

## Development of a gas chromatographic method for demonstrating adulteration of oregano

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Adulteration consists of adding a lower value compound to an initial commercial product. This fraudulent practice remains a major problem in the spice world. Indeed, in this context, the adulterated product is sold at the price of the pure product, inflicting loss to the buyer. Oregano is particularly affected by this behavior due to lower supply than demand. Sumac, olive, myrtle and cistus leaves are often used to adulterate oregano.

The objective of this study is to develop a simple protocol, based on gas chromatography, coupled with a flame ionization detector, to detect and identify some adulteration agents.

The approach implemented in this protocol is based on solvent ultrasonic extraction (hexane or methanol), followed by polar (ZB5MS) or less polar (DB17) gas chromatography.

Four samples of dried oregano obtained from commercial sources were analyzed five times each for the extraction, 100 mg of powder, added with 4 mL of solvent, were sonicated for 20 minutes at 30°C. The thermal chromatographic protocol is from 60°C to 290°C with, heating gradient defined 20°C per minute.

On one hand, analyses of the chromatograms obtained show that the DB17 column exhibits a greater number of peaks than those observed from the ZB5MS column, this, for both nature of solvent. On the other hand, the result obtained from hexane extracts, contain less volatile compounds than the methanolic extract. We observe a higher peaks density from high time retention of the chromatogram. However, these results should be compared with the results obtained for the usual adulterants of oregano in order to verify the relevance of this protocol or define the need to combine the protocols to detect the trace of adulterant.

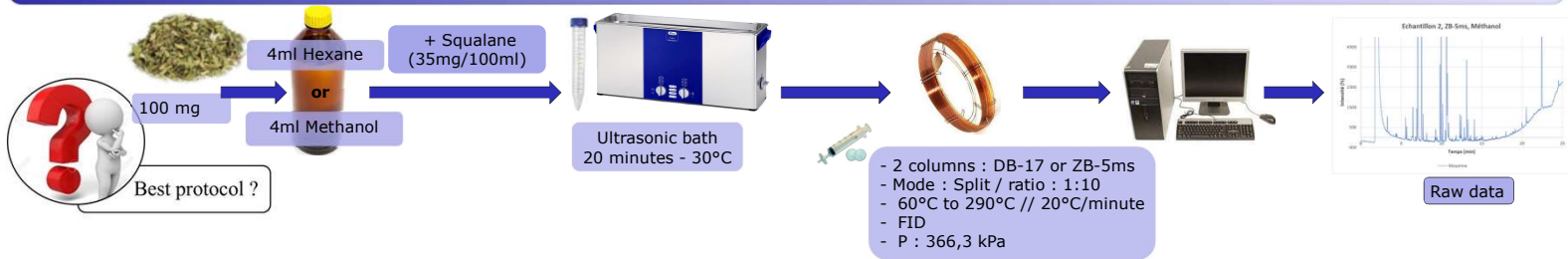
**Keywords:** Oregano, gas chromatography, adulteration

## Introduction

Adulteration is the consequence of adding a less valuable compound in a specific product and sell it at the same price of such original product. This fraudulent practice is common in the world of spices. Oregano is particularly concerned with fraud. The common adulterants of oregano are olive, sumac, cistus and myrtle leaves.

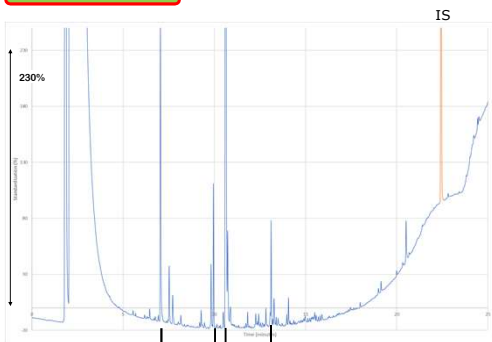
The objective of this study is to develop an analytical protocol based on gas chromatography for detecting oregano adulteration and identify the adulterants.

## Material and Method

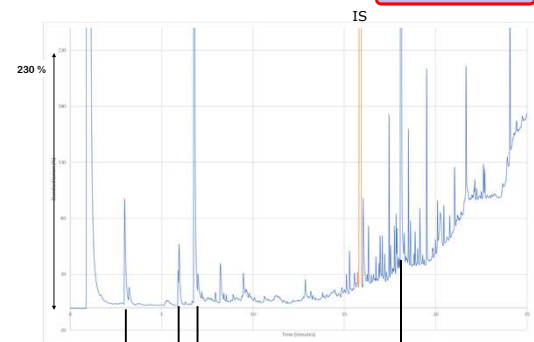


## Results

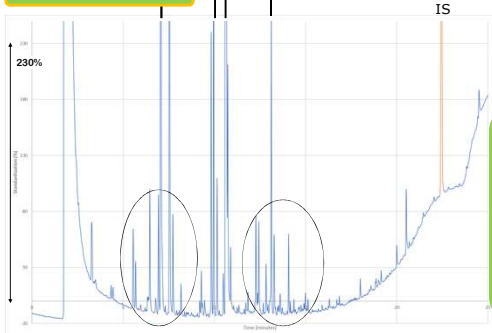
Hexane - ZB-5ms



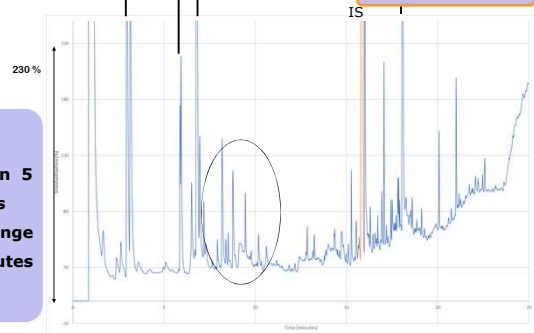
Hexane- DB-17



Methanol - ZB-5ms



Methanol - DB-17



### Treatment of data

#### 1. Averaging

$$\forall T_r ; y_{moy} = \frac{1}{5} \sum_{i=1}^5 y_i$$

With *i* oregano extracts

#### 2. Determination of $IS_{max}$ :

Maximal reponse of signal's internal standart peak

#### 3. Standardization

$$\forall T_r ; y_{st} = \frac{y_{moy}}{IS_{max}} * 100$$

### Results : Chromatograms

#### Hexane vs Methanol

- Chromatograms from methanolic extract contain more peaks that chromatograms from hexane

#### ZB-5ms vs DB-17

- Better separation between 5 and 15 minutes for ZB-5ms  
- Better separation in the range of 0 to 5 and 15 to 25 minutes for DB-17

## Conclusions and perspectives

The chromatograms show that the different column used allow separating the compounds in different way. Indeed, each column separates compounds but in different range of time. The chromatogram with methanol as solvent has more peaks than the chromatogram with hexane.

As each column separates compounds in a different time interval, the combination of both chromatograms could be an interesting way in order to extract a maximum of information. The treatment of the chromatograms with chemometric tools (i.e Principal Component Analysis) could be an interesting approach to extract such information.