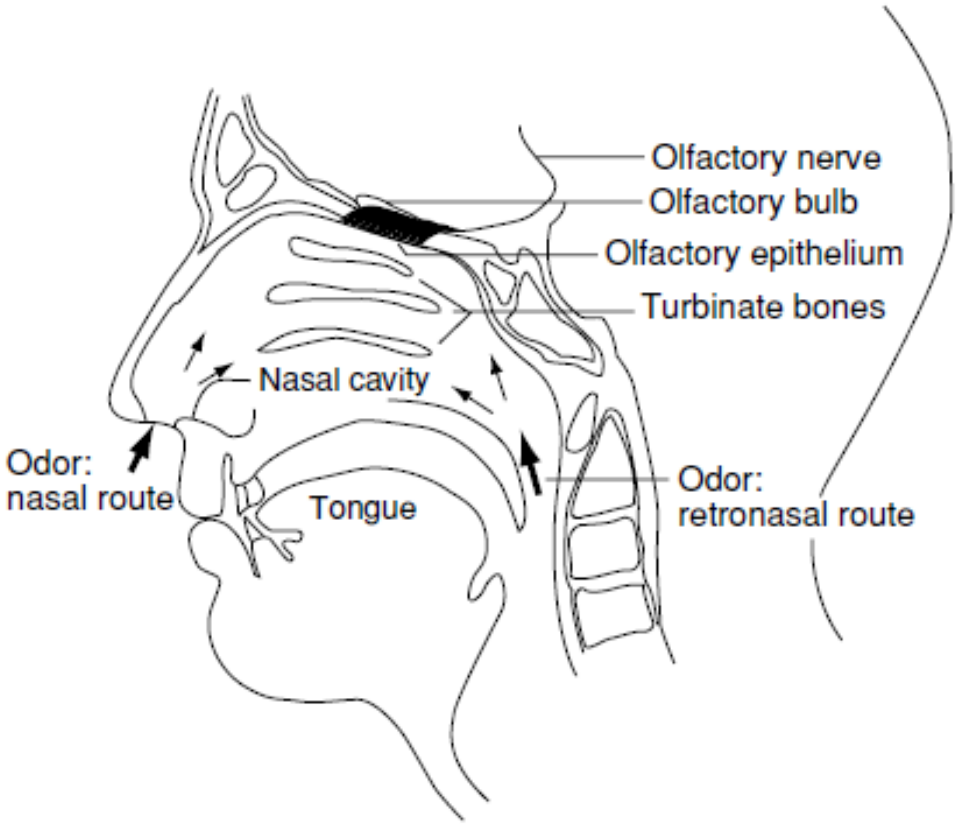
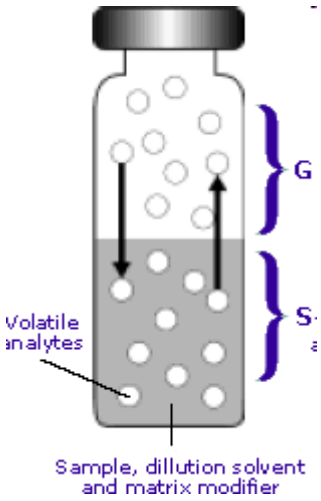
Τεχνικές ανάλυσης ενώσεων υπευθύνων για τη γεύση και την όσφρηση



Στάδια για την ανάλυση πτητικών αρωματικών ενώσεων

Step	*Analytical techniques*
Isolation and concentration	Headspace analysis
	Adsorption
	Distillation
	Extraction
Separation	Gas chromatography
	Preparative liquid chromatography
Identification	Chromatographic retention time
	Mass spectrometry
	Infrared spectroscopy
	Nuclear magnetic resonance spectroscopy
	Chemical synthesis
	Reference compounds
Sensory characterization	Gas chromatography olfactometry
	Sensory panel

Ανάλυση υπερκείμενης φάσης (Headspace)



Στατική

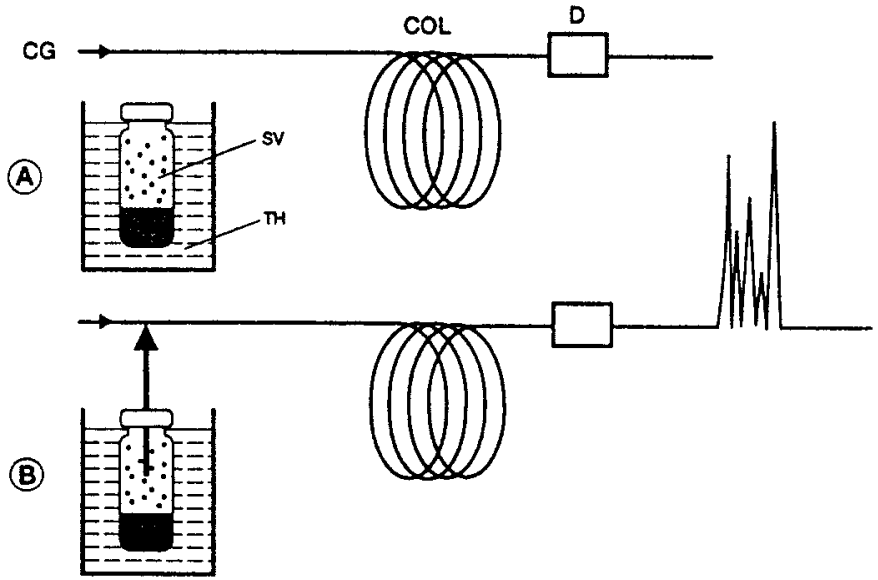
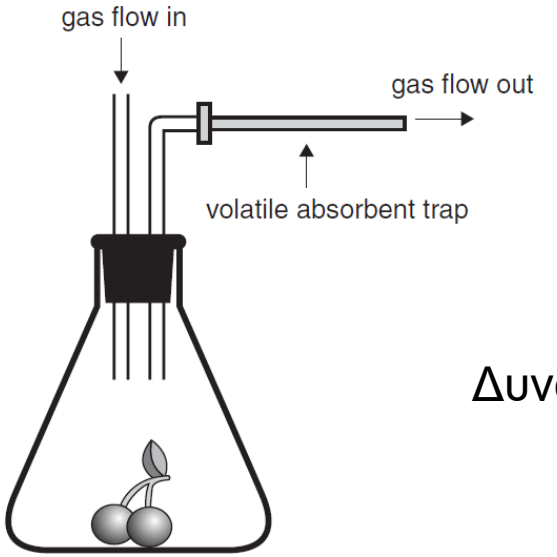


Figure 1.1 Principles of static (equilibrium) headspace–gas chromatography. (A) equilibration and (B) sample transfer. *CG* = carrier gas; *SV* = sample vial, *TH* = thermostat, *COL* = gas chromatographic column, *D* = detector.



Δυναμική

Αέρια Χρωματογραφία ΥΦ σταφυλιού

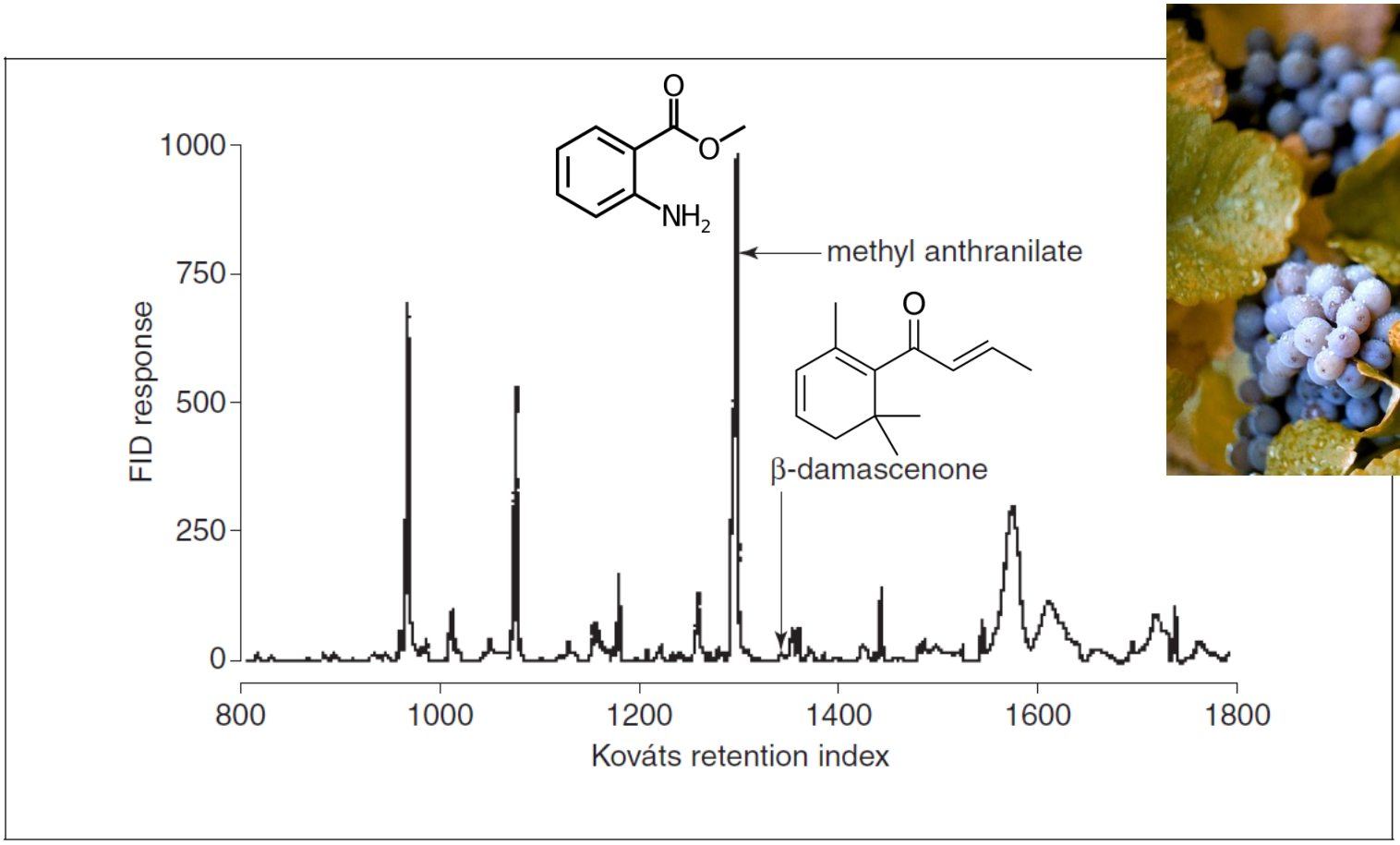


Figure G1.1.3 FID gas chromatogram of a direct injection of the headspace above concentrated extract of Concord grape essence using OV 101 substrate. Note the size of the methyl anthranilate peak and the absence of a convincing peak for β-damascenone.

