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MP 352

Highly sensitive and efficient analysis of aroma components using trapped Headspace GC-MS

Ana Caroline Martimiano[1], Isabela de Oliveira e Silva[1], Rodrigo Ossamu Saga Kitamura[1].
(1) Shimadzu do Brasil Comércio Ltda, Barueri, Brazil.

1. Introduction

The aroma composition of foods has been the subject of considerable research in recent years. It is well known that the presence of volatile compounds and their composition determine the specific aroma of foods and the flavor of the resulting products. Static headspace technique enables high sensitivity analysis of volatile compounds, normally extracting 1 mL of headspace. But in situations where it is necessary to evaluate the total chemical profile, it is known that components in small quantities also affect the overall aroma. Thus, the HS-20 NX was used running in "Trap mode", where by using trapped headspace sampling, it is possible to extract much more volume and analyze, not only the majority components, but all the compounds with high sensitivity.

The system used to achieve the highest sensitivity is the HS-20 NX coupled to the GCMS-QP2020 NX single quadrupole mass spectrometer (**Fig. 1**).

Fig. 1 GCMS-QP2020 NX + HS-20 NX.

2. Methods

❖ The HS-20 NX allows sampling in either loop or trap mode. In the case of trap mode, an electronic cooling system is integrated, enabling compound concentration and high-sensitivity analysis (**Fig. 2**). The gas in the headspace is directed to a tube with trap material, which is cooled to concentrate the components present in the sample. Subsequently, through the thermal desorption process, heating of the trap, the concentrated compounds are released and introduced into the GC.

