

# Detorque Evaluation of Dental Abutment Screws after Immersion in a Fluoridated Artificial Saliva Solution

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## Keywords

Dental abutments; dental implants; screw loosening; torque; corrosion.

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## Abstract

**Purpose:** Implant-abutment connections still present failures in the oral cavity due to the loosening of mechanical integrity by detorque and corrosion of the abutment screws. The objective of this study was to evaluate the detorque of dental abutment screws before and after immersion in fluoridated solutions.

**Materials and Methods:** Five commercial implant-abutment assemblies were assessed in this investigation: (C) Conexão<sup>®</sup>, (E) Emfils<sup>®</sup>, (I) INP<sup>®</sup>, (S) SIN<sup>®</sup>, and (T) Titanium Fix<sup>®</sup>. The implants were embedded in an acrylic resin and then placed in a holding device. The abutments were first connected to the implants and torqued to 20 Ncm using a handheld torque meter. The detorque values of the abutments were evaluated after 10 minutes. After applying a second torque of 20 Ncm, implant-abutment assemblies were withdrawn every 3 hours for 12 hours in a fluoridated solution over a period of 90 days. After that period, detorque of the abutments was examined. Scanning electronic microscopy (SEM) associated to energy dispersive spectroscopy (EDS) was applied to inspect the surfaces of abutments.

**Results:** Detorque values of systems C, E, and I immersed in the fluoridated solution were significantly higher than those of the initial detorque. ANOVA demonstrated no significant differences in detorque values between designs S and T. Signs of localized corrosion could not be detected by SEM although chemical analysis by EDS showed the presence of elements involved in corrosive processes.

**Conclusion:** An increase of detorque values recorded on abutments after immersion in fluoridated artificial saliva solutions was noticed in this study. Regarding chemical analysis, such an increase of detorque can result from a corrosion layer formed between metallic surfaces at static contact in the implant-abutment joint during immersion in the fluoridated solutions.

Since the noteworthy findings on titanium properties such as corrosion resistance and biocompatibility, Ti and its alloys have been largely applied in implant-supported oral rehabilitation.<sup>1-3</sup> For instance, dental implants are produced from commercially pure (Cp) Ti, while abutments, overdenture bars, and fixed prosthesis frameworks can be produced from Ti alloys.<sup>1-6</sup> In fact, such applications consider a match of mechanical and biological properties among Cp Ti, Ti alloy, bone, and surrounding tissues.<sup>1,6,7</sup> Notwithstanding a compact and protective TiO<sub>2</sub> film formed on the Ti surface responsible for its high corrosion resistance and biocompatibility, Ti can be degraded in the presence of H<sub>2</sub>O<sub>2</sub>, urea (carbamide) peroxide, lactic acid, and

high fluoride content.<sup>8-14</sup> All of those corrosive substances can be found in the oral cavity at different concentrations and pH values.<sup>8,15</sup> For instance, fluorides can be found in commercial dentifrices (1000 to 1500 ppm F<sup>-</sup>) and topical gels (up to 12,300 ppm F<sup>-</sup> at pH of 4 to 6), while H<sub>2</sub>O<sub>2</sub> (10% to 15%) and carbamide peroxide (8% to 15%) can be applied in dental bleaching treatments.<sup>8,15</sup> Ti degradation can promote a significant material loss, leading to metallic ion release to surrounding tissues.<sup>10,11</sup> As a consequence, metallic ions can stimulate peri-implant inflammations.<sup>16-18</sup>

Failures in oral rehabilitation involving dental implants have been reported in the literature.<sup>19-22</sup> These failures are often