Αέρια Χρωματογραφία ΥΦ συμπυκνώματος (x500) του ίδιου σταφυλιού

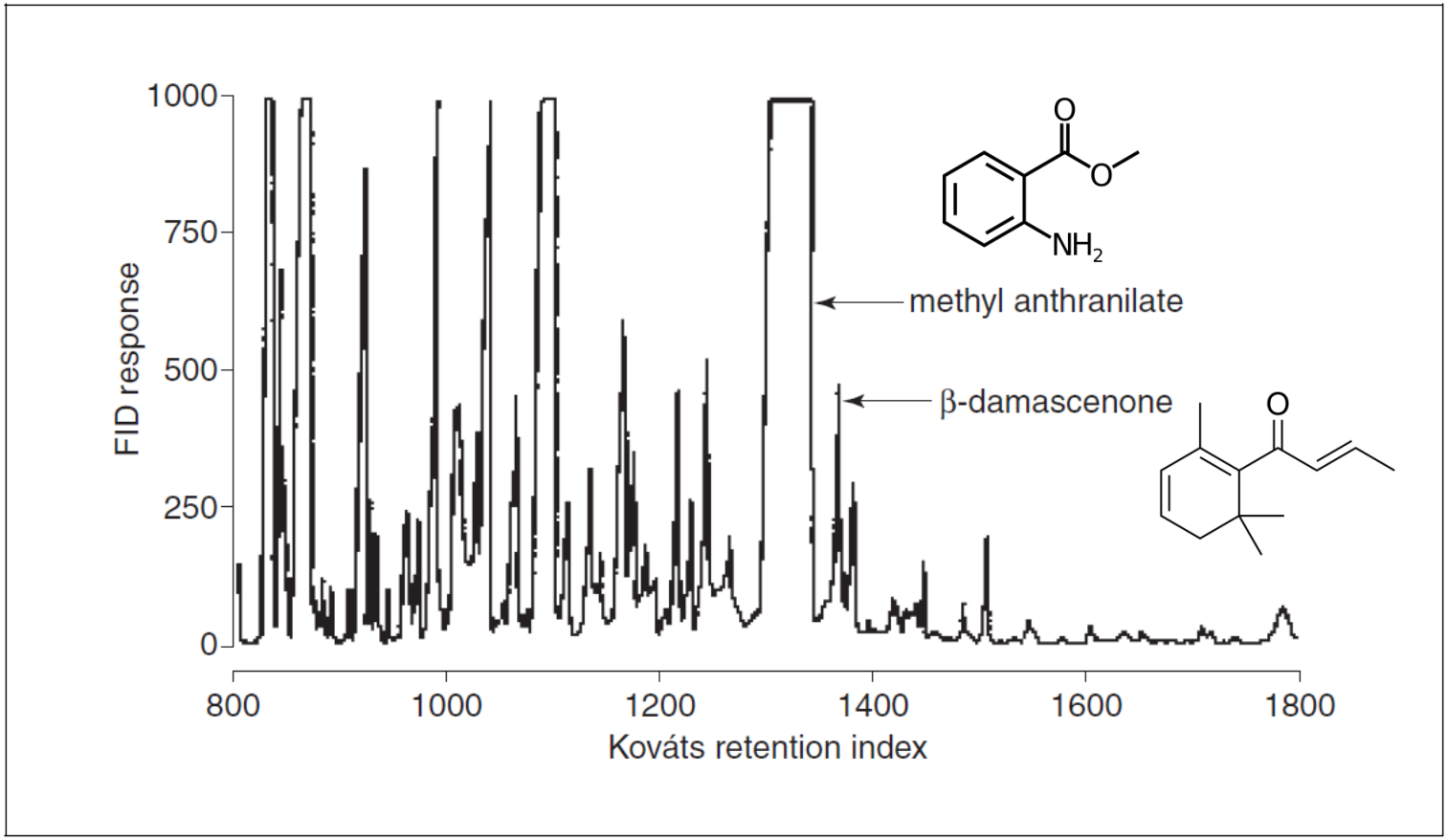


Figure G1.1.4 An FID chromatogram of concentrated extract of the same Concord grape essence shown Figure G1.1.3, drawn to display the data on a linear retention index scale. By simply comparing the index of a peak with the data listed in the flavornet, the odorants that have similar retention indices can be determined. Notice how large the methyl anthranilate peak is, but still no convincing peak for β-damascenone, even though both compounds have the same odor activity (intensity).

Χρόνοι κατακράτησης Kovats στην αέρια χρωματογραφία

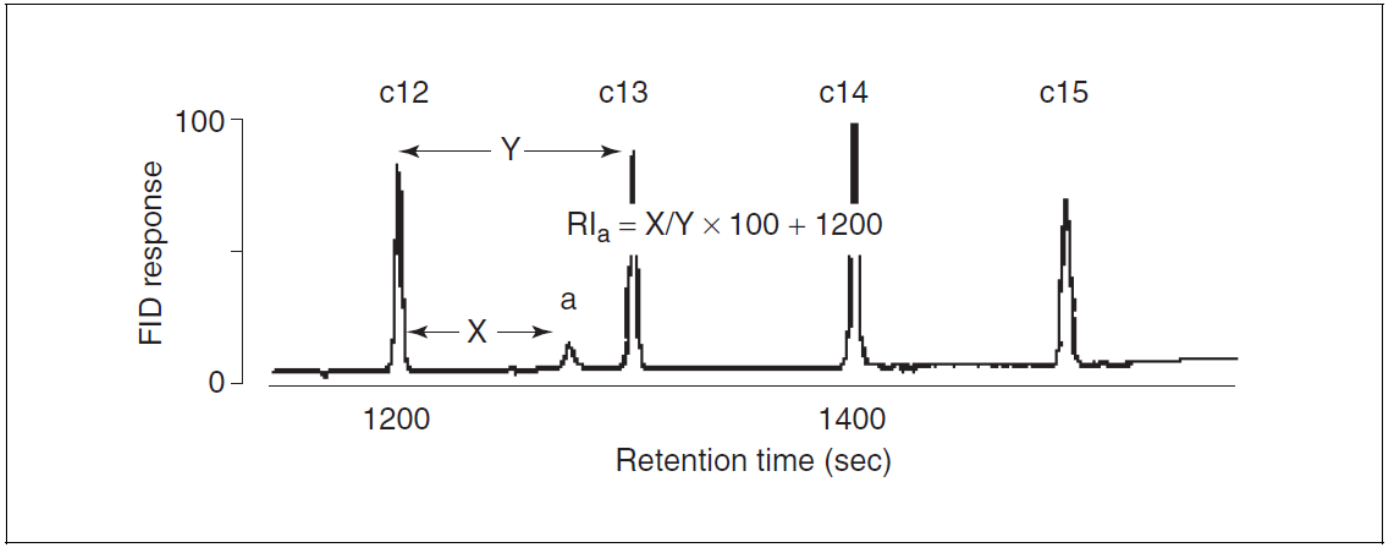


Figure G1.1.2 An FID chromatogram of an indexing standard and parameters used to calculate retention indices. For equations, see Support Protocol, step 3.

$$RI_x = [(T_x - T_n)/(T_{n+1} - T_n) + (n \times 100)$$

Where:

T_x = retention time of a peak (x) to be indexed

T_n = retention time of the paraffin that immediately precedes x

T_{n+1} = retention time of the paraffin that immediately follows x

RI_x = Kováts retention index of x

n = paraffin carbon number of the paraffin that immediately precedes x

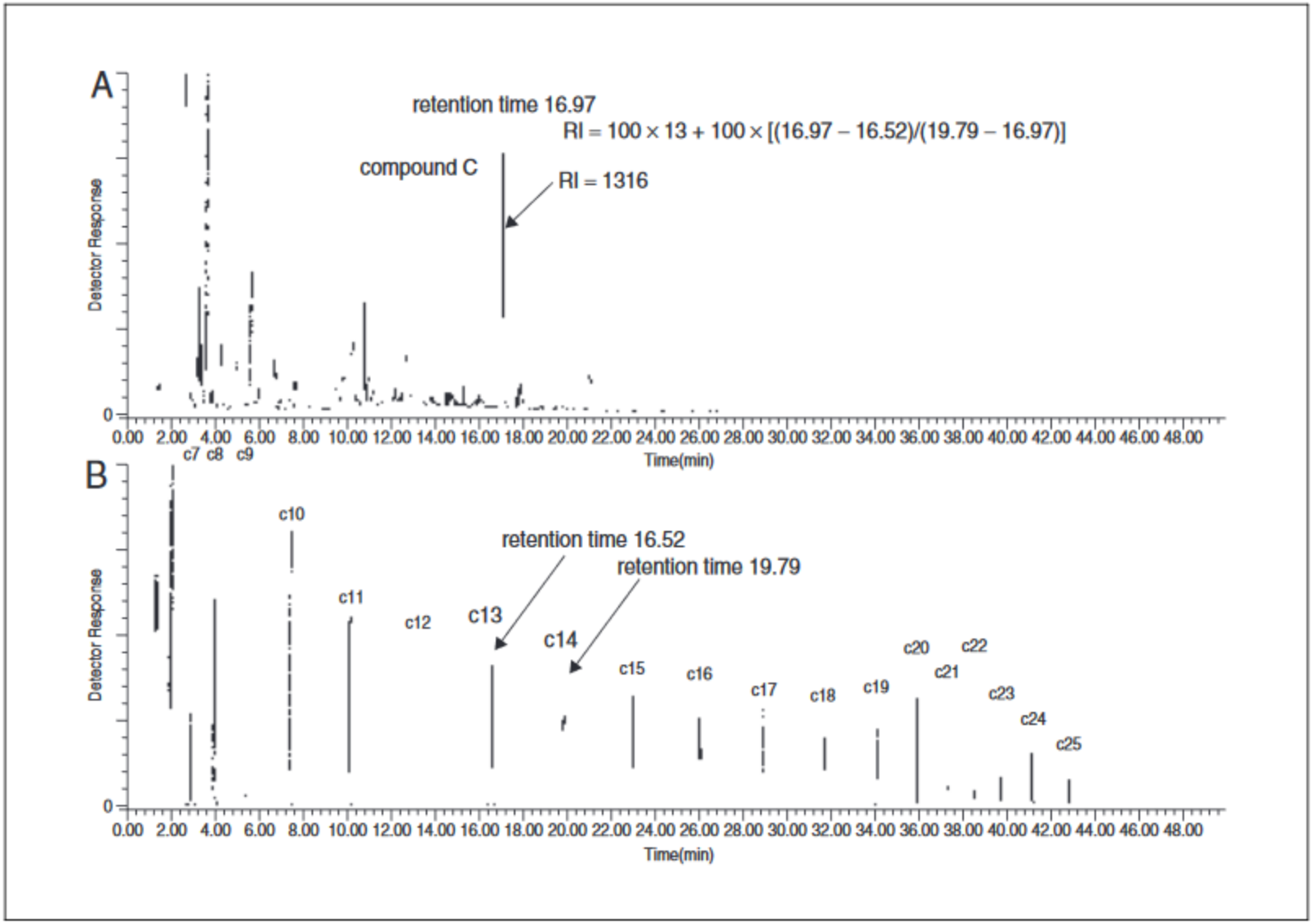
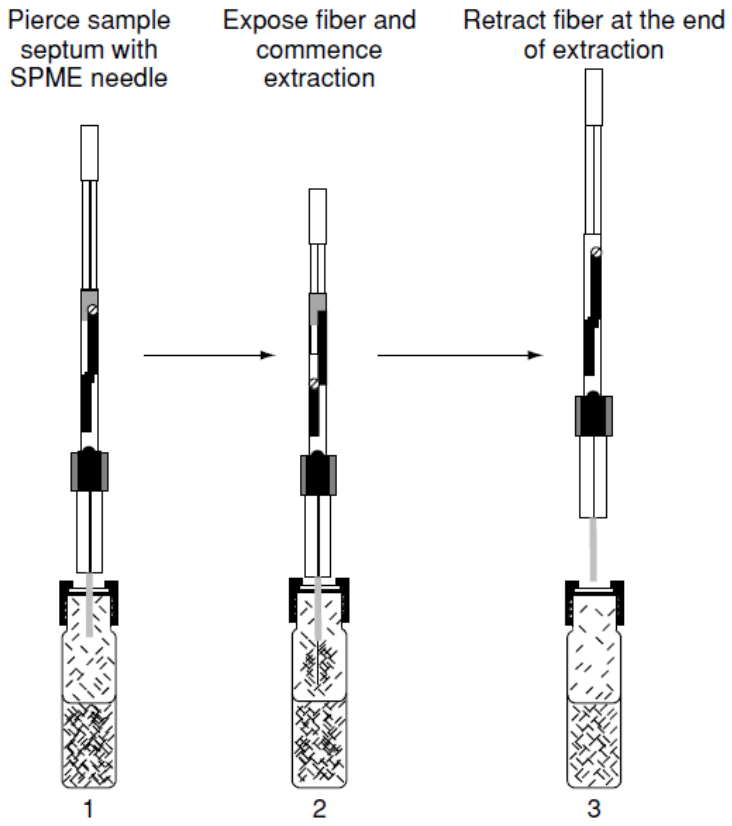


