SHIMADZU

Application Data Sheet

No.39

GCMS
Gas Chromatograph Mass Spectrometer

Analysis of Aroma Components in Peach Juice by GC-MS Using the OPTIC-4 Multimode Inlet and Monolithic Adsorbent (MonoTrap)

The OPTIC-4 is a multimode injection system with a thermal desorption function that enables performing thermal desorption by placing an adsorbent in the injection port liner cup. In combination with the new MonoTrap adsorbent, the system enables analyzing aroma components with high sensitivity.

The MonoTrap is a completely new trapping tool with a proprietary monolithic porous high-purity silica structure that concentrates trace components using the adsorptive capacity provided by its large surface area. Due to a wide variety of adsorbent materials used in the MonoTrap, it can efficiently concentrate many compounds, including polar compounds, enabling ultra-high-sensitivity analysis. This was used as a monolithic material sorptive extraction (MMSE) method for GC-MS pretreatment. In this case, the MonoTrap RGC18 TD (for thermal desorption), a hybrid graphite carbon and octadecyl group type sorbent, was used to concentrate aroma components in peach juice, which were analyzed using a high-polarity capillary column.

Experiment

Collection

30 mL of commercial peach juice and MonoTrap RGC18 TD were placed in a 40 mL vial and stirred for one hour at room temperature for sampling.

Analysis

The MonoTrap material was removed from the vial, its surface lightly rinsed with water, and placed in a specialized liner, then placed in the OPTIC-4 inlet for thermal desorption.

MonoTrap RGC18 TD
Cat.No.1050-74201
(Manufactured by GL Sciences Inc.)

Specialized Liner
Cat.No.1003 -75001
(Manufactured by GL Sciences Inc.)

Table 1: Analysis Conditions

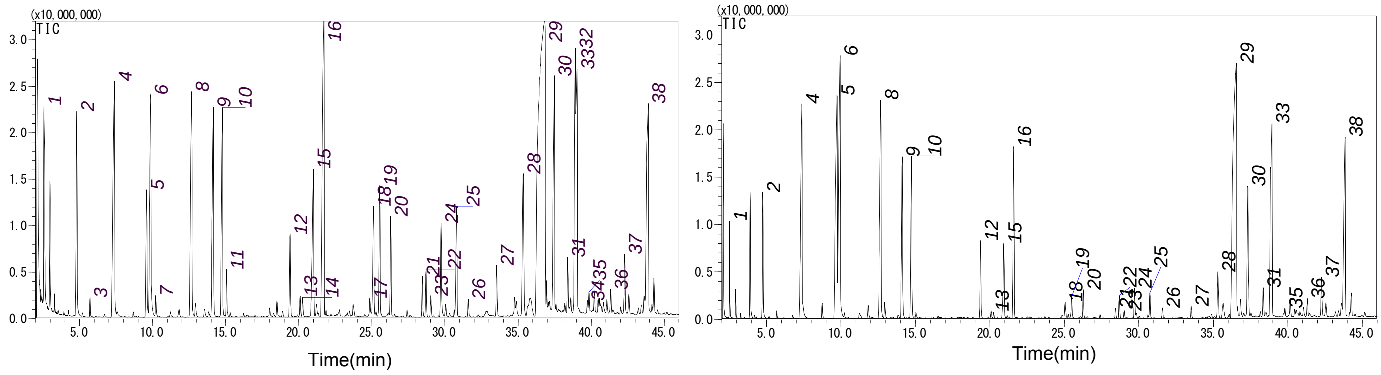
Instrument
Injection(TD) : OPTIC-4 (ATAS GL International BV, Eindhoven, the Netherlands)
GC-MS : GCMS-QP2010 Ultra (Shimadzu).
Column : InertCap Pure-WAX (0.25 mm x 30 m, df=0.25 μm (GL Sciences, Japan)

[Injector]
Thermal desorption temp. : 40 ℃ → (50 ℃/ sec) → 200 ℃
Carrier gas : Helium
Column flow rate : 1.0 mL/min
Split flow rate : 5:50
Cryofocus temperature : Trap –160 ℃
Introduction 250 ℃

[GC]
Column oven temperature :
40 ℃ (5min)→(4 ℃ /min)→250 ℃→(5min)

[MS]
Interface temperature : 250 ℃
Ion source temperature : 200 ℃
Solvent elution time : 0.5 min
Data sampling time : 1.0 – 60 min
Measurement mode : TIC
Mass range : m/z 30-600
Detector voltage : +0.87 kV (absolute value)

