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Stabilization of $\beta$-carotene embedded in a silica matrix and study of its physical properties

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Abstract

This work reports on the behavior of $\beta$-carotene within a silica matrix prepared by the sol–gel method. Results show that highly pure $\beta$-carotene integrates with the silica to form a silica-based composite. The interaction exhibited between the inorganic matrix and the $\beta$-carotene results in a $\pi$-$d$ conjugated system that guarantees the development of the protective mechanism of the $\beta$-carotene and stabilizes the organic molecule, allowing its preservation at high calcination temperature. The spectroscopic analysis, made through fluorescence emission and infrared spectroscopy, shows the presence of $\beta$-carotene within the silica matrix after the composite was calcined at 1000 °C. Furthermore, the structural interactions of the $\beta$-carotene with the silica and the protective function of the $\beta$-carotene change the crystallization route naturally followed by the silica when exposed to increasing temperature. In this case, the amorphous silica matrix is preserved after high-temperature calcination, which is beneficial for biomedical applications as it allows the release and transport of the $\beta$-carotene to take full advantage of its health benefits.

1. Introduction

The $\beta$-carotene ($\beta$C) is a natural red-colored pigment consisted of eight isoprene units forming a conjugated double bond system [1, 2]. This molecule belongs to the carotenoids family who is extensively scattered in nature, being an essential component in some fruits, vegetables, and leaves, as well as in several animals, bacteria, and fungi. The carotenoids synthesized in plants have an essential role in photosynthesis contributing to light-harvesting and because of its protective function from photooxidative damage [3, 4]. These molecules absorb light between 400–500 nm, where chlorophylls fail as light absorbers. The $\beta$C is one of the most extensively investigated carotenoids due to its provitamin-A, antioxidant, and anticarcinogenic activities [5]. However, $\beta$C is prone to isomerization and oxidative degradation, leading to a color fading as well as a loss of health benefits [5–7]. The $\beta$C is sensitive to light, heat, and oxygen. Then, to take advantage of the $\beta$C exceptional biomedical properties needs developing advanced methods allowing its stabilization. In this sense, some theoretical and experimental works report alternatives to protect the $\beta$C from ambient degradation, among that, stand out the incorporation of the $\beta$-carotene inside of carbon nanotubes [8], or the stabilization of carotenoids species obtained directly from carrot juice [9]. Currently, no further studies are dealing with the thermal stabilization of the $\beta$C.

The $\beta$-carotene is composed of polyene chains with 22 carbon atoms regarding its $\pi$-electron structure [10]. This configuration allows the molecules to present a dramatic third-order optical nonlinearity upon the introduction of an electron-withdrawing group at one end of the polyene, which implies that the $\pi$-conjugated
molecules have great potential for future applications in photonics, as ultrafast processing information optical devices \[8, 11\]. The main problem for these novel applications is related to the \(βC\) degradation under ambient conditions, and because its trans-to-cis isomerization occurs under illumination or due to a temperature increase \[12, 13\]. Then, before to think using the \(βC\) in the design of technological devices or for biomedical applications, it is of great importance protecting the \(βC\) from thermal and ambient degradation. \[8, 9, 14\]. Also, the design of composites based on plant molecules together with an inorganic component requires the understanding of structural interactions manifested among the biomolecules and the matrix, as well as of its evolution under operation conditions. In this sense, silica (\(SiO_2\)) highlights as an alternative to encapsulating natural pigments and some biomolecules, although the close arrangement among the matrix and the embedded molecules can generate molecular interactions, the same which modify some of their physicochemical properties \[15\].

The sol-gel method is currently an adequate route to develop silica-based composites, the process based on hydrolysis and polycondensation reactions is of technological importance because of its simplicity and scalability. In this sense, a great variety of molecules can be suitably attached to an inorganic network using sol-gel \[16\]. However, a drawback of this method lies in the hydrolysis and condensation control, as well as in the reaction rate of the precursors \[17\]. Optimal conditions for the sol-gel reactions need to be determined experimentally in each case.

Previous works reported the study of extracts of green-leaves and carrots components embedded in a silica matrix \[9, 18\]. These results show that chlorophylls and carotenoids species can be stabilized within a silica matrix together with the organic components having in green plants and carrots. Once stabilized, the organic pigments support light exposure and high calcination temperatures; also, these molecules retain their main optical properties.

