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GC-QTOF for targeted analysis of complex food matrices

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Introduction

Food industry is bound to ever-increasingly strict safety and quality regulations covering several aspects, e.g. the presence of additives, allergens or pesticide residues, and the profiling of markers connected to for example, organoleptic properties. These demands and specifications need suitable characterization techniques.

Gas Chromatography-Mass Spectrometry (GC-MS) is widely used in food analysis, in particular for flavor analysis. However, identification is often very challenging with common mass spectrometers which provide only nominal mass information. This is especially true for minor components present at trace level and co-eluting with a very complex matrix. In this work we show the benefits arising from using GC-QTOF applied to targeted analysis and identity confirmation of aroma-key compounds in mustard.

Instrumentation

All chromatograms were acquired using an Agilent 7890B GC coupled to an Agilent 7200B QTOF. The QTOF was operated in High Resolution mode (mass resolution better than 13,000 FWHM).

Results

The volatile fraction of a commercial mustard was analyzed by SPME-GC-QTOF (Fig. 1). As can be seen in detail in the zoom-in, at lower concentration levels the matrix is very complex and several peaks are co-eluting. Identification of compounds present at trace level is clearly challenging.

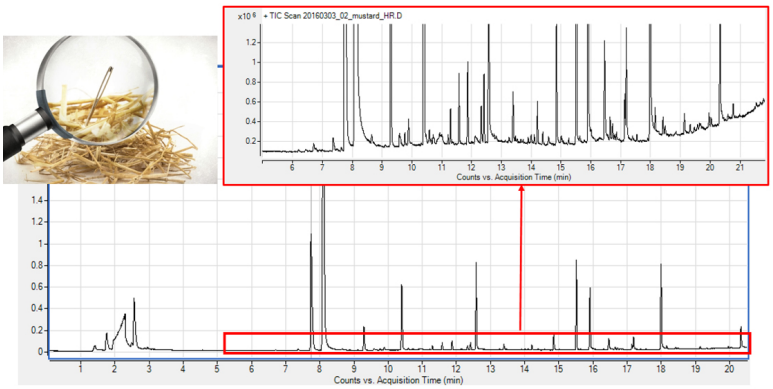

Fig. 1 – HS-SPME-GC-QTOF chromatogram of the mustard sample.

High resolution provides enhanced selectivity also in complex chromatograms thanks to the significantly narrower mass windows. Much cleaner chromatograms are obtained and easier target analysis is possible than if only nominal mass data are available. Fig. 2 shows an example for the peak assignment of benzyl isothiocyanate, a characteristic pungent compound in mustard. When the nominal mass of the molecular ion is extracted the chromatograms shows several compounds but with the exact mass only one peak is present.