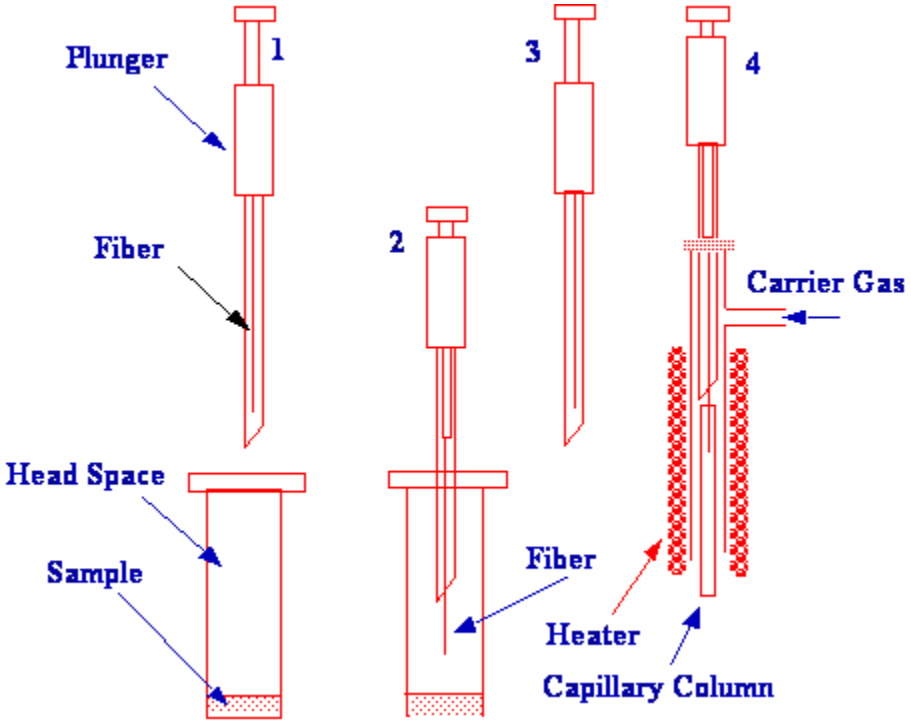
Figure G1.3.1 Determination of the retention index (RI) of an unknown compound C in an aroma extract (**A**) by comparing with a series on *n*-alkanes (**B**) analyzed under the same GC-conditions.

https://www.flavornet.org/index.html

Flavornet Home				Kovats RI	Ethyl Ester RI	Odorants
OV101	DB5	OV17	C20M	Odorant		Odor
[312]	[329]	[420]	677	trimethylamine		fish
418	427	[518]	714	ethanal		pungent, ether
[483]	500	[591]	696	methanethiol		sulfur, gasoline, garlic
[489]	506	[597]	571	propanal		solvent, pungent
500	500	500	500	pentane		alkane
[519]	536	[627]	1037	propanol		alcohol, pungent
527	505	[596]	716	dimethyl sulfide		cabbage, sulfur, gasoline
530	[547]	[591]	848	ethyl formate		pungent
[580]	597	[688]	[945]	methyl ethyl ketone		ether
[592]	609	[700]	[957]	methyl ethyl sulfide		sulfur, garlic
600	600	600	600	hexane		alkane
602	628	[719]	907	ethyl acetate		pineapple
603	[620]	[711]	[968]	methylbutenol		herb
[619]	[636]	[727]	984	pentanone		ether
[630]	647	[738]	1099	isobutanol		wine, solvent, bitter
[631]	648	[739]	1047	trans-crotonaldehyde		flower
[638]	[655]	[746]	1003	methylpentanone		mint
639	641	729	912	methylbutanal		cocoa, almond
641	650	728	910	methylbutanal		malt

Μικροεκχύλιση στερεάς φάσης (Solid Phase Micro-Extraction)





Ενεργός άνθρακας, διάφορες πορώδεις πολυμερικές ρητίνες

Retronasal aroma simulator (RAS)

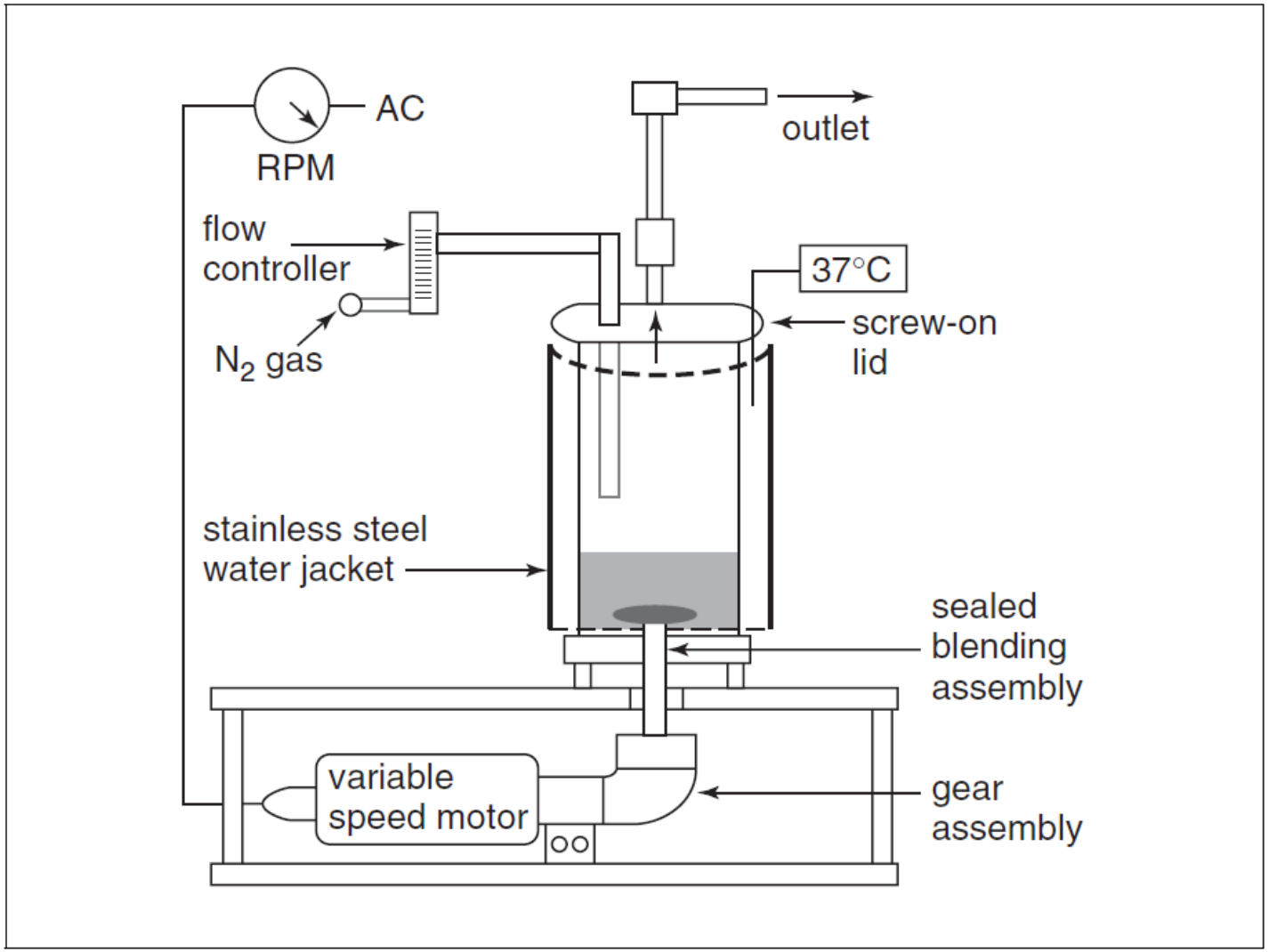


Figure G1.7.2 A diagram of the retronasal aroma simulator (RAS).

