

## Influence of several materials on the bond strength of glass polyalkenoate to dentin

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الأيون مع العاج لإرجاع الأيون: هو مادة مزججة تتلحم بالعاج بواسطة الإتصاق المزيبي. أثناء العلاج السني باستخدام حشوات الزجاج الأيوني هناك العديد من المواد المساعدة قد تستعمل قبل الزجاج الأيوني، وذلك مثل الحشوات المؤقتة، والأدوية المستخدمة في الأتية الجذرية، و الأسمت المؤقت. هذه المواد المساعدة قد يكون لها تأثير معاكس على التصاق الزجاج الأيوني، بالعاج.

الهدف من هذه الدراسة: هو اختبار تأثير استعمال الأسمت المؤقت الحازي على الأوجنول، والحالي منه، ومادات الكالسيوم، واليوناسيوم الودي (دواء للأقضية الجذرية)، وإسمت الأتية الجذرية الراتنجي، على التصاق حشوات الكينك الفضي (زجاج أيوني) مع العاج.

الطريقة: تم تحضير ستين نموذجاً قسمت إلى ستة مجموعات، واحدة منها تركت بدون معالجة العاج السن كعينة تحكم، وكل واحدة من الخمسة مجموعات الباقية عولج عجاج السن بإحدى المواد المساعدة السابقة الذكر، ثم جففت لمدة أسبوع في 61.00% رطوبة و 37 درجة مئوية، ثم إزالة المواد المعالجة بسعد ذلك، ونظف العاج بمحجون الحفان اللاني، ثم حدى السن وغسل جيداً وحفظ بالماء. تم تشكيل اسطوانة قصيرة من الكينك الفضي على سطح العاج بين سطحين متزلقين صمما خصيصاً لهذا الاختبار، ثم شد السطحين المتزلقين لفصلهما بواسطة آلة اختبار، وسجلت قوة الفصل ولم تحليل نتائج الاختبار. أظهرت النتائج أن قوة الفصل لعينة التحكم كانت 2.20 ميجاباسكال، في حين كانت 2.03 و 2.09 و 2.16 و 2.00 ميجاباسكال، بالنسبة لأربعة مجموعات عولجت بأربعة مواد مساعدة مختلفة، ولم يوجد اختلاف ذو أهمية من الناحية الإحصائية بينها وبين عينة التحكم، والمادة الو حسيده التي نسبت تحسناً ملحوظاً في قوة الالتصاق بين الزجاج الفضي والعاج كانت مادة إسمت الأتية الجذرية (AH-26) وأظهرت قيمة 3.50 ميجاباسكال.

Glass ionomer (GI) is a restorative that bonds to dentin by molecular adhesion. During a dental treatment, several adjunctive materials such as temporary fillings, cements and intracanal medicaments may be used prior to the placement of GI which might adversely influence its adhesion to dentin. The purpose of this study was to determine the influence of using eugenol containing and eugenol free zinc oxide temporary cements, calcium hydroxide and iodine potassium iodide intracanal medicaments, and Epoxy resin sealers on the adhesion of Ketac-Silver Aplicap, to dentin. Sixty dentin specimens were prepared and divided to six groups. Each of the five groups was treated with one of the adjunctive materials for one week. The adjunctive material was removed and dentin cleaned with pumice-water slurry, etched, rinsed thoroughly and dried with air. Ketac-silver short cylinders were formed on the dentin between two sliding surface; a specially designed testing apparatus. The two sliding surfaces were pulled apart using a testing machine and the debonding force was recorded. One-way analysis of variance and Tukey's multiple range test were used to analyze the data. While the debonding force was 2.2MPa for the control, it was 2.03,2.09,2.00, and 2.16 MPa for specimens subjected to four of the adjunctive materials and there were no statistically significant differences ( $P < 0.05$ ). The only adjunctive material that caused significant difference in the bond strength between Ketac-Silver and dentin (an improvement) was epoxy resin sealer (AH-26) yielding a debonding value of 3.50 Mpa.

### Introduction

Glass polyalkenoate (ionomer) cements, introduced by Wilson and Kent,<sup>1</sup> are water based. They are derivatives of silicate and zinc polycarboxylate cements consisting of ion-leachable glass powder and polyalkeonic acid. The result of the interaction between the two constituents is a hard cement composed of glass particles surrounded and supported by a matrix arising from the dissolution of the surface of the

glass particles in the acid.

