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Investigation and Profiling of the Aroma Components by GC-SICRIT-QTOF

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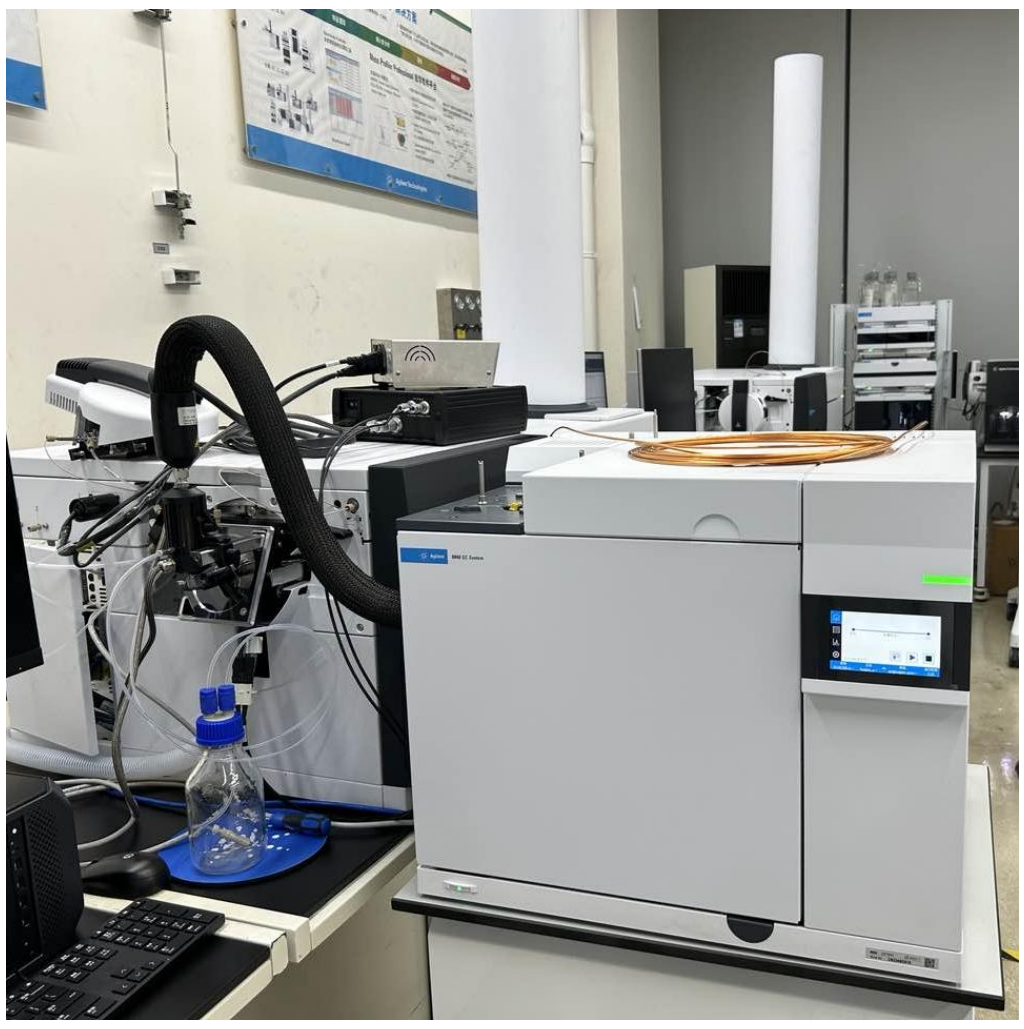
Introduction

Volatile aroma components possess significant value in flavor research. Conventional GC-MS analysis, reliant on the NIST library, is more adept at identifying known components. In contrast, LC-QTOF which exhibits strong identification capabilities, demonstrates limited ionization efficiency for volatile substances.

GC-SICRIT-QTOF, a hybrid technique, synergizes the separation capabilities of GC for volatiles, the ionization strength of SICRIT for alkenes and enols, and the powerful identification capacity of QTOF, highly suitable for the detection and profile of volatile aroma components.



Experimental

Sample preparation

A small amount of the sample is placed in a headspace vial and heated to a certain temperature as needed.

The injection method is relatively flexible: for direct injection, the sample can be placed near the ion source and directly sucked in by negative pressure. If gas chromatography separation is adopted, a gas-tight needle can be used to extract 1mL of volatile gas for testing.

