Characterization and Comparison of Basil Before and After Drying Using GC, GCxGC, and TOFMS

INTRODUCTION

GC and MS are well-established techniques for the characterization of food, GCxGC was crucial for the determination of many of the analytes that differed Representative GC and GCxGC chromatograms for fresh and cured basil are Additional differences between the fresh and cured basil samples were beverage, and flavor samples. Extending the analytical separation to two shown in Figure 2. Some differences between the samples are visually apparent explored with ChromaTOF Tile software. With ChromaTOF Tile, chromatographic between the fresh and cured basil. Some key analyte differences were dimensions with comprehensive two-dimensional gas chromatography while others are obscured in the TIC. GCxGC improves the peak capacity and coeluting in the 1D data, as shown in Figures 4, 6, and 7. GCxGC windows that distinguish the sample groups are identified and the (GCxGC) enhances the peak capacity, allows for exploring more complex chromatographically separates more analytes to help uncover the hidden corresponding features can be determined. Some representative analytes that chromatographically separated these coelutions and then ChromaTOF Tile helped to locate them within the complex data. differences. differ between the samples are shown in Figure 5. samples, and allows for determining more individual analytes within complex samples. TOFMS adds to these benefits and often leads to the identification for these isolated analytes, supporting non-target analyses where analytes of 2.0e10 - Fresh Basil **Chroma Tile** interest and importance can be determined through data evaluation. When 1.5e10 -3-hexen-1-o coupled together, the benefits are often greater than the sum of the individual 1.0e10 techniques. -hexen-1-o Curad Erach

Software tools that compare sets of GCxGC samples facilitate these analyses and can effectively reveal useful information from the rich data. In this work, we evaluate the aroma characteristics for basil before and after drying. This type of characterization can be useful for quality control, product development, and batch comparisons, as well as understanding the sensory profile changes associated with curing.

METHOD

Cured basil was prepared from a fresh basil sample by heating at 60 °C until the moisture level was between 5 and 10 %. Moisture levels were determined with the TGM800 moisture analyzer (LECO, St. Joseph, MI, USA), as shown in Figure 1. Fresh and cured samples were then analyzed by GC and GCxGC-TOFMS on a LECO Pegasus[®] BT 4D, as described in Table 1 and shown in Figure 1. Sample amounts were adjusted based on moisture content to analyze approximately 11-12 mg of plant material (100 mg of fresh and 13 mg of cured). An alkane standard was analyzed with the same methods for retention index (RI) determinations.

AS	LECO L-PAL3 Autosampler
Injection	HS-SPME, 2 min incubate and 5 min extract at 40 °C with triphase fiber
GCxGC	LECO GCxGC QuadJet™ Thermal Modulator
Inlet	250 °C, split 20:1
Carrier Gas	He @ 1.40 mL/min, corrected constant flow
Columns	Column 1: Rxi-5ms, 30 m x 0.25 mm i.d. x 0.25 µm coating (Restek)
	Column 2: Rxi-17Sil MS, 0.6 m x 0.25 mm i.d. x 0.25 µm coating (Restek)
Temperature	2 min 40 °C, ramp 10 °C/min to 250 °C, hold 2 min
Program	Secondary Oven: + 10 °C
Modulation	1.5 s with temperature maintained +15 °C relative to 2nd oven
Transfer Line	250 °C
MS	LECO Pegasus BT
Ion Source Temp	250 °C
Mass range	35-500 m/z
Acquisition Rate	10 spectra/s (GC) and 100 spectra/s (GCxGC)
	LECO TGM800