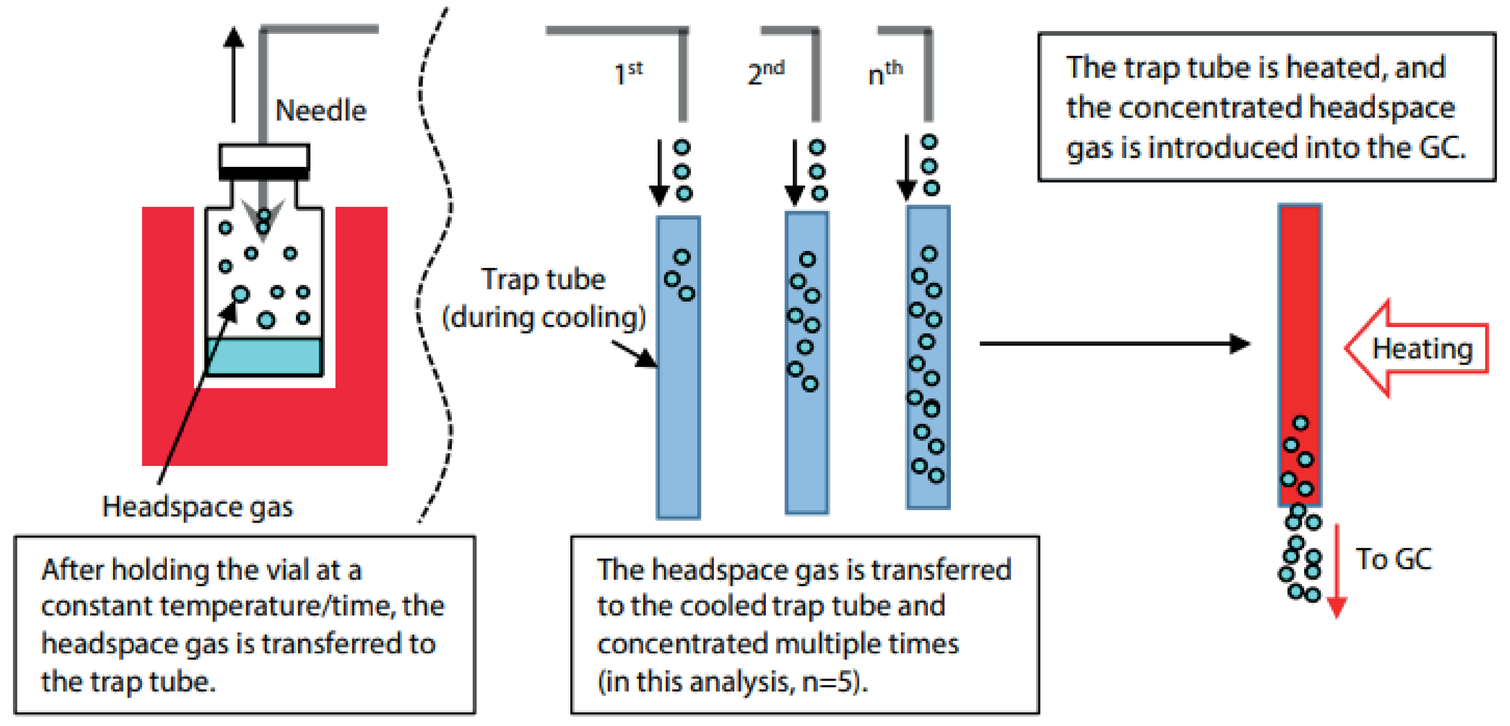

Fig. 2 Sample Concentration in Trap Mode.

2.1 Analytical Method

The analyses were performed in both loop mode and trap mode using 3 and 5 times the sample concentration, with the total chromatographic runtime set at 50.5 minutes. Separation of substances was SH-5MS capillary column 30m x 0.25mm x 0.25µm column. Out of this total runtime, 30 minutes were used for sample incubation on the HS-20 NX, while the remaining 20.5 minutes were dedicated to acquisition on the GC/MS.

2.2 Sample Preparation

To prepare the samples (aroma sample in food), it was only necessary to weigh the sample with 20 mg in a 20 mL vial. 3 vials with 20 mg each were prepared for data acquisition in loop mode, trap mode with 3- and 5-times sample concentration through an electronic cooling trap.

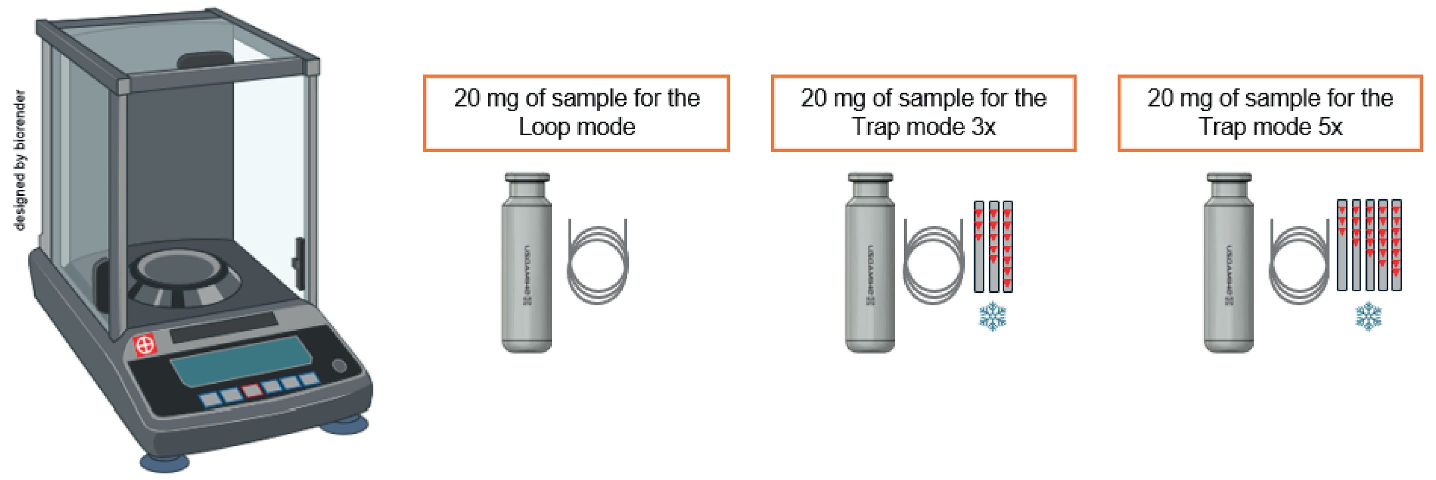

Fig. 3 Sample Preparation for HS-20 NX.

3. Results and Discussion

After the incubation time, the acquisitions were done by loop mode and trap mode with 3- and 5-times sample concentration. In this way, it is possible to analyze, via trap, the presence of compounds at trace levels, which cannot be observed when analyzed in loop mode. 18 compounds could be identified in the sample when analyzed in loop mode, whereas in trap mode, 35 were identified.

