INTRODUCTION

Dental caries structure has two layers, as established by Fusayama: an outer, infected, highly disorganized and non-remineralizable layer, and an inner layer, called caries-affected dentin, which can structurally be recovered by re-mineralization. In caries treatment, the first layer should be removed, whereas the second should be maintained and followed by the adhesive procedures, according to the philosophy of minimal intervention. However, certain chemical, biological and physical modifications occur during caries process and create a distinct situation that hinders adhesion to the caries-affected dentin. First, the ultimate tensile strength of carious dentin itself is lower in comparison with normal dentin, due to loss of minerals. Second, the caries-affected dentin is more permeable than normal, due to less mineral content and fewer cross-links. Furthermore, the lumen of dentinal tubules is obliterated by rhombohedral crystals of Mg-substituted β-TCP (whitlockite minerals) that are larger and more acid-resistant than normal apatites.

This distinct situation promotes the selective demineralization of caries-affected dentin. More permeable and less mineralized intertubular dentin is deeply demineralized, and the tube lumens obliterated by rhombohedral crystal are less affected. This situation could lead to a poorer quality hybrid layer than that occurring in normal dentin. Furthermore, it seems more critical for self-etch adhesives than total-etch adhesives. Moreover, mineralized dentin tube lumen and smear layer could buffer the acidic primers of self-etch adhesives, impairing dentin hybridization.

Thus, it is important to consider the performance of adhesive systems in normal and in caries-affected dentin, when making laboratory assessments, since clinical adhesion occurs in a combination of both. A major difficulty in laboratory studies is the standardization of caries-affected dentin. Some studies use subjective methods such as dentin color, dentin resistance to excavators, or dentin color stained by caries detector solution. An objective method is to measure the fluorescence emitted by bacterial metabolites or through dentin hardness. Since caries-affected-dentin is known to have half the Knoop hardness of normal dentin.

Based on this premise, a portable device for measuring Knoop hardness has recently been developed. This equipment allows dental substrate hardness to be measured, regardless of shape or angle, or even in vivo or in vitro situations.

Therefore, the aim of this study was to compare the microtensile bond strength (µTBS) of currently available self-etch adhesives with an experimental self-etch adhesive to normal and caries-affected dentin, using a portable hardness measuring device, in order to standardize dentin Knoop hardness. Normal (ND) and caries-affected dentin (CAD) were obtained from twenty human molars with class II natural caries. The following adhesive systems were tested: Mega Bond (MB), a 2-step self-etching adhesive; MTB-200 (MTB), an experimental 1-step self-etching adhesive (1-SEA), and two commercially available one-step self-etching systems, G-Bond Plus (GB) and Adper Easy Bond (EB). MB-ND achieved the highest µTBS. The mean µTBS was statistically lower in CAD than in ND for all adhesives tested, and the 2-step self-etch adhesive achieved better overall performance than the 1-step self-etch adhesives.
adhesive systems was also investigated by Scanning Electron Microscope (SEM). The hypotheses tested were that the μTBS to caries-affected dentin was the same as to normal dentin.

MATERIALS AND METHODS

Tooth preparation

After approval from the Federal University of Santa Catarina Review Board (#763.814), 20 extracted human third molars with class II carious lesions (Fig. 1a) and 4 teeth with class I carious lesions were selected and stored in 0.5% chloramine T for at least 1 month until initiating the tests. In exposing the caries-affected dentin of 20 teeth with class II lesions, the enamel portion was trimmed in a gypsum model trimmer (MT-7, J Morita Tokyo, Tokyo, Japan) to allow direct access to the carious dentin. The dentin caries lesions were then removed with a manual excavator assisted by a caries detector dye (1% acid red in polypropylene glycol, Caries Check, Nippon Shika Yakuhin, Shimonoseki, Japan). Initially, the caries-affected dentin was verified by dentin resistance to excavation, color and visual inspection. Then the Cariotester with a dentin indenter was used to standardize dentin hardness. Five measurements were made of the caries-affected dentin cavity, and the Knoop hardness number (KHN) was standardized between 25 and 30 KHN. The enamel was removed from the opposite side of the caries-affected dentin to expose the normal dentin. The enamel was trimmed (MT-7, J Morita Tokyo) until it formed a flat dentin surface, and then ground manually with #600 SiC paper for 60 s to standardize the smear layer; the root portion was trimmed according to the same method.

