Confirmatory methods

Provide full or complementary information enabling the substance to be unequivocally identified and if necessary quantified at the level of interest.

They shall provide information on the chemical structure of the analyte. Methods based only on chromatographic analysis without the use of mass spectrometric detection are not suitable on their own for use as confirmatory methods (suitable combinations of clean-up, chromatographic separation(s) and spectrometric detection)

GC in food analysis:

1. Detection of Volatile Compounds
GC is highly effective in analyzing volatile and semi-volatile compounds, making it ideal for examining flavors, fragrances, and aroma compounds in food. These are critical in assessing food quality, authenticity, and consumer preference.

2. Residue Analysis (Pesticides and Contaminants)
Many pesticides, toxins, and chemical contaminants are volatile or semi-volatile, making GC suitable for identifying and quantifying these residues in food products. Regulatory agencies set strict limits on such contaminants, and GC can measure these at very low concentrations.

3. Fats and Oils Composition GC is used to analyze the fatty acid profile of oils and fats in foods. By using derivatization techniques, GC can separate and identify fatty acid methyl esters (FAMEs), helping in nutritional labeling, adulteration detection, and quality control of oils and fats.

4. Flavor and Aroma Profiling The sensory quality of food relies heavily on flavor and aroma compounds, which are often volatile. GC, sometimes coupled with mass spectrometry (GC-MS), allows for a detailed profile of aroma compounds, aiding food manufacturers in developing consistent flavor profiles and identifying off-flavors.

5. Food Adulteration Detection GC can identify substances that are not naturally present in food or are added in excess, indicating possible adulteration. Examples include added sugars, synthetic flavors, or foreign oils.

6. Nutritional Analysis GC helps in determining the nutritional composition of food, particularly in profiling fatty acids, amino acids (after derivatization), and other small molecules.

7. Monitoring Food Safety and Shelf Life GC helps monitor chemical changes during storage, such as oxidation of fats, which can impact food safety and shelf life. By detecting breakdown products, it helps assess freshness and quality.

Scheme of a gas chromatograph (GC)

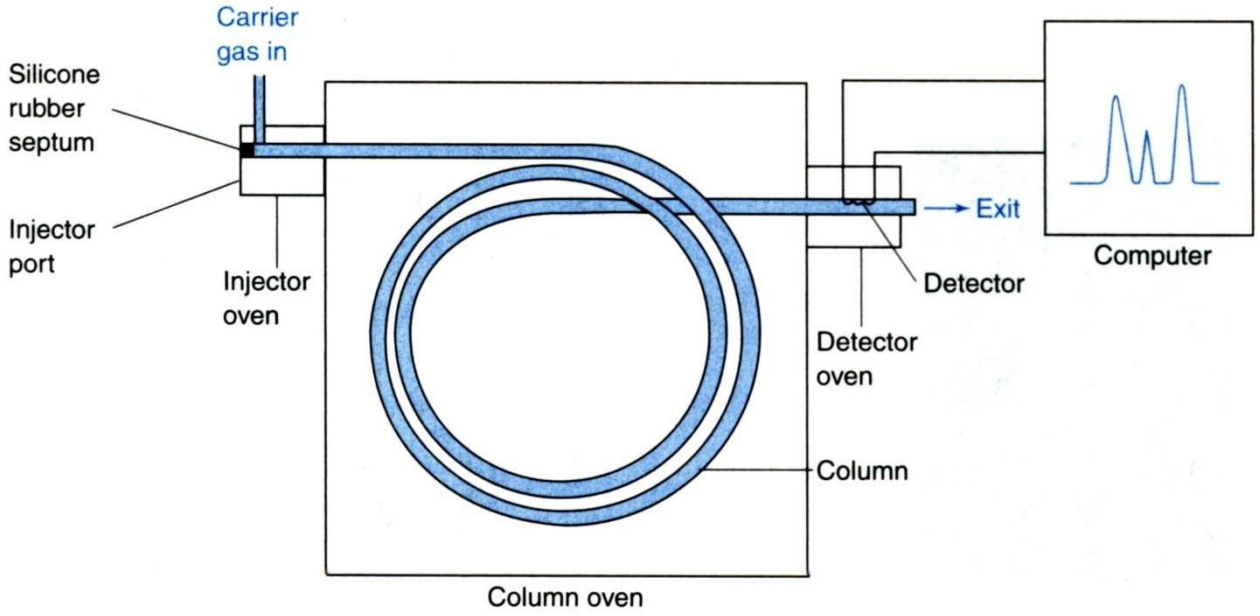
- Carrier gas (high purity, unreactive, cheap): **N_2, He, H_2**
- Flow control
  - constant, reproducible flow rate
- Injection port (sample inlet, microlitre syringe)
- Oven — thermostated at constant T or linear rate
- Column
- Detector
- Data processing
  - retention time (volume)
  - peak area
  - recorder, integrator, microprocessor, computer, etc

Gas Chromatography

Introduction

Instrumentation

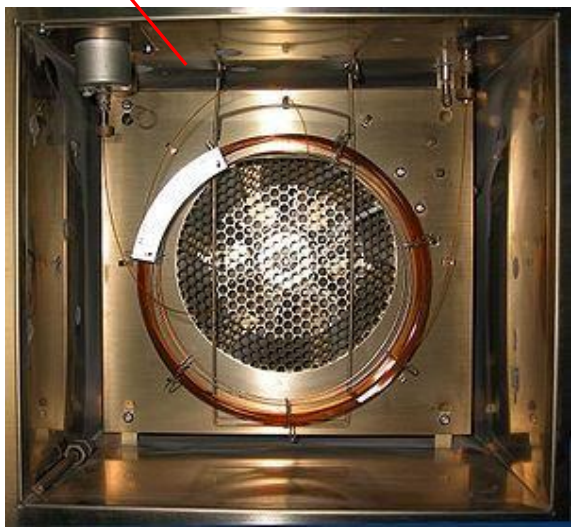
- Process:
  - **Volatile liquid or gas injected through septum into heated port**
  - **Sample rapidly evaporates and is pulled through the column with carrier gas**
  - **Column is heated to provide sufficient vapor pressure to elute analytes**
  - **Separated analytes flow through a heated detector for observation**



GC Columns

Packed columns

- Typically a glass or stainless steel coil.
- 1-5 m total length and 5 mm inner diameter.
- Filled with the st. ph. or a packing coated with the st.ph.



Capillary columns

- Thin fused-silica.
- Typically 10-100 m in length and 250 μm inner diameter.
- Stationary phase coated on the inner surface.
- Provide much higher separation efficiency
- But more easily overloaded by too much sample.

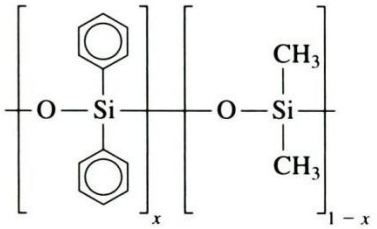
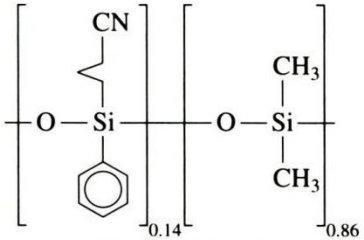
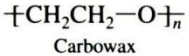
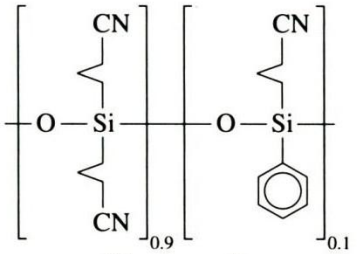
Gas Chromatography

Instrumentation

Choice of liquid stationary phase:

- Based on "like dissolves like"
- Nonpolar columns for nonpolar solutes
- Strongly polar columns for strongly polar compounds
- To reduce "bleeding" of stationary phase:
  - **bond (covalently attached) to silica**
  - **Covalently cross-linked (unbound)**

Table 24-1 Common stationary phases in capillary gas chromatography

Structure	Polarity	Temperature range (°C)
 (Diphenyl)$_x$(dimethyl)$_{1-x}$ polysiloxane	$x = 0$ Nonpolar $x = 0.05$ Nonpolar $x = 0.35$ Intermediate polarity $x = 0.65$ Intermediate polarity	−60°–320° −60°–320° 0°–300° 50°–370°
 (Cyanopropylphenyl)$_{0.14}$ (dimethyl)$_{0.86}$ polysiloxane	Intermediate polarity	−20°–280°
 Carbowax (poly(ethylene glycol))	Strongly polar	40°–250°
 (Biscyanopropyl)$_{0.9}$ (cyanopropylphenyl)$_{0.1}$ polysiloxane	Strongly polar	0°–275°

Gas Chromatography

Retention Index

Retention Time

- Order of elution is mainly determined by *volatility*
  - **Least volatile = most retained**
  - **Polar compounds (ex: alcohols) are the least volatile and will be the most retained on the GC system**

Table 24-2 Polarity of solutes

Nonpolar	Weak intermediate polarity
Saturated hydrocarbons	Ethers
Olefinic hydrocarbons	Ketones
Aromatic hydrocarbons	Aldehydes
Halocarbons	Esters
Mercaptans	Tertiary amines
Sulfides	Nitro compounds (without α-H atoms)
CS_2	Nitriles (without α-atoms)
Strong intermediate polarity	**Strongly polar**
Alcohols	Polyhydroxyalcohols
Carboxylic acids	Amino alcohols
Phenols	Hydroxy acids
Primary and secondary amines	Polyprotic acids
Oximes	Polyphenols
Nitro compounds (with α-H atoms)	
Nitriles (with α-H atoms)	

- Second factor is similarity in polarity between compound and stationary phase

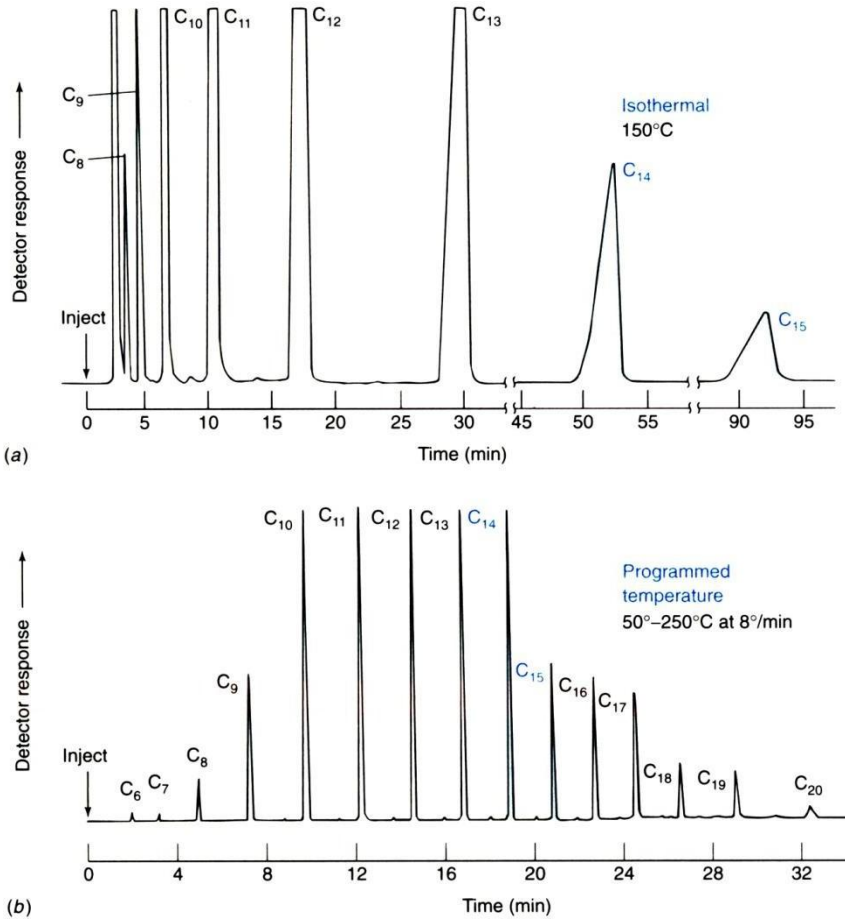
Gas Chromatography

Temperature and Pressure Programming

Improving Column Efficiency

- Temperature programming:
  - **Temperature is raised during the separation (gradient)**
  - ***increases* solute vapor pressure and *decrease* retention time**

