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The effect of fixed and rotary roasting methods on the oxidative stability of pistachio oil

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ABSTRACT

Today, the demand for vegetable oils has increased. Unsaturated fatty acids in pistachio oil are an important component in promoting the health of the human diet. The purpose of this study was to evaluate the oxidative properties of roasted pistachio oil in two fixed and rotary roasters at 160 °C for 15 min during 0, 3, and 6 months of storage. The heat used has led to the destruction of the oil in the roasted pistachios, and with the increase in the storage time, the peroxide value of the samples increased from 0.06 to 0.57 meq/kg in the fixed method and from 0.03 to 0.07 meq/kg in the rotating method, respectively. Free fatty acids and acid value increased from 0.34 to 1.34 % and 0.69 to 2.68 mg KOH/g in the fixed method and from 0.3 to 1.11 % and 0.6 to 2.20 mg KOH/g in the rotating method as a result of storage time for 6 months and lipolysis reactions. Under the effect of roasting heat, the Maillard reaction products played an important role in the thermal stability of the oil ($P<0.05$). The roasted samples at zero storage time have the highest inhibitory percentage of DPPH and the lowest IC50 (3.66 % in the fixed method and 6.9 % in the rotary method). In spite of the fact that the changes made during 6 months of storage in both roasting methods are within the standard range, but due to the fact that in the samples of roasted pistachios in the fixed method, the peroxide value increased and the oxidative stability decreased significantly, it is suggested to use the rotary method in roasting of pistachios.

1. Introduction

Pistachio is intended to be a pragmatic food due to its unsaturated fatty acids, vitamins, minerals, sterols and polyphenols. Raw pistachio kernels contain 45.3% lipid (53% monounsaturated fatty acids, 33% polyunsaturated fatty acids and, 13% total saturated fatty acids) (Pickford et al., 2022) and are situated in the first row of 50 types of food products with the highest antioxidant potential (Hojjati et al., 2013). Pistachios have a positive effect on blood lipid profiles (such as oleic acid, phytosterols, phenolics and tocopherols), which have high antioxidant anti-inflammatory properties and reduce the risk of cardiovascular disease)(Salvador et al., 2020). Nutrients, including polyphenols, carotenoids, and C and E vitamins, are considered antioxidants effective in preventing oxidative reactions, and related diseases (Shakerardekani et al., 2013).

Unsaturated fats reduce heart disease, diabetes and cholesterol. However, more saturated fats are exposed to rancidity. According to exploratory clinical studies, pistachio can control body weight, reduce blood cholesterol, provide healthy antioxidants, maintain anti-inflammatory activity and control glycemic levels (Liu et al., 2014). Also, Tomaino et al. (2010) studied the antioxidant activity and phenolic properties of pistachios and found that the presence of pistachios in the daily diet is effective in maintaining human health without any problems. pistachios are sold in both fresh or salted and roasted forms. Roasting is one of the most common forms of dry processing of pistachio kernels, and its aim is increase attractiveness for consumers. Due to the creation of pleasant odors and colors, this process significantly improves the taste, texture and appearance of pistachio kernels (Sheikhshoaei et al., 2020) Roasting affects pistachio chemical compounds like lipids and amino acids, and increases free fatty acids. During the Maillard reaction, the destruction of lipids increases, and the desired pigment and aroma compounds are formed (Kahyaoglu & Kaya, 2006).

In addition, roasting makes pistachio kernels last longer by eliminating undesirable infected microorganisms. Also, roasting leads to a significant reduction in the antioxidant activity of hazelnuts and walnuts (Ojeda-Amador et al., 2018) while in almonds and pistachios the antioxidant activity remains stable or even slightly increases. We can explain this effect by the loss of polyphenols due to heat treatments,

