GC/MS/MS analysis of b-damascenone in rose oil

Application Note

Food Testing & Agriculture

Authors

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Introduction

Beta-damascenone is one of the most important flavor compounds in the essential oil of roses. Its presence and quantity is considered as *the* marker for the quality of rose oil. β-damascenone (Figure 1) is, however, only present in very low concentrations (100 ppm) in the essential oil.

The analysis of β-damascenone in rose oils is, therefore, a typical application where high resolution and compound specific detection is required. Capillary gas chromatography using highly efficient columns does not offer sufficient resolution to ensure complete separation of β-damascenone from the complex mixture of compounds present in the essential oil. Selective sample preparation, to isolate the compound before GC analysis, is also not feasible because the matrix consists of terpenes and terpenoids with similar volatility and polarity.

Selective detection using GC/MS is limited as well since the mass spectrum of damascenone resembles the spectra of other terpenes. For these reasons, based on its high selectivity, GC/MS/MS was evaluated for this purpose.

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Experimental

A Bulgarian rose oil sample (β-damascenone - 100 ppm) was diluted in chloroform (0.1%). The sample was analyzed as such, without any further pretreatment. After dilution of the oil, 100 pg of β-damascenone is injected into the column using a 1 µL injection. For optimization of GC conditions and MS settings, a standard sample containing 0.1 ppm of β-damascenone in chloroform was also analyzed. Two MS modes of operation are compared. First the standard sample and the rose oil sample were analyzed under standard electron impact MS conditions. Then the same standard and sample were analyzed under MS/MS conditions. For MS/MS operation, the molecular ion was selected as the parent ion.

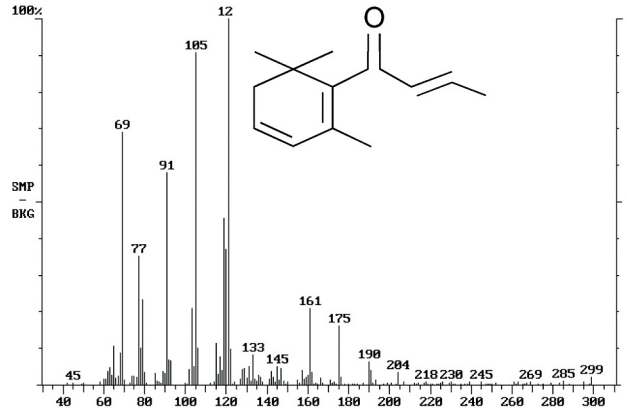

Figure 1: Chemical structure and background subtracted spectrum of β-damascenone

Results

The spectrum of the reference standard of pure β-damascenone analyzed in the EI mode and the chemical structure is shown in Figure 1. Although β-damascenone gives several intense fragments, the ions m/z 69, 77, 91, 105 and 121 are not very specific to β-damascenone (common to other terpenes). The high mass ions 190 and 175 are more characteristic, but present in low abundance. In the MS/MS mode, the signal-to-noise for these smaller ions can be enhanced due to the selectivity of MS/MS. A complex chromatogram is obtained when analyzing the rose oil sample by EI-MS. Upon examination of the spectrum obtained for the expected retention time of β-damascenone, the spectrum (Figure 2) shows additional ion fragments that are not present in Figure 1. The presence of a coeluting compound was confirmed by a library search on this spectrum (caryphyllene MW = 204). From these results, it is obvious that the selectivity of EI-MS is not enough to obtain a good qualitative and quantitative analysis of β-damascenone in the rose oil matrix.

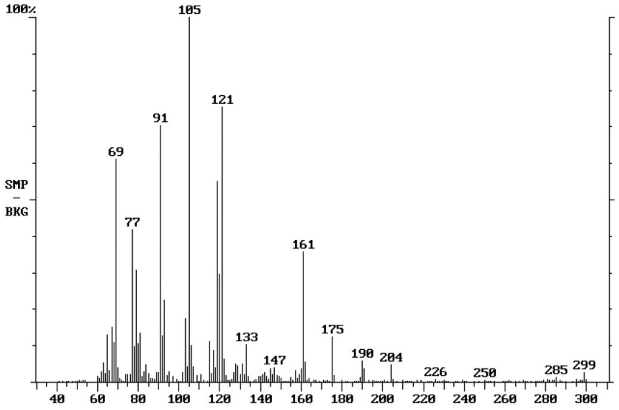

Figure 2: GC/MS spectrum obtained for β-damascenone and a coeluting compound in a rose oil sample.

