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## RESEARCH AND EDUCATION

Assessment of CAD-CAM polymers for digitally fabricated complete dentures

### ABSTRACT

**Statement of problem.** Information on the mechanical properties of the materials used for manufacturing computer-engineered complete dentures is scarce.

**Purpose.** The purpose of this in vitro study was to evaluate the mechanical properties of 3 prepolymerized polymethyl methacrylate (PMMA) resins used in the fabrication of computer-aided design and computer-aided manufacturing (CAD-CAM) milled complete dentures (CDs), as well as 2 denture base polymers used for conventionally fabricated CDs.

**Material and methods.** Three CAD-CAM materials were evaluated: Degos Dental L-Temp, Ivobase CAD, and Zirkozahn Temp Basic Tissue. Two materials used for conventionally manufactured dentures were also included as controls (Palapress and Paladon 65). Each material type was sectioned into bars for flexural strength, nanohardness, elastic modulus, and surface microhardness evaluation ( $n=8/\text{material}$ ). Half of the specimens were stored in water for 30 days while the other half was dry-stored. A 2-way analysis of variance (ANOVA) was conducted to detect the effect of material and storage on the evaluated properties ( $\alpha=.05$ ). Linear contrasts were conducted to compare the differences among the 3 CAD-CAM materials and the conventional ones.

**Results.** Material type and storage had a significant influence on the flexural strength, nanohardness, elastic modulus, and surface hardness of the materials investigated ( $P<.001$ ). The post hoc Scheffé test for flexural strength revealed a nonsignificant difference in the interaction

between Degos L-Temp and Paladon ( $P=1.000$ ). In terms of nanohardness, no difference was found when comparing Palapress with Paladon, as well as IvoBase CAD with Zirkozahn Temp Basic ( $P=1.000$ ). A nonsignificant interaction in terms of surface hardness was also found between IvoBase CAD and Palapress ( $P=.575$ ).

**Conclusions.** The tested materials showed variation in their mechanical properties with satisfactory behavior of the CAD-CAM materials. However, the results obtained when testing the materials used for the conventional fabrication of complete dentures suggest that their use might still be advisable.

## CLINICAL IMPLICATIONS

The mechanical behavior of the materials used for the fabrication of computer-engineered complete dentures varies among different CAD-CAM systems. The results presented here should allow clinicians to make comparisons between the systems investigated with the aim of improving patient care and satisfaction, as well as achieving more predictable treatment outcomes.

## INTRODUCTION

Complete dentures (CDs) continue to represent an important treatment option for an aging population,<sup>1,2</sup> with an expected increased demand for CDs.<sup>3,4</sup> Conventional methods for designing and fabricating CDs involve multiple clinical and laboratory procedures.<sup>5</sup> Additionally, complications related to conventional CDs include fracture, loss of retention, inadequate esthetics, and imprecise occlusal vertical dimension.<sup>6-9</sup> Furthermore, CD underperformance has been attributed to deficient mechanical properties of denture base

resins.<sup>10-13</sup> A variety of methods have been used for enhancing these properties, including modifying the microstructure by blending additives,<sup>14-16</sup> adjusting the liquid-to-powder ratio,<sup>17</sup> and improving the processing protocols, resulting in diverse outcomes.<sup>18-22</sup> The introduction of new manufacturing techniques and new materials have been addressed as potential solutions.<sup>14,23</sup>

Computer-aided design and computer-aided manufacturing (CAD-CAM) has become a new approach for the design and fabrication of CDs,<sup>24,25</sup> with the avoidance of the polymerization shrinkage seen in conventional CDs.<sup>26</sup> The CAD-CAM manufacturing process is subtractive, where the denture bases are milled from fully polymerized acrylic resin blanks,<sup>27</sup> resulting in nondistorted prostheses.<sup>28</sup> Preformed PMMA blanks are polymerized by injection under high temperature and pressure, which prevents shrinkage of the computer-engineered CDs.<sup>24,29</sup> Considering the potentially enhanced physical and mechanical properties of prepolymerized PMMA blanks for CAD-CAM applications, considerable improvements in the quality of CAD-CAM CDs are expected.<sup>30,31</sup>

