# METHOD DEVELOPMENT FOR THE ANALYSIS OF VOLATILE COMPOUNDS IN OLIVE OIL

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Olive oil consists mainly of triglycerides, in about 97 to 99% by weight. The minor compounds are a complex mixture of polar, apolar and amphiphilic substances, such as tocopherols, phenolic compounds, sterols, chlorophyll, carotenoids, terpene acids, monoglycerides and diglycerides, free fatty acids and volatile compounds. These volatiles are the compounds directly responsible for the aroma of the oil. Extra virgin olive oil has a complex aroma with more than 100 volatile compounds identified, among aldehydes, alcohols, esters, hydrocarbons, ketones and furans. The objective of this study was to develop an analytical method for volatile compounds in olive oils using solid-phase microextraction (SPME), gas chromatography coupled to mass spectrometry (GC-MS) and gas chromatography with flame ionization detection (GC-FID). For the SPME, different parameters like flask size (4, 10 and 20 mL), sampling temperatures (40 and 60 °C), headspace conditioning (10 and 60 min) and fiber exposure times (15 and 40 min) were tested. For GC analyses two different internal standards, methyl octanoate and tetradecane, were tested, as well as sub-ambient oven temperatures with liquid nitrogen. A 1 g of sample and a divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PMDS) fiber were used in all the tests. Analytical curves from the FID data were constructed for linearity evaluation, whereas internal standard was used for quantification of compounds such as 3-hexenol, 2-hexanal and limonene. Identification was performed based on mass spectra, co-injection of standards and retention indices data. The best conditions for SPME analysis were sample temperature of 40 °C, 10 min of headspace conditioning and fiber exposure for 40 min, in a 4 mL flask. For the chromatographic analyzes, tetradecane was chosen as the internal standard. Oven temperature program with cryofocusing led to a much better separation and, therefore, better quantitation and identification of the more volatile compounds.

**Keywords:** Olive oil; Volatile compounds; SPME; GC-FID; GC-MS.

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# ANALYSIS OF VOLATILE COMPOUNDS IN EXTRA VIRGIN OLIVE OIL FROM RIO GRANDE DO SUL BY SPME/GC-MS

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Extra virgin olive oil (EVOO) has a complex aroma, which is related to the genetics of the olive tree (cultivar), the edaphoclimatic conditions where it is grown and the conditions of the extraction process. Koroneiki is a Greek variety, whose fruit is small and produces very fruity oil, with herbal and green apple notes besides having certain astringency, with notes of almonds, fig and peels. This cultivar is known for its resistance to water stress and windy conditions, as well as for producing exceptional quality oils with high yield. This work aimed to identify the volatile compounds present in a Koroneiki monovarietal EVOO sample from Rio Grande do Sul (Brazil), through solid-phase microextraction (SPME) and gas chromatography coupled to mass spectrometry (GC-MS). For SPME analysis, a divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PMDS) fiber was used. A 1.0 g sample and 0.2 µL of a standard solution of alkanes ($C_8$-$C_{18}$) were added to a 4 mL vial. The sample was conditioned (10 min, 40 °C) and the SPME fiber was exposed to the headspace of the sample for 40 min at the same temperature. Then, the fiber was transferred to the injector of a GC-MS for thermal desorption for 3 min (at 250 °C), in splitless mode. The oven of the GC was cooled with liquid nitrogen for a focusing effect. The identifications were made through the injection of authentic standards, calculation of the linear retention indices and comparison of the mass spectra to different databases. Eighteen compounds, including (E)-2-hexenal, (Z)-3-hexenal, characteristic compounds of olive oil, produced enzymatically from polyunsaturated fatty acids via the lipoxygenase pathway were identified. These compounds contribute positively to the aroma of EVOO; (E) -2-hexenal is responsible for bitter and astringent odor and (Z) -3-hexenal by green leaves.

**Keywords:** Olive oil; Koroneiki; volatile compounds; SPME; GC-MS.