# Analysis of Essential Oils by Ultra Fast GC: an Effective Technique for 30-Fold Speed Increase

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## Overview

**Purpose:** Ultra Fast Gas Chromatography is applied to the analysis of essential oils. By means of direct resistively heated columns the Ultra Fast GC system achieves very fast temperature programming allowing to shorten the GC analysis time significantly.

**Methods:** Essential Oils are diluted 1 to 200 in cyclohexane and 1 µl of the resulting solution is then injected in split mode in the Ultra Fast GC.

**Results:** The analyses are achieved with about 30-fold speed increase with respect to the conventional GC methods with good accuracy and precision. The separation of critical pairs is optimized by choosing the properly selective stationary phase.

## Introduction

An Essential Oils is the product of hydrodistillation of a plant or of a part of it; they mainly consists of terpenes and their oxygenated derivatives. Essential oils are generally applied in flavor and fragrance formulation in the food, cosmetic and pharmaceutical fields.
Essential Oils from the same species may strongly vary in composition depending on varieties, origin, pedoclimatic and preparation conditions. Therefore their characterization through chemical analysis is fundamental before application, and mainly carried out by both research and quality control laboratories through conventional methods based on GC and GC-MS. Such methods generally require 30-60 minutes for the overall analytical cycle.
This poster reports an alternative method to analyze essential oils based on the ultra fast GC technology [1] implemented on the Finnigan Trace GC Ultra (Thermo Electron), which operates with short narrow bore columns and with heating rates of hundreds degrees per minute. The outcome is a 30-fold reduction of the analysis time.

## Methods

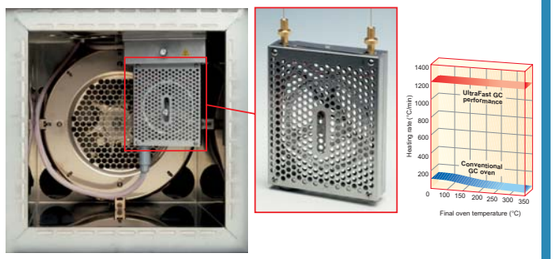
Four essential oils with different complexities were characterized through Ultra Fast GC and conventional GC: chamomile, sage, peppermint and lavender. These oils can be obtained by hydro-distillation according to the method described in the European Pharmacopoeia [2].

**Instrument Configuration:** A Trace GC Ultra (Thermo Electron) equipped with the Ultra Fast option was used. The option provides an ultra fast column module (UFM, figure 2) consisting of a fused silica column combined with a heating element and a temperature sensor such to insure the direct resistive heating of the capillary column. The assembly is held in an "easy to handle" metal cage and can achieve temperature programming rates as high as 20 °C/s. Being placed inside the oven of a conventional Trace GC Ultra (Figure 2), the module cools down fast (around 1 minute from °C to 50 °C) by automatically activating the oven fan at the end of the temperature program. Capillary columns with a broad range of lengths and diameters and featuring different stationary phases can be incorporated in the UF Module (Thermo Electron). The column module inlet was connected to the split-splitless injector and to the Fast FID [3].
The same Gaschromatograph was used to perform the same application in conventional mode, once the Ultra Fast accessory was removed.

**Analytical conditions:** Essential oils were diluted 1:200 in cyclohexane and 1 µl of the solution was injected. Injector temperature was set to 230 °C, and the FID set to 280 °C; all UFM the columns used were 5 m long, 0.1 mm i.d., 0.1 µm film thickness and were heated at 150-500 °C/min. In the conventional GC 25 m long, 0.25 mm i.d., µm f.t. columns were used with heating rates of 10 °C/min. All the capillary columns were supplied by MEGA (Legnano, Italy)

**FIGURE 1. Finnigan TRACE GC Ultra equipped with Ultra Fast Module option and AS3000 autosampler.**

**FIGURE 2. Ultra Fast GC: column module, installation in the Finnigan Trace GC Ultra oven, heating performance.**




## Results

**Chamomile**
Chamomile essential oils are characterized by seven sesqui-terpenoids present at different amounts depending on quality and origin. Apolar columns are not suitable to compensate the loss of efficiency due to operating at ultra fast heating rates, so a highly selective stationary phase is necessary.
The OV1701 features the winning compromise, being able to deliver base-line separation of the most critical pair α-bisabolone oxide A / α-bisabolol at the shortest analysis time.
The reduction in the analysis time with respect to conventional GC is above 30 times (figure 3).

