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THE DETERMINATION OF OIL PROPERTIES OF SOME OLIVE CULTIVARS GROWN IN SÜTÇÜLER, ISPARTA REGION

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Abstract

The aim of this study was to assess the fatty acid compositions, tocopherol contents and some biochemical properties of 'Ayvalık', 'Memecik', and 'Topakaşı' olive cultivars' fruits grown in Mediterrenean region of Turkey, Sütçüler/Isparta. According to mean values the highest value of oleic acid (73.88 %), which is the most dominant acid in olive fruit, was found in Memecik. The highest value for alpha tocopherol content was obtained from Topakaşı (213.63 %) whereas the highest values for beta, gamma and delta tocopherol contenst were obtained from Memecik (2.46, 4.19 and 0.31 % respectively). Memecik had the highest values for chlorophyll (0.47) , carotenoid (0.31) and pheophytin a (2.29) contents according to mean values. According to the knowledge obtained from the research, the fatty acid composition and the quality characteristics of the olive oil are mainly depended on the growing conditions, harvest period and the oil extraction methods. In the study, it is concluded that 2nd harvest period for Memecik, 2nd and 3rd harvest periods for Ayvalık and Topakaşı would be more suitable under the Isparta, Sütçüler growing conditions for high-quality olive oil. This research is the first detailed research on olive in the research area and it is considered that it will be the basis to future scientific studies.

Key words*: Ayvalık, biochemical properties, fatty acid compositions, tocopherol contents.*

INTRODUCTION

The olive (*Olea europaea* L.) is known the oldest cultivated tree in the world (Özbek, 1975). *Olea europaea* is one of the most important and widespread fruit trees in the Mediterranean basin (Conde et al., 2008), especially in Spain, Italy, Greece and Turkey (Dıraman and Dibeklioğlu, 2009).

Olive oil is an essential constituent of Mediterranean diet and is obtained from the fruit of several cultivars. Each one of these cultivars exhibits specific physical and biochemical characteristics, providing oils with different compositions and performances (Matos et al., 2007). Olive oil is a good source of several bioactive compounds such as mono/poly-unsaturated fatty acids, phenols, phytosterols, carotenoids and tocopherols. Due to their antioxidant properties, these bioactive compounds have reducing effect on the risk of chronic degenerative diseases such as coronary heart disease, cancer, obesity, immune and inflammatory responses (Dag et al., 2015).

Turkey is the sixth largest producer of olive oil in the World (4.6 %) (Sevim et al., 2013) and exported 8% on the average between 2004 and 2010. While the production was 130.000 tonnes in 2008, it increased to 160.000 tonnes in the 2010 (Alkan et al., 2012). The economically important Turkish olive cultivars include Ayvalık, Memecik, Gemlik, Erkence, Nizip Yağlık, and Uslu (Dıraman and Dibeklioğlu, 2009). Olive cultivar 'Memecik' has more than 45% of orchard area in Turkey. Memecik is used both as table olive and for extraction of oil. Olive oil is classified as extra virgin, virgin, olive oil. This classification is carried out according to some quality characteristics (acidity, peroxide value, K_{232}, K_{270}, DK values, alkyl esters) (Caporaso et al., 2015).

The quality of olive oil is affected by many factors such as olive variety, climate and soil characteristics of the geographical region, maturity level, cultural practices and extraction methods (İlyasoğlu and Özçelik, 2011). Olives for oil production are generally harvested in November and December in Turkey. However,

olives are sometimes harvested in early period (October) for production high quality olive oil. So, high quality olive oil was obtained in terms of phenolics and other quality parameters (Yıldırım et al., 2016).

The aim of this study was to determine the variation in the fatty acids, tocopherols and biochemical properties of oils obtained from olive cultivars 'Ayvalık', 'Memecik' and 'Topakaşı' harvested at 3 different maturity stages: (1) early harvest period-1 (green skin with pink spots in less than half of the fruit—Beginning of spotting), (2) early harvest period-2 (pink or purple skin in more than half of the fruit—End of spotting), and (3) optimum harvest period (black skin, less than half of pulp to be purple).

