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Book of Abstracts

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Monitoring of Reactive Carbonyl Compounds in Food Matrices via SPME-GC-MS/MS

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Characterizing the volatile profile of thermally treated or aged food matrices is challenging due to the high reactivity of carbonyl compounds. Aldehydes are ideal chemical markers for Strecker degradation and the Maillard reaction. However, their chemical instability makes their analytical determination highly complex. Key molecules such as methional, phenylacetaldehyde, furfural, and branched aldehydes have extremely low olfactory thresholds, often in the ng/L range. Therefore, quantifying them at sensorially relevant levels requires highly sensitive methods with low limits of detection. To stabilize these analytes and prevent evaporative loss or degradation, an optimized protocol was developed. It combines in-situ derivatization using O-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine (PFBHA) with headspace solid-phase microextraction and gas chromatography-tandem mass spectrometry (HS-SPME-GC-MS/MS). Operating the triple quadrupole (QqQ) in Multiple Reaction Monitoring (MRM) mode proved crucial to enhance analytical selectivity. The optimization of collision-induced dissociation parameters allowed for the consistent isolation of diagnostic fragments common to all PFB-oxime derivatives. These include the highly intense pentafluorobenzyl cation $[C_7H_2F_5]^+$ (m/z 181) and the tetrafluorobenzyl cation $[C_7HF_4]^+$ (m/z 161) (resulting from the neutral loss of HF), which facilitate peak detection through retention time alignment. To ensure unambiguous identification, these universal transitions were coupled with analyte-specific transitions of lower intensity, related to the parent aldehyde structure. This approach effectively suppressed chemical background noise and compensated for the matrix effect in highly heterogeneous samples. The method's versatility was preliminarily tested in the baking sector to monitor aldehyde evolution under varying baking conditions. Furthermore, in oenology, this procedure enables accurate quantification of the total aldehyde pool. This accuracy is vital for assessing a wine's aromatic potential, as the free aldehyde fraction responsible for sensory perception is heavily modulated by sulfur dioxide equilibrium and the formation of non-volatile hydroxysulfonate adducts. Ultimately, this robust MS-based approach proves essential for understanding reaction mechanisms and optimizing food production chains, allowing for the precise quantification of reactive analytes that would otherwise remain elusive.

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