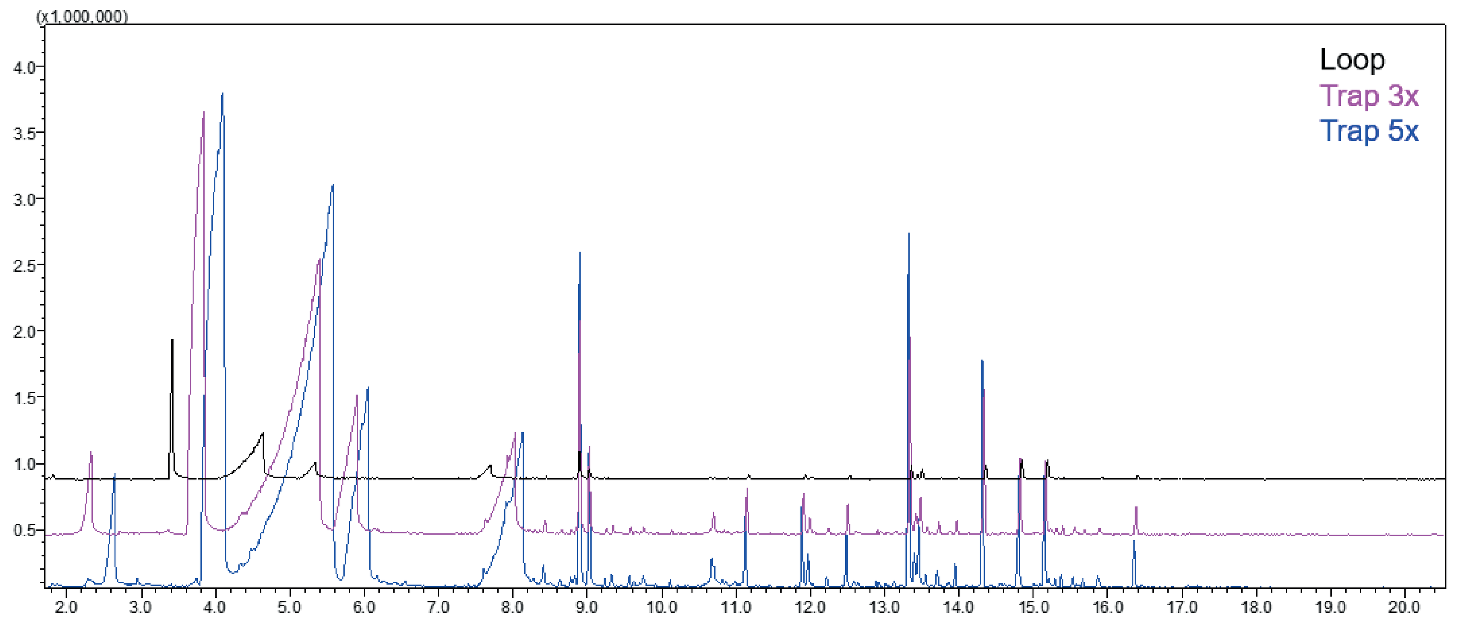

Fig. 4 Comparative chromatogram loop mode and trap mode 3 and 5 times.

Some components such as benzene acetaldehyde, gamma-caprolactone, methyl butyrate, o-guaiacol, that have a decisive influence on the overall aroma and flavor were detected only in the trap mode, due to their low concentrations in the sample. Even concentrating the sample 5-times in the trap, it was possible to detect these compounds that require greater sensitivity along with compounds present in high concentrations in the sample, such as butanoic acid, acetic acid, and others, without losing analysis efficiency.

