

Supplementary Materials

Comparison of Supercritical Fluid Chromatography Hyphenated to An Ultraviolet Detector and Gas Chromatography Hyphenated to a Flame Ionization Detector for Qualitative and Quantitative Analysis of *Citrus* Essential Oils

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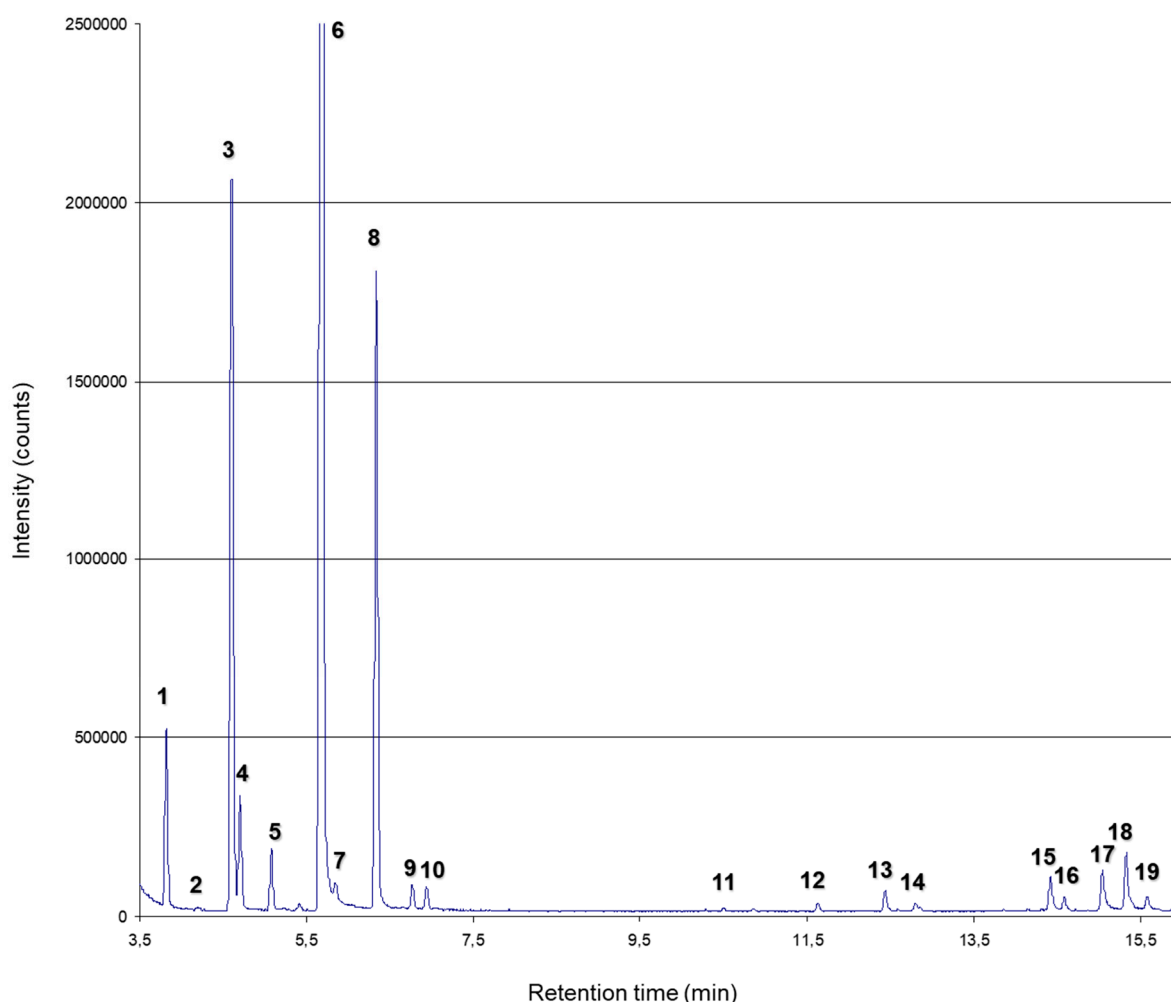


Figure S1. Chromatogram and peak identification obtained by GC-MS using column SUPEL-COWAX® 10 (30 m x 0.25 mm x 0.25 mm); temperature from 75 °C to 100 °C at 5 °C/min then from 100 °C to 220 °C at 6 °C/min; helium as carrier gas at a flow rate of 1 mL/ min. FID temperature was set at 260 °C. (1) α -pinene, (2) camphene, (3) β -pinene, (4) sabinene, (5) myrcene, (6) limonene, (7) β -phellandrene, (8) γ -terpinene, (9) p-cymene, (10) terpinolene, (11) citronellal, (12) linalol, (13) α -E-bergamotene, (14) E-caryophyllene, (15) neral, (16) α -terpineol, (17) β -bisabolene, (18) geranial, (19) geranyl acetate.