MATERIALS AND METHODS

The study was conducted in the Mediterranean region of Turkey, Sütçüler / Isparta (37° 29'40 "N 30° 58'54" E). In this study, olive cultivars 'Ayvalık', 'Memecik' and 'Topakaşı' grafted on seedling rootstocks were used as materials. Olive trees planted at spacing distance of 5 x 4 m were 10 years old. The altitude of the orchard was 250 m. While Ayvalık and Memecik cultivars are grown in a wide area in Turkey, Topakaşı is only grown in a narrow area of Sütçüler-Isparta, Anamur and Tarsus regions of Turkey. Fruit samples were harvested at 3 different stages of maturity according to the coloring of fruit peel and fruit flesh. These are; (1) Pink spots on green ground (maturity index: 2-3, (2) pink-violet (maturity index: 4-5), and (3) purple-black (maturity index: 6-7).

The obtained natural extra virgin olive oil was placed in dark bottle glasses and kept at -80 °C until analysis. The fatty acid composition of olive oil was determined according to Marquard (1987). 1 ml Na- Methylate (0.5 g Na-methylate + 80 ml methanol + 20 ml iso-octane) solution was added on to 1 ml oil sample and esterification was carried out. 0.25 ml of iso-octane was added before injection and the tube was well-shaken. Then 0.5 ml sample was drawn from the upper phase, which became clear, and by means of a microinjection and injected into the GC apparatus (Pelkin Elmer Autosystem XL). GC condiditions: FID dedector; Cp WAX 52 CB 50 m x 0.32 mm. 1.2 µm column; injector and dedector temperatures: 250^{o}C; carrier gas: He; flow rate: 15 mL/min. The oven temperature was held at 60 ^{o}C for 4 min and increased to 175 with increasing 4 ^{o}C per min. After holding at this temperature for 27 min, the temperature was increased to 215 ^{o}C. After holding at 215 ^{o}C for 5 min, the temperature was gradually increased to 240 ^{o}C. Peaks were identified by taking in to account the relative retention times of standards (Sigma-Aldrich Chemicals 189-19) and results were expressed as percentages of peak areas.

The method of Lampi et al. (1999) was used for tocopherol analysis. Tocopherols (α, γ, δ, and β) were determined by HPLC with a RF-10AXL fluorescence detector (Ex 295-Em 330 nm). 20 µl of the oil was injected into the HPLC device equipped with Luna Silica column (250 × 4.6 mm, 5 µm particle size). Mobile phase: heptane: THF (95:5) (v/v); mobile phase flow rate, 1.2 ml/min; Peak identification was performed according to standards (Cabliochem, Germany). The quantity of tocopherols was calculated according to peak area and expressed as mg kg^{-1} oil.

Analysis of carotenoids and chlorophyll were carried out using spectrophotometer (T70+UV/VIS Spectrometer, PG Instruments Ltd-England). The method defined by Minguez-Mosquera et al. (1991) was used for the determination of carotenoids. 7.5 grams of olive oil was dissolved in cyclohexane and was completed to 25 ml. Samples for total carotenoid were read at 470 nm wavelength and the results were calculated as mg carotenoid/kg fat. The oil samples prepared in cyclohexane were measured at 630 nm, 670 nm and 710 nm wavelengths and the amounts of chlorophyll were calculated as mg of pheophytin a/kg oil using the formula given below (Pokorny et al., 1995).

The amount of chlorophyll (equivalent of mg of pheophytin a / kg fat) = 345.3x [A670-(A630 + A710)] / L

A refers to absorbance, L means the ray path (cell thickness, mm) in the equation.

The method defined by Anonymous (2001) was used for absorbance values. 50 mg of oil sample was weighed and 5% cyclohexane was added to prepare a 1% solution of oil in

cyclohexane. The specific absorbance values in UV radiation was determined using K_{232} and K_{270} nm spectrophotometer. The obtained data were analyzed according to One-Way Anova variance analysis method, and significant differences between the averages were determined according to Duncan's test.