Computer-engineered CDs are predominantly fabricated by using scanned data for digital design, followed by either computerized numerical control milling of the denture base, rapid prototyping for trial placement and conventional processing,<sup>32</sup> or printing of the prosthesis.<sup>33</sup> Other advantages of computer-engineered CDs over the conventional processing methods include a reduction in the number of appointments needed,<sup>34</sup> improved fit,<sup>35,36</sup> and electronic archiving.<sup>34</sup> Improved adaptation for milled computer-engineered CDs in comparison with conventional methods of processing has been reported, with the suggestion that the enhanced adaptation provides a more retentive prosthesis.<sup>35</sup> Furthermore, having the possibility of duplicating an existing CD and the ability to digitally archive the information for future treatments improves patient care.<sup>37</sup>

Although the resins used for computer-engineered CDs and the conventional resins are chemically similar, their production process is entirely different. Whether the PMMA resins manufactured under newer protocols have enhanced mechanical properties and might function successfully under clinical conditions requires investigation. Furthermore, evidence relating to the characterization of the mechanical properties of these PMMA resins used for the milling of computer-engineered CDs is scarce. Hence, the purpose of the present in vitro study was to evaluate the mechanical properties of 3 prepolymerized PMMA resins that are used in the manufacturing of CAD-CAM milled CDs, as well as 2 denture base polymers used for conventionally manufactured CDs. The null hypothesis was that no difference would be found in the mechanical properties between the prepolymerized CAD-CAM PMMA blanks and the traditional PMMA denture base polymers used in the conventional manufacturing process of CDs.

## **MATERIAL AND METHODS**

Three CAD-CAM materials used for digitally fabricated dentures were evaluated: LT (Degos Dental L-Temp; Degos Dental GmbH), IB (IvoBase CAD; Ivoclar Vivadent AG), and TB (Zirkonzahn Temp Basic Tissue; Zirkonzahn SRL). Additionally, 2 denture base polymers used for conventionally fabricated dentures were included as controls, an autopolymerizing denture base polymer, PP (Palapress; Kulzer GmbH) and a dental acrylic resin that requires heat-activated polymerization, PD (Paladon 65; Kulzer GmbH). The composition of the materials is shown in Table 1.

The autopolymerizing denture base polymer specimens were made following the manufacturer's recommendation with a powder-liquid ratio of 10 g/7 mL. The heat-polymerized

specimens were fabricated from clear denture base polymer. The powder-liquid ratio was 10 g/4 mL. A mixture of acrylic resin was poured to fill Teflon molds (200×3.5×10 mm). The molds were placed in a hot water polymerization unit (Kulzer GmbH) at 70 °C for 90 minutes, with the molds completely covered with water. The water bath temperature was raised to boiling and maintained at the boiling point for at least 30 minutes. Subsequently, the molds slowly cooled in the water bath. After cooling, the polymerized specimens were removed from the mold, cut to the desired length, and wet ground with successively finer grades of silicon carbide papers from 500 to 1200 grit (Silicon Carbide Grinding Paper; Buehler) (LabPol-21; Struers) to the predetermined dimensions (65×3.2×10 mm).

Thirty-two specimens were fabricated from clear autopolymerizing resin and from the heat-polymerizing denture base polymer. For each material, half of the specimens (n=8/per material) were stored in water at 37 °C for 30 days. The other half was dry stored for an equal number of days under ambient laboratory conditions (23 ±1 °C). Forty-eight specimens were obtained from the CAD-CAM blanks using a low-speed water-cooled diamond saw (Secotom 50; Struers). The specimens were then wet polished with silicon carbide grinding paper 1200 grit (Silicon Carbide Grinding Paper; Buehler) (LabPol-21; Struers). Half of the specimens were dry stored while the other half was kept in water at 37 °C for 30 days (n=8/per material).

The flexural strength was determined with a static 3-point bend test (Model LRX; Lloyds Instruments Ltd) in air.<sup>38-41</sup> The testing machine was programmed to a constant displacement rate of 1 mm/minute, a preload of 1.0 N, and a preload speed of 10 mm/minute. The test was considered finished when the current load was reduced to 50% of the maximum load or was less than 1.0 N. Once the dry-stored specimens were fractured after the 3-point bend test, they were prepared for repair by wetting the surface with methylmethacrylate liquid for 3 minutes,<sup>42</sup>

repaired using a clear autopolymerizing resin (Palapress; Kulzer GmbH), and stored dry under ambient laboratory conditions ( $23 \pm 1$  °C) for 24 hours. Next, a static-3-point bend test was performed to evaluate the flexural strength of the materials after being repaired.

