ITS Academy AgriPuglia
Agribusiness School

Analysis of Volatile Organic Compounds (VOCs) in "Provola dei Nebrodi DOP" during the ripening process

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Introduction

Objective: The goal of this study was the identification of the Volatile Organic Compound (VOC) profile of Provola dei Nebrodi cheese and assess variations in their concentrations during ripening at 8 °C and 13 °C.

DATASET

Treatment		Ripening (days)	Numbers of samples
T0 (cheese after brine)		0	3
8 °C	T1_8	60	3
	T2_8	90	3
	T3_8	150	3
	T4_8	180	3
	T5_8	240	3
13 °C	T1_13	60	3
	T2_13	90	3
	T3_13	150	3
	T4_13	180	3
	T5_13	240	3
Total Samples			33

Methods

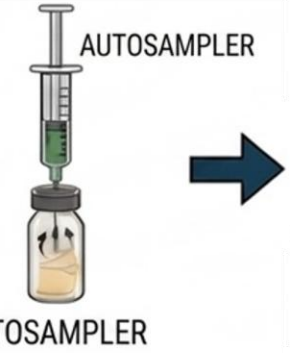
SAMPLING

Sample mass: 2g in 20 ml headspace vials

HEADSPACE

Incubation for 20min at 60 °C and 500 rpm

INJECTION

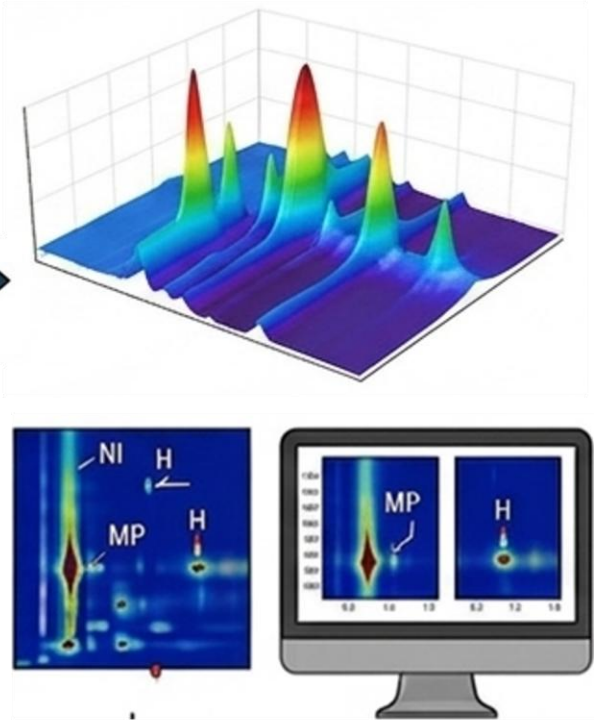


100 µl of each sample headspace was withdrawn

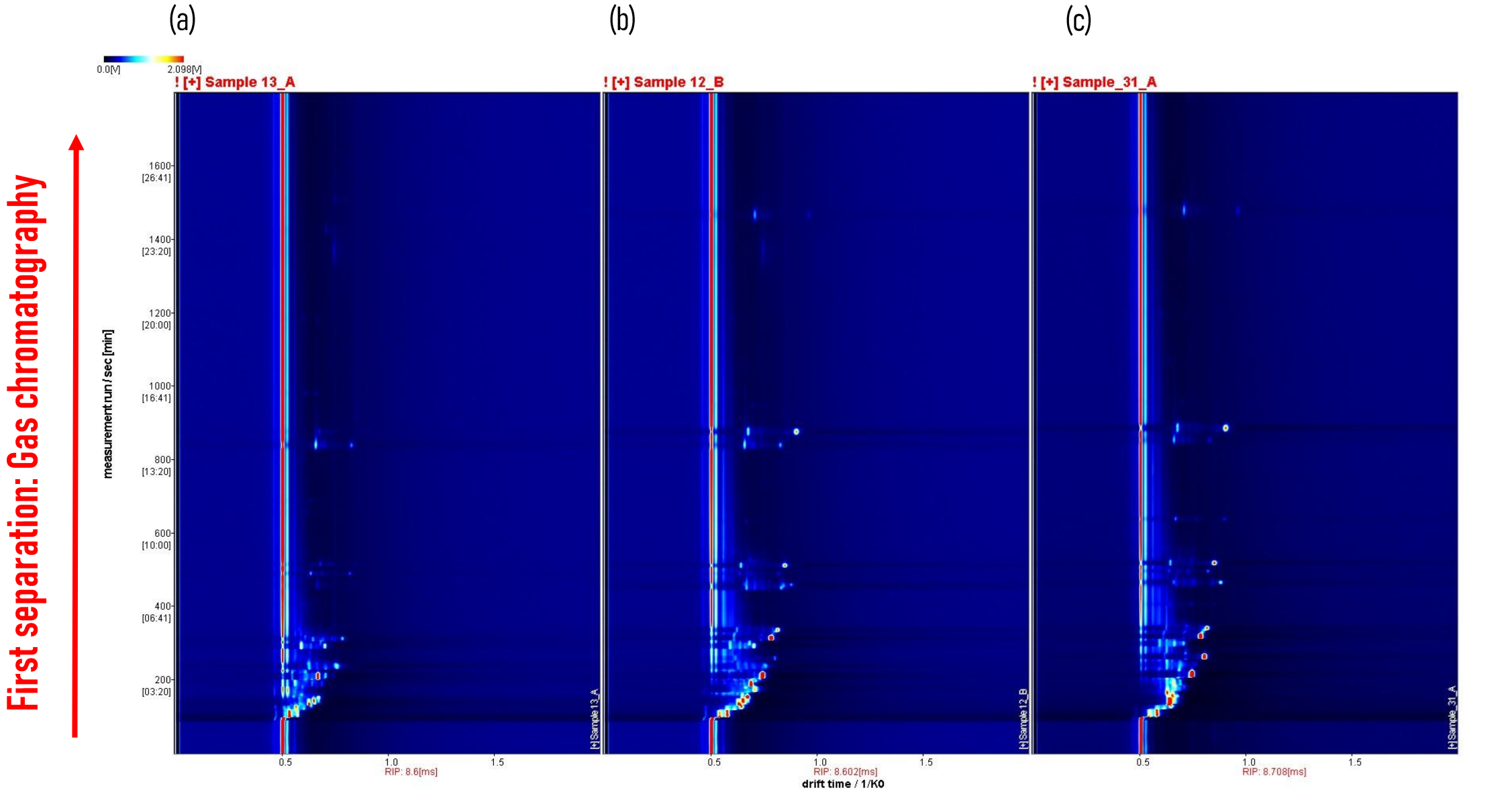
ANALYSIS

Low polar column FS-SE-54-CB-0.5, 30 m, 0.32 mm ID, film thickness 0.5 m (94% methyl-5% phenyl-1% vinylsilicone) for a first separation. IMS equipped with a tritium ionizing radioactive source at 5000 V and a 9.8 cm long drift tube for second separation.