RESULTS AND DISCUSSIONS

1. Fatty acid composition

In this study, oleic (71.80-74.19%), palmitic (11.80-13.72%), linoleic (5.92-8.96%) and stearic (2.05- 2.78%) acid were the major fatty acid in the olive oils. Other fatty acids detected in olive oil were tricosanoic, palmitoleic, linolenic, arachidic, gamalinolenic, heptadecaenoic, eicosatrienoic, behenic, heptadecanoic and myristic acid (Table 1). For all fatty acids except for behenic acid, the cultivar x harvest period interaction was found significant. The oleic and linoleic acid contents of the cultivars were differed according to the fruit maturity periods. The content of oleic acid, which is the main acid in olive cultivars, increased significantly with progress of the maturity in Ayvalık, whereas it decreased in Memecik. The highest oleic acid content was found in Ayvalık as 74.19%, followed by Memecik 73.44% and Topakaşı 72.69% at commercial harvest period (third harvest period). Gurdeniz et al. (2008) reported that the oleic acid contents of Ayvalık and Memecik as 69.58 and 66.32%, respectively at commercial harvest period. Dıraman et al. (2009) found that the amount of oleic acid content of Ayvalık changed between 61.44% and 74.68% at different regions and years. Köseoğlu et al. (2016) investigated the oleic acid component of Memecik at 3 stages of ripening according to skin pigmentation as green, purple, and black and found as 72.37%, 71.23% and 68.92%, respectively.

These different results in literature revealed that the growing conditions and harvest time have important effects on the fatty acid compositions and quality attributes of an olive cultivar.

While the content of linoleic acid decreased in Ayvalık, it increased in Memecik with the progress of maturity. According to average values, the highest content of oleic acid among the varieties was determined as 73.88% in Memecik. The cultivars of Ayvalık and Topakaşı had similar contents of oleic acid. While oleic acid was stable in Topakaşı, linoleic acid increased significantly in the third harvest period. In third maturity stage, oleic acid increased while linoleic acid decreased according to average values.

The contents of palmitic acid decreased at significant levels in Ayvalık and Topakaşı with increased maturity. However, no significant difference was obtained in Memecik. The highest average content of palmitic acid was determined as 13.36% in Ayvalık. Similar to our findings, Dıraman et al. (2009) reported that the variation of palmitic and linoleic acids, which are other major fatty acids for Ayvalık, were as 8.94% - 17.77% and 4.68% - 15.14% respectively in different regions and years. In addition, Uğurlu and Özkan (2011) found that the content of palmitic acid in Memecik was 11.38%.

The content of stearic acid in Ayvalık increased significantly in the third harvest period. The palmitic, linoleic and stearic acid contents were found as 12.65%, 5.92% and 2.78%, respectively at the commercial harvest period (third harvest period) (Table 1).

The contents of tricosanoic acid differed according to the harvest periods of the cultivars. Tricosanoic acid increased in Ayvalık with increasing maturity, while it fluctuated in Memecik.

A similar situation was also observed in Topakaşı. The results of the study showed an increase in palmitoleic acid content increasing maturity. The highest values were determined during the third harvesting period in all cultivars. The highest palmitoleic acid content was determined in Ayvalık with an average of 1.12%, followed by Topakaşı (0.83%) and Memecik (0.79%). Similar to our result, Uğurlu and Özkan (2011) found that palmitoleic acid content was 0.50% in Memecik. The content of linolenic acid increased in all cultivars with incresing maturity. The highest average linolenic acid content was found in Memecik as 0.65%, followed by Ayvalık as 0.54%. Tanılgan et al. (2007) found the linolenic acid content as 0.2% in Ayvalık.

Table 1. Composition of fatty acids (%) obtained from Ayvalık, Memecik and Topakaşı olive cultivars in three different harvesting periods.