Chemical bonding between glass polyalkenoate cements and dentin or enamel has been well documented.<sup>2</sup> The cements are also reported to adhere to a number of other substrates such as stainless steel, tin, tin-oxide plated platinum or gold but not to porcelain, pure platinum or pure gold.<sup>3</sup> Nonetheless, a glass polyalkeonate luting cement enhanced the retention of gold alloy castings when compared with traditional luting cements.<sup>4</sup>

In addition to chemical bonding of glass polyalkenoate cements to cavity walls, the continuity of such bonding is assured. The bond between glass polyalkeonate and tooth material is

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a dynamic one with adhesive bonds being continually broken and reformed as chemical (ion-exchange and water transfer) and biological changes take place.<sup>5</sup> Other advantages of glass polyalkenoate materials is the release of fluoride<sup>6</sup> which leaches into the tooth structure interfacing the restoration,<sup>7</sup> inhibiting recurrent caries,<sup>8</sup> and aiding remineralization.<sup>9</sup> The materials also have low thermal conductivity and a coefficient of thermal expansion similar to that of tooth structure.<sup>10</sup>

Glass polyalkenoates, because of their adhesive characteristics and other advantages have gained wide use in dental practice. The materials are primarily used as a permanent cement, as a base band as a class V filling material. They are also used to build cores, to seal occlusal fissures and as endodontic sealers.<sup>10</sup> Any of these dental procedures usually requires the use of other materials prior to using the glass polyalkenoate. The influence of these other materials on the adhesive potential of the glass polyalkenoate to dentin needs to be studied.

The bonding potential of dental adhesive materials to enamel and dentin is quite sensitive to the prior management of the tooth surface. The effect of several materials on the bond strength of resin composite to dentin had been well studied as reported in the literature. Summitt et al<sup>11</sup> studied the effect of air/water rinse versus water only and rinsing times on resin to etched-enamel bond strength. Gwinnett<sup>12</sup> studied strength of bonding of composite resin to dentin after air drying and re-wetting. Macchi et al<sup>13</sup> studied the influence of several endodontic materials on the bonding of composite resin to dentin. The influence of saliva, plasma, zinc oxide-eugenol cement, non-eugenol zinc oxide cement and handpiece lubricant on bonding composite resin to dentin was studied by Powers et al.<sup>14</sup> Tam and Pilliar<sup>15</sup> studied the influence of conditioning acid when air dried on the bond strength of dentin-composite resin adhesive interface. The effect of etchant type, surface moisture, and resin composite type on the shear bond strength of three dentin adhesives was studied by Perdigão et al.<sup>16</sup> The influence of eugenol in reducing the bond strength between composite resin and dentin is reported.<sup>13,17,18</sup>

In as much as enamel and dentin surface treatment influences their adhesion to composite resins, it is likely to influence their adhesion to glass-ionomer cements. Studies to substantiate or vitiate this conclusion are sparse. Capurro et al<sup>19</sup> found that IRM, Grossman Cement, Dycal and Cavit

had no effect on the bonding of glass ionomer cement to dentin.

The purpose of this study was to determine the influence of five materials including temporary cements, intracanal medicaments and sealer cement materials on the bond strength of Ketac-Silver (a glass-ionomer restorative) to dentin.

## Materials and Methods

A glass polyalkenoate material with proven chemical adhesion to dentin, used for core buildup, was selected for this study (Ketac-Silver Aplicap ESPE Premiere, Norristown, PA, USA). Five materials were used as potential contaminants that may interfere with the glass-ionomer's adhesion to dentin. These were: eugenol-free zinc oxide temporary cement (Temp-Bond NE, Kerr, Romulus, MI, USA), eugenol containing temporary cement (Temp-Bond, Kerr); a calcium hydroxide [Ca(OH)<sub>2</sub>] paste packed in anesthesia cartridges (Calasept, Scania Dental AB, Knivsta, Sweden); biphenol A- diglycidylether based root canal sealing and filling material, an epoxy resin (AH-26, Dentsply Detrey, Konstanz, Germany); and a laboratory prepared 2% Iodine Potassium Iodide (IPI) solution, commonly used as an intracanal medicament.<sup>20</sup>

