Figure 1. Schematic representation of the configuration of the GCx2GC-MS/FID system used.

An application note that shows the benefits and the potential of the Comprehensive two-dimensional gas chromatography (GCxGC) coupled with mass spectrometry (MS) - using MEGA GC columns (MS, FAST and Chiral DEX columns) - for detailed analysis, identification and quantitation of medium-to-high complexity mixtures is here presented.

In this study, a GCx2GC-MS/FID platform (see Figure 1) consisting of one primary column (1D) coupled to two parallel secondary columns (2D) having an identical inner diameter, stationary phase chemistry and film thickness, which, in turn, are connected to two detectors, a fast quadrupole MS and a FID, was adopted for quantitative profiling of essential oils (EOs). Two medium complexity EOs (i.e. *Mentha* and *Lavandula* species) with different quantitation challenges were taken as samples and a selection of quality markers subjected to an extensive method performance evaluation.

The advantages of this approach can be resumed here:

a. to assess data/results over an extended time frame, varied instrumentation and different laboratories;

b. to interpret the biological role of (potential) biomarkers;

c. to evaluate the impact of potent odorants; and/or

d. to define product quality, e.g. relative to a reference standard.

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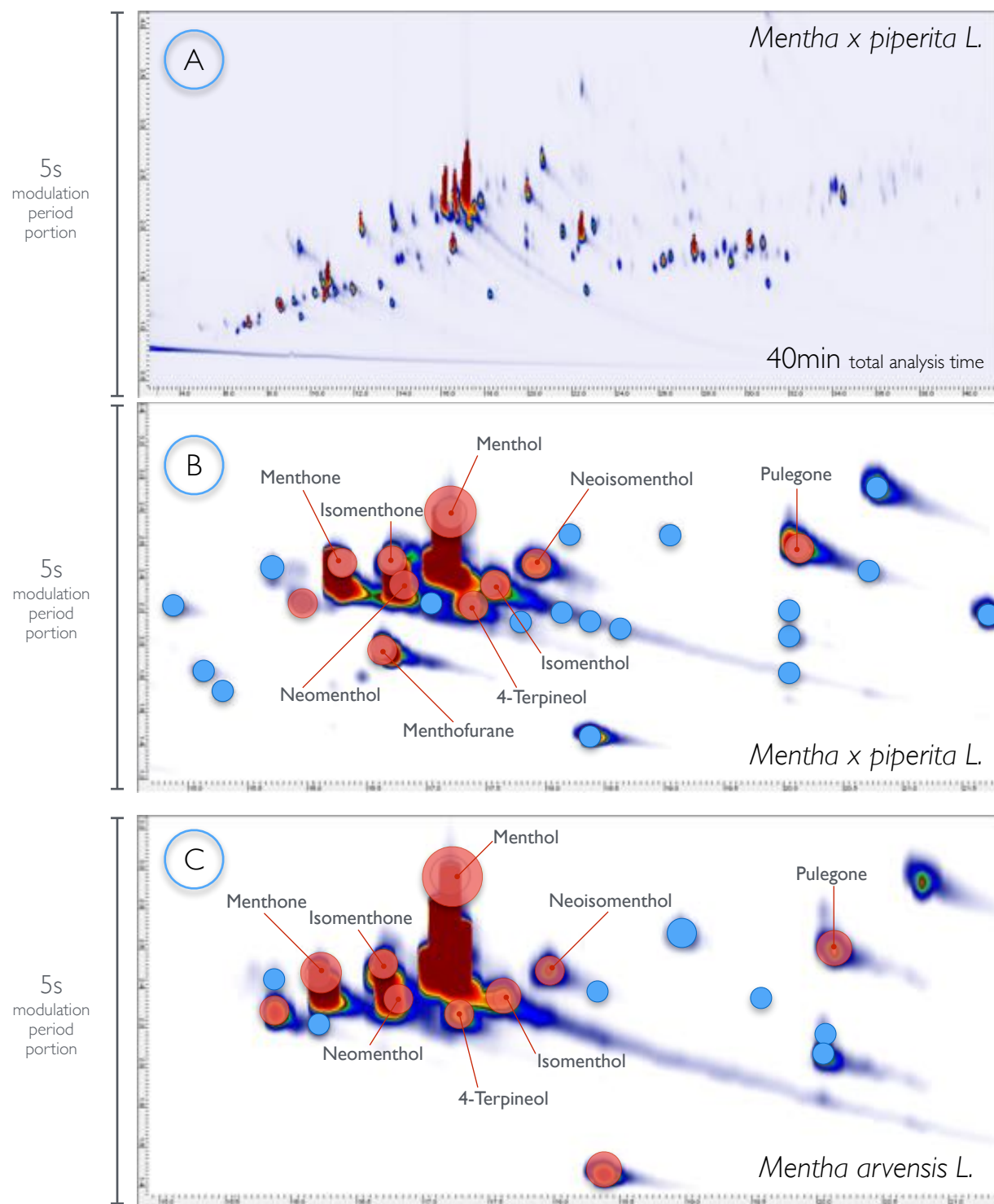
Figure 2.

