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# An In-Depth Stability Study of the Essential Oils from *Mentha* × *piperita*, *Mentha spicata*, *Origanum vulgare*, and *Thymus vulgaris*: The Impact of Thermal and Storage Conditions

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**Abstract:** In recent years, there has been a growing scientific interest in essential oils due to their therapeutic and aromatic properties and as potential alternative natural additives for use as preservatives or antibiotics. However, the literature lacks a comprehensive understanding of their stability and how their composition and properties change over time under various conditions. Through this paper, we aim to enhance the existing literature by providing deeper insights into the stability of essential oils and the sustainability of chemical composition in a time-based approach under various conditions. Therefore, four essential oils of the Lamiaceae family (*Origanum vulgare*, *Thymus vulgaris*, *Mentha spicata*, and *M.* x *piperita*) were evaluated with respect to their chemical variation influenced by several factors, such as thermal and storage conditions. Three types of containers were utilized to store the essential oils, i.e., glass ampoules, glass tubes, and metallic containers with plastic caps, for up to six months in −20 °C, 4 °C, 23 °C (with or without light exposure), 35 °C, and 45 °C. Samples were routinely analyzed by GC-MS and components were subjected to principal component analysis to ascertain whether the identified constituents may be useful in reflecting the stability of the analyzed samples. The main compounds appeared to be more stable, while the degradation of minor constituents (<1.0%) occurred in all four essential oils despite the storage conditions. Overall, apart from a slight variation in the chemical load, essential oils can be considered stable for various applications, especially those stored under low oxygen availability conditions.

**Keywords:** essential oils; *Mentha* × *piperita*; *Mentha spicata*; *Origanum vulgare*; *Thymus vulgaris*; chemical variation; GC-MS; stability study

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## 1. Introduction

Natural plant-derived products and especially essential oils are prone to volatility changes, degradation via oxidation, hydrolysis, isomerization, cyclization, or dehydration reactions, either enzymatically or chemically [1]. Essential oils are generally derived from one or more plant parts such as flowers, leaves, fruits, seeds, bark, and roots [2]. Since ancient times, they have gained significant attention mainly for their therapeutic and aromatic properties. During recent decades, they have been particularly used by the pharmaceutical, agricultural, food, hygiene, cosmetics, and fragrance industries. Moreover, they have attracted the interest of the scientific community because of their pharmacological activities, such as antibacterial, antifungal, antiviral, insecticidal, and antioxidant properties [3,4]. With respect to their chemical profile, essential oils are complex mixtures of multiple chemical constituents, mainly mono-/sesqui-terpenes either as hydrocarbons, or oxygenated; moreover, aliphatic derivatives (alkanes, alkenes, alcohols, aldehydes, ketones, fatty acids, and esters), aromatics and miscellaneous compounds can be found [5]. Most of them are derived from the biosynthetic pathways of mevalonic and shikimic acids and are characterized by low molecular weight [6]. Gas chromatography coupled with mass spectrometry (GC-MS) is considered the gold-standard analytical technique for chemical analysis [7].

Due to their nature, the short- and long-term stability of these substances can be influenced by various factors, their physicochemical properties, and the surrounding environmental conditions such as packaging materials and type, as well as other critical factors, including temperature, humidity, light, and oxygen availability. Such factors impact the quality of the essential oils, and divergent degradation processes may occur through the aforementioned reaction experiments [8]. Since the storage conditions significantly vary depending on the producers and the customers, it is mandatory to build up concrete instructions for their safe storage and their life expectancy.

Consequently, stability studies play a crucial role in ensuring the quality and longevity of essential oils, enabling their optimal utilization in various applications. Thus, this paper presents a comprehensive stability analysis method via monitoring the volatile compounds found in selected essential oils by GC-MS. The analysis is based on the evaluation of the physicochemical properties of the essential oils and the accumulation of compounds under different thermal and storage conditions. To assess the impact of various parameters on the quality and shelf life of essential oils, four commonly used plant species were chosen among others existing in the market, namely *Mentha* × *piperita* L., *M. spicata* L., *Origanum vulgare* L., and *Thymus vulgaris* L. In Greece, these essential oils are among the most commercialized with high financial impact, and they are industrially used in food, cosmetics, and pharmaceutical products. Additionally, three types of containers were utilized to store the oils under different environmental conditions, i.e., glass ampoules, glass tubes with caps, and metallic containers with plastic caps, over a period of up to six months in −20 °C, 4 °C, 23 °C, 35 °C, and 45 °C with and without exposure to light. By investigating these factors, we aim to gain insights into the chemical variation of essential oils and their potential deterioration over time and as a result to evaluate their stability. Moreover, the investigation of the stability of the essential oils is crucial, taking into consideration their applications. Thus, this study examines stability aspects from two perspectives. Firstly, from a qualitative standpoint, it focuses on the total chemical composition, including minor compounds (<1.0%), of the primary state of the essential oils, and secondly, it considers the major constituents that significantly affect their chemical identity, biological properties, and potential applications. To assess the impact of the different storage conditions on the stability of the essential oils, the variance of the identified constituents was evaluated through principal component analysis (PCA). It is worth mentioning that there is a lack of comprehensive information on the stability of essential oils in the existing literature. Up to now, this topic has received limited attention, and only a handful of studies have been conducted on volatile oil stability through thermal and storage experiments [8]. Through this paper, we aim to enhance the existing literature by providing deeper insights into the stability of essential oils.

## 2. Materials and Methods

### *2.1. Essential Oils*

The samples used in this study were acquired through VIORYL S.A. (28th km Athens-Lamia National R.D., Greece), all of them being 100% pure natural essential oils extracted by steam distillation from the aerial parts of *Origanum vulgare* L., *Thymus vulgaris* L., *Mentha spicata* L., and *Mentha* × *piperita* L. All essential oils used in this research were from the same manufacturing batch.

*2.2. Gas Chromatography-Mass Spectrometry Analysis*

We conducted GC–MS analyses using a Hewlett-Packard 7820A-5977B MSD system (Agilent Technologies, Santa Clara, CA, USA) operating in EI mode (70 eV). The system was equipped with an HP-5MS fused silica capillary column (30 m × 0.25 mm; film thickness 0.25 µm) and a split-splitless injector. A volume of 2 µL of the samples was injected. The temperature program started at 60 °C upon injection and increased to 300 °C at a rate of 3 °C/min. It was then held at 300 °C for 10 min. Helium was used as the carrier gas, flowing at a rate of 2.0 mL/min. The samples were dissolved in n-pentane, resulting in a final concentration of 1%, except for *M. piperita* essential oil, which had a concentration of 0.5% due to its high content of major constituents.

*2.3. Identification of Compounds*

Retention indices for all compounds were determined using the van den Dool method [9], referencing a series of n-alkanes from C9 to C25 as a standard. The chemical components were identified by comparing their relative retention times and mass spectra with data from the NIST/NBS and Wiley libraries, as well as from Adams [7] and other literature sources. To validate the results, co-chromatography with authentic compounds (Fluka, Sigma, Germany) was performed in some cases. The relative percentages of components were calculated based on GC peak areas without applying correction factors. Qualitative GC-MS analysis revealed the relative proportion of individual compounds in the sample and the reduction or disappearance of any component leads to a relative increase in the percentage of the remaining substances.

*2.4. Stability Study-Selected Conditions*

The chemical compositions of four essential oils were continuously monitored for a period of up to 6 months to verify the chemical stability. All measurements were performed by GC-MS employing the same conditions. Three types of containers were utilized during this study, i.e., clear glass tubes sealed with caps, metallic containers, and glass ampoules (Industrial Chemistry Laboratory, Department of Chemistry, NKUA, Athens, Greece) (see Supporting Information 1). The parameters regarding the thermal and storage conditions were as follows: i. 6 months of storage in clear glass tubes sealed with caps at room temperature (23 °C) under darkness, ii. 6 months of storage in clear glass tubes sealed with caps under direct sunlight exposure, iii. 3 months of storage in clear glass tubes at 35 °C, iv. 3 months of storage in clear glass tubes at 45 °C, v. same parameters as iii in sealed glass ampoules, vi. same parameters as iv in sealed glass ampoules, vii. 6 months of storage at 4 °C in clear glass tubes sealed with caps, viii. same parameters as vii in metal containers, ix. same parameters as vii in sealed glass ampoules, and x. 6 months of storage at −20 °C in clear glass tubes sealed with caps (see flow chart in Figure 1). Measurement time points were every 10 days for i to vi, every 30 days for vii, and at Day 180 for viii to x. For maintaining the 35 °C and 45 °C, stability climate chambers were used (Memmert UE 200; Memmert UM 400, GmbH+Co. KG, D-91126 Schwabach, Germany), while cooling conditions (4 °C and −20 °C) were achieved by using a refrigerator. Essential oil volumes in each container were at 5 mL for glass tubes, ca. 0.5–1.0 mL for glass ampoules, and 50 mL for metallic containers. In addition, the samples were photographed at regular intervals to record changes in their appearance, mainly in color.

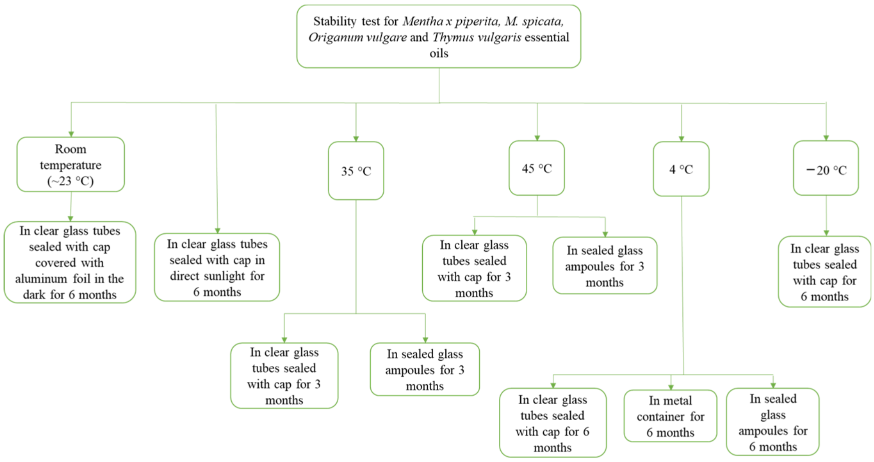


**Figure 1.** Flow chart outlining the selected conditions for the stability study.

### *2.5. Statistical Analysis*

The essential oil contents that exceeded ca. 0.01% of the total oil composition in at least one species were considered as original variables and principal component analysis (PCA) was used to comprehend the similarity among the essential oils in relation to the contents of their chemical constituents. For each plant species, two approaches were employed when contacting PCA. The first approach involved all compounds that were considered original variables. The second approach involved only the main compounds of each essential oil as the original variables. In this approach, the main compounds were selected based on their relative abundance > 1.0%. These data were subjected to multivariate data analysis using SIMCA 16.0.1 (Sartorius Stedim Data Analytics, Umea, Sweden), and prior to PCA, data were log-transformed and pareto-scaled.

## **3. Results and Discussion**

The chemical composition of selected essential oils from the Lamiaceae family, i.e., *O. vulgare*, *T. vulgaris*, *M. spicata*, and *M.* × *piperita*, were analyzed using GC-MS, and the results are presented in Tables 1–20, according to their elution from an HP-5 column. The essential oils were submitted to a stability study by the evaluation of the chemical variation in several conditions (see Figure 1). To conclude whether the identified constituents may be useful in reflecting the stability of the analyzed samples, both all and their main components were subjected to principal component analysis (PCA). The analyses enabled the investigation of thermal degradation and the impact of different conditions on the stability of these selected essential oils.

### *3.1. Individual Compound Analysis: Chemical Composition Variation in Essential Oils under Various Conditions*

#### 3.1.1. *Origanum vulgare* L.

Regarding the initial *O. vulgare* essential oil, a total of 29 compounds were identified, with the main compounds being carvacrol (67.98%), p-cymene (12.29%), *trans*-caryophyllene (5.19%), γ-terpinene (3.98%), caryophyllene oxide (1.96%), linalool (1.94%), α-terpinene (1.27%), limonene (ca. 1.00%), and thymol (0.73%). This composition indicates a high content of phenolic monoterpenes (81.08%). Additionally, smaller percentages of monoterpene and sesquiterpene hydrocarbons (6.57% and 5.92%, respectively), oxygenated monoterpenes (3.46%), and sesquiterpenes (2.05%) were present. These findings are consistent with essential oils derived from oregano subspecies found in Greece [10].

During storage of the *Origanum* aetheroleum at room temperature (ca. 23 °C without exposure to light for 6 months, Table 1), a small percentage of volatile substances were lost from the sample within the first 10 days. Since GC-MS analysis examines the relative proportion of substances in the sample, the reduction or disappearance of these components leads to a relative increase in the percentage of the main substances, without an actual quantitative increase. Within Days 10 to 70, no significant alterations in the chemical profiles were observed. However, deterioration occurred between Days 80 and 130, as α-phellandrene, terpinen-4-ol, α-copaene, and humulene epoxide could no longer be detected. In the following days (140 to 170), traces of thymol and α-humulene were no longer detectable. As a result, the percentage of carvacrol at Day 170 increased from 67.98 (Day 1) to 79.03%, while the remaining detectable substances were α-terpinene, p-cymene, limonene, γ-terpinene, linalool, *trans*-caryophyllene, and caryophyllene oxide.

Similar results were observed in the other experimental conditions (room temperature in direct sunlight, 35 °C, 45 °C, 4 °C, and −20 °C), indicating significant alterations in the essential oil composition under all conditions. In the experiment involving direct sunlight exposure for 6 months (Table 2), the deterioration pattern was consistent with that observed between Days 10 to 70, 80 to 130, and 140 to 170. The components detected in the last measurement (Day 170) are the same as those of the previous experiment (α-terpinene, p-cymene, limonene, γ-terpinene, linalool, *trans*-caryophyllene, and caryophyllene oxide), while the percentage of carvacrol, in this case, increases to 79.12%

Under high-temperature conditions (Tables 3 and 4), the influence of the selected containers on the stability of the essential oil is revealed. The samples were placed for 3 months at a constant temperature of 35 °C and 45 °C in glass ampoules and in glass tubes with caps. The essential oil stored in ampoules exhibited increased stability compared to those in glass tubes despite the high temperature. For example, they managed to preserve α-humulene, borneol, terpinen-4-ol, and thymol for a duration of up to 3 months, while α-phellandrene, α-copaene, β-bisabolene, and α-humulene epoxide remained for a longer period compared to the samples in glass tubes. This difference is likely due to the reduced amount of air content in the ampoules, compared to the glass tubes, leading to lower oxygen availability, which is the main cause of oxidations and negative effects on the stability of essential oils experiments [8].

Finally, Table 5 exhibits the composition of the essential oil after being subjected to refrigeration (4 °C and −20 °C) for 6 months. The impact of container type (glass ampoules, glass tubes, and metal containers) on stability is evident here as well, as it is directly related to the oxygen availability in each case. In the glass ampoules with less air availability and in a well-sealed metal container with a plastic cap, more substances were preserved (borneol, terpinen-4-ol, thymol, and α-humulene). As a result, the percentage of carvacrol is significantly lower at Day 180 (73.32% for the metal container and 74.53% for the ampoule) compared to the glass tubes at 4 °C and −20 °C (79.44% and 79.41% respectively), in which fewer substances are retained. Furthermore, the availability of oxygen plays a crucial role in the stability of essential oils during their storage at low temperatures. This is in line with Henry's law, which states that the solubility of oxygen is higher under such conditions experiments [8].

Regarding the organoleptic characteristics of the essential oil, the photo-documentation revealed a color alteration from pale yellow to intense yellow during storage. Specifically, the change was intense at 35 °C and 45 °C as early as 45 days, while at the same thermal conditions, the color was better preserved in ampoules. Moreover, after 6 months of storage at 45 °C, the color of the sample turned to bright orange. It is noticeable that the storage in the glass tubes at 35 °C and 45 °C resulted in a significant decrease in the sample volumes, which implies extensive evaporation. Finally, under direct exposure to sunlight, the sample color slightly faded (Figure 2).

**Table 1.** Chemical composition (%) of *Origanum vulgare* L. essential oil during 6 months of storage in clear glass tubes sealed with cap at room temperature (23 °C) under darkness.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-Phellandrene | 1004 | 0.16 | 0.09 | 0.09 | 0.11 | 0.11 | 0.10 | 0.08 | 0.08 | 0.06 | - | - | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 1.27 | 0.84 | 0.92 | 0.84 | 0.92 | 0.80 | 0.78 | 0.75 | 0.51 | 0.40 | 0.43 | 0.39 | 0.54 | 0.57 | 0.59 | 0.62 | 0.49 | 0.43 |
| 4 | p-Cymene | 1022 | 12.29 | 12.61 | 14.22 | 13.07 | 13.16 | 12.56 | 13.1 | 13.58 | 13.62 | 12.47 | 12.17 | 11.85 | 13.73 | 14.4 | 14.96 | 15.36 | 13.29 | 12.33 |
| 5 | Limonene + 1,8 Cineol | 1042 | 1.37 | 1.28 | 1.42 | 1.30 | 1.36 | 1.27 | 1.25 | 1.20 | 0.83 | 0.66 | 0.69 | 0.65 | 0.74 | 0.78 | 0.78 | 0.81 | 0.74 | 0.71 |
| 6 | *trans*-β-Ocimene | 1047 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 3.98 | 3.62 | 3.85 | 3.56 | 3.69 | 3.36 | 3.45 | 3.34 | 2.63 | 2.16 | 2.23 | 2.10 | 2.55 | 2.63 | 2.63 | 2.71 | 2.33 | 2.06 |
| 8 | *trans*-Sabinene hydrate | 1069 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 9 | p-Mentha-3,8-diene | 1071 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 10 | Terpinolene | 1087 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | Linalool | 1099 | 1.94 | 1.52 | 1.49 | 1.53 | 1.61 | 1.42 | 1.42 | 1.32 | 0.93 | 0.69 | 0.73 | 0.67 | 0.89 | 0.97 | 0.93 | 0.97 | 0.80 | 0.71 |
| 12 | Borneol | 1156 | 0.53 | 0.4 | 0.37 | 0.40 | 0.43 | 0.36 | 0.38 | 0.33 | 0.23 | 0.16 | 0.19 | - | 0.23 | 0.25 | - | - | - | - |
| 13 | Terpinen-4-ol | 1169 | 0.46 | 0.38 | 0.35 | 0.37 | 0.39 | 0.34 | 0.34 | 0.30 | 0.20 | - | - | - | - | 0.23 | - | - | - | - |
| 14 | α-Terpineol | 1185 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Carvacrol methyl ether | 1237 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | *trans*-Citral (Geranial) | 1266 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Thymol | 1292 | 0.73 | 0.91 | 0.95 | 0.93 | 0.99 | 0.95 | 0.88 | 0.79 | 0.55 | 0.44 | 0.48 | 0.45 | 0.57 | 0.57 | - | - | - | - |
| 18 | Carvacrol | 1301 | 67.98 | 71.19 | 69.88 | 70.55 | 70.30 | 71.29 | 71.64 | 71.69 | 75.3 | 78.62 | 78.63 | 79.29 | 76.54 | 75.77 | 76.41 | 75.89 | 78.20 | 79.03 |
| 19 | Eugenol | 1354 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | α-Copaene | 1368 | 0.13 | 0.09 | 0.08 | 0.10 | 0.10 | 0.09 | 0.07 | 0.08 | - | - | - | - | - | - | - | - | - | - |
| 21 | Geranyl acetate | 1381 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | *cis*-Caryophyllene | 1397 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Caryophyllene | 1411 | 5.19 | 4.87 | 4.58 | 4.83 | 4.68 | 4.87 | 4.54 | 4.50 | 3.60 | 3.16 | 3.20 | 3.26 | 2.96 | 2.71 | 2.73 | 2.68 | 3.03 | 3.40 |
| 24 | Aromadendrene | 1438 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | α-Humulene | 1445 | 0.38 | 0.29 | 0.26 | 0.30 | 0.29 | 0.30 | 0.27 | 0.27 | 0.20 | 0.16 | 0.17 | 0.17 | 0.16 | 0.15 | - | - | - | - |
| 26 | β-Bisabolene | 1500 | 0.14 | 0.08 | - | 0.08 | 0.08 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | δ-Cadinene | 1512 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | Caryophyllene oxide | 1576 | 1.96 | 1.68 | 1.53 | 1.71 | 1.74 | 1.78 | 1.65 | 1.70 | 1.34 | 1.07 | 1.07 | 1.16 | 1.08 | 0.97 | 0.97 | 0.96 | 1.12 | 1.33 |
| 29 | Humulene epoxide | 1590 | 0.09 | 0.07 | - | 0.07 | 0.07 | 0.07 | - | 0.06 | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 6.57 | 5.45 | 5.85 | 5.42 | 5.67 | 5.15 | 5.19 | 5.01 | 3.78 | 3.02 | 3.14 | 2.95 | 3.61 | 3.75 | 3.77 | 3.90 | 3.34 | 2.99 |
| | Oxygenated monoterpenes | | 3.46 | 2.68 | 2.64 | 2.69 | 2.84 | 2.50 | 2.52 | 2.31 | 1.61 | 1.05 | 1.13 | 0.87 | 1.34 | 1.68 | 1.16 | 1.21 | 1.02 | 0.92 |
| | Phenolic monoterpenes | | 81.08 | 84.71 | 85.05 | 84.55 | 84.45 | 84.8 | 85.62 | 86.06 | 89.47 | 91.53 | 91.28 | 91.59 | 90.84 | 90.74 | 91.37 | 91.25 | 91.49 | 91.36 |
| | Sesquiterpene hydrocarbons | | 5.92 | 5.33 | 4.92 | 5.31 | 5.15 | 5.32 | 4.88 | 4.85 | 3.80 | 3.32 | 3.37 | 3.43 | 3.12 | 2.86 | 2.73 | 2.68 | 3.03 | 3.40 |
| | Oxygenated sesquiterpenes | | 2.05 | 1.75 | 1.53 | 1.78 | 1.81 | 1.85 | 1.65 | 1.76 | 1.34 | 1.07 | 1.07 | 1.16 | 1.08 | 0.97 | 0.97 | 0.96 | 1.12 | 1.33 |
| | Total identified | | 99.08 | 99.85 | 99.99 | 99.75 | 99.92 | 99.62 | 99.85 | 99.99 | 100.0 | 99.99 | 99.99 | 99.99 | 99.99 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 2.** Chemical composition (%) of *Origanum vulgare* L. essential oil during 6 months of storage in clear glass tubes sealed with cap under direct sunlight exposure.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-Phellandrene | 1004 | 0.16 | 0.11 | 0.08 | 0.08 | 0.10 | 0.07 | 0.07 | 0.08 | - | - | - | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 1.27 | 1.04 | 0.84 | 0.78 | 0.79 | 0.58 | 0.64 | 0.59 | 0.40 | 0.33 | 0.32 | 0.30 | 0.39 | 0.35 | 0.38 | 0.48 | 0.33 | 0.34 |
| 4 | p-Cymene | 1022 | 12.29 | 13.55 | 13.72 | 13.55 | 13.39 | 12.83 | 13.76 | 13.35 | 13.03 | 13.05 | 12.91 | 12.85 | 14.09 | 13.93 | 14.68 | 14.28 | 14.7 | 12.93 |
| 5 | Limonene + 1,8 Cineol | 1042 | 1.37 | 1.46 | 1.37 | 1.33 | 1.37 | 1.16 | 1.23 | 1.22 | 0.86 | 0.67 | 0.68 | 0.65 | 0.72 | 0.70 | 0.75 | 0.75 | 0.78 | 0.69 |
| 6 | *trans*-β-Ocimene | 1047 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 3.98 | 3.94 | 3.52 | 3.28 | 3.30 | 2.71 | 2.95 | 2.72 | 2.05 | 1.74 | 1.69 | 1.61 | 1.86 | 1.74 | 1.79 | 2.23 | 1.61 | 1.76 |
| 8 | *trans*-Sabinene hydrate | 1069 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 9 | p-Mentha-3,8-diene | 1071 | 0.07 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 10 | Terpinolene | 1087 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | Linalool | 1099 | 1.94 | 1.83 | 1.56 | 1.53 | 1.55 | 1.38 | 1.43 | 1.33 | 0.94 | 0.73 | 0.72 | 0.69 | 0.85 | 0.90 | 0.90 | 0.88 | 0.77 | 0.75 |
| 12 | Borneol | 1156 | 0.53 | 0.52 | 0.41 | 0.42 | 0.42 | 0.34 | 0.39 | 0.36 | 0.24 | 0.18 | 0.19 | - | - | 0.24 | - | - | - | - |
| 13 | Terpinen-4-ol | 1169 | 0.46 | 0.48 | 0.37 | 0.38 | 0.38 | 0.31 | 0.34 | 0.31 | 0.21 | - | - | - | - | - | - | - | - | - |
| 14 | α-Terpineol | 1185 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Carvacrol methyl ether | 1237 | 0.05 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | *trans*-Citral (Geranial) | 1266 | 0.04 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Thymol | 1292 | 0.73 | 1.08 | 0.92 | 0.95 | 0.97 | 0.80 | 0.92 | 0.85 | 0.62 | 0.48 | 0.48 | 0.49 | 0.56 | 0.64 | - | - | - | - |
| 18 | Carvacrol | 1301 | 67.98 | 68.31 | 69.98 | 70.34 | 70.66 | 72.26 | 71.32 | 72.1 | 76.54 | 78.53 | 78.68 | 79.02 | 77.4 | 77.8 | 77.72 | 77.49 | 77.47 | 79.12 |
| 19 | Eugenol | 1354 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | α-Copaene | 1368 | 0.13 | 0.10 | 0.10 | 0.10 | 0.09 | 0.08 | 0.07 | 0.07 | - | - | - | - | - | - | - | - | - | - |
| 21 | Geranyl acetate | 1381 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | *cis*-Caryophyllene | 1397 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Caryophyllene | 1411 | 5.19 | 4.66 | 4.45 | 4.38 | 4.32 | 4.09 | 3.86 | 3.74 | 3.02 | 2.54 | 2.52 | 2.48 | 2.37 | 2.11 | 2.14 | 2.67 | 2.39 | 2.72 |
| 24 | Aromadendrene | 1438 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | α-Humulene | 1445 | 0.38 | 0.30 | 0.27 | 0.28 | 0.27 | 0.25 | 0.23 | 0.22 | 0.16 | 0.14 | 0.14 | 0.14 | - | - | - | - | - | - |
| 26 | β-Bisabolene | 1500 | 0.14 | 0.10 | 0.08 | 0.08 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | δ-Cadinene | 1512 | 0.06 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | Caryophyllene oxide | 1575 | 1.96 | 1.91 | 2.02 | 2.21 | 2.18 | 2.46 | 2.40 | 2.40 | 1.92 | 1.61 | 1.67 | 1.77 | 1.75 | 1.59 | 1.64 | 1.22 | 1.95 | 1.69 |
| 29 | Humulene epoxide | 1590 | 0.09 | 0.08 | 0.08 | 0.09 | 0.08 | 0.09 | 0.09 | 0.09 | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 6.57 | 6.11 | 5.40 | 5.07 | 5.15 | 4.17 | 4.52 | 4.24 | 3.05 | 2.54 | 2.49 | 2.37 | 2.75 | 2.58 | 2.70 | 3.24 | 2.49 | 2.58 |
| | Oxygenated monoterpenes | | 3.46 | 3.34 | 2.75 | 2.73 | 2.76 | 2.38 | 2.53 | 2.37 | 1.65 | 1.11 | 1.11 | 0.89 | 1.07 | 1.35 | 1.13 | 1.11 | 1.00 | 0.96 |
| | Phenolic monoterpenes | | 81.08 | 82.98 | 84.62 | 84.84 | 85.02 | 85.89 | 86.00 | 86.3 | 90.19 | 92.06 | 92.07 | 92.36 | 92.05 | 92.37 | 92.4 | 91.77 | 92.17 | 92.05 |
| | Sesquiterpene hydrocarbons | | 5.92 | 5.20 | 4.90 | 4.84 | 4.75 | 4.42 | 4.16 | 4.03 | 3.18 | 2.68 | 2.66 | 2.62 | 2.37 | 2.11 | 2.14 | 2.67 | 2.39 | 2.72 |
| | Oxygenated sesquiterpenes | | 2.05 | 1.99 | 2.10 | 2.30 | 2.26 | 2.55 | 2.49 | 2.40 | 1.92 | 1.61 | 1.67 | 1.77 | 1.75 | 1.59 | 1.64 | 1.22 | 1.95 | 1.69 |
| | Total identified | | 99.08 | 99.68 | 99.77 | 99.78 | 99.94 | 99.41 | 99.70 | 99.43 | 99.99 | 100 | 100 | 100 | 99.99 | 100 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 3.** Chemical composition (%) of *Origanum vulgare* L. essential oil during 3 months of storage in sealed glass ampoules at a temperature of 35 °C and 45 °C.

