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Self-adhesive Resin Cements: A New Perspective in Luting Technology

Abstract: Many materials are available for the fabrication of indirect restorations such as, metal alloys, resin-based composites and ceramics. Resin cements have long been valued as luting agents for indirect restorations because of their high retentive strength, resistance to wear, and low solubility. However, one of the common discouraging factors regarding their chairside use is the need of multiple-steps (etching, drying, priming and luting) for bonding. Thus the current impetus is towards the use of self-adhesive cements that require no etching, priming or bonding agents to bond to the tooth surface. Their increased popularity can be judged by the commercial availability of more than a dozen self-adhesive resin products/brands, in a short span of time. This article reviews the composition, physical and biological properties, adhesion characteristics and clinical performance of self-adhesive (resin) cements.

Clinical Relevance: Self-adhesive resin cements are dual-cured and adhere to tooth structure without the requirement of a separate etching step and application of an adhesive/bonding agent.

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The reliable adhesion of an indirect restoration is directly related to the luting agent and the associated luting procedure.[1] The intimate contact of a luting agent to the bonding substrate is imperative for an adequate marginal adaptation of the cement on the bonding interfaces, to improve retention and avoid the premature failure of indirect restorations.[2] Patient preference for all-ceramic indirect restorations, for aesthetic reasons, has increased the use of resin cements in clinical practice. With the advent of resin cements, it is now possible to bond to both the tooth surface and the fitting surface of a restoration. Conventional materials, like zinc phosphate and zinc polycarboxylate, are called 'passive' materials as they achieve the retention of the restoration by mechanical interlocking between rough surfaces and the cement. Comparatively, composite resin cements are considered as 'active' materials because of an adhesive interaction with the dentine via micromechanical bonding by the formation of a hybrid layer and bond to dental materials. Currently, resin cements may be classified as: Total-etch, Self-etching, and Self-adhesive cements (Table 1).[3]

Total-etch cements use a phosphoric acid etchant and adhesive to bond the cement to the tooth. These cements have the highest bond strength (cement-to-tooth), with increased retention and superior mechanical properties.[3] However, they require a multi-step application procedure, which may be technique-sensitive, may have higher incidence of post-operative sensitivity, and the clinical outcome is dependent on variables like operator skill, restoration design, material characteristics and the intra-oral conditions.[4]

Self-etching cements, such as *Multilink Automix* (Ivoclar Vivadent), prepare the tooth surface using a self-etch primer followed by the application of the mixed cement over the primer to form a bond between the tooth and the restoration.

The latest subgroups added in resin cements, in clinical practice, are the Self-Adhesive (Resin) Cements (SACs). These cements have evolved as a result of the desire of clinicians/operators to simplify the luting procedures for resin cements and, more importantly, to shorten their '*window of contamination*'.[3] Hence, resin cements based upon phosphoric-acid-modified acrylates, the first so-called self-adhesive (resin) cements (SACs) material (*RelyX Unicem*, 3M ESPE), were developed. SACs are dual-cured resin cements which can bond to an untreated tooth surface that is neither micro-abraded nor pre-treated with an etchant, primer or bonding agent. The cement can be directly applied to the restoration fitting surface that can then be luted to a non-treated tooth surface. They have similar bond strength to self-etching systems, with cementation being accomplished in a single step, thus overcoming the limitations of the conventional total-etch resin cements.[3]

The article discusses the composition, properties, bonding mechanism,

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LUTING AGENT	Total-etch cement	Self-etching Cement	Self-adhesive Cement
Bonding Steps	Multi-step	Two-step	Single-step
Bonding Technique Steps	■ Phosphoric acid etchant ■ Adhesive agent application	■ Self-etching primer ■ Application of mixed cement over primer	■ Dual-cured cements ■ No etching, priming and application of bonding agents
Post-operative Sensitivity	Present	No	No
Bond Strength	Greatest	Intermediate	Equivalent to self-etch
Application	Cementation of high strength ceramic	Cementation of moderate strength pressed all-ceramic	Cementation of high strength ceramic
Commercial Products	■ *RelyX ARC* ■ *Variolink II* ■ *Choice 2 Calibra*	■ *Panavia* ■ *Multilink Automix*	■ *RelyX* ■ *Unicem* ■ *BisCem* ■ *Smart Cem 2* ■ *Maxcem Elite* ■ *Speed-CEM* ■ *RelyX Unicem 2 Automix*

Table 1. Classification of resin cements.

adhesion characteristics and properties of the currently available self-adhesive (resin) cements.