The teeth with caries-affected dentin (CAD) on one side and normal dentin (ND) on the other were randomly divided into 4 groups, according to the adhesive system tested: a two-step self-etching system, MB (Clearfil Mega Bond, Kuraray Noritake Dental, Tokyo, Japan); an experimental one-step self-etching...
system, MTB (MTB-200, Kuraray Noritake Dental); and two commercially available one-step self-etching systems, GB (G-Bond Plus, GC, Tokyo, Japan) and EB (Adper Easy Bond, 3M ESPE, St. Paul, MN, USA). The components, application mode and batch number of the adhesive systems tested are shown in Table 1. The restorative procedure was performed with 2 increments, each 2 mm high, of composite resin (Clearfil AP-X, Kuraray Noritake Dental), light activated for 40 s at 500 mW/cm². A C4 shade was used on the caries-affected dentin side, and an A2 shade was used on the normal dentin side, to differentiate the dentin sides (Fig. 1b). The restored specimens were stored in distilled water for 24 h at 37°C.

Microtensile bond strength (µTBS) test
To test the µTBS, the teeth were cut serially transversal to the adhesive interfaces in X and Y directions, with a low speed diamond saw in a high-precision cutting instrument, with copious water-cooling (Isomet, Buehler, Lake Bluff, IL, USA), to obtain 1.0×1.0 mm sticks, with a cross-sectional area of approximately 1.0 mm². Each stick used in this study was formed by two portions of composite resin and one portion of dentin in the center (Fig. 1c). The cross-sectional area of the sticks obtained was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan).

The sticks were divided into two groups. The caries-affected interface was tested in one group, and the sound dentin, in the other. For this purpose, one end of each stick was attached to a Ciucchi’s metallic jig with cyanoacrylate resin (Model Repair, Dentsply-Sankin, Otahara, Japan), and the opposite end was attached below the adhesive interface that was not tested (Fig. 1d). The µTBS test was performed in a universal testing machine (EZ-Test, Shimadzu, Kyoto, Japan) at 1 mm/min crosshead speed until failure. The µTBS (MPa) values were calculated by dividing the load (N) at the time of fracture by the cross-sectional area of each stick (mm²). After completing the µTBS test, all the sticks were retested with the Caritester, in order to ensure that the tested substrate was caries-affected. The µTBS data obtained was subjected to two-way ANOVA and multiple post-hoc comparisons were made by the Games-Howell test, both at a 5% significance level. The failure modes were observed at 20× magnification on a stereomicroscope (Magnifier Light, As One, Osaka, Japan), and were classified as adhesive, cohesive in

<table>
<thead>
<tr>
<th>System</th>
<th>Ingredients</th>
<th>Batch number</th>
<th>Procedures</th>
</tr>
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<tbody>
<tr>
<td>Clearfil MegaBond (Kuraray Noritake Dental, Tokyo, Japan)</td>
<td>Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water. Bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, photo-initiator, silanated colloidal silica.</td>
<td>01087A</td>
<td>Apply the primer over the substrate; leave it in place for 20 s. Dry the primer with mild air for more than 5 s. Apply the adhesive over the substrate and gently air blow. Light-cure with a dental curing unit for 10 s.</td>
</tr>
<tr>
<td>MTB-200 (Kuraray Noritake Dental)</td>
<td>10-MDP; HEMA; Bis-GMA; Hydrophilic aliphatic dimethacrylate; Hydrophobic aliphatic methacrylate; Colloidal silica; dl-Camphorquinone; Accelerators; Initiators; Water; Ethanol; Sodium fluoride.</td>
<td>090911</td>
<td>Apply the bond over the substrate; leave it in place for 20 s. Dry the bond with mild air for more than 5 s. Light-cure with a dental curing unit for 10 s.</td>
</tr>
<tr>
<td>G-Bond PLUS (GC, Tokyo, Japan)</td>
<td>Phosphoric acid ester monomer, 4-MET, Dimethacrylate monomer, Water, Acetone, Photo-initiator, Nano-silica filler</td>
<td>1009101</td>
<td>Apply the bond over the substrate; leave it in place for 10 s. Dry the bond with maximum air pressure for 5 s. Light-cure with a dental curing unit for 10 s.</td>
</tr>
<tr>
<td>Adper Easy Bond Self-etch Adhesive (3M ESPE, St. Paul, MN, USA)</td>
<td>HEMA; Bis-GMA; Methacrylated phosphoric esters; 1,6 hexanediol dimethacrylate; Methacrylate functionalized polyalkenoic acid (Vitrebond™Copolymer); Finely dispersed bonded silica filler with 7 nm primary particle size; Ethanol; Water; Initiators based on camphorquinone; Stabilizers.</td>
<td>423278</td>
<td>Apply the bond over the substrate for 20 s. Air thin the bond for 5 s. Light-cure with a dental curing unit for 10 s.</td>
</tr>
</tbody>
</table>
dentin or resin, and mixed.

