JSB is an authorised partner of Agilent Technologies Value Added Reseller

GC×GC-QTOF: a comprehensive approach to tackle identification challenges in complex food matrices

Author application note: Daniela Peroni, JSB © 2016

Introduction

Comprehensive two-dimensional gas chromatography coupled to High Resolution Mass Spectrometry (GC×GC-HRMS) is an extremely powerful method for detailed profiling of highly complex samples. The high resolution and accurate mass measurements give substantial advantages to tackle identification challenges. Identity confirmation of target compounds and identification of unknowns can be performed with more confidence than with nominal mass systems. Here we show the benefits arising from using SPME-GC×GC-QTOF as identification tool for the complex volatiles fraction of different food matrices.

Experimental details

Agilent 7890A GC with a cryogen-free Zoex ZX2 thermal modulator and an Agilent 7200B QTOF detector.

Results and discussion

3.1 Targeted compound analysis

The high resolution provides enhanced selectivity also in complex chromatograms thanks to the significantly narrower mass windows possible. This means that much cleaner chromatograms and easier target analysis is possible than with nominal mass data (Fig. 1).

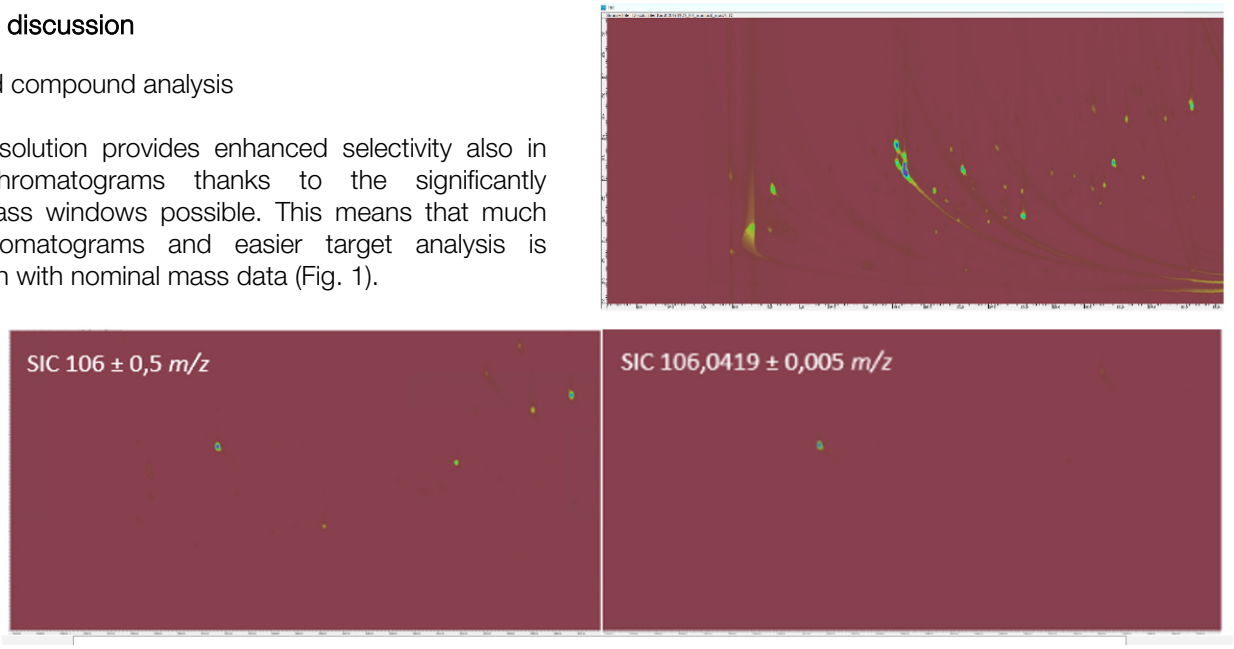


Fig. 1 – TIC chromatogram of a MUSTARD sample (top) and Selected Ion Chromatograms (SIC) of the molecular fragment of benzaldehyde by nominal mass and accurate mass.

Accurate mass data can be used to narrow down the list of possible library matches and confirm identification. The accurate masses measured are compared to the exact masses predicted for the structure of the suggested library match. This process can be applied to molecular/major ions as well as to several fragments (Table 1). If the errors obtained are very small for all fragments, identification confidence is significantly improved.

