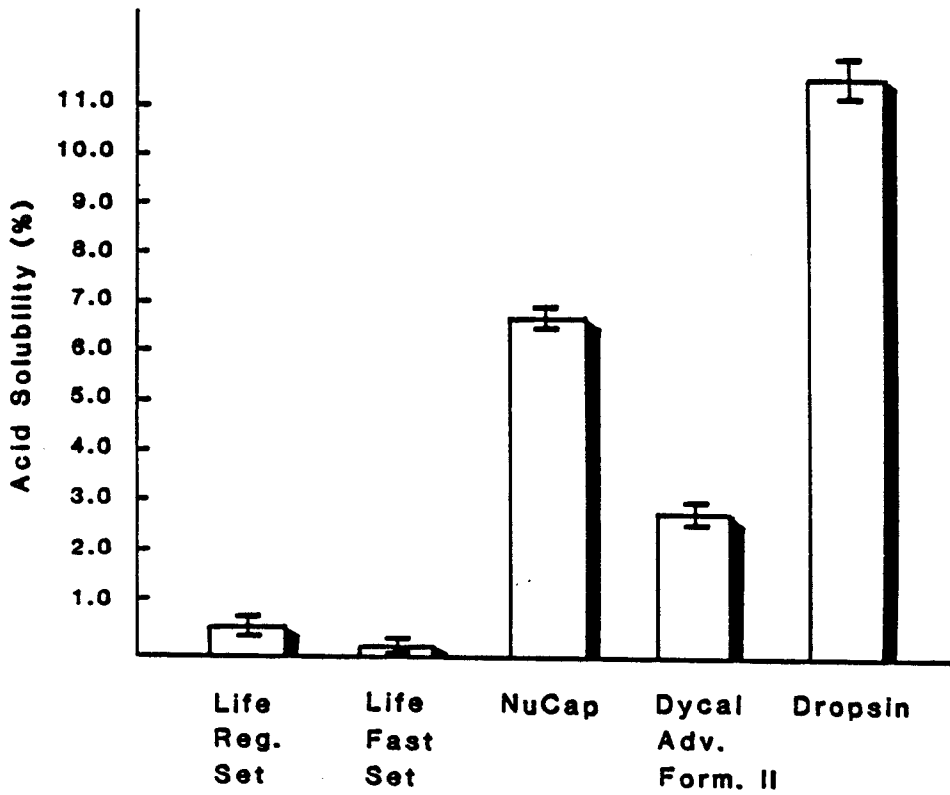


Fig. 7. Acid Solubility of Hard Setting Calcium Hydroxide Base Materials.

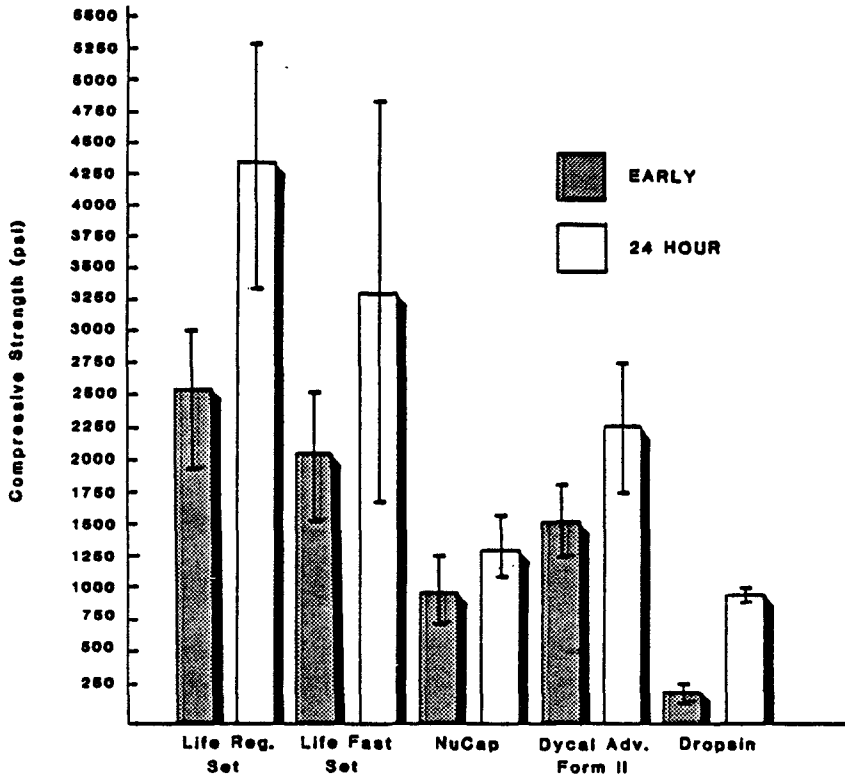


Fig. 8. Early and 24 Hour Compressive Strengths of Hard Setting Calcium Hydroxide Base Materials.

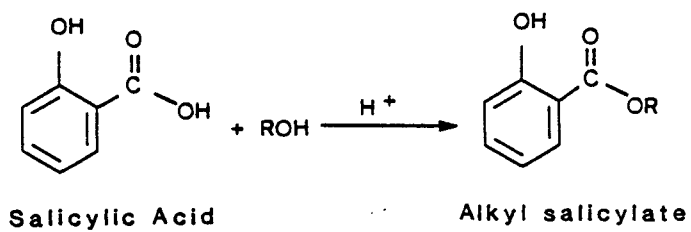
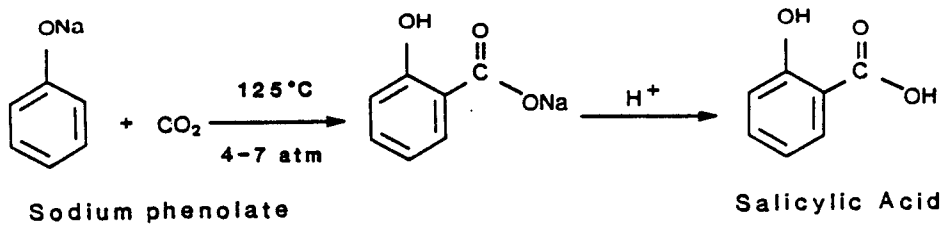
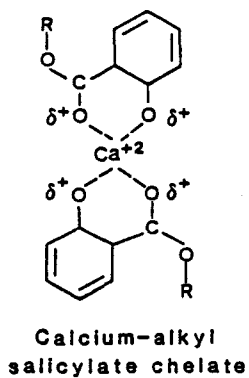


Fig. 9. Kolbe Reaction





DYCAL

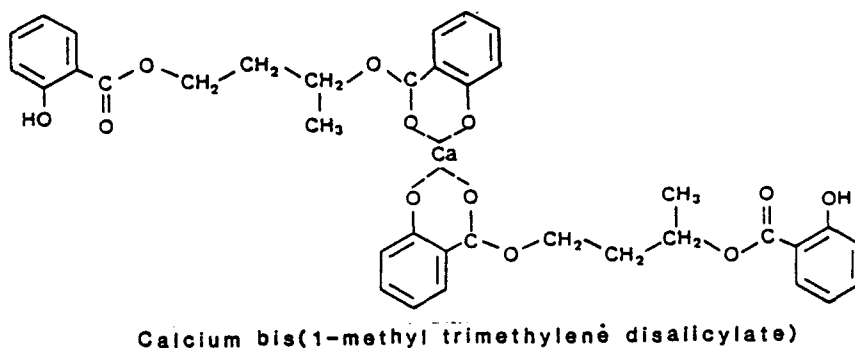
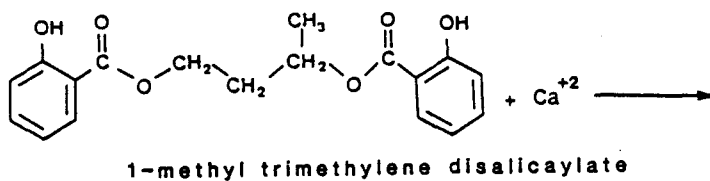