Συσκευές προσομοίωσης του στόματος

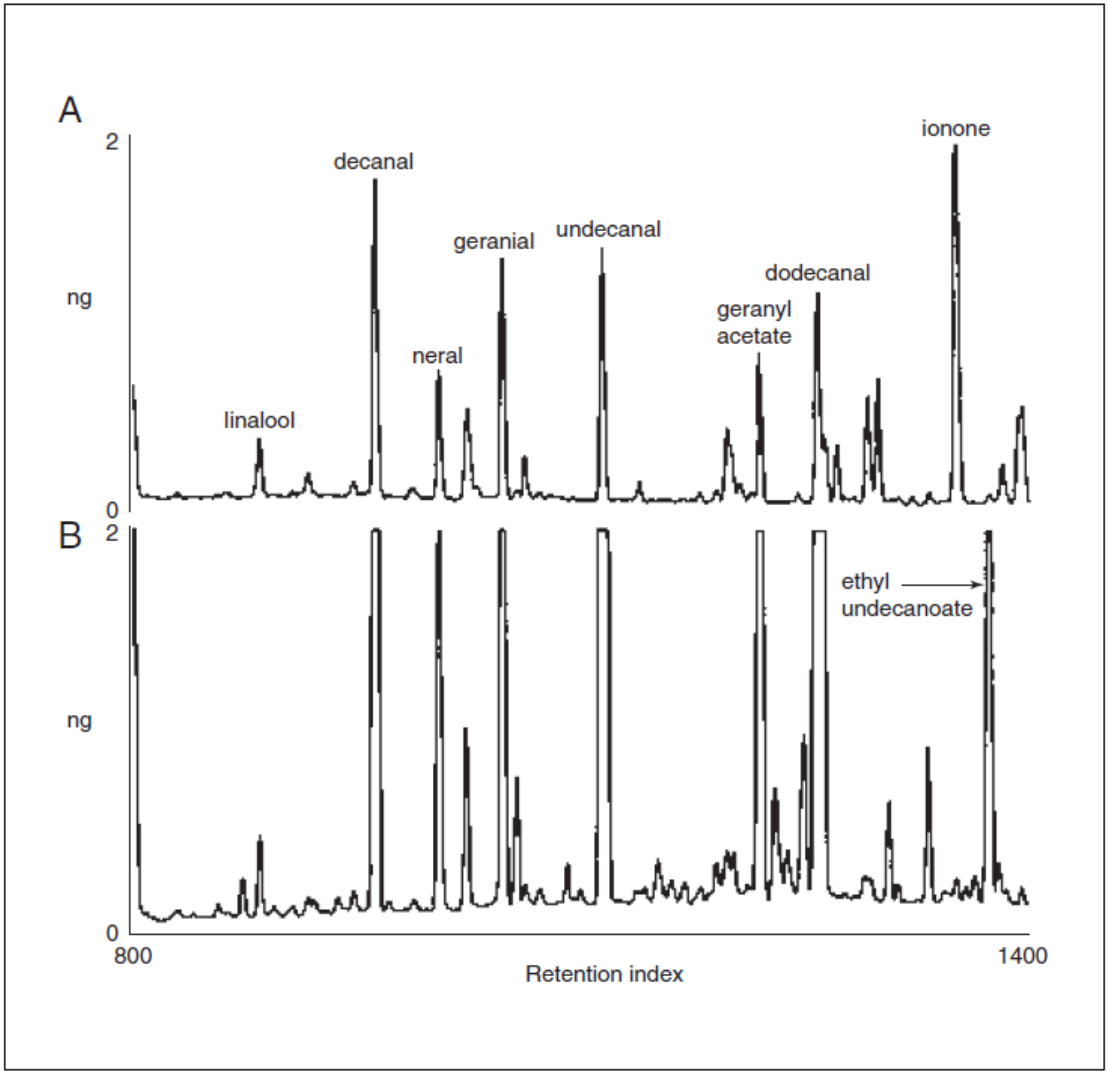


Figure G1.7.1 (**A**) A gas chromatogram of a beverage headspace sampled in the dynamic conditions of an RAS mouth simulator. (**B**) A gas chromatogram of the headspace from the same beverage under static near-equilibrium conditions. Reprinted with permission from Deibler and Acree (2000a). Copyright (2000) American Chemical Society.

Model mouth

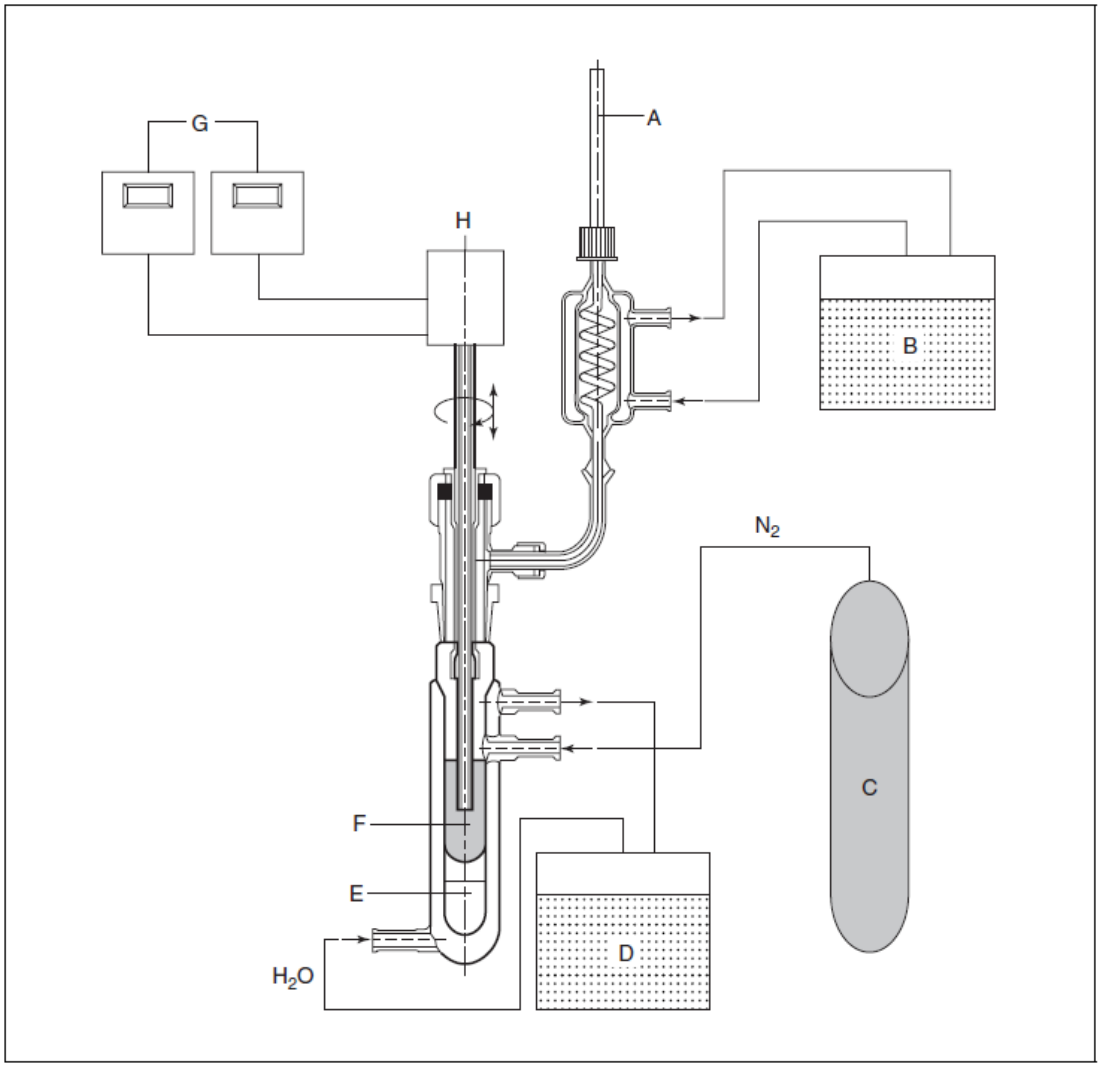


Figure G1.7.3 A diagram of the model mouth. A, trap; B, ethanol bath (–10°C); C, nitrogen gas source; D, water bath (37°C); E, sampling material (in sample flask); F, plunger; G, voltage controllers; H, motors.

Artificial saliva

From van Ruth et al. (1997):
5.208 g $NaHCO_3$
1.369 g $K_2HPO_4 \cdot 3H_2O$
0.877 g NaCl
0.477 g KCl
0.441 g $CaCl_2 \cdot 2H_2O$
0.5 g NaN_3
2.160 g mucin (porcine stomach mucin; Sigma-Aldrich)
200,000 U α-amylase (hog pancreas α-amylase; Sigma-Aldrich)
Bring to 1 liter with distilled water
Adjust to pH 7

Model mouth

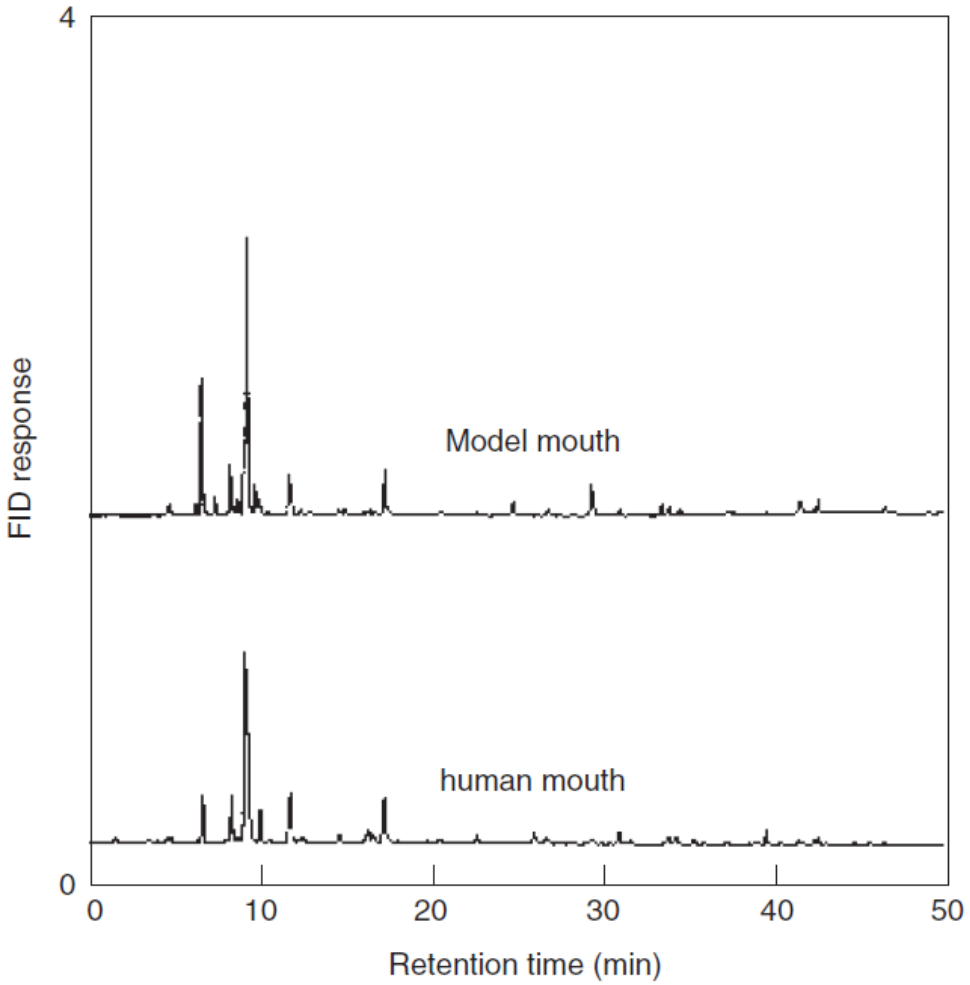


Figure G1.7.4 A model mouth and human mouth comparison shows a gas chromatogram of volatile compounds released from rehydrated French beans in the model mouth (n = 6; upper chromatogram) and in the mouth of assessors (n = 12, lower chromatogram).

