1 **Analysis of the ageing effect on the cyclic tension-compression loading behaviour of bitumen**
2 **and mastics**

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6 **Abstract**

7 Within the bituminous mixture the binder is subjected to different stress-state modes but the cyclic loading response is
8 usually characterized on shear mode. This research investigates the evolution with ageing of the undamaged linear
9 rheological behaviour and resistance to cyclic loading of bituminous materials using a tension-compression strain-
10 sweep test. The results show that, similarly to the shear mode response, mineral fillers may induce a greater stiffening
11 effect than ageing treatment, although the comparative evolution with ageing of materials may differ from that based on
12 the shear mode response. The damage progression rate increases faster at lower temperatures and for the stiffer
13 materials (mastics), where the flow deformation contribution to crack formation is smaller. The testing temperature is
14 determinant for the evaluation of the effect of ageing and filler on fatigue resistance.

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17 **Keywords:** Bituminous mastic; Filler-bitumen interaction; Ageing; Rheological properties; Fatigue testing; tension-
18 compression test.

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1. Introduction

Maintenance and rehabilitation of road and runway pavements involve periodical (about every 8 years [1]) replacement of the top bituminous layers when the level of distresses reach the point that it may compromise the whole pavement structure and/or the users serviceability in the short- to medium-term. Traffic loading, environmental action and ageing of bitumen are acknowledged as the main factors of the initiation and progression of distress [2].

The bitumen suffers various chemical changes, often called ageing, during the fabrication phase of bituminous mixtures and in the field. Most of these changes are caused by oxidation during the mixing procedure with the aggregates due to the high temperatures and air exposure. Other mechanisms, such as loss of the lighter components by evaporation and aggregate absorption, are considered less important. Oxidation continues in the field though at a much lower rate because the access to air is very limited with the exception of open surface courses or poorly compacted materials [2]. Additionally, ageing of the road surface is also caused by UV-radiation [3]. It is known that the chemical composition and proportion of functional groups of bitumen have a decisive influence over its resistance to these ageing mechanisms. However, the filler effect on bitumen ageing is still under debate in the scientific community. The bitumen interacts with the filler through physical and chemical mechanisms that can impact the ageing process of the bitumen. Thus, it was reported in [4,5] that the filler affects the evolution with ageing induced by oxidation or UV-radiation of the molecular size distribution of bitumen components. However, several studies have reported a beneficial effect of hydrated lime [6], which has not been acknowledged for mineral fillers [7,8].

Traditionally, the bitumen ageing index has been defined as the ratio of the viscosity values in the aged and unaged states [9]. However, ageing has consequences in all relevant properties, such as the stiffness, adhesion, cohesion, brittleness at lower temperature, ductility at intermediate temperature, elastic/plastic recovery at higher temperatures, etc., which can be more easily related to in-service pavement performance. Therefore, the characterization of the ageing effect on bitumen properties has been of interest to the scientific community for many years [8], and more recently attention has turned to the effect on mastic properties. Different studies [10–12] have investigated the evolution with ageing of the rheological behaviour of bitumen and mastics by means of dynamic shear rheometer (DSR) testing, and all have reported that the shear stiffness complex modulus increases to a greater extent in bitumen than in mastics. In addition, Moraes [4] found that this beneficial effect can also extend to the shear stiffness master curve slope change with ageing. However, the same author reported that though the glass-transition temperature of bitumen was reduced with filler incorporation the evolution trend with ageing was similar in bitumen and mastics.

Although most of the cited studies characterise the shear loading response of the bitumen and mastic, within the bituminous mixture the binder is subjected to different stress-state modes (shear, compression and tension). Thus, the

tension behaviour is especially important when a tensile stress is applied to the bituminous mixture because the aggregate interlock influence is rather limited [13]. From this, uniaxial tension-compression cyclic tests have been proposed to evaluate the cracking resistance of bitumen and mastics [14,15]. Miró et al. [16,17] showed the ability of this type of test to differentiate the fatigue behaviour of different bitumens, on various conditions of ageing and temperature, and to evaluate the effect of filler incorporation.