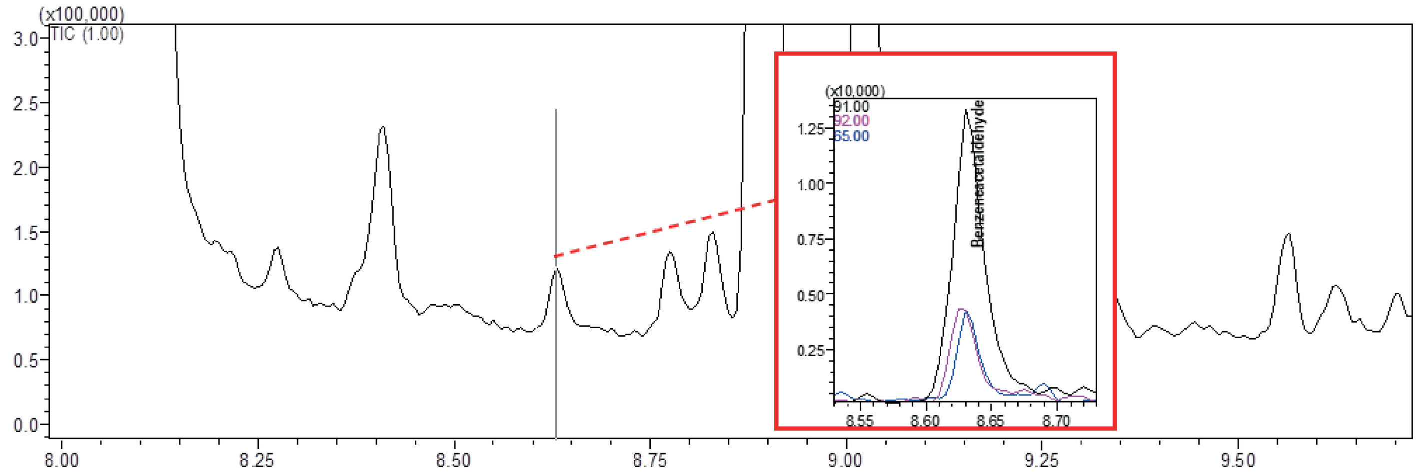

Fig. 5 Benzene acetaldehyde mass chromatogram.

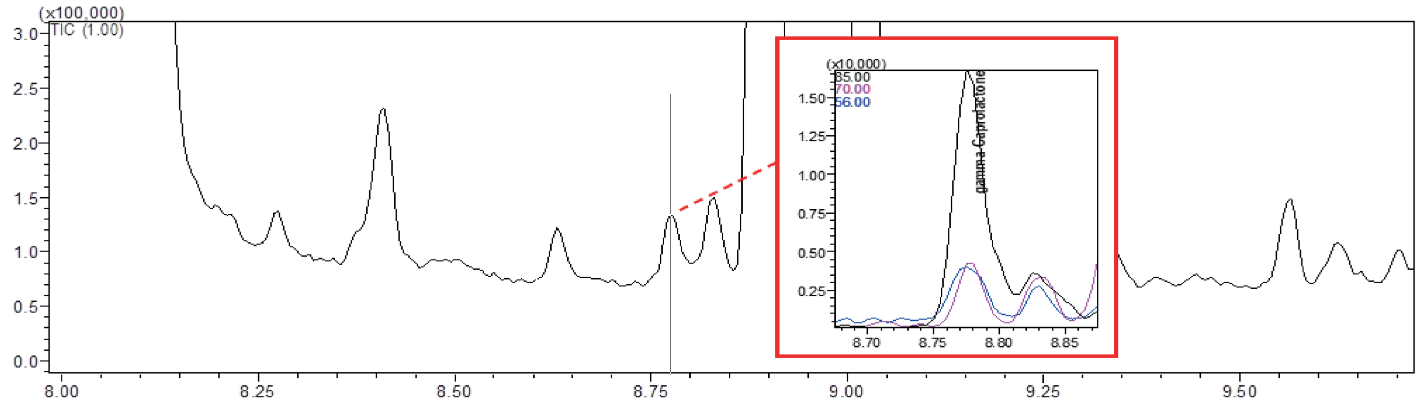

Fig. 6 Gamma-caprolactone mass chromatogram.