### Fabrication of test specimens

A previously used apparatus to prepare testing specimens was used.<sup>18</sup> Each specimen consisted of parts A and B. The configuration of each specimen is illustrated in Fig. 1. The A portion of the specimen was fabricated by embedding a freshly extracted human permanent molar tooth into acrylic resin (Orthoresin; De Trey Dentsply

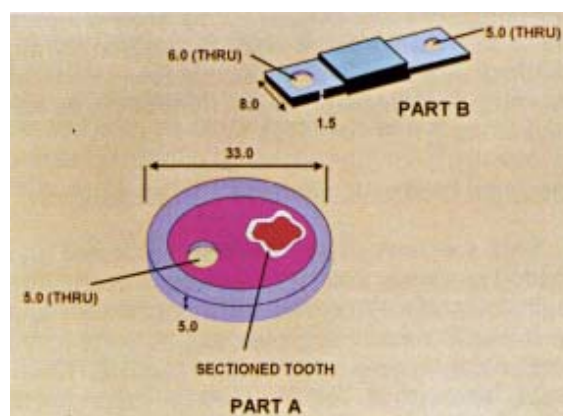


Fig. 1. Diagram of specimen configuration (all dimensions are in millimeters).

S.A., Bois Colombes, France), which was then sectioned to a disc 33 mm in diameter and 5 mm thick. Both sides of each disc were flattened. The solid caries-free dentinal surface of the embedded tooth was exposed in one side of the disc. Specimen sectioning and flattening were accomplished by using a fine diamond disc (Brasseler GmbH, Lemgo, Germany). An opening, 5 mm in diameter, was placed in the acrylic to facilitate mounting on the testing machine (Fig. 2).

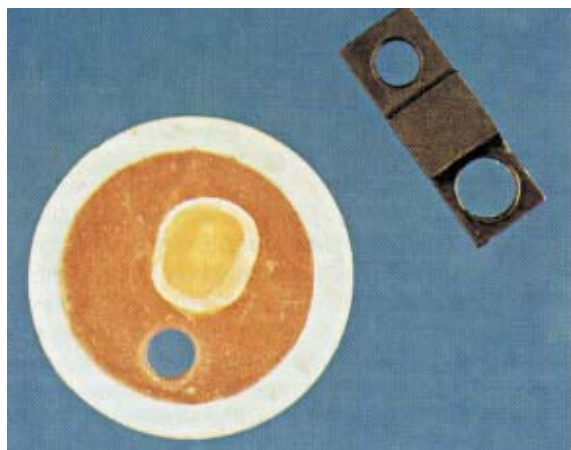


Fig. 2. Part A and B of the test specimen.

The second component (part B) of each specimen was fabricated based on the testing apparatus designed by Hammad et al.<sup>21,22</sup> Only the B portion of Hammad's testing device was used in this study. It was fabricated by machining sheets of solid Delrin (Almac Plastics, Minneapolis, MN) acrylic resin to conform precisely to the diagram depicted in Fig. 1. Part B was cast in a base-metal alloy (Wirron 99, Bego, Germany) following the manufacturer's instructions. Part B served as a matrix to hold the Ketac-Silver in position during bonding procedures and provided a means of attaching the specimen to the debonding device (Figs. 2 and 3).

#### Specimen preparation and material application

Sixty specimens (part A) were fabricated and divided randomly into six groups of 10 each. The teeth in group 1 received no contaminant and served as a control. In groups 2, 3, 4, 5 and 6, the dentin was coated with Temp-Bond NE, Temp-Bond, IPI, AH-26, or Ca (OH)<sub>2</sub>, respectively. Manufacturer's instructions were followed carefully. After contaminant application and setting, the specimens were stored in 100%

humidity at 37°C for 1 week. Following storage, contaminants were removed from the dentin, except for the IPI group, using a Hollenback carving instrument. The exposed dentin surfaces of all six groups were cleaned with pumice-water slurry and a prophylaxis rubber cup mounted on a slow-speed handpiece. Specimens were then rinsed thoroughly with distilled water and dried with air. ESPE Ketac conditioner was then applied over the dentin surface for 10 seconds then thoroughly rinsed with distilled water for 30 seconds and dried with air. The matrix of each specimen (part B) was aligned over the dentin surface of its corresponding part A and then filled with Ketac-Silver Aplicap core material using the syringe procedure (Fig. 3). The core material was used according to the manufacturer's recommendation and applied to all groups. Specimens were left undisturbed until complete setting was attained. The specimens were allowed to set according to the manufacturer's instructions. All specimens were fabricated by one investigator.