Olive cultivars	First harvest	Second harvest	Third harvest	Mean Values
Oleic acid (C18:1)				
Ayvalık	71.80bB	71.93bB	74.19aA	72.64
Memecik	74.72aA	73.48aB	73.44bB	73.88
Topakaşı	72.29b	72.88a	72.69c	72.62
Mean	72.93	72.76	73.44	**Lsd:0.7012**
Palmitic acid (C16:0)				
Ayvalık	13.72aA	13.70aA	12.65B	13.36
Memecik	11.80b	12.01c	12.28	12.03
Topakaşı	13.59aA	12.96bB	12.44C	12.99
Mean	13.03	12.89	12.46	**Lsd:0.4984**
Linoleic acid (C18:2)				
Ayvalık	8.96aA	8.01bB	5.92cC	7.63
Memecik	7.41cC	8.75aA	8.26bB	8.14
Topakaşı	8.27bB	8.24bB	8.76aA	8.42
Mean	8.21	8.33	7.65	**Lsd:0.4416**
Stearic acid (C18:0)				
Ayvalık	2.46aB	2.38aB	2.78aA	2.54
Memecik	2.18bA	2.05bB	2.09cAB	2.11
Topakaşı	2.28b	2.33a	2.39b	2.33
Mean	2.31	2.25	2.42	**Lsd:0.1168**
Tricosanoic acid (C23:0)				
Ayvalık	0.77cC	0.94B	1.13aA	0.94
Memecik	1.28aA	0.93C	1.14aB	1.11
Topakaşı	0.90b	0.94	0.90b	0.91
Mean	0.98	0.93	1.05	**Lsd:0.08924**
Palmitoleic acid (C16:1)				
Ayvalık	0.85aB	1.21aA	1.32aA	1.12
Memecik	0.71bB	0.81bAB	0.85bA	0.79
Topakaşı	0.77ab	0.83b	0.89b	0.83
Mean	0.78	0.95	1.02	**Lsd:0.1282**
Linolenic acid (C18:3)				
Ayvalık	0.46bB	0.49bB	0.67aA	0.54
Memecik	0.54aB	0.68aA	0.72aA	0.65
Topakaşı	0.47bAB	0.44bB	0.51bA	0.47
Mean	0.49	0.54	0.63	**Lsd:0.06176**
Arachidic acid (C20:0)				
Ayvalık	0.33bC	0.39B	0.46A	0.39
Memecik	0.44a	0.41	0.45	0.43
Topakaşı	0.46a	0.43	0.44	0.44
Mean	0.41	0.41	0.45	**Lsd:0.04317**
Gamma-linolenic acid (C18:3)				
Ayvalık	0.22bC	0.30bB	0.38aA	0.30
Memecik	0.38a	0.37a	0.41a	0.38
Topakaşı	0.36a	0.33b	0.35b	0.34
Mean	0.32	0.33	0.38	**Lsd:0.03939**
Heptadecanoic acid (C17:0)				
Ayvalık	0.18B	0.21aA	0.19bAB	0.19
Memecik	0.17A	0.15bA	0.09cB	0.13
Topakaşı	0.18B	0.23aA	0.23aA	0.21
Mean	0.18	0.19	0.17	**Lsd:0.02659**
Eicosatrienoic acid (C20:1)				
Ayvalık	0.10cB	0.18aA	0.08bC	0.12
Memecik	0.24aA	0.18aB	0.06cC	0.16
Topakaşı	0.18bA	0.13bB	0.18aA	0.16
Mean	0.17	0.16	0.11	**Lsd:0.01174**
Behenic acid (C22:0)				
Ayvalık	0.12	0.15	0.13	0.13
Memecik	0.12	0.13	0.14	0.13
Topakaşı	0.13	0.14	0.13	0.13
Mean	0.12B	0.14A	0.13AB	**Lsd:0.01509**
Heptadecenoic acid (C17:1)				
Ayvalık	0.04bB	0.13aA	0.12aA	0.09
Memecik	0.04bB	0.07bA	0.07bA	0.06
Topakaşı	0.14aA	0.13aA	0.10aB	0.12
Mean	0.07	0.11	0.09	**Lsd:0.02176**
Myristic acid (C14:0)				
Ayvalık	0.010cB	0.020A	0.010bB	0.013
Memecik	0.017bB	0.020A	0.020aA	0.019
Topakaşı	0.020a	0.020	0.020a	0.020
Mean	0.016	0.020	0.020	**Lsd:0.002998**

Each value is expressed as mean ±standard deviation. Means followed by different capital letters (years) in the row are signifi cantly different ($p<0.05$). Means followed by different small letters in the columns (cultivars) are signifi cantly different ($p<0.05$).

Our findings are similar those of İlyasoğlu and Özçelik (2011) in terms of palmitic, palmitoleic, stearic, oleic, linoleic and linolenic acid components of Memecik.

The arachidic and gamma-linolenic acid contents increased only in Ayvalık at the significant level with increasing maturity. However, there was no significant difference between the harvest periods for the other two cultivars. The highest average arachidic acid was found in Topakaşı and the highest gamma-linolenic acid was found in Memecik. Dıraman and Dibeklioğlu (2014) found similar results in terms of arachidic acid contents of Ayvalık and Memecik.