**SEM observation**

Another 4 teeth with occlusal caries were selected for adhesive interface analysis in a Field Emission Scanning Electron Microscope (Fe-SEM) (S-4000, Hitachi, Tokyo, Japan). To this end, the occlusal enamel was trimmed (MT-7, J Morita Tokyo) and the caries removed in the same manner as that of the test groups. The teeth were then treated with one of adhesive systems of the study and restored. After storage in distilled water for 24 h, the teeth were sectioned mesiodistally to obtain slices with caries-affected/normal dentin and adhesive layer interface. The slices were polished sequentially with SiC papers (#600, #800 and #1000) and diamond pastes (6 µm; 3 µm; 1 µm DP-Paste, Struers, Denmark), and treated with 5% HCl for 30 s, followed by NaOCl for 5 min; each step was rinsed with water in an ultrasonic cleaner. After storage in a dry state for 24 h, the specimens were Pt-Pd sputter-coated for 120 s and analyzed by Fe-SEM (S-4000, Hitachi). Post photomicrographic evaluation was performed using Image J public domain software (version 1.47, National Institute of Health, Washington, DC, USA).

**RESULTS**

### µTBS test, fracture mode and Knoop Hardness Number (KHN)

The mean µTBS values (MPa) and standard deviation (SD) of the tested adhesives are shown in Table 2. There was a significant difference in mean µTBS between normal dentin and caries-affected dentin for all tested adhesives (p<0.05) (Table 2). When each adhesive system and dentin condition was analyzed by the Games-Howell post-hoc test (Table 2), MB-ND achieved the highest mean µTBS of all groups tested (p<0.05), followed by MB-CAD, GB-ND, MTB-ND and EB-ND, which were statistically similar (p<0.05); EB-ND was statically similar to GB-CAD (p<0.05); GB-CAD, MTB-CAD and

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>n</th>
<th>Normal dentin</th>
<th>Significance within the same rows</th>
<th>n</th>
<th>Caries-affected dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>MB</td>
<td>18</td>
<td>77.23 (±14.23)a</td>
<td>p&lt;0.05</td>
<td>24</td>
<td>42.98 (±15.07)b</td>
</tr>
<tr>
<td>MTB</td>
<td>21</td>
<td>43.28 (±12.98)b</td>
<td>p&lt;0.05</td>
<td>20</td>
<td>22.83 (±12.58)d</td>
</tr>
<tr>
<td>GB</td>
<td>28</td>
<td>46.36 (±19.34)b</td>
<td>p&lt;0.05</td>
<td>18</td>
<td>28.40 (±11.59)cd</td>
</tr>
<tr>
<td>EB</td>
<td>19</td>
<td>40.31 (±17.32)bc</td>
<td>p&lt;0.05</td>
<td>21</td>
<td>20.19 (±6.19)d</td>
</tr>
</tbody>
</table>

Means with identical lowercase letters are not significantly different (Games-Howel test, p>0.05).