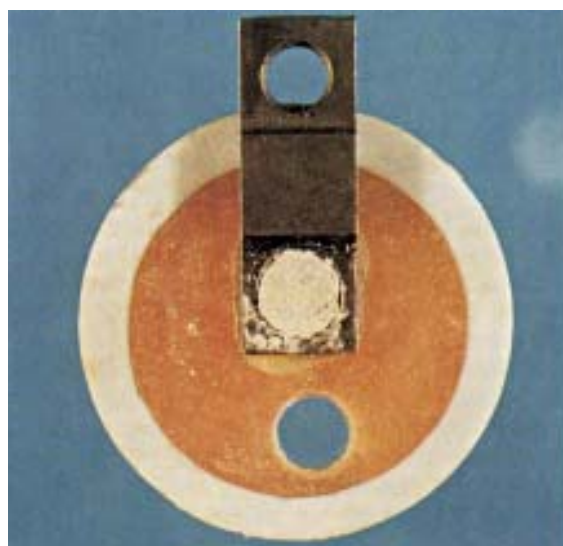


Fig. 3. The Ketac-silver material was applied through the hole in part B over the dentinal surface of part A.

#### Method of testing

The longitudinal ends of parts A and B of each specimen were mounted by means of hooks attached to the upper and lower jaws of a universal testing of an Accuforce Elite test machine (Model E500, Ametek, Largo, FL, USA). Force was applied at a crosshead speed of 0.05 in/min until failure occurred (Fig. 4). The presence of two flexible

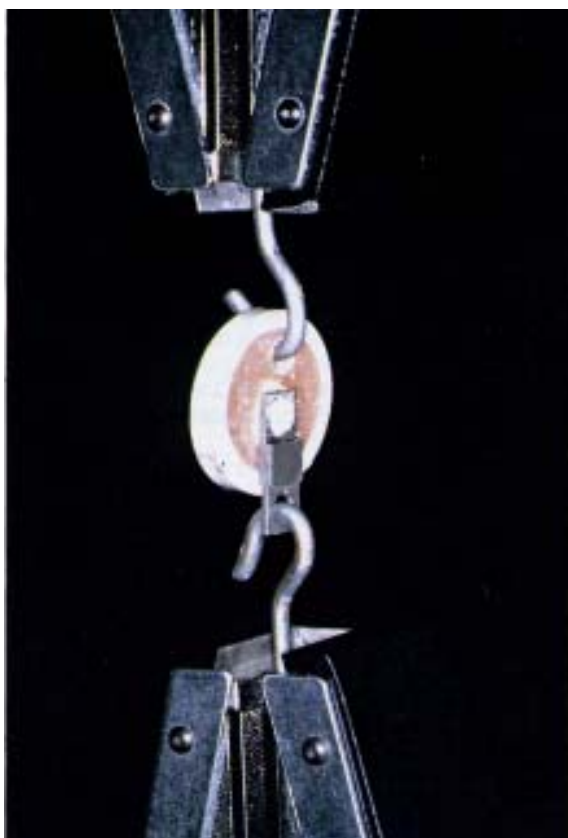


Fig. 4. The specimen in the testing apparatus.

joints, coupled with the slow loading, permitted the specimen to align itself to a uniaxial direction during testing procedures. However, because the bonding interface was not aligned at the middle of the thickness of each specimen, forces were not primarily directed at the dentin-core material interface, and thus forces were directed diagonally. This developed a combination of shear and tensile stresses to simulate complex clinical stress situations. All testings were performed by one investigator. The data obtained were analyzed statistically using a one-way ANOVA and Tukey's post hoc test.

### Results

The bond strength results for the five experimental groups and the control are shown in Table 1. The table suggests that except for AH26, the use of the four other materials caused little change in the adhesion of Ketac-Silver to dentin.

Before selecting the proper statistical tests, it was necessary to test the homogeneity of the variances obtained with the various groups.