| No. | Compounds | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-Phellandrene | 1004 | 0.16 | 0.09 | 0.10 | 0.07 | 0.11 | 0.11 | 0.06 | 0.04 | - | 0.09 | 0.09 | 0.08 | 0.10 | 0.10 | 0.08 | 0.09 | - |
| 2 | δ-3-Carene | 1006 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 1.27 | 0.80 | 0.87 | 0.77 | 0.93 | 0.81 | 0.63 | 0.41 | 0.52 | 0.80 | 0.92 | 0.86 | 0.88 | 0.90 | 0.83 | 0.80 | 0.53 |
| 4 | p-Cymene | 1022 | 12.29 | 12.7 | 13.66 | 13.16 | 13.40 | 12.21 | 13.30 | 11.67 | 12.10 | 12.7 | 13.44 | 13.08 | 13.06 | 13.57 | 13.13 | 13.78 | 12.27 |
| 5 | Limonene + 1,8 Cineol | 1042 | 1.37 | 1.30 | 1.37 | 1.32 | 1.39 | 1.19 | 1.24 | 1.02 | 0.83 | 1.30 | 1.37 | 1.31 | 1.39 | 1.38 | 1.26 | 1.24 | 0.83 |
| 6 | *trans*-β-Ocimene | 1047 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 3.98 | 3.60 | 3.60 | 3.29 | 3.73 | 3.31 | 2.88 | 2.00 | 2.68 | 3.60 | 3.79 | 3.58 | 3.68 | 3.8 | 3.61 | 3.46 | 2.71 |
| 8 | *trans*-Sabinene hydrate | 1069 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 9 | p-Mentha-3,8-diene | 1071 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 10 | Terpinolene | 1087 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | Linalool | 1099 | 1.94 | 1.50 | 1.55 | 1.53 | 1.58 | 1.38 | 1.48 | 1.23 | 0.97 | 1.50 | 1.55 | 1.50 | 1.47 | 1.56 | 1.47 | 1.31 | 0.97 |
| 12 | Borneol | 1156 | 0.53 | 0.30 | 0.43 | 0.41 | 0.41 | 0.38 | 0.40 | 0.33 | 0.21 | 0.30 | 0.41 | 0.40 | 0.35 | 0.39 | 0.40 | 0.35 | 0.24 |
| 13 | Terpinen-4-ol | 1169 | 0.46 | 0.30 | 0.37 | 0.37 | 0.38 | 0.35 | 0.36 | 0.29 | 0.22 | 0.30 | 0.37 | 0.37 | 0.35 | 0.37 | 0.36 | 0.31 | 0.22 |
| 14 | α-Terpineol | 1185 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Carvacrol methyl ether | 1237 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | *trans*-Citral (Geranial) | 1266 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Thymol | 1292 | 0.73 | 0.90 | 0.93 | 0.94 | 0.94 | 2.28 | 1.03 | 5.10 | 0.59 | 0.9 | 0.91 | 0.92 | 0.89 | 1.01 | 0.92 | 0.83 | 0.59 |
| 18 | Carvacrol | 1301 | 67.98 | 71.00 | 70.04 | 70.79 | 70.08 | 70.71 | 71.35 | 71.6 | 76.69 | 71.00 | 69.83 | 70.79 | 71.27 | 70.46 | 71.16 | 71.31 | 76.36 |
| 19 | Eugenol | 1354 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | α-Copaene | 1368 | 0.13 | 0.10 | 0.10 | 0.09 | 0.09 | 0.09 | 0.09 | 0.07 | - | 0.10 | 0.09 | 0.09 | 0.08 | 0.09 | 0.08 | 0.07 | - |
| 21 | Geranyl acetate | 1381 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | *cis*-Caryophyllene | 1397 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Caryophyllene | 1411 | 5.19 | 4.80 | 4.58 | 4.50 | 4.72 | 4.42 | 3.88 | 3.05 | 3.81 | 4.80 | 4.84 | 4.78 | 4.67 | 4.63 | 4.67 | 4.52 | 3.80 |
| 24 | Aromadendrene | 1418 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | α-Humulene | 1445 | 0.38 | 0.20 | 0.28 | 0.28 | 0.29 | 0.26 | 0.24 | 0.19 | 0.21 | 0.20 | 0.29 | 0.29 | 0.28 | 0.28 | 0.28 | 0.26 | 0.21 |
| 26 | β-Bisabolene | 1500 | 0.14 | 0.08 | 0.08 | 0.07 | 0.07 | 0.07 | 0.06 | - | - | 0.08 | 0.07 | 0.07 | - | - | - | - | - |
| 27 | δ-Cadinene | 1512 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | Caryophyllene oxide | 1576 | 1.96 | 1.60 | 1.91 | 2.03 | 1.61 | 1.48 | 2.52 | 2.56 | 1.16 | 1.60 | 1.61 | 1.60 | 1.52 | 1.46 | 1.51 | 1.39 | 1.10 |
| 29 | Humulene epoxide | 1590 | 0.09 | 0.07 | 0.08 | 0.08 | 0.06 | 0.06 | 0.10 | 0.10 | - | 0.07 | 0.06 | 0.06 | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 6.57 | 5.40 | 5.53 | 5.05 | 5.74 | 5.06 | 4.44 | 3.16 | 3.78 | 5.40 | 5.76 | 5.44 | 5.63 | 5.77 | 5.40 | 5.22 | 3.82 |
| | Oxygenated monoterpenes | | 3.46 | 2.49 | 2.76 | 2.71 | 2.79 | 2.47 | 2.61 | 2.16 | 1.65 | 2.49 | 2.74 | 2.66 | 2.59 | 2.73 | 2.61 | 2.34 | 1.68 |
| | Phenolic monoterpenes | | 81.08 | 84.60 | 84.63 | 84.89 | 84.42 | 85.2 | 85.68 | 88.37 | 89.38 | 84.6 | 84.18 | 84.79 | 85.22 | 85.04 | 85.21 | 85.92 | 89.22 |
| | Sesquiterpene hydrocarbons | | 5.92 | 5.18 | 5.04 | 4.94 | 5.17 | 4.84 | 4.27 | 3.31 | 4.02 | 5.18 | 5.29 | 5.23 | 5.03 | 5.00 | 5.03 | 4.85 | 4.01 |
| | Oxygenated sesquiterpenes | | 2.05 | 1.67 | 1.99 | 2.11 | 1.67 | 1.54 | 2.62 | 2.66 | 1.16 | 1.67 | 1.67 | 1.66 | 1.52 | 1.46 | 1.51 | 1.39 | 1.10 |
| | Total identified | | 99.08 | 99.34 | 99.95 | 99.7 | 99.79 | 99.11 | 99.62 | 99.66 | 99.99 | 99.34 | 99.64 | 99.78 | 99.99 | 100 | 99.76 | 99.72 | 99.83 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 4.** Chemical composition (%) of *Origanum vulgare* L. essential oil during 3 months of storage in clear glass tubes sealed with cap at a temperature of 35 °C and 45 °C.

| | | | | 35 °C | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 1 | α-Phellandrene | 1004 | 0.16 | 0.09 | - | - | - | - | - | - | - | 0.08 | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 1.27 | 0.71 | 0.50 | 0.39 | 0.37 | 0.44 | 0.43 | 0.44 | 0.44 | 0.72 | 0.50 | 0.38 | 0.36 | 0.41 | 0.41 | 0.46 | 0.43 |
| 4 | p-Cymene | 1022 | 12.29 | 14.06 | 13.13 | 12.38 | 12.24 | 13.07 | 13.4 | 14.09 | 14.04 | 14.24 | 13.51 | 12.51 | 12.36 | 12.84 | 12.71 | 13.95 | 13.90 |
| 5 | Limonene + 1.8 Cineol | 1042 | 1.37 | 1.22 | 0.86 | 0.71 | 0.67 | 0.70 | 0.71 | 0.73 | 0.75 | 1.20 | 0.88 | 0.68 | 0.65 | 0.65 | 0.71 | 0.76 | 0.73 |
| 6 | *trans*-β-Ocimene | 1047 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 3.98 | 3.25 | 2.47 | 2.03 | 1.93 | 2.16 | 2.09 | 2.10 | 2.05 | 3.23 | 2.47 | 2.00 | 1.91 | 2.04 | 2.03 | 2.13 | 1.99 |
| 8 | *trans*-Sabinene hydrate | 1069 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 9 | p-Mentha-3.8-diene | 1071 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 10 | Terpinolene | 1087 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | Linalool | 1099 | 1.94 | 1.34 | 0.95 | 0.72 | 0.70 | 0.83 | 0.91 | 0.90 | 0.86 | 1.35 | 0.93 | 0.72 | 0.70 | 0.79 | 0.90 | 0.92 | 0.83 |
| 12 | Borneol | 1156 | 0.53 | 0.33 | 0.24 | 0.19 | - | - | - | - | - | 0.34 | 0.23 | 0.19 | - | - | - | - | - |
| 13 | Terpinen-4-ol | 1169 | 0.46 | 0.30 | 0.22 | - | - | - | - | - | - | 0.31 | 0.21 | - | - | - | - | - | - |
| 14 | α-Terpineol | 1185 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Carvacrol methyl ether | 1237 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | *trans*-Citral (Geranial) | 1266 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Thymol | 1292 | 0.73 | 0.76 | 0.59 | 0.48 | 0.48 | 0.54 | 0.61 | - | - | 0.81 | 0.58 | 0.48 | 0.47 | 0.50 | 0.68 | - | - |
| 18 | Carvacrol | 1301 | 67.98 | 71.68 | 75.9 | 78.69 | 79.14 | 78.01 | 78.09 | 77.97 | 77.91 | 71.47 | 75.62 | 78.69 | 79.15 | 78.46 | 78.8 | 78.01 | 78.26 |
| 19 | Eugenol | 1354 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | α-Copaene | 1368 | 0.13 | 0.07 | - | - | - | - | - | - | - | 0.07 | - | - | - | - | - | - | - |
| 21 | Geranyl acetate | 1381 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | *cis*-Caryophyllene | 1397 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Caryophyllene | 1411 | 5.19 | 4.11 | 3.47 | 2.97 | 2.95 | 2.86 | 2.51 | 2.50 | 2.58 | 4.11 | 3.42 | 2.95 | 2.92 | 2.81 | 2.54 | 2.54 | 2.55 |
| 24 | Aromadendrene | 1438 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | α-Humulene | 1445 | 0.38 | 0.24 | 0.19 | 0.16 | 0.16 | - | - | - | - | 0.24 | 0.19 | 0.16 | 0.15 | 0.16 | - | - | - |
| 26 | β-Bisabolene | 1500 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | δ-Cadinene | 1512 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | Caryophyllene oxide | 1576 | 1.96 | 1.72 | 1.48 | 1.28 | 1.36 | 1.39 | 1.23 | 1.26 | 1.37 | 1.76 | 1.46 | 1.24 | 1.32 | 1.34 | 1.22 | 1.23 | 1.31 |
| 29 | Humulene epoxide | 1590 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 6.57 | 4.90 | 3.57 | 2.92 | 2.77 | 3.09 | 3.02 | 3.05 | 3.02 | 4.87 | 3.59 | 2.86 | 2.73 | 2.91 | 2.94 | 3.12 | 2.93 |
| | Oxygenated monoterpenes | | 3.46 | 2.34 | 1.67 | 1.12 | 0.90 | 1.04 | 1.12 | 1.12 | 1.09 | 2.36 | 1.63 | 1.11 | 0.90 | 0.99 | 1.11 | 1.15 | 1.05 |
| | Phenolic monoterpenes | | 81.08 | 86.5 | 89.62 | 91.55 | 91.86 | 91.62 | 92.10 | 92.06 | 91.95 | 86.52 | 89.71 | 91.68 | 91.98 | 91.8 | 92.19 | 91.96 | 92.16 |
| | Sesquiterpene hydrocarbons | | 5.92 | 4.42 | 3.66 | 3.13 | 3.11 | 2.86 | 2.51 | 2.50 | 2.58 | 4.42 | 3.61 | 3.11 | 3.07 | 2.97 | 2.54 | 2.54 | 2.55 |
| | Oxygenated sesquiterpenes | | 2.05 | 1.72 | 1.48 | 1.28 | 1.36 | 1.39 | 1.23 | 1.26 | 1.37 | 1.76 | 1.46 | 1.24 | 1.32 | 1.34 | 1.22 | 1.23 | 1.31 |
| | Total identified | | 99.08 | 99.88 | 100 | 100 | 100 | 100 | 99.98 | 99.99 | 100 | 99.93 | 100 | 100 | 99.99 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 5.** Chemical composition (%) of *Origanum vulgare* L. essential oil during 6 months of storage at 4 °C (in clear glass tubes sealed with cap, in metal containers, and in sealed glass ampoules) and at −20 °C (in clear glass tubes sealed with cap).

| | | | | | | 4 °C Glass Tubes | | | | 4 °C Metal | 4 °C Ampoules | −20 °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No** | **Compound** | **KI** | **1st** | **30th** | **60th** | **90th** | **120th** | **150th** | **180th** | **180th** | **180th** | **180th** |
| 1 | α-Phellandrene | 1004 | 0.16 | 0.09 | 0.09 | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.02 | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 1.27 | 0.89 | 0.85 | 0.54 | 0.47 | 0.53 | 0.46 | 0.71 | 0.68 | 0.45 |
| 4 | p-Cymene | 1022 | 12.29 | 13.20 | 13.49 | 12.72 | 12.03 | 13.37 | 11.9 | 14.96 | 13.88 | 11.96 |
| 5 | Limonene + 1,8 Cineol | 1042 | 1.37 | 1.33 | 1.31 | 0.83 | 0.65 | 0.73 | 0.66 | 1.00 | 0.97 | 0.67 |
| 6 | *trans*-β-Ocimene | 1047 | 0.03 | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 3.98 | 3.68 | 3.70 | 2.70 | 2.41 | 2.47 | 2.36 | 3.00 | 3.00 | 2.20 |
| 8 | *trans*-Sabinene hydrate | 1069 | 0.02 | - | - | - | - | - | - | - | - | - |
| 9 | p-Mentha-3,8-diene | 1071 | 0.07 | - | - | - | - | - | - | - | - | - |
| 10 | Terpinolene | 1087 | 0.08 | - | - | - | - | - | - | - | - | - |
| 11 | Linalool | 1099 | 1.94 | 1.53 | 1.45 | 0.95 | 0.79 | 0.81 | 0.75 | 1.15 | 1.10 | 0.69 |
| 12 | Borneol | 1156 | 0.53 | 0.41 | 0.39 | 0.24 | - | - | - | 0.32 | 0.32 | - |
| 13 | Terpinen-4-ol | 1169 | 0.46 | 0.38 | 0.35 | 0.21 | - | - | - | 0.31 | 0.28 | - |
| 14 | α-Terpineol | 1185 | 0.05 | - | - | - | - | - | - | - | - | - |
| 15 | Carvacrol methyl ether | 1237 | 0.05 | - | - | - | - | - | - | - | - | - |
| 16 | *trans*-Citral (Geranial) | 1266 | 0.04 | - | - | - | - | - | - | - | - | - |
| 17 | Thymol | 1292 | 0.73 | 0.92 | 0.91 | 0.57 | 0.43 | - | - | 0.74 | 0.73 | - |
| 18 | Carvacrol | 1301 | 67.98 | 70.38 | 70.51 | 75.91 | 78.76 | 77.97 | 79.44 | 73.32 | 74.53 | 79.41 |
| 19 | Eugenol | 1354 | 0.03 | - | - | - | - | - | - | - | - | - |
| 20 | α-Copaene | 1368 | 0.13 | 0.09 | 0.08 | - | - | - | - | - | - | - |
| 21 | Geranyl acetate | 1381 | 0.01 | - | - | - | - | - | - | - | - | - |
| 22 | *cis*-Caryophyllene | 1397 | 0.01 | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Caryophyllene | 1411 | 5.19 | 4.90 | 4.74 | 3.76 | 3.27 | 3.16 | 3.42 | 3.13 | 3.29 | 3.58 |
| 24 | Aromadendrene | 1438 | 0.01 | - | - | - | - | - | - | - | - | - |
| 25 | α-Humulene | 1445 | 0.38 | 0.30 | 0.28 | 0.21 | 0.18 | - | - | 0.20 | 0.20 | - |
| 26 | β-Bisabolene | 1500 | 0.14 | 0.08 | - | - | - | - | - | - | - | - |
| 27 | δ-Cadinene | 1512 | 0.06 | - | - | - | - | - | - | - | - | - |
| 28 | Caryophyllene oxide | 1576 | 1.96 | 1.65 | 1.56 | 1.20 | 1.01 | 0.96 | 1.01 | 1.07 | 1.02 | 1.04 |
| 29 | Humulene epoxide | 1590 | 0.09 | 0.06 | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 6.57 | 5.59 | 5.56 | 3.82 | 3.34 | 3.51 | 3.28 | 4.41 | 4.36 | 3.12 |
| | Oxygenated monoterpenes | | 3.46 | 2.72 | 2.58 | 1.65 | 0.99 | 1.03 | 0.95 | 2.08 | 1.99 | 0.89 |
| | Phenolic monoterpenes | | 81.08 | 84.5 | 84.91 | 89.2 | 91.22 | 91.34 | 91.34 | 89.02 | 89.14 | 91.37 |
| | Sesquiterpene hydrocarbons | | 5.92 | 5.37 | 5.10 | 3.97 | 3.45 | 3.16 | 3.42 | 3.33 | 3.49 | 3.58 |
| | Oxygenated sesquiterpenes | | 2.05 | 1.71 | 1.56 | 1.20 | 1.01 | 0.96 | 1.01 | 1.07 | 1.02 | 1.04 |
| | Total identified | | 99.08 | 99.89 | 99.71 | 99.84 | 100 | 100 | 100 | 99.91 | 100 | 100 |

It is worth mentioning that most essential oils are either completely transparent or pale yellow. The yellow color is caused by bisabolol, *trans*-caryophyllene, carvone, linalool, or other yellowish compounds, and in some cases, the color derives from minor components that are <0.5% in the chemical load. Colored essential oil components are usually a consequence of the conjugation of double bonds, i.e., three or more double bonds in alternating sequences (e.g., eugenol) [11,12]. In addition, essential oil discoloration may result due to the oxidation/degradation of some ingredients, such as in the case of *trans*-caryophyllene (yellow color), which is oxidized to caryophyllene oxide (colorless) [13], resulting in the discoloration of the essential oils following their direct exposure to sunlight (see Figure 2B), while other compounds may also contribute in this phenomenon.