RESULT: AROMATIC PROFILE

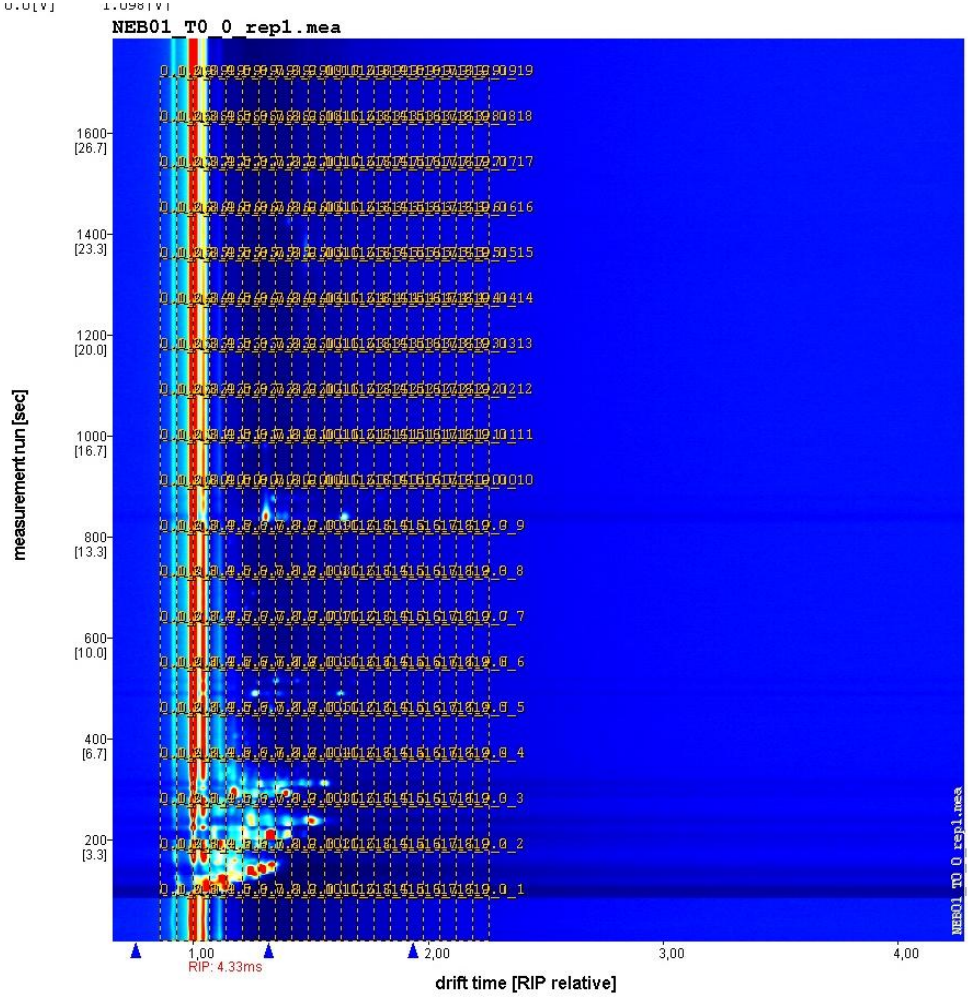


Results

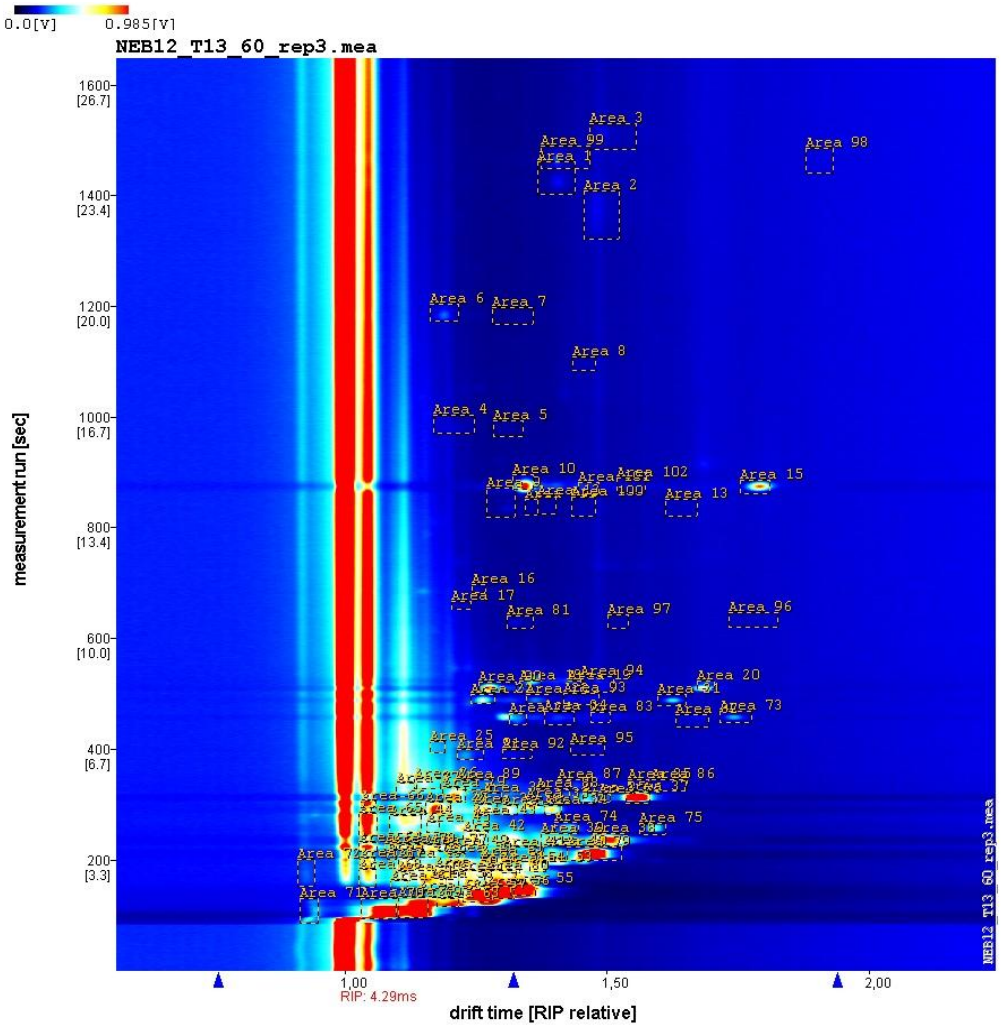


GC-IMS spectra: (a) sample