Temperature gradient improves resolution while also decreasing retention time



Gas Chromatography

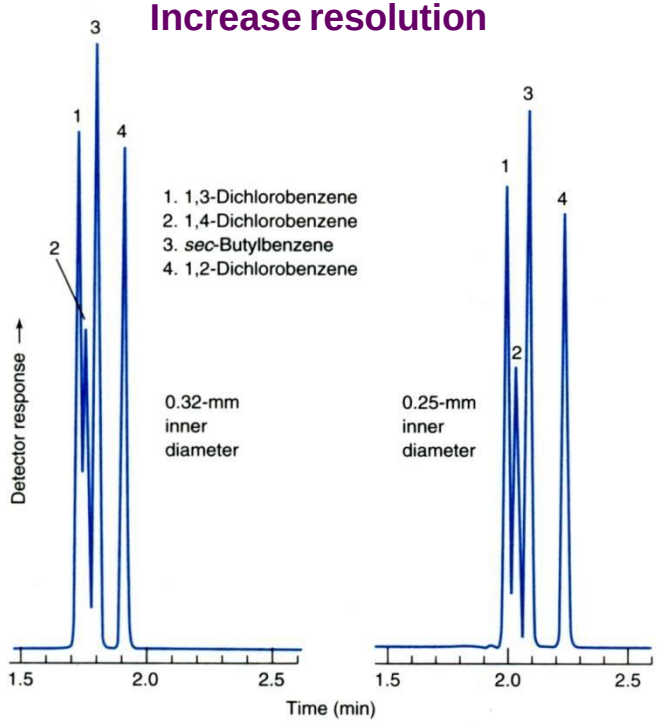
Instrumentation

Open Tubular Columns

- Increasing Resolution

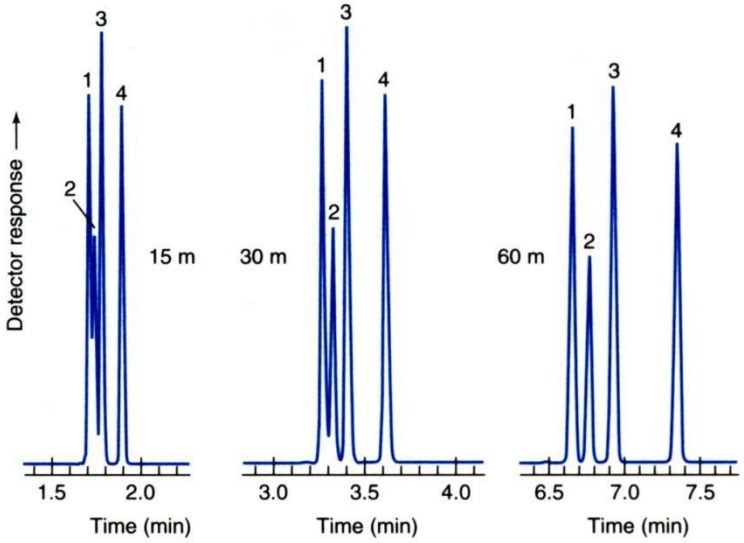
Decrease tube diameter

Increase resolution



Increase Column Length

Increase resolution



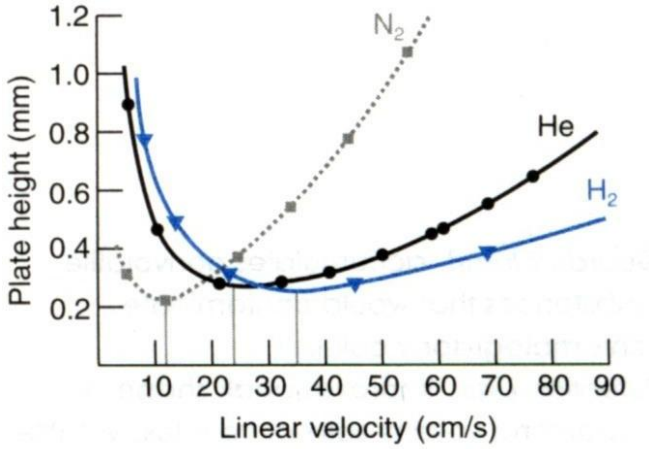
Gas Chromatography

Temperature and Pressure Programming

Improving Column Efficiency

- Pressure Programming:
  - ***Increase* pressure → *increases* flow of mobile phase (carrier gas)**
  - ***Increase* flow → *decrease* retention time**

Van Deemter curves indicate that column efficiency is related to flow rate



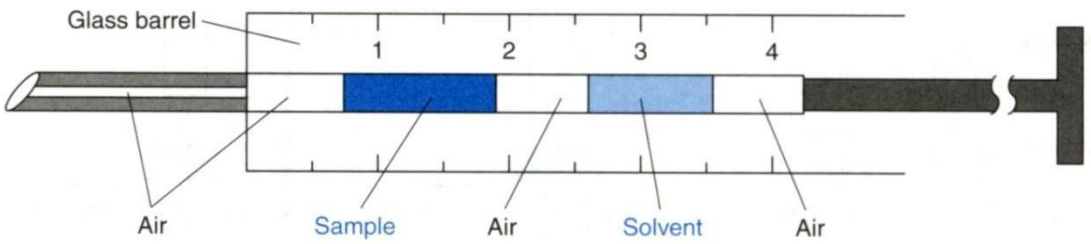
- Pressure is rapidly reduced at the end of the run
  - **Time is not wasted waiting for the column to cool**
  - **Useful for analytes that decompose at high temperatures**

Gas Chromatography

Sample Injection (old)

1) "Sandwich" Injection

- Separate sample with air bubbles and solvent



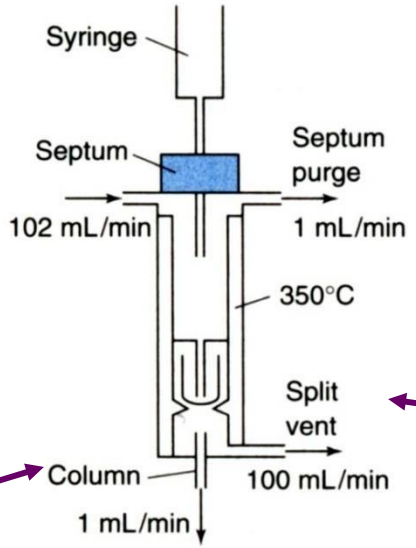
- **Air bubble prevents depletion of most volatile compounds before sample injection is complete (barrier between oven and sample during injection)**
- **Solvent is used to pushes out sample, but bubble prevents mixing**
- **Final air bubble pushes out solvent**
- **Gas-tight syringe is required for gas samples**
- **Injection volume is typically 0.1-2 μL**

Gas Chromatography

Sample Injection

Split Injection

- Delivers only 0.2-2% of sample to the column
  - **Split ratio of 50:1 to 600:1 (sample discarded)**
- For samples where analytes of interest are >0.1% of sample
  - **Best resolution is obtained with smaller amount of sample**
  - **≤ 1 μL with ≤ 1 ng of each compound (0.5 mL of gas volume)**



Split injection

Remainder of the sample is flushed from injector port to column

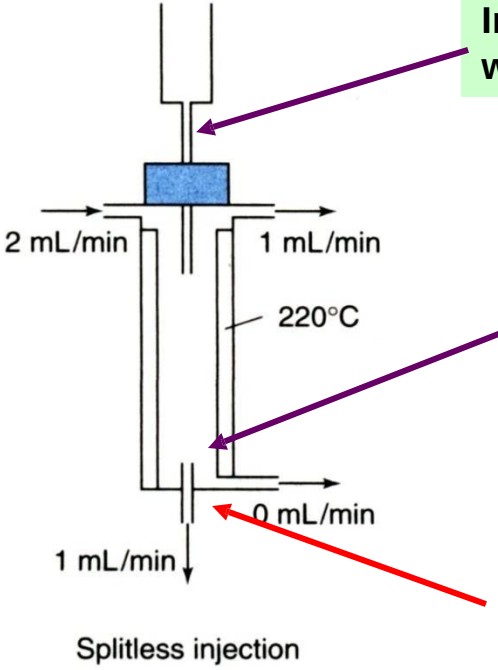
After mixing, pressure regulator controls the fraction of sample discarded

Gas Chromatography

Sample Injection

2.) Splitless Injection

- Delivers ~80% of sample to the column
- For trace analysis, where analytes of interest are < 0.01% of sample
  - **Large volume (~2 μL) injected slowly (2s)**
- No mixing chamber or split vent
  - **Injection temperature is lower (220°C)**
  - **40°C *below* the boiling point of the solvent**



Splitless injection

Injecting larger volume, don't want broad peaks

Lower temperature "traps" solvent in a narrow band at the head of the column

Raise temperature to volatize sample and start separation

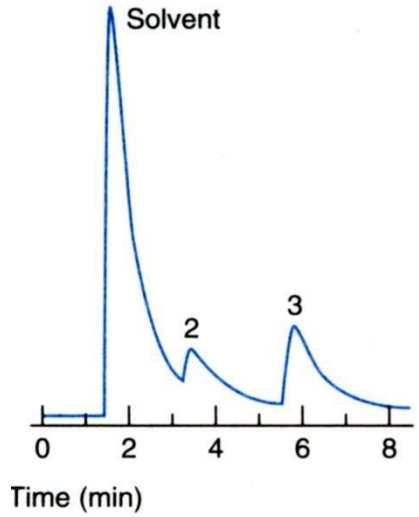
Gas Chromatography

Sample Injection

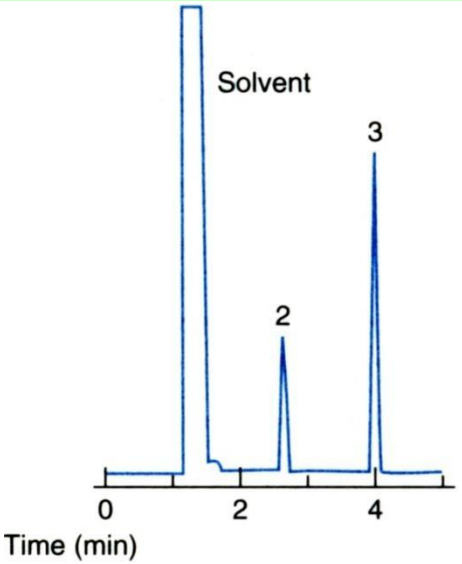
2.) Splitless Injection

- "Solvent trapping" significantly improves the performance of splitless injections
  - **Initial lower temperature of column during injection keeps larger volume into a narrow band**
  - **Chromatography is initiated by raising column temperature**
  - **<u>Cold trapping</u> – condense solutes in narrow band at the beginning of column by using an initial temperature 150°C below boiling points of solutes of interest**