Composition

Self-adhesive resin cements are based on filled polymers designed to adhere to tooth structure without the requirement of a separate adhesive or etchant. A variety of self-adhesive products are available commercially by different manufacturers, having different application protocol, working and setting time and chemical compositions (Table 2).[5,6] Though all are based on an auto-adhesive technology,[5,6] there are variations among the materials' mechanical properties and bonding performances. *RelyX Unicem* (3M ESPE) was the first introduced and most extensively investigated SAC. The basic components of a SAC consist of an organic matrix with multifunctional phosphoric acid methacrylates or acidic monomers, along with traditional fillers. Apart from the monomers and fillers, redox initiator systems, photo-initiator components (camphorquinone-based) and pigments are also added.

The organic matrix is a blend of polymerizable methacrylates, dimethacrylates and polymethacrylates, along with acid functionalized monomers, predominantly methacrylate monomers with either carboxylic acid groups, as with 4-methacryloxyethyl trimellitic anhydride (4-META) and pyromellitic glycerol dimethacrylate (PMGDM), or phosphoric acid groups, as with 2-methacryloxyethyl phenyl hydrogen phosphate (Phenyl-P), 10-methacryloxydecyl dihydrogen phosphate (MDP).[7] These monomers lead to demineralization of enamel and dentine and form a stable salt, mainly involving calcium. The concentration of the acidic monomers must be low enough to avoid excessive hydrophilicity but high enough to achieve bonding to dentine and enamel. Thus the matrix provides the mechanism for the tooth (hydroxyapatite)-to-cement bond. To enhance the hydrolytic stabilities of acidic monomers, various new monomer formulations have been developed for application in self-etch resins like, neutral monomers based on bisacrylamide structures {bisacrylamide-N,N-diethyl-1,3-propane, 10-(N-methylacrylamide) decylphosphonic acid and 3-(N-propylacrylamide) propylidenebisphosphonic acid}, monomers with hydrolytically stable ether rather than an ester linkage {2,4,6-trimethylphenyl-2-[4-(dihydroxyphosphoryl)-2-oxabutyl]-acrylate} and monomers with allyl ether reactive groups {allyloxyethylphosphate; 2-(allyloxymethyl)-2-ethylpropane-1,3-diyl bis(dihydrogen phosphate)}.[7]

The filler content ranges from 60–75 wt% and is composed of barium fluoroaluminoborosilicate glass, strontium calcium aluminosilicate glass, quartz, colloidal silica, ytterbium fluoride and other glass fillers. The acid-soluble glass fillers provide for the neutralization of resin acidity and release of sodium, calcium, silicate and fluoride ions that either take part in the setting reaction or are released locally. The fillers determine the physical behaviour and mechanical properties of cement. The filler content of SACs is less compared with that of compomers. Same fluoride release is seen in some SACs, containing fluoride ion-releasing fillers or by the inclusion of sodium fluoride or related salts in the cements, but is likely to be clinically insignificant.

Owing to the low pH of the acidic component of SACs, an acid-tolerant oxidant, like cumene hydroperoxide, is used in the acidic part of the formulation and various thioureas, like benzoyl thiourea, are added as reducing agents in the non-acidic part. To induce visible light-curing, camphorquinone and/or diphenyl-(2,4,6-trimethylbenzoyl) phosphine oxide (TPO) is added to the acidic components, while a tertiary amine, such as ethyl N,N-dimethylaminobenzoate or N,N-dimethylaminobenzonitrile is incorporated in the non-acidic components. The acid resistance of initiators can be improved by the addition of sodium arylsulphates and aryl borate salts.