In terms of heptadecanoic acid content, there was an increase in Topakaşı in the second harvest period and this increase remained constant during the third harvest period. On the other hand, Ayvalık showed an increase in terms of heptadecanoic acid in the second harvest period and but it was decreased in the third harvest period. The content of heptadecanoic acid decreased in Memecik with increasing maturity and it decreased at significant level in the third harvest period. The highest average heptadecanoic content was determined in Topakaşı as 0.21%, followed by Ayvalık as 0.19%.

Eicosatrienoic acid content increased significantly at the second harvest period in Ayvalık and Topakaşı. the content of eicosatrienoic acid showed a decreasing at significant level in Memecik with progress of maturity. The highest eicosatrienoic content was found in Memecik and Topakaşı as 0.16%. There was no significant difference between cultivars and harvest periods in terms of behenic acid content.

In terms of heptadecanoic content, significant increases observed in the second harvest period in Ayvalık and Memecik and this increase remained stable in the third harvest period. On the contrary, a significant decrease was observed in Topakaşı in the third harvest period. The highest average content of heptadecanoic acid was determined in Topakaşı as 0.12%.

While the content of myristic acid increased during the second harvest period, it decreased in third harvest period in Ayvalık. The fatty acid components of Ayvalık and Memecik

obtained in this research were similar with those of Dıraman (2010) and Dıraman and Dibeklioğlu (2014).

2. Tocopherol contents

One of the most important minor chemical components in olive oils is tocopherols. Beside their health benefits, they also enhance the oxidative stability of olive oils due to their antioxidant properties (Dag et al., 2015). In terms of all tocopherol components, the cultivar x harvest period interaction was found significant.

Among the different tocopherol forms, alpha (α) tocopherol was the major one in all cultivars. The highest α-tocopherol contents were determined in the second harvest period in Ayvalık and Memecik while the highest value was found in the third harvest period in Topakaşı.

The highest average α-tocopherol content was determined in Topakaşı (213.63 mg/kg oil), followed by Ayvalık (212.97 mg/kg oil) (Table 2). Beta (β) tocopherol content increased significantly during the second harvest period in all cultivars and decreased to the lowest level in the third harvest period. The highest average content of β-tocopherol was determined in Memecik (2.46 mg/kg oil). While the content of gamma (γ) tocopherol showed an increasing in Ayvalık with increasing maturity, a decreasing was found in Topakaşı. It increased at second harvest period and then decreased at third harvest period in Memecik.

The highest average content of γ-tocopherol was determined in Memecik (4.19 mg/kg oil), followed by Ayvalık (2.31 mg/kg oil). The highest delta (δ) tocopherol content was determined in the first harvest period in Ayvalık and Topakaşı. The highest average δ-tocopherol content was found in the Memecik as 0.31 mg/kg oil (Table 2). Uğurlu and Özkan (2011) reported that the values of α, β, γ and δ-tocopherol were 205.45 mg/kg oil, 1.645 mg/kg oil, 6.065 mg/kg oil and 0.325 mg/kg oil, respectively in Memecik. These results are similar to our average results obtained in Memecik.

The highest K_{232} values were determined in the second harvest period in all cultivars and the lowest values were determined in the third harvest period. The highest average K_{232} value was determined as 1.57 in the Ayvalık while the lowest value was 1.38 in Memecik (Table 3). The highest average K_{270} value was determined as 0.14 in Ayvalık. Similar to our results, oil quality parameters of K_{232} and K_{270} were found as 1.493 and 0.098, respectively in Memecik by Uğurlu and Özkan (2011).

Table 2. Composition of tocopherol obtained from Ayvalık, Memecik and Topakaşı olive cultivars in three different harvest periods.