T0_0; (b) sample T8_240; (c) sample T13_240

Approach No. 1: Equal Width



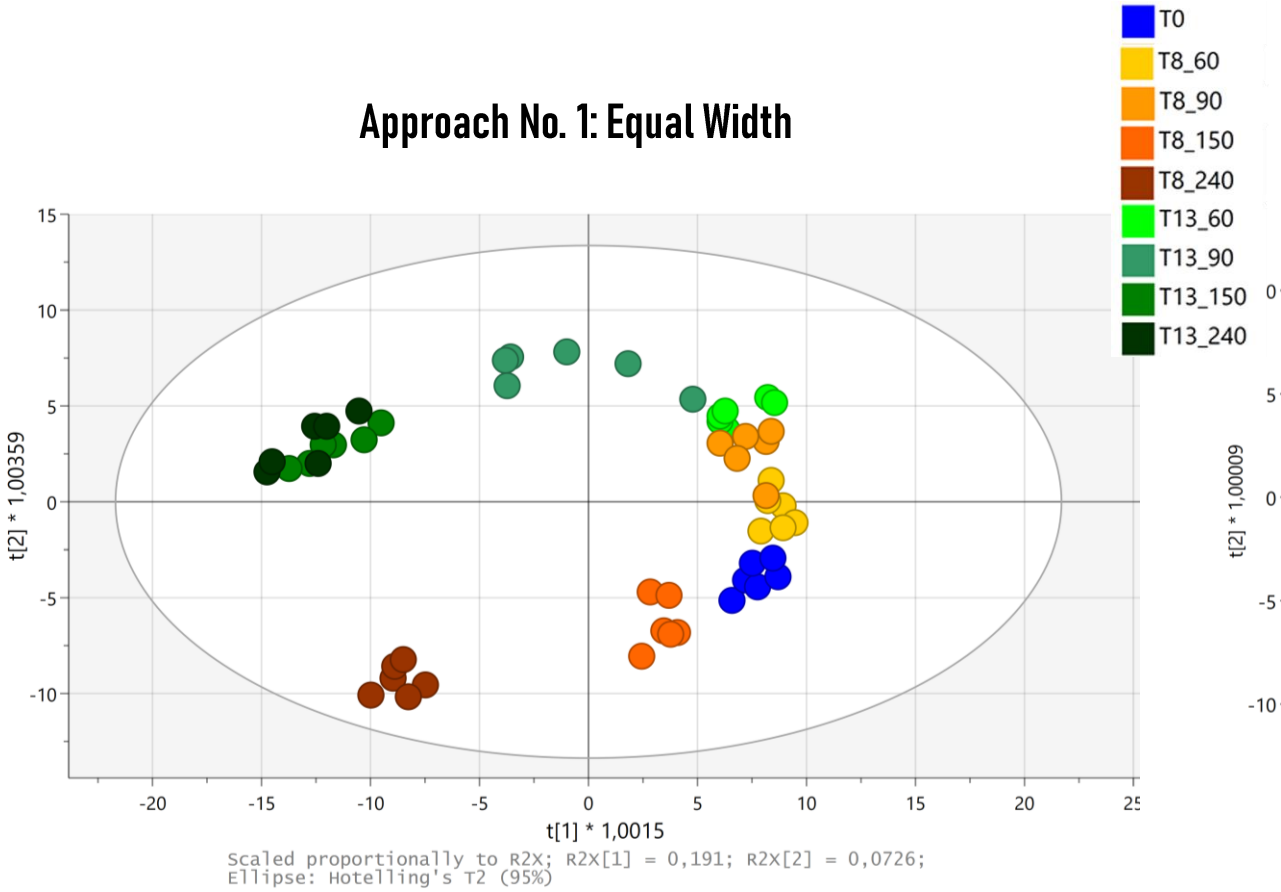
Approach No. 2: User-defined Area Selection