Without "Solvent trapping"



With "Solvent trapping"



Gas Chromatography

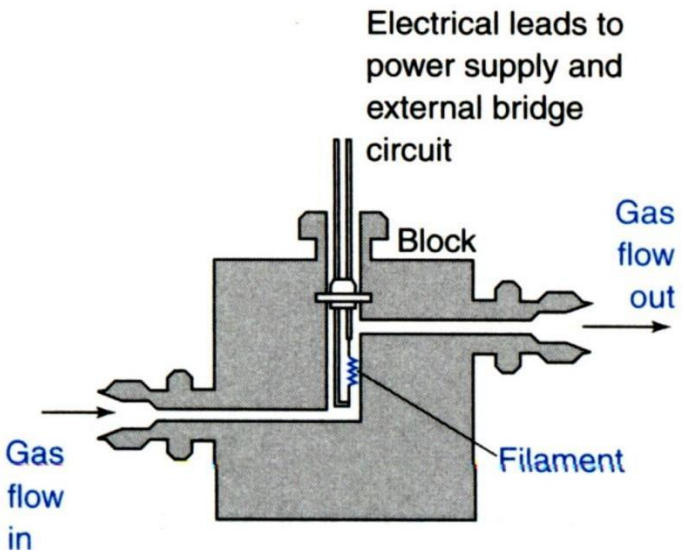
Detectors

Thermal Conductivity Detector

- Measures amount of compound leaving column by its ability to remove heat
  - **It has high thermal conductivity, then the presence of any compound will lower the thermal conductivity increasing temperature of filament**
- As heat is removed from filament, the resistance (R) of filament changes
  - **Causes a change in an electrical signal that can be measured**
- Responds to *all* compounds (universal)
  - **Signal changes in response to flow rate of mobile phase and any impurities present**
  - **Not very sensitive**

Ohm's Law: V =IR

Based on Ohm's law, monitored potential (V) or current (I) Changes as resistance (R) of filament changes due to presence of compound

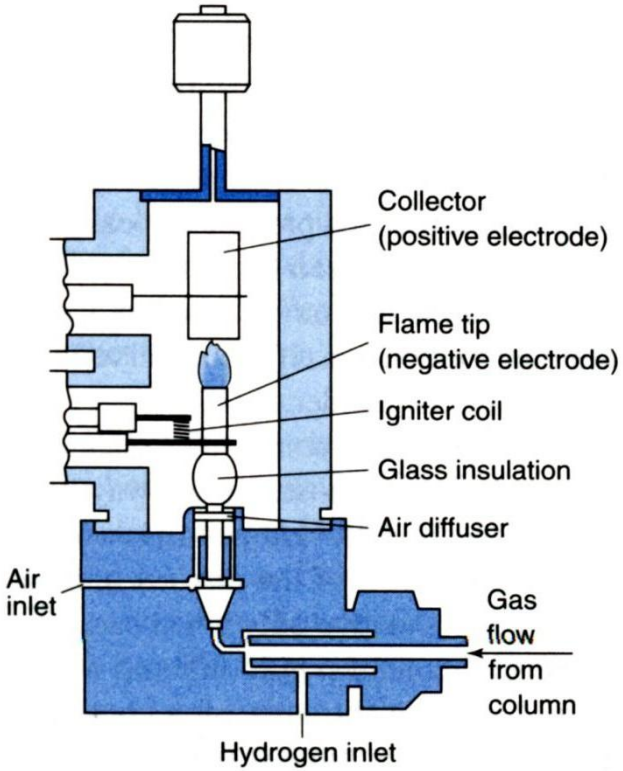


Gas Chromatography

Detectors

Flame Ionization Detector

- **Mobile phase leaving the column is mixed with H_2 and air and burned in a flame**
  - **Carbon present in eluting solutes produces CH radicals which produce CHO^+ ions**
  - **Electrons produced are collected at an electrode and measured**
- **Responds to almost all organic compounds and has good limits of detection**
  - **100 times better than thermal conductivity detector**
  - **Stable to changes in flow rate and common mobile phase impurities (O_2, CO_2,H_2O,NH_3)**



$$CH + O \longrightarrow CHO^+ + e^-$$

Burn sample and measure amount of produced electrons

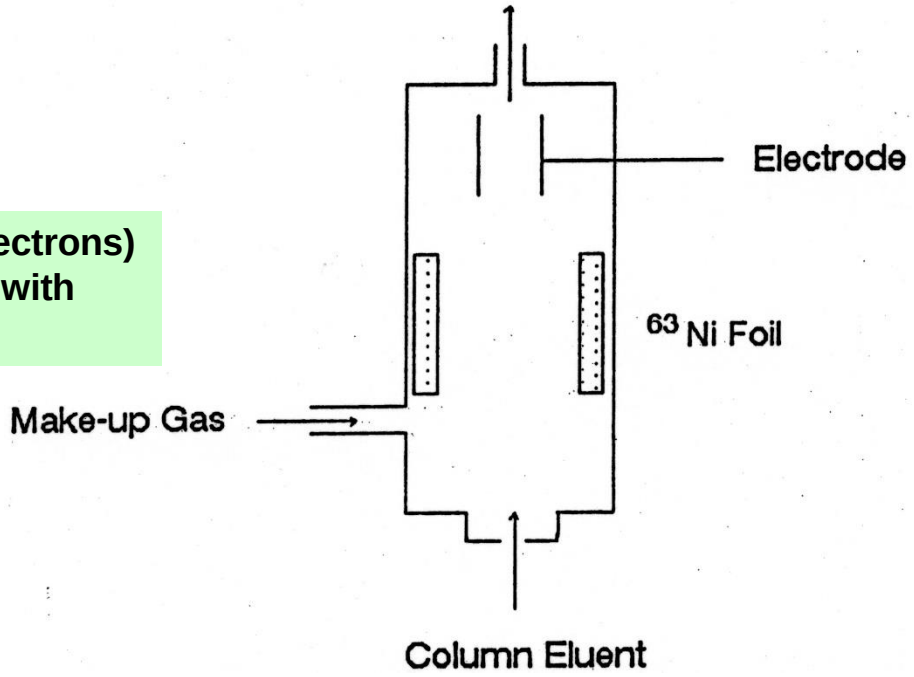
Gas Chromatography

Detectors

Electron Capture Detector

- Sensitive to halogen-containing and other electronegative compounds
- Based on the capture of electrons by electronegative atoms
  - **Compounds ionized by β-rays from radioactive ^{63}Ni**
- Extremely sensitive (~ 5 fg/s)

Steady current (flow of electrons) disrupted by compounds with high electron affinity

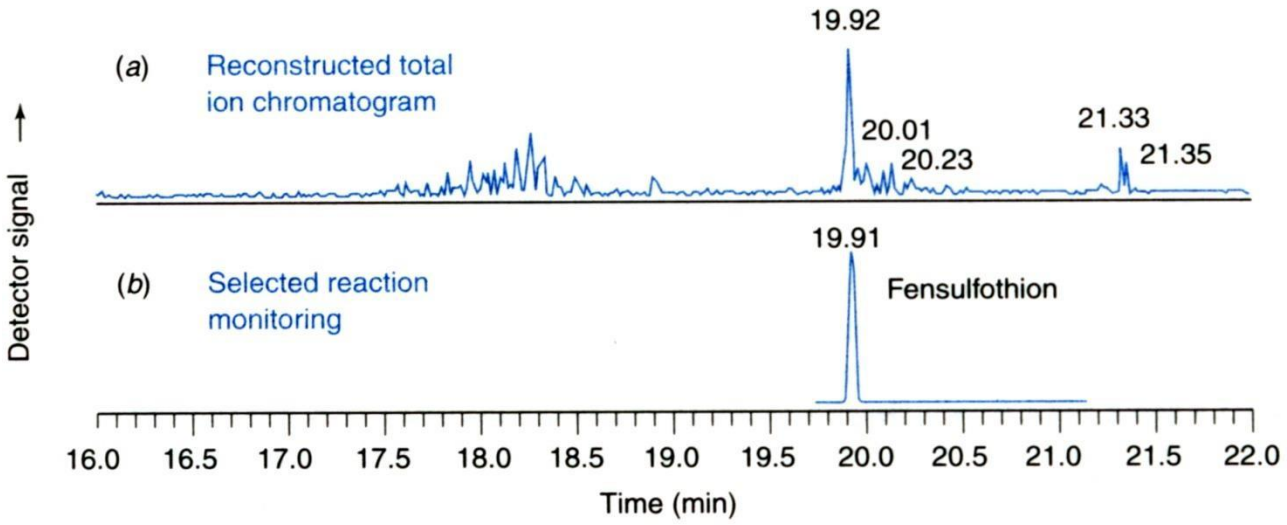


Gas Chromatography

Detectors

Mass Spectrometry

- Detector of Choice → *But Expensive!*
- Sensitive and provides an approach to identify analytes
- Selected ion monitoring – monitor a specific mass/charge (mz) compared to scanning over the complete spectra
  - **Simplifies complex chromatogram**
  - **Increases sensitivity by 10^2-10^3**



Gas Chromatography

Detectors

Other Detectors

- Respond to limited class of analytes
- Modification of previous detectors
- Nitrogen-Phosphorous detector
  - **Modified flame ionization detector**
  - **Extremely sensitive for compounds containing N and P**
  - **Important for drugs and pesticides**
- Flame photometric detector
  - **Measures optical emission from P (536 nM) , S (394 nM), Pb, Sn, and other select elements after passing sample through flame (flame ioniation detector)**
- Photionization detector
  - **Uses a ultraviolet source to ionize aromatic and unsaturated compounds, electrons produced are measured (Electron capture detector)**
- Sulfur/nitrogen chemiluminescence detector
  - **Collects exhaust of flame ionization detector**
  - **S and N converted to SO and NO**
  - **Mix with O_3 form excited state of SO_2 (emits blue light) and NO_3**

Gas Chromatography

Sample Preparation

Transform sample into form suitable for analysis

- Extraction, concentration, removal of interfering species or chemically transforming (derivatizing)

Liquid-Liquid extraction

separate compound based on their relative solubilities in two different immiscible liquids, usually water (polar) and an organic solvent (non-polar). Use simple labware or dedicated equipment