The incorporation of natural species in silica has a strong effect on the structural behavior because the mutual interactions control the silica crystallization, and allow to obtain exotic silica-phases or some of their isomorphic structures at ambient pressure and relative low calcination temperatures. In this sense, there are very few works studying the thermostability of organic molecules embedded in a silica matrix. We reported the stabilization of carotenoid species obtained directly from carrots when they were integrated into a silica matrix, prevalecting the \(βC\) even at high calcination temperatures \[9\]. Also, this organic multi-component system induces a silica crystallization above 800 °C from \(α\)-quartz to \(α\)- and \(β\)-cristobalites, with traces of \(β\)-tridymite and stishovite-type structure \[9\]. Some reports indicate that chlorophyll species could induce an unusual devitrification process in the silica at relatively low temperatures \[18, 19\].

Although \(βC\) stabilization from ambient conditions is possible inside of carbon nanotubes, its obtention could be difficult. Moreover, when \(βC\) stabilizes from carrot juice in a silica matrix, the organic stuff contained in the juice could modify the physicochemical behavior of the \(βC\). In both cases, these factors could restrict the potential applicability of the \(βC\). Then, stabilizing pure \(βC\) in a low-cost and straightforward way is highly necessary to use it in biomedical or photonics applications. Besides, the thermal stabilization of pure \(βC\) has not been properly studied when it forms a functional composite. Then, this work studied the thermal stabilization of high purity \(β\)-carotene after incorporated in a silica matrix, and its effect on the structural behavior of the matrix at high calcination temperatures. Besides, the optical properties associated with the \(βC\) were analyzed as a silica composite in various devitrification states. Our results suggest that \(βC\) avoids the silica crystallization preserving their amorphous states even though at high calcination temperatures. Amorphous materials are desirable in biomedical applications because they have a higher solubility than the crystalline structures and allows releasing embedded components in a controllable way \[20\].

2. Experimental

2.1. Fabrication silica composite with pure \(β\)-Carotene

High purity \(β\)-carotene were bought from Sigma Aldrich and used to fabricate the silica composites without any further purification. The \(βC\) was maintained at a temperature below of \(-2\) °C in dark conditions to avoid its degradation. The silica matrix was obtained using TEOS, tetraethyl orthosilicate (\(SiC_8H_{20}O_4\)), bought from Sigma Aldrich. Deionized water and ethanol (\(CH_2=CH_2-OH\)) also were used as initial chemical reagents.

One of the most critical variables during the sol-gel process is the molar ratio among water and the alkoxide because that controls the sol growth kinetics and its gelation. As the condensation and hydrolysis reactions take place in a mutual solvent, the ethanol to TEOS molar ratio used to form the silica composite was 4:1, and the water to TEOS ratio was 1:1. These relations were obtained experimentally to enhance the hydrolysis reaction and to assure an amorphous structure for the silica matrix. Then, according to the molar ratios, the catalyzing solution was prepared using 10.7 mL of TEOS dissolved in 10.2 mL of ethanol were subjected at constant stirring for 15 min at room temperature. The hydrolyzation initiates when 9 mL of deionized water was slowly dropped
to the ethanol-TEOS solution at a rate of 60 drops per minute. During the hydrolyzation reaction, 1 mg of \( \beta C \) maintained at \(-2^\circ C\) was rapidly added to participate in the condensation process. The methodology described above ensures that the \( \beta C \) participates in the condensation process to form a silica-based composite. Also, under these fabrication conditions, the degradation of the \( \beta C \) was avoided because it was quickly stabilized in contact with silica. The incorporation of pure \( \beta C \) accelerates the drying process obtaining soft pieces of the gel after 24 h. Then, the obtained gel was grounded to form a fine powder and after, it was heated 60 min in a Lindberg oven under atmospheric conditions at temperatures of 100 °C, 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C.