Fig. 10. Chelated Calcium Salicylate Complex

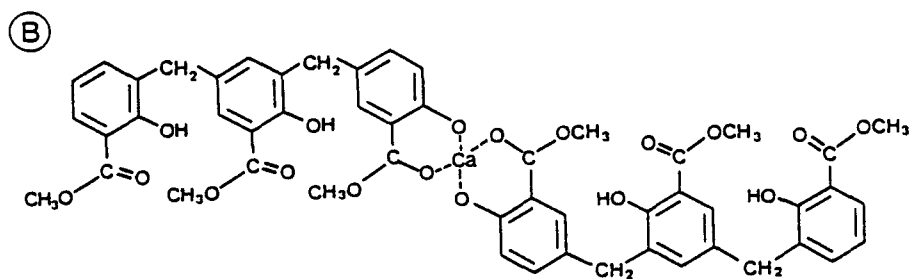
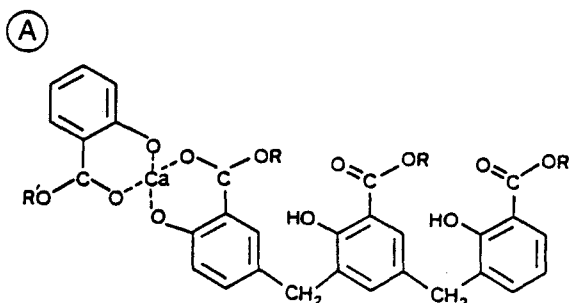
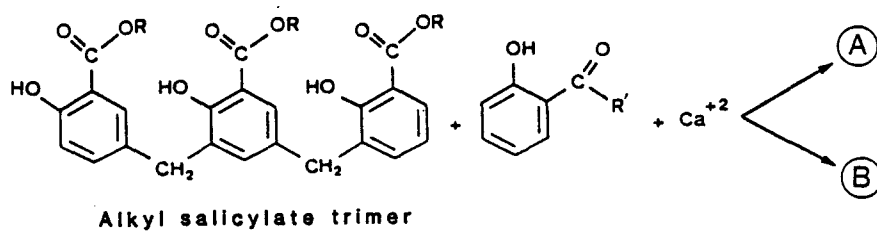


Fig. 11. Resin Polymer

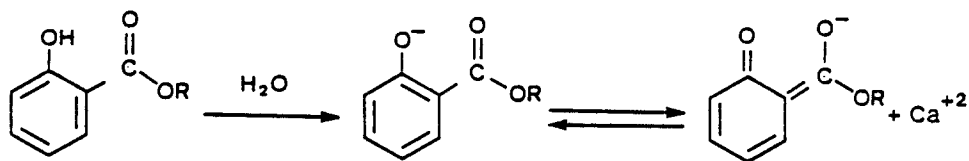


Fig. 12. Ionized Alkyl Salicylate

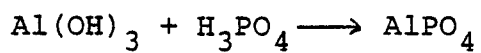
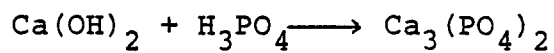
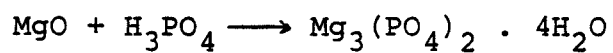
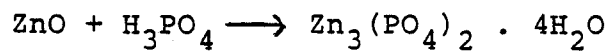


Fig. 13. Zinc Phosphate Cement Reactions

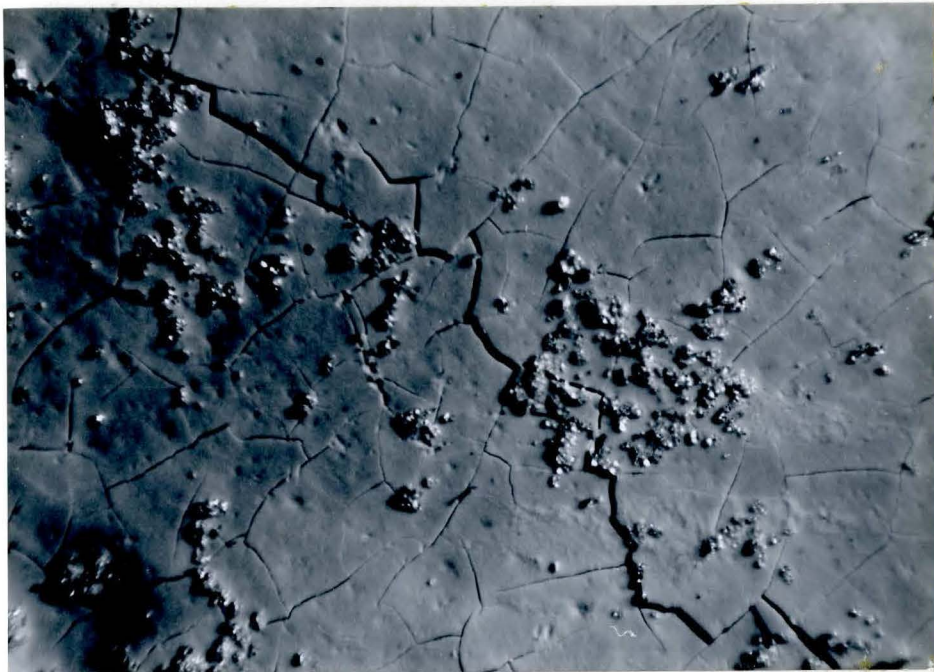


Fig. 14. Photomicrograph (16 x) of the surface of Life Regular Set after a 23-hour submersion in water.

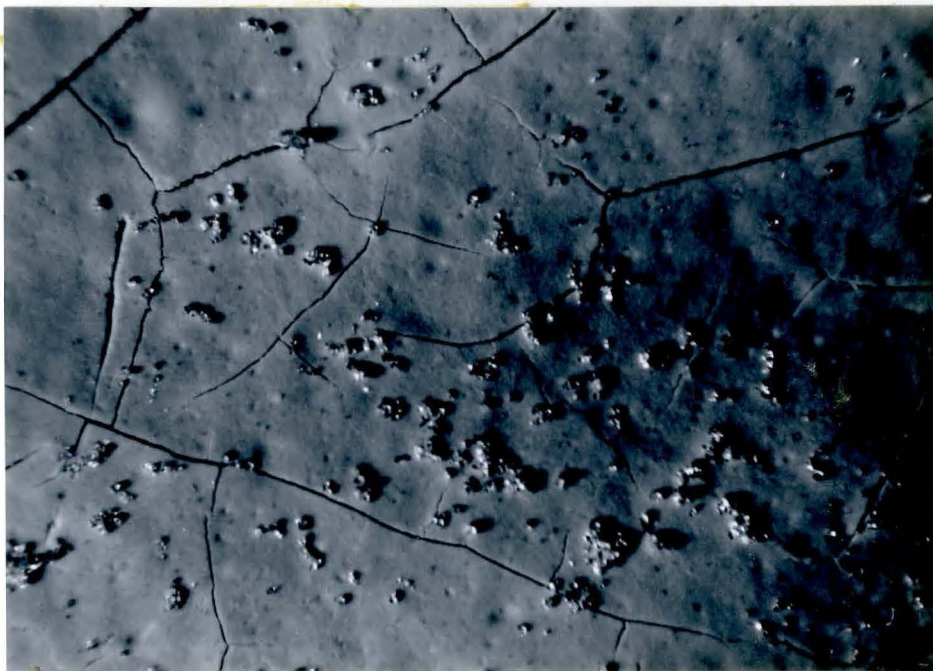


Fig. 15. Photomicrograph (16 x) of the surface of Life Fast Set after a 23-hour submersion in water.

