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Application News

GC-MS HS-20 NX Trap/GCMS-QP 2020 NX

# Aroma Component Analysis Using Trapped Headspace (THS) Sampling

A. Aono

## User Benefits

- An electrically-cooled focusing trap enables higher analyte concentrations and analysis of a wider range of low-boiling-point and high-boiling-point components in a single analysis and shorter time.
- More than 20 times more sensitive than static headspace analysis.
- User can switch between "Trap Mode" and "Loop Mode" according to the concentration of the sample.

## ■ Introduction

Multipurpose autosamplers are often used to concentrate samples for aroma component analysis, but these autosamplers have issues such as high procurement cost and complex operation and maintenance.

The HS-20 NX Trap is a trap headspace sampler with an electrically-cooled trap built into a headspace sampler used for water quality testing and other applications, and is cheaper and simpler to operate than multipurpose autosamplers.

Running the HS-20 NX Trap in "Loop Mode" uses static headspace (SHS) sampling to extract 1 mL of headspace, while running in "Trap Mode" uses trapped headspace (THS) sampling to extract approx. 90 % of vial headspace (approx. 18 mL of a 20 mL vial with a solid sample). This increased extraction allows for easy detection and qualitative analysis of not just major components, but components in small quantities that also affect the overall aroma. The electrically-cooled trap can be cooled to temperatures below ambient to prevent reduced yields of low-boiling-point components.

This article compares using SHS sampling and THS sampling to analyze aroma components in food and spice.

## ■ Equipment Setup and Analysis Conditions

Table 1 shows the conditions that were used to analyze aroma components on a GCMS-QP2020 NX gas chromatograph mass spectrometer and the HS-20 NX Trap headspace sampler.

Table 1 Analysis Conditions

| | |
|---|---|
| **GC-MS Conditions** | |
| Model | : GCMS-QP2020 NX |
| Column | : SH-I-624Sil MS Cap.<br>(0.32 mm I.D. × 30 m, d.f. = 1.8 µm) |
| Column Temp. | : 40 °C (2 min) – 10 °C/min – 200 °C (6 min)<br>Total 24 min |
| Injection Mode | : Split 1:20 |
| Carrier Gas Controller | : Fixed linear velocity mode (He) |
| Linear Velocity | : 43.2 cm/sec |
| Interface Temp. | : 250 °C |
| Ion Source Temp. | : 200 °C |
| SCAN | : m/z 35-400 |
| **HS Sampling Conditions** | |
| Oven Temperature | : 70 °C |
| Equilibration Time | : 45 min |
| Sample Line Temp. | : 150 °C |
| Transfer Line Temp. | : 150 °C |
| Vial Stirring | : Off |
| Vial Volume | : 20 mL |
| Vial Pressurization Time | : 0.5 min |
| Vial Press. Equilib. Time | : 0.1 min |
| Loading Time | : 0.5 min |
| Load Equilib. Time | : 0 min |
| Multi Injection | : 3 |
| Vial Pressure | : 100.0 kPa (He) |
| Trap Cooling Temp. | : -10 °C |
| Trap Heating Temp. | : 250 °C |
| Trap Adsorbent | : Tenax TA 60/80 mesh 37 mg |
| Injection Time | : 5.0 min |
| Needle Flush Time | : 5.0 min |

## ■ Trapped Headspace (THS) Sampling

Fig. 1 shows the principle behind THS sampling. The sample vial is heated for a certain period, then a needle is inserted to pressurize the sample vial. For solid samples, the vial pressure is 10 kPa when held at 80 °C and 22 kPa when held at 100 °C, hence at least double these pressures are used for pressurization and the vial is held pressurized for 1 minute (pressurizing time). The headspace is then allowed to enter the trap (load time) and approx. 50 % of the molecules in the headspace are concentrated in the trap. This cycle of pressurization and trap loading is repeated in a procedure called "Multi Injection." Performing two loading cycles will concentrate approx. 75 % of headspace molecules within the trap, and performing three loading cycles will concentrate approx. 87.5 % of headspace molecules within the trap.

The trap can also be electrically-cooled, which has the advantage of enabling high breakthrough volumes for even low-boiling-point components (Table 2). Although performing three loading cycles requires breakthrough volumes over 0.060 L, by cooling the trap to -10 °C low-boiling-point components like ethanol and acetaldehyde can also be concentrated on a single adsorbent trapping material.