Product Name	Composition	Manufacturer
RelyX Unicem	Base paste: Methacrylate monomer containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator, stabilizers Catalyst paste: Methacrylate monomers, alkaline fillers, silanted fillers, initiator, stabilizers, pigments	3M ESPE, Seefeld, Germany
Clearfil SA Cement	Paste A: Bis-GMA, TEGDMA, MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated colloidal silica, silanated barium glass filler, dl-camphorquinone, benzoyl peroxide, intiator Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, silanated barium glass, silanated colloidal silica, surface treated sodium fluoride, accelerators and pigments	Kuraray Europe GmbH, Frankfurt, Germany
G-Cem	Powder: Fluoroaluminosilicate glass, initiator, pigment Liquid: 4-MET, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer	GC EUROPE, Leuven, Belgium
Smart Cem 2	Base paste: Urethane dimethacrylate, di- and tri-methacrylate resins, strontium fluoride. Catalyst paste: Barium boron fluoroaluminosilicate glass; urethane dimethacrylate resin, urethane modified bis-GMA, dipenta-erythritol penta-acrylate phosphate, hydrophobic amorphous silicon dioxide	Dentsply, Konstanz, Germany
Speed Cem	Dimethacrylates, ytterbium trifluoride copolymers, glass filler, silicon dioxide, adhesive monomer, initiators, stabilizers and pigments	Ivoclar, Schaan, Liechtenstein, Germany

Table 2. Product names, composition and manufacturers' details for commercially available self-adhesive cements.

Dispensing/delivery systems

They are usually available as two-part systems: one component (usually the liquid component) contains the conventional mono-, di- and/or multi-methacrylate monomers like, Bis-GMA, UDMA, HEMA, TEGDMA, along with photo-initiator systems and the second component (usually the powder component) contains the filler combinations. This segregation is necessary to:

- Avoid a premature acid–base reaction between the acidic monomer and the ion-leachable glass;
- Allow separation of the redox initiator components required for free radical polymerization (chemical cure);
- Avoid de-activation of the photoinitiator system (tertiary amine or other electron-donating compound) by the acidic monomers and spontaneous polymerization process that may limit the shelf life of cement;

They are dispensed as three basic systems:[7]

- Capsule delivery systems;
- Hand-mixed paste–paste systems;
- Auto-mixing syringes.

The capsule delivery systems have been shown to produce a stronger bond with better retention than other mixing systems owing to the well controlled proportions of the mixed cement.[3,8] However, the newly introduced *Unicem2 Automix* has shown a significant improvement in dentine and enamel bond strengths.[3]

Setting/curing mechanism

The setting reaction is initiated following the radical polymerization that can be activated either chemically (self-cure) or by light (light exposure).[9] However, better monomer conversion and superior mechanical properties have been reported with dual-cured materials (both chemically and light activation).[10] Thus, most of the available SACs cure via dual cure, chemical and photochemical, based on the addition of photo-initiators along with the redox initiators. The acid–base reaction between the acidic functionality on the monomers and the acid-soluble glass/tooth surface is initiated upon mixing of the two components. However, upon mixing, due to the presence of inhibitor, chemical curing (free radical redox polymerization) commences gradually, with the photo-initiation providing a rapid boost to the setting process, resulting in improved final conversion and adhesive strength, as compared to self-cure alone.[7,11] The working time is approximately two minutes, with setting times ranging from 3–7 minutes. Following placement of restoration and removal of excess cement, an augmented photo-curing can be done with a 20- to 40-s exposure to visible light from a conventional dental curing unit.

Studies have also reported a significant reduction in degree of conversion, strength, micro-hardness and wear resistance of dual-cured resin cements when cured using chemical cure only.[12,13,14]

Bonding characteristics

Bonding mechanism

Self-adhesive resin cements are designed specifically to interact with the dentine substrate with minimal additional surface preparation. The bonding mechanism of SACs is based on chemical interaction and micro-mechanical retention with the adhesive substrate,[15,16,17] along with the simultaneous demineralization/infiltration of the smear layer and the underlying tooth structure. The multifunctional monomers chemically react with the hydroxyapatite. These phosphorylated methacrylates create a low pH on contacting water or moisture from the tooth. Like self-etch adhesives, the pH of a freshly mixed cement may range from 1.5–3. This low pH etches the tooth structure (enamel and dentine) and the cement penetrates the etched tooth surface, creating a micromechanical bond with the tooth when the cement polymerizes. In the initial acid-base reaction, as the cement sets, water is formed which produces the cement's initial hydrophilicity that improves the marginal adaptation and the pH increases to neutrality. During the secondary setting reaction, the produced water is consumed by the forming cement matrix, developing a hydrophobic matrix, which has low solubility, low expansion and long-term stability.[3] Usually, the pH reaches neutrality during the setting reaction. Some materials include calcium hydroxide in the non-acidic part (*RelyX Unicem*, 3M, ESPE, St Paul, MN, USA) for a more rapid neutralization process. Some of these cements also form a weak chemical bond with calcium in the tooth in addition to the micromechanical bond.