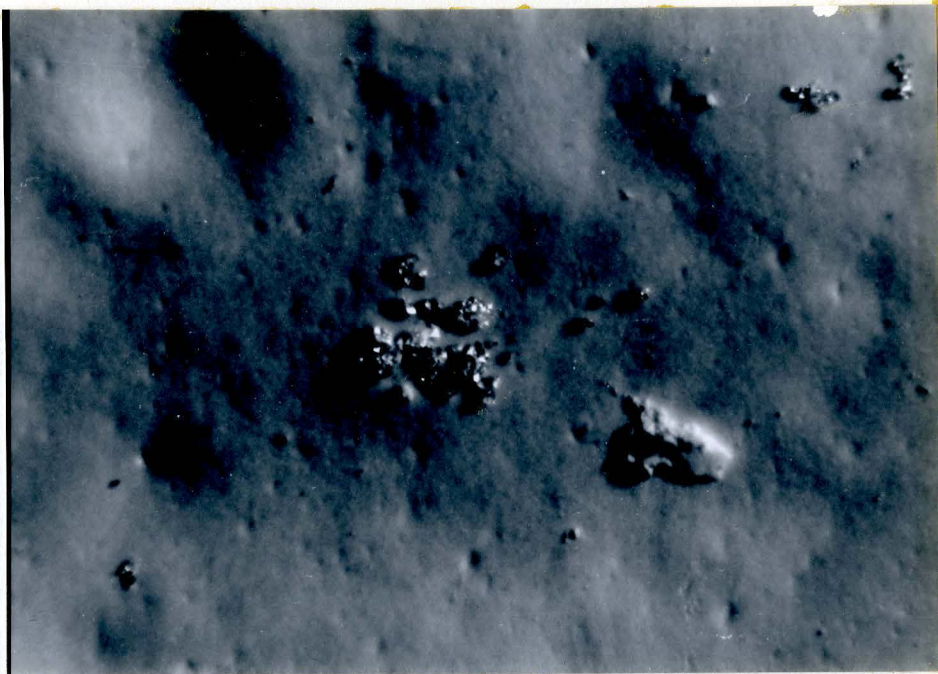


Fig. 16. Photomicrograph (16 x) of the surface of Nu Cap after a 23-hour submersion in water.

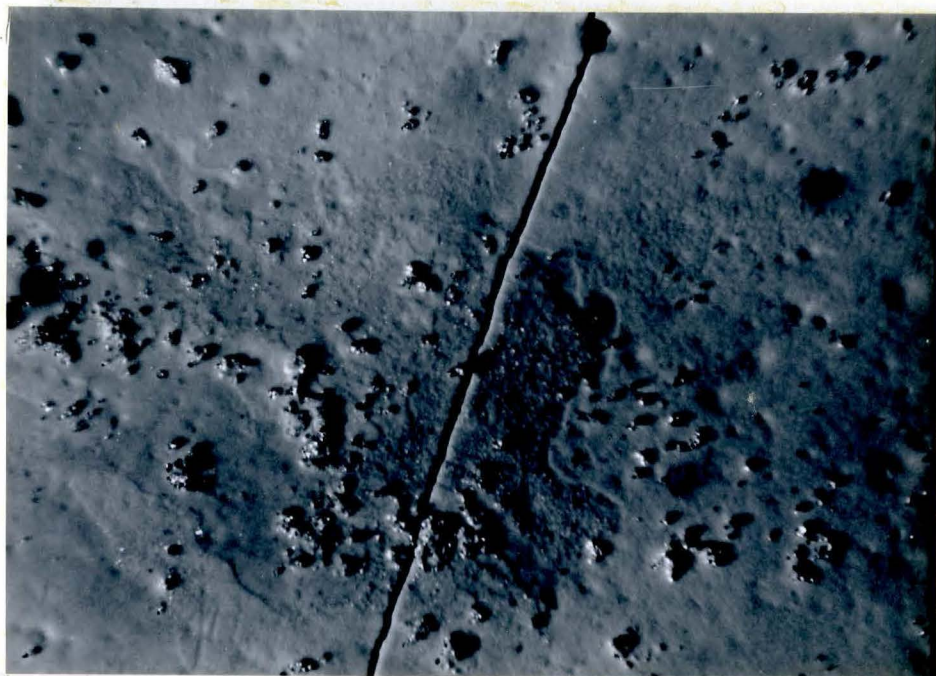


Fig. 17. Photomicrograph (16 x) of the surface of Dycal Advanced Formula II after a 23-hour submersion in water.



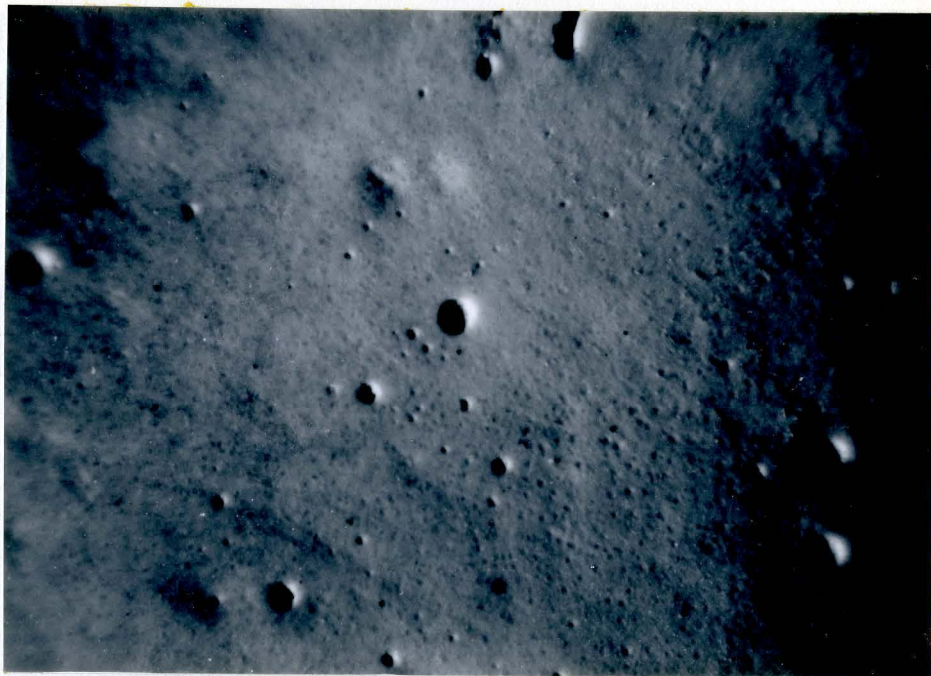


Fig. 18. Photomicrograph (16 x) of the surface of Dropsin after a 23-hour submersion in water.

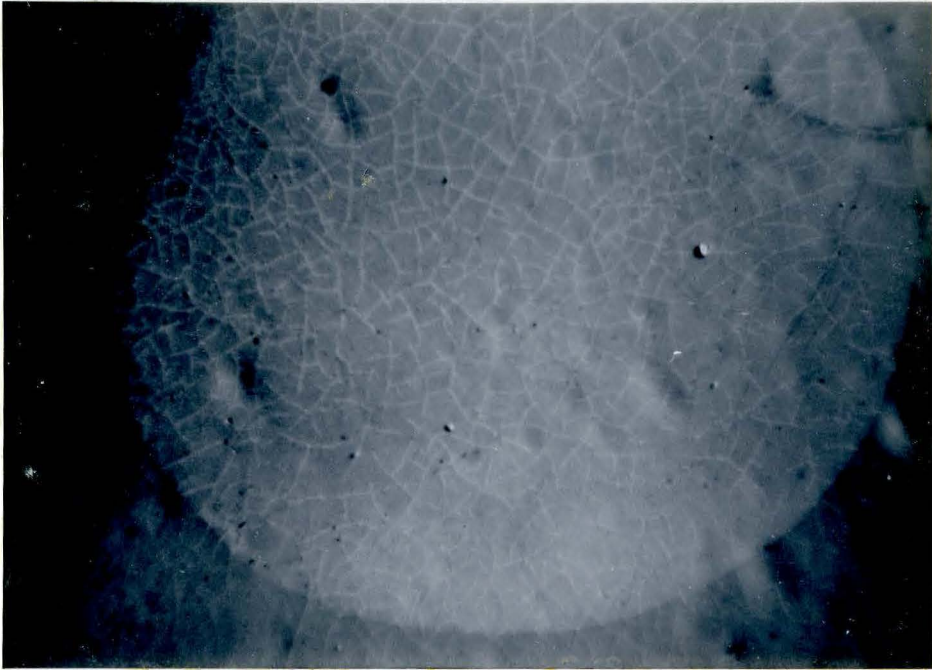


Fig. 19. Photomicrograph (25 x) of acid-etched surface of Life Regular Set.