### Fracture mode distribution of specimens in each group after µTBS.

<table>
<thead>
<tr>
<th>Adhesive (%)</th>
<th>Mixed (%)</th>
<th>Cohesive failure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>In dentin (%)</td>
<td>In composite (%)</td>
</tr>
<tr>
<td>MB Normal dentin</td>
<td>56.25</td>
<td>25.0</td>
</tr>
<tr>
<td>Caries-affected dentin</td>
<td>58.33</td>
<td>25.0</td>
</tr>
<tr>
<td>MTB Normal dentin</td>
<td>76.20</td>
<td>14.30</td>
</tr>
<tr>
<td>Caries-affected dentin</td>
<td>85.0</td>
<td>15.0</td>
</tr>
<tr>
<td>GB Normal dentin</td>
<td>67.86</td>
<td>17.86</td>
</tr>
<tr>
<td>Caries-affected dentin</td>
<td>77.78</td>
<td>16.67</td>
</tr>
<tr>
<td>EB Normal dentin</td>
<td>52.63</td>
<td>31.58</td>
</tr>
<tr>
<td>Caries-affected dentin</td>
<td>76.20</td>
<td>23.80</td>
</tr>
</tbody>
</table>

Adhesive=failure in adhesive interface between dentin and resin.
Mixed=failures that occurred partially in adhesive interface and in the dentin or resin composite.
Cohesive=failures that occurred totally in dentin or in resin composite.
Fig. 2 SEM images of normal and caries-affected dentin-adhesive interfaces.

All the samples of SEM images of CAD, (a) MB, (c) MTB, (e) GB and (g) EB, are characterized by an irregular and not well-defined hybrid layer. The hybrid layer of these images had an approximate 2–3 µm thick hybrid layer; however, this layer was not uniform in pattern or thickness. Only a few resin tags could be seen in the images; these were also not well-defined. (b) Shows an MB image of the normal dentin-adhesive interface with an approximately 0.5 µm thick hybrid layer, regular resin tag formation and some lateral branches; (d) Shows an MTB image of normal dentin with an approximately 0.4 µm thick hybrid layer; some resin tags can be observed and few lateral branches; (f) Shows a GB image of normal dentin with an approximately 0.2 µm thick hybrid layer; short resin tags can be observed; (h) Shows the EB dentin-adhesive interface with normal dentin, with an approximately 0.4 µm thick hybrid layer; few and short resin tags can be observed. HL: hybrid layer, T: resin tags, LB: lateral branches, A: adhesive layer, CR: composite resin.

Table 4 Knoop Hardness Number and standard deviation (SD) for adhesives tested in sound dentin and caries-affected dentin

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>Sound dentin</th>
<th>Caries-affected dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>MB</td>
<td>42.93 (±5.28)a</td>
<td>28.08 (±4)b</td>
</tr>
<tr>
<td>MTB</td>
<td>44.48 (±4.06)a</td>
<td>28.35 (±3.26)b</td>
</tr>
<tr>
<td>GB</td>
<td>41.89 (±4.6)a</td>
<td>27.78 (±3.31)b</td>
</tr>
<tr>
<td>EB</td>
<td>46.64 (±2.75)a</td>
<td>28.02 (±3.2)b</td>
</tr>
</tbody>
</table>

Means with identical lowercase letters are not significantly different (Games-Howel test, p>0.05).

EB-CAD did not differ statistically ($p>0.05$).

There were statistically differences between adhesives tested; MB was statistically different from GB, MTB and EB ($p<0.05$), whereas there were no differences among the last three ($p>0.05$).

The fracture modes are shown in Table 3 and were predominantly adhesive for all groups tested. The KHN of normal dentin and caries-affected dentin are shown in Table 4; there were statistical differences between normal dentin and caries-affected dentin ($p<0.05$).

**SEM observations**

A thicker hybrid layer (about 2–3 µm thick) can be observed in all groups of the caries-affected dentin interface SEM images (Figs. 2-a, -c, -e and -g), in comparison to normal dentin (ranging between 0.2–0.5 µm thick) (Figs. 2-b, -d, -f and -h).

SEM images of the interface between the MB (Fig. 2-a), MTB (Fig. 2-c) and GB (Fig. 2-e) adhesives and the caries-affected dentin area also showed irregular and not-well defined resin tags formation. The same pattern could be observed in relation to the EB (Fig. 2-g) caries-affected interface, however, with fewer resin tags than those observed in other groups.