Solid-phase microextraction

- Extract analytes from complex mixture *without* solvent
- Uses a fused-silica fiber coated with stationary phase
  - **Stationary phase similar to those used in GC**
- Expose Fiber to sample to extract compounds and then inject fiber into GC to evaporate analytes

Gas Chromatography

Sample Preparation

Purge and Trap

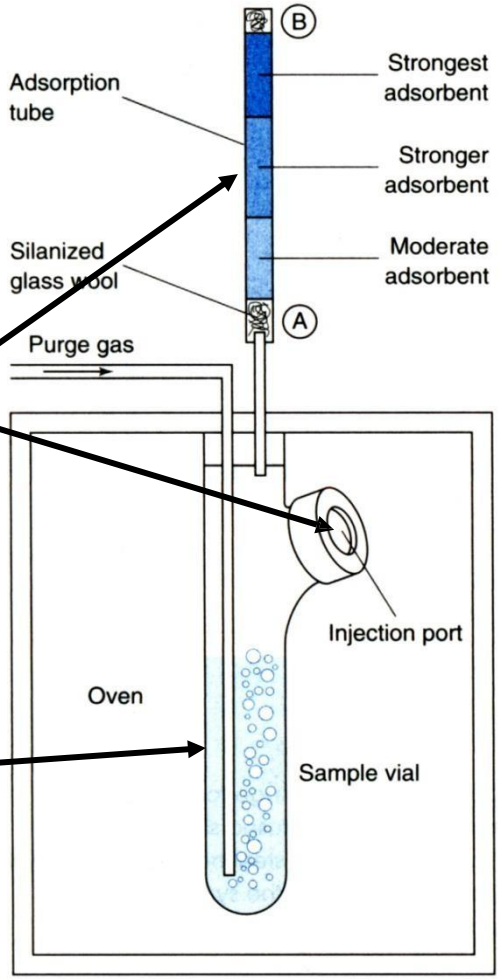
- Removes volatile analytes from liquids or solids concentrates sample and transfer to GC
- Goal is to remove 100% of analyte

Connect port to GC

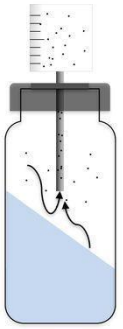
Heat column to 200°C to transfer analytes to GC

Analytes are captured on adsorbent column

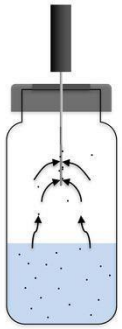
Bubble purge gas (He) through heated sample to evaporate analytes



GC-MS Sampling Methods



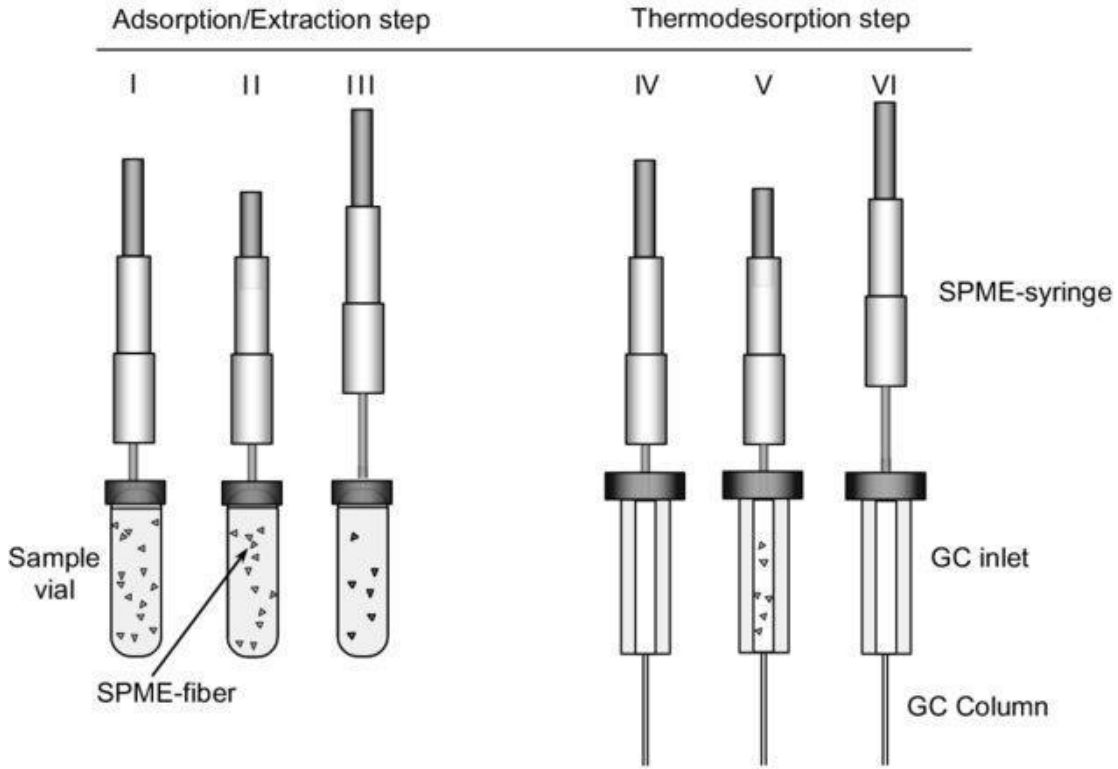
Direct Headspace Sampling:
- Quantification of VOCs using 1 mL of headspace gas.



Solid Phase Microextraction (SPME):
- Concentration of VOCs on absorbent fiber.

Micro solid phase extraction (SPME)

Extraction depend on time of exposure, material of the fiber, not 100%



Automated sampling (SPME)

Gas Chromatography

Method Development in GC

How to Choose a Procedure for a Particular Problem

- Many Satisfactory Solutions
- The order in which the decision should be made should consider:
  1. **Goal of the analysis**
  2. **Sample preparation**
  3. **Detector**
  4. **Column**
  5. **Injection**

- Goal of the analysis
  - **Qualitative vs. quantitative**
  - **Resolution vs. sensitivity**
  - **Precision vs. time**
  - **Interest in a specific analyte**

- Sample preparation
  - **Cleaning-up a complex sample is essential**
  - **Garbage in → garbage out**

- Choosing the Detector
  - **Detect a specific analyte(s) or everything in the sample**
  - **sensitivity**
  - **Identify an unknown (MS, FTIR)**

FATTY ACIDIC PROFILE OF RICOTTA CHEESE

LIPID EXTRACTION

Solubilisation and protein precipitation

2.5 g of ricotta cheese + 5 mL of Ethanol + 750 µL of ammonium hydroxide 25% in water mixed and shacked thoroughly for protein precipitation.

Extraction in diethyl ether

12.5 mL of petroleum ether were added, the sample was mixed and shaken carefully, and 12.5 mL of diethyl ether were added. The sample was mixed and shaken again, and the supernatant was recovered in a flask

Improvement of the recovery of extracted analytes

The extraction was repeated other two times with half volumes.

Pre-concentration of the analytes

At the end of the three extraction, the solvent was evaporated with the use of a Strike-Rotating Evaporator when the solvent was completely evaporated, the flask with the lipids was heated in the air oven for 20 min at 60 °C then cooled at room temperature in a dryer.

DERIVATISATION

TRANS-METHYLATION OF THE FATS

- 60 mg of lipid + 1mL of hexane + 500 mL of CH_3ONa

INJECTION

- 1 µL of methylated extract

Trace GC (ThermoScientific, Waltham, United States) + flame ionization detector (FID)

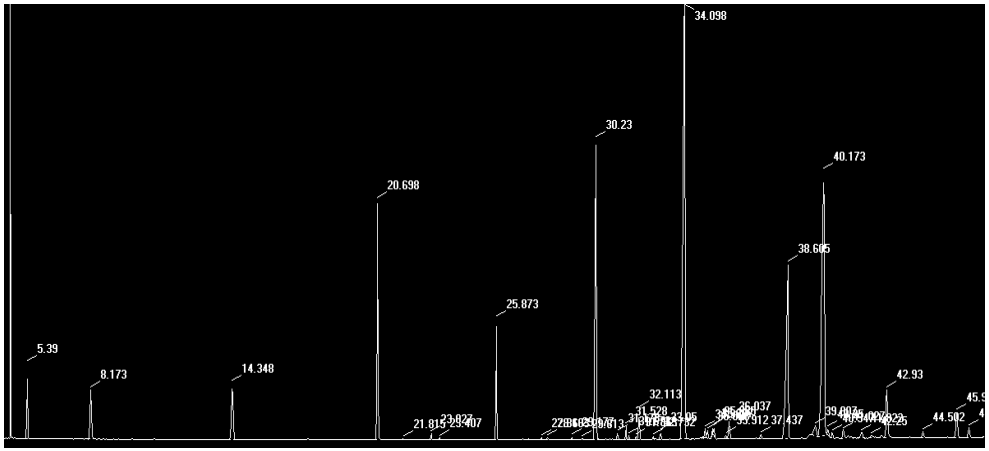
- Column: Restek Rt-2560 100 m, 0.25 mm ID,
(highly polar phase; biscyanopropyl polysiloxane—not bonded Stationary phase selectivity optimized for isomer separation to ensure accurate quantification of critical cis/trans FAMEs)
- Run time: 56 min
- Carrier gas: Hydrogen
- Flow rate: 1 ml/min
- Injector and Detector temperature: 280 °C
- Oven temperature:

Rate (°C/min)	Temperature (°C)	Hold Time (min)
	80 °C	10 min
4 °C/min	172 °C	30 min
4 °C/min	190 °C	10 min

- The identification of each fatty acid methyl ester was made comparing the retention time of peaks samples with the peaks of a mix of analytical standards (F.A.M.E. Mix C8-C24, Supelco).
- Peaks areas were quantified using ChromeCard Software and the results for each compound were expressed as percentage of each individual FA of the total FA.

RESULTS

Innosa, D., Bennato, F., Ianni, A., Martino, C., Grotta, L., Pomilio, F., & Martino, G. (2020). Influence of olive leaves feeding on chemical-nutritional quality of goat ricotta cheese. *European Food Research and Technology*, 1-8.