Table 1. Instrument Conditions

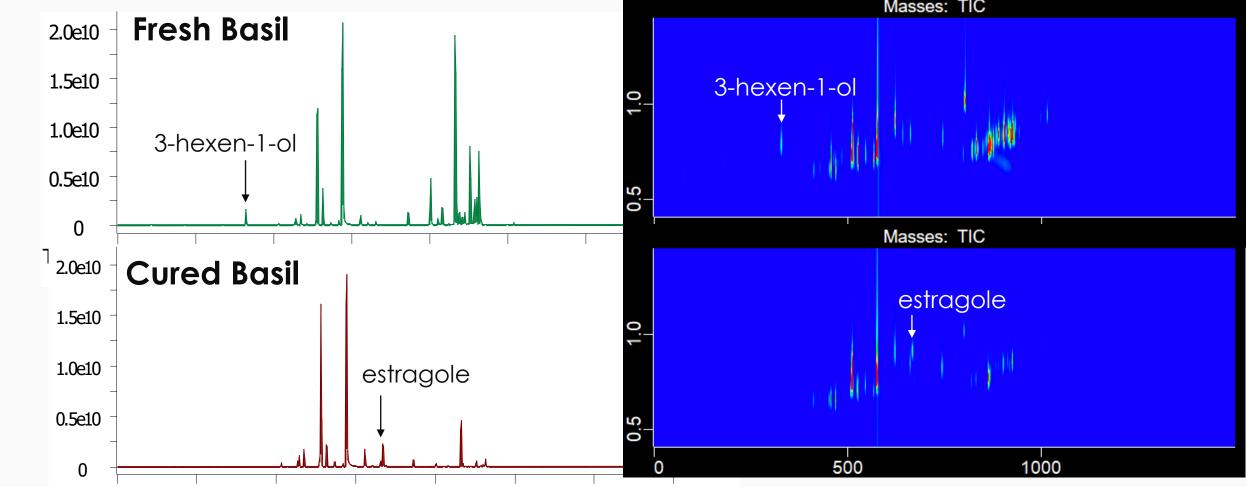


Figure 1. Fresh and cured basil were analyzed. The moisture level in the samples was measured with LECO's TGM800 and aroma profiling was done with LECO's Pegasus BT 4D.

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Fresh and Cured Basil









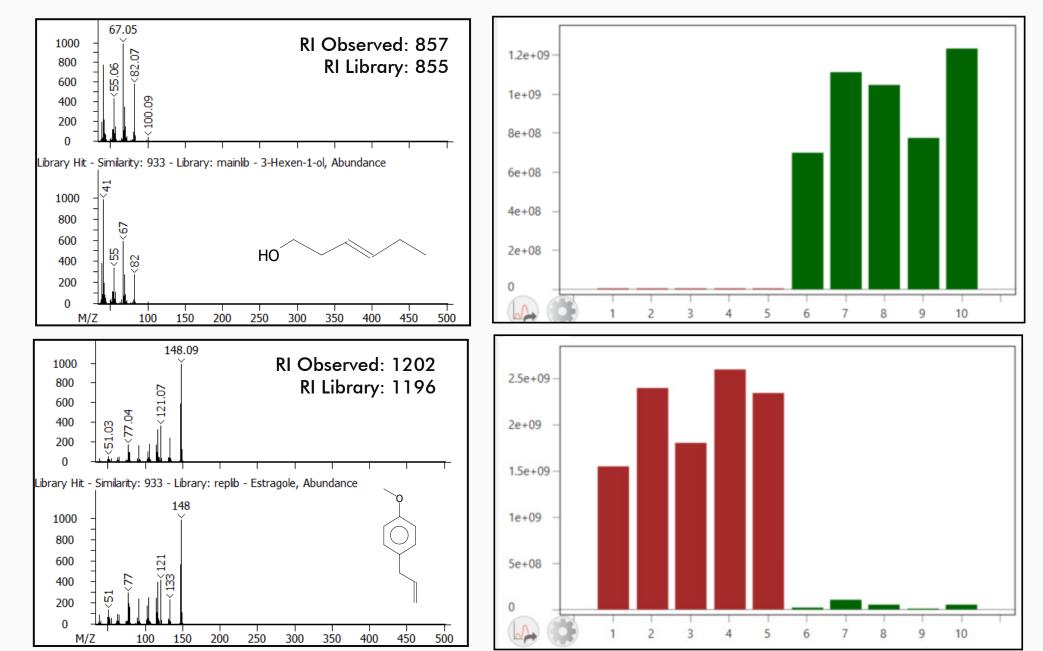


Figure 3. Representative analytes, 3-hexen-1-ol and estragole, differ between fresh and cured basil. These differences were visually apparent in both the GC and GCxGC data as highlighted in Figure 2. Both were readily found with the data analysis tools and are indicated with asterisks in Figure 5.