#### 3.1.2. *Thymus vulgaris* L.

As for the initial composition of *T. vulgaris* essential oil, a total of 45 chemical substances were identified (Table 6). The main compounds included thymol (55.77%), p-cymene (16.79%), γ-terpinene (6.32%), carvacrol (4.97%), linalool (3.72%), *trans*-caryophyllene (2.48%), α-terpinene (2.03%), terpinen-4-ol (1.83%), and borneol (1.44%). The essential oil exhibited a high content of phenolic monoterpenes (78.27%), significant amounts of monoterpene hydrocarbons (8.97%) and oxygenated monoterpenes (7.97%), and smaller quantities of sesquiterpene hydrocarbons (3.57%) and oxygenated sesquiterpenes (0.59%). These findings are consistent with essential oils derived from *Thymus* spp. growing wild in Greece [14].

During the stability control at room temperature without light exposure (in glass tubes) (Table 6), components with initial percentages lower than 0.10% were lost within the first 10 days. An exception was observed for *cis*-β-ocimene, which initially increased over time, indicating its formation during the breakdown of other substances in the mixture before finally starting to decrease, as eventually its degradation took place. This is probably due to the degradation of linalool, which produces *cis*-β-ocimene among others [15]. The chemical composition remained relatively stable until Day 70, except for the loss of γ-cadinene on Day 20. From Days 80 to 100, significant changes occurred, with multiple components disappearing from the sample. These included α-phellandrene, *cis*-sabinene hydrate, α-terpinolene, *trans*-sabinene hydrate, camphor, α-terpineol, aromadendrene, α-humulene, viridiflorene, δ-cadinene, and spathulenol. Finally, after 170 days, only the main compounds and *cis*-β-ocimene remained detectable, leading to an increase in thymol percentage from 58.56% in the initial sample to 68.03%.

In the experiment where *Thymi* aetheroleum was exposed to direct sunlight (Table 7), a similar pattern of composition change was observed, with significant alterations occurring within the first 10 days and in the time interval of 80 to 100 days. An increase in the percentage of *cis*-β-ocimene was also observed. After 6 months, the same substances as before (i.e., at room temperature without light exposure experiment) were detected, with a thymol percentage of 68.01%.

At high-temperature conditions of 35 °C and 45 °C (Tables 8 and 9, respectively), encapsulating the essential oil in ampoules demonstrated enhanced stability compared to placing it in capped glass tubes. After 3 months in glass ampoules, only α-terpineol was no longer detectable at both temperatures, while the other substances were well preserved, resulting in a small increase in thymol percentage (60.72% at 35 °C and 60.36% at 45 °C vs. 55.77% in the original essential oil). In contrast, rapid deterioration occurred when the sample was placed in glass tubes at the same temperatures. Within the first 10 to 30 days, most components were no longer detectable. After 3 months, only the main compounds and *cis*-β-ocimene remained in the essential oil, with thymol increasing to 65.16% at 35 °C and 65.57% at 45 °C.

**Table 6.** Chemical composition (%) of *Thymus vulgaris* L. essential oil during 6 months of storage in clear glass tubes sealed with cap at room temperature (23 °C) under darkness.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-Phellandrene | 1004 | 0.21 | 0.12 | 0.12 | 0.13 | 0.18 | 0.16 | 0.16 | 0.13 | 0.13 | 0.11 | - | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.07 | 0.07 | 0.07 | 0.07 | 0.08 | 0.07 | 0.07 | 0.07 | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 2.03 | 1.69 | 1.72 | 1.54 | 1.79 | 1.65 | 1.56 | 1.45 | 1.26 | 1.01 | 0.94 | 0.82 | 0.79 | 1.03 | 1.07 | 1.00 | 0.89 | 0.78 |
| 4 | p-Cymene | 1022 | 16.79 | 16.98 | 17.07 | 17.5 | 17.92 | 17.6 | 18.18 | 18.43 | 18.81 | 20.09 | 23.31 | 19.27 | 18.69 | 22.17 | 22.65 | 21.57 | 20.72 | 18.92 |
| 5 | *cis*-β-Ocimene | 1028 | 0.01 | 0.66 | 0.67 | 0.66 | 0.72 | 0.76 | 0.68 | 0.59 | 0.59 | 0.41 | 0.37 | 0.34 | 0.32 | 0.37 | 0.38 | 0.38 | 0.36 | 0.32 |
| 6 | *trans*-β-Ocimene | 1047 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 6.32 | 6.01 | 5.86 | 5.58 | 5.91 | 5.72 | 5.75 | 5.32 | 5.05 | 4.36 | 3.87 | 3.72 | 3.52 | 4.23 | 4.22 | 4.05 | 3.79 | 3.62 |
| 8 | *cis*-Sabinene hydrate | 1069 | 0.29 | 0.18 | 0.18 | 0.18 | 0.21 | 0.19 | 0.18 | 0.15 | 0.15 | 0.11 | - | - | - | - | - | - | - | - |
| 9 | α-Terpinolene | 1087 | 0.29 | 0.23 | 0.24 | 0.21 | 0.24 | 0.21 | 0.19 | 0.17 | 0.15 | 0.10 | - | - | - | - | - | - | - | - |
| 10 | 2,4-Decadienal | 1091 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | *trans*-Sabinene hydrate | 1094 | 0.04 | 0.06 | 0.06 | 0.08 | 0.08 | 0.08 | 0.08 | 0.06 | - | - | - | - | - | - | - | - | - | - |
| 12 | Linalool | 1101 | 3.72 | 3.37 | 3.36 | 3.17 | 3.39 | 3.21 | 3.22 | 2.98 | 2.84 | 2.21 | 1.76 | 1.82 | 1.76 | 2.16 | 2.10 | 2.05 | 1.95 | 1.87 |
| 13 | Camphor | 1134 | 0.09 | 0.06 | 0.28 | 0.18 | 0.17 | 0.06 | 0.09 | 0.06 | 0.04 | - | - | - | - | - | - | - | - | - |
| 14 | Borneol | 1157 | 1.44 | 1.14 | 1.21 | 1.08 | 1.15 | 1.15 | 1.11 | 0.99 | 0.91 | 0.69 | 0.51 | 0.59 | 0.57 | 0.67 | 0.64 | 0.65 | 0.60 | 0.63 |
| 15 | Terpinen-4-ol | 1169 | 1.83 | 1.05 | 1.12 | 1.01 | 1.09 | 1.06 | 1.02 | 0.93 | 0.85 | 0.64 | 0.47 | 0.53 | 0.54 | 0.62 | 0.59 | 0.59 | 0.56 | 0.58 |
| 16 | p-Cymen-8-ol | 1175 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | α-Terpineol | 1189 | 0.25 | 0.26 | 0.26 | 0.24 | 0.26 | 0.25 | 0.23 | 0.11 | - | - | - | - | - | - | - | - | - | - |
| 18 | Estragole | 1199 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | *trans*-Dihydro carvone | 1207 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Thymol, methyl ether | 1237 | 0.48 | 0.32 | 0.38 | 0.32 | 0.35 | 0.33 | 0.30 | 0.29 | 0.26 | 0.20 | 0.15 | 0.16 | 0.19 | 0.21 | 0.21 | - | - | - |
| 21 | Geranial | 1270 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | Carvone oxide | 1276 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | Thymol | 1292 | 55.77 | 58.56 | 57.86 | 59.19 | 57.75 | 58.82 | 58.87 | 59.67 | 61.15 | 63.97 | 63.89 | 67.61 | 68.18 | 63.67 | 63.38 | 64.75 | 66.11 | 68.03 |
| 24 | Carvacrol | 1301 | 4.97 | 5.58 | 5.53 | 5.45 | 5.27 | 5.49 | 5.24 | 5.27 | 4.84 | 4.11 | 3.25 | 3.57 | 3.81 | 3.51 | 3.56 | 3.70 | 3.75 | 3.93 |
| 25 | Thymol acetate | 1349 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Eugenol | 1351 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | Isoledene | 1365 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | α-Copaene | 1368 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Gurjunene | 1400 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | *trans*-Caryophyllene | 1410 | 2.48 | 2.12 | 2.15 | 2.07 | 2.02 | 1.99 | 1.96 | 1.95 | 1.75 | 1.50 | 1.26 | 1.34 | 1.39 | 1.18 | 1.20 | 1.26 | 1.27 | 1.32 |
| 31 | Aromandendrene | 1431 | 0.18 | 0.12 | 0.14 | 0.12 | 0.12 | 0.11 | 0.11 | 0.11 | 0.10 | - | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.15 | 0.09 | 0.10 | 0.11 | 0.11 | 0.12 | 0.08 | 0.11 | 0.12 | - | - | - | - | - | - | - | - | - |
| 33 | Alloaromadendrene | 1452 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | *cis*-Cadina-1(6),4-diene | 1462 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | γ-Muurolene | 1476 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | Viridiflorene | 1489 | 0.14 | 0.10 | 0.10 | 0.10 | 0.10 | 0.100 | 0.09 | 0.10 | 0.09 | - | - | - | - | - | - | - | - | - |
| 37 | α-Muurolene | 1494 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | γ-Cadinene | 1511 | 0.09 | 0.06 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 39 | *trans*-Calamenene | 1508 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 40 | δ-Cadinene | 1516 | 0.18 | 0.17 | 0.21 | 0.19 | 0.23 | 0.17 | 0.16 | 0.14 | 0.18 | 0.10 | - | - | - | - | - | - | - | - |
| 41 | Geranyl butanoate | 1555 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 42 | Spathulenol | 1571 | 0.15 | 0.09 | 0.11 | 0.10 | 0.10 | 0.09 | 0.08 | 0.09 | 0.08 | - | - | - | - | - | - | - | - | - |
| 43 | Caryophyllene oxide | 1576 | 0.43 | 0.30 | 0.34 | 0.30 | 0.29 | 0.30 | 0.31 | 0.34 | 0.32 | 0.26 | 0.22 | 0.23 | 0.24 | 0.18 | - | - | - | - |
| 44 | Geranyl 2-methyl butanoate | 1596 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 45 | Caryophylla-4(12),8(13)-dien-5α-ol | 1637 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |

**Table 6.** *Cont.*

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Monoterpene hydrocarbons | | 8.97 | 8.78 | 8.68 | 8.19 | 8.92 | 8.57 | 8.41 | 7.73 | 7.18 | 5.99 | 5.18 | 4.88 | 4.63 | 5.63 | 5.67 | 5.43 | 5.04 | 4.72 |
| | Oxygenated monoterpenes | | 7.97 | 6.12 | 6.47 | 5.94 | 6.35 | 6.00 | 5.93 | 5.28 | 4.79 | 3.65 | 2.74 | 2.94 | 2.87 | 3.45 | 3.33 | 3.29 | 3.11 | 3.08 |
| | Phenolic monoterpenes | | 78.27 | 81.44 | 80.84 | 82.46 | 81.29 | 82.24 | 82.59 | 83.66 | 85.06 | 88.37 | 90.60 | 90.61 | 90.87 | 89.56 | 89.8 | 90.02 | 90.58 | 90.88 |
| | Sesquiterpene hydrocarbons | | 3.57 | 2.66 | 2.77 | 2.59 | 2.58 | 2.49 | 2.40 | 2.41 | 2.24 | 1.60 | 1.26 | 1.34 | 1.39 | 1.18 | 1.20 | 1.26 | 1.27 | 1.32 |
| | Oxygenated sesquiterpenes | | 0.59 | 0.39 | 0.45 | 0.40 | 0.39 | 0.39 | 0.39 | 0.43 | 0.40 | 0.26 | 0.22 | 0.23 | 0.24 | 0.18 | - | - | - | - |
| | Total identified | | 99.37 | 99.39 | 99.21 | 99.58 | 99.53 | 99.69 | 99.72 | 99.51 | 99.67 | 99.87 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column. ** storage time in days.

**Table 7.** Chemical composition (%) of *Thymus vulgaris* L. essential oil during 6 months of storage in clear glass tubes sealed with cap under direct sunlight exposure.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-Phellandrene | 1004 | 0.21 | 0.20 | 0.12 | 0.13 | 0.15 | 0.13 | 0.15 | 0.14 | 0.12 | 0.11 | - | - | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.07 | 0.09 | 0.08 | 0.07 | 0.07 | 0.05 | 0.07 | 0.07 | 0.06 | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 2.03 | 1.89 | 1.75 | 1.59 | 1.49 | 1.30 | 1.45 | 1.46 | 1.31 | 1.05 | 0.90 | 0.82 | 0.85 | 0.96 | 1.00 | 0.95 | 0.88 | 0.88 |
| 4 | p-Cymene | 1022 | 16.79 | 17.4 | 17.57 | 17.57 | 16.79 | 16.03 | 17.00 | 18.41 | 19.47 | 20.13 | 19.71 | 19.2 | 19.07 | 20.94 | 21.43 | 21.23 | 20.82 | 18.54 |
| 5 | *cis*-β-Ocimene | 1028 | 0.01 | 0.70 | 0.69 | 0.69 | 0.68 | 0.58 | 0.63 | 0.64 | 0.53 | 0.42 | 0.36 | 0.33 | 0.33 | 0.35 | 0.36 | 0.36 | 0.34 | 0.35 |
| 6 | *trans*-β-Ocimene | 1047 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 6.32 | 6.19 | 6.02 | 5.62 | 5.41 | 5.11 | 5.34 | 5.36 | 5.25 | 4.49 | 4.08 | 3.74 | 3.88 | 4.12 | 4.14 | 3.94 | 3.76 | 3.72 |
| 8 | *cis*-Sabinene hydrate | 1069 | 0.29 | 0.20 | 0.19 | 0.18 | 0.17 | 0.17 | 0.16 | 0.16 | 0.14 | 0.11 | - | - | - | - | - | - | - | - |
| 9 | α-Terpinolene | 1087 | 0.29 | 0.27 | 0.24 | 0.21 | 0.19 | 0.17 | 0.17 | 0.17 | 0.16 | 0.10 | - | - | - | - | - | - | - | - |
| 10 | 2,4-Decadienal | 1091 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | *trans*-Sabinene hydrate | 1094 | 0.04 | 0.07 | 0.07 | 0.08 | 0.08 | 0.07 | 0.07 | 0.06 | - | - | - | - | - | - | - | - | - | - |
| 12 | Linalool | 1101 | 3.72 | 3.57 | 3.34 | 3.12 | 2.85 | 3.16 | 2.92 | 2.93 | 2.84 | 2.30 | 1.84 | 1.81 | 1.91 | 2.16 | 2.12 | 1.99 | 1.93 | 1.93 |
| 13 | Camphor | 1134 | 0.09 | 0.06 | 0.06 | 0.18 | 0.17 | 0.05 | 0.08 | 0.05 | 0.05 | - | - | - | - | - | - | - | - | - |
| 14 | Borneol | 1157 | 1.44 | 1.32 | 1.19 | 1.08 | 1.00 | 1.08 | 0.99 | 0.99 | 0.88 | 0.73 | 0.59 | 0.59 | 0.62 | 0.67 | 0.65 | 0.62 | 0.59 | 0.64 |
| 15 | Terpinen-4-ol | 1169 | 1.83 | 1.16 | 1.09 | 1.00 | 0.94 | 1.01 | 0.93 | 0.93 | 0.84 | 0.67 | 0.53 | 0.53 | 0.58 | 0.63 | 0.61 | 0.58 | 0.55 | 0.60 |
| 16 | p-Cymen-8-ol | 1175 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | α-Terpineol | 1189 | 0.25 | 0.30 | 0.24 | 0.24 | 0.24 | 0.24 | 0.11 | 0.11 | 0.10 | - | - | - | - | - | - | - | - | - |
| 18 | Estragole | 1199 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | *trans*-Dihydro carvone | 1207 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Thymol, methyl ether | 1237 | 0.48 | 0.36 | 0.35 | 0.31 | 0.28 | 0.10 | 0.28 | 0.28 | 0.26 | 0.21 | 0.21 | 0.16 | 0.20 | 0.22 | 0.21 | 0.20 | - | - |
| 21 | Geranial | 1270 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | Carvone oxide | 1276 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | Thymol | 1292 | 55.77 | 56.88 | 57.72 | 59.47 | 61.91 | 61.30 | 62.11 | 60.47 | 60.49 | 63.52 | 66.62 | 67.65 | 67.23 | 65.07 | 64.59 | 65.1 | 66.08 | 68.01 |
| 24 | Carvacrol | 1301 | 4.97 | 5.58 | 5.54 | 5.23 | 4.87 | 5.41 | 4.70 | 4.83 | 4.73 | 4.10 | 3.54 | 3.58 | 3.76 | 3.61 | 3.62 | 3.70 | 3.72 | 3.90 |
| 25 | Thymol acetate | 1349 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Eugenol | 1351 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | Isoledene | 1365 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | α-Copaene | 1368 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Gurjunene | 1400 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | *trans*-Caryophyllene | 1410 | 2.48 | 2.13 | 2.12 | 1.98 | 1.80 | 2.14 | 1.77 | 1.83 | 1.80 | 1.60 | 1.39 | 1.38 | 1.37 | 1.26 | 1.27 | 1.32 | 1.33 | 1.43 |
| 31 | Aromandendrene | 1431 | 0.18 | 0.13 | 0.12 | 0.11 | 0.10 | 0.12 | 0.10 | 0.12 | 0.10 | - | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.15 | 0.11 | 0.10 | 0.11 | 0.11 | 0.10 | 0.08 | 0.07 | 0.10 | - | - | - | - | - | - | - | - | - |

**Table 7.** *Cont.*

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 33 | Alloaromadendrene | 1452 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | *cis*-Cadina-1(6),4-diene | 1462 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | γ-Muurolene | 1476 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | Viridiflorene | 1489 | 0.14 | 0.11 | 0.10 | 0.09 | 0.08 | 0.10 | 0.08 | 0.09 | 0.09 | - | - | - | - | - | - | - | - | - |
| 37 | α-Muurolene | 1494 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | γ-Cadinene | 1511 | 0.09 | 0.06 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 39 | *trans*-Calamenene | 1515 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 40 | δ-Cadinene | 1516 | 0.18 | 0.17 | 0.18 | 0.18 | 0.23 | 0.27 | 0.16 | 0.20 | 0.13 | 0.10 | - | - | - | - | - | - | - | - |
| 41 | Geranyl butanoate | 1555 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 42 | Spathulenol | 1571 | 0.15 | 0.10 | 0.12 | 0.09 | 0.09 | 0.09 | 0.07 | 0.07 | 0.08 | - | - | - | - | - | - | - | - | - |
| 43 | Caryophyllene oxide | 1576 | 0.43 | 0.30 | 0.30 | 0.28 | 0.25 | 0.32 | 0.26 | 0.28 | 0.30 | 0.23 | 0.23 | 0.19 | 0.19 | - | - | - | - | - |
| 44 | Geranyl 2-methyl butanoate | 1596 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 45 | Caryophylla-4(12),8(13)-dien-5α-ol | 1637 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 8.97 | 9.34 | 8.90 | 8.31 | 7.99 | 7.34 | 7.81 | 7.84 | 7.43 | 6.17 | 5.34 | 4.89 | 5.06 | 5.43 | 5.50 | 5.25 | 4.98 | 4.95 |
| | Oxygenated monoterpenes | | 7.97 | 6.68 | 6.18 | 5.88 | 5.45 | 5.78 | 5.26 | 5.23 | 4.85 | 3.81 | 2.96 | 2.93 | 3.11 | 3.46 | 3.38 | 3.19 | 3.07 | 3.17 |
| | Phenolic monoterpenes | | 78.27 | 80.22 | 81.18 | 82.58 | 83.85 | 82.84 | 84.09 | 83.99 | 84.95 | 87.96 | 90.08 | 90.59 | 90.26 | 89.84 | 89.85 | 90.23 | 90.62 | 90.45 |
| | Sesquiterpene hydrocarbons | | 3.57 | 2.71 | 2.68 | 2.47 | 2.32 | 2.73 | 2.19 | 2.31 | 2.22 | 1.70 | 1.39 | 1.38 | 1.37 | 1.26 | 1.27 | 1.32 | 1.33 | 1.43 |
| | Oxygenated sesquiterpenes | | 0.59 | 0.40 | 0.42 | 0.37 | 0.34 | 0.41 | 0.33 | 0.35 | 0.38 | 0.23 | 0.23 | 0.19 | 0.19 | - | - | - | - | - |
| | Total identified | | 99.37 | 99.35 | 99.36 | 99.61 | 99.95 | 99.1 | 99.68 | 99.52 | 99.83 | 99.87 | 100 | 99.98 | 99.99 | 99.99 | 100 | 99.99 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 8.** Chemical composition (%) of *Thymus vulgaris* L. essential oil during 3 months of storage in sealed glass ampoules at a temperature of 35 °C and 45 °C.

| | | | | 35 °C | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** |
| 1 | α-Phellandrene | 1004 | 0.21 | 0.12 | 0.09 | 0.15 | 0.16 | 0.15 | 0.17 | 0.16 | 0.14 | 0.13 | 0.05 | 0.15 | 0.16 | 0.18 | 0.17 | 0.16 | 0.15 |
| 2 | δ-3-Carene | 1006 | 0.07 | 0.06 | 0.07 | 0.07 | 0.07 | 0.07 | 0.07 | 0.07 | 0.06 | 0.08 | 0.07 | 0.07 | 0.07 | 0.07 | 0.07 | 0.07 | 0.06 |
| 3 | α-Terpinene | 1012 | 2.03 | 1.57 | 1.48 | 1.70 | 1.57 | 1.57 | 1.58 | 1.56 | 1.37 | 1.80 | 0.88 | 1.75 | 1.64 | 1.68 | 1.60 | 1.54 | 1.39 |
| 4 | p-Cymene | 1022 | 16.79 | 16.30 | 18.29 | 17.36 | 16.15 | 16.59 | 17.18 | 18.45 | 18.62 | 17.38 | 19.57 | 17.49 | 16.96 | 17.37 | 16.26 | 18.25 | 18.62 |
| 5 | *cis*-β-Ocimene | 1028 | 0.01 | 0.67 | 0.67 | 0.68 | 0.69 | 0.63 | 0.64 | 0.66 | 0.53 | 0.73 | 0.65 | 0.68 | 0.72 | 0.71 | 0.65 | 0.62 | 0.51 |
| 6 | *trans*-β-Ocimene | 1047 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 6.32 | 5.84 | 4.83 | 5.99 | 5.89 | 5.68 | 5.97 | 6.05 | 5.79 | 6.35 | 2.89 | 6.12 | 5.96 | 6.21 | 5.91 | 5.9 | 5.82 |
| 8 | *cis*-Sabinene hydrate | 1069 | 0.29 | 0.18 | 0.18 | 0.18 | 0.18 | 0.18 | 0.17 | 0.17 | 0.14 | 0.20 | 0.17 | 0.18 | 0.18 | 0.19 | 0.16 | 0.15 | 0.13 |
| 9 | α-Terpinolene | 1087 | 0.29 | 0.23 | 0.22 | 0.22 | 0.20 | 0.20 | 0.19 | 0.18 | 0.17 | 0.25 | 0.22 | 0.24 | 0.20 | 0.21 | 0.20 | 0.14 | 0.17 |
| 10 | 2,4-Decadienal | 1091 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | *trans*-Sabinene hydrate | 1094 | 0.04 | 0.06 | 0.07 | 0.07 | 0.09 | 0.07 | 0.06 | 0.08 | - | 0.08 | 0.07 | 0.06 | 0.08 | 0.08 | 0.07 | 0.04 | - |
| 12 | Linalool | 1101 | 3.72 | 3.34 | 3.28 | 3.14 | 3.11 | 3.13 | 3.03 | 3.05 | 2.87 | 3.42 | 3.40 | 3.32 | 3.09 | 3.23 | 3.04 | 2.87 | 2.83 |
| 13 | Camphor | 1134 | 0.09 | 0.14 | 0.08 | 0.08 | 0.19 | 0.19 | 0.05 | 0.17 | 0.05 | 0.24 | 0.27 | 0.27 | 0.19 | 0.20 | 0.08 | 0.08 | 0.05 |
| 14 | Borneol | 1157 | 1.44 | 1.15 | 1.12 | 1.08 | 1.11 | 1.14 | 1.00 | 1.01 | 0.88 | 1.19 | 1.25 | 1.25 | 1.08 | 1.13 | 1.02 | 0.96 | 0.91 |
| 15 | Terpinen-4-ol | 1169 | 1.83 | 1.08 | 1.05 | 1.01 | 1.03 | 1.06 | 0.96 | 0.95 | 0.86 | 1.11 | 1.11 | 1.10 | 1.03 | 1.04 | 0.98 | 0.90 | 0.87 |
| 16 | p-Cymen-8-ol | 1175 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | α-Terpineol | 1189 | 0.25 | 0.27 | 0.27 | 0.24 | 0.23 | 0.26 | 0.12 | - | - | 0.26 | 0.27 | 0.26 | 0.22 | 0.23 | 0.11 | - | - |
| 18 | Estragole | 1199 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | *trans*-Dihydro carvone | 1207 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Thymol, methyl ether | 1237 | 0.48 | 0.35 | 0.35 | 0.33 | 0.30 | 0.30 | 0.29 | 0.28 | 0.25 | 0.34 | 0.34 | 0.37 | 0.30 | 0.31 | 0.29 | 0.26 | 0.26 |