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which are created by the formation of active antioxidant compounds due to the Millard reaction. In addition, it should be noted that most pistachio antioxidant compounds, such as polyphenols, are located in the skin and their elimination is associated with the loss of antioxidant activity. Therefore, roasted pistachios in suitable conditions show excellent phenolic compounds and antioxidant activities and are recommended for daily consumption (Chang et al., 2016). Of course, it should be noted that roasting, while improving the overall acceptability of the product, reduces its nutritional value. Various studies have been performed on dried pistachio kernels roasting. These studies include the study of changes in pistachio fat oxidation reactions (Tavakolipour et al., 2009) evaluation of oxidation resistance and prediction of the storage of pistachio oil (Dini et al., 2016). With various commercial pistachios, the storage time of the Ahmad Aghaei cultivar was 1.5 times more than Fandoghi, Akbari and Kaleghoochi varieties (Dini et al., 2016). In this work, two fixed and rotating roasters were used for roasting pistachio nuts at 160 °C for 15 min and the oxidative stability of oils (peroxide value, free fatty acids, acid value, thermal resistance, and DPPH inhibitory percentage) during storage time (0, 3 and 6 months) was evaluated at room temperature (20± °C).

2. Materials and methods

2.1. Preparation of raw materials and sample

First, some Fandoghi pistachio varieties were dried in the sunlight for two days in the Jalalabad pistachio processing unit of Sirjan City (Kerman province, Iran) after passing the washing, peeling and grading stages, and then transferred to the roasting unit for roasting. First, some pistachios in the raw sample were roasted in a roasting machine (Tabrizkar Co., Iran) at 160 °C for 15 min, and 4 kg was sampled for evaluation, which is considered a rotary roasting method of study, and 4 kg separated from the raw samples for roasting in the oven and roasted in a laboratory oven (400 UNB Memmert model, Germany) at 160 °C for 15 min, which is considered as the fixed roasting method of study. After roasting all pistachios, they were cooled to room temperature and the first 0-time tests of the samples was carried out. The rest of the pistachios were stored at room temperature and in the dark place until further experiments at 3- and 6-months' storage time.

2.2. Oil extraction

400 g of each sample was soaked in 1000 ml of n-hexane for 24 h and filtered using a Buchner funnel. The obtained oils were again passed through a 0.045-micron nylon filter. It was then evaporated by a vacuum rotary evaporator (Rotavapor model 461, Büchi, Flawilat) 40 °C for 20 min and was kept at −18 °C until the experiments were performed.

2.3. Determination of peroxide value by iron oxidation method

The organic oxidation method of xylenol orange is based on the ability of lipid peroxides to oxidize Iron ions at low pH, which was performed according to the method stated by Wrolstad (2001). According to the absorption values (560 nm) using a spectrophotometer (model 2200-EU, read from the following formula, we calculated and reported the amount of peroxide value of the samples:

$$\mathrm{PV} = \frac{(A - B) \times M}{W \times\ 55.24\ \times\ 2}$$

Whereas A= read absorption, B= blank absorption, M= standard line slop, W= oil sample weight

2.4. Measurement of free fatty acid and acid value

This method reflects the amount of free acid groups in the oil, as well as the acidity (acid value) of the level of free fatty acids in the oil that do not bind to the Glycerol. To measure free hydroxides and acid value, the method described in the book by Wrolstad (2001) was used. The amount of free fatty acids and acid value was reported according to the following formulas.

$$\text{FFA as oleic acid} = \frac{ml\ NaOH \times NaOH\ normality\ \times\ 28.2}{weight\ of\ sample(gr)}$$

$$\text{Acid value (oleic acid)} = \text{FFA}\ \times\ 1.99$$

2.5. Measurement of oxidation stability (rancidity)

To measure the thermal resistance of the samples a rancimat device used at a temperature of 110 °C and the amount of 2.5 gs (Ampofo et al., 2022). The rancimat method measures the secondary products resulting from the oxidation of oils and fats (including Aldehydes, ketones and acids) and is a method based on which the oxidative stability of oils can be predicted. Oxidative stability was determined by Rancimat (Metrohm model 743). The trading device automatically reported the calculation of induction time-oxidative stability in hours with an approximation of 1 hour.