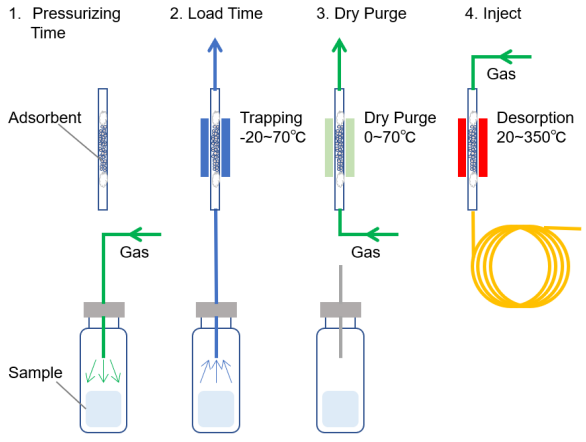


Fig. 1 Principle behind THS Sampling

Table 2 Trap Temperature and Breakthrough Volumes (L) with Tenax TA Adsorbent

| Compounds | -20 °C | -10 °C | 0 °C | 10 °C | 20 °C |
|---|---|---|---|---|---|
| Chloromethane | 0.107 | 0.069 | 0.031 | 0.020 | 0.009 |
| Methanol | 0.173 | 0.110 | 0.048 | 0.031 | 0.013 |
| Vinyl chloride | 0.194 | 0.129 | 0.063 | 0.042 | 0.020 |
| Acetone | 4.835 | 2.935 | 1.036 | 0.629 | 0.222 |
| Pentane | 4.662 | 2.795 | 0.929 | 0.557 | 0.185 |
| Water | 0.010 | 0.007 | 0.005 | 0.004 | 0.002 |
| Ethanol | 1.283 | 0.788 | 0.292 | 0.179 | 0.067 |
| Acetaldehyde | 0.276 | 0.178 | 0.081 | 0.053 | 0.024 |

## ■ Example Aroma Component Analyses

Fig. 2 shows the results obtained from analyzing aroma components generated by almonds. Using THS sampling, even low-boiling-point components like acetaldehyde and pentane were detected with high yield. Components with a major impact on the overall aroma but present in small quantities, such as dimethyl disulfide and acetal, were not detected with SHS sampling but were detected with THS sampling.

As shown in Fig. 3, when cumin seeds were analyzed with THS sampling, many aroma components present in small quantities were also detected alongside major components such as cuminaldehyde.

These examples demonstrate that THS sampling with an electrically-cooled trap can be used to analyze low-boiling-point components such as ethanol and isobutyl aldehyde alongside major aroma components in a single analysis and with a single adsorbent trapping material.