MS Conditions

SICRIT ion source (Plasmion GmbH, Germany)	
Mode	Direct Screening & GC Coupling
Amplitude	1500 V
Frequency	12500 Hz
Transfer line Temp.	235 °C (For GC Coupling)
Agilent 6546 Q-TOF Mass Spectrometer	
Polarity	Positive
Gas Temp	235 °C
Fragmentor	150
Mass Range	20-800
Mode	Scan & Auto MS/MS

GC Conditions (for Sichuan pepper separation)

Agilent 8860 GC System	
Column	DB-WAX (30 m× 0.25 mm, 0.25 µm)
Injection	1 mL
Injection Mode	Splitless
Inlet Temperature	100 °C
Oven Temperature Program	50 °C for 3 min; 2 °C/min to 110 °C; 3 min hold; 4 °C/min to 200 °C; 3 min hold; 10 °C/min to 230 °C; 3 min hold;
Carrier Gas	Helium
Column Flow	1 mL/min

Results and Discussion

Target Extraction and Confirmation

Without sample preparation, volatile aroma components can be directly analyzed 6546 QTOF equipped with the SICRIT ion source, obtain MS and Auto MS/MS spectra within 1 min.

For example, in positive ion mode, we can observe $[M+H]^+$ and $[M+NH_4]^+$ ions for common organic solvents such as water, alcohol, acetonitrile and so on. If we spike ethanol vapor to nitrogen gas, less than 0.1% can be detected with 10e4~5 intensity (Figure 1).

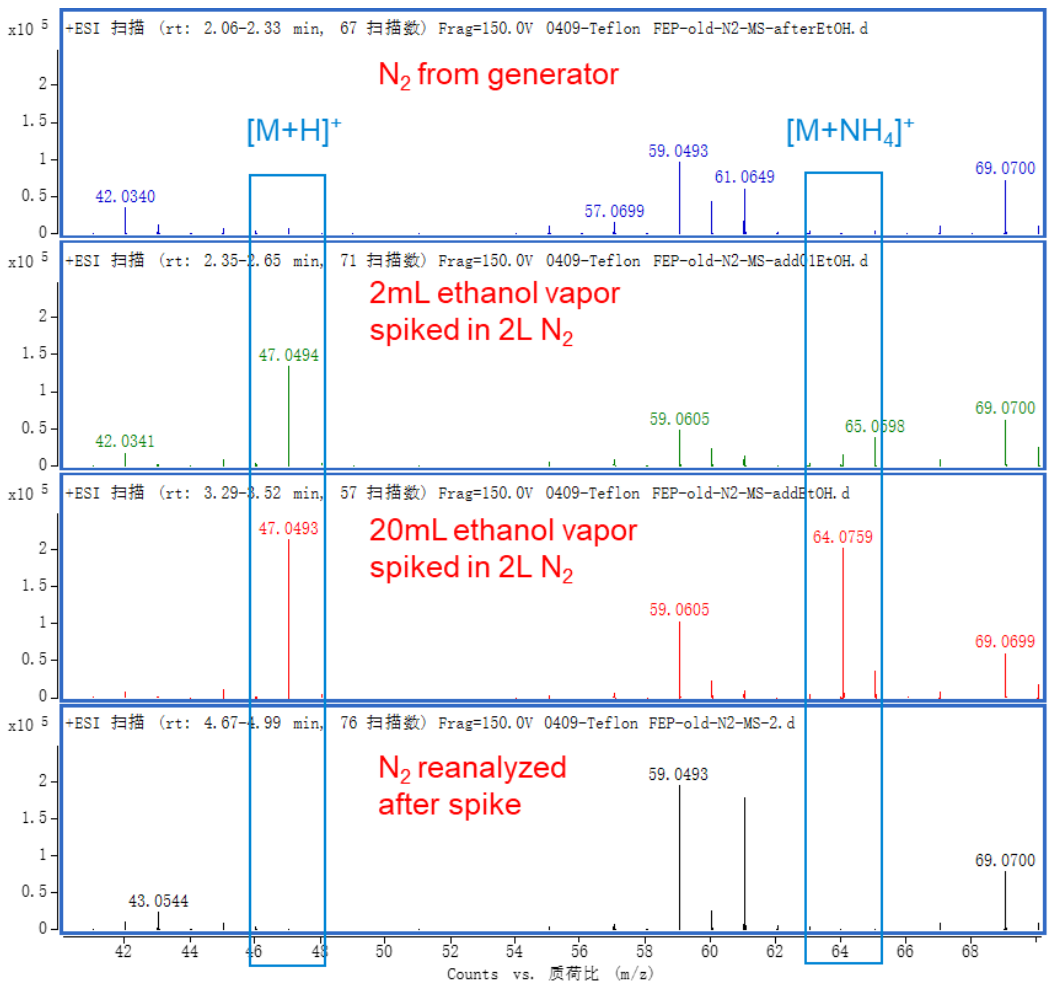


Figure 1. Ethanol Vapor Spiked MS spectra.