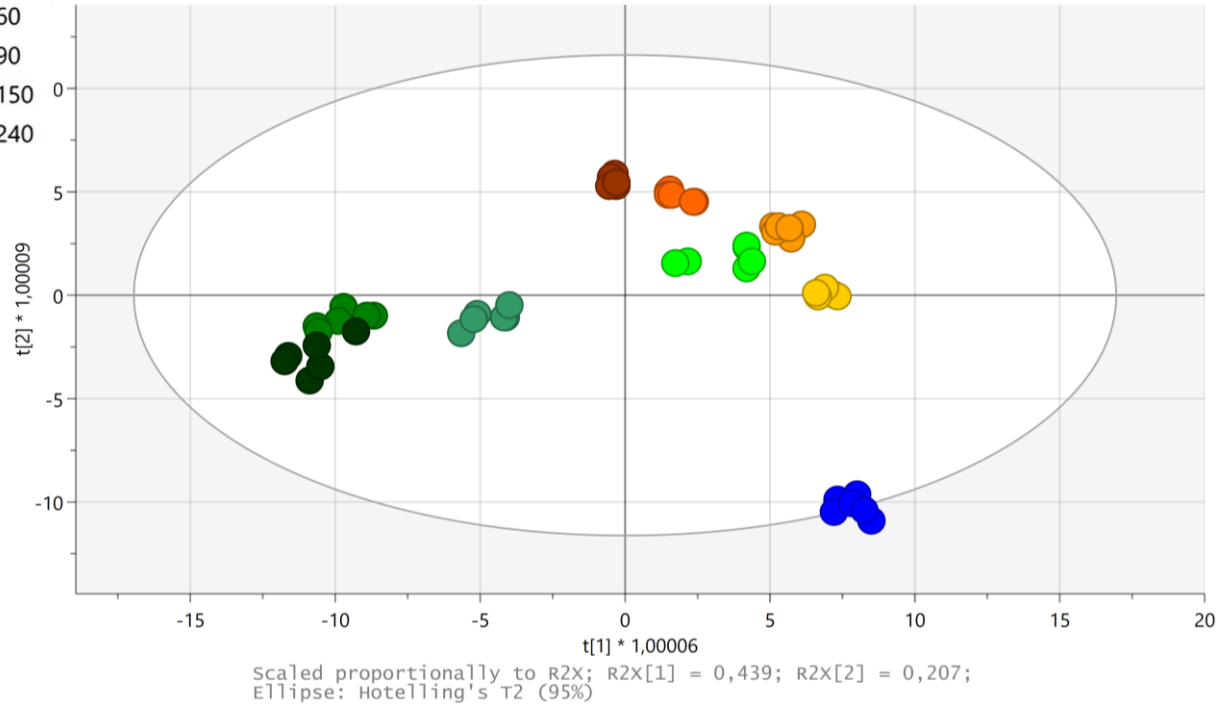
Evaluated Approaches

OPLS-DA (Orthogonal Partial Least Squares Discriminant Analysis_Comparison between Approach 1 and Approach 2