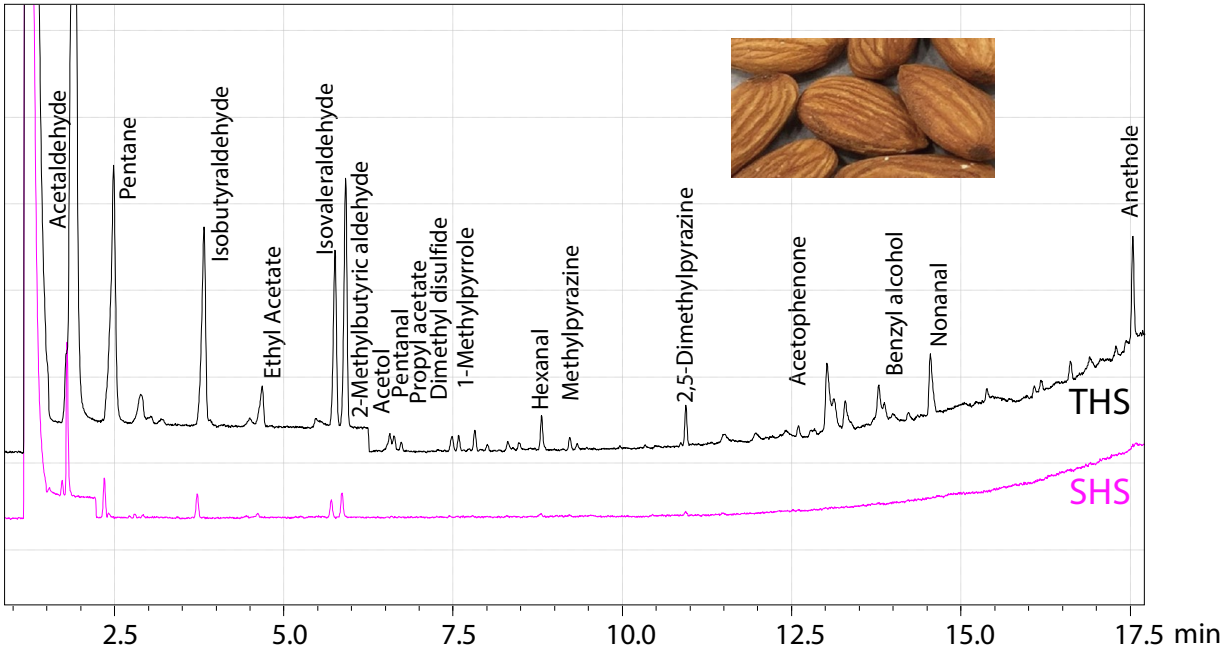


Fig. 2 Aroma Components of Almonds

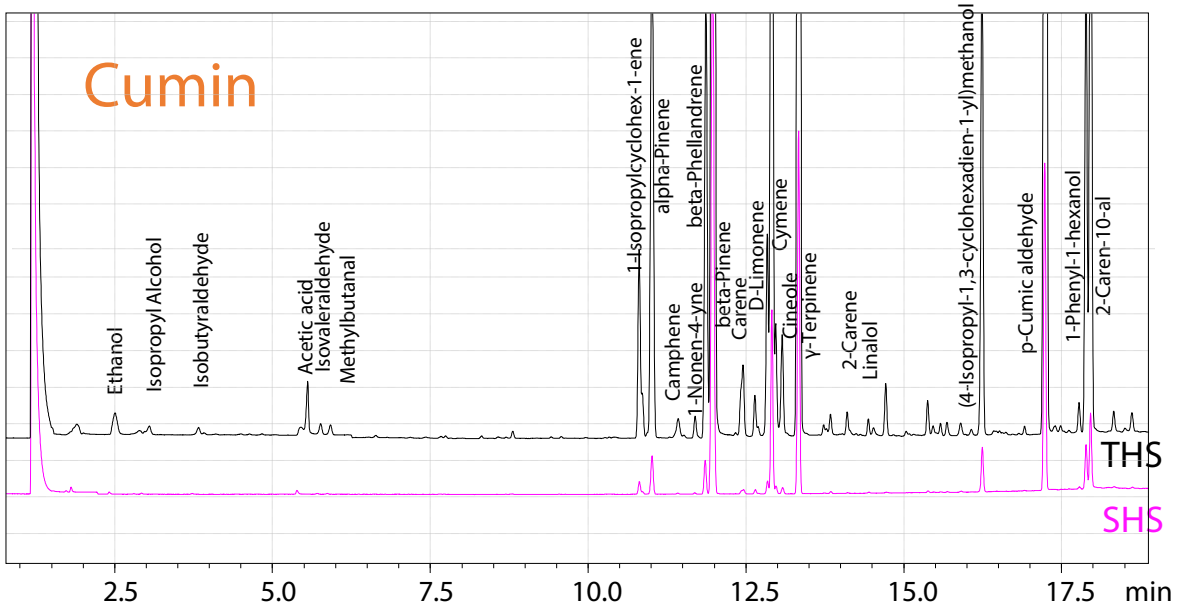


Fig. 3 Aroma Components of Cumin Seeds

## ■ Conclusion

THS sampling is used in fields of environmental science to analyze volatile organic compounds in water and compounds that cause musty odors. This article shows that THS sampling can also be used for highly sensitive analysis of aromas produced by food. Compared to other systems used to concentrate samples for aroma analysis, the HS-20 NX Trap is low-cost and enables faster sample extraction and analysis. The HS-20 NX Trap can also be switched between "Trap Mode" and "Loop Mode" to accommodate high-concentration sample analysis and low-concentration sample analysis on a single system.

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Shimadzu Corporation
Analytical & Measuring Instruments Division
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