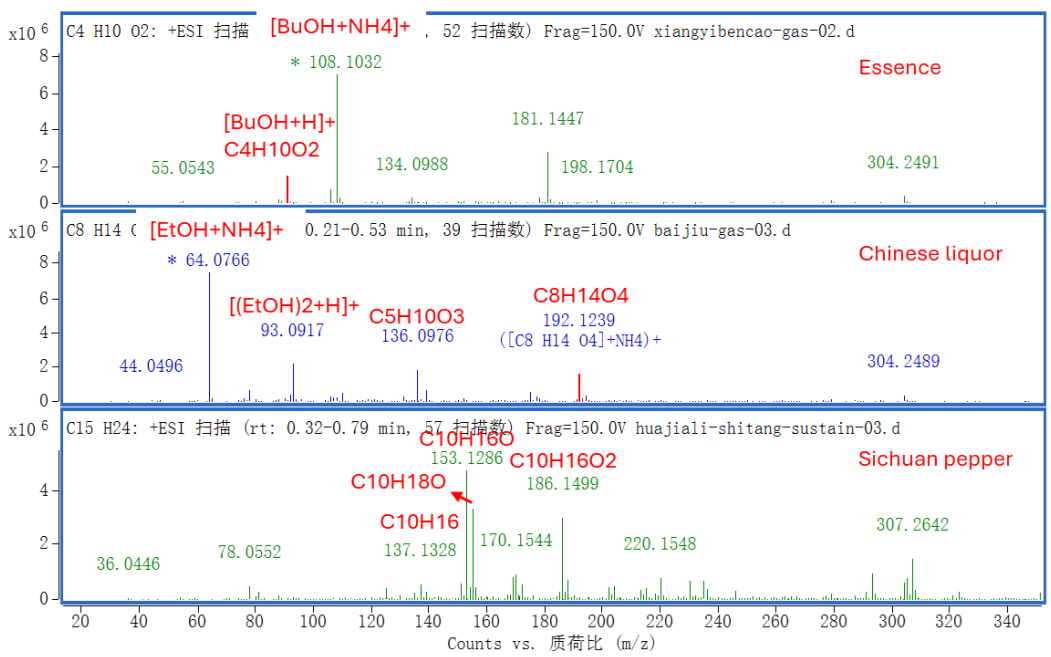


Figure 2. Vapor of Complex Mixtures.

It is also possible to find volatilizable components in complex mixtures. Figure 2 shows some examples. Butanediol is a major component in essence. Besides of alcohol, it is also possible to find esters in Chinese liquor. Alkenes and enols are main aroma components in Sichuan pepper. Their formulas are proved by accurate mass and isotope patterns.