**Table 8.** *Cont.*

| No. | Compound | KI * | 1st ** | 35 °C |  |  |  |  |  |  |  | 45 °C |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** |
| 21 | Geranial | 1270 | 0.07 | 0.06 | 0.05 | - | - | - | - | - | - | 0.08 | - | - | - | - | - | - | - |
| 22 | Carvone oxide | 1276 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | Thymol | 1292 | 55.77 | 59.16 | 58.84 | 59.15 | 60.67 | 60.1 | 60.89 | 59.14 | 60.72 | 57.29 | 58.88 | 57.38 | 59.65 | 58.8 | 61.37 | 60.51 | 60.36 |
| 24 | Carvacrol | 1301 | 4.97 | 5.58 | 5.49 | 5.11 | 5.26 | 5.27 | 4.84 | 4.93 | 4.75 | 5.55 | 5.77 | 5.52 | 5.27 | 5.18 | 4.99 | 4.72 | 4.77 |
| 25 | Thymol acetate | 1349 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Eugenol | 1351 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | Isoledene | 1365 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | α-Copaene | 1368 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Gurjunene | 1400 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | *trans*-Caryophyllene | 1410 | 2.48 | 2.17 | 1.88 | 2.01 | 1.96 | 1.96 | 1.87 | 1.93 | 1.82 | 2.06 | 1.34 | 2.12 | 1.97 | 1.96 | 1.87 | 1.83 | 1.80 |
| 31 | Aromandendrene | 1431 | 0.18 | 0.12 | 0.12 | 0.11 | 0.11 | 0.11 | 0.10 | 0.10 | 0.10 | 0.12 | 0.13 | 0.14 | 0.10 | 0.11 | 0.10 | 0.09 | 0.10 |
| 32 | α-Humulene | 1445 | 0.15 | 0.11 | 0.08 | 0.13 | 0.13 | 0.13 | 0.10 | 0.10 | 0.10 | 0.10 | 0.08 | 0.14 | 0.11 | 0.07 | 0.11 | 0.07 | 0.11 |
| 33 | Alloaromadendrene | 1452 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | *cis*-Cadina-1(6),4-diene | 1462 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | γ-Muurolene | 1476 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | Viridiflorene | 1489 | 0.14 | 0.10 | 0.09 | 0.10 | 0.09 | 0.09 | 0.09 | 0.09 | 0.09 | 0.10 | 0.07 | 0.10 | 0.09 | 0.09 | 0.09 | 0.09 | 0.09 |
| 37 | α-Muurolene | 1494 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | γ-Cadinene | 1511 | 0.09 | 0.06 | 0.06 | 0.05 | - | - | - | - | - | 0.06 | 0.06 | 0.07 | - | - | - | - | - |
| 39 | *trans*-Calamenene | 1515 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 40 | δ-Cadinene | 1516 | 0.18 | 0.19 | 0.16 | 0.21 | 0.20 | 0.21 | 0.14 | 0.21 | 0.15 | 0.17 | 0.21 | 0.23 | 0.22 | 0.20 | 0.15 | 0.17 | 0.18 |
| 41 | Geranyl butanoate | 1555 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 42 | Spathulenol | 1571 | 0.15 | 0.11 | 0.09 | 0.10 | 0.08 | 0.09 | 0.07 | 0.07 | 0.08 | 0.10 | 0.10 | 0.12 | 0.08 | 0.09 | 0.08 | 0.06 | 0.08 |
| 43 | Caryophyllene oxide | 1576 | 0.43 | 0.34 | 0.49 | 0.28 | 0.26 | 0.3 | 0.26 | 0.27 | 0.27 | 0.30 | 1.08 | 0.32 | 0.26 | 0.26 | 0.26 | 0.25 | 0.26 |
| 44 | Geranyl 2-methyl butanoate | 1596 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 45 | Caryophylla-4(12),8(13)-dien-5α-ol | 1637 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
|  | Monoterpene hydrocarbons |  | 8.97 | 8.49 | 7.36 | 8.81 | 8.58 | 8.30 | 8.62 | 8.68 | 8.06 | 9.34 | 4.76 | 9.01 | 8.75 | 9.06 | 8.60 | 8.43 | 8.10 |
|  | Oxygenated monoterpenes |  | 7.97 | 6.28 | 6.10 | 5.80 | 5.94 | 6.03 | 5.39 | 5.43 | 4.80 | 6.58 | 6.54 | 6.44 | 5.87 | 6.00 | 5.46 | 5.00 | 4.90 |
|  | Phenolic monoterpenes |  | 78.27 | 81.39 | 82.97 | 81.95 | 82.38 | 82.26 | 83.2 | 82.8 | 84.34 | 80.56 | 84.56 | 80.76 | 82.18 | 81.66 | 82.91 | 83.74 | 84.01 |
|  | Sesquiterpene hydrocarbons |  | 3.57 | 2.75 | 2.39 | 2.61 | 2.36 | 2.50 | 2.30 | 2.33 | 2.26 | 2.61 | 1.89 | 2.80 | 2.38 | 2.43 | 2.32 | 2.25 | 2.28 |
|  | Oxygenated sesquiterpenes |  | 0.59 | 0.45 | 0.58 | 0.38 | 0.34 | 0.39 | 0.33 | 0.34 | 0.35 | 0.40 | 1.18 | 0.44 | 0.34 | 0.35 | 0.34 | 0.31 | 0.34 |
|  | Total identified |  | 99.37 | 99.36 | 99.4 | 99.55 | 99.73 | 99.48 | 99.84 | 99.68 | 99.81 | 99.49 | 98.93 | 99.45 | 99.63 | 99.6 | 99.63 | 99.73 | 99.52 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 9.** Chemical composition (%) of *Thymus vulgaris* L. essential oil during 3 months of storage in clear glass tubes sealed with cap at a temperature of 35 °C and 45 °C.

| No. | Compound | KI * | 1st ** | 35 °C |  |  |  |  |  |  |  | 45 °C |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** |
| 1 | α-Phellandrene | 1004 | 0.21 | 0.13 | 0.12 | - | - | - | - | - | - | 0.14 | 0.12 | - | - | - | - | - | - |
| 2 | δ-3-Carene | 1006 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 2.03 | 1.36 | 1.08 | 0.87 | 0.85 | 0.87 | 1.00 | 1.03 | 0.99 | 1.34 | 1.08 | 0.87 | 0.85 | 0.88 | 1.00 | 1.04 | 0.98 |
| 4 | p-Cymene | 1022 | 16.79 | 19.53 | 19.8 | 19.56 | 19.09 | 18.82 | 20.87 | 21.65 | 20.98 | 19.02 | 19.88 | 19.38 | 19.19 | 18.83 | 21.13 | 21.44 | 20.97 |
| 5 | *cis*-β-Ocimene | 1028 | 0.01 | 0.58 | 0.43 | 0.33 | 0.33 | 0.32 | 0.36 | 0.35 | 0.39 | 0.62 | 0.42 | 0.34 | 0.33 | 0.33 | 0.35 | 0.39 | 0.37 |

**Table 9.** *Cont.*

| | | | | 35 °C | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 6 | *trans*-β-Ocimene | 1047 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 6.32 | 5.25 | 4.61 | 3.88 | 3.80 | 3.89 | 4.14 | 4.14 | 4.02 | 5.27 | 4.62 | 3.86 | 3.79 | 3.89 | 4.15 | 4.11 | 3.87 |
| 8 | *cis*-Sabinene hydrate | 1069 | 0.29 | 0.15 | 0.11 | - | - | - | - | - | - | 0.15 | 0.11 | - | - | - | - | - | - |
| 9 | α-Terpinolene | 1087 | 0.29 | 0.15 | 0.13 | - | - | - | - | - | - | 0.15 | 0.11 | - | - | - | - | - | - |
| 10 | 2,4-Decadienal | 1091 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | *trans*-Sabinene hydrate | 1094 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 12 | Linalool | 1101 | 3.72 | 2.79 | 2.37 | 1.82 | 1.82 | 1.90 | 2.14 | 2.08 | 2.01 | 2.76 | 2.29 | 1.81 | 1.83 | 1.92 | 2.16 | 2.10 | 1.96 |
| 13 | Camphor | 1134 | 0.09 | - | - | - | - | - | - | - | - | 0.15 | - | - | - | - | - | - | - |
| 14 | Borneol | 1157 | 1.44 | 0.88 | 0.73 | 0.58 | 0.59 | 0.62 | 0.67 | 0.65 | 0.64 | 0.85 | 0.73 | 0.59 | 0.59 | 0.63 | 0.67 | 0.66 | 0.61 |
| 15 | Terpinen-4-ol | 1169 | 1.83 | 0.82 | 0.68 | 0.53 | 0.54 | 0.58 | 0.63 | 0.61 | 0.59 | 0.81 | 0.67 | 0.54 | 0.54 | 0.59 | 0.63 | 0.62 | 0.58 |
| 16 | p-Cymen-8-ol | 1175 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | α-Terpineol | 1189 | 0.25 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 18 | Estragole | 1199 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | *trans*-Dihydro carvone | 1207 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Thymol, methyl ether | 1237 | 0.48 | 0.23 | 0.21 | 0.16 | 0.16 | 0.20 | 0.22 | 0.21 | - | 0.24 | 0.20 | 0.16 | 0.16 | 0.20 | 0.22 | 0.21 | - |
| 21 | Geranial | 1270 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | Carvone oxide | 1276 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | Thymol | 1292 | 55.77 | 60.59 | 63.44 | 67.2 | 67.67 | 67.44 | 65.08 | 64.45 | 65.16 | 61.23 | 63.62 | 67.33 | 67.59 | 67.56 | 64.84 | 64.52 | 65.57 |
| 24 | Carvacrol | 1301 | 4.97 | 4.66 | 4.15 | 3.51 | 3.57 | 3.78 | 3.62 | 3.56 | 3.83 | 4.58 | 4.04 | 3.55 | 3.56 | 3.78 | 3.58 | 3.62 | 3.74 |
| 25 | Thymol acetate | 1349 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Eugenol | 1351 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | Isoledene | 1365 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | α-Copaene | 1368 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Gurjunene | 1400 | 0.02 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | *trans*-Caryophyllene | 1410 | 2.48 | 1.85 | 1.60 | 1.38 | 1.40 | 1.40 | 1.27 | 1.27 | 1.39 | 1.78 | 1.59 | 1.39 | 1.39 | 1.39 | 1.27 | 1.28 | 1.35 |
| 31 | Aromandendrene | 1431 | 0.18 | 0.10 | 0.09 | - | - | - | - | - | - | 0.09 | 0.08 | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.15 | - | - | - | - | - | - | - | - | 0.09 | - | - | - | - | - | - | - |
| 33 | Alloaromadendrene | 1452 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | *cis*-Cadina-1(6),4-diene | 1462 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | γ-Muurolene | 1476 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | Viridiflorene | 1489 | 0.14 | 0.09 | - | - | - | - | - | - | - | 0.09 | - | - | - | - | - | - | - |
| 37 | α-Muurolene | 1494 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | γ-Cadinene | 1511 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 39 | *trans*-Calamenene | 1515 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 40 | δ-Cadinene | 1516 | 0.18 | 0.16 | 0.11 | - | - | - | - | - | - | 0.14 | 0.10 | - | - | - | - | - | - |
| 41 | Geranyl butanoate | 1555 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 42 | Spathulenol | 1571 | 0.15 | 0.09 | - | - | - | - | - | - | - | 0.08 | - | - | - | - | - | - | - |
| 43 | Caryophyllene oxide | 1576 | 0.43 | 0.27 | 0.22 | 0.18 | 0.18 | 0.18 | - | - | - | 0.27 | 0.21 | 0.17 | 0.17 | - | - | - | - |
| 44 | Geranyl 2-methyl butanoate | 1596 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 45 | Caryophylla-4(12),8(13)-dien-5α-ol | 1637 | 0.01 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 8.97 | 7.47 | 6.37 | 5.08 | 4.98 | 5.08 | 5.50 | 5.52 | 5.40 | 7.52 | 6.35 | 5.07 | 4.97 | 5.10 | 5.5 | 5.54 | 5.22 |
| | Oxygenated monoterpenes | | 7.97 | 4.64 | 3.89 | 2.93 | 2.95 | 3.10 | 3.44 | 3.34 | 3.24 | 4.72 | 3.80 | 2.94 | 2.96 | 3.14 | 3.46 | 3.38 | 3.15 |
| | Phenolic monoterpenes | | 78.27 | 85.01 | 87.6 | 90.43 | 90.49 | 90.24 | 89.79 | 89.87 | 89.97 | 85.07 | 87.74 | 90.42 | 90.5 | 90.37 | 89.77 | 89.79 | 90.28 |
| | Sesquiterpene hydrocarbons | | 3.57 | 2.20 | 1.80 | 1.38 | 1.40 | 1.40 | 1.27 | 1.27 | 1.39 | 2.19 | 1.77 | 1.39 | 1.39 | 1.39 | 1.27 | 1.28 | 1.35 |
| | Oxygenated sesquiterpenes | | 0.59 | 0.36 | 0.22 | 0.18 | 0.18 | 0.18 | - | - | - | 0.35 | 0.21 | 0.17 | 0.17 | - | - | - | - |
| | Total identified | | 99.37 | 99.68 | 99.88 | 100 | 100 | 100 | 100 | 100 | 100 | 99.85 | 99.87 | 99.99 | 99.99 | 100 | 100 | 99.99 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 10.** Chemical composition (%) of *Thymus vulgaris* L. essential oil during 6 months of storage at 4 °C (in clear glass tubes sealed with cap, in metal containers and in sealed glass ampoules) and at −20 °C (in clear glass tubes sealed with cap).

| | | | | | | 4 °C Glass | | | | 4 °C Metal | 4°C Ampoules | −20 °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compounds** | **KI *** | **1st **** | **30th** | **60th** | **90th** | **120th** | **150th** | **180th** | **180th** | **180th** | **180th** |
| 1 | α-Phellandrene | 1004 | 0.21 | 0.14 | 0.13 | 0.12 | - | - | - | - | 0.15 | 0.15 |
| 2 | δ-3-Carene | 1006 | 0.07 | 0.07 | 0.07 | - | - | - | - | - | - | - |
| 3 | α-Terpinene | 1012 | 2.03 | 1.64 | 1.56 | 1.04 | 0.88 | 1.20 | 0.83 | 1.07 | 1.31 | 1.34 |
| 4 | p-Cymene | 1022 | 16.79 | 17.15 | 16.71 | 18.99 | 18.43 | 22.45 | 18.26 | 20.89 | 21.8 | 22.98 |
| 5 | *cis*-β-Ocimene | 1028 | 0.01 | 0.67 | 0.65 | 0.4 | 0.32 | 0.40 | 0.32 | 0.37 | 0.46 | 0.48 |
| 6 | *trans*-β-Ocimene | 1047 | 0.04 | - | - | - | - | - | - | - | - | - |
| 7 | γ-Terpinene | 1054 | 6.32 | 5.72 | 5.79 | 4.80 | 4.22 | 4.86 | 4.16 | 4.71 | 5.46 | 5.09 |
| 8 | *cis*-Sabinene hydrate | 1069 | 0.29 | 0.18 | 0.17 | 0.11 | - | - | - | - | 0.13 | 0.14 |
| 9 | α-Terpinolene | 1087 | 0.29 | 0.22 | 0.20 | 0.11 | - | - | - | - | 0.17 | 0.17 |
| 10 | 2,4-Decadienal | 1091 | 0.02 | - | - | - | - | - | - | - | - | - |
| 11 | *trans*-Sabinene hydrate | 1094 | 0.04 | 0.07 | 0.07 | - | - | - | - | - | - | - |
| 12 | Linalool | 1101 | 3.72 | 3.21 | 3.19 | 2.33 | 1.90 | 2.15 | 1.90 | 2.08 | 2.42 | 2.44 |
| 13 | Camphor | 1134 | 0.09 | 0.04 | 0.08 | - | - | - | - | - | 0.11 | 0.11 |
| 14 | Borneol | 1157 | 1.44 | 1.12 | 1.10 | 0.74 | 0.61 | 0.66 | 0.64 | 0.67 | 0.80 | 0.81 |
| 15 | Terpinen-4-ol | 1169 | 1.83 | 1.03 | 1.04 | 0.68 | 0.57 | 0.61 | 0.58 | 0.60 | 0.71 | 0.73 |
| 16 | p-Cymen-8-ol | 1175 | 0.07 | - | - | - | - | - | - | - | - | - |
| 17 | α-Terpineol | 1189 | 0.25 | 0.25 | 0.23 | - | - | - | - | - | 0.10 | 0.09 |
| 18 | Estragole | 1199 | 0.06 | - | - | - | - | - | - | - | - | - |
| 19 | *trans*-Dihydro carvone | 1207 | 0.07 | - | - | - | - | - | - | - | - | - |
| 20 | Thymol, methyl ether | 1237 | 0.48 | 0.34 | 0.33 | 0.21 | 0.20 | 0.21 | 0.20 | 0.20 | 0.24 | 0.24 |
| 21 | Geranial | 1270 | 0.07 | 0.04 | - | - | - | - | - | - | - | - |
| 22 | Carvone oxide | 1276 | 0.04 | - | - | - | - | - | - | - | - | - |
| 23 | Thymol | 1292 | 55.77 | 59.13 | 60.15 | 64.07 | 67.57 | 62.69 | 67.75 | 64.39 | 60.22 | 59.80 |
| 24 | Carvacrol | 1301 | 4.97 | 5.32 | 5.11 | 4.10 | 3.73 | 3.50 | 3.92 | 3.70 | 4.32 | 3.81 |
| 25 | Thymol acetate | 1349 | 0.05 | - | - | - | - | - | - | - | - | - |
| 26 | Eugenol | 1351 | 0.08 | - | - | - | - | - | - | - | - | - |
| 27 | Isoledene | 1365 | 0.02 | - | - | - | - | - | - | - | - | - |
| 28 | α-Copaene | 1368 | 0.04 | - | - | - | - | - | - | - | - | - |
| 29 | α-Gurjunene | 1400 | 0.02 | - | - | - | - | - | - | - | - | - |
| 30 | *trans*-Caryophyllene | 1410 | 2.48 | 2.06 | 1.94 | 1.56 | 1.39 | 1.27 | 1.44 | 1.32 | 1.41 | 1.33 |
| 31 | Aromandendrene | 1431 | 0.18 | 0.12 | 0.11 | 0.08 | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.15 | 0.16 | 0.13 | - | - | - | - | - | - | - |
| 33 | Alloaromadendrene | 1452 | 0.07 | - | - | - | - | - | - | - | - | - |
| 34 | *cis*-Cadina-1(6),4-diene | 1462 | 0.04 | - | - | - | - | - | - | - | - | - |
| 35 | γ-Muurolene | 1476 | 0.06 | - | - | - | - | - | - | - | - | - |
| 36 | Viridiflorene | 1489 | 0.14 | 0.10 | 0.09 | - | - | - | - | - | - | - |
| 37 | α-Muurolene | 1494 | 0.05 | - | - | - | - | - | - | - | - | - |
| 38 | γ-Cadinene | 1511 | 0.09 | 0.06 | - | - | - | - | - | - | - | - |
| 39 | *trans*-Calamenene | 1515 | 0.05 | - | - | - | - | - | - | - | - | - |
| 40 | δ-Cadinene | 1516 | 0.18 | 0.19 | 0.21 | 0.15 | - | - | - | - | - | 0.08 |
| 41 | Geranyl butanoate | 1555 | 0.08 | - | - | - | - | - | - | - | - | - |
| 42 | Spathulenol | 1571 | 0.15 | 0.10 | 0.09 | - | - | - | - | - | - | - |
| 43 | Caryophyllene oxide | 1576 | 0.43 | 0.30 | 0.30 | 0.21 | 0.17 | - | - | - | 0.18 | 0.20 |
| 43 | Geranyl 2-methyl butanoate | 1596 | 0.03 | - | - | - | - | - | - | - | - | - |
| 45 | Caryophylla-4(12),8(13)-dien-5α-ol | 1637 | 0.01 | - | - | - | - | - | - | - | - | - |

**Table 10.** *Cont.*

| | | | 4 °C Glass | | | | | | | 4 °C Metal | 4°C Ampoules | −20 °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compounds** | **KI *** | **1st **** | **30th** | **60th** | **90th** | **120th** | **150th** | **180th** | **180th** | **180th** | **180th** |
| | Monoterpene hydrocarbons | | 8.97 | 8.46 | 8.40 | 6.47 | 5.42 | 6.46 | 5.31 | 6.15 | 7.55 | 7.23 |
| | Oxygenated monoterpenes | | 7.97 | 5.94 | 5.88 | 3.86 | 3.08 | 3.42 | 3.12 | 3.35 | 4.27 | 4.32 |
| | Phenolic monoterpenes | | 78.27 | 81.94 | 82.3 | 87.37 | 89.93 | 88.85 | 90.13 | 89.18 | 86.58 | 86.83 |
| | Sesquiterpene hydrocarbons | | 3.57 | 2.69 | 2.48 | 1.79 | 1.39 | 1.27 | 1.44 | 1.32 | 1.41 | 1.41 |
| | Oxygenated sesquiterpenes | | 0.59 | 0.40 | 0.39 | 0.21 | 0.17 | - | - | - | 0.18 | 0.20 |
| | Total identified | | 99.37 | 99.43 | 99.45 | 99.7 | 99.99 | 100 | 100 | 100 | 99.99 | 99.99 |

* Kovats index on HP-5 column; ** storage time in days.