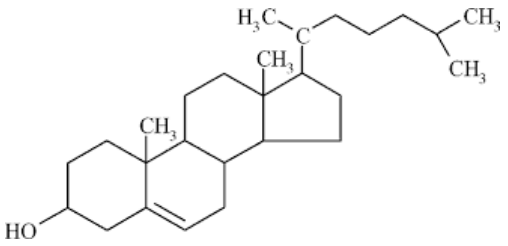


Fatty Acids[1]	CG	EG	P
C4:0	1.25 ± 0.19	1.26 ± 0.36	
C6:0	1.87 ± 0.38	1.82 ± 0.38	
C8:0	2.70 ± 0.83	2.48 ± 0.38	
C10:0	10.15 ± 2.35	8.80 ± 1.06	
C12:0	4.51 ± 0.52	3.86 ± 0.32	
C14:0	11.39 ± 0.17	10.74 ± 0.36	*
C15:0	0.87 ± 0.04	0.86 ± 0.01	
C16:0	25.70 ± 0.90	25.55 ± 0.59	
C17:0	0.61 ± 0.05	0.62 ± 0.02	
C18:0	11.90 ± 1.07	12.62 ± 0.75	
C20:0	0.19 ± 0.06	0.25 ± 0.04	
C22:0	0.06 ± 0.02	0.10 ± 0.01	**
SFA	71.21 ± 6.58	68.96 ± 4.29	**
C14:1	0.41 ± 0.01	0.42 ± 0.01	
C16:1	0.27 ± 0.04	0.32 ± 0.02	*
C18:1 trans11	0.55 ± 0.04	0.68 ± 0.07	*
C18:1 cis9	19.98 ± 1.96	21.60 ± 1.08	
C18:1 cis11	0.21 ± 0.05	0.22 ± 0.05	
MUFA	21.42 ± 2.10	23.24 ± 1.23	**
C18:2	2.45 ± 0.19	2.58 ± 0.18	
CLA	1.09 ± 0.12	1.19 ± 0.13	*
C18:3	1.00 ± 0.02	1.16 ± 0.06	*
PUFA	4.54 ± 0.39	4.92 ± 0.37	*
Others	2.82 ± 0.25	2.88 ± 0.23	

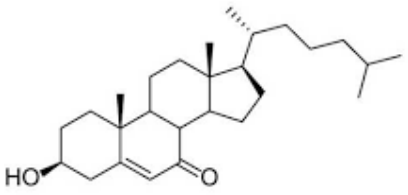
[1]Data are reported as mean percentage of total FAME ± S.D.
SFA: saturated fatty acid; MUFA: monounsaturated fatty acid; PUFA: polyunsaturated fatty acid; CLA: rumenic acid; *P<0.05; **P<0.01; ns: not significant.

Conventional diet (CG) diet enriched with olive leaves (EG)

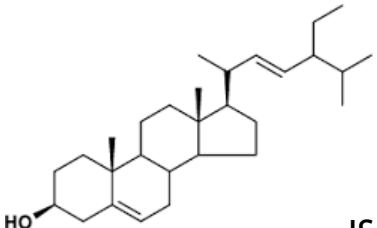
CHOLESTEROL AND 7-KETOCHOLESTEROL IN EGGS



cholesterol



7-keto-cholesterol



Stigmasterol

IS internal standard !!!

An internal standard is a substance added in a known amount to standards, samples, and blanks during an analysis. In chromatography and spectroscopy, the ratio of the signal for the internal standard and the analyte is calculated.

CHOLESTEROL AND 7-KC EXTRACTION

- 10 g of egg yolk + 10 mL of Ethanol + 500 µL 20% of Buthylated hydroxy toluene in methanol (to prevent oxidation) + 100 µL of Stigmasterol (Internal Standard) + 100 µL of KOH 50% water solution
- at 60 °C in a water bath for 20 min
- 2 ml of distilled water + 8 ml of petroleum ether + 1 g of NaCl were added to the samples
- Sample shaken, sonicated and centrifuged to separate into phases
- The supernatant containing non-saponifiable lipids as cholesterol were recovered into round bottom flasks
- Other 2 ml of distilled water + 16 ml of petroleum ether were added
- the phases were again allowed to separate. After recovering the supernatant a third and last extraction was repeated on each sample.
- the solvent was evaporated to dryness at 38 °C
- the non-saponifiable lipids were recovered with 1 mL of hexane and transferred into an 8-mL vial.

DERIVATIZATION OF CHOLESTEROL

- the sample was evaporated with a nitrogen flow
- derivatized with Pyridine and BSTFA (2:1 ratio) O-bis(trimethylsilyl) trifluoroacetamide (BSTFA) into their Si(CH3)3 derivatives .
- 1 min of incubation
- sample was evaporated and recovered with 1 ml of hexane.

INJECTION

- 1 µL of derivatized extract

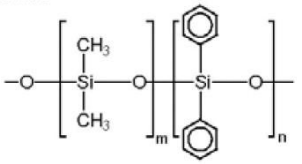
Trace GC (ThermoScientific, Waltham, United States) + flame ionization detector (FID)

- Column: Restek XTI-5 30 m, 0.32 mmID, 0.25 µm as column
- Run time: 56 min
- Carrier gas: Hydrogen
- Flow rate: 1 ml/min and the
- Injector and Detector temperature: 280 °C
- Oven temperature:

Rate (°C/min)	Temperature (°C)	Hold Time (min)
	175 °C	1 min
5 °C/min	230 °C	0 min
10 °C/min	300 °C	5 min

95% Dimethyl-(5%) diphenylpolysiloxane, bonded and crosslinked phase.

- Specially manufactured column to fulfil the level of inertness required by the EPA methods for the analysis of semivolatile compounds, designed for methods 625, 1625, 8270 and CLP protocols
- Inertness and minimum absorption for acidic, basic and neutral compounds



Structure of Poly(dimethyldiphenyl)siloxane

- The analytical method for the identification of cholesterol was calibrated in the range from 100 to 700 µg/ml (r^2 0.9949)
- Peaks areas were quantified using ChromeCard Software
- Results for each compound were expressed as mg/g of egg yolk

RESULTS

Innosa, D., Ianni, A., Palazzo, F., Martino, F., Bennato, F., Grotta, L., & Martino, G. (2019). High temperature and heating effect on the oxidative stability of dietary cholesterol in different real food systems arising from eggs. *European Food Research and Technology*, *245*(7), 1533-1538.

Table 2 Cholesterol and 7-KC obtained from fresh yolks and hard-boiled eggs for 5, 10 and 20 minutes. Data are expressed as mg/g of yolk for cholesterol and as µg/mg of yolk for 7-KC.

	Fresh Yolks	Hard-boiled eggs 5’	Hard-boiled eggs 10’	Hard-boiled eggs 20’
Cholesterol (mg/g)	$15.57^{a}\pm0.44$	$16.13^{a}\pm0.65$	$12.36^{b}\pm1.62$	$12.10^{b}\pm1.65$
7-ketocholesterol (µg/mg)	n.d.	n.d.	n.d.	n.d.

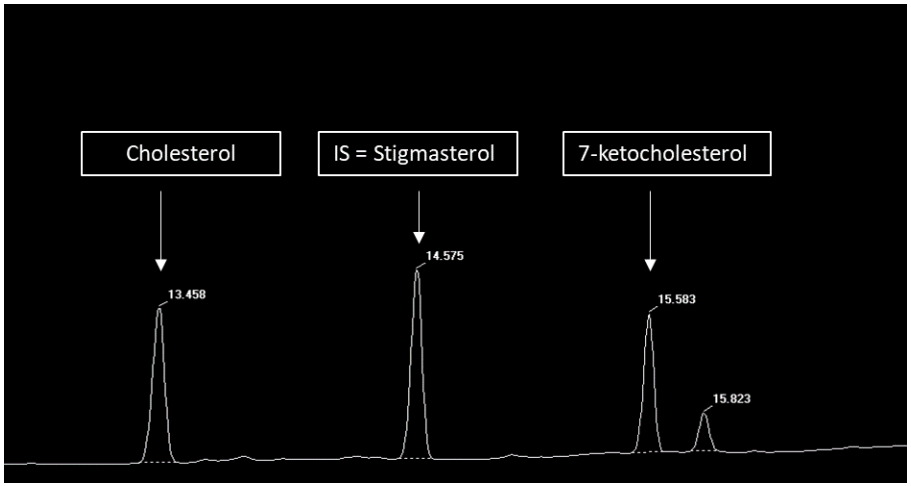


Figure 2 Chromatogram of Cholesterol, Internal standard and 7-KC standard

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journal homepage: www.elsevier.com/locate/talanta

Direct determination of acrylamide in potato chips by using headspace solid-phase microextraction coupled with gas chromatography-flame ionization detection

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Acrylamide has been classified as a "probable human carcinogen" (Group 2A) by the International Agency for Research on Cancer (IARC). Acrylamide formation in foods is mainly attributed to the Maillard browning reaction, which is an interaction between the amino group of asparagine free amino acid and the carbonyl groups of reducing sugars such as glucose and fructose, at higher temperatures and low moisture contents.

Acrylamide Colour Chart

Visibly display this colour chart so that staff preparing chips can check that they are a light golden colour and not overcooked. This chart could be covered or laminated to protect it in the kitchen.

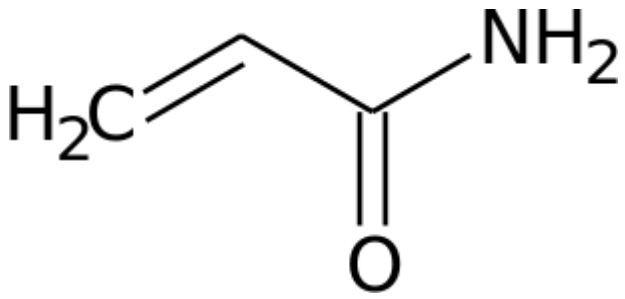
Image 1 - 24 micrograms acrylamide per kilogram

Image 2 - 130 micrograms acrylamide per kilogram

Image 3 - 690 micrograms acrylamide per kilogram

Image 4 - 1590 micrograms acrylamide per kilogram

EU Regulation 2017/2158 sets a benchmark level for French fries (ready-to-eat) of 500 µg/kg.



Processed foods with high levels of Acrylamide like French fries, potato chips, crisp bread as well as Various baked products and cereal formulations show a wide range of acrylamide level, different in category and brand of food.

Most used method are GC–MS and LC–MS–MS, the study reports the development of GC-FID coupled to HS-SPME to reduce the cost of the analysis

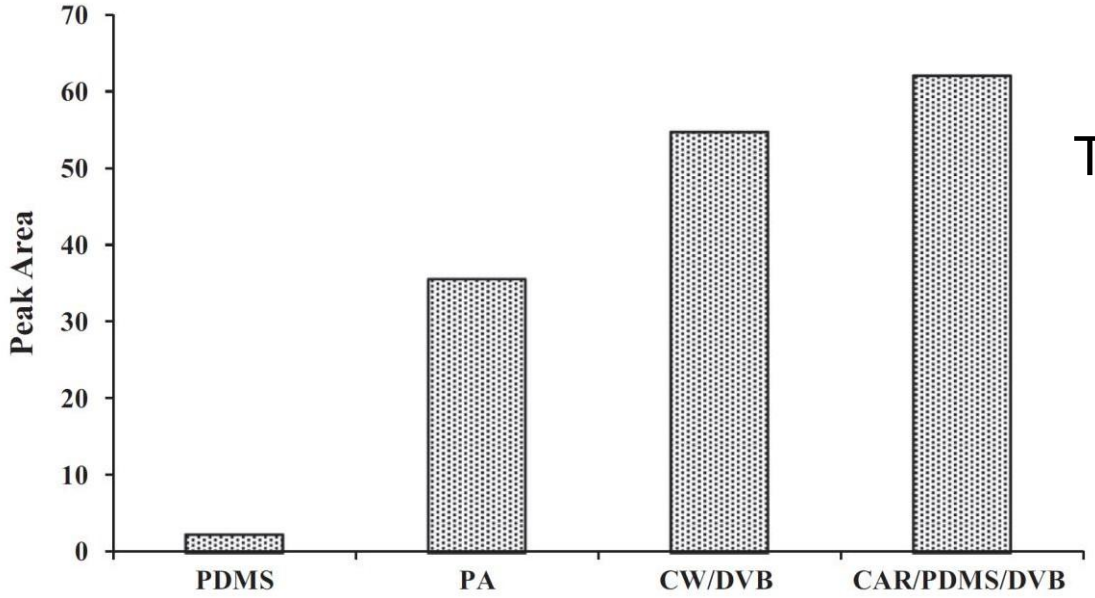
- Different commercial fibers were tested
- The extractions were performed in10-mL sample vials with crimp caps and PTFE-coated Silicone septa(Supelco). An A3229 Unicam Pro GC gas chromatograph (USA), equipped with a BP20 polar capillary column (polyethyleneglycol, 30m x 0.25 mm x 0.25 μm) from SGE Company and a flame ionization detector (FID), was employed for the chromatography.
- Temperature was started from 45 °C held for 2min and then raised to 150 °C at the rate of 10 °C/ min. The temperature of the detector and injector was held at 200 and 230 °C, respectively. Under these conditions, acrylamide was eluted at a retention time of 4.038 min.