Eighty specimens ( $2 \times 10 \times 10$  mm) were obtained ( $n=16$ /material). They were wet ground flat with 1200 grit (Silicon Carbide Grinding Paper; Buehler). The specimens were then cleaned in deionized water in an ultrasonic cleaning device (Quantrex 90; L&R Ultrasonics) for 10 minutes. Half of the specimens were stored dry while the rest were stored in distilled water at 37 °C for 30 days. Surface microhardness testing (VHN) was performed on selected portions of the specimens with a Vickers hardness testing machine (Duramin-5; Struers). The force used was 245.2 mN for 15 seconds. One indentation was made on each specimen to obtain the surface microhardness value, and the deformation of the indentation was measured after 3 seconds from the point of releasing the load.

Nanoindentation was used to measure the nanohardness and modulus of elasticity of the tested materials. Four indentations were made on each specimen ( $n=8$ /material) with the aid of a  $\times 20$  objective lens for accuracy and using a nanomechanical tester (TI 980 TriboIndenter; Bruker) equipped with a Berkovich diamond indenter tip of nominal radius of approximately 100 nm. The loading and unloading rates used were 0.5 mN/second, with a dwell time of 10 seconds. The maximum load was set to 5.0 mN.

A specimen of each material was placed in tetrahydrofuran (THF) solvent (Sigma-Aldrich) for 10 seconds and allowed to dry under ambient laboratory conditions for 24 hours. This was conducted in order to identify differences in the materials' cross-linking densities. The gold-sputtered surfaces were examined with a scanning electron microscope (SEM) (JSM 5500; Jeol) to analyze the polymer structure of the different CAD-CAM materials.

All data for flexural strength, surface hardness, nanohardness, and modulus of elasticity were collected and statistically analyzed. A 2-way analysis of variance (ANOVA) was conducted to detect the effect of material and storage as the independent variables of the evaluated properties ( $\alpha=.05$ ). A 1-way ANOVA was conducted to identify the effect that the repair procedure had on the flexural strength of the materials investigated. Linear contrasts were conducted to compare the differences between the three CAD-CAM materials and the conventional ones. Statistical software (IBM SPSS Statistics, v24; IBM Corp.) was used to conduct all analyses.

## RESULTS

In terms of flexural strength for dry and water-stored specimens, the 2-way ANOVA revealed a statistically significant difference according to material, storage, and their interaction ( $P<.001$ ). The post hoc Scheffé test revealed a nonsignificant difference on the interaction between LT and PD ( $P=1.000$ ). When evaluating flexural strength by comparing the nonrepaired and repaired samples, a statistically significant difference was found ( $P<.001$ ), except for the interaction between LT and PD ( $P=.685$ ) and between IB and PP ( $P=.995$ ) (Fig. 1). Figure 2 shows the maximum bend stress for each material, and Figure 3 displays the 2 scenarios found in the specimens after fracture. Some specimens had more space than others for the addition of the repair resin.

A statistically significant difference was found for the surface hardness of dry- and water-stored specimens, according to material, storage, and their interaction ( $P<.001$ ), except for the interaction between IB and PP ( $P=.575$ ) (Fig. 4). A statistically significant difference was found for nanohardness among the materials ( $P<.001$ ). However, no difference was found when PP was



compared with PD or IB with TB ( $P=1.000$ ) (Fig. 5). A statistically significant difference was found for the modulus of elasticity among the materials ( $P<.001$ ), with the highest difference being between PP and TB ( $P<.001$ ) (Fig. 6).

Linear contrasts were also conducted to compare the 3 CAD-CAM materials (LT, IB, and TB) versus the 2 conventional methods (PP and PD). A statistically significant difference was found among them in terms of bend stress ( $P=.009$ ), surface hardness ( $P=.009$ ), nanohardness ( $P<.001$ ), and elastic modulus ( $P=.003$ ).