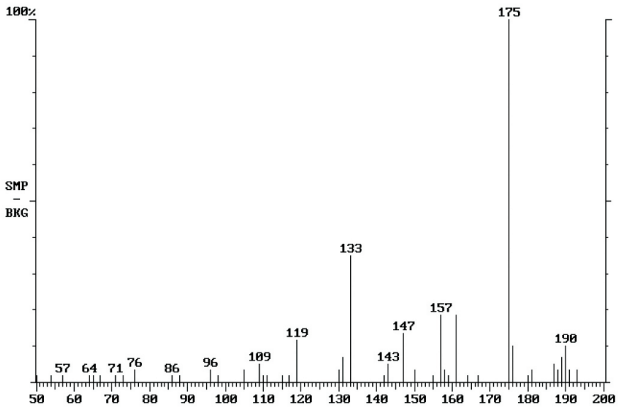

Figure 3: β-damascenone spectrum obtained from a rose oil sample by GC/MS/MS.

For MS/MS analysis the molecular ion (m/z 190) was isolated as the parent ion, and fragmented by non-resonant collision induced dissociation. The resulting spectrum is shown in Figure 3. The spectrum indicates the strong fragmentation of the molecular ion into ion 175 plus other unique ions (133, 14?). An increase in signal-to-noise is also observed with GC/MS/MS compared to GC/MS. This is due to the selectivity of the MS/MS process which removes the background noise contributed by the matrix. Figure 4 shows the signal-to-noise comparison of GC/MS and GC/MS/MS.

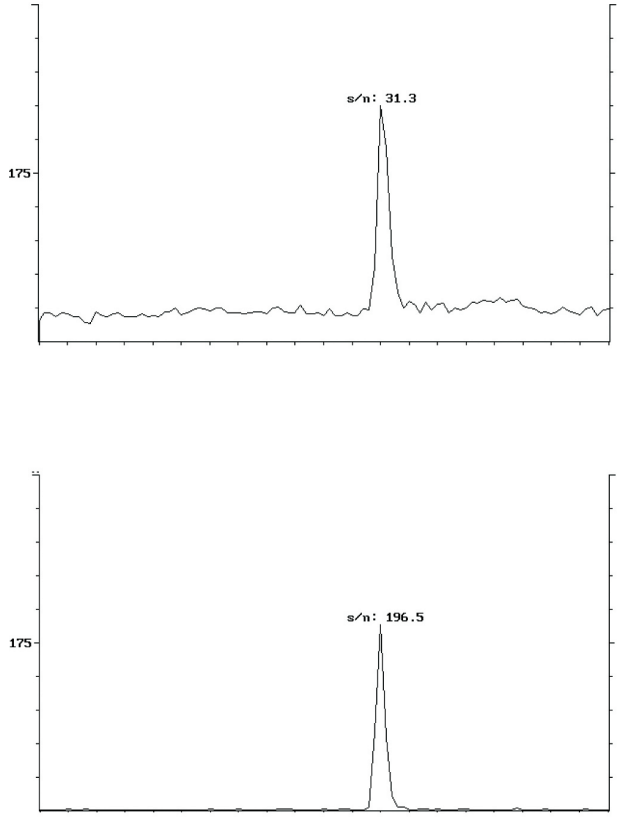

Figure 4: GC/MS analysis of rose oil (top) and GC/MS/MS analysis of rose oil (bottom).

Conclusion

Capillary GC in combination with MS/MS detection can be used for the very specific determination of trace compounds in complex matrices. Due to the high specificity, MS/MS offers a much more accurate determination of β~damascenone in rose oil.

Gas Chromatograph

Column: Arylene modified 5% phenyl/95% methyl PDMS, Agilent equivalent:

CP-Sil 8 CB Low Bleed/MS,

0.25 mm x 30 m x 0.25 µm, Part no. CP5860

Flow Rate: 1 mL/min.

Oven Program: 50 °C for 1 min., then 15 °C/min to 305 °C.

Injector: SPI 30 °C for 0.15 min. then 150 °C/min to 250 °C and hold 15 min.

Transfer Line: 280 °C

Recommended capillary column

Agilent CP-Sil 8 CB Low Bleed/MS

0.25 mm x 30 m, df = 0.25 µm, Part no. CP5860

Mass Spectrometer

Mass Range: 40-300 u

s/Scan: 1

Multiplier Delay: 5 min.

Threshold: 0

Ion Trap Temperature: 220 °C

Mass Defect: 0

Background Mass: 39 u

Target: GC/MS 21000, GC/MS/MS 5000

MS/MS

Parent ion: 190 u

Range: 3 u

Amplitude: 31 volts non-resonant

RF level: 48 u

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