2.6. Measurement of percentage of inhibition (DPPH)

A stable free radical called diphenylpicrylhydrazyl (DPPH) was used to determine the antioxidant power of compounds (Tomaino et al., 2010). In order to measure the antioxidant activity of the samples, 2 ml of 100 micromolar DPPH dissolved in methanol mixed with 2 ml of 5, 10, 20 and 40 ppm oil concentrations. the resulting mixture placed at room temperature for 30 min, and then the samples absorbtion was measured at 520 nm by a spectrophotometer (model 2200-EU, China) and the amount of antioxidant activity by was reported using the following formula.

$$\mathrm{DPPH} = \frac{B - A}{B}$$

Whereas A= sample absorption, B= absorption of control samples

The blank sample contains a mixture of 2 ml methanol and 2 ml oil with used concentrations and a sample contain 2 ml of DPPH and 2 ml of oil with the used concentrations considered as a negative control. Also the IC_{50} was calculated after measuring the percentage of oils inhibitory. In this way, the graph of inhibition percentages was drawn in different concentrations and the concentration with half the inhibition was reported as the IC_{50} value of each oil sample. IC_{50} is defined as the half-life or concentration that has half of the inhibitory rate. Therefore, the sample with the lowest IC_{50} will have the highest stability because the lowest concentration has had the highest inhibition.

3. Results and discussion

3.1. Peroxide value of roasted pistachios

Peroxide value determines the stability of oil against oxidative degradation. It is widely used to measure peroxides and forms of hydroperoxide due to oxidation in oils (Suri et al., 2020). Based on the results (Table 1), the peroxide value of samples roasted in the fixed and rotary methods has increased significantly during the storage time. The peroxide value of roasted pistachio in rotary method (0.07 meq/kg) was lower than fixed method (0.57meq/kg) after 6 months storage. The maximum permissible amount of peroxide value in processed pistachios according to ISIRI (2016) is equal to 1 meq/kg. According to the data obtained from all samples, the peroxide values are within the standard range.

Nikzadeh and Sedaghat (2008) reported that by increasing the roasting temperature from 90 to 150°C, the peroxide value increases

Table 1
The effect of roasting method and storage time on peroxide value, free fatty acids, acid value, and oxidative stability of roasted pistachio.

sample	peroxide value (meq/kg)	free fatty acids (%)	acid value (mg KOH/g)	oxidative stability (hr)
Fixed roaster (0 month)	0.06± 0.00 b*	0.34± 0.06 c	0.69± 0.12 c	14.6±1.4 e
Fixed roaster (3 months)	0.07± 0.00 b	0.82± 0.10 b	1.63± 0.21 b	23.7±5.3 d
Fixed roaster (6 months)	0.57± 0.00 a	1.34± 0.11 a	2.68± 0.22 a	42.7±1.2 b
Rotary roaster (0 month)	0.03± 0.00 d	0.30± 0.05 c	0.60± 0.11 c	23.4±2.1 d
Rotary roaster (3 months)	0.05± 0.00 c	0.70± 0.12 b	1.39± 0.25 b	32.8±2.2 c
Rotary roaster (6 months)	0.07± 0.00 b	1.11± 0.06 a	2.20± 0.13 a	47.5±1.4 a

* Values with unmatched letters in each column have a significant difference at the 5 % level.

significantly. Therefore, roasting the samples of this research at the temperature of 160°C and staying at this temperature for 15 min, leads to an increase in the peroxide value. Kashani and Valadon (1983) declared that the effect of storage time on the peroxide value is significant and stated that with increasing the storage time, the peroxide value increases significantly. The increase in the peroxide value after roasting indicates some damage to the oil. Kashani and Valadon (1983) after roasting pistachio at 145 °C for 30 min, showed that salting and roasting had a significant effect on the peroxide value of lipids of pistachio kernels. Also, in relation to other nut seeds, It has also been stated that roasting in the microwave has also led to an increase in the peroxide value (Ling et al., 2016). Similar to this research, increasing the peroxide value of hazelnuts and soybeans has shown a sharp upward trend in storage temperature (KhoshnoudiNia & Sedaghat, 2019).

3.2. Free fatty acid and acid value of roasted pistachios

The acid value acts as an indicator of the oil quality (Tenyang et al., 2017). As can be seen in Table 1, the acid value has increased over time. The samples of roasted pistachios in the two devices are not significantly different after 0, 3 and 6 months of storage. Also, the interaction effect of the roasting method and storage time on the acid value doesn't show a significant change ($P = 0.23$). Similarly, in other studies, an increase has been reported in the amount of acid value in peppermint and poppy seed oil by roasting in the microwave (Ghafoor et al., 2019; Özcan et al., 2019). Increasing the acidity of the oil is the hydrolytic degradation of triglycerides and the formation of free radicals during roasting at high temperatures (Suri et al., 2020).