The study described in this paper investigated the effect of ageing on the rheological and mechanical behaviour of bitumen and mastics because it is accepted that asphalt pavements can be made more resistant to cracking by selecting proper bitumens and fillers that prevent premature and excessive ageing of bituminous mixture. To this end, the bituminous materials were subjected to the EBADE test, which consists in a strain-sweep cyclic tension-compression test applied to cylindrical specimens [14]. The bitumen and the mastics, fabricated with two different mineral fillers, were aged to different levels in the laboratory, and their loading response was evaluated at different test temperatures to provide a comprehensive analysis of the ageing effect.

2. Materials and experiments

2.1 Materials

In this study a paving grade bitumen, grade 50/70 [18], was used with a needle penetration at 25°C of $49 \cdot 10^{-1}$ mm and a softening point of 48.2 °C. Before, in [19], good ageing resistance was reported for this bitumen due to its balanced chemical composition. The bituminous mastics were fabricated with this bitumen and two different mineral fillers. The fillers (F1 and F2) were both obtained from grinding rock to dust. Filler F1 is a commercially available limestone filler and filler F2 is a baghouse filler collected from an asphalt plant working with granite aggregates. The fillers' main properties are indicated in Table 1. Filler F2 has a much lower absorbing capacity due to its larger Rigden voids. This means that for the same amount of filler added to a certain bitumen quantity, filler F2 increases the viscosity of bitumen more in comparison to filler F1.

2.2 Laboratory experiments

2.2.1 Fabrication of mastics

The mastics were fabricated with a filler volumetric concentration (30%) that represents average conditions of filler incorporation in conventional dense bituminous mixtures. The fillers were oven dried (150 °C) before the fabrication of

mastics. The (hot) filler was manually added to the bitumen at 150 °C under mechanical agitation at 400 rpm, and the agitation was continued for approximately 10 min after adding all filler to ensure a homogenous mixture at the end. After 24 h at room temperature, the mastic was heated, manually homogenised and then poured into small containers for testing (no ageing treatment) or onto plates for following ageing treatment.

2.2.2 Ageing treatment

The Pressure Ageing Vessel (PAV) (Model 9300, Prentex) was used to induce different levels of ageing in bitumen and mastics. The bituminous samples were conditioned for different periods of time (5 h and 25 h) in PAV, using the most typical conditions of film thickness, temperature and air pressure (3.2 mm, 100 °C and 2.1 MPa). The conditioning periods of 5 h and 25 h aimed to simulate the short- and long-term ageing conditions experienced by bitumen in the field, respectively. The reason for not implementing the Rolling Thin Film Oven Test (RTFOT) procedure [20] is that it is not recommended for high viscosity binders (or mastics). Thus, the equivalency between RTFOT and PAV reported by Migliori and Corté [21] was used. They concluded from the analysis of several paving grade bitumens that the ageing state of bitumens was similar when conditioned with RTFOT and PAV 5h, and with RTFOT+PAV 20h and PAV 25h.

2.2.2 Uniaxial strain sweep test (EBADE)

The EBADE test protocol consists of a cyclic test for the evaluation of the fatigue behaviour of bituminous materials. Cylindrical specimens with a diameter of 20 mm and 40 mm high are subjected to a cyclic compression-tension loading protocol in which the strain amplitude is periodically increased. Therefore, the test protocol induces failure in a significantly shorter period than if a constant strain amplitude (time-sweep) was maintained. This test protocol has been successfully implemented before with bitumens and mastics at different temperatures [14,16]. Hence, one of the advantages of this test protocol is that it allows testing at lower temperatures (< 15°C) than is often possible with the dynamic shear rheometer [22].

For the preparation of the cylindrical specimens, the bitumen/mastic was heated in an oven for 30-60 min at 145-165 °C (the time and temperature depending on the size and material of the sample) and then poured into the moulds (see Figure 1a). The moulds were covered with non-stick paper to allow easy extraction of specimens before testing. After cooling, the moulds with specimens were conditioned in a temperature-controlled chamber at the test temperature. Before testing, the top of the specimen was cut with a hot blade to obtain specimens of approximately 40 mm in height. The specimen was then assembled in the testing machine, gluing the specimen to the top and bottom plates to permit tension loading (see Figure 1b).