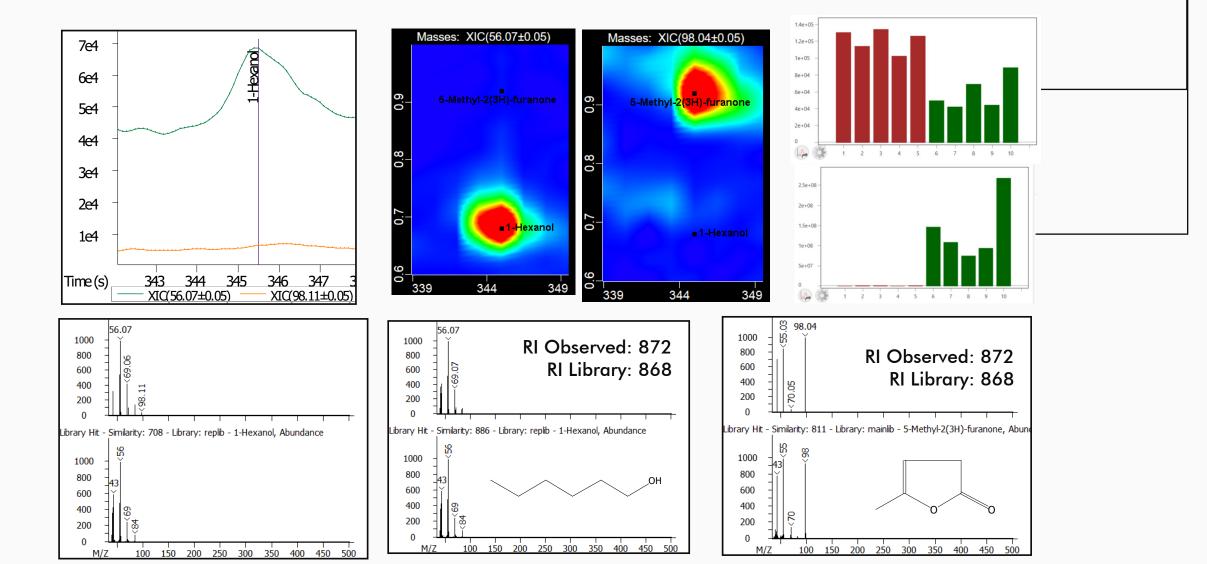


Figure 4. Other analyte differences were obscured in the 1D GC data. Two analytes that coelute in the GC data were chromatographically resolved with GCxGC, allowing for improved spectral similarity scores and revealing additional differences between the fresh and cured basil. These were also readily found with the data analysis tools, as indicated in Figure 5.