Sample

For more realistic evaluation and optimization of the experimental conditions in HS-SPME experiments, it is recommended touse a real sample matrix without analytes, especially for solid samples. Therefore,100 g of raw and non-heated potato was cut using an electric slicing machine and the resulted slices were rinsed in distilled water to remove starch from their surface. Then, potato slices were dried by using a household fruit-dryer (Fuma Starfruit-dryer,modelFU-731) at 45 °C for 20 hand homogenized by means of a Kenwood kitchen blender (Kenwood, model BL 530, UK). This sample was considered as the blank (control) or model solid sample matrix, for using throughout the optimization experiments.

Acrylamide extraction

The SPME fiber was fitted into a manual holder and located into the headspace above the sample for the extraction ofacrylamide (30 min at 60 °C). The fiber was then withdrawn into the needle and immediately injected into GC-FID system. Desorption of the analyte was performed at 230 °C for 2 min. Acrylamide was determined in triplicate by using the GC-FID system.



Type of fiber

Fig. 1. Comparison of different commercial fibers for extraction of acrylamide from the potato model matrix (error bars represent SD for three replicate experiments).

Extraction temperature

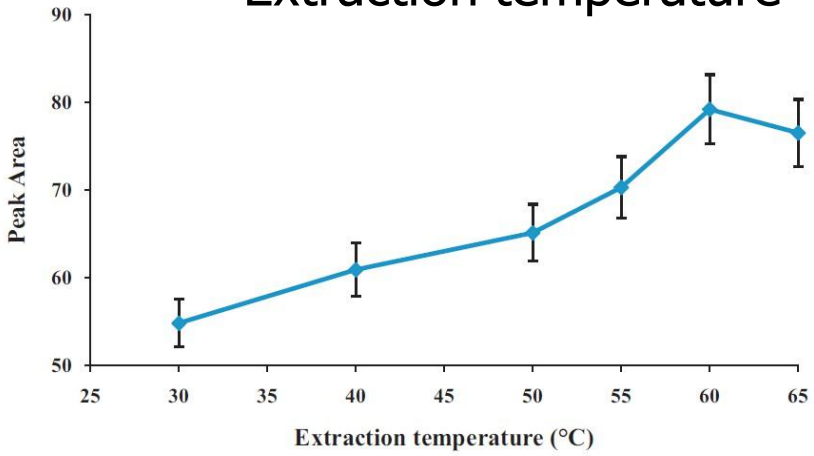


Fig. 2. Effect of extraction temperature on the extraction efficiency of acrylamide using the proposed HS-SPME-GC-FID method (conditions: fiber, CAR/PDMS/DVB; extraction time, 30 min).

Extraction time

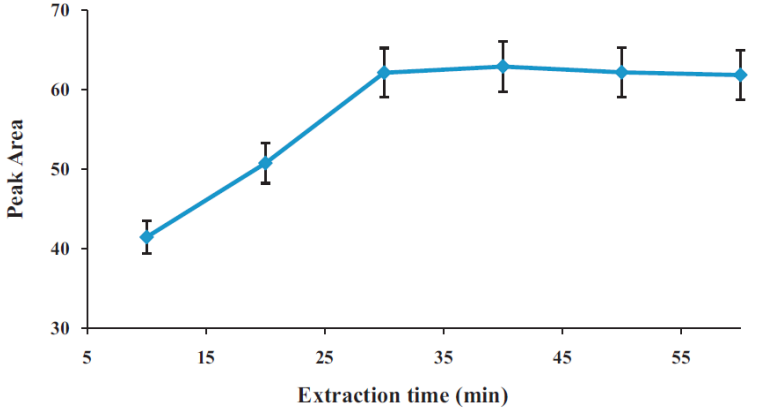


Fig. 3. Effect of extraction time on the extraction efficiency of the HS-SPME strategy used to extract acrylamide (conditions: fiber, CAR/PDMS/DVB; extraction temperature, 60 °C).

Addition of a modifier (water)

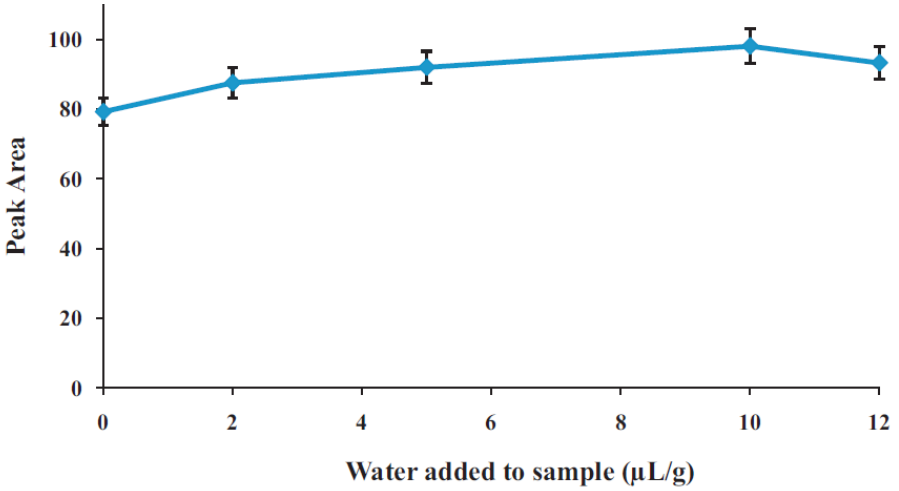


Fig. 4. Changes trend of extraction efficiency of acrylamide *vs.* addition of different volumes of water to sample matrix (conditions: fiber, CAR/PDMS/DVB; extraction temperature, 60 °C; extraction time, 30 min).

Desorption time and temperature

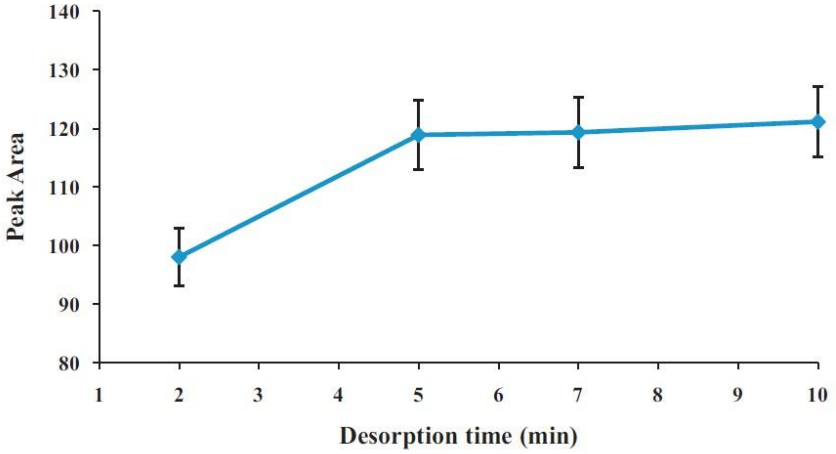


Fig. 5. Influence of fiber's retaining time in GC injector on the amount of desorbed acrylamide (conditions: fiber, CAR/PDMS/DVB; extraction temperature, 60 °C; extraction time, 30 min; water added to sample, 10 µL g^{-1}).

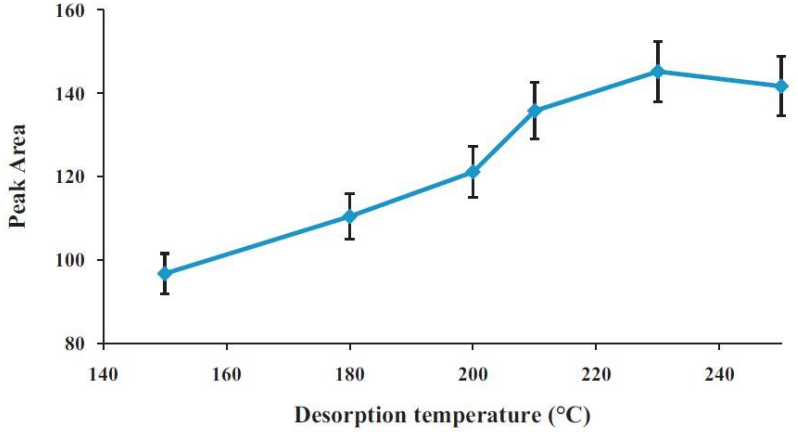


Fig. 6. Effect of different desorption temperatures on the peak area of acrylamide extracted by the proposed HS-SPME-GC-FID procedure (conditions: fiber, CAR/PDMS/DVB; extraction temperature, 60 °C; extraction time, 30 min; water added to sample, 10 µL g^{-1}; desorption time, 2 min).

Analytical performance

Linear Range (or linear dynamic range) = 0.77–50 mg/g (regression coefficient (r^2) = 0.9977.
Limit of detection (LOD) = 0,23 mg/g (3 x Signal to Noise ratio)
Limit of Quantification(LOQ) = 0.77 mg/g (10 x Signal to Noise ratio)

RECOVERY

Table 1
Recovery percent for potato chips and French fries samples (without acrylamide) spiked with 20 μg g^{-1} of acrylamide and determined using the proposed HS-SPME-GC-FID method.

Sample	Acrylamide determined (μg g^{-1})	Recovery (%)	RSD (%)
Potato chips 1	18.64	93.2	8.0
Potato chips 2	16.46	82.3	5.2
Potato chips 3	15.92	79.6	4.1
French fries 1	17.42	87.1	7.4
French fries 2	19.14	95.7	6.7
French fries 3	17.58	87.9	5.1

REPRODUCIBILITY

COMPARISON WITH A VALIDATED INDEPENDENT METHOD!

Table 2
Concentration of acrylamide in potato chips samples obtained by the proposed HS-SPME-GC-FID method and a validated Soxhlet-GC–MS extraction procedure ($n=3$).