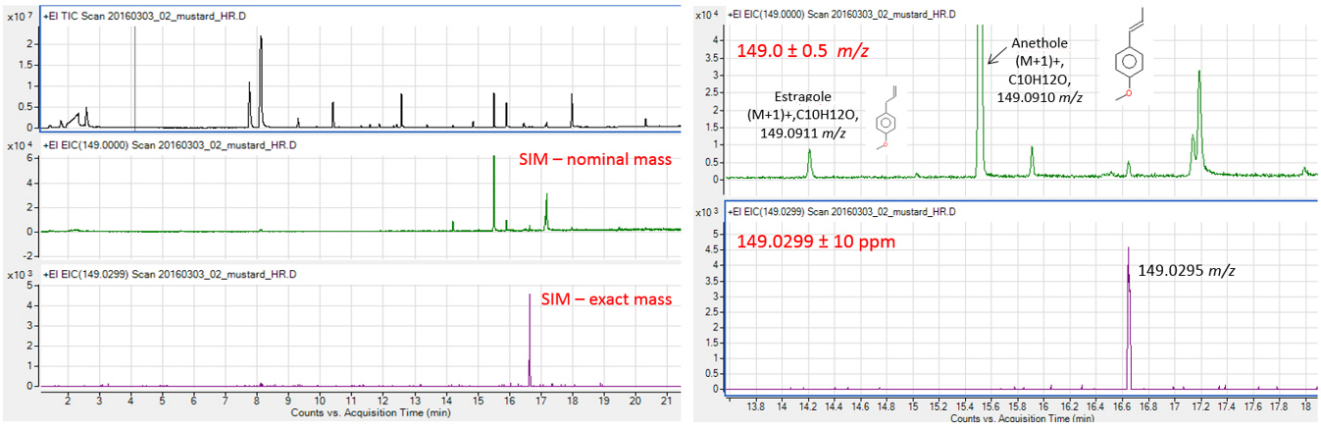

Fig. 2 – TIC chromatograms and extracted chromatograms with nominal and accurate mass for the molecular ion of benzyl isothiocyanate (C8H7NS, molecular mass: 149.0299 m/z).

Accurate mass information greatly contributes to reliable peak identification. The Formula Calculator provides the most likely formulas for any accurate mass measured.

Figure 3 shows the MS spectrum of the peak at 15.5 min identified as anethole by NIST.
If identification is correct, the fragment 148 *m/z* is the molecular ion. This is confirmed by the fact that the accurate mass measured and the proposed formula match those expected for the molecular formula of anethole.

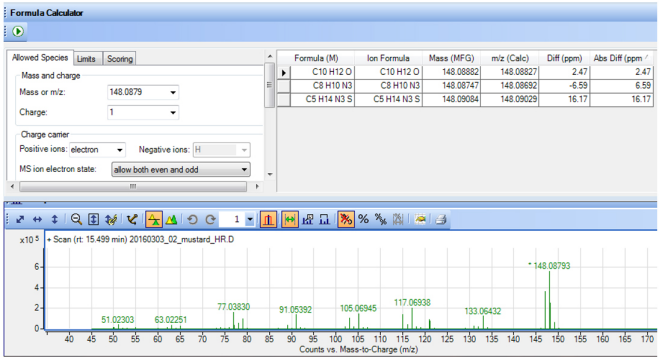

Fig. 3 – Example of mass accuracy calculation for the molecular ion of anethole.

This process can be applied to molecular/major ions as well as to several fragments as shown in Table 1. If the errors obtained are very small for all fragments, identification confidence is significantly improved.

Table 1 – Identity confirmation of target aroma-key compounds in mustard by multi-fragment evaluation of mass accuracy.

Compound	Fragment	Formula	Exact mass	Measured mass	Difference	Error (ppm)
Allyl isothiocyanate	99	C4H5NS+ (M+)	99.0137	99.0137	<0.0001	0.22
	72	C2H2NS	71.9903	71.9904	0.0001	-2.14
	58	CNS+	57.9746	57.9745	0.0001	-0.93
Camphor	152	C10H16O(M+)	152.1196	152.1191	0.0005	3.07
	137	C9H13O+	137.0961	137.0959	0.0002	1.40
	95	C7H11+	95.0855	95.0852	0.0003	3.12
Anethole	148	C10H12O+ (M+)	148.0883	148.0879	0.0004	2.47
	133	C9H9O+	121.0648	121.0645	0.0003	2.24
	117	C9H9+	117.0699	117.0694	0.0005	3.73
Allylnitrile	67	C4H5N (M+)	67.0417	67.0417	<0.0001	-0.74
	66	C4H4N	66.0338	66.0338	<0.0001	0.69
	52	C3H2N	52.0182	52.0184	0.0002	-2.59
Phenethyl isothiocyanate	163	C9H9NS+ (M+)	163.0450	163.0448	0.0002	1.36
	105	C8H9+	105.1699	105.0696	0.0003	2.63
	91	C7H7+	91.0542	91.0541	0.0001	1.39
Benzyl isothiocyanate	149	C8H7NS+ (M+)	142.0294	142.0295	0.0001	-0.59
	91	C7H7+	91.0542	91.0540	0.0002	2.49
	77	C6H5+	77.0386	77.0386	<0.0001	0.35

Conclusions

- The mass accuracy of the QTOF makes it an excellent tool for identification.
- The High Resolution grants greatly enhanced selectivity and sensitivity even for highly complex samples.
- The excellent mass accuracy can be used to discriminate between identification choices with the same nominal mass but different formula and thus different exact masses.
- Multi-fragment evaluation of mass accuracy data improves the confidence of compound identifications.

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