When investigating the chemical load of *T. vulgaris* essential oil under refrigeration conditions of 4 °C and −20 °C, it is notable that the preservation of its composition was similar to the above experiment (in high-temperature conditions) (Table 10), without significant improvement compared to other conditions, as it would be expected. In glass tubes at 4 °C, the number of undetected components increased in each monthly measurement, and after 6 months, the compounds were reduced to the main ones along with *trans*-β-ocimene and thymol methyl ether. The percentage of thymol in this measurement was 67.75%. The same substances were found in the sample stored in a metal container at the same temperature after 6 months. However, in the latter case, a smaller increase in the percentage of thymol (64.39%) was observed, while a significant increase in p-cymene (20.89% from 16.79% in the original sample) was noted. On the contrary, the sample stored in a glass ampoule exhibited better stability at the same temperature. Apart from the substances preserved in the previous two conditions, α-phellandrene, *cis*-sabinene hydrate, α-terpinolene, camphor, α-terpineol, and caryophyllene oxide were also detected after 6 months. In this condition, the percentage of thymol increased to 60.22%, and the percentage of p-cymene also showed a significant increase (21.80%). Furthermore, increased retention of the substances was observed at −20 °C in glass tubes compared to the same container at 4 °C. After 6 months, more compounds remained in the sample at −20 °C, including α-phellandrene, *cis*-sabinene hydrate, α-terpinolene, camphor, α-terpineol, caryophyllene oxide, and δ-cadinene. Moreover, the percentage of thymol was 59.80%, and the largest increase was observed in the percentage of p-cymene at 22.98%. It is worth mentioning that p-cymene is speculated to increase as a result of the transformation of α-phellandrene, α- and γ-terpinene, as well as in other cases of limonene [16].

The essential oil of *T. vulgaris* had a bright orange color. It is interesting that, after storage under direct sunlight, the intensity of the color decreased. No significant difference in color was observed during storage in the glass tubes at high temperatures of 35 °C and 45 °C (probably due to the initial color), while during storage in the ampoules at 45 °C after 3 months and at 35 °C and 45 °C after 6 months, the color turned to dark orange-red (Figure 2).

#### 3.1.3. *Mentha spicata* L.

In the essential oil of *M. spicata* (spearmint), a total of 39 compounds were identified (Table 11). The major constituents included carvone (73.69%), limonene (10.09%), terpinen-4-ol (ca. 2.28%), menthol (ca. 2.28%), *cis*-dihydrocarvone (2.24%), caryophyllene (1.75%), and p-menthone (0.96%). The composition of the essential oil indicates a high content of oxygenated monoterpenes (84.92%), followed by monoterpene hydrocarbons (10.09%), sesquiterpene hydrocarbons (3.74%), and traces of oxygenated sesquiterpenes (0.6%), and phenolic monoterpenes (0.27%). These findings are in agreement with reports of the percentage composition of this particular essential oil in the literature [17].

Under room temperature conditions and in the absence of light (in glass tubes) (Table 11), several components initially present in low proportions no longer appeared in the mixture after Day 10. It is worth noting that during the degradation of spearmint essential oil, certain compounds that are not originally present in the chemical composition are produced, namely neo-menthol, menthol, α-terpineol, and carveol. Specifically, α-terpineol results from sabinene hydrate with C-C bond cleavage [16], neo-menthol and menthol are produced from limonene or in other cases pulegone [18], and carveol is probably derived from limonene [19]. The chemical accumulation remains during the period of 10 to 30 days, while in the measurement after 40 days, isopulegol, carveol, and *cis*-β-farnesene are no longer detected. Subsequently, between 70 and 120 days, additional substances disappear from the sample, including *trans*-dihydrocarvone, neo-menthol, α-, iso-menthol, p-cymene, and caryophyllene oxide. Finally, in the last four measurements (Days 140–170), nine components are detected in the sample, namely limonene, p-menthone, menthol, terpinen-4-ol, *cis*-dihydrocarvone, carvone, dihydro carveol acetate, β-bourbonene, and caryophyllene, with carvone being the predominant compound, constituting 88.85% of the mixture.

**Table 11.** Chemical composition (%) of *Mentha spicata* L. essential oil during 6 months of storage in clear glass tubes sealed with cap at room temperature (23 °C) under darkness.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | p-Cymene | 1017 | 0.27 | 0.45 | 0.23 | 0.25 | 0.22 | 0.22 | 0.23 | 0.23 | 0.17 | 0.12 | 0.11 | - | - | - | - | - | - | - |
| 2 | Limonene | 1022 | 10.09 | 9.86 | 10.29 | 10.38 | 10.18 | 10.45 | 10.28 | 10.03 | 8.79 | 8.37 | 7.53 | 6.95 | 7.02 | 8.84 | 8.56 | 7.63 | 7.61 | 7.06 |
| 3 | *cis*-Sabinene hydrate | 1069 | 0.11 | 0.28 | 0.08 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 4 | *trans*-p-Mentha-2,8-dien-1-ol | 1115 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | *cis*-Limonene oxide | 1126 | 0.09 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 6 | *trans*-Limonene oxide | 1130 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | Isopulegol | 1138 | 0.15 | 0.11 | 0.08 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 8 | p-Menthone | 1144 | 0.96 | 0.88 | 0.75 | 0.8 | 0.67 | 0.64 | 0.65 | 0.64 | 0.46 | 0.38 | 0.34 | 0.31 | 0.31 | 0.44 | 0.40 | 0.32 | 0.35 | 0.34 |
| 9 | Isomenthone | 1154 | 0.69 | 0.39 | 0.32 | 0.34 | 0.27 | 0.25 | 0.26 | 0.26 | 0.19 | 0.06 | - | - | - | - | - | - | - | - |
| 10 | Neomenthol | 1156 | - | 0.24 | 0.19 | 0.21 | 0.18 | 0.17 | 0.16 | 0.17 | - | - | - | - | - | - | - | - | - | - |
| 11 | Menthol | 1165 | - | 3.01 | 2.55 | 2.72 | 2.32 | 2.26 | 2.14 | 2.22 | 1.60 | 1.29 | 1.21 | 1.01 | 0.98 | 1.37 | 1.26 | 1.06 | 1.05 | 0.98 |
| 12 | Terpinen-4-ol + Menthol | 1169 | 4.56 | 1.24 | 1.10 | 1.18 | 1.03 | 0.99 | 0.94 | 0.96 | 0.70 | 0.60 | 0.55 | 0.48 | 0.47 | 0.61 | 0.57 | 0.51 | 0.51 | 0.49 |
| 13 | α-Terpineol | 1183 | - | 0.63 | 0.46 | 0.52 | 0.47 | 0.40 | 0.43 | 0.41 | 0.24 | 0.18 | 0.23 | - | - | - | - | - | - | - |
| 14 | *cis*-Dihydrocarvone | 1189 | 2.24 | 1.88 | 1.72 | 1.82 | 1.51 | 1.34 | 1.41 | 1.34 | 0.93 | 0.75 | 0.78 | 0.70 | 0.69 | 1.11 | 0.79 | 0.70 | 0.86 | 0.73 |
| 15 | *trans*-Dihydrocarvone | 1195 | 0.84 | 0.21 | 0.21 | 0.16 | 0.17 | 0.16 | 0.16 | - | - | - | - | - | - | - | - | - | - | - |
| 16 | Carveol | 1213 | - | 0.12 | 0.16 | 0.16 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Carvone | 1233 | 73.69 | 75.71 | 77.1 | 76.44 | 78.89 | 79.63 | 78.83 | 80.09 | 84.200 | 86.21 | 87.19 | 88.62 | 88.89 | 86.05 | 86.96 | 88.32 | 88.15 | 88.85 |
| 18 | Isopiperitone | 1255 | 0.37 | 0.39 | 0.38 | 0.39 | 0.43 | 0.35 | 0.35 | 0.34 | 0.21 | 0.17 | 0.18 | 0.18 | 0.17 | 0.17 | - | - | - | - |
| 19 | Carvenone | 1256 | tr | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Menthyl acetate | 1290 | 0.25 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | Dihydro carveol acetate | 1303 | 0.61 | 0.51 | 0.5 | 0.50 | 0.44 | 0.41 | 0.40 | 0.40 | 0.34 | 0.28 | 0.28 | 0.28 | 0.28 | 0.26 | 0.27 | 0.26 | 0.26 | 0.29 |
| 22 | Neoisoverbanol acetate | 1330 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Carveyl acetate | 1336 | 0.16 | 0.13 | 0.11 | 0.10 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 24 | α-Copaene | 1367 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | β-Bourbonene | 1379 | 0.83 | 0.71 | 0.71 | 0.70 | 0.67 | 0.60 | 0.58 | 0.6 | 0.49 | 0.41 | 0.42 | 0.41 | 0.40 | 0.37 | 0.38 | 0.40 | 0.40 | 0.45 |
| 26 | β-Elemene | 1384 | 0.16 | 0.13 | 0.12 | 0.12 | 0.11 | 0.10 | 0.10 | 0.11 | - | - | - | - | - | - | - | - | - | - |
| 27 | *trans*-Jasmone | 1387 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | *cis*-Caryophyllene | 1397 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | *trans*-Caryophyllene | 1410 | 1.75 | 1.48 | 1.50 | 1.51 | 1.40 | 1.27 | 1.24 | 1.26 | 1.09 | 0.90 | 0.91 | 0.8 | 0.79 | 0.77 | 0.80 | 0.80 | 0.81 | 0.81 |
| 30 | β-Copaene | 1423 | 0.17 | 0.14 | 0.15 | 0.15 | 0.13 | 0.13 | 0.12 | - | - | - | - | - | - | - | - | - | - | - |
| 31 | Aromadendrene | 1432 | 0.10 | 0.08 | 0.07 | 0.10 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.19 | 0.15 | 0.15 | 0.15 | 0.15 | 0.12 | 0.12 | 0.13 | 0.11 | - | - | - | - | - | - | - | - | - |
| 33 | *cis*-β-Farnesene | 1452 | 0.18 | 0.13 | 0.10 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | Germacrene D | 1477 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | α-Amorphene | 1482 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | α-Muurolene | 1494 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 37 | δ-Cadinene | 1515 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | Caryophyllene oxide | 1576 | 0.55 | 0.45 | 0.44 | 0.44 | 0.37 | 0.33 | 0.37 | 0.39 | 0.36 | 0.28 | 0.27 | 0.25 | - | - | - | - | - | - |
| 39 | Viridiflorol | 1584 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 10.09 | 9.86 | 10.29 | 10.38 | 10.18 | 10.45 | 10.28 | 10.03 | 8.79 | 8.37 | 7.53 | 6.95 | 7.02 | 8.84 | 8.56 | 7.63 | 7.61 | 7.06 |
| | Oxygenated monoterpenes | | 84.92 | 85.8 | 85.71 | 85.51 | 86.38 | 86.6 | 85.73 | 86.83 | 88.87 | 89.92 | 90.76 | 91.58 | 91.79 | 90.01 | 90.25 | 91.17 | 91.18 | 91.68 |
| | Phenolic monoterpenes | | 0.27 | 0.45 | 0.23 | 0.25 | 0.22 | 0.22 | 0.23 | 0.23 | 0.17 | 0.12 | 0.11 | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.74 | 2.82 | 2.80 | 2.82 | 2.00 | 2.22 | 2.16 | 2.10 | 1.69 | 1.31 | 1.33 | 1.21 | 1.19 | 1.14 | 1.18 | 1.20 | 1.21 | 1.26 |
| | Oxygenated sesquiterpenes | | 0.60 | 0.45 | 0.44 | 0.44 | 0.37 | 0.33 | 0.37 | 0.39 | 0.36 | 0.28 | 0.27 | 0.25 | - | - | - | - | - | - |
| | Total identified | | 99.62 | 99.38 | 99.47 | 99.4 | 99.75 | 99.82 | 98.77 | 99.58 | 99.88 | 100 | 100 | 99.99 | 100 | 99.99 | 99.99 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 12.** Chemical composition (%) of *Mentha spicata* L. essential oil during 6 months of storage in clear glass tubes sealed with cap under direct sunlight exposure.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | p-Cymene | 1017 | 0.27 | 0.40 | 0.26 | 0.27 | 0.22 | 0.25 | 0.23 | 0.23 | 0.23 | 0.12 | 0.11 | - | - | - | - | - | - | - |
| 2 | Limonene | 1022 | 10.09 | 10.72 | 10.69 | 10.37 | 10.21 | 9.37 | 10.31 | 10.19 | 8.24 | 7.8 | 7.55 | 6.98 | 7.18 | 8.26 | 8.35 | 7.73 | 7.67 | 7.29 |
| 3 | *cis*-Sabinene hydrate | 1069 | 0.11 | 0.13 | 0.09 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 4 | *trans*-p-Mentha-2,8-dien-1-ol | 1115 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | *cis*-Limonene oxide | 1126 | 0.09 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 6 | *trans*-Limonene oxide | 1130 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | Isopulegol | 1138 | 0.15 | 0.09 | 0.09 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 8 | p-Menthone | 1144 | 0.96 | 0.74 | 0.79 | 0.81 | 0.60 | 0.60 | 0.65 | 0.67 | 0.45 | 0.35 | 0.33 | 0.31 | 0.36 | 0.44 | 0.42 | 0.33 | 0.35 | 0.33 |
| 9 | Isomenthone | 1154 | 0.69 | 0.33 | 0.34 | 0.34 | 0.22 | 0.24 | 0.26 | 0.27 | - | - | - | - | - | - | - | - | - | - |
| 10 | Neomenthol | 1156 | - | 0.19 | 0.20 | 0.20 | 0.16 | 0.16 | 0.16 | 0.18 | 0.05 | 0.05 | - | - | - | - | - | - | - | - |
| 11 | Menthol | 1165 | - | 2.55 | 2.65 | 2.73 | 2.17 | 2.09 | 2.16 | 2.33 | 1.59 | 1.21 | 1.18 | 1.03 | 1.06 | 1.40 | 1.33 | 1.08 | 1.08 | 1.02 |
| 12 | Terpinen-4-ol + Menthol | 1169 | 4.56 | 1.08 | 1.12 | 1.17 | 0.94 | 0.91 | 0.93 | 1.00 | 0.69 | 0.57 | 0.55 | 0.49 | 0.50 | 0.61 | 0.59 | 0.51 | 0.51 | 0.48 |
| 13 | α-Terpineol | 1183 | - | 0.53 | 0.55 | 0.49 | 0.40 | 0.35 | 0.45 | 0.46 | 0.32 | 0.18 | 0.22 | - | - | - | - | - | - | - |
| 14 | *cis*-Dihydrocarvone | 1189 | 2.24 | 1.64 | 1.74 | 1.80 | 1.27 | 1.19 | 1.40 | 1.52 | 1.01 | 0.73 | 0.75 | 0.70 | 0.73 | 0.81 | 0.86 | 0.76 | 0.69 | 0.71 |
| 15 | *trans*-Dihydrocarvone | 1195 | 0.84 | 0.18 | 0.19 | 0.22 | 0.22 | 0.16 | 0.16 | 0.18 | - | - | - | - | - | - | - | - | - | - |
| 16 | Carveol | 1213 | - | 0.17 | 0.17 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Carvone | 1233 | 73.69 | 76.85 | 76.32 | 76.48 | 80.08 | 80.83 | 79.94 | 79.04 | 84.81 | 86.98 | 87.33 | 88.73 | 88.4 | 86.71 | 87.11 | 88.26 | 88.37 | 88.74 |
| 18 | Isopiperitone | 1255 | 0.37 | 0.36 | 0.39 | 0.40 | 0.55 | 0.30 | 0.33 | 0.34 | 0.23 | 0.17 | 0.17 | - | - | - | - | - | - | - |
| 19 | Carvenone | 1256 | TR | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Menthyl acetate | 1290 | 0.25 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | Dihydro carveol acetate | 1303 | 0.61 | 0.47 | 0.49 | 0.50 | 0.38 | 0.43 | 0.40 | 0.43 | 0.32 | 0.28 | 0.28 | 0.27 | 0.28 | 0.27 | 0.27 | 0.27 | 0.27 | 0.30 |
| 22 | Neoisoverbanol acetate | 1330 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Carveyl acetate | 1336 | 0.16 | 0.11 | 0.11 | 0.10 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 24 | α-Copaene | 1367 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | β-Bourbonene | 1379 | 0.83 | 0.67 | 0.70 | 0.71 | 0.58 | 0.65 | 0.59 | 0.65 | 0.48 | 0.44 | 0.44 | 0.42 | 0.42 | 0.38 | 0.40 | 0.41 | 0.41 | 0.44 |
| 26 | β-Elemene | 1384 | 0.16 | 0.12 | 0.12 | 0.13 | 0.11 | 0.11 | 0.1 | 0.11 | - | - | - | - | - | - | - | - | - | - |
| 27 | *trans*-Jasmone | 1387 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | *cis*-Caryophyllene | 1397 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | *trans*-Caryophyllene | 1410 | 1.75 | 1.37 | 1.40 | 1.38 | 1.18 | 1.19 | 1.14 | 1.24 | 0.95 | 0.82 | 0.79 | 0.77 | 0.75 | 0.65 | 0.67 | 0.65 | 0.65 | 0.69 |
| 30 | β-Copaene | 1423 | 0.17 | 0.13 | 0.14 | 0.15 | 0.13 | 0.13 | 0.12 | 0.13 | - | - | - | - | - | - | - | - | - | - |
| 31 | Aromadendrene | 1432 | 0.10 | 0.07 | 0.09 | 0.11 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.19 | 0.11 | 0.14 | 0.14 | 0.12 | 0.12 | 0.12 | 0.13 | - | - | - | - | - | - | - | - | - | - |
| 33 | *cis*-β-Farnesene | 1452 | 0.18 | 0.10 | 0.10 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | Germacrene D | 1477 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | α-Amorphene | 1482 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | α-Muurolene | 1494 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 37 | δ-Cadinene | 1515 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | Caryophyllene oxide | 1576 | 0.55 | 0.43 | 0.45 | 0.52 | 0.30 | 0.49 | 0.40 | 0.46 | 0.33 | 0.31 | 0.30 | 0.30 | 0.31 | 0.29 | - | - | - | - |
| 39 | Viridiflorol | 1584 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 10.09 | 10.72 | 10.69 | 10.37 | 10.21 | 9.37 | 10.31 | 10.19 | 8.24 | 7.80 | 7.55 | 6.98 | 7.18 | 8.26 | 8.35 | 7.73 | 7.67 | 7.29 |
| | Oxygenated monoterpenes | | 84.92 | 85.50 | 85.24 | 85.58 | 86.99 | 87.26 | 86.84 | 86.42 | 89.47 | 90.17 | 90.81 | 91.53 | 91.33 | 90.24 | 90.58 | 91.21 | 91.27 | 91.58 |
| | Phenolic monoterpenes | | 0.27 | 0.40 | 0.26 | 0.27 | 0.22 | 0.25 | 0.23 | 0.23 | 0.23 | 0.12 | 0.11 | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.74 | 2.57 | 2.69 | 2.69 | 2.12 | 2.20 | 2.07 | 2.26 | 1.43 | 1.26 | 1.23 | 1.19 | 1.17 | 1.03 | 1.07 | 1.06 | 1.06 | 1.13 |
| | Oxygenated sesquiterpenes | | 0.60 | 0.43 | 0.45 | 0.52 | 0.30 | 0.49 | 0.40 | 0.46 | 0.33 | 0.31 | 0.30 | 0.30 | 0.31 | 0.29 | - | - | - | - |
| | Total identified | | 99.62 | 99.62 | 99.33 | 99.43 | 99.84 | 99.57 | 99.85 | 99.56 | 99.7 | 99.66 | 100 | 100 | 99.99 | 99.82 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 13.** Chemical composition (%) of *Mentha spicata* L. essential oil during 3 months of storage in sealed glass ampoules at a temperature of 35 °C and 45 °C.