ChromaTOF[®] Tile to Find Features of Interest

											Cured	Fresh
Name	Formula	Similarity	CAS	Quant mass	R.I. calc	R.I. lib	aroma	descriptor	Med RT1	Med RT2	1 2 3 4 5	6 7 8 9
3-Hexen-1-ol, (Z)-	C ₆ H ₁₂ O	889	928-96-1	67	858	857	green	fresh green cut grass foliage vegetable herbal oily	328.5	0.79		
3-Hexenal	C ₆ H ₁₀ O	901	4440-65-7	69	802	810	green	leafy green stem tomato melon apple	270.0	0.70		
2-Hexen-1-ol, (Z)-	C ₆ H ₁₂ O	894	928-94-9	82	869	868	green	green cortex leafy green bean nasturtium herbal soapy ald	340.5	0.73		
1-Hexanol	C ₆ H ₁₄ O	817	111-27-3	56	872	868	herbal	ethereal fusel oil fruity alcoholic sweet green	343.5	0.70		
y-Hexanolactone	C ₆ H ₁₀ O ₂	843	695-06-7	85	1058	1056	tonka	herbal coconut sweet coumarin tobacco	534.0	1.11		
Methyleugenol	C11H14O2	891	93-15-2	178	1405	1403	spicy	sweet fresh warm spicy clove carnation cinnamon	833.9	1.01		
sopropyl myristate	C17H34O2	837	110-27-0	228	1827	1825	mild	faint oily fatty	1123.4	0.79		
3-Octanol	C ₈ H ₁₈ O	944	589-98-0	83	995	993	earthy	earthy mushroom herbal melon citrus woody spicy minty	472.5	0.68		
arnesyl bromide	C15H25Br	831	6874-67-5	68	1699	N.A.			1042.4	0.99		
2,4-Hexadienal, (E,E)-	C ₆ H ₈ O	887	142-83-6	81	913	911	green	sweet green spicy floral citrus	387.0	0.83		
1,4-Dibutyl benzene-1,4-dicarbox	C16H22O4	947	1962-75-0	149	1976	1963			1211.9	1.15		
x-elemene	C15H24	833	5951-67-7	204	1459	1454			874.4	0.82		
δ-Guaiene	C15H24	876	3691-11-0	189	1503	1505			907.4	0.85		
p-Chavicol	C ₉ H ₁₀ O	927	501-92-8	134	1254	1255	phenolic	phenolic medicinal herbal	711.0	1.00		
Naphthalene, 1,2,4a,5,6,8a-hexahy	C15H24	832	17627-24-6	161	1590	N.A.			968.9	0.91		
Epicubenol	C15H26O	904	19912-67-5	204	1632	1627			997.4	0.92		
α-cadinene	C15H24	913	24406-05-1	204	1550	1538	woody	woody dry	940.4	0.85		
Hexanal				82	802	N.A.	green	fresh green fatty aldehydic grass leafy fruity sweaty	270.0	0.70		
1-Octen-3-ol	C ₈ H ₁₆ O	826	3391-86-4	57	979	980	earthy	mushroom earthy green oily fungal raw chicken	456.0	0.72		
-Muurolene	C15H24	856	30021-74-0	204	1477	1477	woody	herbal woody spice	887.9	0.84		
Gleenol	C15H26O	914	72203-99-7	121	1607	1586			980.9	0.89		
1,1,4a-Trimethyl-5,6-dimethylene	C15H24	865		204	1518	N.A.			917.9	0.84		
Azulene, 1,2,3,5,6,7,8,8a-octahydro	C15H24	855	3691-11-0	204	1512	1505			913.4	0.85		
Nerolidol, trans	C15H26O	878	40716-66-3	69	1569	1564	floral	floral green citrus woody waxy	953.9	0.82		
Naphthalene, 1,2,3,5,6,7,8,8a-octa	C15H24	883	10219-75-7	93	1489	1494			896.9	0.83		
5-Cadinene	C15H24	888	483-76-1	105	1535	1524	herbal	thyme herbal woody dry	929.9	0.84		
Caryophyllene	C15H24	910	87-44-5	93	1435	1419	spicy	sweet woody spice clove dry	856.4	0.80		
Patchoulene	C15H24	851	1405-16-9	204	1469	1467			881.9	0.84		
Oxime-, methoxy-phenyl	C ₈ H ₉ NO ₂	808		133	901	N.A.			375.0	0.66		
3-Sesquiphellandrene	C15H24	864	20307-83-9	69	1533	1524	herbal	herbal fruity woody	928.4	0.82		
Furan, 2-ethyl-	C ₆ H ₈ O	739	3208-16-0	81	704	703	chemical	chemical beany ethereal cocoa bready malty coffee nutty	184.5	0.61		
Cyclopropanebutanoic acid, 2-[[2	C25H42O2	729	56051-53-7	74	1932	N.A.			1186.4	0.83		
2-Naphthalenol, 2,3,4,4a,5,6,7-oct	C15H26O2	848		81	1710	N.A.			1049.9	1.03		
1-Butanol, 2-methyl-	C5H12O	907	137-32-6	57	739	739	ethereal	ethereal fusel alcoholic fatty greasy winey whiskey leathery	214.5	0.68		
epi-α-Cadinol	C15H26O	899	5937-11-1	204	1654	1640	balsamic	balsamic earthy	1012.4	0.94		
Cedrene	C15H24	851	11028-42-5	204	1405	1422	woody	cedarwood woody	833.9	0.76		
Penten-1-ol, (E)-	C ₅ H ₁₀ O	893	1576-96-1	57	773	769	mushroom	mushroom	244.5	0.70		
x-Cedrene	C15H24	864	469-61-4		1417	1411	woody	woody cedar sweet fresh	842.9	0.78		
ongifolene	C15H24	849	475-20-7	204	1401	1406	woody	sweet woody rose medical fir needle	830.9	0.77		
5-Elemene	C15H24	823	20307-84-0		1346	1338	herbal	sweet herbal woody	787.4	0.74		
2-(4a,8-Dimethyl-2,3,4,4a,5,6-hex	C15H24O	845		161	1562	N.A.			949.4	0.87		
2-Hexenal, (E)-	C6H100	840	6728-26-3	83	850	854	green	green banana aldehydic fatty cheesy	321.0	0.79		