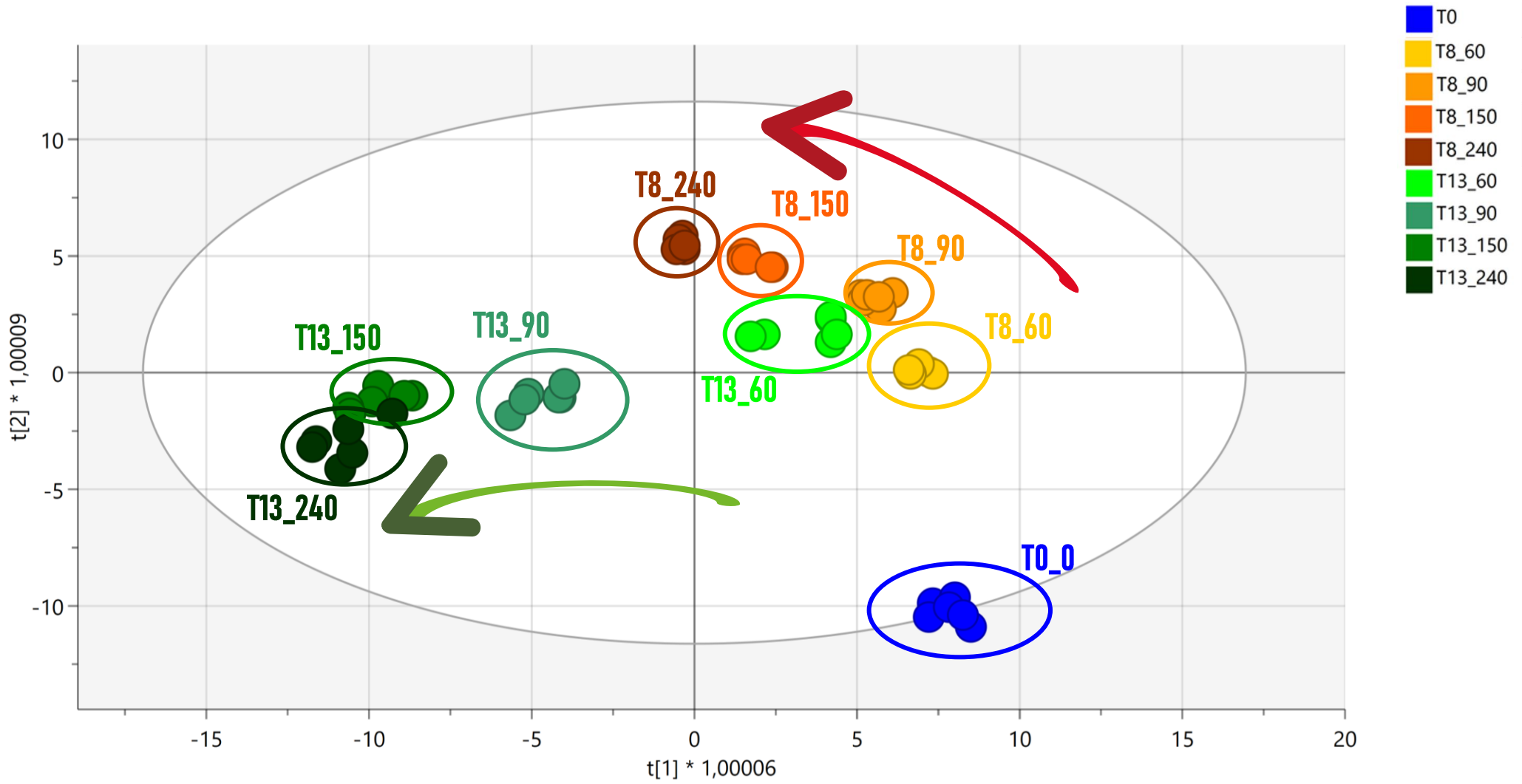
Approach No. 1: Equal Width



Approach No. 2: User-defined Area Selection

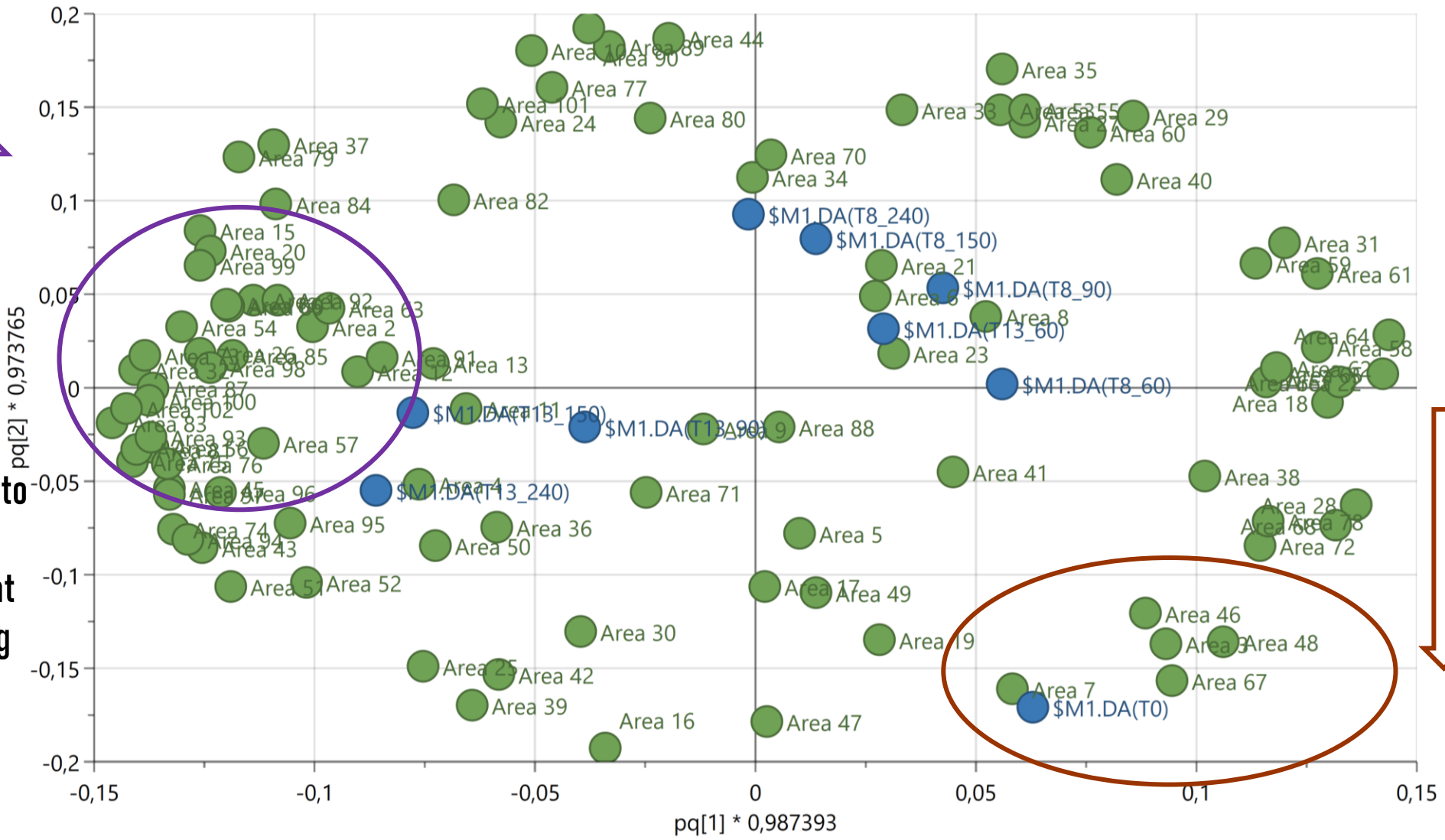


OPLS-DA (Orthogonal Partial Least Squares Discriminant Analysis)_Score plot

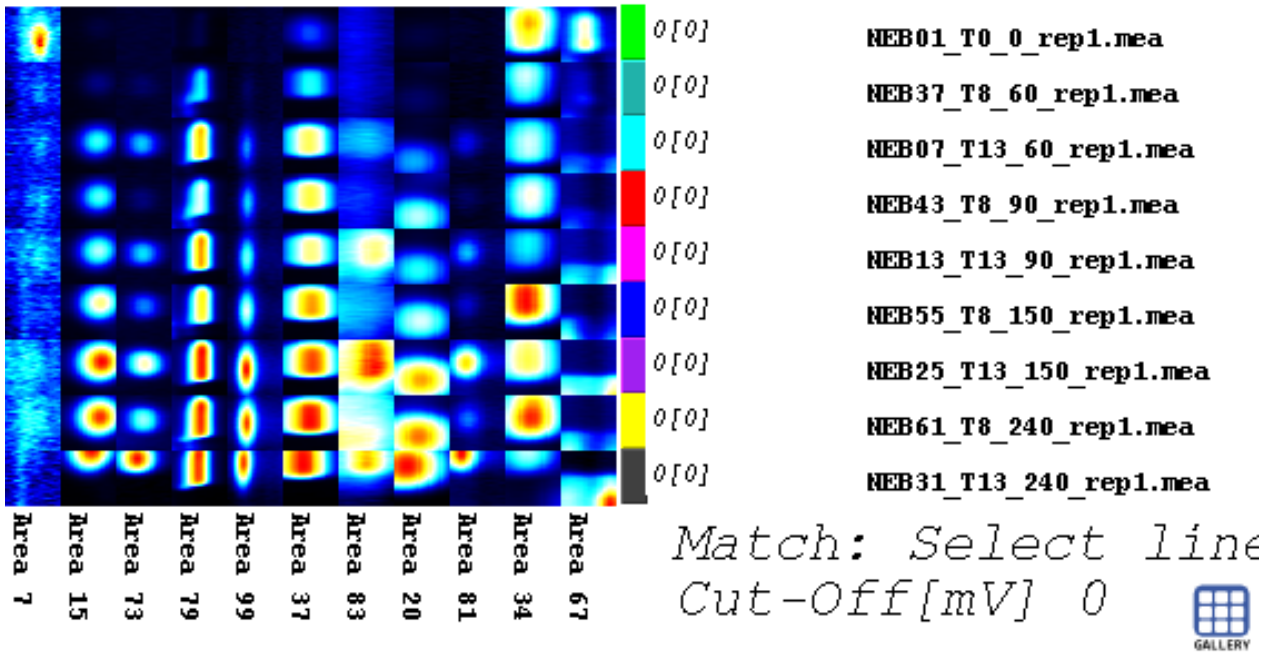


The values on the axes (R2X[1]=0.439 and R2X[2]=0.207) represent the amount of variance explained by each principal component.
The first component (p[1]) accounts for approximately 43.9% of the variance.
The second component (p[2]) accounts for approximately 20.7% of the variance.
The first two components explain a total of about 65% of the variance in the dataset. R^2X: 0.944; Q2: 0.87

OPLS-DA (Orthogonal Partial Least Squares Discriminant Analysis)_Loading plot