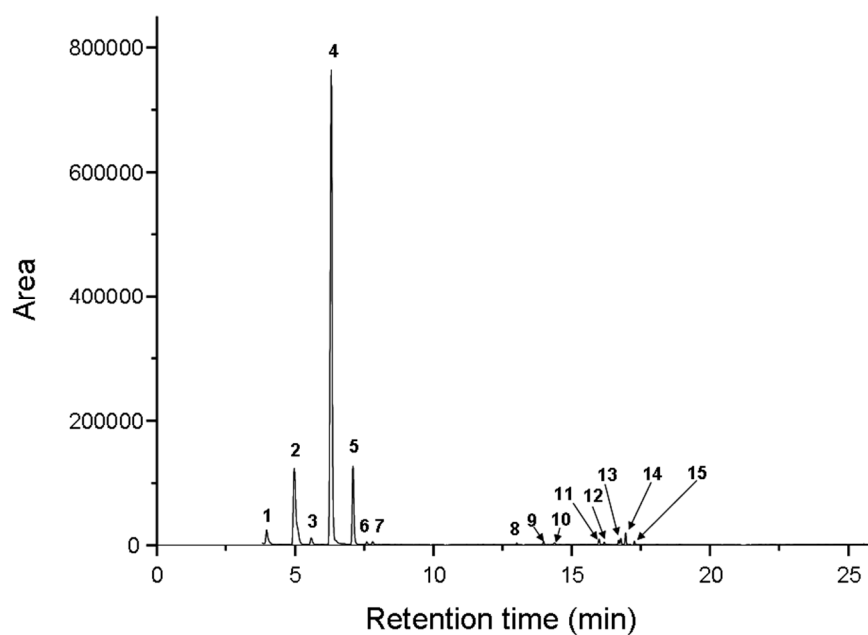


Figure S2. Chromatogram and peak identification obtained by GC-FID using column SUPEL-COWAX® 10 (30 m × 0.25 mm, 0.25 mm film thickness); temperature from 75 °C to 100 °C at 5 °C/min then from 100 °C to 220 °C at 6 °C/min; helium as carrier gas at a flow rate of 1 mL/ min. FID temperature was set at 260 °C. (1) α -pinene, (2) β -pinene, (3) myrcene, (4) limonene, (5) γ terpinene, (6) p-cymene, (7) terpinolene, (8) linalol, (9) α -E-bergamotene, (10) E-caryophyllene, (11) neral, (12) α -terpineol, (13) β -bisabolene, (14) geranial, (15) geranyl acetate.