The cross-linking densities of the materials differed in terms of their polymeric structure (Fig. 7). PP demonstrated an eventual multiphasic polymeric structure composed of polymer beads of linear polymer, likely PMMA, and a surrounding cross-linked matrix. LT, TB without the exposure to THF, and PD did not show a multiphasic polymer structure. When exposing TB to THF, a different polymeric structure was seen, which might be because of material damage since the surface looked slightly burned. IB showed some porosities on the surface and some particles that might have been inorganic fillers.

## **DISCUSSION**

The purpose of this study was to investigate the mechanical properties of 3 CAD-CAM materials used for computer-engineered complete dentures and 2 PMMA denture base polymers used for the conventional fabrication of the same kind of prostheses. The mechanical properties evaluated were flexural strength, nanohardness, elastic modulus and surface hardness. The null hypothesis was rejected as significant differences were found among the materials investigated.

High mechanical strength is an essential prerequisite for successful denture base materials. However, clinical reports on the fracture of complete dentures have indicated that the

mechanical properties of PMMA are not completely satisfactory with regard to longevity of the denture base.<sup>8,9</sup> A variety of approaches have been reported to improve the mechanical properties of acrylic-based materials; however, some of them were not transferred into clinical applications due to processing difficulties or high costs.<sup>20-22</sup>

A variety of factors can affect the initiation and propagation of cracks and the consequent fracture of denture base materials, including poor fit, anatomic notches, and poor design.<sup>20</sup> In those situations, a denture base is loaded under flexure fatigue, and, once the maximum mechanical capacity of the material is exceeded, it fractures. The 3-point bend test is the most common method used for measuring flexural properties of denture base materials adopted by international standards for polymer materials.<sup>38</sup> The use of this specification for flexural testing to compare the flexural strength, flexural modulus, and fracture energy of various denture base materials has been reported.<sup>39-41</sup> Flexural strength for prepolymerized resins for CAD-CAM applications, in addition to a heat-polymerizing and an autopolymerizing resin, were investigated in the present study. Although not recommended, autopolymerizing denture base polymers are still often used for complete denture fabrication or relining, which is why they were included in this study. However, the autopolymerizing resin included in this study (Palapress; Kulzer GmbH) is not recommended by the manufacturer for complete dentures but only for removable partial dentures. The load-deflection graphs obtained in this study were clearly different between the materials investigated, indicating dissimilarity in the mechanical behavior of the denture base materials depending on the kind of material and on the different processing methods (Fig. 2).

Heat-polymerizing denture base polymer is widely used for the fabrication of CDs<sup>14</sup> because of its physical and mechanical characteristics, ease of processing, and affordability.

However, polymerization drawbacks have been reported for conventional denture processing, including denture porosity, crazing, and volumetric and linear shrinkage.<sup>12,13</sup>

In the current study, the heat-polymerizing denture base material investigated, PD, along with a CAD-CAM prepolymerized resin, LT, had the highest values of flexural strength, consistent with those found in a similar report on heat-polymerizing denture base.<sup>43</sup> PMMA blanks are polymerized under high temperature and pressure, which promotes the formation of longer polymer chains, leading to a higher degree of monomer conversion and lower values of residual monomer, as well as minimal porosity.<sup>28</sup> Additionally, the processing conditions of CAD-CAM blanks decrease the intermolecular distances.<sup>44</sup> This could explain the behavior of the CAD-CAM material investigated, LT.

The elastic modulus is a parameter with clinical relevance for CDs since denture base materials with high elastic moduli are more resistant to elastic deformation, allowing the fabrication of dentures with thinner bases. The tested CAD-CAM materials showed a high modulus of elasticity as was reported in a previous study.<sup>45</sup> This high modulus of elasticity means that these materials might take more force to deform before fracture. However, a denture base resistant to deformation provides a stable occlusion and appropriate positioning of the mandible.

Surface hardness provides information on the cross-linking density of a material and its resistance to wear.<sup>18</sup> The results of the present study showed a significant difference in the surface hardness of the materials investigated, except between IB and PP. The highest mean values for dry- and water-stored specimens were found in the heat-polymerizing denture base polymer (PD). This might be associated with the heat-induced free radical polymerization process of the resin, which is connected to the formation of a partial cross-linked polymer chain

because of the presence of minor quantities of cross-linking dimethacrylate monomers, resulting in superior hardness. It is not known whether partial cross-linking occurs during the polymerization process of the CAD-CAM resins with the addition of inorganic fillers.<sup>11</sup>