The initial strain amplitude was 0.076 %, and it was increased by the same amount (0.076 %) every 5000 cycles. The loading frequency was 10 Hz. Loading was stopped when the load registered was very low or the specimen broke (see Figure 1c and Figure 2a). At the lowest test temperature (2.5 °C), some specimens failed (brittle failure close to the plates) immediately after the increase of the applied strain during the first strain levels. This type of failure was more related to the test procedure than to the material's resistance to fatigue cracking. Therefore, the test procedure at 2.5 °C was then changed by reducing the increase in the strain rate during the first steps (the initial three steps were divided into six steps with 0.038 % increase every 2500 cycles).

The specimen behaviour was evaluated for the evolution of the stiffness modulus (E^*) and dissipated energy density (DED) (see Figure 2b). The behaviour exhibited during the first strain amplitude is usually stable and therefore it was used to characterize the rheological behaviour of the undamaged material (modulus E^*_{ini} , and phase angle ϕ_{ini}). The specimen's failure was defined at the point that the dissipated energy density had decreased by half of the peak value as recommended previously [14].

The EBADE test protocol was implemented with the bituminous materials conditioned at different temperatures (2.5 °C, 10 °C and 17.5 °C) to characterize their fatigue resistance in varied rheological conditions. A minimum of 2 specimens were used in testing for every material-temperature combination.

3. Results and discussion

3.1 Effect of ageing on initial stiffness modulus

As expected, the filler and the oxidative ageing, induced by the Pressure Ageing Vessel, had a significant effect in the rheological behaviour of the undamaged bituminous materials (see Table 2). The rheological behaviour of the undamaged material was characterized by the initial modulus (E^*_{ini}) and phase angle (ϕ_{ini}). The materials became stiffer and more elastic (reduction of phase angle) with both variables. The effect on the complex stiffness modulus caused by the filler was greater than that caused by the ageing treatment, and this trend was seen at the different test temperatures. On the other hand, the ageing treatment induced a greater reduction in phase angle than the filler. For the unaged state (PAV 0h), the E^* values of BF1 and BF2 were 2.68-2.82 and 2.97-3.18 times greater than that of B, respectively. And the effect of ageing (see Figure 3) on E^* value varied between 1.12 and 1.61. The E^* value ratio increased with the PAV conditioning time and the test temperature, although the bitumen was no more affected than the mastics. These results do not agree with the previous analysis of these materials [19], which showed smaller shear modulus (G^*) value ratios obtained at 20°C for the mastics (especially with BF2). However, it should be emphasised that the differences in E^* value ratios among the tested materials are not large (except for BF2-25h at 17.5 °C)

142 In the literature [23] several physical mechanisms have been postulated to explain the differences in behaviour of
143 bitumen in tension and shear. One of these mechanisms is the transient behaviour of the asphaltene network structure
144 with applied loading and temperature. All bitumen groups have less mobility and form stronger bonds with the
145 temperature reduction. In addition, it has been hypothesized [24] that the filler particles are surrounded by a bitumen
146 layer with less mobility (fixed layer), and these “filler-bitumen particles” can interact when the filler concentration is
147 above a certain level. It is therefore possible that with the stiffening of the bitumen medium induced by ageing
148 treatment there was an easier stress transfer between “filler-bitumen particles” upon tension-compression loading,
149 which resulted in high tension-compression stiffness values.

150 **3.2 Effect of ageing on cyclic loading behaviour**

151 Figures 4-6 show the evolution of the stiffness modulus and dissipated energy density with loading cycles in the
152 EBADE test for the bitumen and mastics. Each figure shows the results obtained at a single temperature (2.5 °C, 10 °C
153 and 17.5 °C). These test temperatures were selected to characterize the loading response of materials under varied
154 rheological conditions, implying different trends for damage evolution with accumulated cycles. The lowest
155 temperature conditions are close to the stiff and brittle behaviour, whereas the highest temperature approximates to the
156 soft viscoelastic and ductile behaviour. Hence, the intermediate temperature (10 °C) represents the loading response of a
157 viscoelastic material that should be less affected by excessive flow or fast damage growth.

