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Headspace-Solid Phase Micro Extraction-Gas Chromatography-Quadrupole Mass Spectrometry-based metabolomics for kinetics tracking of natural herbicides' volatile residues: a simple non-destructive method

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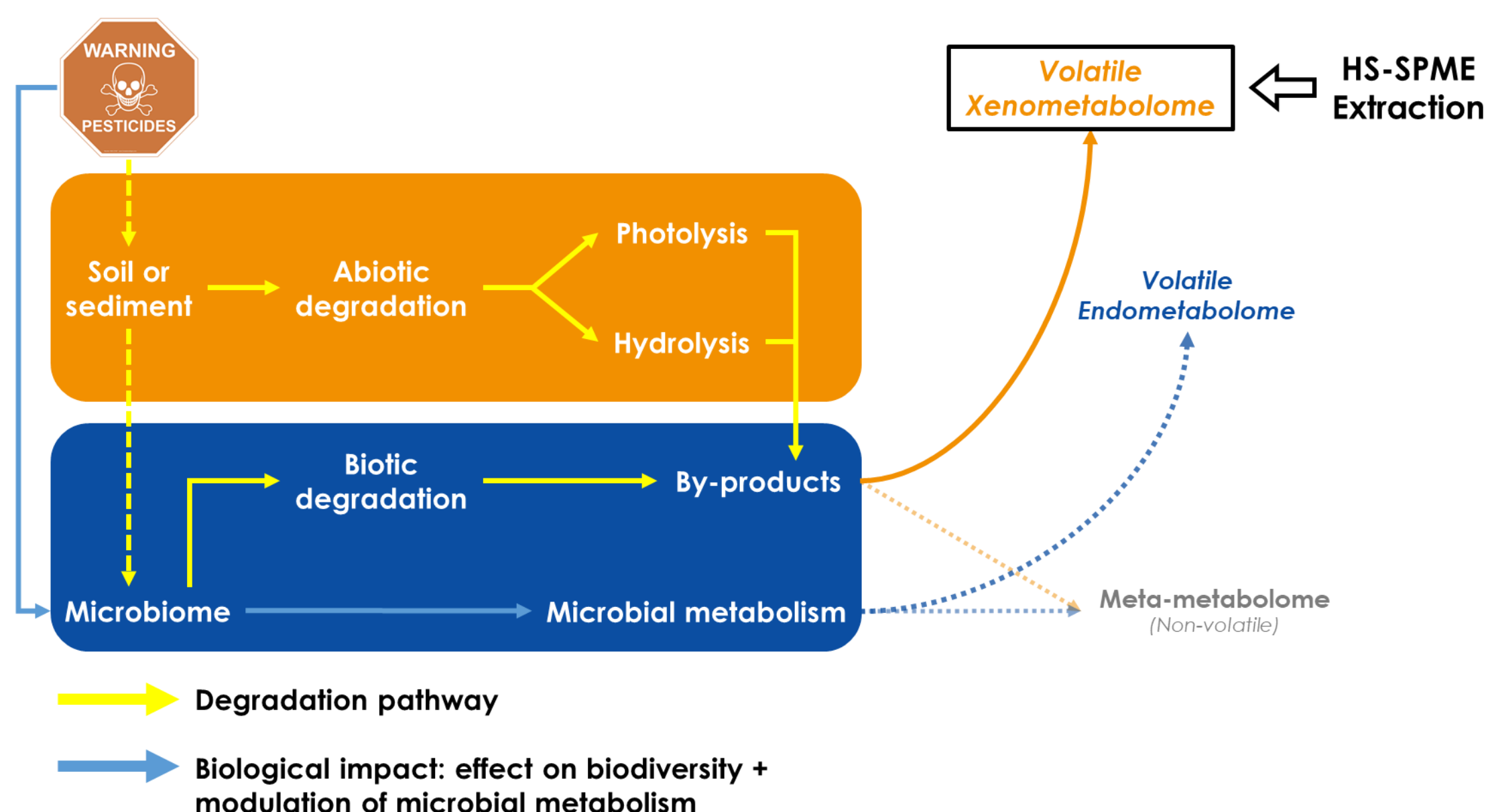
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Context