On the other hand, the SEM image of MB-ND (Fig. 2-h) showed regular resin tag formation, and even some lateral branches. Resin tags and lateral branches were also observed in MTB-ND (Fig. 2-d), but in a smaller number than those observed in MB-ND. The SEM image of GB-ND (Fig. 2-f) showed regular resin tag formation, like that of MB-ND, but shorter, whereas the SEM images of EB-ND (Fig. 2-h) showed short and sparse resin tags.

**DISCUSSION**

The µTBS of caries-affected dentin was statistically lower in the adhesive systems tested in this study. These results were in agreement with others studies, which showed a lower µTBS in caries-affected dentin compared with normal dentin$^{5,7,10,16,17}$. These findings reject the null hypothesis that there were no differences in bond strength between normal dentin and caries-affected dentin.

The results of the present study could have been influenced by the method used to obtain the final substrates of caries-affected and normal dentin, in so far as the caries-affected dentin surfaces were not flat, like those of others studies$^{5,7,10,16,17}$. The caries were removed manually with a hand excavator until the point determined in the materials and methods, leaving the final shape of the caries-affected surface irregular; furthermore, the smear layer was not standardized by other methods. This situation was clinically similar but more challenging to test. The cross-sectional area used to determine the µTBS was 1 mm², adopted for the purpose of minimizing differences between the sticks in relation to the bonded area$^{18,19}$. At the same time, this methodology allowed testing the normal and caries-affected dentin in the same tooth, thus minimizing the influence of tooth differences.

Although, the caries-affected dentin results obtained in the present study follow a clinical protocol, care was taken to minimize the variability by standardizing the Knoop hardness using a manual system to determine hardness. This instrument was validated by a study that compared it with a fluorescent laser device (DIAGNoDent, KAVO, Biberach, Germany)$^{14}$. In the study referred to, an inverse relationship was found between the Knoop hardness obtained by Cariotester and the fluorescence obtained by Diagnodent. This relationship suggests that caries progression can be evaluated by Knoop hardness. Another study found that there was a positive correlation between the degree of remineralization of carious dentin and the amount of penetration of the tip of Cariotester, thus demonstrating that carious lesion remineralization can be monitored in vitro by this device$^{15}$.

The Knoop hardness of caries-affected dentin found in this study is in agreement with other studies; however, the hardness of normal dentin was slightly lower than that found in the same studies$^{9,10,17}$. The present study uses the normal dentin of the axial wall, which was found to have a lower Knoop hardness than the dentin of pulpal wall$^{29}$.

Some authors suggest that aggressive acids could dissolve the whitlockite crystals in the dentin tubule lumen, resulting in better adhesive performance; however, the literature is controversial in relation to the advantages to bond strength of phosphoric acid versus self-etch primer etching capacity$^{5,7,8,10,17,21}$. Furthermore, one concern of the total etch approach relates to the discrepancy between demineralized depth and resin-infiltrated depth. This could result in inadequately enveloped, unprotected collagen fibrils. Using Raman microspectroscopy, it was seen that the phosphoric acid used to test the adhesive system was incapable of removing the rhombohedral crystals of Mg-substituted β-TCP, and that the pattern of demineralization was irregular and deeper than in the caries-affected dentin. Moreover, the demineralized depth of normal dentin was approximately 7–8 µm, whereas that of caries-affected dentin was approximately 14–16 µm$^{30}$. Based on this, the best solution would be for the less sensitive self-etch adhesive technique to work the same way in both normal and caries-affected dentin. However, in the current study all adhesives tested performed worse in the caries-affected than in the normal dentin.

Clearfil Mega Bond is considered as a gold standard for self-etch adhesive systems, achieving bond strengths comparable or even superior to etch-and-rinse adhesives$^{22,23}$. In the current study, this adhesive showed statistically higher µTBS to normal dentin than all the other adhesive groups. SEM image showed resin tags that were longer and had better quantity, and even lateral branches. However, in the caries-affected group, the µTBS of Clearfil Mega Bond was statistical lower in caries-affected versus normal dentin. SEM image showed a thick hybrid layer, less resin tag quantity
and absence of lateral branches. Studies attributed the better results of this adhesive system in dentin to the 10-MDP functional monomer, which bonded chemically to hydroxyapatites; however, in caries-affected dentin, the dentin was less crystallized. This dentin condition could have been influenced negatively to the µTBS; nevertheless, this adhesive group presented cohesive failures in dentin, indicating higher bonding to dentin^{23-26}.