2.2. Characterization methods

The behavior of the crystalline structure of silica after incorporating \( \beta C \) as a function of the calcination temperature was analyzed with a Siemens D5000 diffractometer using a cobalt source (\( \lambda = 1.7890 \) Å). All the x-ray diffractograms were examined using the Rietveld refinement method implemented in the MAUD program [21]. Previously, the Rietveld refinement method proved to be useful to characterize amorphous compounds [22, 23]. Infrared spectra were obtained with a Bruker vertex 70 FTIR using diffuse reflectance mode. For all the samples, 0.05 g. of the silica composite was mixed with 0.3 g. of KBr to make measurable pellets. The emission spectra were obtained from a USB 4000 Ocean Optics fluorescence spectrometer using a reflectance diffused 45° configuration with a double fiber-optic. The excitation was done with an Omnicrome argon-ion laser of 532 nm and filter HNF-532-1.0. SEM micrographs were obtained with a JEOL FEI Inspect F50 scanning electron microscopy working with an accelerating voltage of 20 kV. All the measurements were done at room temperature.

3. Results and discussion

3.1. Stabilization of the \( \beta \)-Carotene in a silica matrix

Figure 1 shows the infrared spectrum (FT-IR) of the \( \beta C \), the \( \alpha \)-quartz, and the spectrum of the composite resulting when the \( \beta C \) was embedded in silica without any heat treatment, sample labeled as RT. The absorption bands of the \( \beta C \) are indicated in blue, while the bands of the \( \alpha \)-quartz are shown in red. The FT-IR spectrum of the silica reveals the silanol groups between 950–964 cm\(^{-1}\), which are the characteristic FT-IR bands of the \( \alpha \)-quartz. The predominant absorbance bands at 1077 cm\(^{-1}\) were assigned to Si–O–Si asymmetric stretching vibrations [24, 25]. On the other hand, the FT-IR spectrum of the \( \beta \)-carotene exhibits a narrow band at 1400 cm\(^{-1}\) associated with C–H stretching vibration mode, the bands observed between 1000–1200 cm\(^{-1}\) were attributed to the presence of the C–O and C=C–O stretching vibration modes. The bands between 960 and 1005 cm\(^{-1}\) were assigned to an out-of-plane deformation mode of the polyene chains with a \( \beta \)-polyene rings contribution giving an in-phase wagging vibration C=C–H and methyl groups. Table 1 shows the characteristic absorption bands of the \( \alpha \)-quartz and the \( \beta \)-carotene. All the contributions are in agreement with the vibration modes reported in the literature [9, 26–28]. The \( \beta C \) incorporated in a silica matrix was exposed several days to ambient conditions and after measured using infrared spectroscopy. The obtained FTIR spectrum exhibits the absorption bands associated with the \( \alpha \)-quartz and the \( \beta \)-carotene, except that the more significant silica bands hide away some minor contributions of the \( \beta C \) in the range of 1000 to 1200 cm\(^{-1}\), as the spectra of figure 1 show. Also, the contribution observed at 1385 cm\(^{-1}\) in the RT sample was identified as the \( \pi \)-d interaction between the pyrrole-type ring of the \( \beta C \) and the empty d orbital of the silica. This interaction is the main responsible for
stabilizing the conjugated double bond of the βC chains and assures its incorporation with the silica matrix [9, 29]. Then, resulting from a mutual interaction among the pure βC and the silica, the structure of the βCi is stabilized, avoiding its degradation under light exposure and ambient conditions.

### 3.2. Effect of the β-Carotene on the silica crystallization

Samples of silica matrix with embedded βC were subjected to different calcination temperatures from 100 °C to 1000 °C, the physical transition of the silica matrix was analyzed through the different vibrational modes of the composite. Figure 2 shows the effect of calcination temperature on the absorption bands of the silica matrix with embedded βC. There is observed that the βC is functionally preserved as part of a silica-based composite when the temperature increases, although the absorption bands of the silica show some changes associated with a gradual reconfiguration of the silica structure. The samples subjected to a heat-treatment between 100 °C and 400 °C exhibited small changes in the absorption bands of the Si-O and Si-OH bonds, indicating slight structural changes related to alterations in the atoms induced by the βC presence. At 600 °C, the silanol band is