Despite their known risks, herbicides are still essential for agriculture. Thus, natural herbicides are increasingly recommended to replace synthetic ones. However, there are still limitations in studying the environmental fate of several of them. The main reason is the lack of methods dedicated for this type of complex bio-herbicides. Hence, in the framework of the Environmental Metabolic Footprinting (EMF) approach, the current work presents a method dedicated to analyze the volatile residues of herbicides applied on soil. The aim is to track the evolution of the volatile compounds issued from the herbicide by an untargeted metabolomics-based kinetics, in order to determine the "resilience time" of the gaseous phase above the soil. The approach aims to explore the environmental fate of these herbicides. In fact, it will supplement other methods based on the EMF approach recently developed. Moreover, it can estimate the exposure of farmers, insects and plants to potential toxic volatile substances. The method is based on Headspace-Solid Phase Micro Extraction-Gas Chromatography-Electronic Impact Ionization-Quadrupole Mass Spectrometry (HS-SPME-GC-EI-Q MS). The HS-SPME provides a non-destructive extraction. This allows reducing the number of samples. The GC separation technique provides high reproducible analysis for volatile compounds. In addition, it allows the calculation of the Retention Index (RI) as a tool for the molecular characterization. On the other hand, despite the low resolution of the Quadrupole mass analyzer, the Electronic Impact Ionization provides reproducible MS fragmentation patterns used for spectral library search and fast putative identification of unknown compounds.