Bonding to enamel and dentine

The low pH created during the chemical reaction etches dentine more easily than enamel. Thus SACs have better bonding to dentine than to enamel. The bond-strength with enamel, may be improved by pre-etching of enamel with phosphoric acid, so called *'selective enamel etching'*, and formation of enhanced microscopic irregularities produced by the stronger acid. However, a reduction in bond strength to dentine is observed following phosphoric acid pre-treatment due to formation of an impenetrable thick collagenous matrix produced following acid demineralization that result in a weak interface.[15]

Bonding to ceramic

Bonding of resin cement to ceramic can be achieved through micromechanical and/or chemical bonding mechanisms. As the phosphate ester group can chemically bond to metal oxides (such as zirconium dioxide), phosphate ester monomer (MDP monomer) based resin cements may bond to zirconia ceramics.[18] MDP-based metal primer coated intaglio surfaces have shown to increase the bond strength between zirconia and the resin cement significantly.[19]

A further enhancement in bond strength can be achieved by conditioning of the ceramic surfaces, which is strongly dependent on the type and the microstructure of the ceramic fitting surface. A durable bond to glass ceramics can be achieved by treatment of ceramic surface with hydrofluoric acid (HF), which attacks the glass phase to produce a retentive surface,[20] followed by application of a silane coupling agent that promotes a chemical bond between the silica phase of the ceramic and the methacrylate groups of the silane coupling agent.[21] Silane coupling agents also increase the substrate surface energy and improve the surface wettability to resin cements.[22] High-content alumina and zirconia based ceramics may be roughened by the use of a tribochemical silica-coating procedure (Rocatec/Cojet Technique, 3M ESPE), as they are resistant to hydrofluoric acid.[23] This method involves cleaning the surfaces with 110 µm high purity alumina at 250 KPa for 14s, followed by tribochemical coating with 30- or 110-micron silica particles coated with alumina. Silane is then applied to this layer that bonds the cement to the silica-coated surface. Strengthening of ceramic, by about 15% has also been observed because of the silica coating. Self-adhesive resin cements yield the highest shear bond strength to tribochemical silica/silane-coated surfaces.[24] The Rocatec system has been shown to improve the bond strength between non-APM (adhesive-phosphate-monomer) containing luting cements and zirconia ceramic significantly.[25] Sandblasting also improves the bonding with high-strength ceramics by increasing the surface area of the intaglio surface of the restoration.

Recently, a new method has been introduced to increase the bond strength of zirconia by selective infiltration etching. In this method, the ceramic surface is coated with glass-containing conditioning agent and is heated to above its glass transition temperature, forming a surface layer of silica glass over the ceramic surface. Upon cooling, the glass is dissolved in acidic bath, creating a porous retentive surface.[26] Aboushelib *et al*[27] reported increased bond strength with selective infiltration etching and novel silane-based zirconia primers. Alternatively, laser[28] like Er, Cr:YSGG and CO_2 has been introduced to enhance micromechanical retention and improve the bond strength of resin to zirconia and alumina ceramics.