**Table 1** Specifications of the dental implant systems used in this study

| Group | Connection | Dimension (mm)                                | Chemical composition            | Commercial brand  | Batch                    |
|-------|------------|---|---------------------------------|---|--------------------------|
| 1 (C) | EH         | Impl.: 3.75 × 13<br>Abut.: 4.0 × 2.0          | Ti-based alloy<br>Ti6Al4V alloy | CONEXÃO <sup>®</sup> (São Paulo, Brazil)                | 4121844266<br>5010315002 |
| 2 (E) | IH         | Impl.: 4 × 13<br>Abut.: 4.0 × 2.0<br>3.5 × 15 | Ti-based alloy<br>Ti6Al4V alloy | EMFILS <sup>®</sup> (São Paulo, Brazil)                 | 050205<br>050108         |
| 3 (I) | EH         | Impl.: 4.0 × 15<br>Abut.: 4.0 × 3,0           | Ti-based alloy<br>Ti6Al4V alloy | INP <sup>®</sup> (São Paulo, Brazil)                    | 050419<br>020205         |
| 4 (S) | EH         | Impl.: 4 × 11.5<br>Abut.: 4.1 × 3.5           | Ti-based alloy<br>Ti6Al4V alloy | SIN <sup>®</sup> (Água Rasa, Brazil)                    | D4070<br>B3021           |
| 5 (T) | EH         | Impl.: 3.75 × 13<br>Abut.: 4.1 × 2.0          | Ti-based alloy<br>Ti6Al4V alloy | TITANIUM FIX <sup>®</sup> (São José dos Campos, Brazil) | 513/05<br>029/05         |

EH = external hexagon; IH = internal hexagon.

associated with several factors such as microbial colonization, corrosion of materials, peri-implant inflammations, implant-abutment design, overload distribution, and bone loss.<sup>10,11,16,20</sup> Considering the implant-abutment joints, a loosening of mechanical integrity of implant-abutment connections by abutment unscrewing is reported as being caused by detorque in 1% to 40% of cases.<sup>23-26</sup> Implant-abutment misfit, surface characteristics, unsatisfactory position, and structural geometry of implants can generate or increase overloads distributed through the structural materials onto the bone.<sup>20,22,27-30</sup> In fact, a relative motion like micro-sliding occurs between contacting surfaces during mastication,<sup>31,32</sup> leading to a simultaneous wear and corrosion process of structural materials.<sup>10,11,33,34</sup> The penetration of oral fluids like fluorides and acidic substances between microgaps formed in the implant-abutment connection can be responsible for the corrosion of structural materials, including abutment screws.<sup>10,11,34,35</sup> Also, the penetration of fluids can be followed by microorganisms with their extracellular matrix and metabolic products.<sup>35-39</sup> Souza *et al*<sup>11</sup> revealed low friction on Ti in the presence of biofilms or mucin. The low friction can decrease the torque in implant-abutment connections. On the other hand, several acidic substances (e.g., lactic acid) that can decrease the pH of surrounding areas can be released or accumulated by biofilms. The corrosion of Ti caused by the presence of lactic acid has been reported in previous studies.<sup>9,10</sup> In fact, the internal connection of implant-abutment joints is a retentive area for biofilms and corrosive substances that can accelerate the corrosion of structural materials and consequently promote the loosening of mechanical integrity of dental implant connections.

The objective of this study was to investigate the influence of a fluoridated artificial saliva on detorque of dental abutment screws. Also, surfaces were inspected by scanning electron microscopy (SEM) associated to spectroscopy energy dispersive analysis.

## Materials and methods

### Evaluation of initial weight and detorque of abutments

Fifty commercial dental implant systems were divided into five groups of 10 implant-abutment systems ( $n = 10$ ) (Table 1).

The implants were embedded in acrylic resin and then placed in a metallic holding device (Fig 1A). The abutments were connected to the implants by applying a torque of 20 Ncm with the use of a handheld torque meter (TONICHI ATG09CN, Tonichi America Corp, Northbrook, IL) coupled to a metallic holding device.<sup>40</sup> Metallic holding devices can avoid oblique loads during torque application. After 10 minutes, the initial detorque of the abutments was evaluated using the same handheld torque meter. Then, a second torque of 20 Ncm was applied to establish the implant-abutment connection for detorque evaluation after immersion in fluoridated solution (Fig 1A). Gravimetric analysis (SA120, Scientech, Boulder, CO) was applied to evaluate the abutment weight before connection to dental implants.