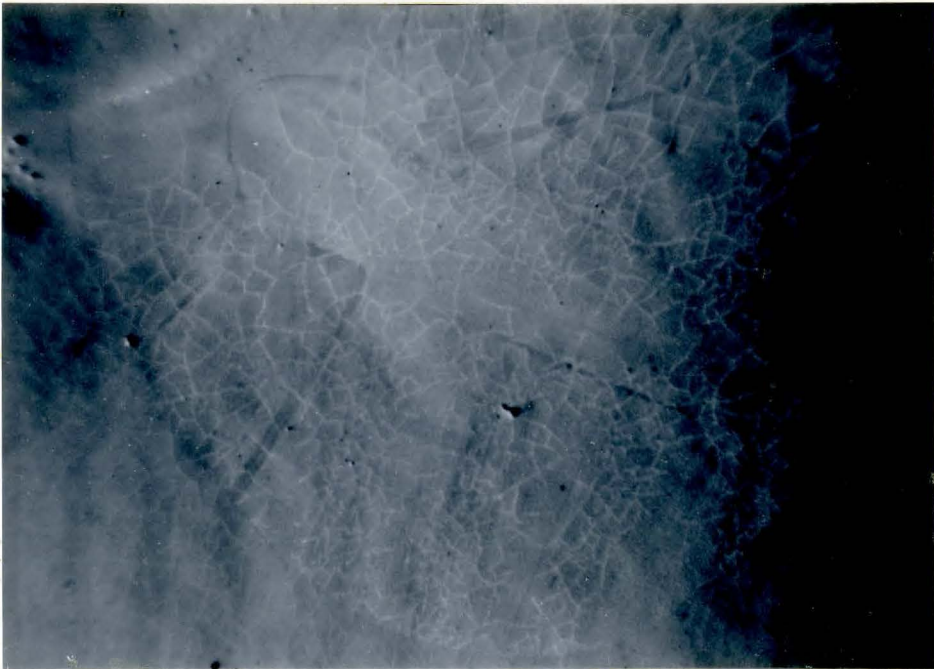


Fig. 20. Photomicrograph (25 x) of acid-etched surface of Life Fast Set.

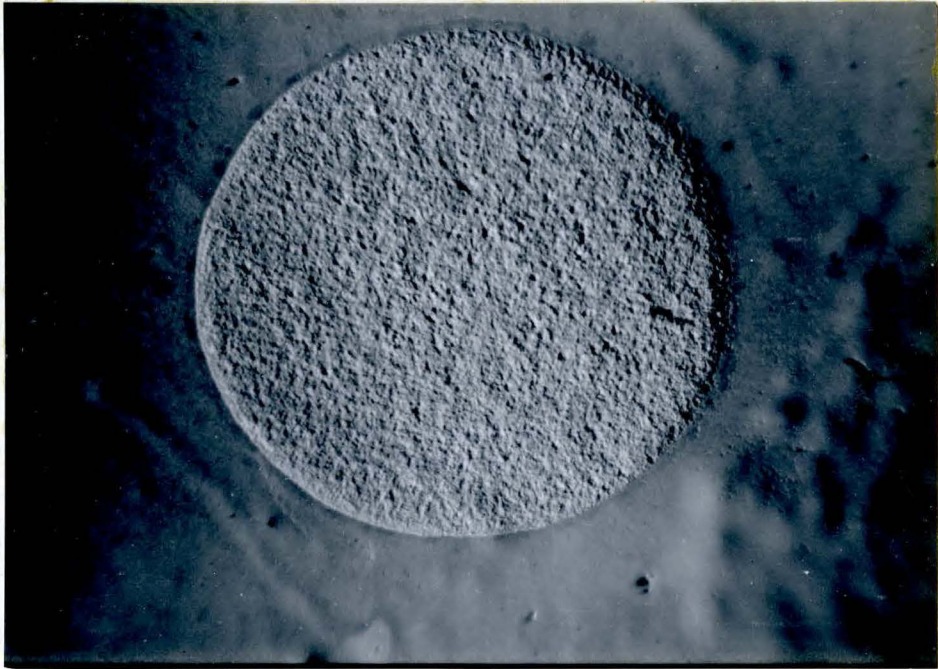
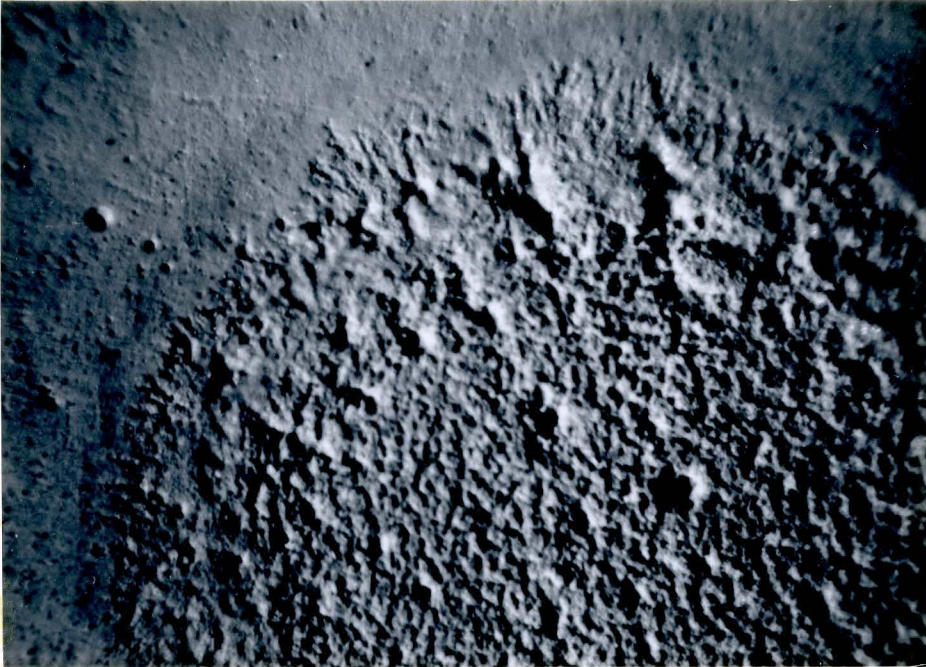


Fig. 21. Photomicrograph (25 x) of acid-etched surface of Nu Cap.



Fig. 22. Photomicrograph (25 x) of acid-etched surface of Dycal Advanced Formula II.

In this investigation water solubility, acid solubility and compressive strength of various calcium hydroxide containing bases were studied.



Correlations between water solubility and acid solubility of the materials tested.

1. Life regular Set and Life Fast Set had the highest early compressive strength. Drosin had the lowest early compressive strength.

**Fig. 23.** Photomicrograph (25 x) of a base material because of Drosin. This material may cause pulpal irritation, due to high acid solubility and its low initial compressive strength which could lead to the

## SUMMARY

In this investigation water solubility, acid solubility and compressive strength of various calcium hydroxide containing bases were studied.

1. Dropsin was the least water soluble of all materials tested by this method. However, Dycal Advanced Formula II had the lowest water solubility for those materials consisting of a calcium hydroxide salicylate mixture. Life Regular Set had the highest water solubility, while Life Fast Set and Nu Cap fell between the extremes.

2. Life Fast Set and Regular Set are essentially insoluble in 37% phosphoric acid. Dropsin is the most acid soluble of the materials tested. There was no correlation between water solubility and acid solubility of the materials tested.

3. Life Regular Set and Life Fast Set had the highest early compressive strength. Dropsin had the lowest early compressive strength.