Αέρια Χρωματογραφία - Οσφρομετρία

Ανιχνευτής : Ανθρώπινη μύτη

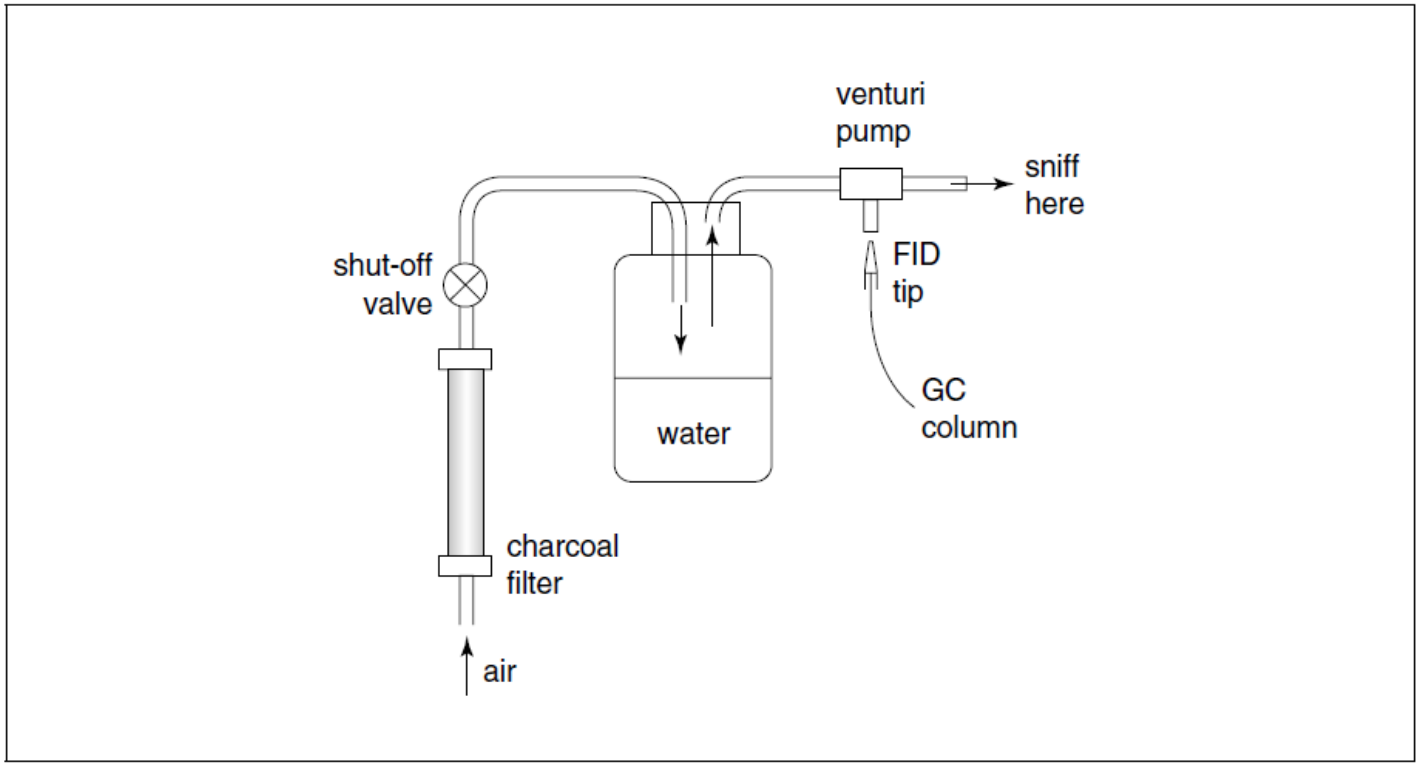


Figure G1.8.1 Diagram of the sniff port constructed from a laboratory filter (based on Acree et al., 1976; see Acree, 1997) showing the filter pump (with the check ball removed) attached to a humidifier, shut-off valve, and charcoal filter. The vacuum side of the pump is positioned over a flame ionization detector (FID) with the hydrogen gas turned off. The make-up gas helps lift the narrow (<0.2-mm-o.d.) gas chromatography (GC) effluent stream into the much larger olfactometry air stream without loss of resolution, and the 300 ml/min air combustion gas produced by the FID also prevents loss of resolution.

Table G1.8.1 Single Sniff Run: Start and Stop Times of Odors Detected by GC/O[a]

Start RI	Stop RI	Odor
797	800	Sweet
800	804	Sweat
831	837	Fruity
837	839	Green
839	846	Burnt sugar
882	886	Fruity
904	910	Cat urine
948	951	Minty/fruity
952	956	Mushroom
1021	1036	Cotton candy
1037	1052	Burnt sugar
1052	1072	Caramel
1072	1079	Floral
1079	1095	Fresh
1115	1123	Meat
1130	1136	Skunky
1142	1149	Sweat
1187	1195	Sweet
1216	1233	Foxy
1239	1258	Skunky
1270	1282	Foxy
1284	1307	Vanilla
1331	1347	Apple
1348	1361	Skunky/plastic
1393	1403	Plastic
1403	1413	Plastic
1419	1428	Plastic
1428	1440	Sweet
1482	1491	Cherry

[a]Data from GC/O run shown in Figure G1.8.4 (Niagara grapes; OV-101 index). The odor descriptors were the most frequent descriptors used by the sniffer over several GC/O runs of the same sample. RI, retention index.

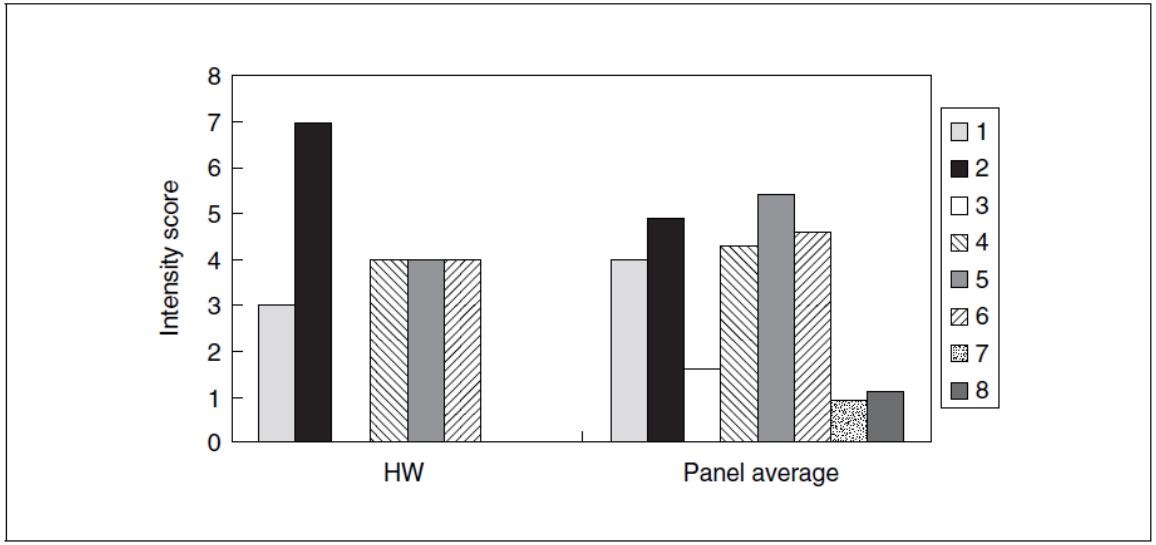


Figure G1.8.2 Time intensity chromatogram (see Basic Protocol 3), showing intensity scores of eight volatile compounds in a reference mix for a single sniffer (HW) and for the panel average (n = 8). Notice that sniffer HW showed no response to compounds 3, 7, and 8. This kind of specific anosmia is not uncommon, requiring the use of multiple sniffers or testing to eliminate anosmics.

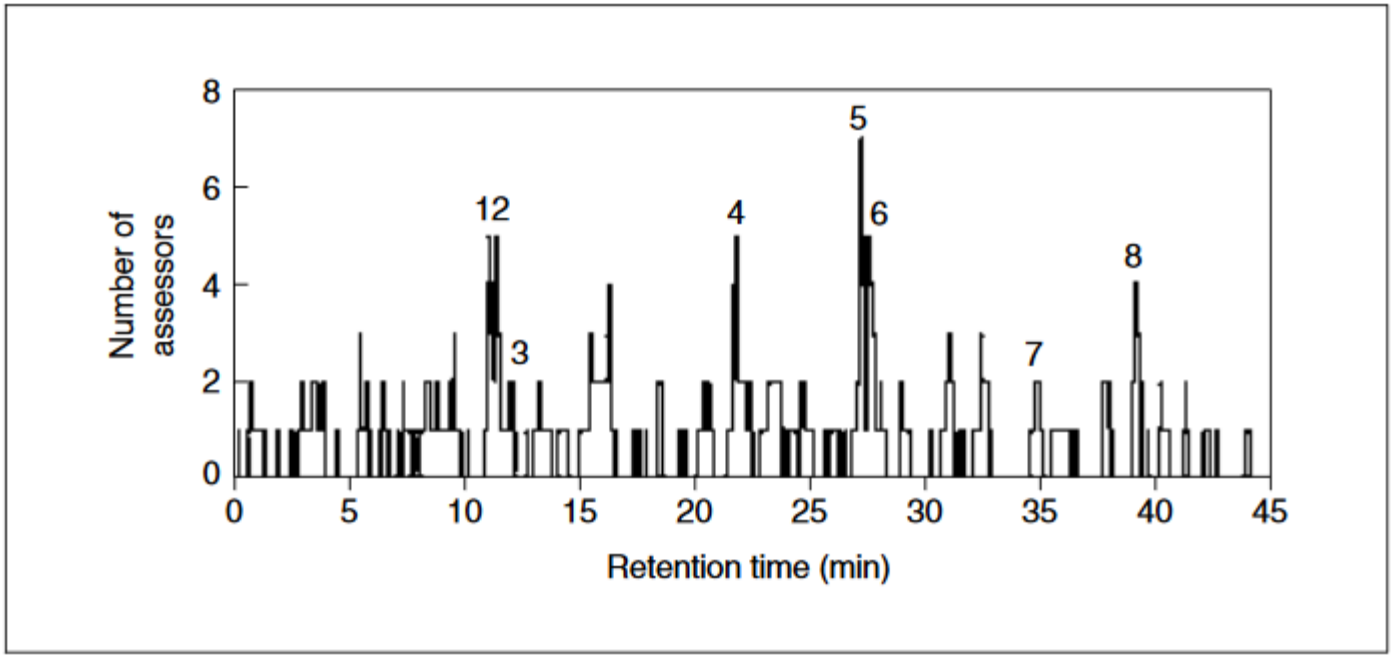


Figure G1.8.3 Sniffing chromatogram of eight volatile compounds in a reference mix obtained by the detection frequency method using eight assessors (see Alternate Protocol 1). Compounds: 1, 100 ng 2-butanone; 2, 20 ng diacetyl; 3, 500 ng ethyl acetate; 4, 100 ng 3-methyl-1-butanol; 5, 20 ng ethyl butyrate; 6, 100 ng hexanal; 7, 100 ng 2-heptanone; 8, 500 ng α-pinene.