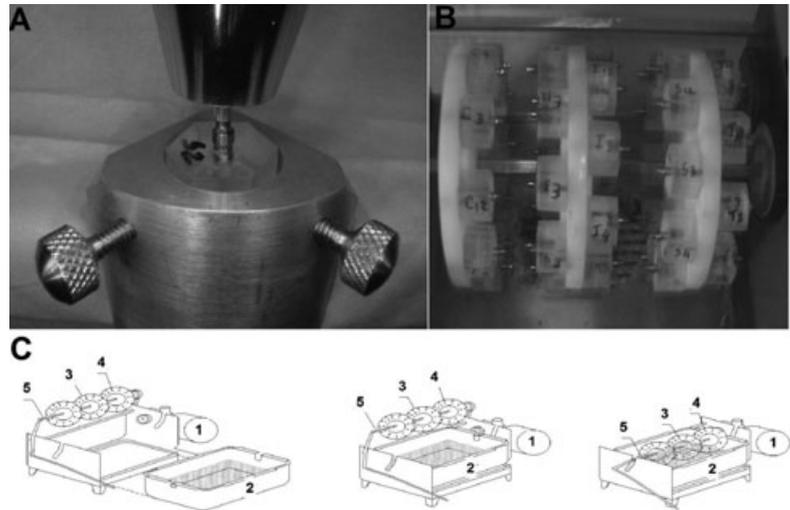
### Evaluation of detorque and abutment weight after immersion in fluoridated solutions

The implant-abutment assemblies torqued at 20 Ncm were placed on nylon disks (Fig 1B). Then, those nylon disks were coupled to an electric rotator device for immersion tests (Fig 1C). On rotating at 1 rpm for 12 hours, the implant-abutment assemblies were immersed daily in a fluoridated artificial saliva solution at 37°C and withdrawn every 3 hours over a period of 90 days. The fluoridated artificial saliva solution was prepared by diluting a commercial dental dentifrice (Contente<sup>®</sup>, Suavetex, Uberlândia, Brazil) in an artificial saliva solution, in a proportion of 1/3 (wt./v.), under vortex for 30 minutes. The compositions of commercial dentifrice and artificial saliva are shown in Table 2.

After 90 days, the final detorque of the abutments was evaluated using the handheld torque meter. Also, the weight of abutments after immersion was evaluated by gravimetric analysis.

### Scanning electron microscopy of dental implants and abutment screws

The implant-abutment assemblies were cleaned in distilled water for 15 minutes and then in acetone for 5 minutes using an ultrasonic bath. Then, implant-abutment joints were inspected by SEM (LEO-Stereocam 440, LEO Elettron Microscopy Ltd., Cambridge, UK) associated with energy



**Figure 1** (A) Connection between abutment and dental implants applying a 20 Ncm torque using a handheld torque meter. (B) Implant-abutment assemblies placed on acrylic disks for immersion tests. (C) Schematic illustration of the electric rotator device coupled to an immersion bath (1—electric driver; 2—immersing bath; 3—acrylic disk; 4—implant-abutment assemblies; 5—holder).

**Table 2** Composition of the dental dentifrice and fluoridated artificial saliva used as a stock solution (pH 5.5) in this study

| Dental dentifrice       |                     | Artificial saliva solution                      |                     |
|-------------------------|---------------------|---|---------------------|
| Compound                | Concentration (g/l) | Compound  | Concentration (g/l) |
| NaFPO <sub>4</sub>      | 1500 ppm            | NaPO <sub>4</sub>                               | 0.780               |
| CaCO <sub>4</sub>       | —                   | NaCl  | 0.5                 |
| Sodium laureth sulfate  | —                   | KCl   | 0.5                 |
| Sodium saccharin        | —                   | CaCl  | 0.795               |
| Sorbitol                | —                   | NaS   | 0.05                |
| Carboxymethyl cellulose | —                   | (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> | 0.3                 |
| Methyl-paraben          | —                   | Citric acid                                     | 0.05                |
| H <sub>2</sub> O        | —                   | NaCO <sub>4</sub>                               | 0.1                 |
|                         |                     | Urea  | 1.0                 |

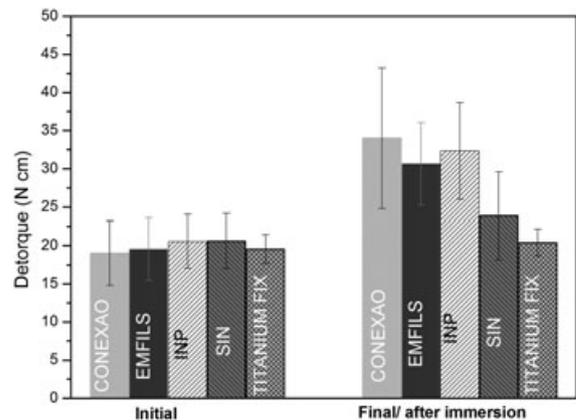
dispersive spectroscopy (EDS) for chemical analysis. After that, the abutments were unscrewed and cleaned using the same protocol for the implant-abutment assemblies. Finally, the abutment screw surfaces were inspected by SEM-EDS. Images were obtained by secondary electron (SE) and back-scattered electron (BSE) modes at 20 kV.