Table 1. Descriptive statistics for bond strength.

Contaminant	N	Mean MPa	Std. deviation	Std. error	Min	Max	95% confidence interval for mean	
							Lower bound	Upper bound
Control	10	2.2070	0.9219	0.2915	1.11	3.35	1.5475	2.8665
Temp-Bond	10	2.0300	0.4407	0.1394	1.34	2.68	1.7148	2.3452
Temp-								
Bond-NE	10	2.0950	0.4741	0.1499	1.56	2.90	1.7559	2.4341
IPI	10	2.0080	0.8570	0.2710	1.11	3.35	1.3949	2.6211
AH-26*	10	3.5010	0.7424	0.2348	2.23	4.68	2.9699	4.0321
Ca(OH)2	10	2.1630	0.6408	0.2026	1.11	3.12	1.7046	2.6214

\* Shows significant differences at  $P \leq 0.05$

Homogeneity of variance test was significant; however, not significant enough to preclude using a parametric one way analysis of variance (ANOVA). The results of parametric one-way ANOVA is depicted in Table 2. The test showed highly significant differences between groups at ( $P < 0.001$ )

Table 2. Results of parametric ANOVA in comparing groups.

	Sum of Squares	Df	Mean Squares	F	Sig.
Between Groups	16.631	5	3.326	6.730	.0001
Within Groups	26.686	54	0.494		
Total	43.317	59			

Due to the mild significance of the variance homogeneity test, it was felt necessary to also use non-parametric ANOVA. The results of non-parametric ANOVA, also showed highly significant differences between the groups at ( $P < 0.001$ ). Tukey's post hoc test showed that the bond strength of Ketac-silver to dentin treated by AH-26 was significantly higher than that for dentin treated with all other materials and than that for untreated dentin (the control) as illustrated in Table 1.

### Discussion

In the present study, the contaminants applied to dentin were kept on it for one week and then removed by carving followed by pumice-water slurry and thoroughly rinsed with water. This procedure did not achieve the decontaminated dentin surface which could have facilitated adherence of resin composite to dentin.<sup>13,14,18</sup>

Accordingly, one may safely conclude that traces of these contaminants did remain on the dentin surfaces. As Table 1 shows, four of the contaminants had no effect on the strength of the bond between the glass-ionomer and dentin suggesting that the glass-ionomer liquid, a polyacrylic acid, dissolved away the traces of the contaminants thus eliminating their effect in reducing the bond strength. Similar finding was reported by Capurro et al.<sup>19</sup> Further, when three polyacrylic acid etchants were used, the bond strength between glass-ionomer cement and dentin was increased<sup>23</sup> emphasizing the etching and thorough cleaning ability of the polyacrylic acid.

On the other hand, the use of AH-26, an epoxy resin, as a contaminant caused an unexpected increase in the bond strength between the glass-ionomer cement and the dentin (Table 1). This increase suggests that the traces of AH-26 remaining on the specimens of this study after cleaning were not dissolved by the polyacrylic acid of the glass-ionomer cement. This explanation is quite reasonable when one considers that AH-26 was found to be extremely difficult to dissolve except with chloroform applied for more than 30 minutes.<sup>24</sup>

The presence of these traces of AH-26 increased the bond strength between the dentin and the glass ionomer cement. This increase suggests that AH-26 attaches to dentin with more strength than does the glass-ionomer. Other studies showed that canal sealed with AH-26 leaks less than those sealed with Ketac-endo (a glass-ionomer) suggesting that AH-26 attaches to the dentin even stronger than to the glass ionomer.<sup>25</sup>

Our data suggests that remnants of most contaminants used on dentin before using a glass-ionomer cement are not likely to influence its bonding to dentin, since the liquid of the cement dissolves them away. However, remnants of AH-26 are not dissolved by the liquid of the glass-ionomer and their presence enhance the glass-ionomer cement bond to dentin.

### Conclusions

1. Remnants of zinc oxide, eugenol, iodine potassium iodide and calcium hydroxide appear not to influence the bond strength of GI to dentin when polyacrylic acid is used as a conditioner.
2. Remnants of AH-26 enhance the GI bond to dentin and do not dissolve by polyacrylic acid.