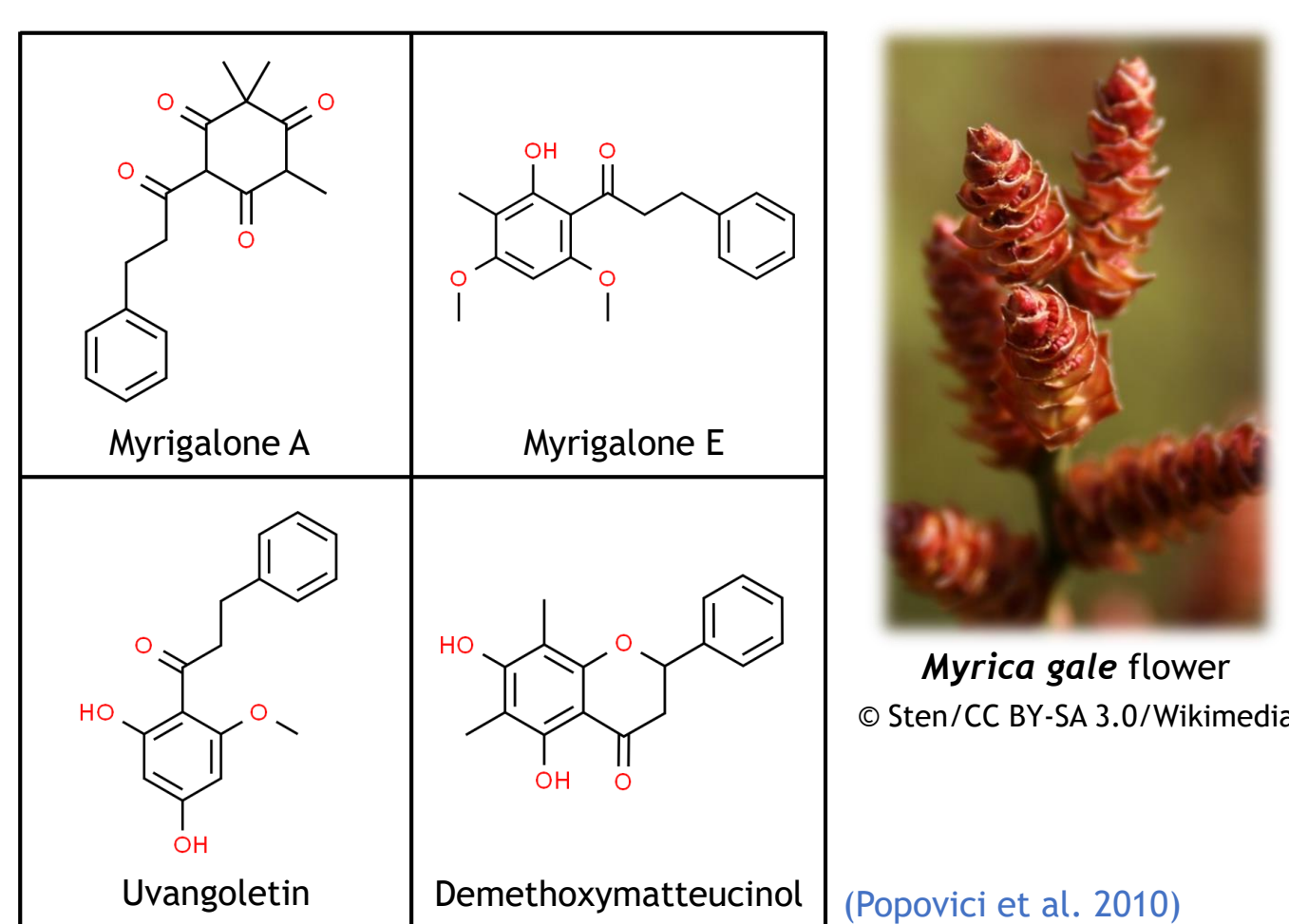
1. Introduction

1.1. Environmental-Analytical concept



1.2. *Myrica gale* extract

- ✓ **β-Triketone** natural herbicide
- ✓ **Complex mixture** of metabolites
- ✓ **Active compound: Myrigalone A**
- ✓ **Several unknown compounds**



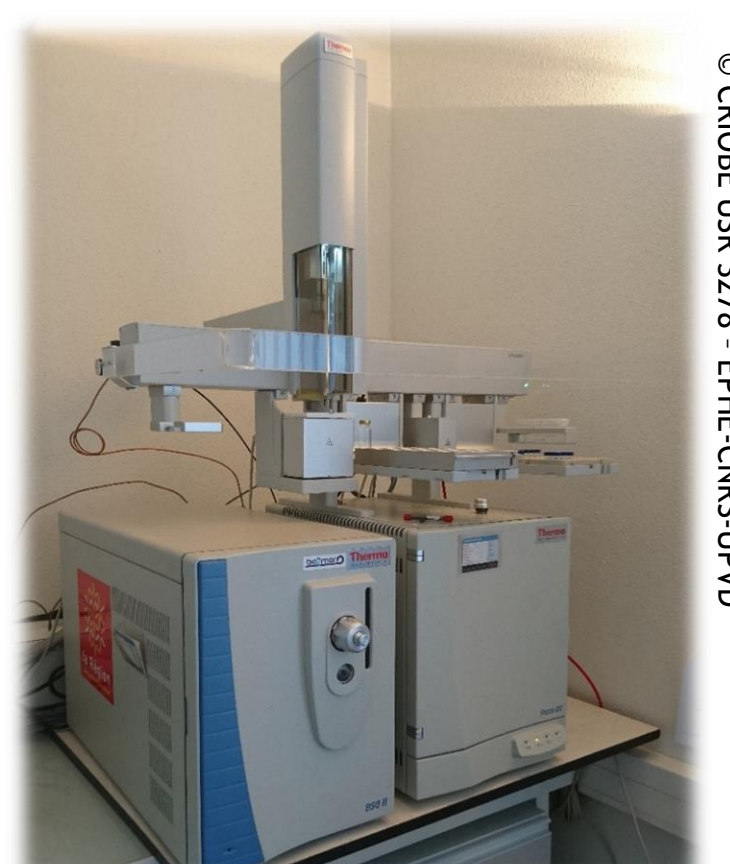
2. Material and Methods

2.1. Sample preparation

- ✓ **6 g of soil** incubated in a 20 mL crimped vial
- ✓ **Two 18Gx1.5"** needles to assure aerobic conditions
- ✓ **Control (Ctrl) vs. Pesticide-spiked (MyrN) soil:**
 - 8 kinetics points (Day 1 to 38), 3 replicates/group
- ✓ **24h day/night cycle** in climatic chamber:
 - 28 °C/18 °C, 40%/65% RH, light/dark