| | | | | **35 °C** | | | | | | | | **45 °C** | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 1 | p-Cymene | 1017 | 0.27 | 0.41 | 0.27 | 0.27 | 0.21 | 0.26 | 0.23 | 0.24 | 0.22 | 0.42 | 0.23 | 0.26 | 0.21 | 0.25 | 0.24 | 0.23 | 0.20 |
| 2 | Limonene | 1022 | 10.09 | 10.48 | 11.09 | 10.96 | 9.89 | 9.33 | 10.22 | 10.14 | 8.12 | 10.05 | 9.84 | 10.6 | 10.46 | 8.53 | 10.09 | 10.19 | 8.43 |
| 3 | *cis*-Sabinene hydrate | 1069 | 0.11 | 0.23 | 0.08 | 0.10 | 0.10 | 0.10 | 0.08 | - | - | 0.22 | 0.09 | 0.09 | 0.09 | 0.09 | 0.07 | - | - |
| 4 | *trans*-p-Mentha-2,8-dien-1-ol | 1115 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | *cis*-Limonene oxide | 1126 | 0.09 | - | - | - | - | - | - | - | - | 0.09 | 0.09 | - | - | - | - | - | - |
| 6 | *trans*-Limonene oxide | 1130 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | Isopulegol | 1138 | 0.15 | 0.10 | 0.10 | 0.10 | - | - | - | - | - | 0.10 | 0.09 | 0.09 | - | - | - | - | - |
| 8 | p-Menthone | 1144 | 0.96 | 0.85 | 0.76 | 0.83 | 0.67 | 0.77 | 0.69 | 0.65 | 0.43 | 0.82 | 0.73 | 0.78 | 0.64 | 0.72 | 0.69 | 0.64 | 0.43 |
| 9 | Isomenthone | 1154 | 0.69 | 0.37 | 0.31 | 0.35 | 0.26 | 0.32 | 0.27 | 0.25 | - | 0.35 | 0.29 | 0.33 | 0.24 | 0.29 | 0.27 | 0.25 | - |
| 10 | Neomenthol | 1156 | - | 0.22 | 0.19 | 0.22 | 0.17 | 0.19 | 0.17 | 0.17 | - | 0.21 | 0.18 | 0.20 | 0.19 | 0.19 | 0.17 | 0.16 | - |
| 11 | Menthol | 1165 | - | 2.83 | 2.54 | 2.73 | 2.35 | 2.62 | 2.25 | 2.23 | 1.54 | 2.82 | 2.55 | 2.63 | 2.28 | 2.42 | 2.29 | 2.22 | 1.56 |
| 12 | Terpinen-4-ol + Menthol | 1169 | 4.56 | 1.19 | 1.08 | 1.16 | 1.02 | 1.03 | 0.99 | 0.98 | 0.67 | 1.16 | 1.02 | 1.12 | 0.99 | 0.99 | 1.01 | 0.96 | 0.69 |
| 13 | α-Terpineol | 1183 | - | 0.57 | 0.54 | 0.57 | 0.47 | 0.54 | 0.43 | 0.45 | 0.33 | 0.57 | 0.50 | 0.56 | 0.42 | 0.52 | 0.45 | 0.38 | 0.31 |
| 14 | *cis*-Dihydrocarvone | 1189 | 2.24 | 1.84 | 1.66 | 1.78 | 1.49 | 1.68 | 1.51 | 1.46 | 1.00 | 1.81 | 1.65 | 1.71 | 1.35 | 1.60 | 1.54 | 1.30 | 0.99 |
| 15 | *trans*-Dihydrocarvone | 1195 | 0.84 | 0.21 | 0.19 | 0.21 | 0.16 | 0.17 | 0.18 | 0.16 | - | 0.20 | 0.18 | 0.19 | 0.16 | 0.16 | 0.18 | - | - |
| 16 | Carveol | 1213 | - | 0.18 | 0.18 | 0.17 | 0.17 | 0.20 | 0.15 | - | - | 0.18 | 0.18 | 0.17 | 0.17 | 0.19 | 0.12 | - | - |
| 17 | Carvone | 1233 | 73.69 | 75.71 | 76.89 | 75.83 | 79.43 | 78.95 | 78.96 | 79.53 | 85.36 | 76.26 | 77.89 | 76.54 | 79.27 | 79.79 | 79.07 | 80.06 | 84.98 |
| 18 | Isopiperitone | 1255 | 0.37 | 0.39 | 0.43 | 0.43 | 0.46 | 0.31 | 0.33 | 0.35 | 0.27 | 0.40 | 0.31 | 0.40 | 0.40 | 0.30 | 0.32 | 0.29 | 0.22 |
| 19 | Carvenone | 1256 | tr | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Menthyl acetate | 1290 | 0.25 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | Dihydro carveol acetate | 1303 | 0.61 | 0.51 | 0.45 | 0.49 | 0.44 | 0.46 | 0.42 | 0.42 | 0.30 | 0.51 | 0.47 | 0.48 | 0.40 | 0.45 | 0.42 | 0.41 | 0.32 |
| 22 | Neoisoverbanol acetate | 1330 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Carveyl acetate | 1336 | 0.16 | 0.12 | 0.12 | 0.12 | 0.10 | 0.10 | - | - | - | 0.12 | 0.10 | 0.11 | - | - | - | - | - |
| 24 | α-Copaene | 1367 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | β-Bourbonene | 1379 | 0.83 | 0.70 | 0.64 | 0.69 | 0.63 | 0.63 | 0.63 | 0.60 | 0.47 | 0.73 | 0.69 | 0.69 | 0.59 | 0.64 | 0.64 | 0.59 | 0.47 |
| 26 | β-Elemene | 1384 | 0.16 | 0.13 | 0.11 | 0.12 | 0.11 | 0.11 | 0.11 | 0.11 | - | 0.13 | 0.12 | 0.12 | 0.11 | 0.11 | 0.11 | 0.10 | - |
| 27 | *trans*-Jasmone | 1387 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | *cis*-Caryophyllene | 1397 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | *trans*-Caryophyllene | 1410 | 1.75 | 1.50 | 1.15 | 1.43 | 1.10 | 0.61 | 1.33 | 1.29 | 1.04 | 1.51 | 0.75 | 1.41 | 1.30 | 0.86 | 1.32 | 1.27 | 1.04 |
| 30 | β-Copaene | 1423 | 0.17 | 0.15 | 0.13 | 0.14 | 0.13 | 0.11 | 0.13 | 0.12 | - | 0.15 | 0.13 | 0.14 | 0.13 | 0.13 | 0.13 | 0.12 | - |
| 31 | Aromadendrene | 1432 | 0.10 | 0.08 | 0.09 | 0.09 | - | - | - | - | - | 0.09 | 0.07 | 0.07 | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.19 | 0.15 | 0.12 | 0.15 | 0.13 | 0.10 | 0.13 | 0.13 | - | 0.15 | 0.11 | 0.14 | 0.12 | 0.12 | 0.13 | 0.12 | - |
| 33 | *cis*-β-Farnesene | 1452 | 0.18 | 0.13 | 0.12 | 0.12 | - | - | - | - | - | 0.12 | 0.10 | 0.10 | - | - | - | - | - |
| 34 | Germacrene D | 1477 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | α-Amorphene | 1482 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | α-Muurolene | 1494 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 37 | δ-Cadinene | 1515 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | Caryophyllene oxide | 1576 | 0.55 | 0.45 | 0.44 | 0.44 | 0.34 | 0.99 | 0.39 | 0.38 | 0.25 | 0.48 | 0.96 | 0.43 | 0.32 | 0.80 | 0.40 | 0.38 | 0.29 |
| 39 | Viridiflorol | 1584 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 10.09 | 10.48 | 11.09 | 10.96 | 9.89 | 9.33 | 10.22 | 10.14 | 8.12 | 10.05 | 9.84 | 10.6 | 10.46 | 8.53 | 10.09 | 10.19 | 8.43 |
| | Oxygenated monoterpenes | | 84.92 | 85.32 | 85.52 | 85.09 | 87.29 | 87.44 | 86.43 | 86.65 | 89.90 | 85.82 | 86.32 | 85.4 | 86.6 | 87.71 | 86.6 | 86.67 | 89.5 |
| | Phenolic monoterpenes | | 0.27 | 0.41 | 0.27 | 0.27 | 0.21 | 0.26 | 0.23 | 0.24 | 0.22 | 0.42 | 0.23 | 0.26 | 0.21 | 0.25 | 0.24 | 0.23 | 0.20 |
| | Sesquiterpene hydrocarbons | | 3.74 | 2.84 | 2.36 | 2.74 | 2.10 | 1.56 | 2.33 | 2.25 | 1.51 | 2.88 | 1.97 | 2.67 | 2.25 | 1.86 | 2.33 | 2.20 | 1.51 |
| | Oxygenated sesquiterpenes | | 0.60 | 0.45 | 0.44 | 0.44 | 0.34 | 0.99 | 0.39 | 0.38 | 0.25 | 0.48 | 0.96 | 0.43 | 0.32 | 0.80 | 0.40 | 0.38 | 0.29 |
| | Total identified | | 99.62 | 99.5 | 99.68 | 99.5 | 99.83 | 99.58 | 99.6 | 99.66 | 100 | 99.65 | 99.32 | 99.36 | 99.84 | 99.15 | 99.66 | 99.67 | 99.93 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 14.** Chemical composition (%) of *Mentha spicata* L. essential oil during 3 months of storage in clear glass tubes sealed with cap at a temperature of 35 °C and 45 °C.

| | | | 35 °C | | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 1 | p-Cymene | 1017 | 0.27 | 0.25 | 0.17 | 0.11 | - | - | - | - | - | 0.21 | 0.15 | 0.11 | - | - | - | - | - |
| 2 | Limonene | 1022 | 10.09 | 10.68 | 8.72 | 7.55 | 6.94 | 7.14 | 8.18 | 8.24 | 7.65 | 9.52 | 9.06 | 7.46 | 6.94 | 7.13 | 8.17 | 8.23 | 7.61 |
| 3 | *cis*-Sabinene hydrate | 1069 | 0.11 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 4 | *trans*-p-Mentha-2,8-dien-1-ol | 1115 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | *cis*-Limonene oxide | 1126 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 6 | *trans*-Limonene oxide | 1130 | 0.08 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 7 | Isopulegol | 1138 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 8 | p-Menthone | 1144 | 0.96 | 0.69 | 0.44 | 0.33 | 0.32 | 0.33 | 0.44 | 0.42 | 0.35 | 0.56 | 0.47 | 0.32 | 0.32 | 0.33 | 0.45 | 0.41 | 0.34 |
| 9 | Isomenthone | 1154 | 0.69 | 0.29 | 0.06 | - | - | - | - | - | - | 0.23 | 0.19 | - | - | - | - | - | - |
| 10 | Neomenthol | 1156 | - | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 11 | Menthol | 1165 | - | 2.40 | 1.56 | 1.18 | 1.03 | 1.07 | 1.41 | 1.32 | 1.07 | 1.91 | 1.63 | 1.17 | 1.03 | 1.07 | 1.42 | 1.29 | 1.07 |
| 12 | Terpinen-4-ol + Menthol | 1169 | 4.56 | 0.96 | 0.68 | 0.54 | 0.49 | 0.49 | 0.61 | 0.59 | 0.51 | 0.81 | 0.73 | 0.54 | 0.49 | 0.50 | 0.62 | 0.57 | 0.50 |
| 13 | α-Terpineol | 1183 | - | 0.41 | 0.30 | - | - | - | - | - | - | 0.38 | 0.25 | - | - | - | - | - | - |
| 14 | *cis*-Dihydrocarvone | 1189 | 2.24 | 1.35 | 0.99 | 0.76 | 0.70 | 0.75 | 0.82 | 0.85 | 0.88 | 1.20 | 0.93 | 0.76 | 0.69 | 0.73 | 0.87 | 0.80 | 0.85 |
| 15 | *trans*-Dihydrocarvone | 1195 | 0.84 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Carvone | 1233 | 73.69 | 79.77 | 84.69 | 87.49 | 88.51 | 88.21 | 86.69 | 87.17 | 88.13 | 82.34 | 83.84 | 87.62 | 88.7 | 88.42 | 86.80 | 86.99 | 87.91 |
| 18 | Isopiperitone | 1255 | 0.37 | 0.25 | 0.24 | 0.17 | 0.16 | 0.16 | 0.17 | - | - | 0.26 | 0.22 | 0.18 | - | - | - | - | - |
| 19 | Carvenone | 1256 | tr | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 20 | Menthyl acetate | 1290 | 0.25 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | Dihydro carveol acetate | 1303 | 0.61 | 0.38 | 0.32 | 0.27 | 0.27 | 0.28 | 0.27 | 0.28 | 0.27 | 0.36 | 0.35 | 0.28 | 0.27 | 0.28 | 0.27 | 0.27 | 0.27 |
| 22 | Neoisoverbanol acetate | 1330 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Carveyl acetate | 1336 | 0.16 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 24 | α-Copaene | 1367 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | β-Bourbonene | 1379 | 0.83 | 0.53 | 0.47 | 0.43 | 0.43 | 0.41 | 0.38 | 0.39 | 0.40 | 0.52 | 0.52 | 0.42 | 0.41 | 0.42 | 0.38 | 0.39 | 0.39 |
| 26 | β-Elemene | 1384 | 0.16 | 0.10 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | *trans*-Jasmone | 1387 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | cis-Caryophyllene | 1397 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | *trans*-Caryophyllene | 1410 | 1.75 | 1.09 | 1.02 | 0.86 | 0.84 | 0.83 | 0.72 | 0.74 | 0.74 | 1.09 | 1.07 | 0.83 | 0.8 | 0.77 | 0.68 | 0.69 | 0.70 |
| 30 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 31 | Aromadendrene | 1432 | 0.1 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.19 | 0.11 | - | - | - | - | - | - | - | 0.11 | 0.11 | - | - | - | - | - | - |
| 33 | *cis*-β-Farnesene | 1452 | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 34 | Germacrene D | 1477 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 35 | α-Amorphene | 1482 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 36 | α-Muurolene | 1494 | 0.03 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 37 | δ-Cadinene | 1515 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 38 | Caryophyllene oxide | 1576 | 0.55 | 0.38 | 0.33 | 0.30 | 0.31 | 0.33 | 0.31 | - | - | 0.38 | 0.36 | 0.32 | 0.34 | 0.35 | 0.34 | 0.35 | 0.36 |
| 39 | Viridiflorol | 1584 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 10.09 | 10.68 | 8.72 | 7.55 | 6.94 | 7.14 | 8.18 | 8.24 | 7.65 | 9.52 | 9.06 | 7.46 | 6.94 | 7.13 | 8.17 | 8.23 | 7.61 |
| | Oxygenated monoterpenes | | 84.92 | 86.68 | 89.28 | 90.74 | 91.48 | 91.29 | 90.41 | 90.63 | 91.21 | 88.05 | 88.61 | 90.87 | 91.5 | 91.33 | 90.43 | 90.33 | 90.94 |
| | Phenolic monoterpenes | | 0.27 | 0.25 | 0.17 | 0.11 | - | - | - | - | - | 0.21 | 0.15 | 0.11 | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.74 | 1.83 | 1.49 | 1.29 | 1.27 | 1.24 | 1.10 | 1.13 | 1.14 | 1.72 | 1.70 | 1.25 | 1.21 | 1.19 | 1.06 | 1.08 | 1.09 |
| | Oxygenated sesquiterpenes | | 0.60 | 0.38 | 0.33 | 0.30 | 0.31 | 0.33 | 0.31 | - | - | 0.38 | 0.36 | 0.32 | 0.34 | 0.35 | 0.34 | 0.35 | 0.36 |
| | Total identified | | 99.62 | 99.82 | 99.99 | 99.99 | 100 | 100 | 100 | 100 | 100 | 99.88 | 99.88 | 99.9 | 99.99 | 100 | 100 | 99.99 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 15.** Chemical composition (%) of *Mentha spicata* L. essential oil during 6 months of storage at 4 °C (in clear glass tubes sealed with cap, in metal containers, and in sealed glass ampoules) and at −20 °C (in clear glass tubes sealed with cap).

| | | | 4 °C Glass | | | | | | | 4 °C Metal | 4 °C Ampoules | −20 °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **30th** | **60th** | **90th** | **120th** | **150th** | **180th** | **180th** | **180th** | **180th** |
| 1 | p-Cymene | 1017 | 0.27 | 0.25 | 0.23 | - | - | - | - | 0.17 | 0.16 | - |
| 2 | Limonene | 1022 | 10.09 | 10.08 | 9.69 | 6.84 | 7.22 | 7.30 | 7.30 | 10.18 | 9.34 | 7.41 |
| 3 | *cis*-Sabinene hydrate | 1069 | 0.11 | 0.09 | - | - | - | - | - | - | - | - |
| 4 | *trans*-p-Mentha-2,8-dien-1-ol | 1115 | 0.06 | - | - | - | - | - | - | - | - | - |
| 5 | *cis*-Limonene oxide | 1126 | 0.09 | - | - | - | - | - | - | - | - | - |
| 6 | *trans*-Limonene oxide | 1130 | 0.08 | - | - | - | - | - | - | - | - | - |
| 7 | Isopulegol | 1138 | 0.15 | 0.08 | - | - | - | - | - | - | - | - |
| 8 | p-Menthone | 1144 | 0.96 | 0.82 | 0.64 | 0.30 | 0.36 | 0.33 | 0.33 | 0.53 | 0.52 | 0.33 |
| 9 | Isomenthone | 1154 | 0.69 | 0.35 | 0.26 | - | - | - | - | 0.12 | 0.13 | - |
| 10 | Neomenthol | 1156 | - | 0.21 | 0.17 | - | - | - | - | - | - | - |
| 11 | Menthol | 1165 | - | 2.76 | 2.24 | 0.99 | 1.06 | 0.98 | 0.97 | 1.51 | 1.52 | 1.01 |
| 12 | Terpinen-4-ol + Menthol | 1169 | 4.56 | 1.21 | 0.97 | 0.48 | 0.50 | 0.50 | 0.49 | 0.84 | 0.80 | 0.49 |
| 13 | α-Terpineol | 1183 | - | 0.50 | 0.42 | - | - | - | - | 0.31 | 0.27 | - |
| 14 | *cis*-Dihydrocarvone | 1189 | 2.24 | 1.89 | 1.35 | 0.66 | 0.75 | 0.70 | 0.67 | 1.34 | 0.94 | 0.67 |
| 15 | *trans*-Dihydrocarvone | 1195 | 0.84 | 0.84 | - | - | - | - | - | - | - | - |
| 16 | Carveol | 1213 | - | 0.18 | - | - | - | - | - | - | - | - |
| 17 | Carvone | 1235 | 73.69 | 76.09 | 80.72 | 88.93 | 88.47 | 88.59 | 88.62 | 82.81 | 84.16 | 88.35 |
| 18 | Isopiperitone | 1255 | 0.37 | 0.36 | 0.29 | 0.16 | - | - | - | 0.18 | 0.20 | - |
| 19 | Carvenone | 1256 | tr | - | - | - | - | - | - | - | - | - |
| 20 | Menthyl acetate | 1290 | 0.25 | - | - | - | - | - | - | 0.11 | - | - |
| 21 | Dihydro carveol acetate | 1303 | 0.61 | 0.51 | 0.40 | 0.26 | 0.28 | 0.28 | 0.29 | 0.31 | 0.31 | 0.29 |
| 22 | Neoisoverbanol acetate | 1330 | 0.06 | - | - | - | - | - | - | - | - | - |
| 23 | *trans*-Carveyl acetate | 1336 | 0.16 | 0.11 | - | - | - | - | - | - | - | - |
| 24 | α-Copaene | 1367 | 0.06 | - | - | - | - | - | - | - | - | - |
| 25 | β-Bourbonene | 1379 | 0.83 | 0.73 | 0.59 | 0.41 | 0.41 | 0.40 | 0.43 | 0.42 | 0.44 | 0.44 |
| 26 | β-Elemene | 1384 | 0.16 | 0.14 | 0.11 | - | - | - | - | - | - | - |
| 27 | *trans*-Jasmone | 1387 | 0.06 | - | - | - | - | - | - | - | - | - |
| 28 | cis-Caryophyllene | 1397 | 0.03 | - | - | - | - | - | - | - | - | - |
| 29 | *trans*-Caryophyllene | 1410 | 1.75 | 1.56 | 1.31 | 0.97 | 0.95 | 0.92 | 0.90 | 0.89 | 0.95 | 1.01 |
| 30 | β-Copaene | 1423 | 0.17 | 0.15 | - | - | - | - | - | - | - | - |
| 31 | Aromadendrene | 1432 | 0.10 | 0.20 | - | - | - | - | - | - | - | - |
| 32 | α-Humulene | 1445 | 0.19 | 0.16 | 0.13 | - | - | - | - | - | - | - |
| 33 | *cis*-β-Farnesene | 1452 | 0.18 | 0.10 | - | - | - | - | - | - | - | - |
| 34 | Germacrene D | 1477 | 0.05 | - | - | - | - | - | - | - | - | - |
| 35 | α-Amorphene | 1482 | 0.07 | - | - | - | - | - | - | - | - | - |
| 36 | α-Muurolene | 1494 | 0.03 | - | - | - | - | - | - | - | - | - |
| 37 | δ-Cadinene | 1515 | 0.06 | - | - | - | - | - | - | - | - | - |
| 38 | Caryophyllene oxide | 1576 | 0.55 | 0.43 | 0.33 | - | - | - | - | 0.28 | 0.26 | - |
| 39 | Viridiflorol | 1584 | 0.05 | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 10.09 | 10.08 | 9.69 | 6.84 | 7.22 | 7.30 | 7.30 | 10.18 | 9.34 | 7.41 |
| | Oxygenated monoterpenes | | 84.92 | 86.00 | 87.46 | 91.78 | 91.42 | 91.38 | 91.37 | 88.06 | 88.85 | 91.14 |
| | Phenolic monoterpenes | | 0.27 | 0.25 | 0.23 | - | - | - | - | 0.17 | 0.16 | - |
| | Sesquiterpene hydrocarbons | | 3.74 | 3.04 | 2.14 | 1.38 | 1.36 | 1.32 | 1.33 | 1.31 | 1.39 | 1.45 |
| | Oxygenated sesquiterpenes | | 0.60 | 0.43 | 0.33 | - | - | - | - | 0.28 | 0.26 | - |
| | Total identified | | 99.62 | 99.8 | 99.85 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

Deterioration of the essential oil follows a similar pattern during storage at room temperature and in direct sunlight (Table 12). Components with low concentration become undetectable after 10 days, and the gradual loss of substances eventually leads to the presence of only the nine above-mentioned compounds. The formation of neo-menthol, menthol, α-terpineol, and carveol in this condition further supports their status as degradation products of the essential oil [16]. The percentage of carvone increases to 88.74% after 170 days, compared to 73.69% in the initial sample.

The enhanced stability achieved through encapsulating the essential oil In glass ampoules is evident once again in the experiments conducted at 35 °C and 45 °C (Table 13). Despite the higher temperature, which typically leads to reduced stability, a greater number of substances can be found in the final measurement (Day 80). In addition to the previously mentioned compounds, p-cymene, α-terpineol, isopiperitone, and caryophyllene oxide are preserved. Nevertheless, the sample still undergoes changes with the loss of substances and the formation of degradation products, similar to the previous conditions. The percentage of carvone increases to 85.36% at 35 °C and 84.98% at 45 °C, compared to 73.69% in the original sample after a 3-month period. Under the same temperature conditions, but in a different container (clear glass with a cap; Table 14), the composition shows a different pattern. The essential oils rapidly lose compounds with low concentrations, and gradually, the remaining substances diminish until, after 3 months, only the nine aforementioned components remain. The observed degradation products in these conditions are neo-menthol, menthol, and α-terpineol (carveol is not formed). The percentage of carvone increases significantly to 88.13% and 87.91% at 35 °C and 45 °C, respectively.

Finally, in cooling conditions (Table 15), the effect of the container on the preservation of the essential oil components becomes strongly evident. At 4 °C in clear glass tubes, the composition of the final sample after 6 months remains similar to that of the previous conditions, while the formation of all four degradation products is observed. The percentage of carvone significantly increases to 88.62%. In contrast, when stored in a metal container at the same temperature for 6 months, additional compounds such as p-cymene, iso-menthone, α-terpineol, isopiperitone, menthyl acetate, and caryophyllene oxide are preserved in the mixture, and the percentage of carvone increases to only 82.81%. Similarly, the same additional ingredients, except for menthyl acetate, are retained in the glass ampoules, and the concentration of carvone increases slightly more (84.16%), but not as much as in the glass tubes (88.62%). Moreover, the final composition of the sample that remains for 6 months in glass test tubes at −20 °C does not differ significantly from that at 4 °C in the same container, from which we conclude that at low temperatures, the effect of the container is significant.

After storage in various conditions, the color of *M. spicata* essential oil changed from pale yellow to a more intense yellow, being more distinct at high temperatures (35 °C and 45 °C) and especially in glass tubes with caps. The color intensity was maintained for a longer period in the ampoules at the same temperatures, but after 6 months, the color turned dark yellow. Similarly to the previous ones, the sample color stored under sunlight faded to pale yellow (Figure 2).

### 3.1.4. *Mentha* × *piperita* L.

In the *M.* × *piperita* (peppermint) essential oil, a total of 33 compounds were identified (Table 16) with the main ones being isomenthol (36.59%), menthone (21.70%), isomenthone (10.17%), menthyl acetate (6.56%), neomenthol (6.42%), limonene (4.95%), isopulegol (2.00%), α-terpineol (1.92%), *trans*-caryophyllene (1.35%), isopiperitone (1.31%), neoisomenthol (1.15%), and pulegone (1.10%). This indicates that the essential oil is particularly abundant in oxygenated monoterpenes (82.94%), while monoterpene hydrocarbons (4.95%), sesquiterpene hydrocarbons (3.66%), and other minor chemical groups such as phenolic monoterpenes (0.15%), and oxygenated sesquiterpenes (0.12%). Moreover, other compounds (7.02%) from different categories are found in smaller quantities. This observed composition is consistent with other reports of essential oil analysis of the same species [20].

Most of the substances present in the mixture at low concentrations (<0.20%) could no longer be detected after storage for just 10 days at room temperature in the absence of light (in glass tubes) (Table 16). In continuation, the chemical composition remained relatively stable until Day 30. Of note, γ-terpinene, not present in the initial analysis of peppermint essential oil, became detectable within measurements of 10 to 30 days. This suggests that this substance is a degradation product of a main compound of the sample, probably limonene [21]. In the measurement after 40 days, the absence of 3-octanol and p-cymene was observed. By Day 60, α-guaiene could no longer be detected. During the period between 80 and 140 days, the composition gradually changed, leading to a reduction in the number of detectable substances. Eventually, in the last four measurements, only six compounds remained detectable: limonene, menthone, isomenthone, neomenthol, isomenthol, and menthyl acetate. The percentage of isomenthol, therefore, increased from 36.59% in the initial sample to 52.33% in the final measurement after 6 months.

When storing the essential oil at room temperature and in glass tube containers but in direct sunlight (Table 14), a more rapid reduction in the number of substances in the mixture is observed. For example, 3-octanol cannot be detected in the sample even after 10 days and p-cymene after 30 days. In this condition, no formation of γ-terpinene occurred unless this process was completed during the first 10 days and the substance was not detected. After the gradual loss of substances, the final result is that the mixture consists of the same six substances (as in the previous condition) in the last four measurements, and the percentage of isomenthol increased to 52.33%.