Sample	Acrylamide determined ($\mu g\ g^{-1}$) by	
	The proposed HS-SPME-GC-FID	**Soxhlet-GC–MS**
Potato chips 1	6.15 ± 6.2	8.56 ± 5.3
Potato chips 2	4.87 ± 7.4	6.91 ± 3.1
Potato chips 3	7.38 ± 2.3	9.01 ± 5.0

MASS SPECTROMETRY

- MS has been described as the smallest scale in the world, because of the size of what it weighs→ molecules
- Ions are generated by inducing either the loss or gain of a charge from a neutral species
- These ions are electrostatically directed into a mass analyzer where they are separated according to m/z and finally detected
- An MS spectrum that can provide molecular mass and even structural information

GC-MS Pros

1. **High Sensitivity and Specificity**
   Mass spectrometers provide high sensitivity, enabling the detection of trace compounds at very low concentrations. **The combination of retention time (from GC) and mass spectral data (from MS) provides high specificity,** making it easier to distinguish between compounds that might otherwise co-elute.
2. **Enhanced Identification Capabilities**
   Mass spectrometers generate mass spectra (mass-to-charge ratio data), which are unique for each compound and act like fingerprints. This allows for precise identification, especially when using spectral libraries for compound comparison.
3. **Quantitative and Qualitative Analysis**
   GC-MS can perform both qualitative (identification) and quantitative (concentration measurement) analyses, making it highly versatile for detailed sample profiling.
4. **Versatility Across Multiple Applications**
   GC-MS is widely used across fields, including food analysis (for flavor and aroma profiling), environmental testing (for pesticides, pollutants), forensic analysis (for drugs, explosives), and pharmaceutical testing. It's adaptable to many different compound types and applications.
5. **Detection of Unknowns**
   With MS, analysts can identify unknown compounds by comparing their mass spectra to known databases or libraries, which is valuable for detecting contaminants or new compounds in complex mixtures.
6. **Structural Information**
   The MS detector provides valuable structural information about analytes, helping in understanding compound composition and molecular structure, which is crucial in research and development.
7. **Automation and Throughput**
   Modern GC-MS systems are automated and support high-throughput analysis, allowing for quicker sample processing and data analysis, especially with advanced software for data interpretation.

GC-MS cons

1.High Cost and Maintenance

GC-MS systems are expensive to purchase and maintain. They require specialized equipment, consumables, and trained personnel for operation, which can be a barrier for smaller labs or facilities.

2.Complex Operation and Interpretation

MS requires skilled operators to interpret data, manage calibrations, and troubleshoot issues. Misinterpretation of mass spectra can lead to inaccurate conclusions, requiring a higher level of expertise.

3.Limitations to Volatile and Thermally Stable Compounds

GC-MS is generally limited to volatile, thermally stable compounds. Non-volatile or thermally labile substances cannot be easily analyzed without prior derivatization, which can add time, complexity, and potential for error.

4.Matrix Interferences

Complex sample matrices (such as food, soil, or biological samples) can interfere with GC-MS analysis. These interferences may require additional sample preparation steps, increasing time and cost.

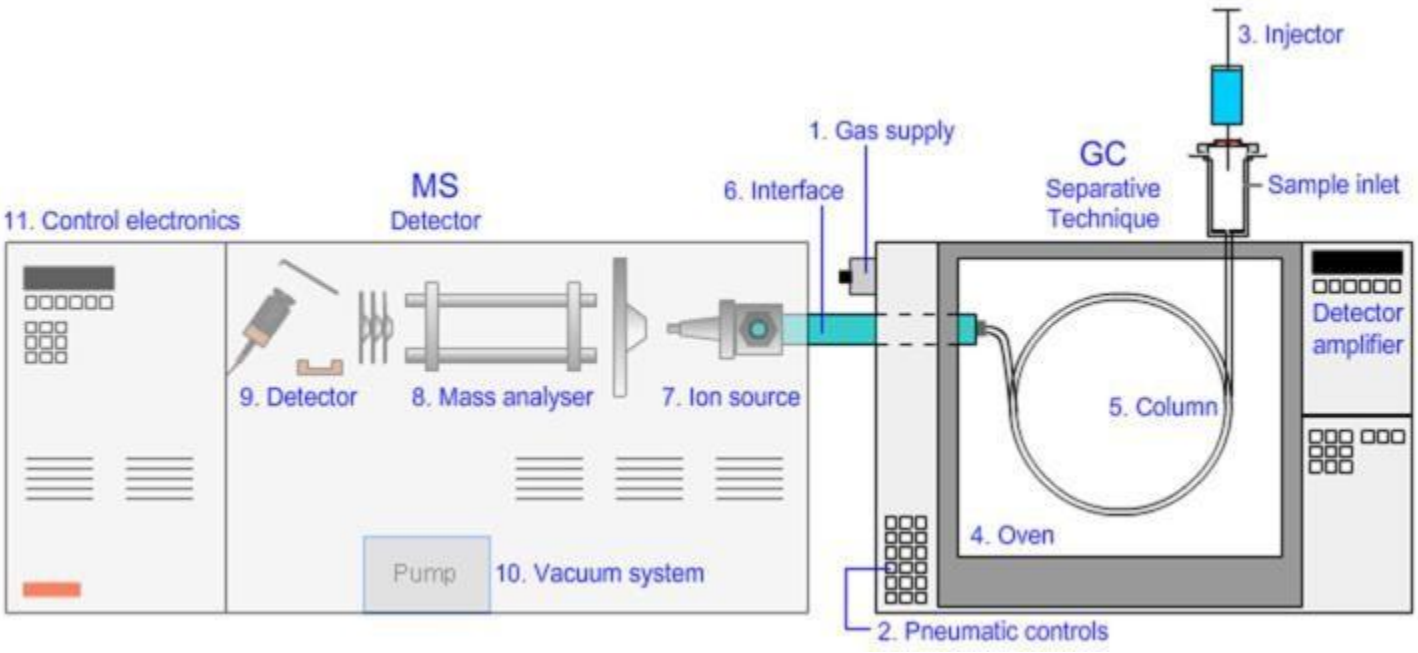
5.Sensitivity to Contamination

MS detectors are highly sensitive and can be affected by contamination from previous samples, column bleed, or carrier gas impurities. Maintaining cleanliness and quality control is critical, which can require extra time and effort.

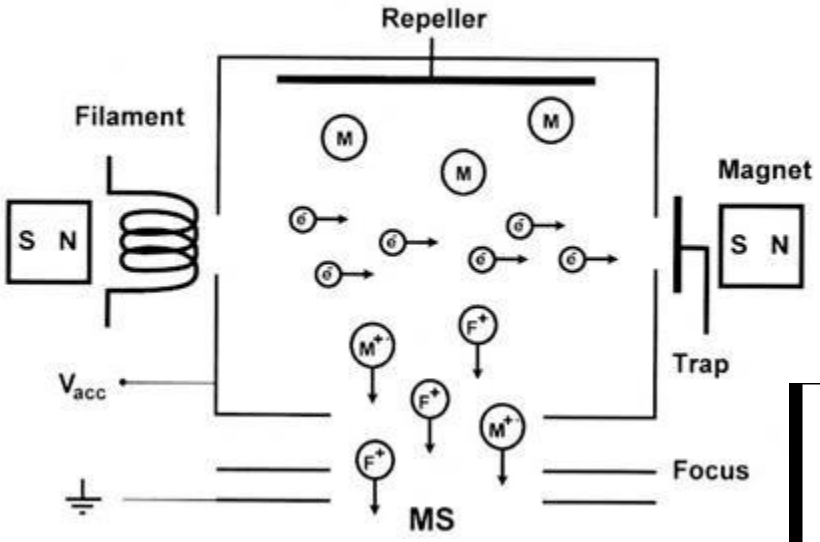
6.Slow Analysis Times for Complex Samples

1. While GC-MS is powerful, analyzing very complex mixtures can be time-consuming due to necessary calibration, optimization, and possible derivatization steps, reducing throughput compared to simpler analytical methods.

GC-MS scheme

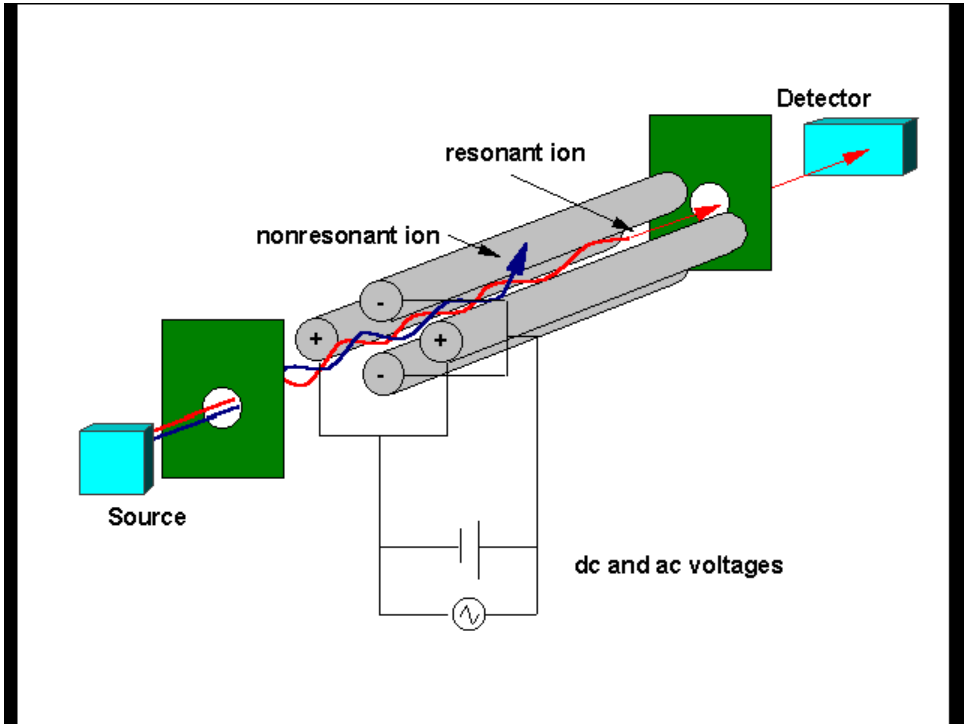

3. Injector
1. Gas supply
GC
Separative
Technique
Sample inlet
MS
Detector
6. Interface
11. Control electronics
Detector
amplifier
9. Detector
8. Mass analyser
7. Ion source
5. Column
4. Oven
Pump
10. Vacuum system
2. Pneumatic controls

ELECTRONIC IMPACT ION SOURCE GC-MS (HARD SOURCE- HIGH ENERGY)