The mechanical properties of CAD-CAM resins may also allow the fabrication of overdentures without metal or fiber reinforcement, as crack propagation and eventual fracture may be prevented in areas where an attachment system requires thinning of the denture bases. Clinical evidence is needed to confirm those assumptions. Additionally, the behavior of CAD-CAM resins under dynamic loading conditions needs to be studied because the majority of denture fractures are caused by fatigue.<sup>46</sup>

## **CONCLUSIONS**

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The tested materials showed variation in their mechanical properties with satisfactory behavior of the CAD-CAM materials.
2. However, the results obtained when testing the materials used for the conventional fabrication of complete dentures suggest that their use might still be advisable since the evaluated CAD-CAM denture base resins did not generally have better mechanical properties than manually processed denture base polymers.

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**TABLE**

Table 1. Materials used

<b>Brand</b>	<b>Code</b>	<b>Composition according to manufacturer</b>	<b>Manufacturer</b>
L-Temp	LT	Poly(methyl methacrylate)	Degos Dental GmbH
Temp Basic Tissue	TB	Poly(methyl methacrylate)	Zirkonzahn
IvoBase CAD	IB	Poly(methyl methacrylate)	Ivoclar Vivadent AG
Palapress	PP	Liquid: methylmethacrylate (> 90%); tetramethylene dimethacrylate (0–5%); 2-(2H-Benzotriazol-2-yl)-4-methylphenol (< 1%), N,N-dimethyl-p-toluidine (< 1%) Powder: polymethylmethacrylate (> 95%); Bis(p-Chlorbenzoyl) peroxide (0–5%)	Kulzer GmbH
Paladon 65	PD	Liquid: methylmethacrylate (>90%), BDMA(0–5%) Powder: Methacrylate copolymonomers (0–5%),BPO<1%	Kulzer GmbH

## FIGURES

Figure 1. Mean flexural strength values for nonrepaired and repaired specimens. Same letters indicate nonsignificant difference between materials ( $P > .05$ ). Error bars represent standard deviations. LT, (egos L-Temp; IB, IvoBase, PP, Palapress, TB, Zirkonzahn Temp Basic; PD, Paladon.

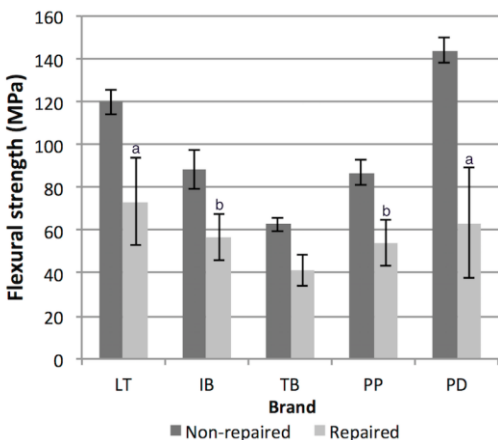


Figure 2. Maximum bend stress for each material and behavior under applied force. LT, (egos L-Temp; IB, IvoBase, PP, Palapress, TB, Zirkonzahn Temp Basic; PD, Paladon.

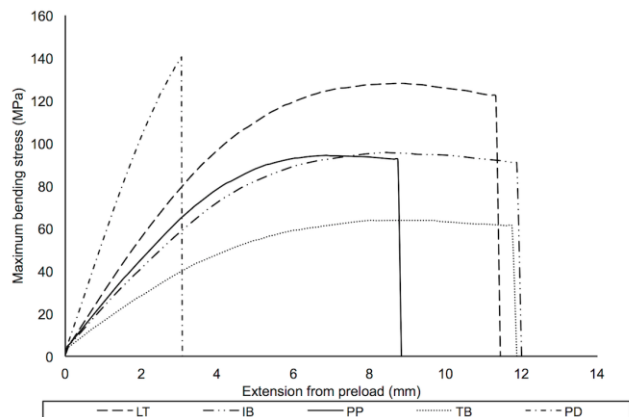


Figure 3. Representative fracture types. Some specimens had more space for addition of repair resin.