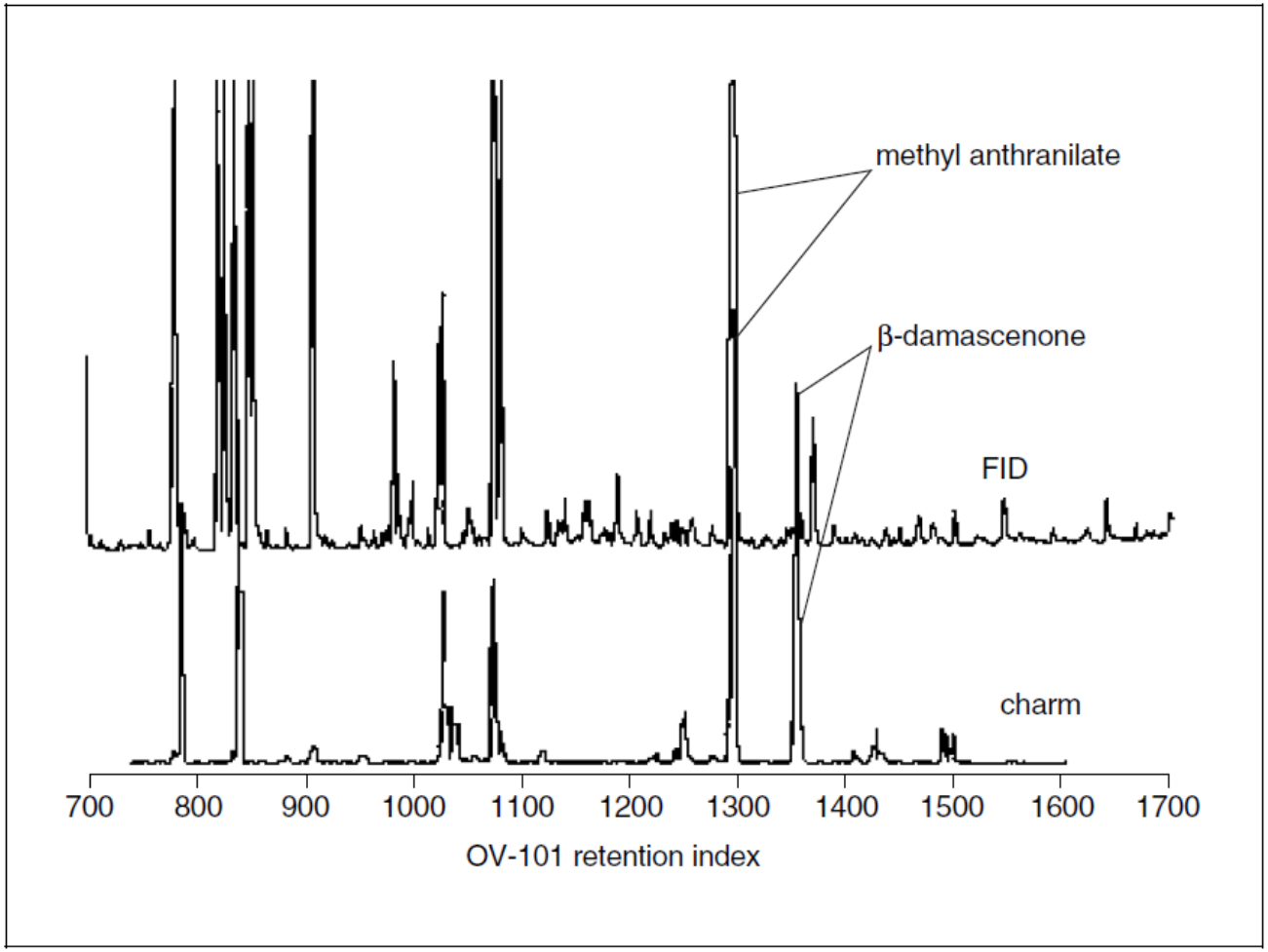


Figure G1.8.4 An FID chromatogram of concentrated extract of Niagara grape juice drawn to display the data on a linear retention index scale where the *y* axis is flame ionization response (upper trace). Below it is the charm chromatogram, where the *y* axis is dilution value. By simply comparing the index of a peak with the data listed in the Flavornet (see Internet Resource), it is possible to determine which odorants have similar retention indices. Notice how large the methyl anthranilate peak is, whereas there is no convincing peak for β-damascenone in the FID chromatogram, even though both compounds have the same potency in the charm chromatogram.

Characterization of Food: Emerging Methods
A.G. Gaonkar (Editor)

Chapter 17

New Developments in Methods for Analysis of Volatile Flavor Compounds and their Precursors

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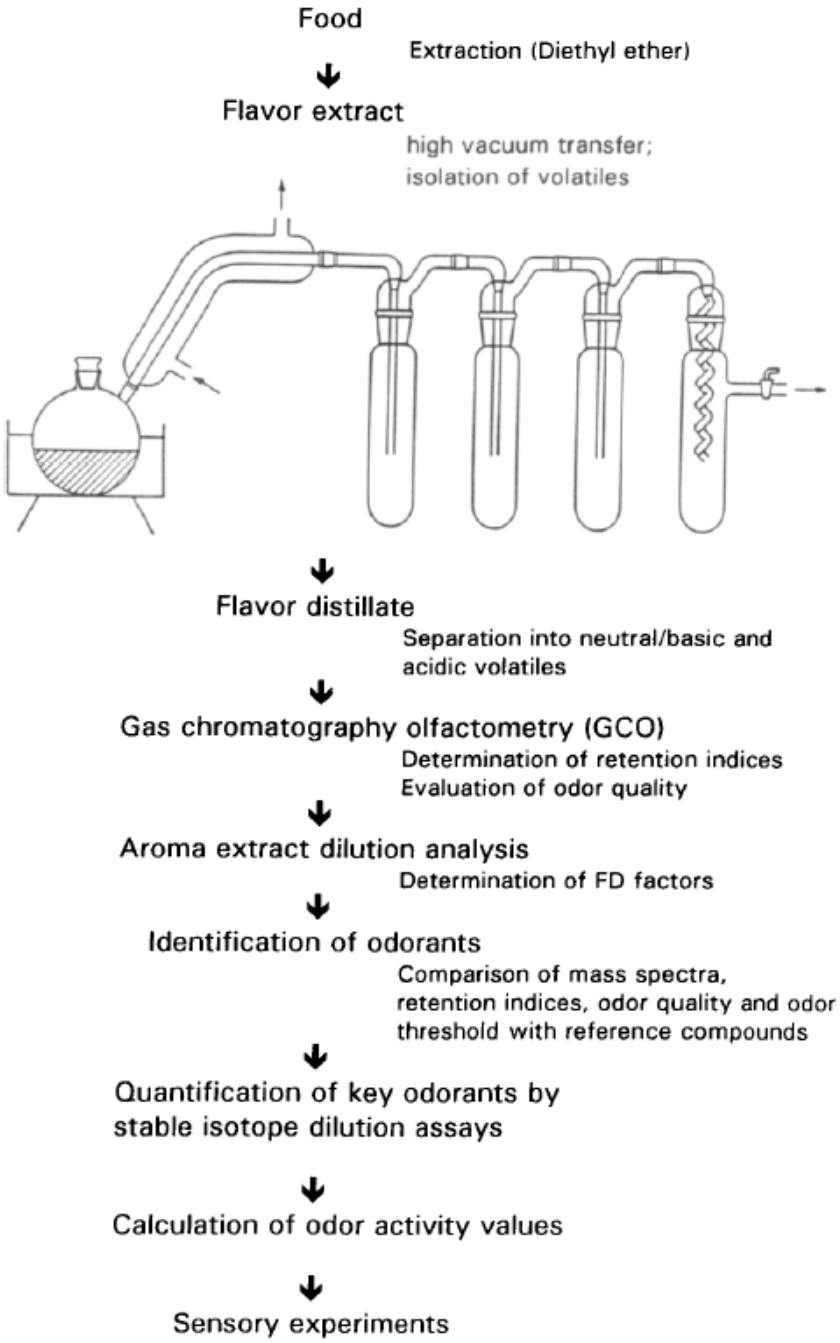


Figure 1. Experimental design for the isolation and characterization of key odorants in foods

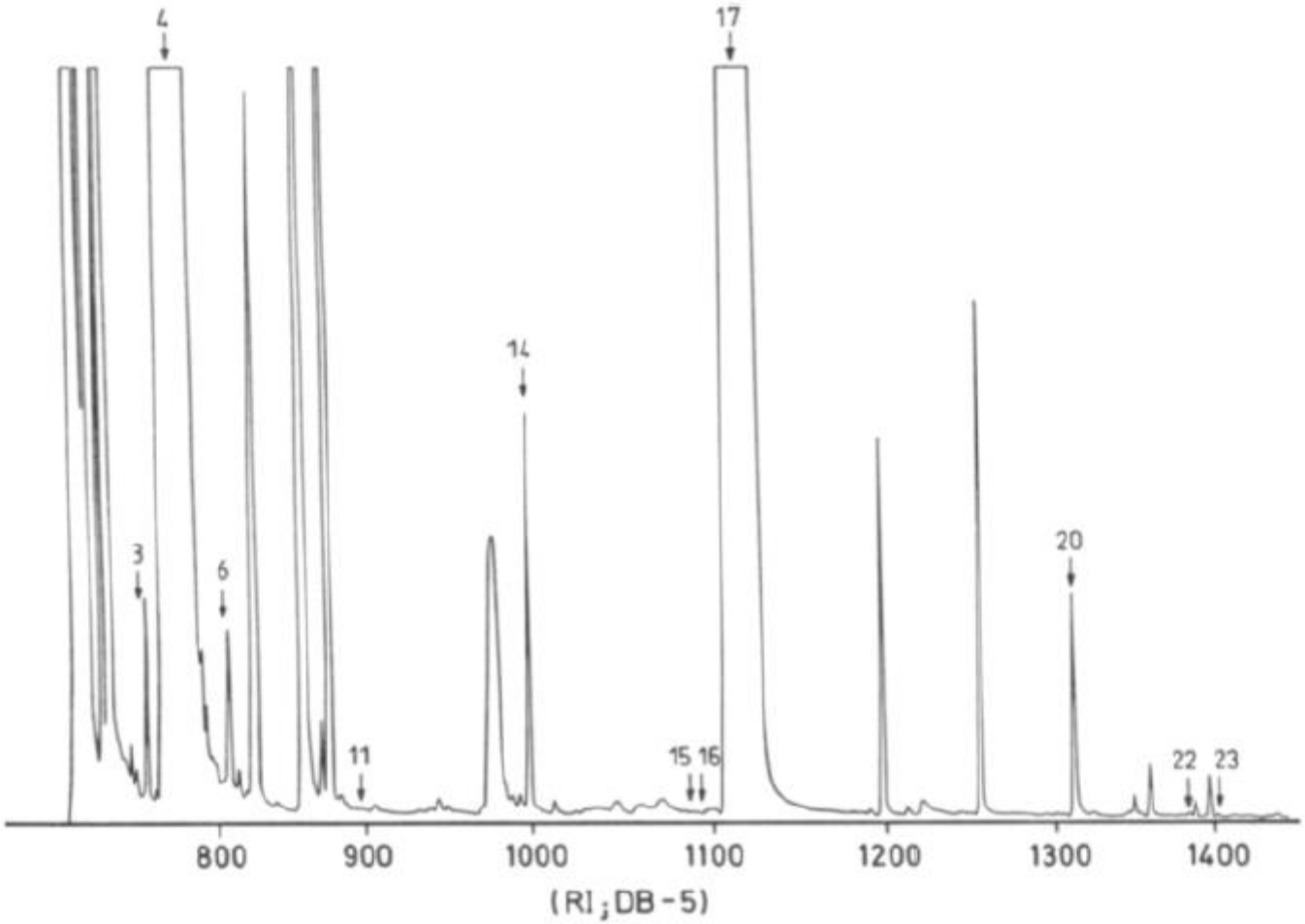


Figure 2. Gas chromatogram of the neutral/basic volatile fraction isolated from a pale lager beer. Numbers characterize an odor-active position.