During the stability experiment at high temperatures of 35 °C and 45 °C in glass ampoules (Table 15), the essential oil composition remains relatively stable. Apart from the loss of volatile components in small concentrations observed in the first measurement, only p-cymene, δ-elemene, and α-guaiene could no longer be detected in the mixture after 3 months. Formation of γ-terpinene is observed only at 35 °C, probably because at 45 °C the process of its formation and degradation takes place at a higher rate, and as a result, its occurrence within the analyses may have been overlooked. In the final measurement after 80 days, the essential oil composition at both temperatures consists of 13 compounds (limonene, isopulegol, menthone, isomenthone, neomenthol, iso-menthol, neoisomenthol, α-terpineol, pulegone, isopiperitone, menthyl acetate, β-bourbonene, and *trans*-caryophyllene), and the percentage of iso-menthol increased to 45.95% at 35 °C and 47.10% at 45 °C from 36.59% in the initial sample.

At the same temperatures (35 °C and 45 °C) but in glass tubes (Table 14), the alteration of the composition occurs at a higher rate. No formation of γ-terpinene was observed at both temperature conditions. The samples after 3 months (Day 80) consisted of the same six substances (limonene, menthone, isomenthone, neomenthol, isomenthol, and menthyl acetate), and the percentage of isomenthol increased to 50.48% at 35 °C and 50.38% at 45 °C from 36.59% in the initial sample.

**Table 16.** Chemical composition (%) of *Mentha* × *piperita* L. essential oil during 6 months of storage in clear glass tubes sealed with cap at room temperature (23 °C) under darkness.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3-Octanol | 991 | 0.42 | 0.28 | 0.28 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 2 | p-Cymene | 1019 | 0.15 | 0.25 | 0.22 | 0.10 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | Limonene | 1022 | 4.95 | 2.43 | 4.85 | 4.48 | 4.41 | 4.35 | 4.35 | 3.94 | 3.37 | 3.31 | 3.53 | 3.65 | 4.04 | 4.37 | 4.82 | 4.39 | 4.30 | 4.02 |
| 4 | γ-Terpinene | 1054 | - | 0.17 | 0.16 | 0.13 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | Linalool | 1097 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 6 | Isopulegol | 1141 | 2.00 | 1.48 | 1.76 | 1.60 | 1.44 | 1.43 | 1.42 | 1.34 | 1.11 | 0.99 | 1.02 | - | - | - | - | - | - | - |
| 7 | Menthone | 1150 | 21.70 | 22.4 | 22.95 | 22.57 | 22.78 | 22.77 | 22.94 | 22.35 | 21.9 | 22.04 | 22.08 | 23.6 | 23.64 | 25.32 | 25.12 | 24.08 | 24.01 | 22.71 |
| 8 | Isomenthone | 1159 | 10.17 | 9.74 | 10.16 | 10.08 | 9.75 | 9.71 | 9.66 | 9.42 | 8.68 | 8.47 | 8.76 | 9.13 | 9.13 | 9.14 | 9.67 | 9.33 | 9.30 | 8.82 |
| 9 | Neomenthol | 1163 | 6.42 | 6.89 | 6.98 | 6.88 | 6.73 | 6.74 | 6.58 | 6.60 | 6.02 | 5.58 | 5.86 | 5.80 | 5.80 | 5.55 | 6.00 | 5.91 | 5.90 | 5.56 |
| 10 | Isomenthol | 1178 | 36.59 | 44.18 | 40.11 | 40.75 | 42.54 | 42.67 | 43.77 | 44.61 | 48.01 | 49.21 | 49.61 | 50.45 | 49.49 | 48.45 | 48.64 | 50.46 | 50.74 | 52.33 |
| 11 | Neoisomenthol | 1182 | 1.15 | 0.99 | 0.99 | 1.01 | 0.96 | 0.95 | 0.91 | 0.91 | 0.85 | 0.78 | 0.84 | - | - | - | - | - | - | - |
| 12 | α-Terpineol | 1189 | 1.92 | 1.57 | 1.60 | 1.63 | 1.45 | 1.45 | 1.41 | 1.42 | 1.35 | 1.31 | 1.35 | 1.35 | 1.58 | 1.33 | - | - | - | - |
| 13 | Dihydro carveol | 1192 | 0.21 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 14 | *cis*-Piperitol | 1198 | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Pulegone | 1231 | 1.10 | 0.82 | 0.85 | 0.91 | 0.74 | 0.75 | 0.68 | 0.73 | 0.68 | 0.70 | - | - | - | - | - | - | - | - |
| 16 | Carvone | 1236 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Isopiperitone | 1245 | 1.31 | 0.94 | 0.98 | 1.10 | 0.8 | 0.78 | 0.72 | 0.78 | 0.70 | 0.74 | - | - | - | - | - | - | - | - |
| 18 | Dihydroedulan II | 1281 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | Menthyl acetate | 1292 | 6.56 | 5.86 | 6.00 | 6.35 | 6.27 | 6.31 | 5.78 | 5.98 | 5.79 | 5.73 | 5.85 | 6.02 | 6.32 | 5.84 | 5.75 | 5.83 | 5.75 | 6.56 |
| 20 | δ-Elemene | 1331 | 0.36 | 0.27 | 0.29 | 0.30 | 0.28 | 0.28 | 0.27 | 0.28 | - | - | - | - | - | - | - | - | - | - |
| 21 | Longicyclene | 1362 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | α-Ylangene | 1364 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | α-Copaene | 1369 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 24 | β-Bourbonene | 1379 | 0.66 | 0.49 | 0.52 | 0.55 | 0.52 | 0.51 | 0.49 | 0.52 | 0.47 | - | - | - | - | - | - | - | - | - |
| 25 | β-Elemene | 1386 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Longifolene | 1396 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | *trans*-Caryophyllene | 1410 | 1.35 | 1.04 | 1.08 | 1.15 | 1.11 | 1.10 | 1.02 | 1.12 | 1.07 | 1.13 | 1.09 | - | - | - | - | - | - | - |
| 28 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | β-Gurjunene | 1434 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | α-Guaiene | 1442 | 0.31 | 0.20 | 0.22 | 0.24 | 0.21 | 0.20 | - | - | - | - | - | - | - | - | - | - | - | - |
| 31 | γ-Muurolene | 1477 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | Germacrene D | 1484 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 33 | Caryophyllene oxide | 1576 | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 4.95 | 2.60 | 5.01 | 4.61 | 4.41 | 4.35 | 4.35 | 3.94 | 3.37 | 3.31 | 3.53 | 3.65 | 4.04 | 4.37 | 4.82 | 4.39 | 4.30 | 4.02 |
| | Oxygenated monoterpenes | | 82.94 | 89.01 | 86.38 | 86.53 | 87.19 | 87.25 | 88.09 | 88.16 | 89.30 | 89.82 | 89.52 | 90.33 | 89.64 | 89.79 | 89.43 | 89.78 | 89.95 | 89.42 |
| | Phenolic monoterpenes | | 0.15 | 0.25 | 0.22 | 0.10 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.66 | 2.00 | 2.11 | 2.24 | 2.12 | 2.09 | 1.78 | 1.92 | 1.54 | 1.13 | 1.09 | - | - | - | - | - | - | - |
| | Oxygenated sesquiterpenes | | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | -- |
| | Others | | 7.02 | 6.14 | 6.28 | 6.52 | 6.27 | 6.31 | 5.78 | 5.98 | 5.79 | 5.73 | 5.85 | 6.02 | 6.32 | 5.84 | 5.75 | 5.83 | 5.75 | 6.56 |
| | Total identified | | 98.84 | 100 | 100 | 100 | 99.99 | 100 | 100 | 100 | 100 | 99.99 | 99.99 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 17.** Chemical composition (%) of *Mentha* × *piperita* L. essential oil during 6 months of storage in clear glass tubes sealed with cap under direct sunlight exposure.

| No. | Compound | KI * | 1st ** | 10th ** | 20th ** | 30th ** | 40th ** | 50th ** | 60th ** | 70th ** | 80th ** | 90th ** | 100th ** | 110th ** | 120th ** | 130th ** | 140th ** | 150th ** | 160th ** | 170th ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3-Octanol | 991 | 0.42 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 2 | p-Cymene | 1019 | 0.15 | 0.21 | 0.24 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | Limonene | 1022 | 4.95 | 4.07 | 4.29 | 4.36 | 4.01 | 3.94 | 4.09 | 4.02 | 3.72 | 3.41 | 3.41 | 3.62 | 4.10 | 4.00 | 4.41 | 4.23 | 4.36 | 4.12 |
| 4 | Linalool | 1097 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | Isopulegol | 1141 | 2.00 | 1.40 | 1.49 | 1.47 | 1.43 | 1.65 | 1.43 | 1.38 | 1.14 | 1.03 | 1.01 | - | - | - | - | - | - | - |
| 6 | Menthone | 1150 | 21.70 | 22.79 | 22.83 | 22.83 | 22.14 | 22.57 | 22.73 | 22.57 | 22.58 | 22.25 | 22.33 | 23.24 | 23.51 | 25.08 | 25.51 | 24.06 | 24.04 | 23.50 |
| 7 | Isomenthone | 1159 | 10.17 | 9.63 | 9.87 | 10.00 | 9.49 | 9.50 | 9.58 | 9.46 | 8.90 | 8.72 | 8.93 | 9.10 | 9.10 | 9.25 | 9.59 | 9.44 | 9.53 | 9.13 |
| 8 | Neomenthol | 1163 | 6.42 | 6.62 | 6.81 | 6.87 | 6.88 | 6.94 | 6.64 | 6.65 | 6.04 | 5.82 | 5.92 | 5.72 | 5.72 | 5.65 | 5.85 | 6.03 | 6.07 | 5.84 |
| 9 | Isomenthol | 1178 | 36.59 | 43.39 | 42.28 | 42.14 | 43.45 | 43.68 | 43.65 | 44.44 | 47.31 | 49.84 | 50.28 | 50.13 | 48.75 | 49.01 | 47.53 | 50.41 | 50.38 | 51.81 |
| 10 | Neoisomenthol | 1182 | 1.15 | 0.93 | 0.98 | 0.98 | 0.97 | 0.96 | 0.92 | 0.92 | 0.83 | 0.82 | 0.80 | 0.80 | 0.84 | 0.55 | - | - | - | - |
| 11 | α-Terpineol | 1189 | 1.92 | 1.48 | 1.56 | 1.56 | 1.51 | 1.52 | 1.48 | 1.42 | 1.30 | 1.36 | 1.28 | 1.28 | 1.56 | 1.36 | 1.46 | - | - | - |
| 12 | Dihydro carveol | 1192 | 0.21 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 13 | *cis*-Piperitol | 1198 | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 14 | Pulegone | 1231 | 1.10 | 0.78 | 0.82 | 0.78 | 0.76 | 0.74 | 0.73 | 0.68 | 0.58 | - | - | - | - | - | - | - | - | - |
| 15 | Carvone | 1236 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | Isopiperitone | 1245 | 1.31 | 0.88 | 0.81 | 0.81 | 0.74 | 0.77 | 0.75 | 0.65 | 0.58 | - | - | - | - | - | - | - | - | - |
| 17 | Dihydroedulan II | 1281 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 18 | Menthyl acetate | 1292 | 6.56 | 5.85 | 6.00 | 6.19 | 6.52 | 5.79 | 6.01 | 6.07 | 5.70 | 5.85 | 6.04 | 6.11 | 6.42 | 5.10 | 5.65 | 5.83 | 5.62 | 5.60 |
| 19 | δ-Elemene | 1331 | 0.36 | 0.27 | 0.28 | 0.28 | 0.29 | 0.28 | 0.29 | 0.28 | - | - | - | - | - | - | - | - | - | - |
| 20 | Longicyclene | 1362 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | α-Ylangene | 1364 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | α-Copaene | 1369 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | β-Bourbonene | 1379 | 0.66 | 0.49 | 0.51 | 0.51 | 0.54 | 0.51 | 0.52 | 0.52 | 0.46 | - | - | - | - | - | - | - | - | - |
| 24 | β-Elemene | 1386 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | Longifolene | 1396 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | *trans*-Caryophyllene | 1411 | 1.35 | 1.00 | 1.02 | 1.02 | 1.06 | 0.94 | 0.97 | 0.94 | 0.86 | 0.90 | - | - | - | - | - | - | - | - |
| 27 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | β-Gurjunene | 1434 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Guaiene | 1442 | 0.31 | 0.20 | 0.20 | 0.20 | 0.21 | 0.21 | 0.21 | - | - | - | - | - | - | - | - | - | - | - |
| 30 | γ-Muurolene | 1476 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 31 | Germacrene D | 1484 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | Caryophyllene oxide | 1576 | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 4.95 | 4.07 | 4.29 | 4.36 | 4.01 | 3.94 | 4.09 | 4.02 | 3.72 | 3.41 | 3.41 | 3.62 | 4.10 | 4.00 | 4.41 | 4.23 | 4.36 | 4.12 |
| | Oxygenated monoterpenes | | 82.94 | 87.9 | 87.45 | 87.44 | 87.37 | 88.33 | 87.91 | 88.17 | 89.26 | 89.84 | 90.55 | 90.27 | 89.48 | 90.9 | 89.94 | 89.94 | 90.02 | 90.28 |
| | Phenolic monoterpenes | | 0.15 | 0.21 | 0.24 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.66 | 1.96 | 2.01 | 2.01 | 2.01 | 1.94 | 1.99 | 1.74 | 1.32 | 0.90 | - | - | - | - | - | - | - | - |
| | Oxygenated sesquiterpenes | | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Others | | 7.02 | 5.85 | 6.00 | 6.19 | 6.52 | 5.79 | 6.01 | 6.07 | 5.70 | 5.85 | 6.04 | 6.11 | 6.42 | 5.10 | 5.65 | 5.83 | 5.62 | 5.60 |
| | Total identified | | 98.84 | 99.99 | 99.99 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

* Kovats index on HP-5 column ** storage time in day.

**Table 18.** Chemical composition (%) of *Mentha* × *piperita* L. essential oil during 3 months of storage in sealed glass ampoules at a temperature of 35 °C and 45 °C.

| | | | | 35 °C | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 1 | 3-Octanol | 991 | 0.42 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 2 | p-Cymene | 1019 | 0.15 | 0.51 | 0.14 | - | - | - | - | - | - | 0.39 | 0.13 | - | - | - | - | - | - |
| 3 | Limonene | 1022 | 4.95 | 4.16 | 4.61 | 4.35 | 4.41 | 4.38 | 4.52 | 4.16 | 4.20 | 3.91 | 4.51 | 4.28 | 4.13 | 4.12 | 4.36 | 4.27 | 3.49 |
| 4 | γ-Terpinene | 1054 | - | 0.26 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | Linalool | 1097 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 6 | Isopulegol | 1141 | 2.00 | 1.49 | 1.56 | 1.67 | 1.41 | 1.44 | 1.49 | 1.40 | 1.13 | 1.43 | 1.54 | 1.51 | 1.43 | 1.52 | 1.47 | 1.40 | 1.15 |
| 7 | Menthone | 1150 | 21.7 | 22.15 | 22.83 | 23.11 | 22.76 | 22.64 | 23.08 | 22.67 | 22.89 | 22.45 | 22.69 | 22.37 | 22.22 | 22.00 | 23.00 | 22.92 | 22.29 |
| 8 | lsomenthone | 1159 | 10.17 | 9.48 | 9.96 | 9.88 | 9.70 | 9.67 | 9.71 | 9.50 | 9.22 | 9.58 | 9.95 | 9.59 | 9.49 | 9.41 | 9.71 | 9.63 | 8.78 |
| 9 | Neomenthol | 1163 | 6.42 | 6.78 | 6.95 | 6.83 | 6.76 | 6.71 | 6.64 | 6.61 | 5.96 | 6.79 | 6.94 | 6.74 | 6.85 | 6.85 | 6.66 | 6.52 | 6.11 |
| 10 | Isomenthol | 1178 | 36.59 | 43.09 | 41.67 | 42.03 | 43.00 | 43.61 | 44.04 | 43.9 | 45.95 | 43.71 | 41.86 | 43.47 | 43.33 | 44.28 | 43.33 | 43.5 | 47.10 |
| 11 | Neoisomenthol | 1182 | 1.15 | 0.97 | 0.99 | 0.96 | 0.94 | 0.95 | 0.47 | 0.90 | 0.82 | 0.95 | 0.99 | 0.96 | 0.94 | 0.95 | 0.91 | 0.89 | 0.82 |
| 12 | α-Terpineol | 1189 | 1.92 | 1.54 | 1.55 | 1.54 | 1.42 | 1.46 | 0.93 | 1.43 | 1.30 | 1.50 | 1.57 | 1.51 | 1.47 | 1.52 | 1.43 | 1.42 | 1.36 |
| 13 | Dihydro carveol | 1192 | 0.21 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 14 | *cis*-Piperitol | 1198 | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 15 | Pulegone | 1231 | 1.10 | 0.76 | 0.80 | 0.82 | 0.71 | 0.74 | 0.70 | 0.74 | 0.65 | 0.74 | 0.81 | 0.78 | 0.77 | 0.77 | 0.73 | 0.80 | 0.76 |
| 16 | Carvone | 1236 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 17 | Isopiperitone | 1245 | 1.31 | 0.84 | 0.88 | 0.92 | 0.72 | 0.81 | 0.75 | 0.76 | 0.63 | 0.80 | 0.87 | 0.86 | 0.78 | 0.82 | 0.74 | 0.78 | 0.73 |
| 18 | Dihydroedulan II | 1281 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 19 | Menthyl acetate | 1292 | 6.56 | 5.92 | 6.03 | 5.88 | 6.29 | 5.79 | 5.85 | 6.02 | 5.68 | 5.81 | 6.15 | 5.92 | 6.45 | 5.96 | 5.84 | 6.01 | 5.85 |
| 20 | δ-Elemene | 1331 | 0.36 | 0.28 | 0.28 | 0.29 | 0.28 | 0.27 | 0.28 | 0.29 | - | 0.27 | 0.28 | 0.28 | 0.29 | 0.29 | 0.28 | 0.28 | - |
| 21 | Longicyclene | 1362 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | α-Ylangene | 1364 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | α-Copaene | 1369 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 24 | β-Bourbonene | 1379 | 0.66 | 0.51 | 0.51 | 0.51 | 0.51 | 0.49 | 0.50 | 0.52 | 0.51 | 0.48 | 0.44 | 0.50 | 0.52 | 0.45 | 0.50 | 0.51 | 0.48 |
| 25 | β-Elemene | 1386 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | Longifolene | 1396 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 27 | *trans*-Caryophyllene | 1411 | 1.35 | 1.05 | 1.03 | 1.00 | 1.08 | 1.03 | 1.04 | 1.10 | 1.02 | 0.96 | 1.06 | 1.03 | 1.11 | 1.06 | 1.04 | 1.07 | 1.07 |
| 28 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | β-Gurjunene | 1434 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | α-Guaiene | 1442 | 0.31 | 0.21 | 0.21 | 0.21 | - | - | - | - | - | 0.21 | 0.21 | 0.20 | 0.21 | - | - | - | - |
| 31 | γ-Muurolene | 1476 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | Germacrene D | 1484 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 33 | Caryophyllene oxide | 1576 | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 4.95 | 4.42 | 4.61 | 4.35 | 4.41 | 4.38 | 4.52 | 4.16 | 4.20 | 3.91 | 4.51 | 4.28 | 4.13 | 4.12 | 4.36 | 4.27 | 3.49 |
| | Oxygenated monoterpenes | | 82.94 | 87.1 | 87.19 | 87.76 | 87.42 | 88.03 | 87.81 | 87.91 | 88.55 | 87.95 | 87.22 | 87.79 | 87.28 | 88.12 | 87.98 | 87.86 | 89.1 |
| | Phenolic monoterpenes | | 0.15 | 0.51 | 0.14 | - | - | - | - | - | - | 0.39 | 0.13 | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.66 | 2.05 | 2.03 | 2.01 | 1.87 | 1.79 | 1.82 | 1.91 | 1.53 | 1.92 | 1.99 | 2.01 | 2.13 | 1.80 | 1.82 | 1.86 | 1.55 |
| | Oxygenated sesquiterpenes | | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Others | | 7.02 | 5.92 | 6.03 | 5.88 | 6.29 | 5.79 | 5.85 | 6.02 | 5.68 | 5.81 | 6.15 | 5.92 | 6.45 | 5.96 | 5.84 | 6.01 | 5.85 |
| | Total identified | | 98.84 | 100 | 100 | 100 | 99.99 | 99.99 | 100 | 100 | 99.96 | 99.98 | 100 | 100 | 99.99 | 100 | 100 | 100 | 99.99 |

* Kovats index on HP-5 column; ** storage time in days.

**Table 19.** Chemical composition (%) of *Mentha* × *piperita* L. essential oil during 3 months of storage in clear glass tubes sealed with cap at a temperature of 35 °C and 45 °C.

| | | | | 35 °C | | | | | | | | 45 °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** | **10th **** | **20th **** | **30th **** | **40th **** | **50th **** | **60th **** | **70th **** | **80th **** |
| 1 | 3-Octanol | 991 | 0.42 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 2 | p-Cymene | 1019 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 3 | Limonene | 1022 | 4.95 | 4.33 | 3.88 | 3.58 | 3.60 | 4.07 | 4.08 | 4.49 | 4.28 | 3.65 | 3.62 | 3.52 | 3.53 | 3.52 | 3.65 | 3.65 | 4.29 |
| 4 | Linalool | 1097 | 0.14 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 5 | Isopulegol | 1141 | 2.00 | 1.32 | 1.15 | 1.01 | - | - | - | - | - | 1.20 | 1.11 | 1.02 | 1.02 | 1.02 | 1.45 | 1.20 | - |
| 6 | Menthone | 1150 | 21.70 | 23.83 | 23.12 | 22.28 | 22.97 | 22.93 | 25.07 | 25.11 | 24.03 | 22.55 | 22.59 | 22.6 | 22.22 | 22.03 | 22.04 | 22.55 | 23.99 |
| 7 | Isomenthone | 1159 | 10.17 | 9.22 | 9.10 | 8.90 | 9.01 | 8.84 | 9.23 | 9.50 | 9.45 | 8.85 | 8.91 | 9.02 | 9.12 | 9.17 | 9.26 | 8.85 | 9.49 |
| 8 | Neomenthol | 1163 | 6.42 | 6.25 | 5.97 | 5.94 | 5.88 | 5.57 | 5.64 | 5.80 | 5.94 | 6.07 | 5.98 | 5.96 | 6.00 | 5.90 | 5.63 | 6.07 | 6.01 |
| 9 | Isomenthol | 1178 | 36.59 | 44.63 | 45.94 | 48.18 | 48.04 | 47.76 | 45.31 | 46.95 | 50.48 | 46.42 | 46.66 | 48.62 | 49.02 | 49.72 | 49.42 | 49.05 | 50.38 |
| 10 | Neoisomenthol | 1182 | 1.15 | 0.86 | 0.83 | 0.82 | 0.81 | 0.81 | 0.75 | - | - | 0.82 | 0.81 | 0.78 | 0.78 | 0.78 | 0.74 | 0.82 | - |
| 11 | α-Terpineol | 1189 | 1.92 | 1.33 | 1.32 | 1.32 | 1.54 | 1.54 | 1.40 | 1.46 | - | 1.36 | 1.33 | 1.33 | 1.33 | 1.31 | 1.31 | 1.36 | - |
| 12 | Dihydro carveol | 1192 | 0.21 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 13 | *cis*-Piperitol | 1198 | 0.18 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 14 | Pulegone | 1231 | 1.1 | 0.64 | 0.70 | - | - | - | - | - | - | 0.71 | 0.69 | 0.69 | 0.69 | 0.69 | 0.66 | 0.71 | - |
| 15 | Carvone | 1236 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 16 | Isopiperitone | 1245 | 1.31 | 0.67 | 0.73 | 1.04 | 1.09 | 1.09 | 1.04 | 1.08 | - | 0.76 | 0.76 | 0.76 | 0.74 | 0.69 | 0.66 | 0.59 | - |
| 17 | Dihydroedulan II | 1281 | 0.04 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 18 | Menthyl acetate | 1292 | 6.56 | 5.47 | 5.74 | 5.90 | 6.03 | 6.24 | 5.41 | 5.61 | 5.81 | 5.81 | 5.80 | 5.70 | 5.55 | 5.17 | 5.16 | 5.15 | 5.84 |
| 19 | δ-Elemene | 1331 | 0.36 | - | - | - | - | - | - | - | - | 0.28 | 0.28 | - | - | - | - | - | - |
| 20 | Longicyclene | 1362 | 0.09 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 21 | α-Ylangene | 1364 | 0.06 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 22 | α-Copaene | 1369 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 23 | β-Bourbonene | 1379 | 0.66 | 0.48 | 0.48 | - | - | - | - | - | - | 0.49 | 0.49 | - | - | - | - | - | - |
| 24 | β-Elemene | 1386 | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 25 | Longifolene | 1396 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 26 | *trans*-Caryophyllene | 1411 | 1.35 | 0.95 | 1.04 | 1.03 | 1.03 | 1.14 | - | - | - | 1.02 | 0.97 | - | - | - | - | - | - |
| 27 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 28 | β-Gurjunene | 1434 | 0.07 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 29 | α-Guaiene | 1442 | 0.31 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 30 | γ-Muurolene | 1476 | 0.05 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 31 | Germacrene D | 1484 | 0.17 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 32 | Caryophyllene oxide | 1576 | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 4.95 | 4.33 | 3.88 | 3.58 | 3.60 | 4.07 | 4.08 | 4.49 | 4.28 | 3.65 | 3.62 | 3.52 | 3.53 | 3.52 | 3.65 | 3.65 | 4.29 |
| | Oxygenated monoterpenes | | 82.94 | 88.75 | 88.86 | 89.49 | 89.34 | 88.54 | 88.44 | 89.90 | 89.90 | 88.74 | 88.84 | 90.78 | 90.92 | 91.31 | 91.17 | 91.2 | 89.87 |
| | Phenolic monoterpenes | | 0.15 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.66 | 1.43 | 1.52 | 1.03 | 1.03 | 1.14 | - | - | - | 1.79 | 1.74 | - | - | - | - | - | - |
| | Oxygenated sesquiterpenes | | 0.12 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| | Others | | 7.02 | 5.47 | 5.74 | 5.90 | 6.03 | 6.24 | 5.41 | 5.61 | 5.81 | 5.81 | 5.80 | 5.70 | 5.55 | 5.17 | 5.16 | 5.15 | 5.84 |
| | Total identified | | 98.84 | 99.98 | 100 | 100 | 100 | 99.99 | 97.93 | 100 | 99.99 | 99.99 | 100 | 100 | 100 | 100 | 99.98 | 100 | 100 |

* Kovats index on HP-5 column ** storage time in days.