Conditions:

Injection: Split 280°C, 1:20 split ratio, 1µL injection volume.

Sample dilutions: 5, 2, 1mg/mL and 500µg/mL in dichloromethane or cyclohexane.

Detector: FID 280°C, MS operating in EI mode at 70 eV, transfer line 280°C, scan range set to m/z 40-250 with a scan rate of 12500 amu/s. Split ratio MS/FID is 50:50.

Oven Program: 50°C (1min), 3°C/min, 270°C, 10°C/min, 280°C (10min).

Carrier Gas: Helium, 296 kPa head pressure, constant flow.

Modulator: two-stage loop thermal modulator, modulation time 5 s.

Column set:

1D GC column:
MEGA-SE52 MS - 0.25mm, 0.25µm, 30m
Catalog Code: MS-SE52-025-025-30

2D GC columns:
MEGA-1701 FAST - 0.10mm, 0.10µm, 1.4m
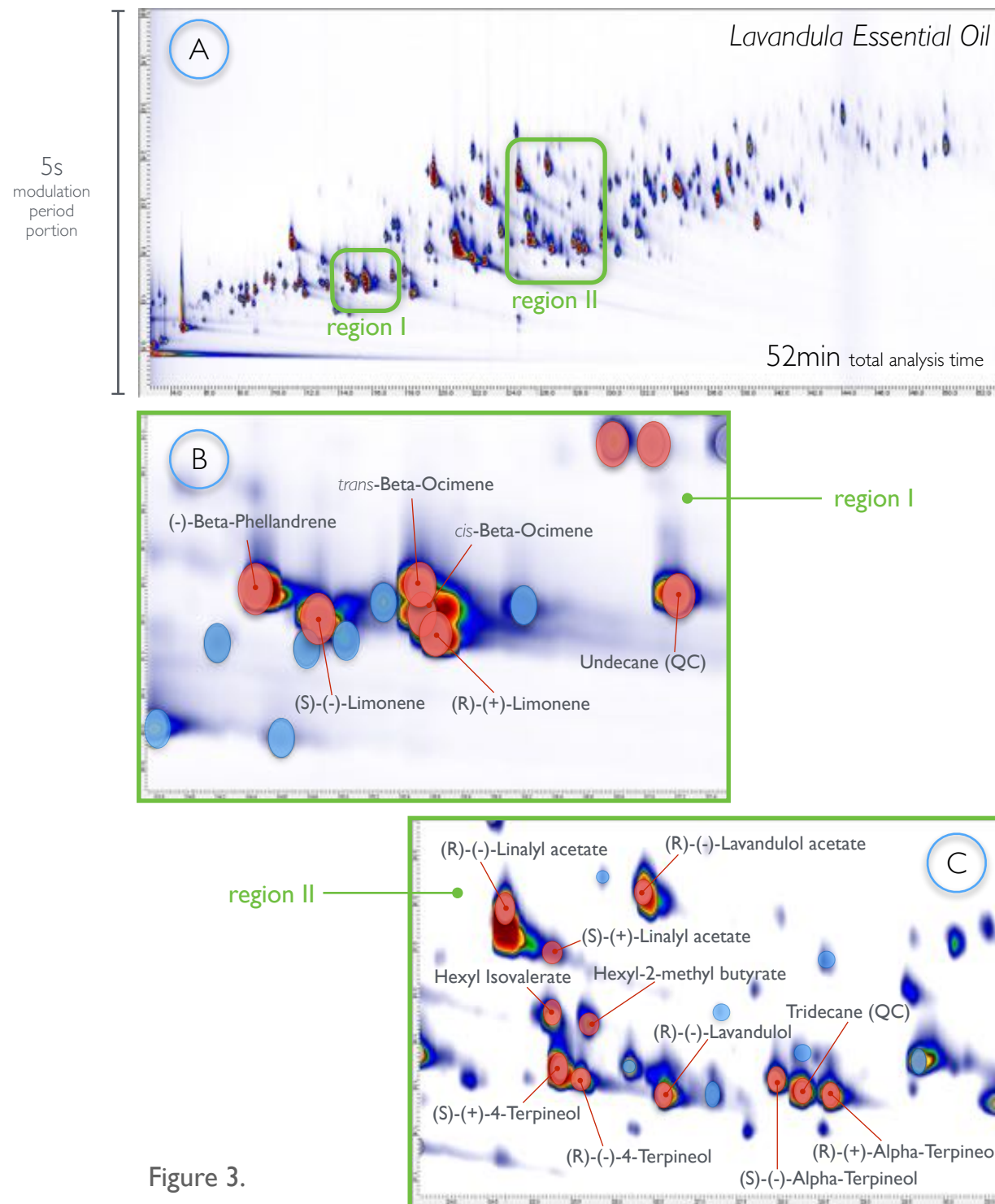Catalog Code: F-1701-010-010-1-4