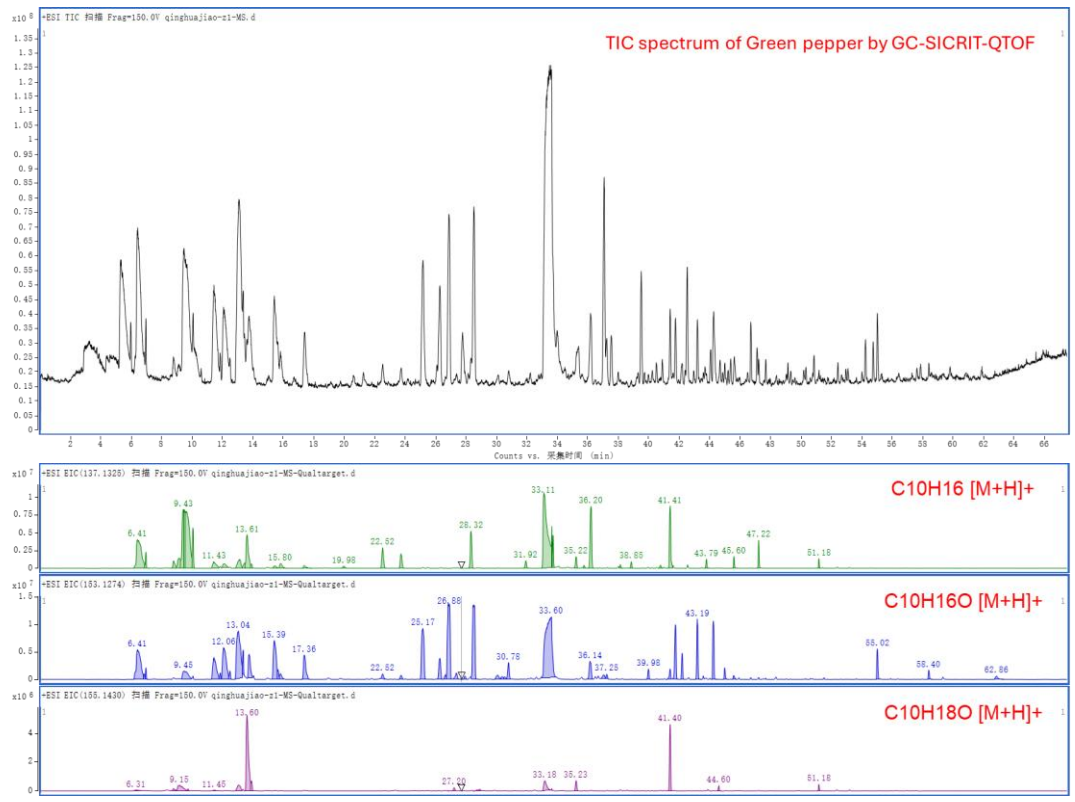


Figure 3. TIC and Major components EIC spectra of Green Pepper.

GC Coupling for isomers separation

Pepper samples contain many isomers with the same formula but different aromas. We use 8860 GC to separate these isomers, combined with SICRIT-QTOF for ionization and acquiring mass spectra.

The system demonstrates a robust response to the volatile gas components of Sichuan pepper, with a significant number of signals observed in the total ion chromatogram (TIC, Figure 3).

For the known volatile components, the GC-SICRIT-QTOF system can identify many isomers by quasi-molecular ion extraction. For example, C10H16, includes myrcene with peppery aroma, limonene with the flavor and fragrance of oranges, sabinene with woody and spicy aroma. RT, MS and MS/MS spectra can be used for identification by the support of reference and PCDL database.

Results and Discussion

Non-target extraction and discovery of new aroma component.

Non-targeted molecular feature extraction (MFE) was applied to the GC-SICRIT-QTOF data of pepper vapor. A total of 1,947 components were discovered with an intensity greater than 5,000 and MFE score exceeding 70.

Qualitative analysis can be performed using accurate mass, isotopes, and MS/MS spectra with the assistance of SIRIUS software. Figure 4 shows a new component in green pepper, methyl 2-furoate, which shows fruity aroma.

