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Combined Analytical-Statistical Metrics/Strategy for the Quantification of Volatile Compounds in Oily Matrices

Abstract: The quantification of volatile compounds in virgin olive oil (VOO) is a complex task due to the wide range of concentrations and chemical families present, as well as interferences from the oily matrix. Obtaining reliable results in an analytical quality environment necessitates the use of appropriate methodological calibration.

This study implements statistical and analytical metrics/strategies for the quantification of volatile compounds in oily matrices. This task was performed comparing four calibration procedures for quantifying volatile compounds in VOO: external matrix-matched standard (EC), external standard with internal standard (IS), standard addition (AC), and standard addition with IS. Volatile compounds were analysed using Dynamic Headspace - Gas Chromatography (DHS-GC-FID).

For EC and EC with IS calibrations, a single calibration curve was generated, and sample signals were interpolated. In contrast, AC and AC with IS involved incrementally adding standards to each sample, creating a calibration curve per sample, and extrapolating to determine the sample concentration.

The results indicated that AC and AC with IS methods exhibited higher variability compared to EC-based approaches. The EC method proved to be the most effective for quantifying volatile compounds in virgin olive oil within the context of this study. This suggests that for routine analysis and quality control of VOO volatile profiles, external matrix-matched standard calibration offers a more reliable and less variable approach. The use of an internal standard alongside external calibration can further enhance the accuracy and precision of the quantification by correcting for potential variations in sample introduction and instrument response.

The findings of this research contribute to the ongoing efforts to standardize and improve the analytical methods used to assess the quality and authenticity of virgin olive oil, where the volatile profile plays a crucial role in defining its sensory characteristics and overall quality.