Butanal, 3-methyl-	C ₅ H ₁₀ O	936	590-86-3	57	653	652	aldehydic	ethereal aldehydic chocolate peach fatty	154.5	0.60
4-Carene, (1S,3R,6R)-(-)-	C10H16	886	5208-49-1	93	1222	N.A.	none		684.0	0.74
+)-Borneol acetate	C12H20O2	902	20347-65-3	95	1291	1293	camphoreous	pine needle camphor herbal balsamic	742.5	0.83
,6-Dimethyl-1,3,5,7-octatetraene	C10H14	917	460-01-5	119	1133	1131			604.5	0.77
,4-Cyclohexadiene, 3-ethenyl-1,2	C10H14	829	62338-57-2	134	1261	N.A.			717.0	0.88
2-Oxabicyclo[2.2.2]octan-6-one, 1	C10H16O2	889	107598-08-3	141	1220	1217			682.5	0.97
Octanal	C ₈ H ₁₆ O	882	124-13-0	84	1003	1003	aldehydic	aldehydic waxy citrus orange peel green herbal fresh fatty	480.0	0.71
rans-Sabinyl acetate	C12H18O2	842	139757-62-3	91	1305	1297			754.5	0.85
5-methyl 2(3H)-Furanone				98	873	N.A.	coconut	sweet solvent nutty tonka coumarin tobacco	345.0	0.92
o-Cymene	C10H14	896	99-87-6	134	1027	1025	terpenic	fresh citrus terpene woody spice	504.0	0.74
2(4H)-Benzofuranone, 5,6,7,7a-te	C11H16O2	710	17092-92-1	111	1550	1532	musk	musk coumarin	940.4	1.22
1,3-Cyclohexadiene-1-methanol,	C11H18O	781	102676-97-1	107	1264	N.A.			720.0	0.87
Myrtenyl acetate	C12H18O2	868	1079-01-2	91	1330	1327	herbal	herbal fresh sweet fruity citrus	774.0	0.87
Methyl vinyl ketone	C ₄ H ₆ O	764	78-94-4	55	593	576	sweet	sweet	123.0	0.61
eature 01429				98	1128	N.A.			600.0	1.16
2,4-Heptadienal, (E,E)-	C7H10O	915	4313-03-5	81	998	1012	fatty	fatty green oily aldehydic vegetable cake cinnamon	475.5	0.81
eature 57063				112	905	N.A.			378.0	0.82
eature 01748				119	1247	N.A.			705.0	0.98
eature 13358				71	933	N.A.			408.0	0.77
2,6-Octadienal, 3,7-dimethyl-, (E)	C10H16O	846	141-27-5	69	1273	1270	citrus	citrus lemon	727.5	0.88
2-Butenal, 3-methyl-	C₅H ₈ O	927	107-86-8	84	792	782	fruity	sweet fruity pungent brown nutty almond cherry	261.0	0.76
Acetic acid, octyl ester	C10H20O2	929	112-14-1	70	1209	1210	floral	green earthy mushroom herbal waxy	673.5	0.76
1S,4R,5R)-1,3,3-Trimethyl-2-oxab	C12H20O3	770	81781-24-0	137	1347	1343			787.4	0.89
Myroxide	C10H16O	884	28977-57-3	79	1143	1141			613.5	0.80
2-Furanmethanol, 5-ethenyltetrah	C10H18O2	917	5989-33-3	59	1075	1074	earthy	earthy floral sweet woody	550.5	0.71
Alpha,alpha,4-trimethylbenzyl car	C17H19NO:	775	7366-54-3	132	1187	N.A.			654.0	0.91
1,2,3,5,6,7-Hexahydro-inden-4-on	C ₉ H ₁₂ O	725	22118-01-0	108	1001	N.A.			478.5	0.94
Acetoin	C ₄ H ₈ O ₂	767	513-86-0	88	714	713	buttery	sweet buttery creamy dairy milky fatty	193.5	0.73
2-Cyclopenten-1-one, 2-(2-buten	C10H14O	833	17190-71-5	150	1182	1184			649.5	0.78
Pentanal	C ₅ H ₁₀ O	799	110-62-3	58	701	700	fermented	fermented bready fruity nutty berry	181.5	0.64
,2-Epoxy-3-propyl acetate	C ₅ H ₈ O ₃	788	6387-89-9	74	775	N.A.			246.0	0.85
Butanoic acid, ethyl ester	C ₆ H ₁₂ O ₂	872	105-54-4	88	806	802	fruity	fruity juicy fruit pineapple cognac	274.5	0.65
Estragole	C10H12O	912	140-67-0	148	1201	1196	anisic	sweet sassafrass anise spice green herbal fennel	666.0	0.91
Benzyl chloride	C ₇ H ₇ Cl	872	100-44-7	126	1017	1023	none		493.5	0.91
Acetic acid	C ₂ H ₄ O ₂	911	64-19-7	60	581	610	acidic	sharp pungent sour vinegar	120.0	0.57
Pyrazine	C ₄ H ₄ N ₂	875	290-37-9	80	749	736	nutty	pungent sweet corn like roasted hazelnut barly	223.5	0.83