Qualitative Analysis: From Areas to Compound Identification



Areas	Kovats Retention Index	Potential volatile compound	Effect
Area 7	1100	2-Phenylethanol	Decreases during
Area 67	787	2-hexanone	Decreases during
Area 15	1058	2-octenal	Increases with ripening
Area 79	757	3-methyl-3-buten-1-ol	Increases with ripening
Area 99	1146	2-nonenal	Increases with ripening
Area 37	858	1-hexanol	Increases with ripening
Area 83	936	Pentanoic acid	Increases with ripening
Area 20	960	2-heptanal	Increases with ripening
Area 81	999	2-octanone	Increases with ripening
Area 34	834	Butyric acid	Increases with ripening

Identification Tools: Comparison of mass spectra with NIST Libraries.
Verification via Kovats Retention Indices (RI).

Key Findings

- **Methodological Effectiveness**: The analytical method proved to be extremely effective in monitoring and discriminating the different stages of the ripening process.
- **Treatment Impact**: A significant impact of different treatments on the volatile profile was observed, highlighting how processing conditions directly influence the chemical fingerprint.
- **Model Predictivity and Scalability**: The robust predictive performance of the OPLS-DA model makes this approach suitable for broader applications, including large-scale quality control and process optimization.
- **Qualitative Identification**: The user-driven peak area selection approach allowed for the precise isolation of key volatile markers. This enabled the qualitative identification of the characteristic compounds that define each specific maturation phase.