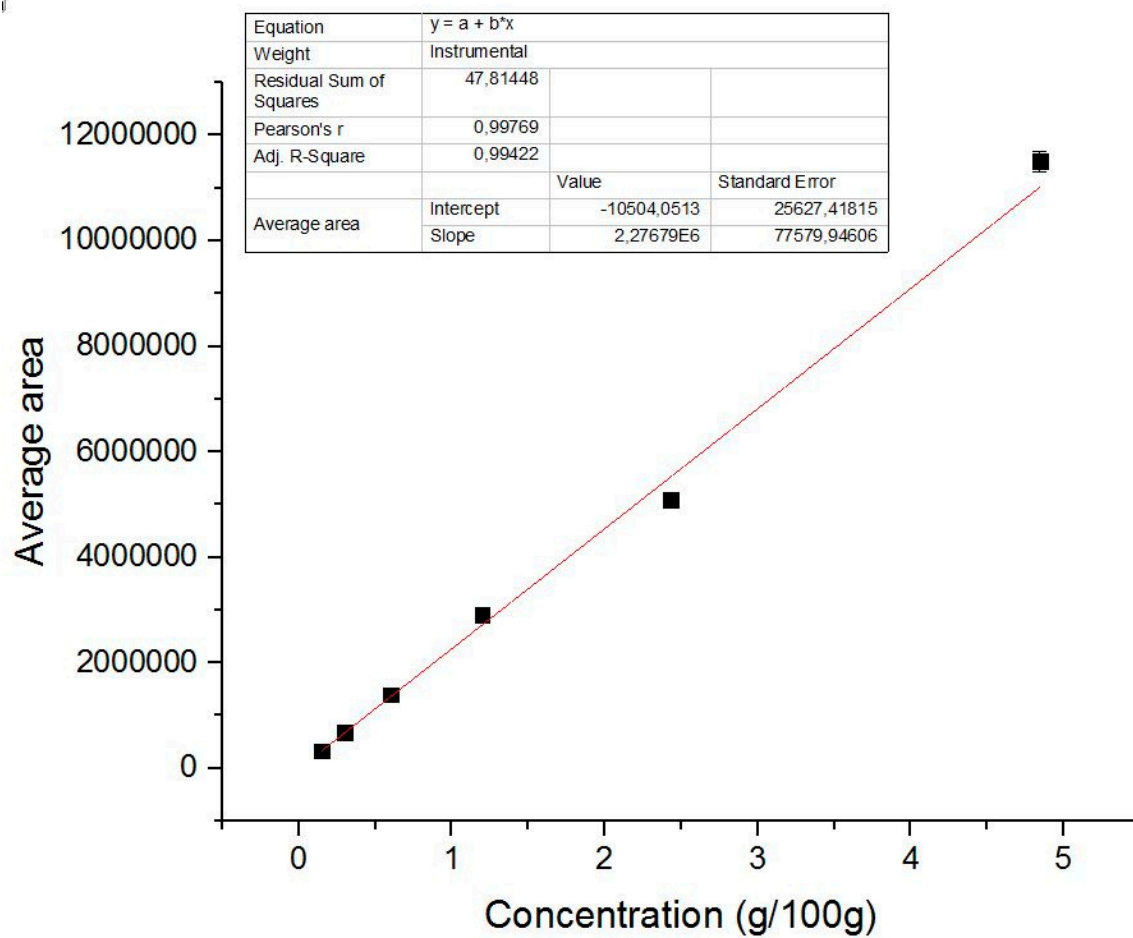


Figure S3. GC-FID R-limonene calibration curve obtained using Origin software.

Table S1. Raw data used to obtain the GC-FID calibration curve. Standard concentration used between 4.85 and 0.15 g/100 g of limonene. Injections were performed in triplicate. Chromatographic conditions: injection volume was set at 0.1 µL using a split ratio of 100:1. The following parameters were used for analyses: injector temperature 230 °C, column SUPELLOWAX® 10 (30 m x 0.25 mm x 0.25 µm); temperature from 75 °C to 100 °C at 5 °C/min then from 100 °C to 220 °C at 6 °C/min; helium as carrier gas at a flow rate of 1 mL/ min. FID temperature was set at 260°C.

	Standard concentration (g/100g)	Area number 1	Area number 2	Area number 3	Area average	Standard deviation	CV%
Level 1	4.85	11724420	11384934	11405466	11504940	190352	1.65
Level 2	2.43	5035676	5060222	5185914	5093937	80594	1.58
Level 3	1.20	2907819	2989087	2850549	2915818	69615	2.39
Level 4	0.60	1371185	1409233	1410993	1397137	22492	1.61
Level 5	0.30	672275	682172	699481	684643	13770	2.01
Level 6	0.15	322907	320431	332309	325216	6267	1.93