2.2. Analysis protocol

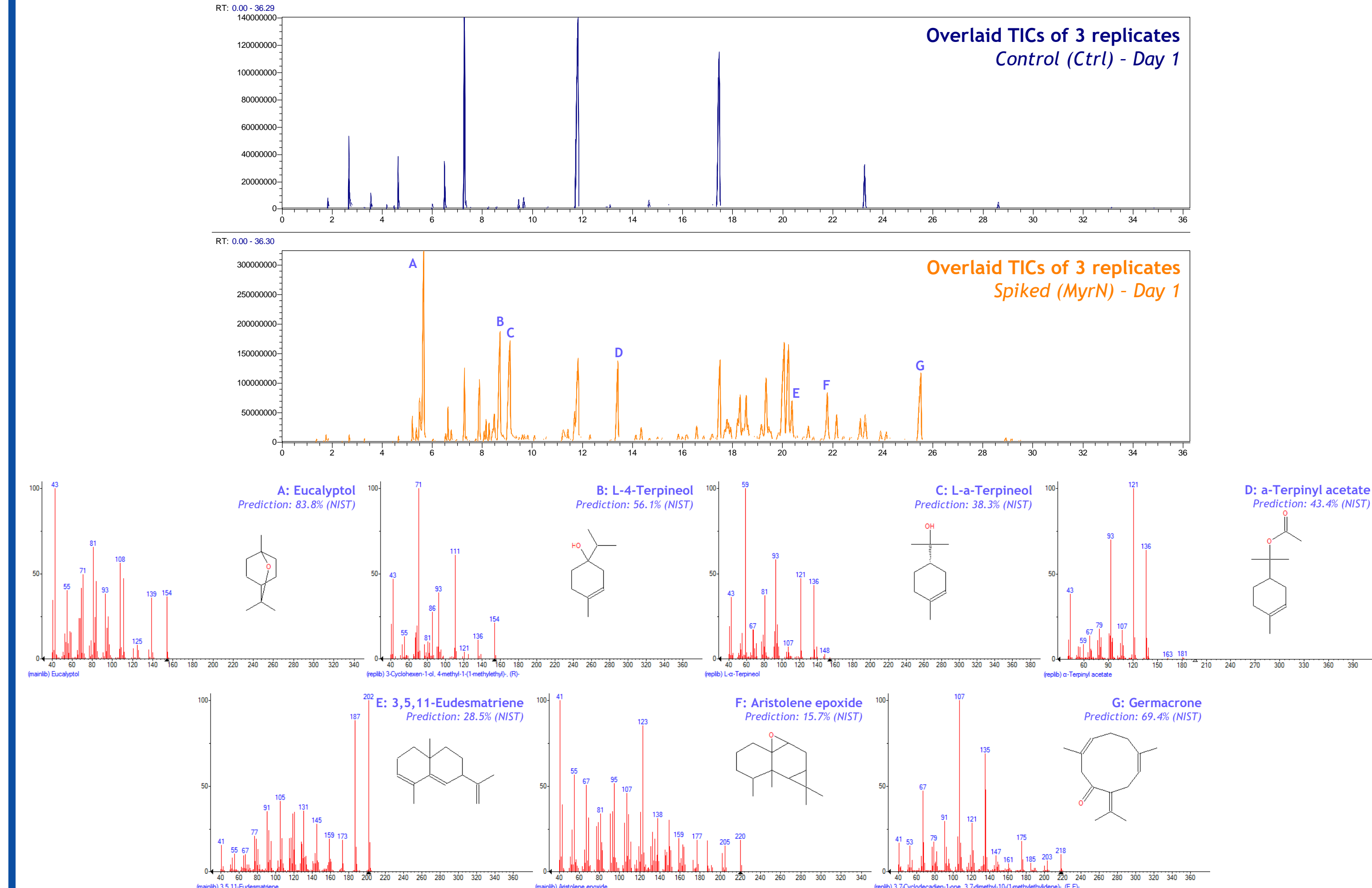
- ✓ Automated HS-SPME extraction (Thermo Triplus):
 - Fiber type: DVB/CAR/PDMS
 - Extraction time/temperature: 30 min/40 °C
- ✓ Thermo Focus GC, DB-5MS column, 35 min/run
- ✓ Thermo DSQ II EI-Quad MS: *m/z* 40-400 scan range