**Statistical analysis**

The results were statistically analyzed via two-way ANOVA, using a significance level of *p* < 0.05. Tukey’s analysis was applied to compare groups, while Pearson analysis investigated the correlation between weight increase and detorque values.

**Results**

Detorque values of abutments are shown in Figure 2. An increase of detorque values was noticed after immersion in fluoridated solution for 90 days. Tukey’s multiple comparison test indicated no significant difference between groups T and S; however, the detorque values of groups C, E, and I were sig-



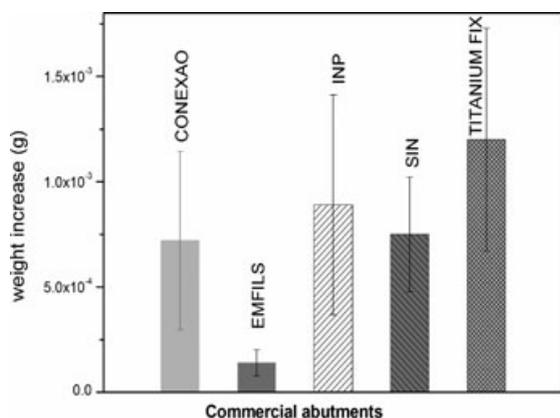
**Figure 2** Detorque values before (initial) and after immersion in fluoridated artificial saliva solution for 90 days.

nificantly higher compared to the initial detorque values. Also, an increase of abutment weight was noticed after immersion in fluoridated solution for 90 days (Fig 3).

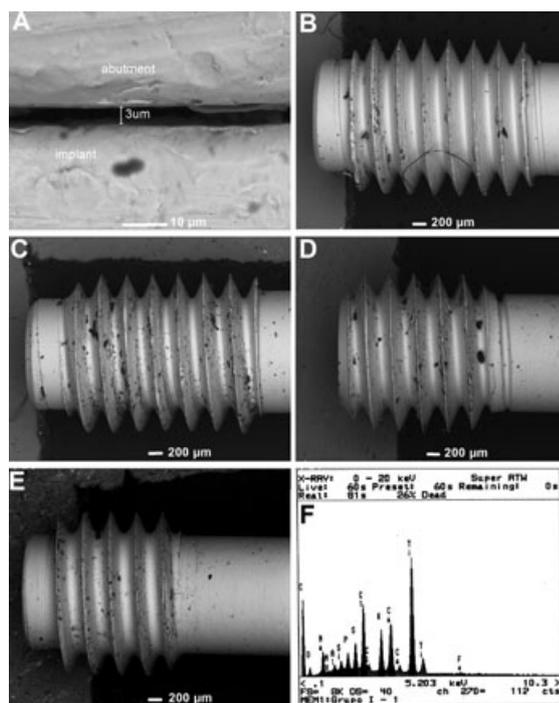
The weight of all abutments (C, E, I, S, T) increased after immersion in the fluoridated solution, although group E presented the lowest values. Concerning weight, there was no significant difference among groups C, S, I, and T. Pearson analyses indicated no significant correlation between detorque and weight increase.

The topography of an implant-abutment assembly and abutment screws is shown in Figure 4A-E. Figure 4A reveals an implant-abutment joint with a vertical misfit (microgap dimension) of about 3 μm.

Black spots were observed on abutment screw surfaces after detorquing (Fig 4C-E) and suggest the adsorption of compounds from the fluoridated artificial saliva solution. The elemental analysis (Fig 4F) on black spots reveals a high intensity of Cl, C, Na, and K, suggesting the reaction of chlorides and dentifrice compounds taking place on titanium.