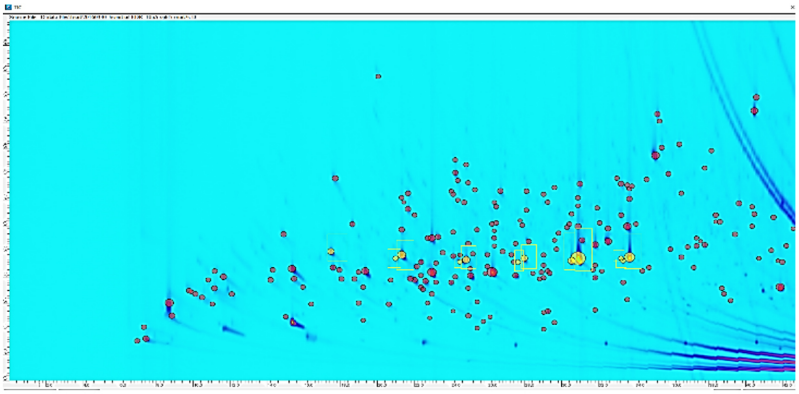
Table 1 – Identity confirmation by multi-fragment mass accuracy evaluation for aroma compounds in a TEA infusion.

Compound	Fragment	Library match formula	Exact mass	Measured mass	Suggested formula	Error (ppm)
Linalyl acetate	196	C12H20O2+	196.1458	196.1458	C12H20O2+	0.23
	154	C10H18O+	154.1352	154.1352	C10H18O+	0.20
	136	C10H16+	136.1247	136.1244	C10H16+	1.65
	121	C9H13+	121.1012	121.1010	C9H13+	1.57
Caryophyllene	204	C15H24+	204.1872	204.1871	C15H24+	0.78
	189	C14H21+	189.1638	189.1637	C14H21+	0.15
	147	C11H15+	147.1168	147.1167	C11H15+	1.03
	133	C10H13+	133.1012	133.1006	C10H13+	4.47
Citral	152	C10H16O+	152.1196	152,1198	C10H16O+	1.36
	137	C9H13O+	137.0961	137.0966	C9H13O+	3,34
	123	C9H15+	123.1168	123.1168	C9H15+	0,53
	94	C7H10+	94.0777	94.0775	C7H10+	1.86

3.2 Group characterization

The enhanced selectivity can be used, in combination with the ordered 2D chromatograms, for group characterization. Peaks can be filtered by accurate masses for specific functional groups and retention times windows. Fig. 3 shows the mono-unsaturated aldehydes selected among over 200 peaks found for an OLIVE OIL sample. Constrains: (i) m/z 83,049 ±0,05 (C5H7O+) is one of the two most abundant fragments and (ii) second dimension retention time <2,5 s. All C6-C11 isomers, from 2-hexenal to 2-undecenal, are successfully found. No additional, undesired peaks are included in the search.

Fig. 3 – Group search of mono-unsaturated aldehydes (yellow) in OLIVE OIL.



3.3 Fingerprinting

The 2D pattern and MS information of a sample is a unique fingerprint. The degree of similarity between samples can be determined by pair-wise comparisons of templates of compounds built on their respective patterns (Table 2). This approach is very effective to differentiate and discriminate food samples based on origin or treatment. Markers can be selected as the compounds with the highest variability and identified based on their accurate mass information

Table 2 – Pattern similarity index (as matching %) between 7 brands of Earl Grey TEA analysed as leaves. The patterns contain from 245 to 473 peaks/sample.

	Template Matching (%)						
	template 1	template 2	template 3	template 4	template 5	template 6	template 7
sample 1		52,7	66,3	62,5	74,6	68,0	59,8
sample 2	41,9		50,1	54,1	60,1	49,4	38,7
sample 3	53,5	58,0		58,0	75,6	67,7	54,1
sample 4	24,8	60,8	39,8		61,4	40,7	28,8
sample 5	54,6	59,6	67,4	62,2		64,3	50,1
sample 6	50,9	52,2	63,8	55,1	67,3		54,8
sample 7	65,7	55,9	75,8	66,1	74,6	79,2	

Conclusions

- HS-SPME-GC×GC-HRMS can be used to characterize in depth the complex volatiles profiles of different food matrices.
- Results obtained for olive oil, mustard and tea samples are shown as feasibility examples of the different work flows.
- The high peak capacity can be exploited for target analysis, group characterization as well as fingerprinting applications.
- High Resolution provides enhanced selectivity and “information density”.
- High mass accuracy is an extremely powerful profiling tool for identity confirmation and identification of unknowns.

HEAD OFFICE

JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

INFO@GO-JSB.COM
WWW.GO-JSB.COM

SALES AND SERVICE

NETHERLANDS
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 32 087 00 18
F +31 (0) 32 087 00 19

GERMANY, AUSTRIA, SWITZERLAND
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

BELGIUM
Grensstraat 7, Box 3
1831 Diegem
T +32 (0) 27219211
F +32 (0) 27207622

UK & IRELAND
Cedar Court, Grove Park Business Est. White
Waltham, Maidenhead, Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

SHARPER PEAKS, BETTER SEPARATIONS!