158 Although the materials had significantly different rheological behaviours across the tested temperature range, in
159 general, the evolution trend in E^* and DED with loading cycles was similar in all tested materials and test temperatures.
160 The stiffness modulus had a small variation with loading cycles during the first strain amplitude which was then
161 followed by a continuous decrease at a nearly constant rate. The initially stiffer materials (aged) had a somewhat faster
162 decrease in modulus during second stage, which may correspond to faster damage progression. Moreover, the bitumen,
163 regardless of the ageing level, had a much slower decrease in modulus than the mastics.

164 Damage progression in the specimens is better perceived from the DED evolution graphs. Several mechanisms, other
165 than damage alone, contribute to the stiffness modulus reduction and the DED variation at constant strain amplitude.
166 Heating, thixotropy and non-linearity are important contributions to this change [14]. For the undamaged material, the
167 DED is proportional to the stiffness modulus, the phase angle and the strain amplitude, and the global trend (DED
168 increases with strain amplitude) confirmed this (see Figure 4b). However, for a constant applied strain amplitude
169 (intermediate level), the DED value showed an initial fast decrease with the loading cycles, which is mostly explained
170 by thixotropy and heating [25,26]. Thermal modelling of this test has shown that heating can account for 80-90% of the
171 reversible stiffness reduction observed in the test, leaving the rest 10-20% to other reversible phenomena such as

thixotropy [27]. Moreover, this effect was more pronounced in the DED curves obtained at 17.5 °C (see Figure 6b), where a greater thixotropy effect is expected in the material. Thixotropy in bitumen has been explained as resulting from the microstructural rearrangement due to the weaker bonds between chemical groups, and the temperature effect on the destructure-restructure mechanism has been confirmed by Mouillet et al. [26,28]. The decrease rate of DED after this initial drop is therefore indicative of damage progression, and in general the slope increased with the imposed strain amplitude and the initial stiffness of the material. Regardless of the material and ageing conditioning, close to the end of loading (before fracture) the DED slope was very steep, indeed it was almost vertical.

However, the mechanisms of damage were not the same at different temperatures. At 17.5 °C, the specimens' fracture progression was dominated by flow, with necking and tearing around the crack zone (see Figure 1c), whereas at 2.5 °C, the progression was dominated by microcracking and the deformation around the crack zone was not meaningful. Nevertheless, the 25 h aged bitumen was the only material showing brittle fracture, though it lasted more than 20,000 cycles. At 10 °C, the cracking zone showed an intermediate behaviour. As expected, the materials that resisted more loading cycles showed larger deformation around the crack zone because the ultimate strain level imposed was greater.

To complement the analysis of the ageing effect on cyclic loading behaviour, Figure 7 plots the results of the linear amplitude sweep (LAS) test performed previously with these materials (unaged and aged 25 h) at 25°C and reported in [19]. The LAS is a strain sweep test protocol implemented in the dynamic shear rheometer that was developed for the evaluation of the fatigue performance of binders [29]. Both test quantities (G^* and DED) had a similar variation with loading cycles to that which was observed in the EBADE test. However, the effect of ageing was considerably greater both in terms of initial stiffness (G^*) and of the DED evolution and ultimate deformation before failure. In addition, as the strain amplitude was linearly increased with loading cycles the thixotropy effect mentioned before was not observed.

3.3 Effect of ageing on fatigue resistance

The fatigue resistance in the EBADE test was determined from the applied strain at the failure point. The failure point was defined when the DED had decreased by half of the maximum DED during the test, and this point was always close to the specimen's complete fracture. Figure 8 plots the average ultimate strain value versus the PAV conditioning time at different test temperatures. There is a clear effect of the incorporation of the filler and the temperature on the fatigue resistance, although the ageing effect varied with the material and test temperature. It should be stressed, however, that the failure strain is a discrete variable (increase of 0.076% every 5000 cycles).

Regardless of the ageing state of the materials, the bitumen lasted more deformation and cycles than the mastics, though

the difference between bitumen and mastics reduced significantly with the increase of test temperature. These results can be explained by the lower stiffness and greater ductility of the bitumen. As suggested by different authors [24,30], a part of the bitumen is immobilized due to the interaction with the filler and therefore the ductility of the mastic is significantly reduced. In addition, as explained by Klüppel [31] in the analysis of cracking propagation in filled elastomers, the introduction of rigid particles leads to a hydrodynamic amplification of the macroscopic deformation within the elastomer matrix, i.e. local deformation is higher than external deformation. Hence, based on these physical mechanisms, there was a fast propagation of microcracking through the mastic specimens at the lower test temperature because the “free” bitumen was stiffer and less able to sustain deformation without cracking.