**Figure 3** Increase of abutment weight after immersion in fluoridated artificial saliva solution for 90 days.



**Figure 4** SEM micrographs of (A) an implant-abutment joint, (B) control group abutment, (C) Conexão abutment, (D) SIN abutment, and (E) INP abutment after immersion. Images obtained by BSE mode at 20 kV. (F) Elemental analysis by EDS of INP abutment after immersion.

**Discussion**

For implant-abutment connection, a torque is applied on abutment screws generating a preload on the structural materials.<sup>26</sup> Thus, the composition of structural materials and screw characteristics can determine preload, whereas a torque up to 75% to 80% of material strength is recommended.<sup>30</sup> For instance, a Cp Ti grade 1 abutment screw can generate a preload of 120 Ncm when torqued to 75% of its strength, while a Ti alloy screw can generate a 400 Ncm preload after the same torque.<sup>23</sup> The initial unscrewing of abutments can occur due to the compres-

sion of screw heads leading to a lower friction and decrease of preload.<sup>29,41</sup> Schulte and Coffey<sup>42</sup> revealed that 81% of gold screws and 71% of Ti abutment screws presented torque loosening after applying a 10 Ncm initial torque. In our study, initial detorque occurred, although it was less significant compared to previous findings.<sup>43-45</sup>

After immersion in fluoridated artificial saliva, detorque values of abutments tested in this study were higher than those before immersion. A galvanic corrosion can be responsible for that, considering implant and abutment (metallic materials) are coupled in the presence of an electrolyte (artificial saliva).<sup>46-50</sup> An electrical current distribution is established between a Ti alloy abutment (anode) and Cp Ti implant (cathode) when the artificial saliva solution penetrates into microgaps. As a result, there is an increase of chemical reactivity between implant and abutment surfaces forming a reaction layer that maintains the torque on abutment screws (Fig 2). Results from chemical analysis (Fig 4F) indicate the presence of a complex reaction layer composed of Cl, Na, S, K, Ca, P, O and Ti onto Ti-based structures. That reaction layer can promote an implant-abutment adhesion on static contact; however, a rupture of the reaction layer and wear of structural materials of the implant-abutment joint might occur due to the micromovements during mastication. Then, a new reaction layer can be formed in contact with the environment, and the corrosion remains with a progressive material loss. Additionally, compression on abutment screws and micromovements can decrease the torque and friction on the thread, leading to abutment screw loosening.<sup>26,41,51</sup>

The weight increase shown in Figure 3 may occur due to the wear of implant connection surfaces by abutment unscrewing. That can promote a deposition of implant material (less hard) on the abutment screw surface (harder) associated with the reaction layer formation; however, the wear during detorquing depends on the mechanical properties of the structural materials. Also, the complex reaction layer can vary in composition, thickness, and density depending on the composition of abutment and implants, or else on the size of microgaps along the inner implant-abutment joints. As mentioned in the literature, microgaps accumulate oral fluids such as fluorides and corrosive substances<sup>21,30,36,37</sup> that can increase the period and intensity of the chemical reaction between structural materials and the environment. Thus, the extent of the galvanic corrosion depends on the corrosion resistance of the metallic materials, their processing time, and the assembling of the implant system.<sup>46,49</sup> Additionally, different localized corrosion processes, such as crevice and pitting corrosion, can be associated with galvanic corrosion in the marginal gap between abutment and implant.<sup>46</sup> Such confounding variables can affect the relation between weight increase and detorque values, as noticed in our results.

Concerning corrosion of structural materials, Kuphasuk *et al*<sup>52</sup> revealed a higher corrosion resistance recorded on Cp Ti than that on Ti6Al4V alloy. Concerning the effect of fluorides, Zavanelli *et al*<sup>33</sup> investigated the corrosion and fatigue behavior of Cp Ti and Ti6Al4V in an artificial saliva solution with or without fluorides. Both materials presented the worst fatigue corrosion behavior in the fluoridated artificial saliva solution. Schiff *et al*<sup>14</sup> revealed a significant decrease of the corrosion resistance of Cp Ti and Ti6Al4V in Fusayama's

