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Qualitative and Quantitative Determination of Furanocoumarins in Citrus Oils by LC/TOF

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Introduction

Furanocoumarins are natural compounds found in plants, including citrus fruits, parsley, celery and carrots, and may serve as a defense mechanism against predators such as insects and mammals. In humans, they have been shown to have photogenotoxic / photocarcinogenic properties when exposed to long wavelength UVA on skin, as well as act as irreversible inhibitors of cytochrome P450 3A enzymes in the liver and small intestine. The International Fragrance Association (IFRA) recently set safety standards of furocoumarins in fragrance and cosmetic products (5 ppm).¹

The chemical structure of furanocoumarins consists of a furan ring fused with a coumarin (Figure 1). The furan ring, fused in various ways, produces several different isomers. The parent compounds of the most common isomers are psoralen and angelicin. Historically, many laboratories used long (45 min) LC/UV detection methods to separate the isomers chromatographically. Here, a 15 min LC/TOF method is shown to be linear, accurate and precise for the characterization and quantitation of fifteen common furanocoumarins including isomers in citrus oils.

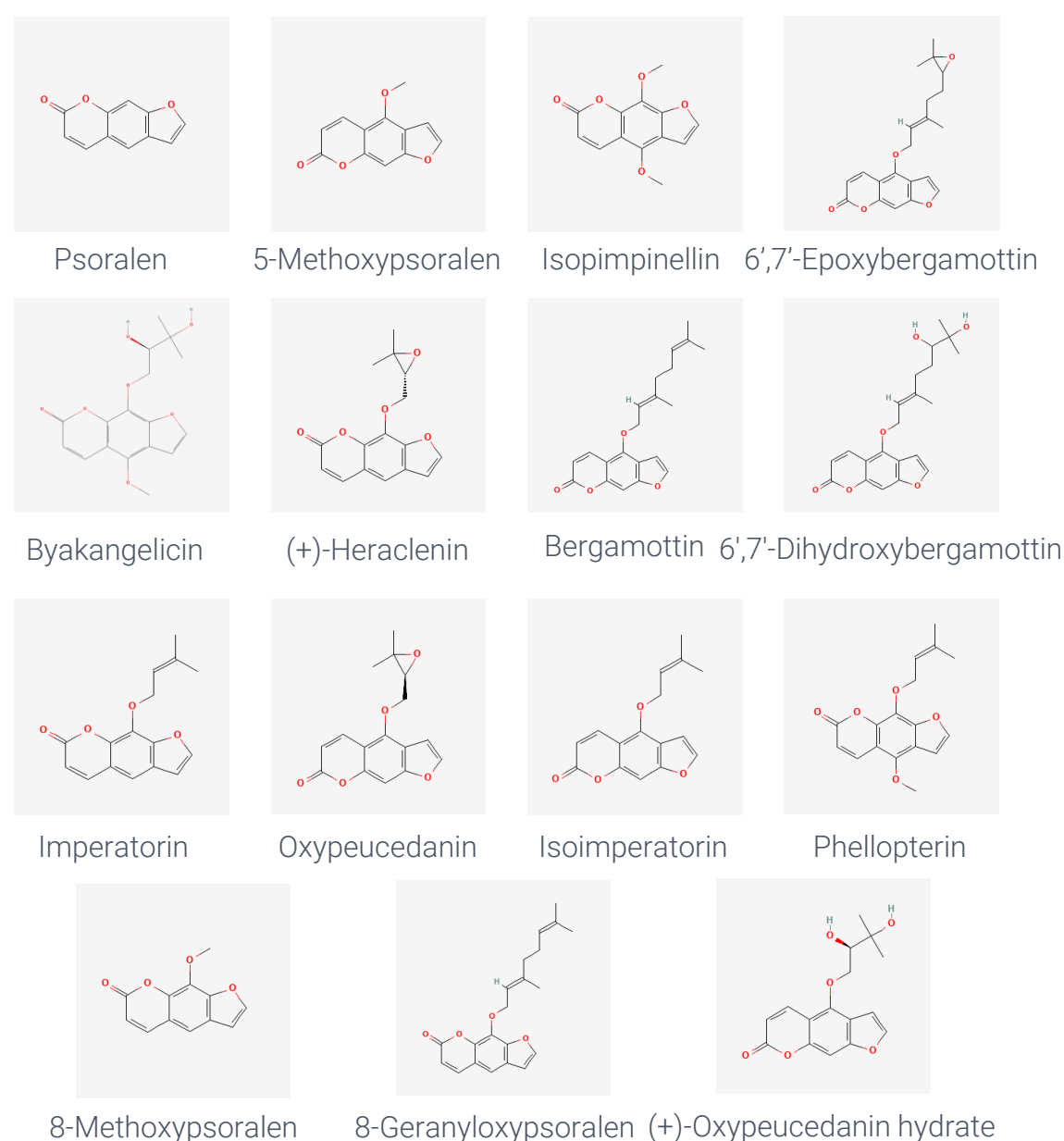


Figure 1. Chemical structures of furanocoumarins under investigation.

Experimental

Instrumentation

All experiments in this study were performed using an [Agilent 1290 Infinity III LC](#) consisting of an Agilent 1290 Infinity III Multisampler, an Agilent 1290 Infinity Flexible Pump, and an Agilent 1260 Infinity III Multicolumn Thermostat coupled to an [Agilent Time-of-Flight Mass Spectrometer](#) (G6230B). The system was controlled by Agilent MassHunter software, version 11. Data processing was performed using the same Agilent MassHunter software.

Samples, Standards and Consumables

Column:

- InfinityLab Poroshell 120 SB-C8, 3.0 x 100 mm, 2.7 μ m (Agilent PN 685975-306).

Solvents:

- LC-MS grade Water (Agilent PN 5191-4498)
- 99.5% Formic Acid (Agilent PN G2453-85060)
- LC-MS grade Acetonitrile (ACN) LCMS Grade (Millipore Sigma PN AX0156-6)

Standards:

- Furocoumarin Mix (Supelco PN 93102, 16 components in Acetonitrile, Certified Reference Material, TraceCERT®)



Figure 2. Agilent 6230C Time-of-Flight Mass Spectrometer coupled to a 1290 Infinity III LC stack.