| Wavenumber (cm⁻¹) | Vibration mode | Wavenumber (cm⁻¹) | Vibration mode |
|-------------------|----------------|-------------------|----------------|
| 480               | Si–O (rock.)   | 940               | C=C–H (wag.)   |
| 795               | Si–O–Si (sym. str.) | 967               | methylene CH₂ (rock.) |
| 810               | Si–O (str.)    | 1010–1170         | C–O, C–C–O (str.) |
| 865               | Si–OH (bend.)  | 1365              | C–H, –CH₃ (sym. Bend.) |
| 964               | Si–OH (sc. silanol groups) | 1400              | C–H (str.) |
| 1095              | Si–O (asym. str.) | 1425              | CH₃ and CH₂ of β-carotene |
| 1200              | Si–O (st.)     | 1523              | C=C (str.)     |
| 1637              | O–H (water molecule) | 1620              | H=C≡H (str. of β-carotene) |
|                   |                | 1670              | H=C=CH (str. of β-carotene) |

**Figure 2.** FT-IR spectra of the silica matrix with embedded β-carotene measured as a composite at different calcination temperatures.

**Table 1.** FT-IR absorption bands related to the α-quartz and the β-carotene.
incorporating towards the main band of the silica, and at 800 °C is thoroughly combined with the main silica band, while it is observed that a band at 1095 cm⁻¹ narrows due to the formation of a new amorphous-like compound. The changes observed in the spectra of figure 2 indicate that the silica suffers a structural accommodation as a result of the interaction of silanols groups with the βC. After sintering at 1000 °C for 1 hour, a significant fraction of βC remained functional, indicating the β-carotene thermal stabilization resulted from its interaction with the silica matrix.

Figure 3 shows the FT-IR spectrum of the silica with embedded βC after calcined at 1000 °C. In this figure, the band assigned to the βC at 1400 cm⁻¹ is very noticeable. Also, some other absorption bands were observed in this sample. The bands at 425, 470, 525, 710, 787, 1002, 1117 and 1210 cm⁻¹ are associated with a coesite-type structure [30]. Also, this spectrum shows unusually broad bands and some weak features observed over the main stretching bands, indicating the presence of small amounts of crystalline and amorphous-like structures. One of these can be a stishovite-like structure found in similar systems and obtained as a result of the strong tensions generated by the presence of the organic components in the silica matrix [31]. Besides, one of the most significative differences regarding the results obtained using the carrot juice is the apparition of contributions related to the coesite and its coexistence with β-tridymite and β-cristobalite. The above results indicate that the interaction between the silica and the organic components influences the crystallization form of the silica.

Figure 4 shows the experimental x-ray diffractograms of the silica composite with embedded βC at RT, and the ones subjected to a heat treatment at 200 °C, 400 °C, 600 °C, 800 °C, and 1000 °C. The experimental diffractograms are presented with their corresponding Rietveld refinement profile obtained from MAUD, which is a useful program in the quantification of amorphous and crystalline compounds [32, 33]. The phases used to create the initial model were: (i) α-quartz (COD-ID: 1011 097), with trigonal symmetry, P3121 space group, lattice parameters a = 4.913 Å, c = 5.404 Å, and density of 2.65 g cm⁻³; (ii) β-tridymite (COD-ID: 5910147), having an hexagonal symmetry, P6/ mmc space group, lattice parameters a = 5.03 Å, c = 8.22 Å, and density of 2.25 g cm⁻³; (iii) β-cristobalite (COD-ID: 1010921) with cubic symmetry, P213 space group, lattice parameter a = 6.94 Å and density of 2.27 g cm⁻³; and (iv) coesite (COD-ID: 9008083) with monoclinic symmetry, C12/c b1 space group, lattice parameters a = 7.135 Å, b = 12.369 Å, c = 7.173 Å, β = 120.34°, and density of 3.01 g cm⁻³. Notice that x-ray experimental diffractograms were obtained with a cobalt source; therefore, the bands shift to higher 2-theta values regarding the diffractograms obtained with a copper source.