artificial saliva containing 1000 ppm F<sup>-</sup>. That was amplified when the pH decreased from 5.3 down to 2.5.<sup>14</sup> Concerning simultaneous corrosion and wear processes, Souza *et al*<sup>53</sup> reported on a significant weight loss after sliding corrosion tests in artificial saliva containing 227 (pH 5.5) and 12,300 ppm F<sup>-</sup> (pH 6.5), denoting a progressive degradation of Ti when the F<sup>-</sup> concentration increases. In fact, the association between F<sup>-</sup> and H<sup>+</sup> from hydrofluoric acid (HF) depends on the fluoride concentration and pH of the solution. Previous studies revealed a destruction of TiO<sub>2</sub> film and Ti surfaces at an HF concentration of 30 ppm.<sup>12-14</sup> For instance, 227 ppm F at a pH below 3.9 or 500 ppm F at a pH below 4.3 is enough to promote a localized corrosion of Ti.<sup>12</sup> In our study, the low concentration of fluorides in the solutions could not promote a localized corrosion of Ti surfaces. Gil *et al*<sup>54</sup> compared the corrosion behavior and ion release of AuAgPd, AgPdAu, AgPd, NiCr, and Ti alloys in artificial saliva (37°C, pH 6.7) for 250 minutes. Ti specimens presented the lowest chemical reactivity in artificial saliva. Oh and Kim<sup>46</sup> studied the galvanic corrosion of gold, NiCr, AgPd, CoCr, and Ti suprastructures in combination with Ti implants. That study revealed a significantly lower galvanic corrosion in the case of a couple consisting of a Ti abutment and Ti implant compared to the other systems.<sup>46</sup>

The effect of corrosion on the environment is also reported and may be visible *in vivo* when it is severe, and consequently a change of surface coloration or peri-implant inflammation caused by ion release can take place.<sup>17</sup> Guindy *et al*<sup>17</sup> reported the failure of six dental implant systems caused by corrosion of the metallic suprastructure. In that study, areas with clear signs of localized corrosion on implants and inner crown surfaces were detected by light and scanning electron microscopy on all six implants and inner crown surfaces. As result of a chemical reaction between metal and solution, metallic ions can be released to the surrounding environment at low levels for a long period.<sup>17</sup> Metallic ions released to the surrounding tissues can infiltrate into the tissue membranes and stimulate inflammatory cells.<sup>55,56</sup> Guindy *et al*<sup>17</sup> noticed higher contents of metal ions in bone tissue collected from retrieved implants compared to physiologic baseline values detected in healthy bones.

Concerning the role of saliva, Norton<sup>57</sup> revealed no influence of artificial saliva solution on abutment unscrewing; however, oral fluids can penetrate into microgaps, depositing microorganisms and glycoproteins in the implant-abutment internal connection. Souza *et al*<sup>11</sup> reported on the glycoproteins (e.g., mucin) and mixed (*S. mutans* and *C. albicans*) biofilms inducing ultralow friction on Ti surfaces. Biofilms act as lubricants in which polysaccharides, microorganisms, and glycoproteins (viscoelastic materials) distribute loads and decrease the friction on Ti. Ultralow friction on sliding contact areas might therefore cause a loss of the mechanical integrity of implant-abutment joints. On the other hand, biofilms produce acidic substances during metabolism of carbohydrates, promoting corrosion of structural materials.<sup>10</sup>

## Conclusions

Chemical analysis of abutment screw surfaces indicated that a corrosion layer can be formed between metallic surfaces at static contact in the implant-abutment joint during immersion

in artificial saliva solution. The corrosion can occur due to an electrochemical reaction taking place among implant, abutment, and artificial saliva compounds. Consequently, that reaction layer can be responsible for the increase of detorque values recorded on abutments after immersion in fluoridated artificial saliva solutions; however, different commercial implant joints reveal variations in topography, design, chemical composition, and mechanical properties of structural materials that influence the chemical and mechanical properties of the reaction layer formed along the inner joint. In addition, such characteristics of commercial implant systems determine the material transference on the abutment by wear and corrosion processes of implant-abutment assemblies under detorquing and performance of abutment screws. Under the experimental conditions of this work, some interesting aspects should be considered in further research such as: study of synergism between cyclic loading and corrosion of implant-abutment assemblies in the presence of corrosive substances or biofilms, effect of topography and chemical composition of abutment screws and internal connection of implant on detorque, and influence of biological fluids and biofilms on the mechanical integrity of commercial implant-abutment joints.

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