Olive cultivars	First harvest	Second harvest	Third harvest	Mean Values
	Alpha (α) tocopherol (mg/kg oil)			
Ayvalık	186.85cC	229.65aA	222.40bB	212.97
Memecik	205.50bB	215.25bA	200.25cC	207.00
Topakaşı	210.15aB	201.30cC	229.45aA	213.63
Mean	200.83	215.40	217.37	**Lsd:3.581**
	Beta (β) tocopherol (mg/kg oil)			
Ayvalık	1.57cB	1.77aA	1.42bC	1.59
Memecik	2.45aB	2.76aA	2.17aC	2.46
Topakaşı	1.72bB	1.79bA	1.27cC	1.59
Mean	1.91	2.10	1.62	**Lsd:0.01341**
	Gamma (γ) tocopherol (mg/kg oil)			
Ayvalık	1.64bC	2.44bB	2.87bA	2.31
Memecik	3.67aB	5.28aA	3.63aC	4.19
Topakaşı	1.64bA	1.56cB	1.14cC	1.45
Mean	2.32	3.09	2.54	**Lsd:0.02448**
	Delta (δ) tocopherol (mg/kg oil)			
Ayvalık	0.13cA	0.11bB	0.10bC	0.11
Memecik	0.24aC	0.42aA	0.28aB	0.31
Topakaşı	0.17bA	0.07cC	0.09cB	0.11
Mean	0.18	0.19	0.16	**Lsd:0.004240**

Each value is expressed as mean ±standard deviation. Means followed by different capital letters (years) in the row are signifi cantly different ($p<0.05$). Means followed by different small letters in the columns (cultivars) are signifi cantly different ($p<0.05$).

Table 3. Some biochemical properties of cultivars

Olive cultivars	First harvest	Second harvest	Third harvest	Mean Values
	Chlorophyll (mg/kg oil)			
Ayvalık	0.31cA	0.24cB	0.17cC	0.24
Memecik	0.52aA	0.43aB	0.46aB	0.47
Topakaşı	0.37bA	0.32bB	0.22bC	0.30
Mean	0.40	0.33	0.28	**Lsd:0.02665**
	Carotenoid (mg/kg oil)			
Ayvalık	0.26cA	0.23cB	0.20bC	0.23
Memecik	0.35aA	0.28aB	0.29aB	0.31
Topakaşı	0.29bA	0.26bB	0.18cC	0.25
Mean	0.30	0.26	0.22	**Lsd:0.008654**
	Pheophytin-a (mg/kg oil)			
Ayvalık	1.08cA	0.74cB	0.34cC	0.72
Memecik	2.69aA	2.04aC	2.13aB	2.29
Topakaşı	1.46bA	0.90bB	0.46bC	0.94
Mean	1.74	1.23	0.98	**Lsd:0.01731**
	K_{232}			
Ayvalık	1.58aA	1.59aA	1.56aB	1.57
Memecik	1.40cA	1.41bA	1.35cB	1.38
Topakaşı	1.51bB	1.58aA	1.50bB	1.53
Mean	1.49	1.53	1.47	**Lsd:0.01095**
	K_{270}			
Ayvalık	0.15aA	0.13aB	0.15aA	0.14
Memecik	0.12bA	0.11bAB	0.10cB	0.11
Topakaşı	0.12b	0.13a	0.12b	0.13
Mean	0.13	0.13	0.13	**Lsd:0.01187**

Each value is expressed as mean ±standard deviation. Means followed by different capital letters (years) in the row are signifi cantly different ($p<0.05$). Means followed by different small letters in the columns (cultivars) are signifi cantly different ($p<0.05$).

İlyasoğlu and Özçelik (2011) also found similar result to our finding in term of K_{270} value. In

this research, the interaction between chlorophyll, carotenoid and pheophytin-a contents and harvest periods were found significant. Chlorophyll, carotenoid and pheophytin-a contents decreased significantly with increasing maturity. The highest average values of chlorophyll, carotenoid, and pheophytin-a were determined in Memecik (0.47, 0.31 and 2.29, respectively) (Table 3). Our findings are similar to those of Uğurlu and Özkan, (2011) which reported the average value of chlorophyll content of Memecik as 0.49 mg/kg of oil.

CONCLUSIONS

In this research, the effects of cultivar and harvest period on the fatty acid composition were significantly determined.

According to the knowledge obtained from the research, the fatty acid composition and the quality characteristics of the olive oil are mainly depended on the growing conditions, harvest period and the oil extraction methods.

In the study, it is concluded that 2nd harvest period for Memecik, 2nd and 3rd harvest periods for Ayvalık and Topakaşı would be more suitable under the Isparta/Sütçüler growing conditions for high-quality olive oil.

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