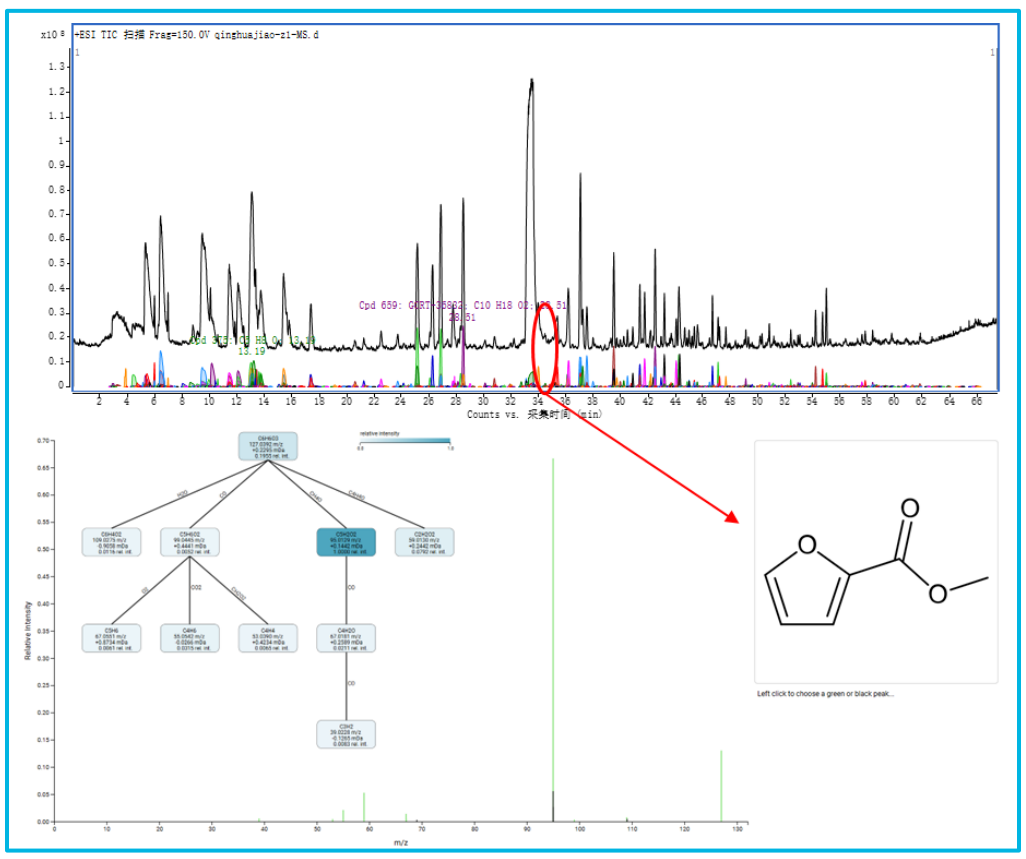


Figure 4. MFE and discovery of Methyl 2-furoate.

Comparative analysis of aroma components of green and red pepper from different origins

Several Sichuan pepper samples with different kinds and from different origins were analyzed by GC-SICRIT-QTOF system and profiled by Mass Profiler Professional (MPP) and related software.

From clustering analysis, red pepper shows obviously difference from green pepper. Principal component analysis (PCA, Figure 5) was applied to find the varying compounds in peppers. Green and red peppers show significant difference, and we can also distinguish peppers from different origins.

A list of markers is related to the key aroma components. For example, m/z 155.1430 which is identified as linalool, shows significant contribution in the distinction between red and green peppers. Consistent with the conclusion of the literature.

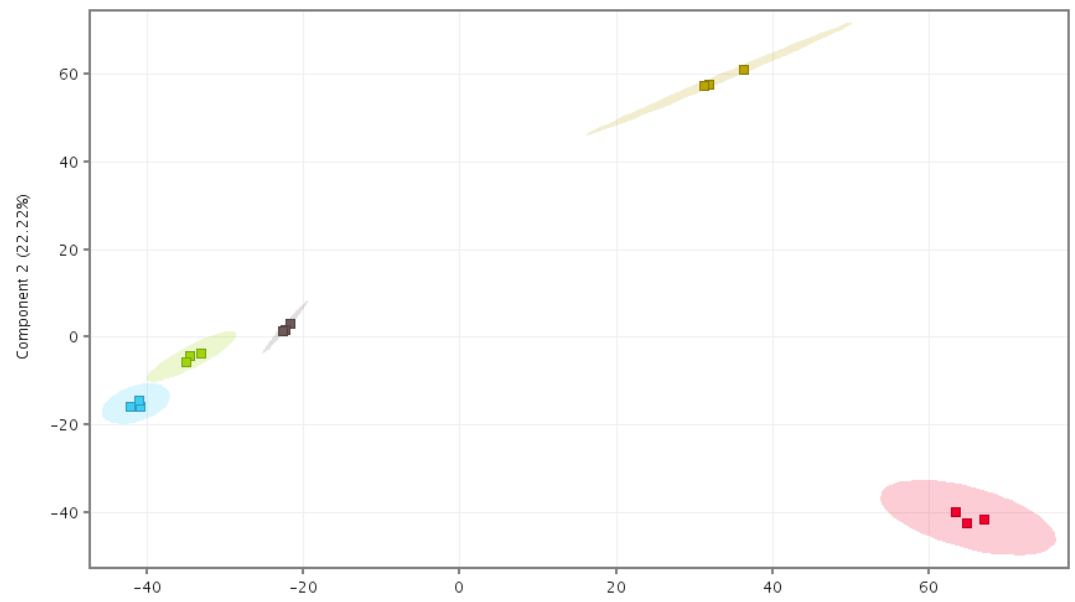


Figure 5. Principal component analysis (PCA) of different peppers from different origins.

Conclusions

- GC-SICRIT-QTOF is a powerful tool for volatile aroma component analysis. It combines the separation capabilities of GC, the ionization strength of SICRIT for low polar components, and the identification power of QTOF.
- Compare to GC-MS, this system shows quasi-molecular ions, which can be used in identification and discover new component using non-targeted extraction. Soft ionization is also suitable for fast screening and profiling without preparation.
- GC-SICRIT-QTOF facilitates the analysis of complex mixtures. It was demonstrated in the research of Sichuan pepper, including the target extraction, unknown analysis and profiling. It will contribute to the development of new food products, fragrances, and other applications in the flavor industry.

References

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