On the other hand, in the literature [32] it is suggested that the filler may increase the cracking toughness of bituminous mixture by the crack pinning mechanism. According to this theory proposed by Evans [33], solid particle inclusions in a brittle medium act as obstacles to crack propagation, forming secondary flaws, which results in an overall increase in strength. Thus, Khedoe et al. [34] reported the increase of the failure stress and strain in direct tensile tests at low temperature by adding filler to a very hard bitumen. From the reported results, the positive contribution of this mechanism might have been minimized because the bitumen’s behaviour was still ductile viscoelastic, not brittle, and/or the specimen was subjected to a deformation-controlled cyclic test with deformation (tension-compression) inversion that induces more complex microcracking progression.

In addition, it is possible that the results between bitumen and mastics came closer at the highest test temperature because of the flow deformation contribution to crack initiation and progression. The filler stiffening effect may have prevented excessive deformation and therefore counter-balanced the other mechanisms contributing to reduce fatigue resistance. It is therefore feasible that at higher temperatures the mastics would hold greater deformation than the bitumens.

Hence, performance-related testing of bituminous materials needs to be performed at different temperatures in order to consider the material’s unique rheological behaviour-temperature relation in the evaluation. Only in this way can the performance of the different materials be properly compared.

Surprisingly, the bitumen did not show signs of being affected by the ageing treatment, and for 25h PAV the fatigue resistance at 17.5 °C was even greater than that of less aged bitumens. It is considered that, as shown before for the LAS test results (see Figure 7), the specimen was able to hold larger strains because the increased stiffness of this bitumen prevented excessive flow deformation in the crack zone.

However, the effect of ageing on fatigue resistance of mastics did change with the test temperature. At the lowest test

temperature, the failure strains were close among different mastics (unaged, aged and with different fillers), and a specific trend is not observed for the filler type or ageing time. In contrast, at higher temperatures the ageing treatment had a significant effect on the mastic with granite filler. Compared to the unaged state, the failure strain of BF2 decreased 17% (10 °C) and 13% (17.5 °C) after PAV 25h conditioning. For the mastic BF1, the unaged and highly aged mastics held the same ultimate deformation at 10 °C and 17.5 °C.

In general, these results agree with the stiffness ratios shown in Figure 3. The mastic BF2 had a greater increase of stiffness with PAV treatment than BF1 and the bitumen, and the fatigue resistance of the specimens was therefore affected (reduction of ductility). The relation between these variables is confirmed by the chart shown in Figure 9, where the stiffness modulus is plotted versus the failure strain. Although the range of stiffness values for the bitumen is narrower (112-684 MPa) than of the mastics (316-1911 MPa), the lines representing the various materials, aged to different levels, tend to overlap, and are adequately fitted with a power-law model.

However, to demonstrate that the increased stiffening of BF2 with ageing is related with the stress transfer between “filler-bitumen particles” upon tension-compression loading which was already stiffer in the unaged condition, and not with the increased ageing of bitumen, in comparison with BF1, it is necessary to investigate the chemical changes in bitumen with ageing.

4. Conclusions

This study investigated the effect of ageing on the cyclic loading behaviour of bitumen and mastics. One paving grade bitumen and two mastics, using different mineral fillers, were aged to different levels (unaged, short-term and long-term) and subjected to a tension-compression strain-sweep test (EBADE) at different temperatures (2.5 °C, 10 °C, 17.5 °C) to characterize the undamaged rheological behaviour and the resistance to cyclic loading. Short- and long-term ageing was simulated by the Pressure Ageing Vessel (PAV), using different treatment periods (5 h and 25 h). The following conclusions can be drawn from the results obtained:

- Mineral fillers, with a moderate filler concentration, may induce a higher stiffening than ageing with PAV; the complex stiffness modulus (E^*) increased by 168-218% with filler (30% v/v mastic) and by only 12-61% with the effect of PAV treatment; the stiffening effect of filler incorporation and ageing treatment increases with the test temperature, which agrees with the results of the shear complex modulus found in the literature; contrary to previous results with these materials, the mastic with granite filler had a higher increase of stiffness than the mastic with limestone and the bitumen;
- The evolution of the stiffness modulus and dissipated energy density (DED) with the loading cycles in

EBADE is similar in shape regardless of the test temperature and characteristics of the materials; however, the specimen's failure is highly affected by these conditions; the EBADE test protocol is a deformation-controlled cyclic test that increases the strain periodically (5000 cycles), and from the analysis of the DED evolution at constant strain amplitude it could be observed that the damage progression increases with the applied strain; the damage progression rate increases faster at lower test temperatures and for the stiffer materials (mastics), where the flow deformation contribution to crack formation is smaller;

- The EBADE test can differentiate the fatigue resistance of different materials at different temperatures; thus, the test performance increases significantly with the test temperature because the material is more ductile; regardless of the ageing level, the fatigue resistance of bitumen was greater than that of the mastics, and it was not significantly affected by ageing, despite the increase in stiffness and phase angle reduction; the filler type and the ageing level do not have a meaningful effect on fatigue resistance at low temperatures; on the other hand, at higher temperatures, the mastic with granite filler had its fatigue performance significantly affected by ageing, which agrees with the results of the stiffness modulus.

Contrary to other studies found in the literature, the results of the EBADE test did not show that the mastics are less affected by ageing than the bitumen. The filler characteristics have a large impact on the physical and mechanical properties of mastics, and ageing of the bitumen is one of the variables that contributes to it. Tension-compression loading of bituminous materials is less common than other loading modes (shear, bending, indirect tension, etc.), but it complements the mechanical characterization of bituminous materials. In addition, the performance evaluation of different bituminous materials can only be made if tested at different temperatures.

Finally, the chemical composition of bitumen, and the evolution with ageing treatment, was not investigated, and this should be addressed in future.

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Table 1 – Filler properties

Filler		Particle density [Mg/m ³]	Gradation (% passing)			Rigden voids [%]	Absorbing capacity [g]
Code	Origin		#2.0 mm	#0.125 mm	#0.063 mm		
F1	Limestone	2.71	100.0	99.0	83.5	26	56
F2	Granite	2.57	100.0	93.1	81.5	42	37

Table 2 – EBADE test results (average values)

Temperature		2.5 °C		10 °C		17.5 °C	
	Ageing PAV	E* _{ini} [MPa]	ϕ _{ni} [°]	E* _{ini} [MPa]	ϕ _{ni} [°]	E* _{ini} [MPa]	ϕ _{ni} [°]
B	0h	557	23.1	281	36.7	112	48.4
	5h	623	22.5	305	33.0	135	43.7
	25	684	19.4	364	28.1	161	39.2
BF1	0h	1553	19.9	753	32.8	316	45.9
	5h	1613	18.4	878	28.7	394	40.8
	25	1763	16.0	934	25.9	450	37.4
BF2	0h	1655	19.7	853	32.1	356	45.6
	5h	1854	17.3	978	27.4	449	40.8
	25	1911	15.4	1139	24.4	574	35.1

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381 Figure 1. EBADE testing: a) specimens in mould; b) specimen mounted for testing; c) specimen failed at end of testing.

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383 Figure 2. Evolution of (a) stress amplitude and (b) stiffness modulus and dissipated energy density with loading cycles.

Figure 3. Variation of the stiffness modulus with ageing treatment.

Figure 4. Evolution of (a) stiffness modulus and (b) dissipated energy density with loading cycles at 2.5°C.

Figure 5. Evolution of (a) stiffness modulus and (b) dissipated energy density with loading cycles at 10°C.

Figure 6. Evolution of (a) stiffness modulus and (b) dissipated energy density with loading cycles at 17.5°C.

Figure 7. Evolution of (a) stiffness modulus and (b) dissipated energy density with loading cycles in LAS test at 25°C [19].

Figure 8. Fatigue resistance results (average failure strain).

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402 Figure 9. Stiffness modulus versus failure strain.

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