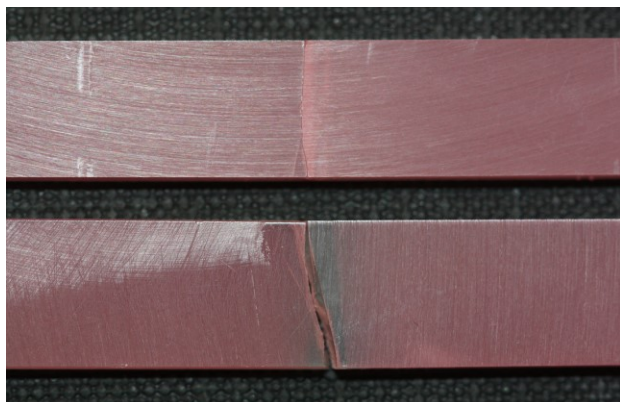


Figure 4. Mean surface hardness values for dry- and water-stored samples.

Same letters indicate nonsignificant difference between materials ( $P > .05$ ). Error bars represent standard deviations. LT, (egos L-Temp; IB, IvoBase, PP, Palapress, TB, Zirkonzahn Temp Basic; PD, Paladon.

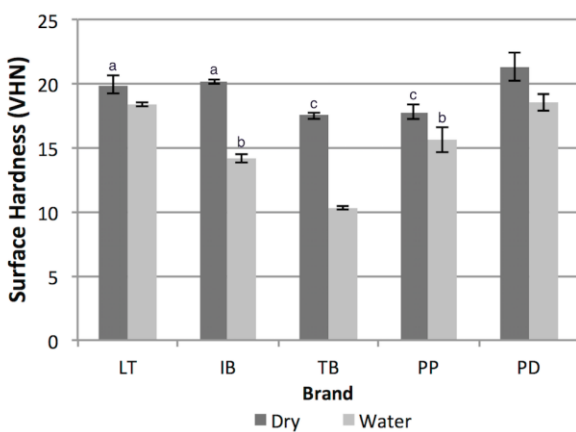


Figure 5. Mean nanohardness values.

Same letters indicate nonsignificant difference between materials ( $P > .05$ ). Error bars represent standard deviations. LT, (egos L-Temp; IB, IvoBase, PP, Palapress, TB, Zirkonzahn Temp Basic; PD, Paladon.

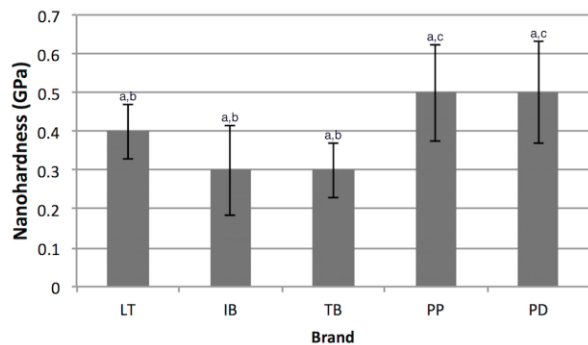


Figure 6. Mean elastic modulus values by nanoindentation.

Same letters indicate nonsignificant difference between materials ( $P > .05$ ). Error bars represent standard deviations. LT, (egos L-Temp; IB, IvoBase, PP, Palapress, TB, Zirkonzahn Temp Basic; PD, Paladon.

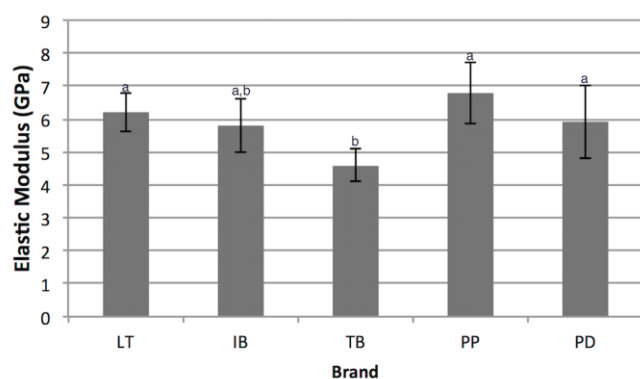


Figure 7. Scanning electron microscope images after surfaces were treated with solvent THF.

Original magnification  $\times 500$ . A, Degos L-Temp. B, IvoBase. C, Zirkonzahn Temp Basic without exposure to THF D, Zirkonzahn Temp Basic after exposure to THF. E, Palapress. F, Paladon.

THF, tetrahydrofuran.