The results shows that by roasting pistachios, free fatty acids ranged from 0.3 to 1.34% (in terms of oleic acid). The percentage of free fatty acid of the samples roasted in rotary machine was not significantly different from the sample roasted in the fixed machine. The percentage of free fatty acids gradually increased significantly during the storage of pistachios for 6-months in all samples. The highest amount of free fatty acids (1.34 % and 1.1 %) is related to the samples roasted in the both fixed and rotary machines after 6 months of storage, respectively.

Since the free fatty acid is less stable than the neutral oil, it is more susceptible to oxidation and spoilage; Therefore, the amount of free fatty acids is an essential characteristic related to the quality and commercial value of oils and fats .Considering the maximum free fatty acid which was observed in fixed machine (6 months storage), it can be concluded that because this amount is less than 5 % (maximum free fatty acid for human consumption); therefore, the quality of roasted pistachio is well maintained during 6 months of storage. An increase in free fatty acids amount expresses hydrolysis phenomenon in pistachio oil. Lipolytic enzymes located just below the thin grain skin will not be able to attack fats in undamaged cells. Initially, in the unroasted samples that are not heated, the enzyme activity is low, the cells are not damaged, and as a result, the percentage of free fatty acids is low. However, with the onset of thermal process at high temperatures, physical changes occur in the cell. As a result, cell damage increases and a significant increase observed in free fatty acids of roasted pistachios at 160 °C. Also, the results obtained by Nikzadeh and Sedaghat (2008) show that in the thermal process with high temperature, the free fatty acids amount released is higher and during storage time this amount will increase over time and this increase is significant. Other results obtained by Mokhtarian et al. (2021) show that during storage time at a constant temperature, with increasing relative humidity, the percentage of free fatty acids in the complex increases, and on the other hand, at a constant relative humidity and higher temperature, the percentage of free fatty acids increases, which indicates that during the storage, the temperature and relative humidity have an auxiliary effect on the increase of lipolysis reaction (Tapia et al., 2020). These changes in other nuts are similar, as Özdemir et al. (2001) stated that roasting is significantly effective on hazelnut free fatty acids, so that with increasing the roasting degree, the amount of free fatty acid in the samples increases. An et al. (2022) examined the increase in free fatty acids in pine nut oil, and found that overtime during storage, the amount of free fatty acid increased.

3.3. Oxidation stability of roasted pistachios

Oxidation stability indicates the sensitivity of the oil to oxidation, which mainly depends on saturation degree and the antioxidant compounds level in the oil. Roasting leads to the start of fat oxidation and the formation of carbonyl compounds, but on the other hand, this process, due to the antioxidant effect of the maillard reaction products, leads to an increase in the stability of seed oils against oxidation during storage (Suri et al., 2020). Study of the effect of oxidative stability of a roasted samples in two fixed and rotary machines shows that the oxidative stability has increased significantly over a period of 6 months' time. Oxidation stability of the samples roasted in the fixed and rotary machine after roasting were respectively, 14.6 and 23.4 h, which were significantly different from each other. Oxidation stability of the roasted samples in the rotary machine was higher. During the 3 months of storage due to the increase in antioxidant activity, the oxidative stability in both roasting methods of samples increased significantly. Also, there has been a significant increase (47.5 hr) in the oxidation stability of rotary method compared to the fixed method (42.7 hr) after 6-months storage time of the roasted pistachios.