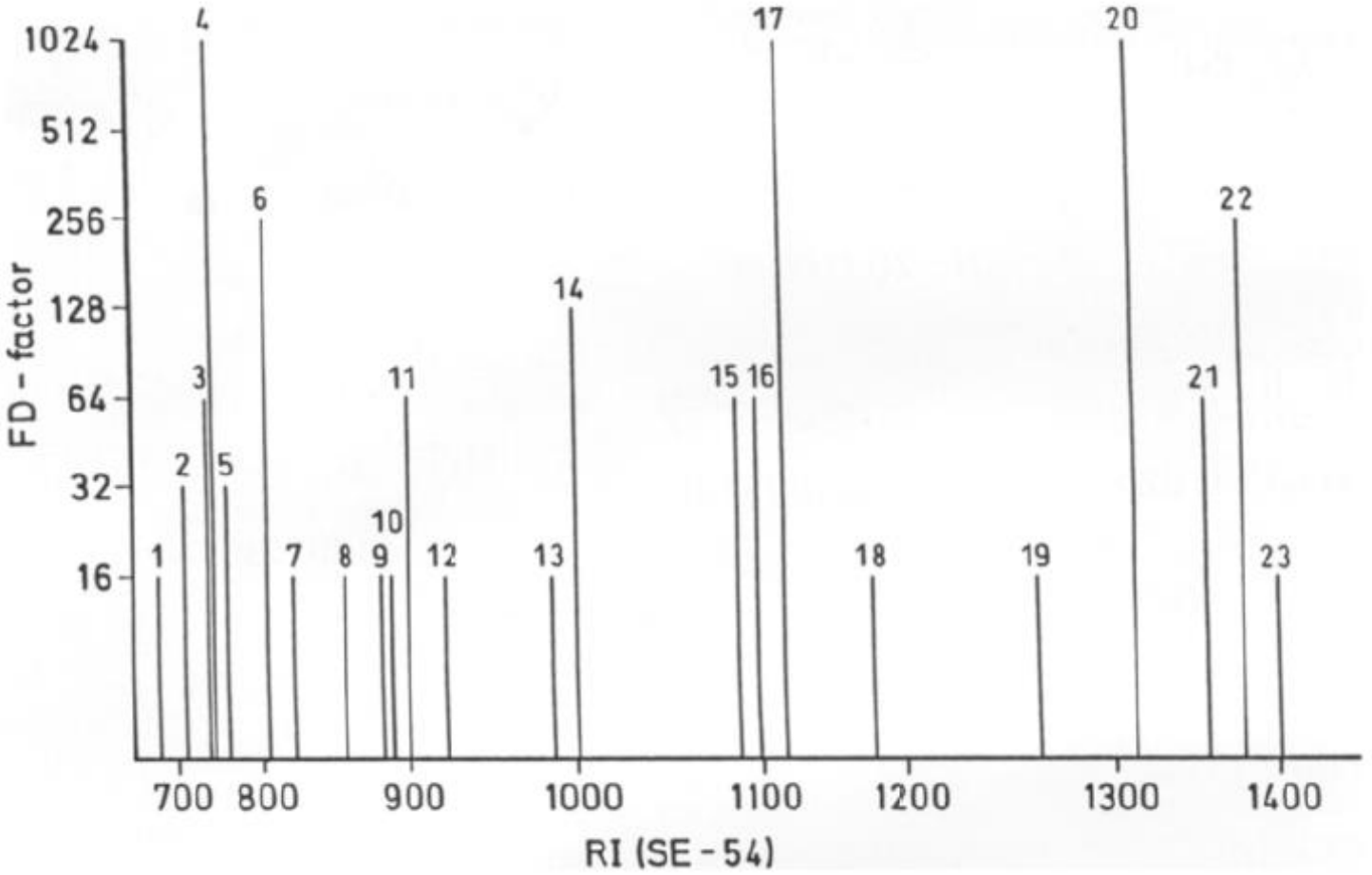


Figure 3. Flavor dilution (FD) chromatogram of the neutral/basic odorants in a pale lager beer. The numbering follows Figure 2. RI; retention index on a silicone SE-54 GC stationary phase [adapted from ref. 18].

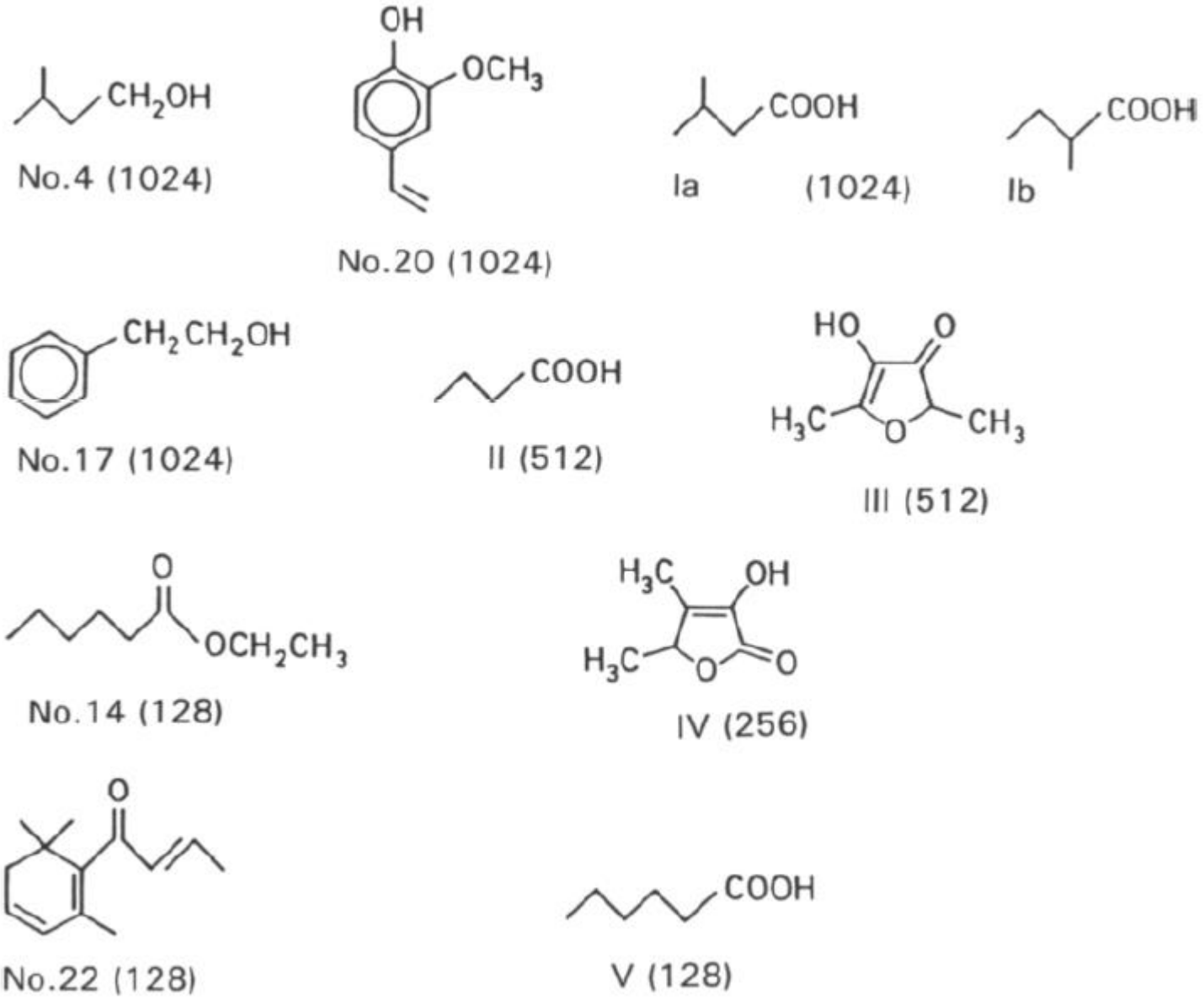


Figure 4. Key odorants in pale lager beer. The numbering follows Figs. 2 and 3. Odorants with roman numbers were identified in the acidic fraction (FD-factor).

3.2. Quantification of odorants

A first step to approach the situation in the food is a calculation of odor activity values (OAV)

$$OAV_x = \frac{\text{concentration}_x}{\text{odor threshold}_x}$$

Table 9
Concentrations (µg/kg) and odor activity values (OAV) of selected key odorants in roasted (180°C, 30 min) black and white sesame seeds [46, 52]

Odorant	Odor threshold[a] (µg/kg oil)	Black		White seeds	
		Conc.	OAV	Conc.	OAV
(E,E)-2,4-Decadienal	180	1103	6	212	1
2-Methoxyphenol	19	2652	139	4974	262
2-Pentylpyridine	5	904	181	255	51
2-Furfurylthiol	0.4	673	1682	2461	6152
2-Ethyl-3,5-dimethylpyrazine	3	394	131	238	79
4-Hydroxy-2,5-dimethyl-3(2H)-furanone	50	11685	234	9155	183
2-Acetyl-1-pyrroline	0.1	n.a.	n.d.	12	120
2-Phenylethylthiol	0.05	12	240	44	880