3. Results

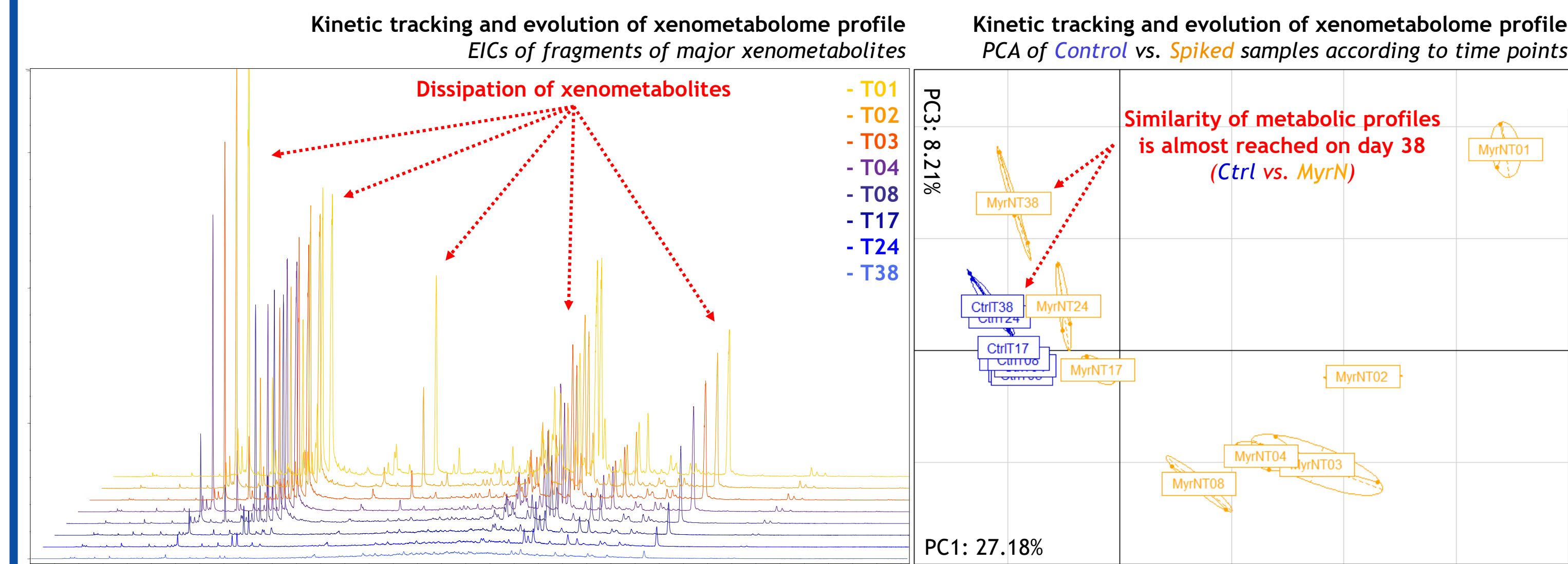
3.1. Extracted profile

- ✓ Detection of a **wide profile of volatile xenometabolome**
- ✓ **No volatile endometabolites** were detected
- ✓ **Low matrix effect** → Low interferences
- ✓ **Putative identification** of several volatile xenometabolites



3.2. Kinetics tracking

- ✓ **38 days kinetics tracking:** application on the same samples
- ✓ **Degradation of xenometabolites:** resilience was almost reached after 38 days



4. Conclusion

- ✓ **Non-destructive, simple, cheap and green** extraction method
- ✓ **Detection and characterization** of volatile xenometabolites
- ✓ **Reliable performance** for kinetics tracking on the same samples

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RGate Profile