Oxidation resistance is one of the important parameters in quality evaluation of oil, which is affected by fatty acids and bioactive components of the oil. Dini et al. (2023) reported that among different pistachio varieties, the oxidation resistance of Kaleghoochi, Fandoghi, Akbari, and Ahmed Aghaei oil was respectively 12. 68, 12.95, 12.24, 14.75, hours, at 110 °C that is less stable for Kaleghoochi (15–16 h) and Akbari varieties (14–16.4 h). Shakerardekani and Yahyazadeh (2020) evaluated the rancimat thermal stability of whole wild pistachio oil, and declared that there was no significant difference in 3 types of Iranian wild pistachios, and the oxidation stability ranged from 17 to 18 h. Other studies have reported that Millard reaction products are useful in increasing the antioxidant activity of seed oils and play an important role in improving oxidative stability (Cai et al., 2021; Nounah et al., 2021). It's expected that the products from Millard reaction increase the oxidative stability of the oil by ending the fat oxidation reactions. The results clearly show that roasting the seeds at the right level can increase the oxidation stability of nut oil through the production of Maillard reaction products. Our current results show that roasted pistachio oil has more oxidation stability than raw oil and oxidation stability can be related to temperature and time of roasting. Also, oils from roasted nuts and kernels are more resistant to oxidative degradation than raw samples (An et al., 2022; Tu et al., 2021). But the suitable heat process should apply to the nuts. The formation of Maillard reaction products can play an important role in increasing the activity of free radicals and

thermal stability of oil (Cai et al., 2021). These results are consistent with previous findings regarding sesame oil, which show greater oxidative stability of roasted sesame samples at higher temperatures (Arab et al., 2022). Data analysis of current study also showed that the oxidation stability of roasted pistachio oil increased during storage. These results are similar with the results of Arab et al. (2022). They reported that the induction time significantly affected by packaging conditions, temperature and the storage time. The induction time in the ninth month storage time decreased significantly compared to other times. Other studies in different oils showed similar results with this study. Aleena et al. (2020) and Lee et al. (2004) studies also confirmed that roasting heat, increases the oxidative stability of sunflower oils. Durmaz et al. (2010) reported that the oxidative stability and antioxidant capacity of apricot kernel oil increased with roasting, due to the formation of antioxidative Maillard reaction products during the roasting. Yang et al. (2018) stated that increasing the roasting temperature could increase the antioxidant stability of Maillard reaction products in camellia seed oil.

3.4. Antioxidant capacity (DPPH) of roasted pistachios

According to results (Table 2), the inhibitory percentage of oils from roasted samples in different concentrations has increased significantly for both roasting methods. With increasing concentrations of alpha and alpha dipicryl hydrazine, the inhibitory percentage of oils, has increased during the 6-months of storage. Among the roasted samples and the storage time (Fig. 1), the samples roasted in the rotary machine in 0 time had the lowest level of IC50 and the highest percentage of inhibition, while the samples roasted in the rotary machine in 6-months' time had a maximum level of IC50, which has the lowest percentage of inhibition. In addition, in relation to the samples roasted in both fixed and rotary machines, the lowest amount of IC50 is observed for the samples roasted at 0 time and the highest amount of IC50 is observed for the samples roasted at 6 months' time.

A significant result of roasting is the increase in antioxidant activity, mainly due to the formation of Maillard reaction products. Maillard reaction in fatty seeds occurs by the interactional of carbonyls with amines during the thermal process (Shakoor et al., 2022). Roasting may eliminate some bioactive compounds, but they can form antioxidant compounds by forming of Maillard reaction products. So, with increasing the content of tocopherols and carotenoids, antioxidant activity increases (Chelghoum et al., 2020). Ling et al. (2016) stated that roasting pistachios increases total phenol and antioxidant capacity and reduces the level of total tocopherols and chlorophylls. If oil is obtained from roasted seeds, phenolic compounds pass through the oil phase better. This effect is probably due to the diffusion of phenolic compounds from finite structures or the chemical change of phenols at higher temperatures (Durmaz & Gökmen, 2011). The increase in antioxidant activity is mainly due to the relatively polar compounds in the oil that have accumulated during the process. The cellular changes that occur during the roasting process cause the cancellation of antioxidants of roasted seeds to be higher than the raw seeds and facilitate the extraction of phytochemicals with oil and phenolic compounds and tocopherols are well extracted from roasted seeds (Chen et al., 2021).

Durmaz and Gökmen (2011) reported that by roasting, the antioxidant activity in methanolic oil extraction increased. Evaluation of the antioxidant capacity of products resulting from the Maillard reaction and phenolic compounds transferred to oil increases the oxidative stability and antioxidant capacity of pistachio oil. This means that the thermal process reduces the activity of lipolytic enzymes in the extracted oils, which provides better oxidative stability in seed oils and roasted kernels. They also stated that roasting pistachios increases their antioxidant capacity. Free radical activity has different functions in nuts, for example, Oxidative stability, presented by DPPH, was significantly different in almonds, pine, cashew nuts except for pistachio nuts that have shown no differences (Ghazzawi & Al-Ismail, 2017). Lipid oxidation, also has a significant effect on the shelf life and sensory properties of pistachios. It depends on many factors such as concentration, enzymatic activity of unsaturated fatty acids, mineral compounds and antioxidants (Khajeh-Ali et al., 2022).