Chromatographic separation of 15 furocoumarins and their respective isomers

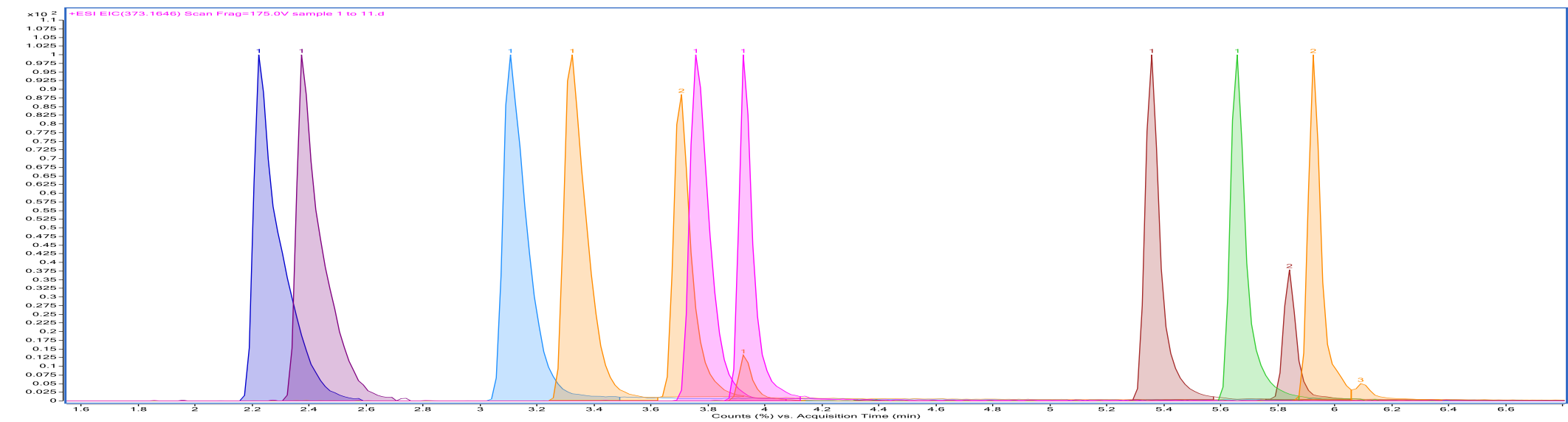


Figure 2. Normalized extracted ion chromatograms of 15 furanocoumarins showing separation of furocoumarins.

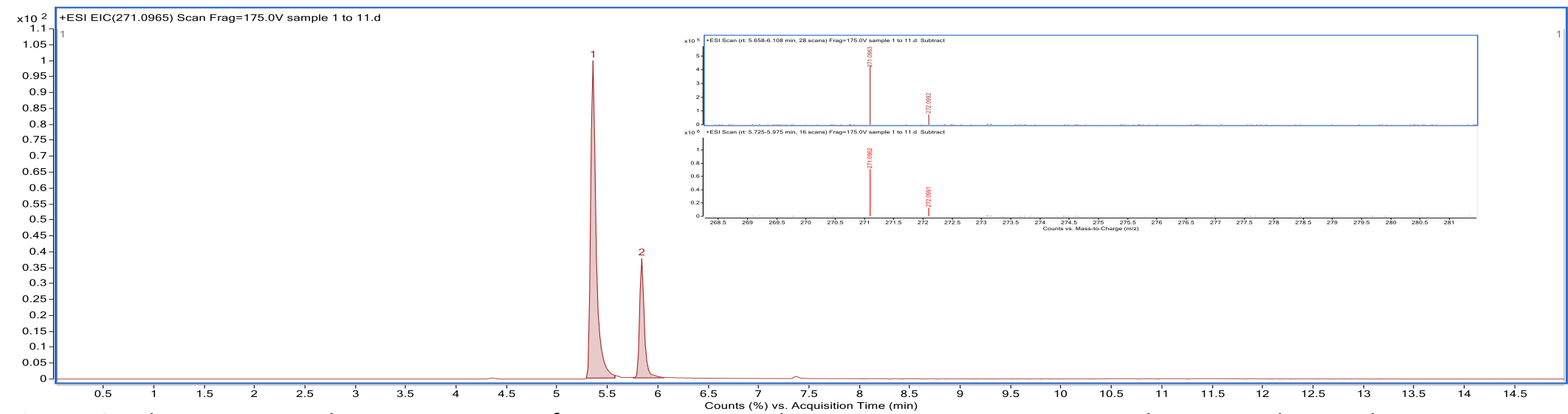


Figure 3. Chromatographic separation of imperatorin and isoimperatorin isomers. The inset shows the mass spectra of each isomer.

Method Precision and Accuracy

Compound	Precision		Accuracy		
	15 Replicate Average (10 ppm)	RSD (%)	Spiked Recovery Average (100 ppb)	STD	RSD (%)
Psoralen	10.4771	1.6	102.53	3.049	2.973
5-Methoxypsoralen	10.2888	2.1	99.94	4.468	4.471
8-Methoxypsoralen	10.5809	1.4	96.66	6.214	6.429
Isopimpinellin	10.3558	1.9	101.92	3.889	3.816
Oxypeucedanin	10.7032	1.3	99.87	5.670	5.678
Oxypeucedanin hydrate	10.6833	1.8	100.96	7.953	7.877
Byakangelicin	10.6076	1.6	100.54	4.058	4.037
(+)-Heraclenin	10.7057	1.6	98.22	2.469	2.514
8-geranyloxypsoralen	10.6131	2.2	100.91	4.750	4.707
Bergamottin	10.6537	1.7	102.35	2.666	2.605
Imperatorin	10.6966	1.3	98.14	5.247	5.346
Isoimperatorin	10.4719	1.5	99.93	4.136	4.139
Phellopterin	10.5079	2.0	99.22	5.340	5.382
6'7'-Epoxybergamottin	10.7593	1.8	101.70	5.188	5.101
6'7'-Dihydroxybergamottin	10.4809	1.2	102.00	5.542	5.433