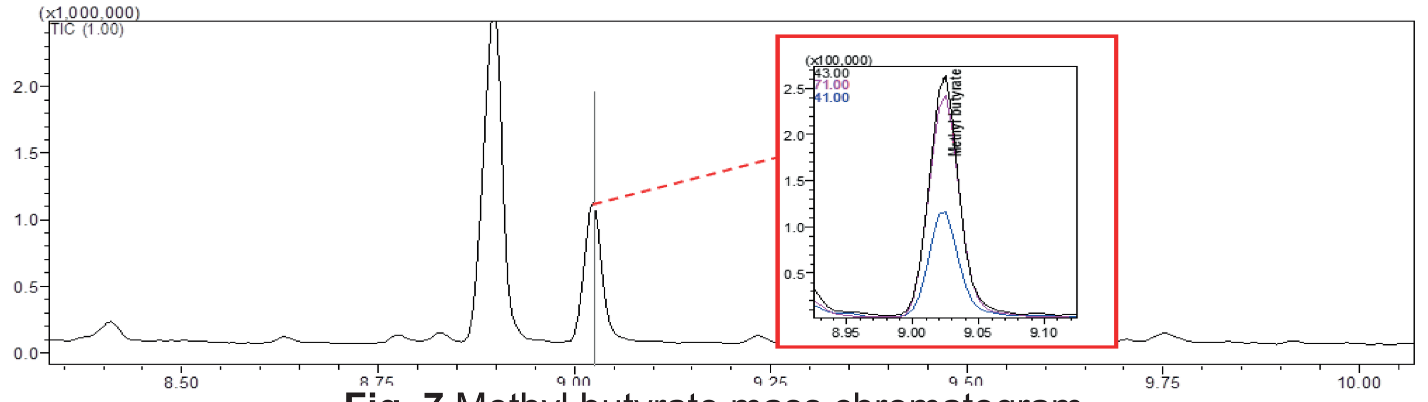

Fig. 7 Methyl butyrate mass chromatogram.

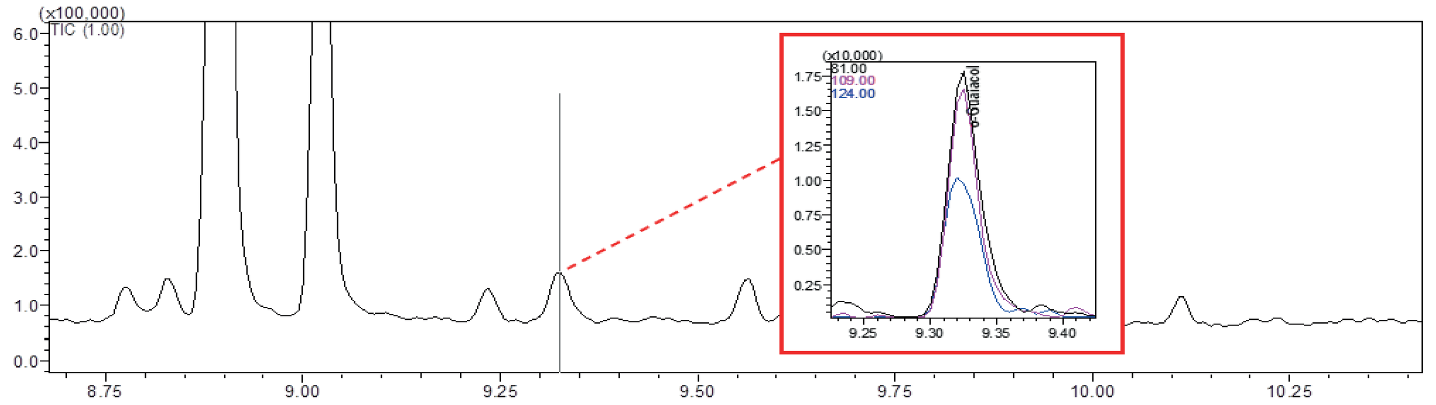

Fig. 8 o-Guaiacol mass chromatogram.

4. Conclusion

The improvement in analysis sensitivity when using trap mode can be observed when comparing the area for some components with a major impact on the overall aroma but present in small quantities. When concentrating the sample 3-times in the trap, the peak intensities increased from 351.17 to 1401.08%. And it was still possible to have a significant increase of another 168%, on average, by concentrating the sample 5-times. The peak intensities increased from 521.48 to 2517.86% compared to loop mode analysis.

These analyses demonstrate that trapped headspace can be used to determine an overall aroma profile in a single analysis and with a single trapping material. The trapped headspace sampler enables ultra sensitivity analysis, identifying compounds at low concentrations which were undetectable with conventional headspace samplers.

COI Disclosure:The authors are affiliated and funded by Shimadzu do Brasil Comércio Ltda.