4. Dropsin cannot be recommended as a base material because of its low initial pH which may cause pulpal irritation, its high acid solubility and its low initial compressive strength which could lead to the

failure of the permanent restoration.

5. The 24-hour compressive strength of Life Regular Set and Life Fast Set was nearly twice that of the other brands tested.

All materials tested, except Dropsin, have sufficient compressive strength to withstand amalgam condensation and masticatory forces.

6. The proposal of "Disappearing Dycal" does not appear to be due to a dissolution of the base material, rather it may be better explained by percolation caused by poor marginal seal of the permanent restoration.



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APPENDIX I

APPENDIX I

10:34:35 03/01/85 109089

```

LISTFNS
GO:COL:CT;DF;E;I;J;M;MSM;N;NM;SSB;SST;SSM;XX;CD
C 13 M=NM*(PGL1)P0
C 22 SSB=COL*0
C 33 'TOTAL NUMBER OF DATA' , (VM+(+(G)0))
C 41 'CORRECTION TERM' , (VCT-(+(G)2)*M)
C 53 'SUM OF SQUARES TOTAL' , (VSST+(+(G)2))-CT)
C 61 HERE=COL-COL+1
C 71 'NUMBER OF DATA IN TEST' , (VCDL) , ' ' , (VNNICOL)+(+(GE;COL)0))
C 87 SSB=SSB+(+(GE;COL)2)*NNICOL
C 97 MCCOLJ=(+(GE;COL)2)*(+(GE;COL)0))
C 107 +HERE=(COL<(PGL1))
C 117 'DEGREES OF FREEDOM' , (VDFP*((N-1)-(PGL1)-1)))
C 123 'SUM OF SQUARES BETWEEN' , (VSSB-SSB-CT)
C 133 'SUM OF SQUARES WITHIN' , (VSSM-SST-SSB)
C 143 'MEAN STANDARD WEIGHT' , (VMSM+SSM+DF)
C 153 'STANDARD ERROR OF MEANS' , (VE*(MSM*((PGL1)+(+(NM))))0.5)
C 163 'DF' , (VDF)
C 177 'ENTER SIGNIFICANT STUDENT RANGES'
C 183 R=0
C 193 'CRITICAL DIFFERENCES' , (VCD-R=E)
C 203 M= SORT M
C 217 I=0
C 227 POINT1:I=I+1
C 233 J=I
C 243 POINT2:J=I+J
C 253 +(M(J)-M(I))>CDE(J-I))/PRINT1 Tests Against
C 263 +(M(J)-M(I))<CDE(J-I))/PRINT2 Critical Difference
C 273 BACK:POINT2=J<(PM)
C 283 +POINT1=I<(PM)-1)
C 293 PRINT1:(VI) , ' AND ' , (VJ) , ' SIGNIFICANT'
C 303 +BACK
C 313 PRINT2:(VI) , ' AND ' , (VJ) , ' NONSIGNIFICANT'
C 323 +BACK
    
```

Loop to Calculate Mean  
Calculates Statistics and Prints

Prints if Significant or Nonsignificant

```

LISTFNS:ALF;NAME:I;FUN:COL;NO:GIO:OPM
C 13 GIO=1
C 23 OPM=60
C 33 NAME=OML 3
C 43 ALF= ABCDEFHIJKLMNOPQRSTUVWXYZAARGDEFGHIJKLMNOPQRSTUVWXYZ0123456789
C 53 NAME=NAME[65:ALF\NAME:]
C 63 I=0
C 73 L=(I+NAME)<I+1)/0
C 83 NO=I+PUN+OCR NAME[I:]
C 93 COL=(NO,1)P'(I) , (V(NO,1)P"1+NO) , ((NO,2)P"1' )
C 103 COLI:J=(I+P)COL)P'
C 113 COL,FUN
C 123 2 1 P'
C 133 =L
    
```

```

LISTVARS:ALF;VAR:I;R:N:GIO
C 13 GIO=1
C 23 R=I+VAR=OML 2
C 33 ALF= ABCDEFHIJKLMNOPQRSTUVWXYZA
C 43 ALF=ALF , 'ABCDEFGHIJKLMNORSTUVWXYZ0123456789'
C 53 VAR=VAR[65:ALF\VAR:]
C 63 I=0
    
```

```

C 73 L=(R<I+1)/0
C 83 2 1 P'
C 93 N=+VAR[I:]
C 103 O= , VAR[I:]
C 113 O= P , V(PH)
C 123 O= PP , V(PPH)
C 133 +VAR[I:]
C 143 =L
    
```

```

R=N ROUND X
C 13 R=(10.5000000001*X=10*N)+10*N } Rounds Number to n decimal
    
```

```

R= SORT X
C 13 'MEANS' , (VX)
C 23 I=0
C 33 XX=X
C 43 THERE:I=0
C 53 J=I+1
C 63 HERE:I=I+1
C 73 X(I)>X(I+1)=(X(I)<X(I+1)))+(X(I)=X(I+1))>(X(I+1)<X(I))
C 83 X(I)>X(I+1)=(X(I)>X(I+1)))+(X(I)=X(I+1))>(X(I+1)>X(I))
C 93 X=XX
C 103 +HERE=(I<(PM)-1)
C 113 +THERE=(I<(PM)-1)
C 123 'SORTED MEANS' , (VXX)
C 133 R=XX
    
```

Sorts Strings of #'s (Bubble Sort)

LISTVARS

ECS  
p 15 5  
pp 2

|       |       |      |      |      |
|-------|-------|------|------|------|
| 118.8 | 99.4  | 29.7 | 56.2 | 9.2  |
| 128   | 142   | 17   | 45.1 | 9    |
| 188.4 | 132.2 | 33.7 | 77.8 | 14.1 |
| 141   | 83.8  | 47.7 | 56.5 | 13.7 |
| 81.6  | 83.6  | 54.2 | 82.8 | 9.2  |
| 99.8  | 111.6 | 22.6 | 82.2 | 8.7  |
| 78.6  | 97.8  | 58.4 | 52.7 | 6.6  |
| 87.1  | 83.6  | 46.1 | 86.8 | 7.5  |
| 186.4 | 99.6  | 46.6 | 0    | 12.3 |
| 126.4 | 75.4  | 50.9 | 0    | 13.9 |
| 140.4 | 72    | 51   | 0    | 0    |
| 124.6 | 88.8  | 74   | 0    | 0    |
| 133.6 | 81.6  | 63   | 0    | 0    |
| 0     | 0     | 37.9 | 0    | 0    |
| 0     | 0     | 39.3 | 0    | 0    |

EARLY COMPRESSIVE STRENGTH (ECS)

| Column # | Material                  |
|----------|---------------------------|
| 1        | Life Regular Set          |
| 2        | Life Fast Set             |
| 3        | Nu Cap                    |
| 4        | Dycal Advanced Formula II |
| 5        | Dropsin                   |

WS  
p 10 5  
pp 2

|      |      |      |      |      |
|------|------|------|------|------|
| 4.86 | 4.41 | 3.32 | 0.73 | 0.86 |
| 5.93 | 3.83 | 2.94 | 1.06 | 0.96 |
| 4.86 | 2.22 | 2.4  | 0.83 | 0.49 |
| 3.23 | 4.4  | 3.97 | 0.96 | 0.98 |

WATER SOLUBILITY (WS)

| Column # | Material                  |
|----------|---------------------------|
| 1        | Life Regular Set          |
| 2        | Life Fast Set             |
| 3        | Nu Cap                    |
| 4        | Dycal Advanced Formula II |
| 5        | Dropsin                   |

|      |      |      |      |      |
|------|------|------|------|------|
| 4.79 | 3.84 | 4.83 | 2.41 | 0.99 |
| 4.21 | 2.95 | 2.73 | 0.72 | 0.92 |
| 5.4  | 3.35 | 4.11 | 0.83 | 0.99 |
| 3.18 | 2.59 | 3.82 | 0.87 | 0.77 |
| 4.87 | 2.32 | 4.87 | 0.72 | 0.64 |
| 3.76 | 3.44 | 0    | 0.52 | 0.86 |