Results and Discussion



MMSE Method

SBSE Method (conventional method))

Fig. 1: Comparison of Chromatograms Obtained Using the MMSE (with MonoTrap) and Conventional (SBSE) Methods

1. Ethyl Acetate
2. Ethyl butanoate
3. Butyl acetate
4. Isoamyl acetate
5. D-Limonene
6. Isobutyl isovalerate
7. 2-Hexenal
8. Hexyl acetate
9. 3-Hexenyl Acetate
10. 2-Hexenyl Acetate
11. 2-Isopropyl-4-methylthiazole
12. Octyl acetate
13. Benzaldehyde
• 2-Methyl-4-propyl-1,3-oxathiane
• p-Menthan-2-one
16. beta-Linalool
17. 2-Methylbutanoic acid
18. gamma-Caprolactone
19. Terpineol
20. Benzyl acetate
21. cis-Geraniol
22. beta-Damascenone
• 5-Methyl-2-(1-methyl-1-sulfanylethyl)cyclohexanone
• trans-Geraniol
25. gamma-Butylbutyrolactone
26. beta-Ionone beta-Ionone
27. gamma-n-Amylbutyrolactone
28. Triacetin
29. delta-Undecalactone
30. delta-Decalactone
31. Eudesm-7(11)-en-4-ol
32. Ethyl caproate
33. delta-Undecalactone
34. n-Decanoic acid
35. delta-Hexylvalerolactone
36. gamma-Dodecalactone
37. delta-Dodecalactone
38. Nootkatone

In Fig. 2, the peak area values obtained for each component by the SBSE method are indicated as "1" to show how MMSE sensitivity compares to SBSE. Because the MonoTrap RGC18 TD material is a hybrid of graphite carbon and octadecyl groups, it is more effective in collecting polar compounds than the SGSE method, which uses agitator paddles coated with PDMS. Therefore, the MonoTrap material enabled highly sensitive analysis.
It is especially useful for sulfur compounds and lactones, such as gamma-caprolactone (18) and 2-Isopropyl-4-methylthiazole (11). Lactones are the components in peaches, other fruits, dairy products, etc., that give them a sweet smell. 2-Isopropyl-4-methylthiazole is used as a fruit and vegetable flavoring.

The monolith manufacturing technology with sol-gel method is new technology developed in Japan by Dr. Soga and Dr. Nakanishi at Kyoto University.
H. Minakuchi, K. Nakanishi, N. Soga, N. Ishizuka, N.Tanaka, Anal. Chem. 1996, 68, 3498-3501.
Nakanishi, K., Pore structure control of silica gels based on phase separation. *J. Porous Materials*, 4 (1997) 67

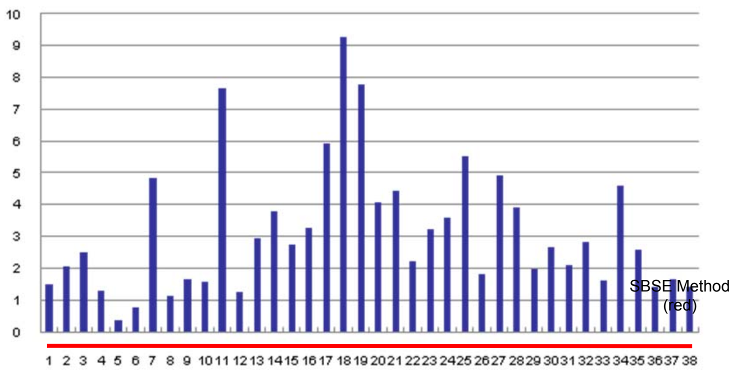


Fig. 2: Sensitivity Comparison Between MMSE and Conventional (SBSE) Methods

Summary

The MMSE method using the MonoTrap monolithic adsorbent enabled the use of a simple procedure to highly concentrate trace aroma components. The high sensitivity is not only due to the high concentration levels, but also due to the improved adsorption efficiency of the graphite carbon contained in the MonoTrap's base silica structure and to the use of a high-polarity deactivated column. In addition, the improved sensitivity for polar compounds, which had previously been an issue when analyzing aroma components, was presumably due to the efficient thermal desorption provided by the OPTIC-4 multimode inlet. The combination of concentration by the MMSE method and the OPTIC-4 multimode inlet provides an excellent alternative to SBSE and other typical concentrating methods for detecting or screening low-concentration compounds in gaseous form.

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