**Table 20.** Chemical composition (%) of *Mentha* × *piperita* L. essential oil during 6 months of storage at 4 °C (in clear glass tubes sealed with cap, in metal containers and in sealed glass ampoules) and at −20 °C (in clear glass tubes sealed with cap).

| | | | | | | 4 °C Glass Tubes | | | | 4 °C Metal | 4 °C Ampoules | −20 °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **No.** | **Compound** | **KI *** | **1st **** | **30th** | **60th** | **90th** | **120th** | **150th** | **180th** | **180th** | **180th** | **180th** |
| 1 | 3-Octanol | 991 | 0.42 | - | - | - | - | - | - | - | - | - |
| 2 | p-Cymene | 1019 | 0.15 | - | - | - | - | - | - | - | - | - |
| 3 | Limonene | 1022 | 4.95 | 4.36 | 4.07 | 3.50 | 4.10 | 4.20 | 4.04 | 4.56 | 4.37 | 4.03 |
| 4 | Linalool | 1097 | 0.14 | - | - | - | - | - | - | - | - | - |
| 5 | Isopulegol | 1141 | 2.00 | 1.48 | 1.40 | - | - | - | - | 1.47 | 1.56 | - |
| 6 | Menthone | 1150 | 21.7 | 22.72 | 22.34 | 22.29 | 22.92 | 22.85 | 22.34 | 24.56 | 24.61 | 23.11 |
| 7 | Isomenthone | 1159 | 10.17 | 9.92 | 9.46 | 9.44 | 8.92 | 8.90 | 8.76 | 9.11 | 9.29 | 8.73 |
| 8 | Neomenthol | 1163 | 6.42 | 6.88 | 6.72 | 6.23 | 5.66 | 5.60 | 5.57 | 6.01 | 6.06 | 5.58 |
| 9 | Isomenthol | 1178 | 36.59 | 42.10 | 44.39 | 49.01 | 48.79 | 49.84 | 50.71 | 43.26 | 43.41 | 49.01 |
| 10 | Neoisomenthol | 1182 | 1.15 | 0.98 | 0.94 | 0.91 | 0.85 | - | - | 0.75 | 0.79 | - |
| 11 | α-Terpineol | 1189 | 1.92 | 1.56 | 1.43 | 1.50 | 1.59 | 1.56 | 1.56 | 1.32 | 1.38 | 1.60 |
| 12 | Dihydro carveol | 1192 | 0.21 | - | - | - | - | - | - | - | - | - |
| 13 | *cis*-Piperitol | 1198 | 0.18 | - | - | - | - | - | - | - | - | - |
| 14 | Pulegone | 1231 | 1.10 | 0.80 | 0.67 | - | - | - | - | 0.93 | 0.94 | - |
| 15 | Carvone | 1236 | 0.05 | - | - | - | - | - | - | - | - | - |
| 16 | Isopiperitone | 1245 | 1.31 | 0.89 | 0.73 | - | - | - | - | 1.07 | 1.02 | |
| 17 | Dihydroedulan II | 1281 | 0.04 | - | - | - | - | - | - | - | - | - |
| 18 | Menthyl acetate | 1292 | 6.56 | 6.19 | 5.96 | 5.92 | 5.90 | 5.80 | 5.77 | 5.10 | 5.03 | 6.54 |
| 19 | δ-Elemene | 1331 | 0.36 | 0.28 | 0.28 | - | - | - | - | - | - | - |
| 20 | Longicyclene | 1362 | 0.09 | - | - | - | - | - | - | - | - | - |
| 21 | α-Ylangene | 1364 | 0.06 | - | - | - | - | - | - | - | - | - |
| 22 | α-Copaene | 1369 | 0.15 | - | - | - | - | - | - | - | - | - |
| 23 | β-Bourbonene | 1379 | 0.66 | 0.52 | 0.50 | - | - | - | - | 0.50 | 0.48 | - |
| 24 | β-Elemene | 1386 | 0.15 | - | - | - | - | - | - | - | - | - |
| 25 | Longifolene | 1396 | 0.07 | - | - | - | - | - | - | - | - | - |
| 26 | *trans*-Caryophyllene | 1411 | 1.35 | 1.09 | 1.10 | 1.20 | 1.27 | 1.25 | 1.25 | 1.07 | 1.05 | 1.40 |
| 27 | β-Copaene | 1423 | 0.17 | - | - | - | - | - | - | - | - | - |
| 28 | β-Gurjunene | 1434 | 0.07 | - | - | - | - | - | - | - | - | - |
| 29 | α-Guaiene | 1442 | 0.31 | 0.21 | - | - | - | - | - | - | - | - |
| 30 | γ-Muurolene | 1476 | 0.05 | - | - | - | - | - | - | - | - | - |
| 31 | Germacrene D | 1484 | 0.17 | - | - | - | - | - | - | - | - | - |
| 32 | Caryophyllene oxide | 1576 | 0.12 | - | - | - | - | - | - | - | - | - |
| | Monoterpene hydrocarbons | | 4.95 | 4.36 | 4.07 | 3.50 | 4.10 | 4.20 | 4.04 | 4.56 | 4.37 | 4.03 |
| | Oxygenated monoterpenes | | 82.94 | 87.33 | 88.08 | 89.38 | 88.73 | 88.75 | 88.94 | 88.48 | 89.06 | 88.03 |
| | Phenolic monoterpenes | | 0.15 | - | - | - | - | - | - | - | - | - |
| | Sesquiterpene hydrocarbons | | 3.66 | 2.10 | 1.88 | 1.20 | 1.27 | 1.25 | 1.25 | 1.57 | 1.53 | 1.40 |
| | Oxygenated sesquiterpenes | | 0.12 | - | - | - | - | - | - | - | - | - |
| | Others | | 7.02 | 6.19 | 5.96 | 5.92 | 5.90 | 5.80 | 5.77 | 5.10 | 5.03 | 6.54 |
| | Total identified | | 98.84 | 99.98 | 99.99 | 100 | 100 | 100 | 100 | 99.71 | 99.99 | 100 |

* Kovats index on HP-5 column; ** storage time in days.

In the refrigerator at low temperatures (4 °C and −20 °C) (Table 15), the number of substances in the essential oil appeared to be better preserved compared to the rest of the conditions (at room and high temperatures) regardless of the container. At 4 °C after 6 months, the composition remained almost stable, consisting of eight substances (limonene, menthone, isomenthone, neomenthol, isomenthol, α-terpineol, menthyl acetate, and *trans*-caryophyllene). The percentage of iso-menthol increased to 50.71% after 6 months. At 4 °C, in both metal containers and glass ampoules, isopulegol, neoisomenthol, pulegone, isopiperitone, and β-bourbonene are additionally preserved. The percentage of isomenthol in these conditions increased less in comparison with higher temperatures (23 °C, 35 °C, and 45 °C) and specifically to 43.26% and 43.41% for metal containers and glass ampoules, respectively. Finally, at −20 °C in glass tubes, the same substances are preserved as at 4 °C in the same container, but again less than those remaining in comparison to metal containers and glass ampoules, suggesting that the type of container affects the stability more significantly at low temperatures than the temperature itself. The concentration of isomenthol increases in this condition (−20 °C) to 49.01%. Finally, the formation of γ-terpinene was not recorded at 4 °C or −20 °C, but this does not mean that the process did not take place at some point during the experiments as less frequent measurements were performed.

The initial sample of *M.* × *piperita* essential oil was clear with a very slight yellowish tint. When it was stored in various conditions, no great difference in its appearance was observed, apart from the sample placed in a glass tube with a cap at 45 °C, which after 3 months turned clear yellow. In the same container at 35 °C, the change to yellow was less intense but clearly visible (Figure 2).

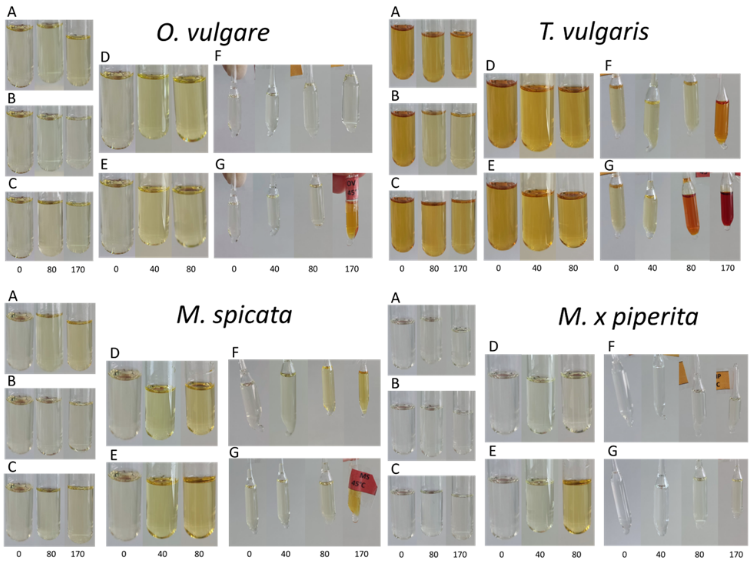


**Figure 2.** Photo-documentation on different days (0, 40, 80, 170) of selected essential oils (*O. vulgare*, *T. vulgaris*, *M. spicata* and *M.* x *piperita*) after storage in various conditions: A. at room temperature in glass tubes sealed with cap; B. in direct sunlight exposure in glass tubes sealed with cap; C. at 4 °C in glass tubes sealed with cap; D. at 35 °C in glass tubes sealed with cap; E. at 45 °C in glass tubes sealed with cap; F. at 35 °C in sealed glass ampoules; G. at 45 °C in sealed glass ampoules.

### *3.2. Multivariable Data Analysis: Principal Component Analysis (PCA)*

To evaluate whether the identified constituents may be useful in reflecting qualitative characteristics and by taking into account their dual potential application: a. from a qualitative standpoint, all components, including minor compounds (<1.0%), may potentially contribute to the overall quality and represent the primary state of the essential oils and b. that the major constituent exceeding 1.0% of total essential oil, significantly may affect their chemical identity, biological properties and potential applications, all tested conditions for each plant species were subjected to principal component analysis (PCA) under these two approaches. Specifically, all sample measurements in different time points, storage, or light conditions within a period of up to 6 months for each plant sample were utilized to distinguish any potential differentiation.

From the qualitative point of view in the PCA score scatter plot (Figure 3) generated from the chemical analysis dataset containing all the above-mentioned features, a clear difference between samples in different time points and a clear separation into three distinct clusters was observed. Based on the clustering, the glass tube containers were clearly separated from the ampoules and the metallic containers. The selected glass containers with caps affect at most the essential oil robustness with a dual contribution of oxygen availability and temperature conditions.

Specifically, the cluster illustrated by a green ellipse contains measurements in times points where the chemical load has been differentiated at most for all essential oils, indicating the impact of the container on the chemical constituents over time. In detail, this cluster contains measurements of the second half of the experiment regarding the samples stored in glass tubes (ca. 90–170 days) in light exposure, 23 °C and 4 °C, with *O. vulgare* essential oil participating only after Day 140, while in high-temperature storage conditions (35 °C and 45 °C), the samples participate in this cluster from earlier time points and even from 30 days in cases of *M. spicata* and *T. vulgaris*. It is worth mentioning that ampoules and metallic containers are absent from this cluster in all conditions except for *T. vulgare* essential oil stored in a metallic container for 180 days (4 °C).

Moreover, a clear clustering of samples (blue ellipse) in accordance with the previous cluster was observed, which includes former time points suggesting a transitional chemical state in all essential oils. The chemical profile of all samples has been differentiated but not as much as in the previous case and consists mostly of time points 80 to 130 in light exposure and 23 °C for glass tubes, as well as the last measurements for 4 °C ampoules and metallic containers except *M. spicata*. Regarding the samples at 35 °C and 45 °C, the measurements at 20 days participate for *T. vulgare* and *M. spicata* while for *O. vulgaris* and *M.* × *piperita* at up to 60 days.

All time points of samples with the most preserved chemical load are clustered together under the red ellipse, e.g., samples up to 70 or 80 days with 4 °C, 23 °C, and light exposure samples in glass tubes as well as ampoules at 35 °C and 45 °C for the first 10 days, while for *M.* × *piperita*, this cluster includes the last measurement at 4 °C for glass tubes and metallic containers.

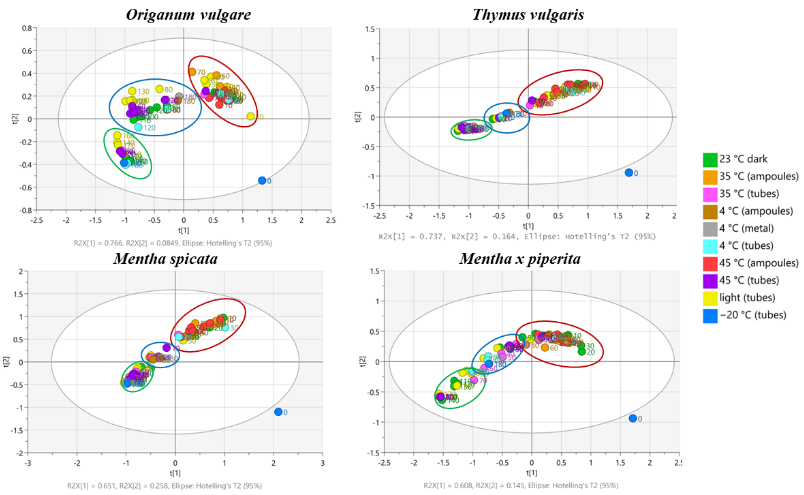


**Figure 3.** Two-dimensional (2D) score scatter plots of PCA, for samples measurements in different time points, storage, or light conditions within a period of up to 6 months (SIMCA-P 16.0.1) distinguishing the different volatiles of selected essential oils into three clusters (green, blue, and red ellipses) in terms of metabolite distribution.

Regarding the PCA analysis for the major constituents, almost no clustering of the different time points and conditions was observable, indicating that the accumulation of the major compounds has no severe impact on their relative composition during the experiment (Figure 4). The plots showed high group similarities and the variables being near each other. As the major constituents have not been significantly influenced, the chemical identity and as a result the biological properties of the essential oil may not have been affected nor other potential uses regarding their predominant compounds. It is of high importance that the preservation of the major compounds demonstrates the actual chemical profile characterizing the essential oils, and these ingredients in fact remain during their application. However, to evaluate the overall stability, apart from the preservation of the main compounds, additional factors should be taken into account, such as organoleptic characteristics including color changes through time (see Supplementary Materials).

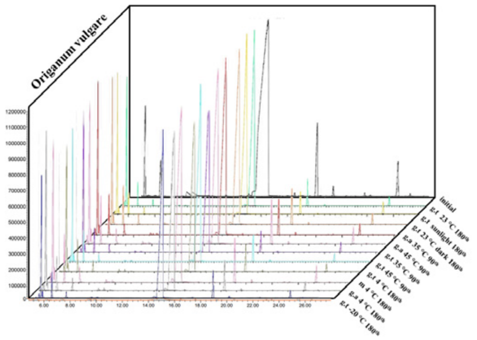


**Figure 4.** A Representative GC-MS chromatographs (from *O. vulgare;* composition %) of the initial essential oil analysis and the last measurements in each condition. g.a: glass ampoules; g.t: glass tubes; m: metallic container.

### 3.3. Essential Oil Stability

Generally, during the prolonged storage of the essential oils in various conditions, the substances that appeared to be more stable were the more abundant in the initial samples, i.e., the main substances. Ensuring the preservation of the major compounds is of utmost importance as it guarantees that the essential oils retain their distinct chemical profile throughout their potential application. These ingredients must preserve their portion at 90%, as they contribute decisively to its organoleptic characteristics and in order to maintain their efficacy, unique properties, and benefits. However, it is important to note that these substances may undergo alterations such as decomposition during storage.

As mentioned above, the degradation or evaporation of the volatiles contained in small amounts (<1.0%) occurs in all four essential oils within the first 10 days, despite the storage conditions. It is noteworthy that, upon opening the sealed container of the commercially available essential oil, its degradation immediately begins to take place. As expected, only the remaining ingredients (major compounds) are present during essential oil applications. As previously discussed, the preservation of trace substances is not feasible regardless of the temperature or container used. However, this limitation does not have an impact on their applications because these minor compounds will not contribute to a final product. Thus, from being complex mixtures (>30 substances), essential oils ended up characterized mainly by their main substances after storage in various conditions.

Moreover, odor perception is another crucial element and a distinct characteristic for essential oils that remain constant and unaltered across each species throughout the 6 months duration of the stability study regardless of the tested conditions and the chemical variation of the minor compounds.

The selected condition of 45 °C involves the speed of reactions estimating essential oil's robustness. For a period of up to 3 months, all essential oils' chemical composition regarding their major compounds showed minor variation under low oxygen availability. The selected containers played a crucial role in their organoleptic properties as in glass tubes with caps color intensity increases after 1.5 months, while in glass ampoules, color remains intact for a period of up to 3 months except for *T. vulgaris* essential oil. Regarding the former sample, it is expected to gain a more intense color as it started with a distinct orange color (see Figure 2). Regarding the selected condition of 35 °C, similar results were observed, and even *T. vulgaris* essential oil color remained stable for up to three months in glass ampoules. Thus, accelerated aging predictions give evidence for a shelf-life of about 2–3 years.

The stability test of the samples in light exposure gave evidence, mainly for the influence of the UVA wavelength range, as the selected container made by tempered glass is known to block 60–70% of UVB. Essential oils by nature are photo- and thermo-sensitive, and color alteration is expected. According to the photo-documentation, direct sunlight exposure causes a slight fading of their color. However, these changes do not significantly affect the major compounds, and vice versa.

The chemical load in all cases was better preserved under cooling conditions of 4 °C or ideally in temperatures less than 23 °C degrees away from direct sunlight. Extreme temperature fluctuations and any exposure to heat sources degrade the quality of the essential oils.

## 4. Conclusions

The present study provides a comprehensive approach to monitoring the quality and stability of essential oils. By considering the physicochemical properties, compound accumulation, and the influence of diverse thermal and storage conditions, valuable insights into the factors affecting essential oil stability were revealed. The findings from this study contribute to a better understanding of essential oil preservation and can guide the development of strategies to optimize their shelf life and maintain their therapeutic properties. Specifically, this study demonstrates the impact of different conditions on the chemical composition of selected essential oils from the Lamiaceae family (*O. vulgare*, *T. vulgaris*, *M. spicata*, and *M. × piperita*). The samples were analyzed using GC-MS, and the components were subjected to PCA.

In light of the limited existing literature, our study faces challenges in establishing a direct correlation between our findings and prior research. The gap in available studies hinders a correlation analysis as only a few investigations have been undertaken in this area. In addition, there are some stability studies on essential oil encapsulation, but not on the raw material itself, emphasizing the need for more targeted research on this topic.

Overall, the results showed that the chemical composition of the initial samples regarding the main constituents was preserved over time, even though minor compounds were negatively affected regardless of the thermal and storage conditions.

Changes observed after subjecting the essential oils to an accelerated aging (heating) process indicated the shelf-life of the samples. The major compounds showed minor variation over a period of up to 3 months, while under low oxygen availability, essential oil color remains stable during the same period according to the photo-documentation. However, these changes do not significantly affect the major compounds, and vice versa. In all cases, essential oil odor remains unaffected throughout the stability study regardless of the tested conditions. Consequently, the final fragrance is mainly influenced by the major compounds that render their aromatic notes, and the contribution of the minor compounds is at an imperceptible level. Chemically, essential oils were better preserved under cooling conditions at 4 °C without sunlight exposure. Extreme temperature variations and any exposure to heat sources degrade the quality of the essential oils. In parallel, the influence of the selected containers has been highlighted. Based on the above, apart from a slight variation in the chemical load, essential oils can be considered stable for various applications, especially those stored under low oxygen availability conditions. It is crucial to conduct further analytical stability studies on essential oils and examine a wider range of conditions, including parameters such as UVB and UVA wavelength range, PH, and other organoleptic characteristics. In addition, it is of interest to study the potential application of antioxidant additives for enhancing the stability of essential oils. This will provide a more comprehensive understanding of how the storage environment affects their degradation process.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/separations10090488/s1, 1. Type of containers used for the stability study, 2. Photo-documentation of selected essential oils, 3. GC-MS chromatograms of the *O. vulgare*, *T. vulgaris*, *M. spicata*, and *M.* × *piperita* essential oils, 4. GC-MS chromatograms of the *Thymus vulgaris* L. essential oil, 5. GC-MS chromatograms of the *Mentha spicata* L. essential oil and 6. GC-MS chromatograms of the *Mentha* × *piperita* L. essential oil.

**Author Contributions:** Formal analysis, E.G. and C.B.; investigation, E.G. and C.B.; writing—original draft preparation, E.G., C.B. and M.-E.G.; writing—review and editing, E.G., C.B., M.-E.G. and H.S.; visualization, E.G., C.B. and M.-E.G.; supervision, H.S. and M.C.R. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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