Table 2
The effect of roasting method and storage time on the percentage of inhibited DPPH of roasted pistachio.

sample	Concentration 5 mg/kg	Concentration 10 mg/kg	Concentration 20 mg/kg	Concentration 40 mg/kg
Fixed roaster (0 month)	53.93± 0.20 p *	61.46± 0.12 n	69.78± 0.16 i	76.39± 0.04 f
Fixed roaster (3 months)	59.56± 0.11o	66.24± 0.02l	74.57± 0.25 g	81.25± 0.21 d
Fixed roaster (6 months)	65.52±0.34 m	71.77± 0.19 h	85.24± 0.12 c	89.78± 0.15 a
Rotary roaster (0 month)	52.86± 0.03q	61.89± 0.06n	66.98± 0.08 k	78.49± 0.98 e
Rotary roaster (3 months)	61.99±0.13 n	69.07± 0.08 j	78.57± 0.05 e	86.27± 0.37 b
Rotary roaster (6 months)	65.39± 0.06 m	73.99± 0.25 g	85.68± 0.12 bc	90.44± 0.13 a

* Values with unmatched letters in each column have a significant difference at the 5 % level; DPPH: diphenylpicrylhydrazyl.

4. Conclusions

The purpose of this study was to evaluate the oxidation stability of roasted pistachio oil by fixed and rotating methods at 160 °C (15 min) during storage times of (0, 3 and 6 months). By roasting and increasing the storage time, the peroxide value, free fatty acid and acid value of nut oil have increased. The thermal resistance (hr) of the samples increases after roasting and during storage due to Maillard reaction products. Roasted samples at 0 time have the highest inhibitory percentage and the lowest IC50. According to the results, the changes observed in two roasting methods and storage times were within the standard range. The temperature and time did not have any special harmful effects on the antioxidant properties and oxidation stability of roasted pistachios. Therefore, it is recommended to use a commercial rotary roaster at a temperature of 160 °C for 15 min to roast pistachio nuts.

Informed consent

Written informed consent was obtained from all study participants.

CRediT authorship contribution statement

A. Shakerardekani: Conceptualization, Data curation, Formal analysis, Methodology, Project administration, Software, Supervision, Validation, Visualization, Writing – original draft. **M. Rahdari:** Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

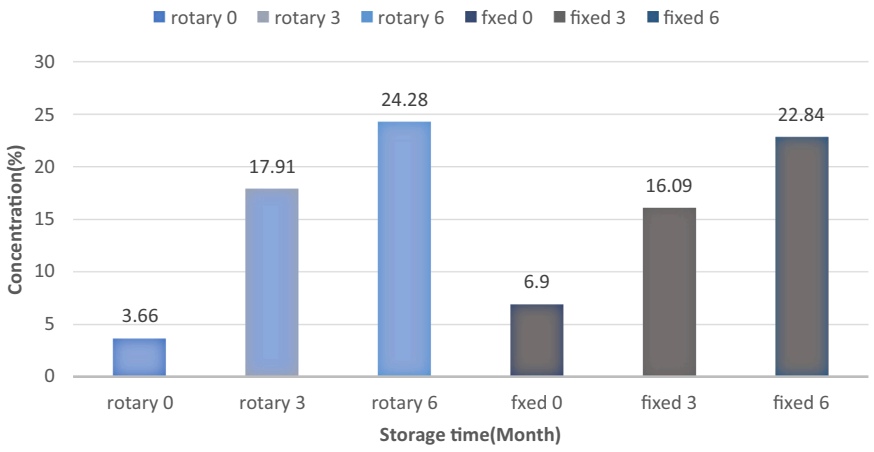

Fig. 1. IC_{50} of roasted pistachios in two fixed and rotating devices during 6 months storage time.

Data availability

Data will be made available on request.

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