# High-Resolution Characterization of Bio-Oils Using Reversed-Phase LC Coupled to an 18 T FT-ICR Mass Spectrometer

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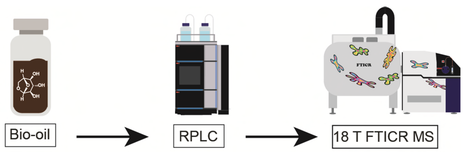
## Introduction

**Direct introduction** Fourier-transform ion cyclotron resonance mass spectrometry is typically used to evaluate upgrading efficiency at the molecular level.[1] To obtain information on the **isomeric content** of these samples, the addition of a **separation method** is required.




Here we evaluate the advantage of **liquid chromatography** coupled to **FTICR MS** equipped with a **18 teslas superconducting magnet** for the characterization of different upgraded.[2]

## 1. Method development

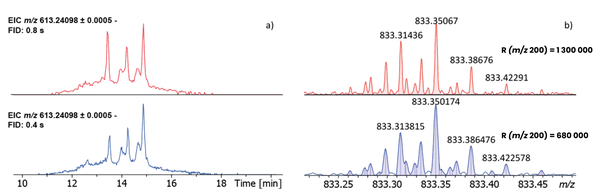



Figure 1. Adjustment of the **FID length** to ensure enough **resolving power** and **scanning rate**

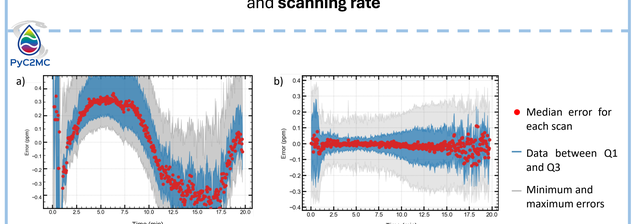



Figure 2. **Attribution error** as a function of time before calibration in (a) and attribution error after calibration, **scan by scan**, using the PyC2MC software

## 2. Advantages of coupling

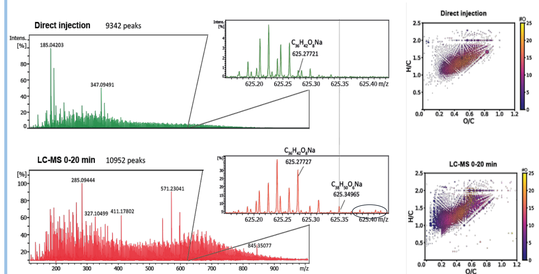



Figure 3. Comparison between **direct infusion** and **LC-FTICR** run on the same sample. Van Krevelen diagrams are given for both mass spectra to highlight the decrease of ionization effect with the chromatography.

Nearly **1000 additional** peaks thanks to **reduced ionization competition**

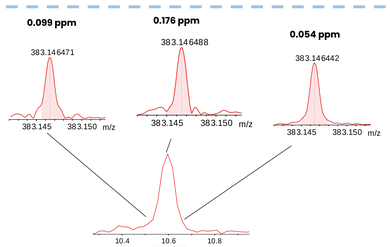



Figure 4. **Mass deviation** between the molecular formula and the peak apex value of the ion at *m/z* 383.14648 at the beginning, middle and end of the **chromatogram**

Despite the absence of ion current control, mass shifts at *m/z* 383.14648 remain **very low** (<0.2 ppm) over a wide TIC range, mainly due to the 18T magnet **limiting space-charge effects.**

## 3. Application to bio-oils

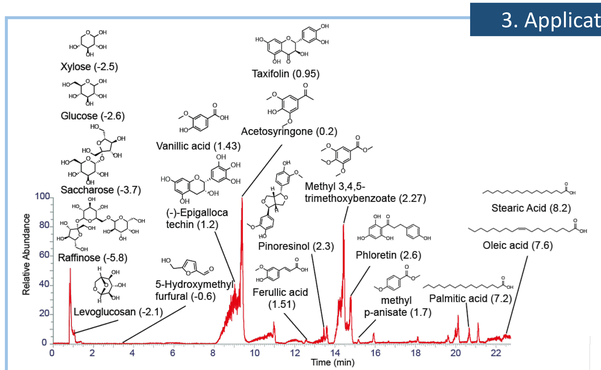



Figure 5. Base peak chromatogram (BPC) of **21** ESI(+) ionized **standards**, LC-FTICR MS coupling. The **logP values** are indicated in parentheses

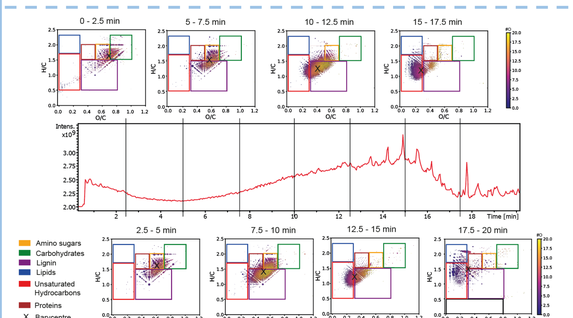



Figure 6. **Van Krevelen diagrams** of the bio-oil obtained with pyrolysis along the **chromatographic analysis**.

Trends in **compound families** according to elution time: carbohydrates, lignin derivatives, unsaturated hydrocarbons, lipids **confirming** the elution order of the standards for the **entire** sample

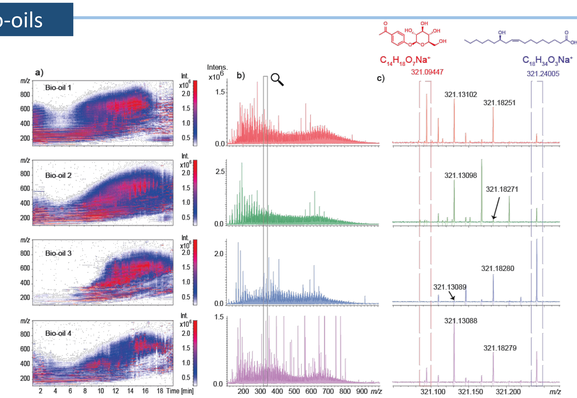



Figure 7. Heatmap, average mass specta and zoom at *m/z* 321 of the **four analyzed bio-oils.**

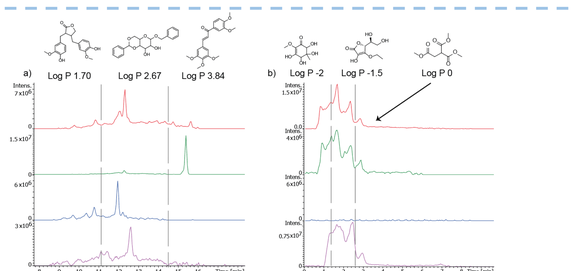



Figure 8. Extracted ion chromatograms of (*m/z* 381.13086 in a) and *m/z* 227.05261 in b)) . Different **isomeric profiles** are observed. Putative **structures** are also provided for each one.

## References

[1] Mase, C. et Al. *Journal of Analytical and Applied Pyrolysis* 2024, 177.
[2] Wootton, C. et Al. *Anal. Chem.* 2024, 96 (28), 11343–11352

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