TABLE 2

Metal-Metal Tensile Bond Strength (kgf/cm^2) in 2000-Time Thermal Cycle Tests: BPO-DEPT System

	EXAMPLE 15 (2-MEDT)	COMPARATIVE EXAMPLE 3 (4-META)	COMPARATIVE EXAMPLE 4 (VBATDT)	COMPARATIVE EXAMPLE 5 (MPMA)	COMPARATIVE EXAMPLE 6 (BMEDS)	COMPARATIVE EXAMPLE 7 (BMPDS)	COMPARATIVE EXAMPLE 8 (BMDDS)
Gold	196	0	69	16	0	64	66
Silver	396	0	0	0	0	0	0
Platinum	338	0	149	5	222	354	351
Palladium	193	27	100	70	118	215	217
Gold alloy	412	0	263	38	236	288	368
Silver alloy	396	105	257	218	106	140	289
Gold/silver/ palladium alloy	451	158	281	115	246	244	332

Notes:
Adhesive resins used: MMA—PMMA/BPO-DEPT resin system.
Precious metals: all in pure state; alloys used: alloys for dental
Gold alloy: "Casting Gold M. C." (type IV) (made by GC K. K.)
Silver alloy: "Sunsilver C. B." (made by Sankin K. K.)
Gold/silver/palladium alloy: "Castwell M. C." (made by GC K. K.)

As is apparent from Table 2 results, 2-MEDT, a compound of the invention, when used in conjunction with general-purpose catalyst BPO-DEPT, exhibits exceedingly higher bond durability and greater storage stability as compared with conventional compounds, such as 4-META, VBATDT, MPMA, BMEDS and BMPDS.

In contrast, it has now been found that the performance of MPMA is very low in bond durability as well as in storage stability. It has also been made clear that VBATDT, BMEDS and BMPDS have a deficiency in respect of bond durability such that its adhesion performance relative to gold and silver in particular is noticeably low.

It may be noted that 10-methacryloyloxydecyl dihydrogen thiophosphate, a thiophosphoric compound, was omitted from evaluation for bond performance, because the compound has unacceptably low shelf stability and tends to generate extremely bad smell, which is a problem from the standpoint of practical use irrespective of whether or not the compound deserves such evaluation.

It has now been found that in contrast to such straight-chain disulfide compound, compounds of the invention which incorporate a cyclic disulfide compound exhibit stable and much higher bond durability in relation to gold, silver and alloys thereof.

With reference to the low bond performance of bis (methacryloylalkyl) disulfide used in combination with BPO-DEPT catalysts as in COMPARATIVE EXAMPLES 6–8, it may be noted that reports to that effect were made by the present inventors in "Dental Material & Equipment", vol. 11, "Lecture Issue 20", pp 234–235 (1992), and also in "Dental Material & Equipment", vol. 12, "Lecture Issue 21", pp 164–165 (1993).

EXAMPLE 16–18 AND COMPARATIVE EXAMPLE 9

Tests of enamel-gold alloy bond strength under shear were carried out by using primer compounds of EXAMPLES 8–10 and adhesive resin cements of BPO-DEPT-barbituric acid derivative system.

An adhesive resin cement powder was prepared by mixing silane-treated silica (75 parts by weight), silane-treated barium sulfate (25 parts by weight), N, N-di(2-hydroxyethyl)-p-toluidine (0.1 part by weight) and 1-benzyl-5-phenyl barbituric acid (1.0 part by weight).

A liquid adhesive resin cement was prepared by mixing a 1:2 (mol) reaction product of 2, 2, 4-trimethylhexamethylene diisocyanate and 2-hydroxyethyl methacrylate (65 parts by weight), triethyleneglycol dimethacrylate (20 parts by weight), ethyleneglycol dimethacrylate (10 parts by weight), 4-acryloxyethyl trimellitic acid (5.0 parts by weight), benzoyl peroxide (0.5 part by weight), and butylated hydroxytoluene (0.05 part by weight).

In conduct of tests, a bovine tooth was embedded in an epoxy resin, and then enamel surface of the tooth was exposed, which was then abrasively polished with waterproof sand paper No. #600 under water pouring. After drying, the enamel surface was subjected to etching with phosphoric acid for 30 seconds, followed by water washing and drying.

A type IV gold alloy of 4 mm diameter and 2 mm height ("Supergold" (type 4): made by Shofu Inc.) was sand-blasted over its adhesion surface with aluminum oxide particles, and was then ultrasonically cleaned in water, followed by drying.

A metal surface to be treated was coated with the primers prepared in EXAMPLE 1–3, followed by air drying. Thereafter, the foregoing powder and liquid adhesive resin cements were mixed in a powder-liquid ratio of 3.5:1.0 (weight ratio) and kneaded into paste, with which paste the enamel surface and the gold alloy surface were bonded to each other.

Further, 2000-time thermal cycle tests were carried out in the same way as in EXAMPLE 8, and thereafter the compressive shear bond strength was measured at a crosshead speed of 1 mm/min.

For comparison purposes, samples with which no primer treatment was made were tested in the same way as above described. The results of the foregoing tests are shown in Table 3.

TABLE 3

Enamel-Gold Alloy Shear Bond Strength (kgf/cm^2) in 2000-Time Thermal Cycle Tests; Barbituric Acid Type

	Adhesive monomer in primer	Enamel-gold alloy shear bond strength (kgf/cm^2) (2000 cycles)
EXAMPLE 16	2-MEDT	305
EXAMPLE 17	6-MHDT	338