PAS  
p 10 5  
pp 2

|                 |                 |                |                |
|-----------------|-----------------|----------------|----------------|
| -6.880000000E-1 | -3.000000000E-2 | -6.470000000E0 | -2.500000000E0 |
| -1.881000000E1  |                 |                |                |
| -5.200000000E-1 | 1.000000000E-22 | -6.480000000E0 | -2.210000000E0 |
| -1.127000000E1  |                 |                |                |
| -3.200000000E-1 | 5.000000000E-2  | -6.640000000E0 | -2.600000000E0 |
| -1.242000000E1  |                 |                |                |
| -3.780000000E-1 | 8.000000000E-2  | -6.790000000E0 | -2.420000000E0 |
| -1.288000000E1  |                 |                |                |
| -3.380000000E-1 | 8.000000000E-2  | -6.730000000E0 | -2.870000000E0 |
| -1.235000000E1  |                 |                |                |
| -4.300000000E-1 | -1.100000000E-1 | -6.490000000E0 | -2.930000000E0 |
| -1.276000000E1  |                 |                |                |
| -4.480000000E-1 | -1.200000000E-1 | -6.830000000E0 | -2.530000000E0 |
| -1.321000000E1  |                 |                |                |
| -1.580000000E-1 | -1.100000000E-1 | -6.820000000E0 | -3.920000000E0 |
| -1.843000000E1  |                 |                |                |
| -4.200000000E-1 | 5.000000000E-2  | -6.840000000E0 | -2.300000000E0 |
| -8.430000000E0  |                 |                |                |
| -2.700000000E-1 | -3.000000000E-2 | -6.270000000E0 | -2.550000000E0 |
| -1.854000000E1  |                 |                |                |

ACID SOLUBILITY (PAS)

| TFHCS<br>P 28 S |          |      |       | 24 HOUR COMPRESSIVE STRENGTH (TFHCS) |  |
|-----------------|----------|------|-------|--------------------------------------|--|
| Column #        | Material |      |       |                                      |  |
| 239             | 211.5    | 57.6 | 118   | 41.7                                 |  |
| 185.5           | 189      | 53.7 | 94.4  | 42.5                                 |  |
| 230             | 218.5    | 52.9 | 156.6 | 42.8                                 |  |
| 140             | 121      | 56.2 | 119.6 | 42.4                                 |  |
| 156.5           | 184      | 63.5 | 114.2 | 59.6                                 |  |
| 172             | 183.5    | 45.7 | 61.6  | 42.3                                 |  |
| 200             | 197.5    | 63   | 64.6  | 39                                   |  |
| 204.5           | 188.5    | 67.1 | 123.6 | 43.8                                 |  |
| 151.5           | 241      | 37.8 | 75.2  | 44.8                                 |  |
| 127             | 140.5    | 45   | 41.6  | 42.4                                 |  |
| 190.5           | 211.5    | 61   | 83.6  | 41.1                                 |  |
| 127.5           | 240      | 59   | 100.8 | 42.9                                 |  |
| 267             | 136      | 67.6 | 66.6  | 57.9                                 |  |
| 261.5           | 179      | 35   | 150.4 | 32.9                                 |  |
| 214.5           | 0        | 44.8 | 139.8 | 35.3                                 |  |
| 159             | 0        | 49.4 | 90    | 32.2                                 |  |
| 125.5           | 0        | 54.2 | 90.2  | 45.7                                 |  |
| 209             | 0        | 55.3 | 0     | 37.3                                 |  |
| 240.5           | 0        | 65.8 | 0     | 31.3                                 |  |
| 229             | 0        | 76.1 | 0     | 44.4                                 |  |

| WS<br>P 12 S |          |      |      |      | WATER SOLUBILITY (WS)<br>(Data Repeat) |  |
|--------------|----------|------|------|------|--|--|
| Column #     | Material |      |      |      |  |  |
| 4.84         | 4.41     | 3.32 | 0.75 | 0.86 |  |  |
| 5.93         | 3.83     | 2.94 | 1.06 | 0.96 |  |  |
| 4.06         | 2.22     | 2.44 | 0.83 | 0.69 |  |  |
| 5.23         | 4.59     | 3.87 | 0.96 | 0.98 |  |  |
| 4.79         | 3.84     | 4.63 | 2.41 | 0.99 |  |  |
| 4.21         | 2.95     | 2.73 | 0.72 | 0.92 |  |  |
| 5.4          | 3.35     | 4.11 | 0.83 | 0.99 |  |  |
| 3.18         | 2.39     | 3.82 | 0.87 | 0.77 |  |  |
| 4.67         | 2.32     | 4.67 | 0.72 | 0.64 |  |  |
| 3.76         | 3.44     | 0    | 0.52 | 0.84 |  |  |

| X<br>P 1<br>5 4 3 2 ROUND PAS |          |       |       |        |  | ACID SOLUBILITY (PAS) |  |
|-------------------------------|----------|-------|-------|--------|--|-----------------------|--|
| Column #                      | Material |       |       |        |  |                       |  |
| -0.4                          | -0.03    | -0.47 | -2.5  | -10.01 |  |                       |  |
| -0.52                         | 0        | -0.40 | -2.21 | -11.27 |  |                       |  |
| -0.32                         | 0.85     | -0.44 | -2.4  | -12.42 |  |                       |  |
| -0.37                         | -0.88    | -0.79 | -2.42 | -12.8  |  |                       |  |
| -0.33                         | -0.88    | -0.73 | -2.67 | -12.35 |  |                       |  |
| -0.45                         | -0.11    | -0.49 | -2.93 | -12.76 |  |                       |  |
| -0.44                         | -0.12    | -0.83 | -2.53 | -13.21 |  |                       |  |
| -0.15                         | -0.11    | -0.82 | -3.92 | -18.43 |  |                       |  |
| -0.42                         | 0.85     | -0.84 | -2.3  | -8.83  |  |                       |  |
| -0.27                         | -0.83    | -0.27 | -2.55 | -18.54 |  |                       |  |

TOTAL NUMBER OF DATA = 59  
 CORRECTION TERM = 266663.089  
 SUM OF SQUARES TOTAL = 92797.60102  
 NUMBER OF DATA IN TEST 1 = 13  
 NUMBER OF DATA IN TEST 2 = 13  
 NUMBER OF DATA IN TEST 3 = 15  
 NUMBER OF DATA IN TEST 4 = 8  
 NUMBER OF DATA IN TEST 5 = 10  
 DEGREES OF FREEDOM = 54  
 SUM OF SQUARES BETWEEN = 74818.18488  
 SUM OF SQUARES WITHIN = 17979.41615  
 MEAN STANDARD WEIGHT = 332.9558545  
 STANDARD ERROR OF MEANS = 5.446742374  
 DF = 54  
 ENTER SIGNIFICANT STUDENT RANGES  
 D1: 3.76 4.28 4.59 4.82  
 CRITICAL DIFFERENCES = 28.47982652 23.31214296 25.0806393 26.25339444  
 MEANS = 112.8238769 91.64615385 44.14 67.5125 10.62  
 SORTED MEANS = 10.62 44.14 67.5125 91.64615385 112.8238769  
 1 AND 2 SIGNIFICANT  
 1 AND 3 SIGNIFICANT  
 1 AND 4 SIGNIFICANT  
 1 AND 5 SIGNIFICANT  
 2 AND 3 SIGNIFICANT  
 2 AND 4 SIGNIFICANT  
 2 AND 5 SIGNIFICANT  
 3 AND 4 SIGNIFICANT  
 3 AND 5 SIGNIFICANT  
 4 AND 5 SIGNIFICANT