The sample RT shows a broad diffraction band centered on 30.4° related to the α-quartz, the band is formed by two reflections, one minor at 24.2° (100) and a major at 31.04° (011). Results show that α-quartz has a density of 2.66 g cm⁻³, lattice parameters a = 4.795 Å and c = 5.649 Å. These values are close to the ones reported for this compound. Then, the x-ray patterns indicate that the characteristics associated with the α-quartz kept at calcination temperatures of 400 °C. After, at 600 °C is observed a broadening and a shift of the x-ray band to low 2-theta angles. Many authors relate this behavior with a structural change that takes place in the silica network [34–37]. In this case, the shift of the x-ray band results from the formation of an amorphous β-tridymite-type structure having reflections at 23.69° (100), 25.14° (002) and 26.87° (101), while the band broadening is due that β-tridymite coexists with the α-quartz. Although both phases have amorphous structures, they also have particular reflections allowing its identification and the evaluation of their structural features using the Rietveld method. At 800 °C, the x-ray diffractogram shows a vanishing of the contribution associated with the α-quartz, being the β-tridymite the majority phase. However, as the spectroscopic analysis showed in figure 2, the β-carotene was stabilized with the silica, this factor favored the formation of an amorphous coesite-like structure, having mainly reflections at 23.59° (021), 30.26° (111), and 33.59° (002). Although at 800 °C, the β-tridymite and
coesite do not reach crystalline arrangements, as they exhibit amorphous-like structures, they also show an increase in their crystallite sizes, indicating an increase on the crystallinity state of the silica phases. The presence of coesite at high calcination temperatures agrees with results obtained from the infrared analysis.

The presence of a coesite-like structure at ambient pressure can result from the interaction of the silica with the $\beta$C, which induces strong structural stress at a molecular level modifying the crystallization behavior of the silica. In this sense, previous work reports that the incorporation of organic species can guide the crystallization route of the silica during the heat-treatment [9, 15, 18, 19]. Finally, the sample heat-treated at 1000 °C shows an experimental x-ray pattern with a sharpened band centered at 25.5°. This behavior is consistent with an increase in

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**Figure 4.** X-ray diffractograms and Rietveld refinement profile of the silica with embedded $\beta$C subjected to different calcination temperatures.
the crystallinity of the phases at increasing the calcination temperature. According to the Rietveld analysis and the infrared spectrum, this sample contains β-tridymite, coesite, and β-cristobalite as a minor phase, but all of them as amorphous type-structures. Then, the embedded pure β-carotene avoids the silica crystallization, even though at high temperatures. These results contrast with the obtained by the silica with embedded carrot juice, in which crystallization occurs mainly in β-cristobalite at 900 °C [9]. Table 2 lists the structural parameters together with a quantitative phase analysis obtained from the Rietveld refinement method for all the samples. The crystallite size is related to the crystallinity degree of the silica; in this sense, low values are associated with highly amorphous silica, while high values indicate an increase in silica ordering. Then, it was observed an increment in the silica crystallization as the calcination temperature increases; however, the phases do not reach a full-crystalline state.

Then, results obtained from x-ray diffraction experiments and the Rietveld refinement match with the analysis derived from the spectroscopic techniques, indicating that the embedded βC exerts an enormous influence on the crystallization of the silica, probably the micro tensions generated among the βC and the silica avoids its full ordering. These results also differ entirely from the ones obtained since the silica crystallization using other types of organic molecules, such as chlorophylls. In this case, the βC promoted the formation of amorphous-type structures.

### 3.3. Morphological behavior of the silica with embedded β-Carotene

Figure 4 shows the SEM micrographs of the silica with embedded β-carotene, the sample without calcination treatment is presented in figure 5 (a), while figures 5 (b) and (c) show the samples subjected to a heat-treatment at 600 °C and 1000 °C, respectively. The sample without heat-treatment is recognized by a porous pattern conformed by particles smaller than 100 nm diameter, as the insert of figure 5(a) shows. In this sense, nanometric particle agglomeration forms the porous structures observed in the RT sample. On the other hand, both the porous structure and the nanometric particle size lost when the silica composite is subjected to high calcination temperatures. After 600 °C, the particles adopted sharped forms with an irregular surface, and the volume of particles increased significantly up to reach several micrometers per length. Finally, when the sample was heat-treated at 1000 °C for 1 hour, the particles become rounded and spherical with uniform size, as the micrograph of figure 5(c) shows. These morphological characteristics are typical in some silica polymorphs, such as in the like-stishovite structure [38, 39].