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### References

1. Wilson AD and Kent BE. The glass ionomer cement: A new translucent dental filling material. *J Appl Chem Biotechnol*1971; 21:313-318.
2. Wilson AD, Crisp S and Lewis BG. Experimental luting agents based on the glass-ionomer cements. *Brit Dent J* 1977; 142:117-122.
3. Holtz P, McLean JW, Sced 1 and Wilson AD. The bonding of glass-ionomer cements to metal and tooth substrates. *Brit Dentj* 1977; 142:41-47.
4. McComb D. Retention of castings with glass-ionomer cement. *J Prosthet Dent* 1982; 48:285-288.
5. Wilson AD, Prosser R and Powis DM. Mechanism of adhesion of polyelectrolyte cements of hydroxyapatite. *J Dent Res* 1983; 62:590-592.
6. Swartz ML, Phillips RW and Clark HE. Long-term fluoride release from glass-ionomer cements. *J Dent Res* 1984; 63:158-160.
7. Retief DH, Bradley EL and Dentone JC et al. Enamel and cementum fluoride uptake from glass-ionomer cement. *Caries Res* 1984; 16:250-257.
8. TyasMJ. Cariostatic effect of glass-ionomer cement. A five year clinical study. *Aust Dent J* 1991; 36: 236-239.
9. Berg JH, Donly KJ and Posnick WR. Glass ionomer-silver restoration: A demineralization-reminerization concept. *Quint Int* 1988; 19: 639-641.
10. Craig RG. *Cements: Restorative Dental Materials*. 10th ed. St. Louis, Missouri: Mosby, 1997:172-208.
11. Summitt JB, Chan DC, Burgess JO et al. Effect of air/water rinse versus water only and of five rinse times on resin-to-etched-enamel shear bond strength. *Oper Dent* 1992; 17:142-151.
12. Gwinnett AJ. Dentin bond strength after air drying and rewetting. *Am J Dent* 1994; 7:144-148.
13. Macchi RL, Capurro MA and Herrera CL et al. Influence of endodontic materials on the bonding of composite resin to dentin. *Endod Dent Traumatol* 1992;8:26-29.
14. Powers JM, Finger WJ and Xie J. Bonding of composite resin to contaminated human enamel and dentin. *J Prosthodont* 1995; 4: 28-32.
15. Tarn LE and Pilliar RM. Effect of dentin surface treatments on the fracture toughness and tensile bond strength of a dentin-composite adhesive interface. *J Dent Res* 1994;73:1530-1538.
16. Perdigao J, Swift EJ and Cloe BC. Effects of etchants, surface moisture, and resin-composite on dentin bond strengths. *Am J Dent* 1993; 6:61-64.

17. Tjan AHL and Nemetz H. Effect of eugenol-containing endodontic sealer on retention of pre-fabricated posts luted with an adhesive composite resin cement. *Quint Int* 1992; 23: 839-844.
18. Al Wazzan KA, Al Harbi AA and Hammad IA. The effect of eugenol-containing temporary cement on the bond strength of two resin composite core materials to dentin. *J Prost hodont* 1997; 6:37-42.
19. Capurro MA, Herrera CL and Machhi RL. Influence of endodontic materials on the bonding of glass ionomer cement to dentin. *Endod Dent Traumatol* 1993; 2: 75-76.20.
20. Ingle JI and Bakland LK. Intercanal Medicaments. In *Endodontics*, 3rd ed. Philadelphia, PA: Williams and Wilkins, 1985:627-640.
21. Hammad IA and Stein RS. A qualitative study for the bond and color of ceramometals. *J Prosthet Dent* 1990;58:431-437.
22. Hammad IA and Talic YF. Design of bond strength tests for metal-ceramic complexes: Review of the literature. *J Prosthet Dent* 1996; 75:602-608.
23. Joynt RB, Davis EL and Wieczowski G et al. Effect of dentinal pretreatment on bond strength between glass-ionomer cement and dentin. *Oper Dent* 1990; 15:173-177.
24. Hansen MG. Relative efficiency of solvents used in endodontics. *J Endod* 1998; 24:38-40.
25. De Gee AJ, Wu MK and WesseLink PR. Sealing properties of Ketac-Endo glass-ionomer cement and AH-26 root canal sealer. *Int Endod J*1994; 27:239-244.