Experimental Continued

Instrument Parameters

LC Gradient

Time	H ₂ O + FA	ACN	H ₂ O
0 Min	5%	30%	65%
10 Min	5%	95%	0%

LC Parameters

Parameter	Setting
Analytical Column	Agilent InfinityLab Poroshell 120 SB-C8, 3.0 x 100 mm, 2.7
Column Oven	60.0 °C
Injection Volume	1.0 µl
Run Time	15.00 min.
Post-run Time	5.00 min.
Mobile Phase Flow Rate	0.6 ml/min

Mass Spectrometer Parameters

Parameter	Value
Ion Source	Dual ESI
Polarity	Positive
Gas Temperature (°C)	325
Drying Gas Flow (l/min)	8
Nebulizer (psig)	35
Capillary Voltage (V)	3500
Fragmentor (V)	175
Skimmer1 (V)	65
Oct 1 RF (V RF)	750
Mass Range (m/z)	100-1700
Acquisition Rate (spectra/sec)	1.0

Results and Discussion -Part 2

Physical properties and coefficient of determination (R²) of furocoumarins (10 – 100 ppb)

Compound	R ²	Formula	H ⁺ adduct (m/z)	Na ⁺ adduct (m/z)	RT (min)
Psoralen	0.997	C ₁₁ H ₆ O ₃	187.039	209.029	3.766
5-Methoxypsoralen	0.998	C ₁₂ H ₈ O ₄	217.0495	239.0315	4.083
8-Methoxypsoralen	0.997	C ₁₂ H ₈ O ₄	217.0495	239.0315	4.652
Isopimpinellin	0.996	C ₁₃ H ₁₀ O ₅	247.0601	269.042	4.736
Oxypeucedanin	0.998	C ₁₆ H ₁₄ O ₅	287.0194	309.0733	5.201
Oxypeucedanin hydrate	0.998	C ₁₆ H ₁₆ O ₆	305.102	327.0839	3.07
Byakangelicin	0.996	C ₁₇ H ₁₈ O ₇	335.1125	357.0945	3.26
(±)-Heraclenin	0.996	C ₁₅ H ₁₆ O ₃	245.1172	267.0992	7.771
8-geranyloxypsoralen	0.998	C ₂₁ H ₂₂ O ₄	339.1591	361.141	5.791
Bergamottin	0.997	C ₂₁ H ₂₂ O ₄	339.1591	361.141	10.578
Imperatorin	0.998	C ₁₆ H ₁₄ O ₄	271.0965	293.0784	7.373
Isoimperatorin	0.998	C ₁₆ H ₁₄ O ₄	271.0965	293.0784	8.005
Phellopterin	0.997	C ₁₇ H ₁₆ O ₅	301.1071	323.089	7.794
6'7'-Epoxybergamottin	0.996	C ₂₁ H ₂₂ O ₅	355.154	377.1359	5.7991
6'7'-Dihydroxybergamottin	0.996	C ₂₁ H ₂₄ O ₆	373.1646	395.1465	3.281

Table 1. Molecular formula, H⁺ and Na⁺ adduct m/zs and retention times for furocoumarins in the standard mix. All furocoumarins in the standard mix showed linear quantitation (R²) from 10 – 100 ppb.

Conclusions

Detection, characterization and quantitation of furocoumarins using an LC/TOF method is shown to be:

- Linear
- Accurate
- Precise

References

¹ Irizar, A., et al., 2025. Phototoxicity and skin damage: A review of adverse effects of some furocoumarins found in natural extracts. Food and Chemical Detection,



<https://www.agilent.com/en/promotions/asms>

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