TFHCS  
P 20 S

|       |       |      |       |      |
|-------|-------|------|-------|------|
| 239   | 211.5 | 57.6 | 118   | 41.7 |
| 185.5 | 169   | 53.7 | 94.4  | 42.5 |
| 230   | 218.5 | 52.9 | 156.6 | 42.8 |
| 140   | 121   | 56.2 | 119.4 | 42.6 |
| 156.5 | 184   | 63.5 | 114.2 | 50.6 |
| 172   | 183.5 | 65.7 | 61.6  | 42.3 |
| 208   | 197.5 | 63   | 64.6  | 39   |
| 204.5 | 188.5 | 67.1 | 123.6 | 43.8 |
| 151.5 | 241   | 37.8 | 73.2  | 44.8 |
| 127   | 160.5 | 63   | 81.6  | 42.4 |
| 196.5 | 211.5 | 61   | 83.6  | 41.1 |
| 127.5 | 240   | 59   | 108.8 | 42.9 |
| 267   | 136   | 67.6 | 64.6  | 37.9 |
| 261.5 | 179   | 55   | 150.8 | 32.9 |
| 214.5 | 8     | 44.8 | 139.8 | 35.3 |
| 159   | 0     | 69.4 | 98    | 32.2 |
| 125.5 | 0     | 54.2 | 90.2  | 45.7 |
| 209   | 0     | 35.3 | 0     | 37.3 |
| 248.5 | 0     | 65.8 | 0     | 31.3 |
| 229   | 0     | 76.1 | 0     | 44.4 |

24 HOUR COMPRESSIVE STRENGTH (TFHCS)

| Column # | Material                  |
|----------|---------------------------|
| 1        | Life Regular Set          |
| 2        | Life Fast Set             |
| 3        | Nu Cap                    |
| 4        | Dycal Advanced Formula II |
| 5        | Dropsin                   |

WS  
P 12 S

PP 2

|      |      |      |      |      |
|------|------|------|------|------|
| 4.84 | 4.41 | 3.32 | 0.73 | 0.86 |
| 5.93 | 3.83 | 2.94 | 1.06 | 0.96 |
| 4.86 | 2.22 | 2.4  | 0.83 | 0.69 |
| 5.23 | 4.59 | 3.97 | 0.96 | 0.98 |
| 4.79 | 3.84 | 4.83 | 2.41 | 0.99 |
| 4.21 | 2.95 | 2.73 | 0.72 | 0.92 |
| 5.4  | 3.35 | 4.11 | 0.83 | 0.99 |
| 5.18 | 2.39 | 3.82 | 0.87 | 0.77 |
| 4.67 | 2.32 | 4.87 | 0.72 | 0.64 |
| 3.76 | 3.44 | 0    | 0.52 | 0.84 |

WATER SOLUBILITY (WS)  
(Data Repeat)

X  
P 3  
S 4 J  
2 ROUND PAS

|       |       |       |       |        |
|-------|-------|-------|-------|--------|
| -8.4  | -8.03 | -6.47 | -2.3  | -10.81 |
| -8.32 | 0     | -6.48 | -2.21 | -11.27 |
| -8.32 | 0.05  | -6.44 | -2.4  | -12.42 |
| -8.37 | -0.08 | -6.79 | -2.42 | -12.8  |
| -8.33 | -0.08 | -6.75 | -2.87 | -12.35 |
| -8.45 | -0.11 | -6.49 | -2.93 | -12.76 |
| -8.44 | -0.12 | -6.83 | -2.53 | -13.21 |
| -8.15 | -0.11 | -6.82 | -3.92 | -10.43 |
| -8.42 | 0.05  | -6.84 | -2.3  | -8.83  |
| -8.27 | -0.03 | -6.27 | -2.55 | -10.54 |

ACID SOLUBILITY (PAS)

| Column # | Material                  |
|----------|---------------------------|
| 1        | Life Regular Set          |
| 2        | Life Fast Set             |
| 3        | Nu Cap                    |
| 4        | Dycal Advanced Formula II |
| 5        | Dropsin                   |

ECS  
GO  
TOTAL NUMBER OF DATA= 59  
CORRECTION TERM= 266663.089  
SUM OF SQUARES TOTAL= 92797.80102  
NUMBER OF DATA IN TEST 1= 13  
NUMBER OF DATA IN TEST 2= 13  
NUMBER OF DATA IN TEST 3= 15  
NUMBER OF DATA IN TEST 4= 8  
NUMBER OF DATA IN TEST 5= 10  
DEGREES OF FREEDOM= 54  
SUM OF SQUARES BETWEEN= 74818.18488  
SUM OF SQUARES WITHIN= 17979.61613  
MEAN STANDARD WEIGHT= 332.9558543  
STANDARD ERROR OF MEANS = 5.446742374  
DF= 54  
ENTER SIGNIFICANT STUDENT RANGES

EARLY COMPRESSIVE STRENGTH (ECS)

- 1 = Dropsin
- 2 = Nu Cap
- 3 = Dycal Advanced Formula II
- 4 = Life Fast Set
- 5 = Life Regular Set

01  
3.76 4.28 4.39 4.82  
CRITICAL DIFFERENCES= 28.47982652 23.31214294 25.0806393 26.25339464  
MEANS= 112.8230769 91.64615385 44.19 67.5125 10.42  
SORTED MEANS= 10.42 44.14 67.5125 91.64615385 112.8230769  
1 AND 2 SIGNIFICANT  
1 AND 3 SIGNIFICANT  
1 AND 4 SIGNIFICANT  
1 AND 5 SIGNIFICANT  
2 AND 3 SIGNIFICANT  
2 AND 4 SIGNIFICANT  
2 AND 5 SIGNIFICANT  
3 AND 4 SIGNIFICANT  
3 AND 5 SIGNIFICANT  
4 AND 5 SIGNIFICANT

0+PAS  
 3.82 4.37 4.7 4.93  
 3.82 4.37 4.7 4.93  
 00

TOTAL NUMBER OF DATA= 50  
 CORRECTION TERM= 985.166152  
 SUM OF SQUARES TOTAL= 960.843448  
 NUMBER OF DATA IN TEST 1= 10  
 NUMBER OF DATA IN TEST 2= 10  
 NUMBER OF DATA IN TEST 3= 10  
 NUMBER OF DATA IN TEST 4= 10  
 NUMBER OF DATA IN TEST 5= 10  
 DEGREES OF FREEDOM= 45  
 SUM OF SQUARES BETWEEN= 940.729388  
 SUM OF SQUARES WITHIN= 20.11406  
 MEAN STANDARD WEIGHT= 0.4469791111  
 STANDARD ERROR OF MEANS = 0.211418895  
 DF= 45  
 ENTER SIGNIFICANT STUDENT RANGES

Q:  
 3.82 4.37 4.7 4.93  
 CRITICAL DIFFERENCES= 0.8076198351 0.9259001779 0.9936683835 1.042294789  
 MEANS= -0.367 -0.046 -0.636 -2.683 -11.542  
 SORTED MEANS= -11.542 -0.636 -2.683 -0.367 -0.046  
 1 AND 2 SIGNIFICANT  
 1 AND 3 SIGNIFICANT  
 1 AND 4 SIGNIFICANT  
 1 AND 5 SIGNIFICANT  
 2 AND 3 SIGNIFICANT  
 2 AND 4 SIGNIFICANT  
 2 AND 5 SIGNIFICANT  
 3 AND 4 SIGNIFICANT  
 3 AND 5 SIGNIFICANT  
 4 AND 5 NONSIGNIFICANT

00  
 TOTAL NUMBER OF DATA= 50  
 CORRECTION TERM= 985.166152  
 SUM OF SQUARES TOTAL= 960.843448  
 NUMBER OF DATA IN TEST 1= 10  
 NUMBER OF DATA IN TEST 2= 10  
 NUMBER OF DATA IN TEST 3= 10  
 NUMBER OF DATA IN TEST 4= 10  
 NUMBER OF DATA IN TEST 5= 10  
 DEGREES OF FREEDOM= 45  
 SUM OF SQUARES BETWEEN= 940.729388  
 SUM OF SQUARES WITHIN= 20.11406  
 MEAN STANDARD WEIGHT= 0.4469791111  
 STANDARD ERROR OF MEANS = 0.211418895  
 DF= 45  
 ENTER SIGNIFICANT STUDENT RANGES