**Sage**
Sage essential oils are medium complexity oils, whose composition can vary greatly depending on its origin. The sample analyzed belongs to the linalool-linalyl acetate chemotype and is characterized by 30 components of differing volatilities. An apolar SE54 column was used. The latter eluting component is a minor diterpenoid called sclareol, that requires to extend the analysis time up to 51 minutes to be eluted in conventional mode. Figure 4 reports the chromatograms acquired both in conventional and ultra fast (1.5 min) modes, pointing out a 30-fold reduction in analysis time.

**FIGURE 3. Comparison between Ultra Fast and conventional GC analysis of Chamomile essential oil using a OV-1701 column.**

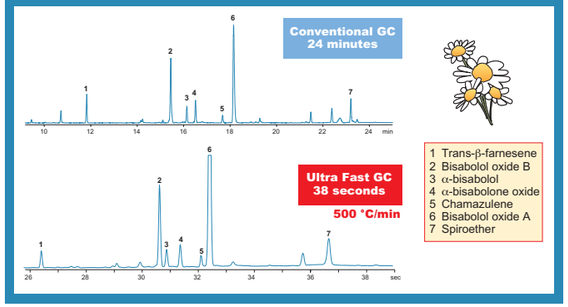
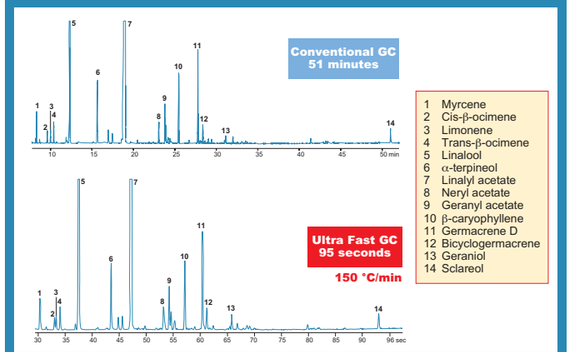
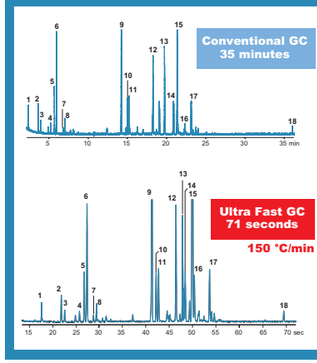



**FIGURE 4. Comparison between Ultra Fast and conventional GC analysis of a Sage essential oil using a SE-54 column**




**Peppermint**
Peppermint essential oils are medium complexity oils characterized by about 30 mono- and sesqui-terpenoids. The concentration of some of them (e.g. pulegone, menthofurane) is legally limited because of their toxicity. This oil is generally analyzed with a polar phase, CarboWax, since apolar phases are not selective enough to provide complete separation of some critical components. Figure 5, showing the peppermint chromatograms acquired in ultra fast (71 seconds) and in conventional (35 minutes) GC, points out an almost 30-fold reduction in the analysis time. The table reports the good accordance between conventional and ultra fast peak area % for all the components.

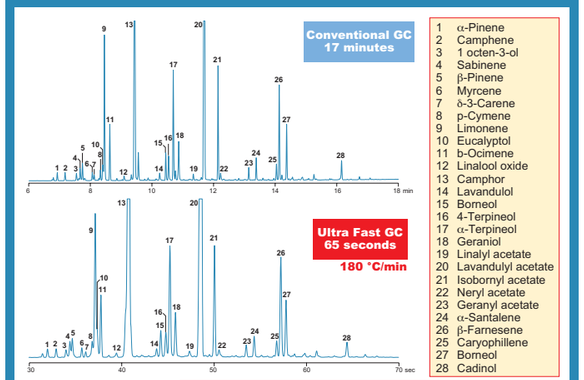
**FIGURE 5. Comparison between Ultra Fast and conventional GC analysis of a Peppermint essential oil using a CarboWax column.**