### 3.4. Thermostabilization of the β-carotene

Figure 6 shows the emission spectra of the βC embedded in a silica matrix as a composite at different calcination temperatures from RT to 1000 °C. The fluorescence emission is attributable to the organic component stabilized.
in the inorganic matrix, and this stabilization allows the \( \beta C \) to keep its photoprotective function. The fluorescence emission has been assigned to carotenoids because they are fluorescent in the green-yellow spectral region between 550–600 nm [40]. Figure 6 (a) shows an increase in the fluorescence emission in the samples at RT and the one calcined at 200 °C, where the fluorescence emission reaches its maximum value. Next, in figure 6(b) is observed a decreasing in the fluorescence emission when calcination temperature goes from 200 to 800 °C. At this point, the fluorescence reaches its minimum value. Figure 6(c) shows an increment in the fluorescence emission that occurred when the sample was calcined from 800 °C to 1000 °C. Finally, figure 6(d) shows that the fluorescence emission intensities of the samples at RT and the one calcinated at 1000 °C are very similar, corroborating the prevalence of the \( \beta \)-carotene as a functional component of the composite.

The fluorescence intensity of the \( \beta C \) embedded in silica shows an increasing-decreasing behavior, which is typical in carotenes and chlorophyll with different light-harvesting complexes (LHC) in a physiological and non-physiological regime. The maximum fluorescence intensity of green leaves is reached at 60 °C, and after they were embedded in a silica matrix, the maximum was reached at 70 °C; next, its fluorescence quenching at temperatures above 70 °C [15, 41]. In contrast, the maximum fluorescence intensity of pure \( \beta C \) embedded in a silica matrix was reached at 200 °C, as figure 7 shows. The increment in the fluorescence intensity is an indicator of thermostability in the photosystem II [42], and the non-photochemical quenching, such as the high-energy-state quenching is activated in the formation of the silica composites.

The interaction between inorganic matrix and organic molecules in a composite forming \( \pi \)-d conjugated assures the establishment of a protective mechanism in which is induced a non-photochemical fluorescence quenching to dissipate the excess of excitation energy. In the case of the \( \beta C \), the interaction with silica is strong, as can be shown in the infrared results. Thus, the decomposition of pure \( \beta C \) that is evident at low temperature is preserved at higher ones and shows a remarkable thermostability when it is embedded in a silica matrix. Also, the fluorescence rise observed above 800 °C coincides with the structural and morphological changes observed in the silica composite, indicating a close relationship among the structural properties of the silica with the
behavior of the \( \beta \)-carotene embedded therein, and the strong effect that the \( \beta \)-carotene causes on the crystallization route of the silica.

The results show that \( \beta \)-carotene induces structural stress on the silica matrix avoiding its crystallization at increasing the temperature. Reciprocally, the \( \beta \)-carotene chains are subjected to molecular stress, increasing its fluorescence emission as a result of its protective function \[41, 42\]. However, after 200 °C, the system relaxes, and fluorescence emission reduces. In this sense, the relationship between the structure and physical properties needs to be studied using first principle calculations \[43\].

4. Conclusion

The positive effects of \( \beta \)-carotene on human health are well known. However, its rapid degradation under ambient conditions does not allow proper absorption and its widespread use is far from being optimal. Our results show that pure \( \beta \)-carotene can be stabilized to endure ambient conditions retaining its functional properties even at high temperatures and long-term exposition to the environment. The stabilization of \( \beta \)-carotene comes as a result of embedding the material into a silica matrix. These two materials interact and form a \( \pi \)-d conjugated system.

Figure 6. Fluorescence spectra for the silica composite with embedded \( \beta \)-carotene at different calcination temperatures showing (a) an increase in the fluorescence as increasing temperature from RT to 400 °C, (b) a diminishing the fluorescence emission between 400 °C to 800 °C, (c) a rise fluorescence intensity from 800 °C to 1000 °C, and (d) comparison between the fluorescence emission of the sample at RT and the one calcined at 1000 °C.

Figure 7. Fluorescence emission behavior of the silica composite with embedded \( \beta \)-carotene as a function of the calcination temperature.
where the silica coating protects the βC while the βC controls the phase formation of the silica at increasing temperature and avoids its crystallization. We note that the obtained results are of particular interest for biomedical applications as the amorphous compounds can be easily dissolved to release the β-carotene, this last property is exclusive to the amorphous phase and it is not possible in the case that silica forms crystalline structures.

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