J:  
 2.86 3.44 3.79 4.04  
 CRITICAL DIFFERENCES= 0.6846377823 0.7272806892 0.801277271 0.8541319722  
 MEANS= -0.367 -0.046 -0.636 -2.683 -11.542  
 SORTED MEANS= -11.542 -0.636 -2.683 -0.367 -0.046  
 1 AND 2 SIGNIFICANT  
 1 AND 3 SIGNIFICANT  
 1 AND 4 SIGNIFICANT  
 1 AND 5 SIGNIFICANT  
 2 AND 3 SIGNIFICANT

2 AND 4 SIGNIFICANT  
 2 AND 5 SIGNIFICANT  
 3 AND 4 SIGNIFICANT  
 3 AND 5 SIGNIFICANT  
 4 AND 5 NONSIGNIFICANT

## ACID SOLUBILITY (PAS)

1 = Dropsin  
 2 = Nu Cap  
 3 = Dycal Advanced Formula II  
 4 = Life Regular Set  
 5 = Life Pasc Set



```

QTFRCS
GO
TOTAL NUMBER OF DATA= 91
CORRECTION TERM= 1091594.603
SUM OF SQUARES TOTAL= 417612.3868
NUMBER OF DATA IN TEST 1= 20
NUMBER OF DATA IN TEST 2= 14
NUMBER OF DATA IN TEST 3= 20
NUMBER OF DATA IN TEST 4= 17
NUMBER OF DATA IN TEST 5= 20
DEGREES OF FREEDOM= 86
SUM OF SQUARES BETWEEN= 328738.9785
SUM OF SQUARES WITHIN= 88873.41632
MEAN STANDARD WEIGHT= 1833.411818
STANDARD ERROR OF MEANS = 7.618727073
DF= 86
ENTER SIGNIFICANT STUDENT RANGES
Q:
3.7 4.2 4.5 4.71
CRITICAL DIFFERENCES= 28.15969017 31.96585371 34.24827183 35.84652452
MEANS= 191.5 169.9442857 59.435 182.0588235 41.675
SORTED MEANS= 41.675 59.435 182.0588235 169.9442857 191.5
1 AND 2 NONSIGNIFICANT
1 AND 3 SIGNIFICANT
1 AND 4 SIGNIFICANT
1 AND 5 SIGNIFICANT
2 AND 3 SIGNIFICANT
2 AND 4 SIGNIFICANT
2 AND 5 SIGNIFICANT
3 AND 4 SIGNIFICANT
3 AND 5 SIGNIFICANT
4 AND 5 NONSIGNIFICANT
GO
TOTAL NUMBER OF DATA= 91
CORRECTION TERM= 1091594.603
SUM OF SQUARES TOTAL= 417612.3868
NUMBER OF DATA IN TEST 1= 20
NUMBER OF DATA IN TEST 2= 14
NUMBER OF DATA IN TEST 3= 20
NUMBER OF DATA IN TEST 4= 17
NUMBER OF DATA IN TEST 5= 20
DEGREES OF FREEDOM= 86
SUM OF SQUARES BETWEEN= 328738.9785
SUM OF SQUARES WITHIN= 88873.41632
MEAN STANDARD WEIGHT= 1833.411818
STANDARD ERROR OF MEANS = 7.618727073
DF= 86
ENTER SIGNIFICANT STUDENT RANGES
Q:
2.8 3.36 3.68 3.92
CRITICAL DIFFERENCES= 21.31883581 25.57284297 28.88747563 29.83405013
MEANS= 191.5 169.9442857 59.435 182.0588235 41.675
SORTED MEANS= 41.675 59.435 182.0588235 169.9442857 191.5
1 AND 2 NONSIGNIFICANT
1 AND 3 SIGNIFICANT
1 AND 4 SIGNIFICANT
1 AND 5 SIGNIFICANT
2 AND 3 SIGNIFICANT
2 AND 4 SIGNIFICANT
2 AND 5 SIGNIFICANT
3 AND 4 SIGNIFICANT
3 AND 5 SIGNIFICANT
4 AND 5 SIGNIFICANT

```

```

UPRS
GO
TOTAL NUMBER OF DATA= 49
CORRECTION TERM= 347.7159367
SUM OF SQUARES TOTAL= 132.9885633
NUMBER OF DATA IN TEST 1= 10
NUMBER OF DATA IN TEST 2= 10
NUMBER OF DATA IN TEST 3= 9
NUMBER OF DATA IN TEST 4= 10
NUMBER OF DATA IN TEST 5= 10
DEGREES OF FREEDOM= 44
SUM OF SQUARES BETWEEN= 116.6721744
SUM OF SQUARES WITHIN= 16.3163889
MEAN STANDARD WEIGHT= 0.3788270202
STANDARD ERROR OF MEANS = 0.1946965898
DF= 44
ENTER SIGNIFICANT STUDENT RANGES
D:
  3.76 4.28 4.39 4.82
CRITICAL DIFFERENCES= 0.7320591775 0.8333014042 0.893657347 0.9384375627
MEANS= 4.887 3.354 3.398888889 0.967 0.866
SORTED MEANS= 0.866 0.967 3.354 3.398888889 4.887
1 AND 2 NONSIGNIFICANT
1 AND 3 SIGNIFICANT
1 AND 4 SIGNIFICANT
1 AND 5 SIGNIFICANT
2 AND 3 SIGNIFICANT
2 AND 4 SIGNIFICANT
2 AND 5 SIGNIFICANT
3 AND 4 NONSIGNIFICANT
3 AND 5 SIGNIFICANT
4 AND 5 SIGNIFICANT
GO
TOTAL NUMBER OF DATA= 49
CORRECTION TERM= 347.7159367
SUM OF SQUARES TOTAL= 132.9885633
NUMBER OF DATA IN TEST 1= 10
NUMBER OF DATA IN TEST 2= 10
NUMBER OF DATA IN TEST 3= 9
NUMBER OF DATA IN TEST 4= 10
NUMBER OF DATA IN TEST 5= 10
DEGREES OF FREEDOM= 44
SUM OF SQUARES BETWEEN= 116.6721744
SUM OF SQUARES WITHIN= 16.3163889
MEAN STANDARD WEIGHT= 0.3788270202
STANDARD ERROR OF MEANS = 0.1946965898
DF= 44
ENTER SIGNIFICANT STUDENT RANGES
-D:
  2.86 3.44 3.79 4.04
CRITICAL DIFFERENCES= 0.3568322467 0.6697562688 0.7379000752 0.7865742226
MEANS= 4.767 3.354 3.398888889 0.967 0.866

!
!

SORTED MEANS= 0.866 0.967 3.354 3.398888889 4.887
1 AND 2 NONSIGNIFICANT
1 AND 3 SIGNIFICANT
1 AND 4 SIGNIFICANT
1 AND 5 SIGNIFICANT
2 AND 3 SIGNIFICANT
2 AND 4 SIGNIFICANT
2 AND 5 SIGNIFICANT
3 AND 4 NONSIGNIFICANT
3 AND 5 SIGNIFICANT
4 AND 5 SIGNIFICANT
)SAVE
10:38:57 03/01/85 TOM
READY )OFF HOLD
OUT=

```

## WATER SOLUBILITY (WS)

- 1 = Dropsin
- 2 = Dycal Advanced Formula II
- 3 = Life Fast Set
- 4 = Nu Cap
- 5 = Life Regular Set

APPROVAL SHEET

The thesis submitted by Thomas Sarna has been read and approved by the following committee:

Dr. James L. Sandrik, Director  
Professor, Dental Materials, Loyola

Dr. Franklin S. Weine  
Professor, Endodontics, Loyola

Dr. Gary N. Taylor  
Professor, Endodontics, 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.

April 1, 1986  
Date

James L. Sandrik  
Director's Signature