| | | Area % | |
|---|---|---|---|
| | | Ultra Fast GC | Conv. GC |
| 1 | α-pinene | 0.96 | 0.94 |
| 2 | β-pinene | 1.37 | 1.41 |
| 3 | Sabinene | 0.62 | 0.64 |
| 4 | Myrcene | 0.35 | 0.39 |
| 5 | p-cymene | 0.56 | 0.58 |
| 6 | Limonene | 2.58 | 2.47 |
| 7 | 1,8 cineole | 6.19 | 6.07 |
| 8 | γ-terpinene | 1.02 | 0.94 |
| 9 | Menthone | 14.18 | 14.21 |
| 10 | Menthofurane | 2.56 | 2.44 |
| 11 | i-menthone | 2.59 | 2.57 |
| 12 | Methyl acetate | 5.69 | 5.74 |
| 13 | Neomenthol | 5.88 | 5.95 |
| 14 | β-cariophyllene | 2.34 | 2.39 |
| 15 | Menthol | 47.26 | 47.49 |
| 16 | Pulegone | 2.58 | 2.54 |
| 17 | Germacrene D | 2.69 | 2.64 |
| 18 | Viridiflorol | 0.56 | 0.58 |

**Lavender**
A sequence of 10 consecutive injections of lavender essential oil was performed to evaluate repeatability. Figure 6 reports the repeatability on retention times and peak areas and the comparison with the conventional GC analysis. Good repeatability was found for both peak areas (RSD% around 1%), and retention times (SD around 40 ms) allowing a reliable identification and quantitation.

**FIGURE 6. Comparison between Ultra Fast and conventional GC analysis of a Lavender essential oil using a SE-54 column.**




| | | Precision | | | | Accuracy | |
|---|---|---|---|---|---|---|---|
| | | Retention times | | Peak areas | | Area % | |
| | | Mean (s) | SD (s) | Mean ($10^4$ counts) | RSD% | Ultra Fast GC | Conv. GC |
| 1 | α-Pinene | 31.80 | 0.05 | 94 | 1.32 | 0.28 | 0.28 |
| 9 | Limonene | 37.12 | 0.04 | 173 | 1.00 | 5.13 | 5.08 |
| 13 | Linalol | 40.79 | 0.04 | 998 | 0.60 | 29.30 | 28.60 |
| 15 | Lavandulol | 44.20 | 0.04 | 33 | 0.77 | 0.94 | 0.98 |
| 16 | Borneol | 44.76 | 0.04 | 34 | 0.76 | 0.95 | 0.98 |
| 18 | α-Terpineol | 45.80 | 0.04 | 57 | 0.78 | 1.67 | 1.65 |
| 20 | Lavandulyl acetate | 50.03 | 0.04 | 128 | 0.64 | 38.30 | 38.60 |
| 22 | Neryl Acetate | 53.45 | 0.04 | 15 | 1.28 | 0.37 | 0.30 |
| 25 | Caryophillene | 57.19 | 0.04 | 127 | 0.63 | 0.56 | 0.58 |
| 26 | β-Farnesene | 57.75 | 0.04 | 69 | 0.82 | 3.74 | 3.40 |
| 28 | Cadinol | 64.35 | 0.04 | 21 | 0.56 | 0.60 | 0.71 |

## Conclusions

A 30-fold reduction of the analysis time in the characterization of essential oils was obtained by applying Ultra Fast GC with direct resistively heated capillary columns. The perfect agreement with the conventional GC method and the high precision of the data obtained prove the suitability of this technique for the routine analysis of these complex mixtures.

## References

[1] C. Bicchi, C. Brunelli, C. Cordero, P. Rubiolo, M. Galli, A. Sironi, J. Chromatogr. A, 1024 (2004) 195-207
[2] European Pharmacopoeia, 3rd Edition, Council of Europe publishing, Strasbourg (France), (1997), 1146
[3] P. Magni, R. Facchetti, D. Cavagnino, S. Trestianu, in: P. Sandra (Ed.), Proceedings of 25th International Symposium of Capillary Chromatography, Riva del Garda, Italy, 13-17 May 2002, KLN05

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