Figure 2 represents the 2D plot of menthol-rich essential oils (EOs):

(A) peppermint (CS PEPP) 2D elution pattern

(B) elution region of menthols in peppermint (*Mentha x piperita L. - CS PEPP*)

(C) elution region of menthols in cornmint (*Mentha arvensis L. - CS ARV*)

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Lavandula Essential Oil - **GCx2GC-MS/FID**

A

Lavandula Essential Oil

5s
modulation
period
portion

region I

region II

52min total analysis time

Conditions:

Injection: Split 280°C, 1:20 split ratio, 1μL injection volume.

Sample dilutions: 5, 2, 1mg/mL and 500μg/mL in dichloromethane or cyclohexane.

Detector: FID 280°C, MS operating in EI mode at 70 eV, transfer line 280°C, scan range set to m/z 40-250 with a scan rate of 12500 amu/s. Split ratio MS/FID is 50:50.

Oven Program: 60°C (1min), 2°C/min, 180°C, 10°C/min, 230°C (5min).

Carrier Gas: Helium, 296 kPa head pressure, constant flow.

Modulator: two-stage loop thermal modulator, modulation time 5 s.

B

trans-**Beta-Ocimene**

(-)-Beta-Phellandrene

cis-**Beta-Ocimene**

region I

Undecane (QC)

(S)-(-)-Limonene   (R)-(+)-Limonene

Column set:

1D GC column:

MEGA-DEX DET Beta - 0.25mm, 0.25μm, 25m
Catalog Code: DEX-DET-B-025-025-25

2D GC columns:

MEGA-1701 FAST - 0.10mm, 0.10μm, 1.4m
Catalog Code: F-1701-010-010-1-4

C

region II

(R)-(-)-Linalyl acetate   (R)-(-)-Lavandulol acetate

(S)-(+)-Linalyl acetate

Hexyl Isovalerate   Hexyl-2-methyl butyrate

(R)-(-)-Lavandulol

Tridecane (QC)

(S)-(+)-4-Terpineol

(R)-(-)-4-Terpineol   (R)-(+)-Alpha-Terpineol

(S)-(-)-Alpha-Terpineol

Figure 3.

Figure 3 represents the 2D plot of *Lavandula angustifolia* Miller essential oil (EO) (A) obtained by MEGA-DEX DET Beta / MEGA-1701 FAST columns combination. Two sub-regions are also shown (B - region I and C - Region II) where components are effectively resolved in the 2D.

Acknowledgments:

Original and full paper (from which some of the results here presented were extracted) is available on the IRIS "AperTO" archive of the University of Turin at this link:

http://hdl.handle.net/2318/1520994

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