Bonding to metals

Currently, only few studies exist on the adhesion strength between SACs and metal alloys used in dentistry.[7,29] The retentive strengths of noble metal alloy crowns cemented with SACs is observed to be equivalent to those luted with polycarboxylate cement and greater than the glass–ionomer, zinc phosphate or zinc oxide eugenol cements.[29] Based on the presence of the more retentive oxide films, SACs bonding to metals vary with varying nobility of metals. The bonding of SACs to base metal alloy is observed to be significantly higher than to the gold alloy.[7]

Properties

Self-adhesive resin cements:

- Are easy to handle;
- Have the property of auto-adhesion,

micro-mechanical retention and are dimensionally stable.[30] These one-step cements are less technique sensitive, with a more limited chance of errors that are commonly observed in multi-step (resin) cements, ie overwetting or overdrying of tooth structure;[31]

- Have better physical properties than conventional cements;[3,7]
- Have good resistance to compression[32] and microhardness;[33]
- Have sufficient film thickness for the cementation of single crowns.[34]
- A wide range of self-adhesive products are available having variable chemical compositions that may differentiate their mechanical properties and bonding performances.[5]

Physical properties

The flexural and compressive strength of SACs, both self-cure and dual-cure, are reported to be equivalent to slightly lower than other resin cements but greater than zinc phosphate, polycarboxylate, GIC, and RM-GIC.[34]

The hardness of SACs is also observed to be lower than that of non-self-adhesive resin cements, both in self- or dual-cure mode.[35]

Abrasion wear of SACs in self-cure mode is equivalent to zinc phosphate and greater than the non self-adhesive resin cement and glass–ionomer. As compared to other resin cements, the SACs have shown better resistance to wear associated with tooth-brushing or food excursions and lesser resistance to wear (abrasion) during the chewing or grinding process.

As compared to dental composite restoratives, SACs are more susceptible to surface erosion because of the more hydrophilic nature of the matrix resin and the less protected surface of the basic fillers due to their interaction with the acidic monomers and absence of a hydrophobic resin coating, such as silane, in the matrix.[6]

Thus, the physical properties of SACs can be considered equivalent to, or slightly lower than, the non self-adhesive resin cements, but greater than the non resin-based cements.[7]

Bond strength

The bond strength of SACs is less than the total-etch and non self-adhesive resin cements, equivalent to self-etch cements and greater than resin-modified glass ionomers.[36,37]

Though studies have reported non-formation of any hybrid layer or real resin tag formations at the dentine-cement interface,[15,16] SACs have been shown to bond to dentine. Limited interaction is also observed between SACs and radicular dentine, leading to poor demineralization and no hybrid layer formation.[38,39] Reduced bond strength to deep or cervical human dentine, as compared to superficial human dentine for *Unicem*, is observed.[16]

As mentioned earlier, SACs bond more efficiently and better to dentine than to enamel.[15,17] Both self-adhesive and self-etch resin cements have similar micro-shear bond strength to human enamel which is greater than glass–ionomer. However, the bond to dentine is generally greater with self-etch resin cements when compared to the SACs. This should be taken into consideration during orthodontic bracket cementation. The failure of *Unicem* to enamel is mostly adhesive, while the failure to dentine is adhesive or a mixed-mode.[7]

The bond strength to enamel can be increased after 'selective enamel etching', ie etching the enamel substrate with 35% phosphoric acid.[15,40,41] On the other hand, conditioning of dentine with 35% phosphoric acid either does not show any improvement in bond strength, or shows a decrease in the same.[15]

As the higher viscosity of the self-etch luting agents, compared with that of separate-etch luting agents, limits their diffusion into dentinal tubules, it is suggested to apply a sustained seating pressure during the setting reaction to allow better resin penetration into dentinal tubules that increases the bond strength values.[42]

Marginal leakage

One of the critical factors determining the clinical success of SACs is their efficiency to adapt well and seal the restoration margins. Marginal adaptation/fit of restorations luted with SACs is observed to be better than the resin-modified glass–ionomer and the compomer cements and equivalent to the conventional total-etch and self-etch resin cements.[43] For all-ceramic restorations *RelyX Unicem* has shown, at the dentine margins, better marginal integrity and equivalent or less microleakage than the other resin cements (like, *Variolink II*, *Panavia F*).[35,43,44] However, the same was not true at the enamel margins. Thus, it is recommended to use a separate phosphoric acid etch for the enamel surfaces.

Durability

Studies examining the effect of fatigue loading on the restorations cemented with SACs observed that these cements can withstand more fatigue cycles than the zinc phosphate but no significant difference is noted between the SACs and the other resin cements.[45] The failures were mainly observed at the crown–cement interface for the SACs, as opposed to resin cements where they were observed at the tooth–cement interface. In contrast, in another study, evaluating the fracture strength of Procera alumina crowns luted with an SAC (*Unicem*) showed that the fracture strength of SACs was significantly lower than the resin cements and equivalent to the zinc phosphate cement.[46]

Thus, further studies are required to assess the durability of cement adhesion for the SACs.