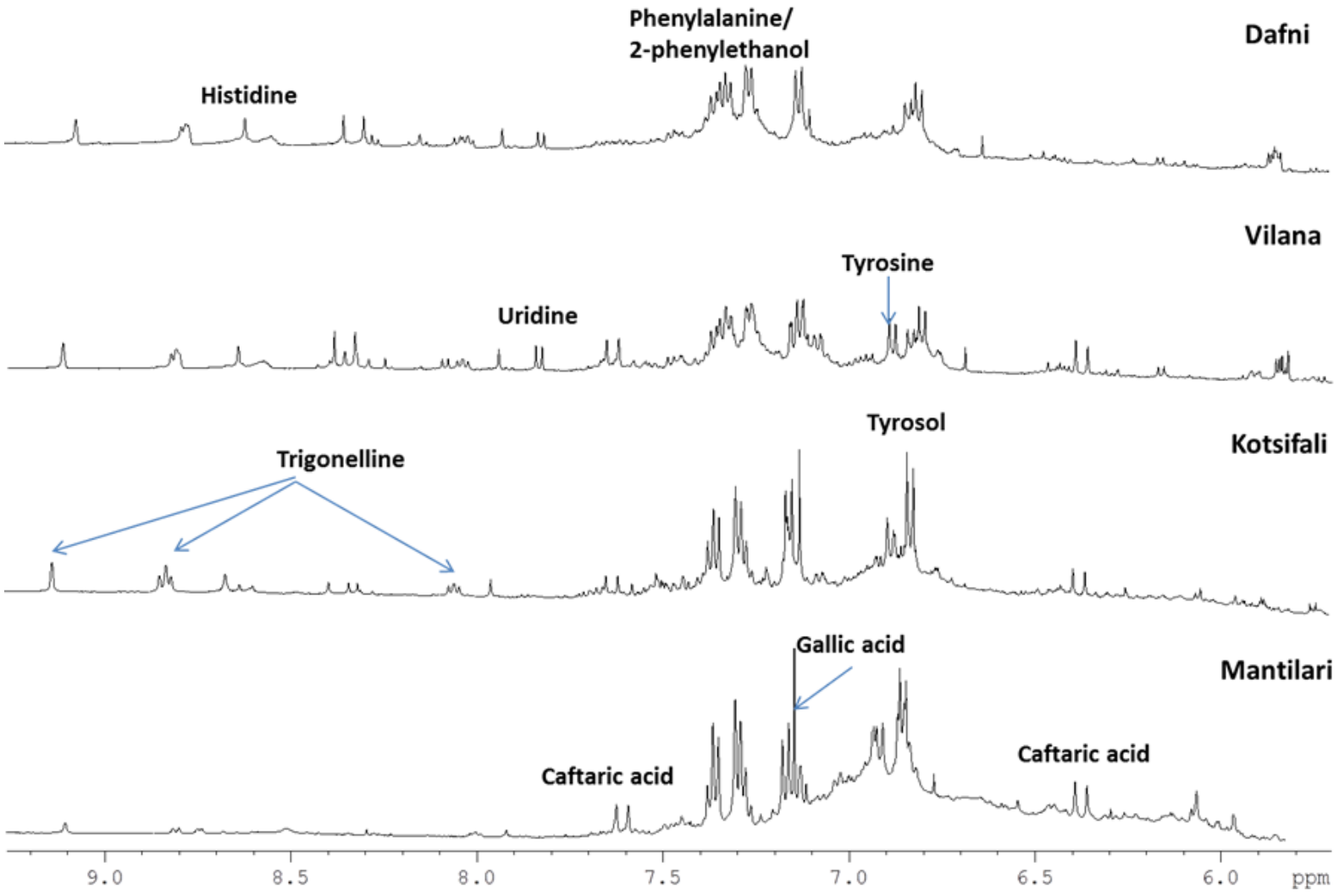
[a] Odor thresholds were determined in sunflower oil.

Wine aging and NMR metabolomics

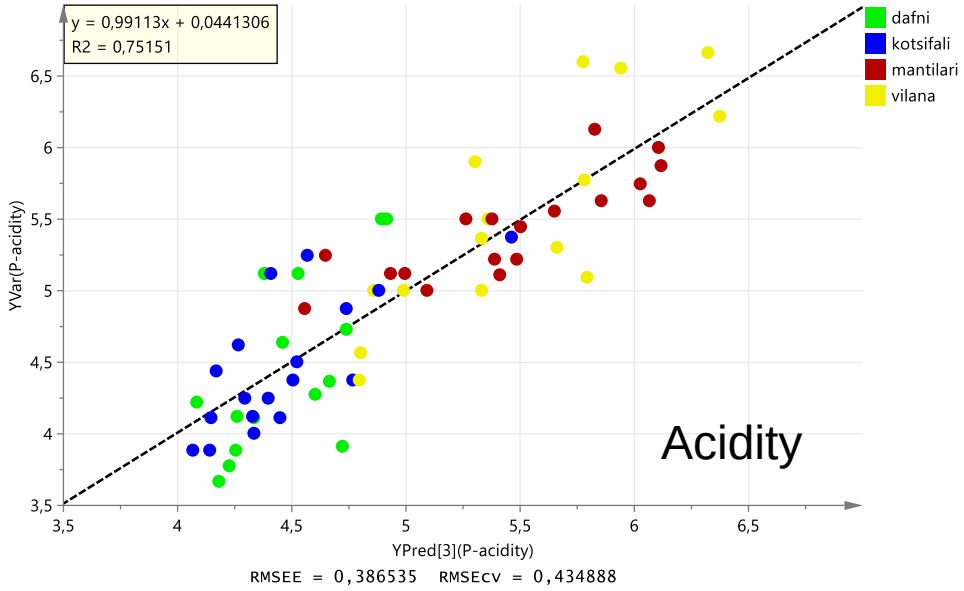
Samples & procedures

- Two white (Vilana, Dafni), two red (Mantilari, Kotsifali) Cretan cultivars
- 4-5 different barrel types (american and french oak, acacia, chestnut, chips etc.)
- Maturation in barrels for 12 months (3-month sampling)
- Wine-in-bottle aging for 12 months
- NMR, sensory panel testing and classical wine chemical analysis

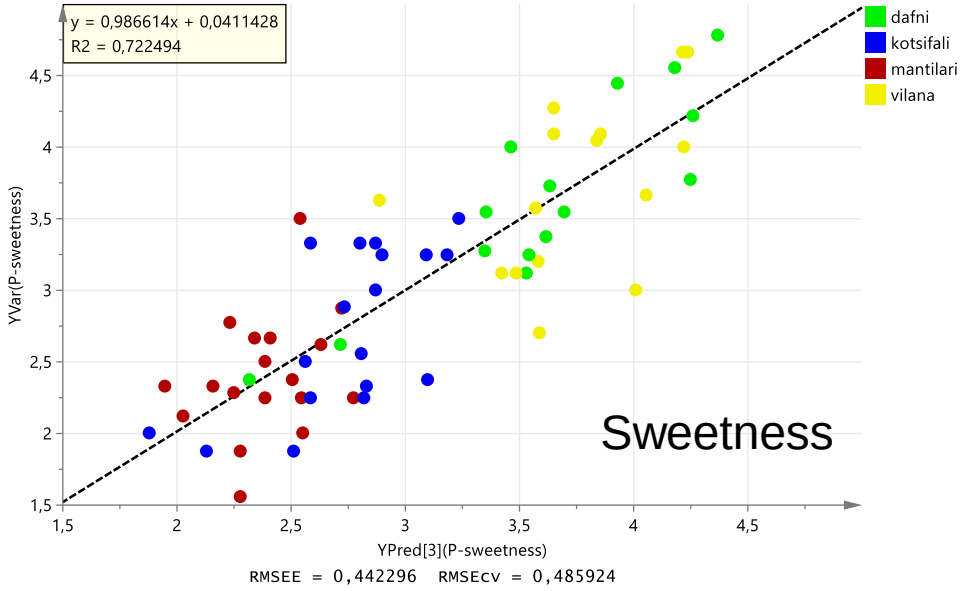
^{1}H NMR spectra of wines, phenolic region



NMR metabolome and sensory panel testing

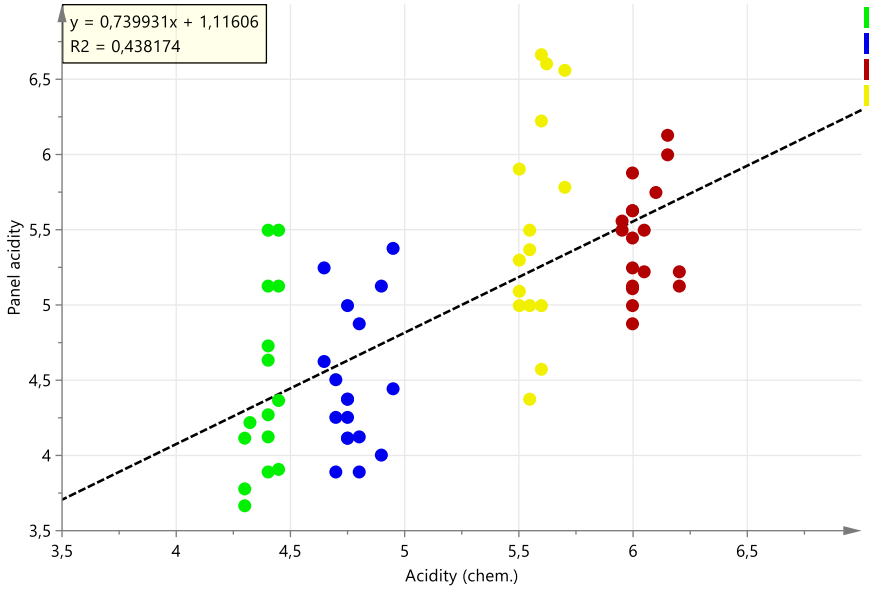


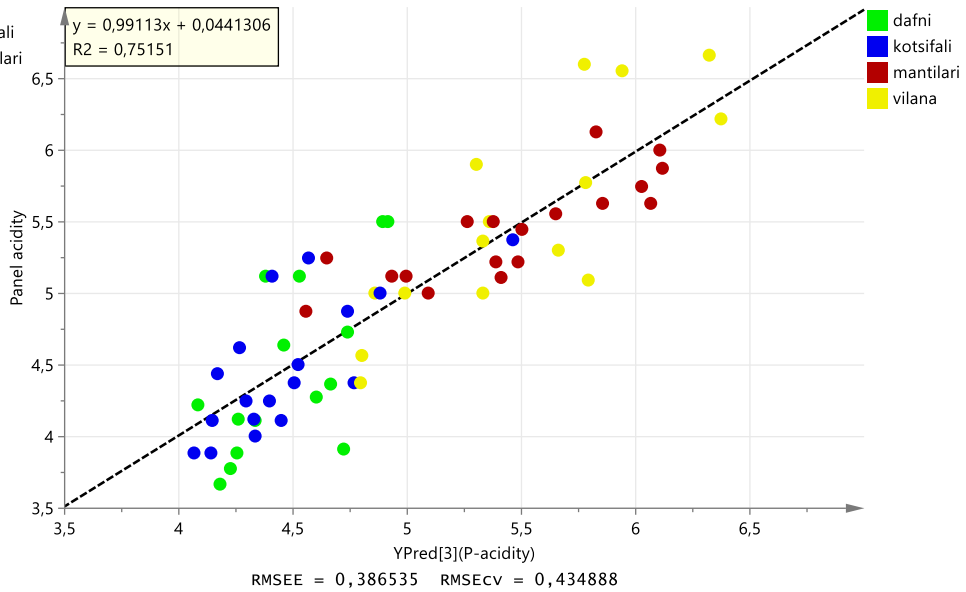
Acetaldehyde
Malic acid
Fumaric acid



α, β-glucose
Trehalose
2,3-BuOH

Titrated acidity and sensory panel testing





Sensory panel acidity correlates much better with NMR metabolites than chemically measured acidity