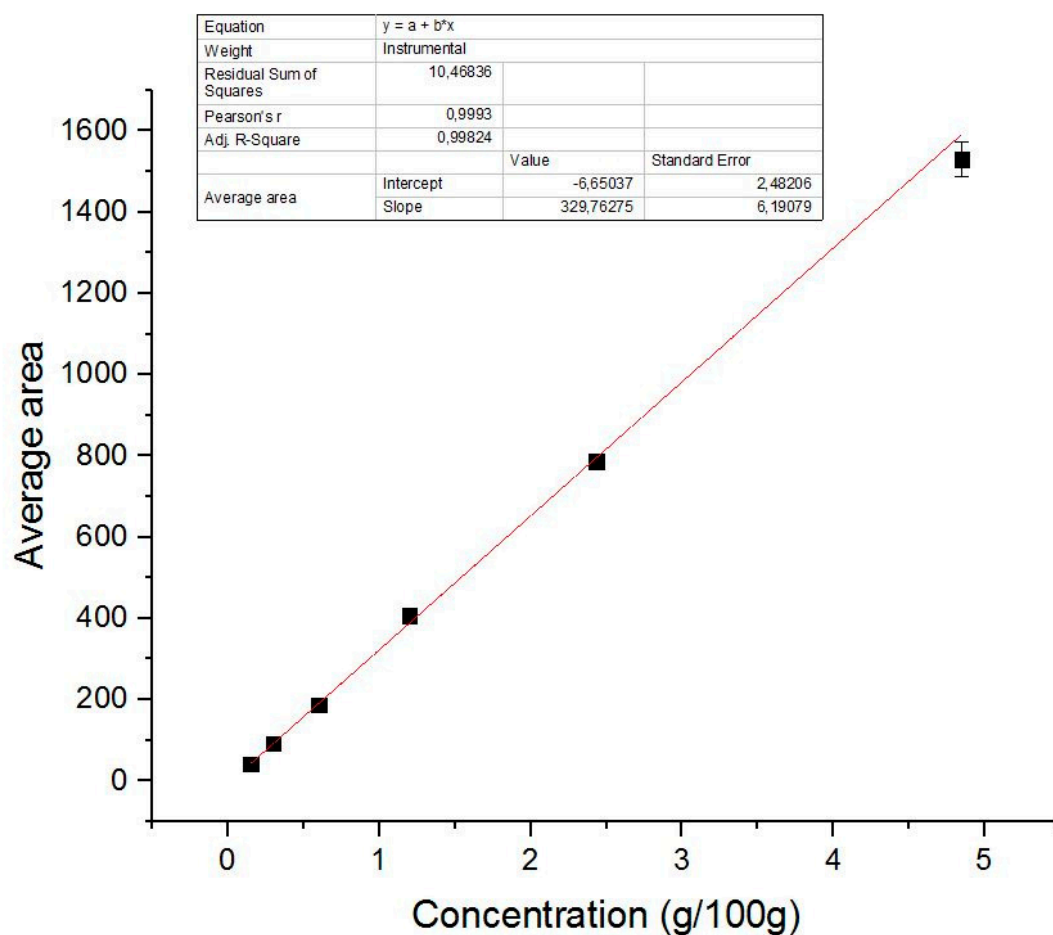


Figure S4. SFC-UV R-limonene calibration curve obtained using Origin software.

Table S2: Raw data used to obtain the SFC-UV calibration curve. Standard concentration used between 4.85 and 0.15 g/100 g of limonene. Injections were performed in triplicate. Chromatographic conditions: Two columns in series were used for chromatography, i.e. a Hypercarb® (150 mm x 4.6 mm x 5 µm, Thermo Scientific, USA) and a Poly-(butylene terephthalate) DCpack PBT (150 mm x 4.6 mm x 5 µm, DAICEL Corporation, USA). The mobile phase consisting of CO₂ (A) and methanol (B) was introduced at a flow rate of 1.5 mL/min according to the following gradient: 0.0–3.0 min (0% B), 3.0–16.0 min (0–10% B), 16.0–17.0 min (10–0% B) and 17.0–19.0 min (0% B). The column temperature was kept at 60 °C and the back-pressure was fixed at 90 bar.

	Standard concentration (g/100g)	Area number 1	Area number 2	Area number 3	Area average	Standard deviation	CV%
Level 1	4.85	1545.7	1561.5	1480.2	1529.1	43.1	2.82
Level 2	2.43	799.8	775.1	782.5	785.8	12.7	1.62
Level 3	1.20	399.0	413.4	408.5	407.0	7.3	1.80
Level 4	0.60	181.3	190.6	187.5	186.5	4.7	2.54
Level 5	0.30	90.3	94.9	94.0	93.1	2.4	2.61
Level 6	0.15	40.8	43.5	42.8	42.4	1.4	3.35