Figure 5. Representative examples of features that differ between the fresh and cured basil are shown. These were located and compiled with ChromaTOF Tile. Tentative identifications are listed.



Improved Peak Capacity with GCxGC

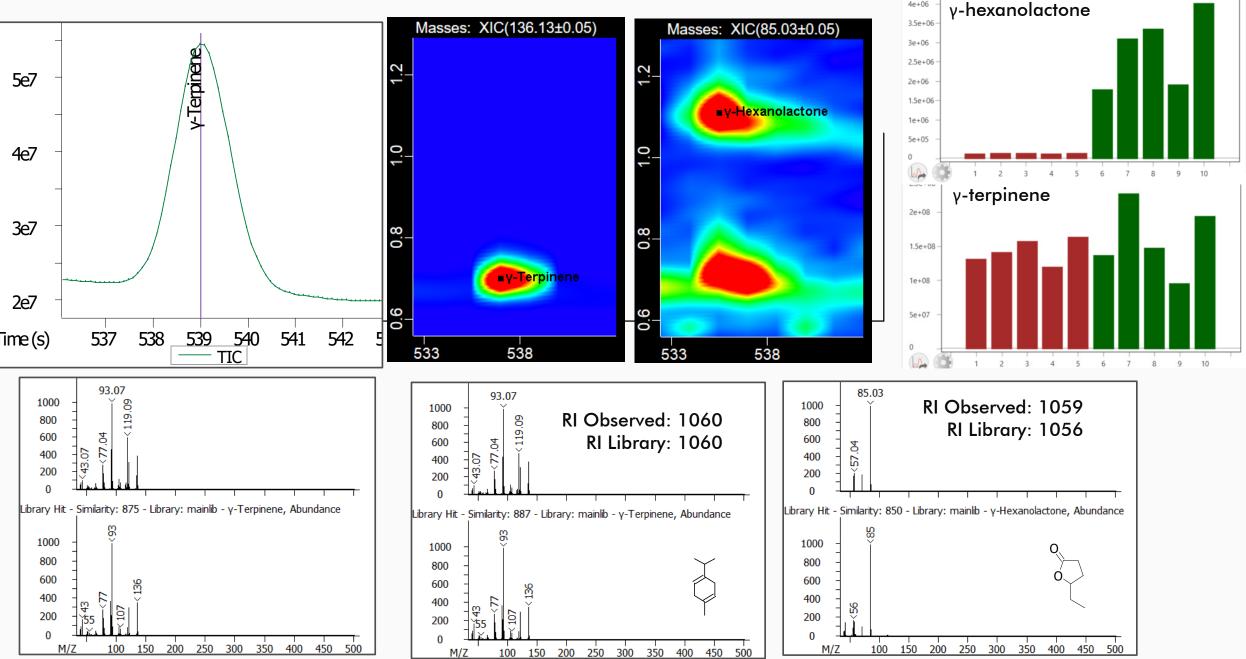


Figure 6. A lactone, observed at higher levels in the fresh basil, was hidden by a terpene in the 1D GC data. The coelution was resolved in the second dimension of the GCxGC data allowing the additional analyte to be identified and highlighted with ChromaTOF Tile.

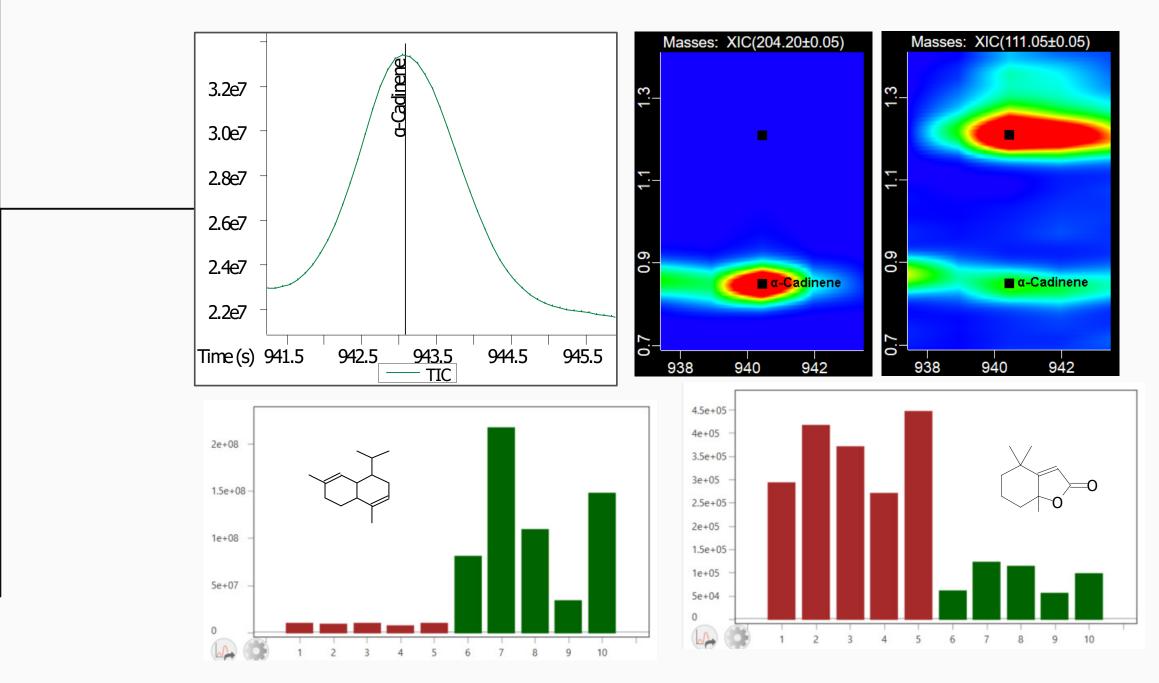


Figure 7. Two analytes that were observed to distinguish the fresh and cured basil were coeluting in the 1D GC data. The coelution was resolved in the second dimension of the GCxGC data and the difference was highlighted with ChromaTOF Tile.

CONCLUSIONS

In this work, LECO's TGM800 was used to determine the moisture level of fresh and cured basil samples and LECO's Pegasus BT4D GCxGC-TOFMS with ChromaTOF Tile were used to highlight differences between the chemicals which contribute to the sensory profiles of fresh and cured basil. This provided a better understanding of the aroma characteristics of these samples. Many differences were observed between the samples and the analytical tools used in this work were crucial for their determination.