Post-operative sensitivity

Unlike the total-etch resin cements, SACs show less incidence of post-operative sensitivity. The acidic monomers etch dentine without opening the dentine tubules and incorporate the smear layer into the shallow hybrid layer. As the smear layer acts as an intermediate bonding substrate during the setting reaction of SACs and near neutral pH is achieved rapidly, a reduction in post-operative sensitivity with these cements is observed. A follow-up for full-coverage restorations luted with SACs reported 0% post-operative sensitivity at the end of 6 months to 1 year.[47,48]

Crown retention

The retentive values for crowns cemented with SACs is reported to be greater than the conventional cements and equivalent to that of self-etch resin cements.[49,50]

Expansion

Self-adhesive resin cements have expansion rates less than resin-modified glass ionomers but higher than total-etch resin cements.[51] Thus they can be recommended for the cementation of weaker ceramic materials without the danger of fracturing the ceramic because of cement expansion.

Biocompatibility

Though studies have reported contrasting results regarding the cytotoxic effects of SACs on dental pulp, it is suggested that they are well tolerated by the pulp, if sufficient underlying dentine barrier is present.[52,53] A slight to moderate inflammatory pulpal response has been observed with SACs owing to their high viscosity and limited penetration in dentine, because of absence of a pre-etching step, as opposed to total-etch cements.[9] No reparative dentine formation and bacterial infiltration is observed with resin cements. However, their degree of monomer conversion and long-term bond durability is still uncertain.[54] A recent study evaluated the bond degradation behaviour of an SAC (*G-Cem*) under simulated oral conditions and showed that they have higher water sorption and solubility than conventional resin cements.[55] This may lead to possible cytotoxic effects and pulpal damage, as partial polymerization of the cements may increase their solubility into fluid solutions under oral conditions.[53]

Clinical performance/success

Burke and colleagues[8] reported that SACs are more convenient to handle and use than the conventional luting agents. Success rate as high as 100% and clinical success ranging from 2–4years has been reported for both metal and ceramic restorations luted with SACs in terms of retention, colour match, post-operative sensitivity wear rate and the overall clinical performance.[56,57,58] A questionnaire study, conducted by a PREP (practitioner-based research group) Panel, to obtain information regarding the use of SACs showed that evaluators rated higher for *RelyX Unicem* in terms of ease of use and handling than the other resin and conventional cements with very low incidence of post-operative sensitivity and de-bonding, and would purchase the material if made available at an appropriate commercial cost.[8] Also, the bonding effectiveness of SACs is not influenced by eugenol-containing provisional cements[59] and placement of endodontic sealers like, *AH Plus*, *Epiphany* and *Sealer 26*.[60]

However, there is still a paucity of long-term *in vivo* studies that assess the performance of self-adhesive luting materials in clinical settings.

As they are dual-cured cements, all the accessible restorative margins should be light-cured to improve the marginal integrity, wear resistance and stain resistance; as a reduction in bond strength, colour stability and wear resistance is usually observed when only self-cure mode is applied to dual-cured cements. Giráldez *et al*[61] recommended that the dual-curing resin cements should always be light irradiated for longer periods than that recommended by manufacturers as it significantly increased their microhardness. Excess cement should be removed before setting to avoid damaging the early weak bond.

Conclusion

As newer materials are introduced into the market, with improvisation over the limitations of previous materials, it is quite difficult to keep track of each prospective material and its properties. Currently, wide choices of self-adhesive cements are available to the clinicians. The article has reviewed the properties of various self-adhesives cements as a whole. According to studies, the bond strength of self-adhesive cement to dentine is better and clinically more satisfactory as compared to the bonding with enamel. However, *in vivo* clinical evaluations are few and short term; so drawing long-term conclusions about the overall effectiveness of these cements in dental practice is not yet possible. Thus the selection of a self-adhesive cement for any clinical application should be evidence-based, depending on its properties and not totally by operator preference.

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