MTB-200 is an experimental one-step self-etch adhesive based on the same 10-MDP functional monomer; however, its behavior in this study was more similar to the other one-step adhesives tested. The mean µTBS was not statistically different between MTB-200 and G-Bond Plus or Adper Easy Bond in normal dentin, or between MTB-200 in normal dentin and Clearfil Mega Bond in caries-affected dentin, whereas the µTBS of MTB-200 was influenced negatively by the caries-affected dentin. The SEM images showed fewer and short resin tags in normal dentin, whereas unclear resin tags were found in the specimens of caries-affected dentin SEM sample. The fracture mode in the caries-affected dentin was predominately adhesive. Despite the results of this study, a previous study showed that additions to MTB-200, such as sodium fluoride, a new hydrophobic methacrylate and new photo-initiators, resulted in enhanced resistance to bonding degradation after a stress condition of 20,000 thermal cycles, as well as superior µTBS, compared to two other 1-step self-etch adhesives (Clearfil S3, Kuraray Noritake Dental; and Beautibond, Shofu, Kyoto, Japan)^27.

The same results were obtained by G-Bond Plus, where the µTBS to caries-affected dentin was statistically lower than to normal dentin. G-Bond Plus is the only adhesive that does not have HEMA in its composition in this study. The SEM of the bonding interface of this adhesive showed a slightly less thick hybrid layer in normal and caries-affected dentin, in comparison with the other adhesive groups. HEMA is considered as a highly hydrophilic co-monomer that prevents phase separation and promotes dentin wetting, improving the bond strength. Owing to its high hydrophilicity, it can penetrate deeper into the dentin; thus, the lack of HEMA in G-Bond Plus may lead to a thinner hybrid layer in the SEM images of normal and caries-affected dentin observed in the present study^{23-26}. This adhesive is very sensitive to air-blowing time. It was shown that there were statistical differences between even small differences in air-blowing time, i.e., 25 s resulted in a significantly higher bond strength than 5 s^{15}. Therefore, more studies are needed to verify the influence of air-blowing time on the bond strength of caries-affected dentin.

There were also statistical differences in the mean µTBS for Adper Easy Bond, between caries-affected dentin and normal dentin. In the SEM image of this adhesive in caries-affected dentin, fewer resin tags can be observed than in other caries-affected adhesive groups. In previous studies, it was suggested that the behavior of Adper Easy Bond is highly dependent on how the substrate is prepared^{22}. These findings could explain the SEM image for caries-affected dentin. The crystals in the dentinal tubes could keep Adper Easy Bond from producing clearer resin tags than the others adhesives. Comparing the SEM images from each group, it is possible to observe that the hybrid layer pattern was influenced by the adhesive system and the dentin condition.

All adhesives tested are composed of one kind of functional monomer: 10-MDP in Clearfil Mega bond and in MTB-200; 4-MET in G-Bond Plus; and Vitrebond™ Copolymer in Adper Easy Bond. These monomers promote a chemical interaction with residual hydroxyapatite. In normal situations, the functional monomers in each adhesive work in conjuction with micromechanical hybridization, and impart additional chemical bonding to the dentin substrate^{24,29}. However, alterations in the proportion of residual hydroxyapatites in intertubular dentin, and a larger amount of water in caries-affected dentin, could result in differences in the caries-affected dentin^{30,31}.

Considering the result of the present study, further research is needed to evaluate the influence of caries-activity-related dentin substrate alterations on these functional monomers, and, consequently, on bond strength. More studies are also needed to produce adhesive systems that are less influenced by different dentin conditions.

**CONCLUSION**

Within the limitations of the present study, the mean µTBS to caries-affected dentin was statistically lower than to normal dentin. Generally, two-step self-etch adhesives outperform one-step self-etch adhesives. The portable hardness measuring equipment proved a viable device for standardization of dentin hardness.

**ACKNOWLEDGMENTS**

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