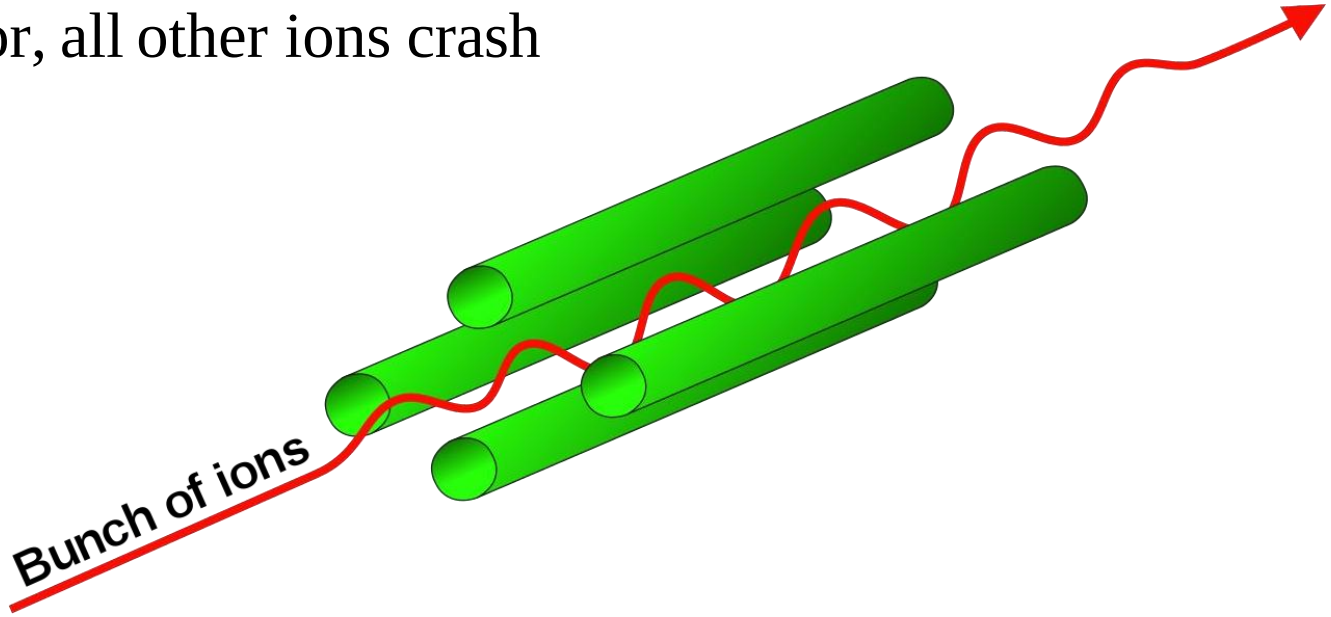


A Mass Spectrometer consists in an ion source, a mass analyzer, and a detector that is capable of measuring the amount of a selected mass-to-charge ratio of ions produced by the source. Each compound coming from GC is ionised and broken. .

QUADRUPOLE – MASS SELECTOR



Application of an oscillating sinusoidal potential to each couple of rods causes only a particular M/Z ratio to pass and reach the detector, all other ions crash



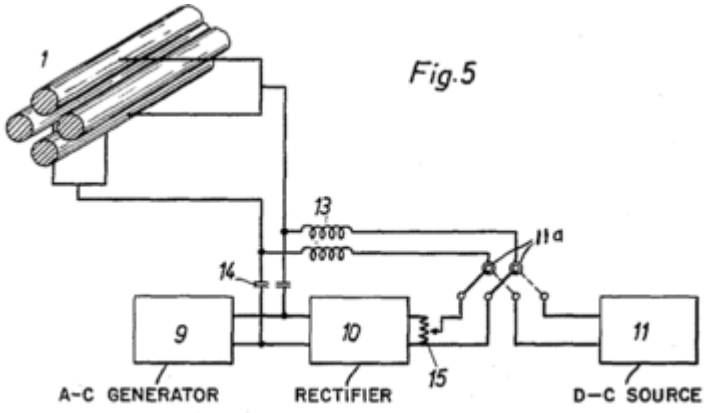
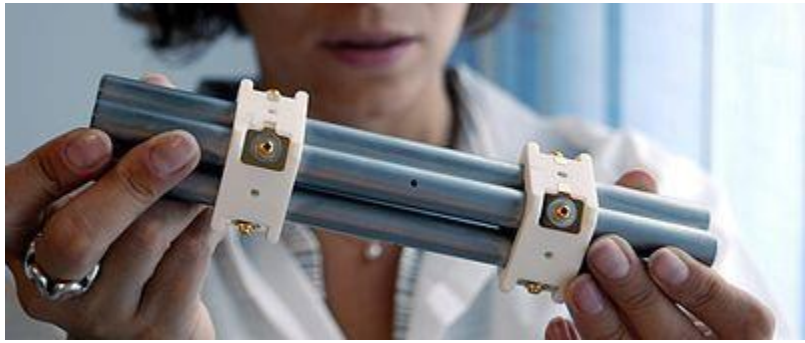
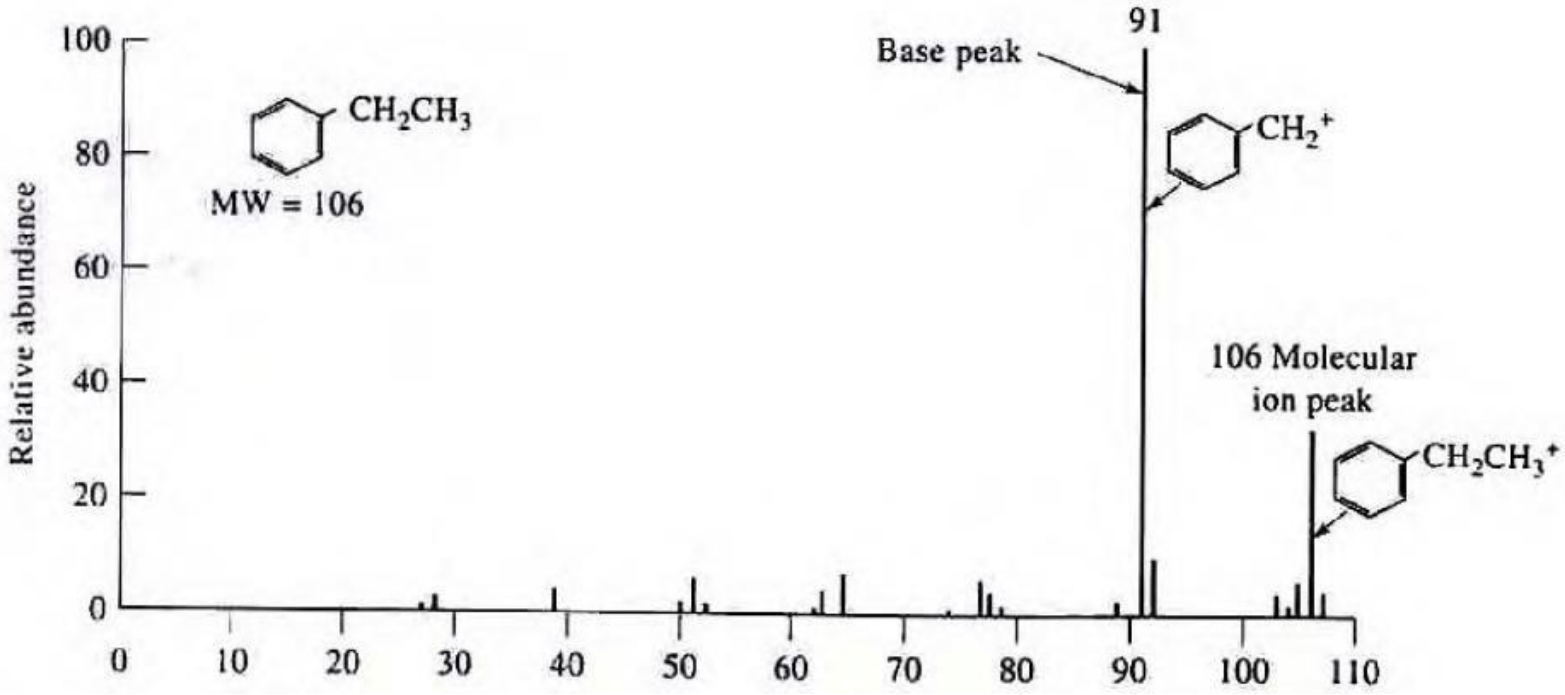


Fig.5




Relative abundance
100
80
60
40
20
0
0
10
20
30
40
50
60
70
80
90
100
110
CH2CH3
MW = 106
91
Base peak
CH2+
106 Molecular
ion peak
CH2CH3+

detection of volatile compounds in blenched carrot samples by GC-MS

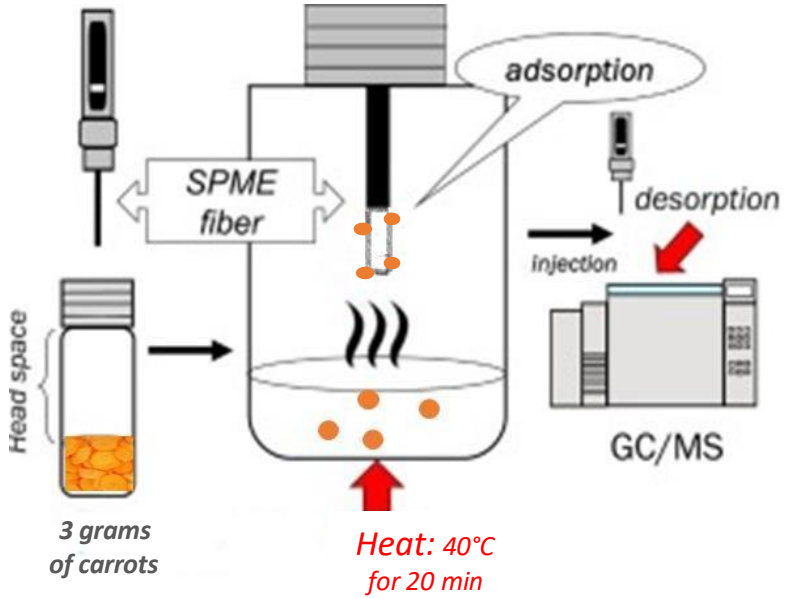
Monitoring and control of vegetable ripening are important parameters in the food industry, since the maturation state during harvest, storage and distribution on the market defines the quality of the end product

The samples were analyzed at different times during storage at different temperatures (-18 C, 4 C, 25 C, and 40 C).

To prevent enzymatic reactions during processing, storage and thawing, the packaged carrots, cut in 1cm height pieces, were blanched at 90 °C for 10 min in a water bath

The results demonstrated that the carrot samples showed clear changes in aroma compounds that were identified and monitored using GC-MS

GC-MS/SPME Procedure



3 g **of blanched carrots were placed in 20 ml gas-tight vials and hermetically sealed with a gas-tight septum**

Carrots was kept for 20 min at 40 °C and then exposed to the fiber (DVB/CAR/PDMS; Divinylbenzene/carboxen/polydimethylsiloxane)

24 **vials were prepared for each temperature tested (-18 °C, 4 °C, 25 °C, 40 °C), in order to have three replicates (three different vials) for each day of measurement (1, 4, 8, 12, 19, 26 days)**

The fiber was then inserted in the desorption chamber where GC analysis was carried out with the following temperature gradient: the column was kept at 40 °C for 6 min, then the temperature was raised up to 250 °C at 4°C/min

Finally, this procedure was also used to extract the headspace of the standards (octanal, terpinolene, α-terpineol, o-cymene, D-limonene, β-pinene) using 2 µL of pure standards into 20 ml vials.

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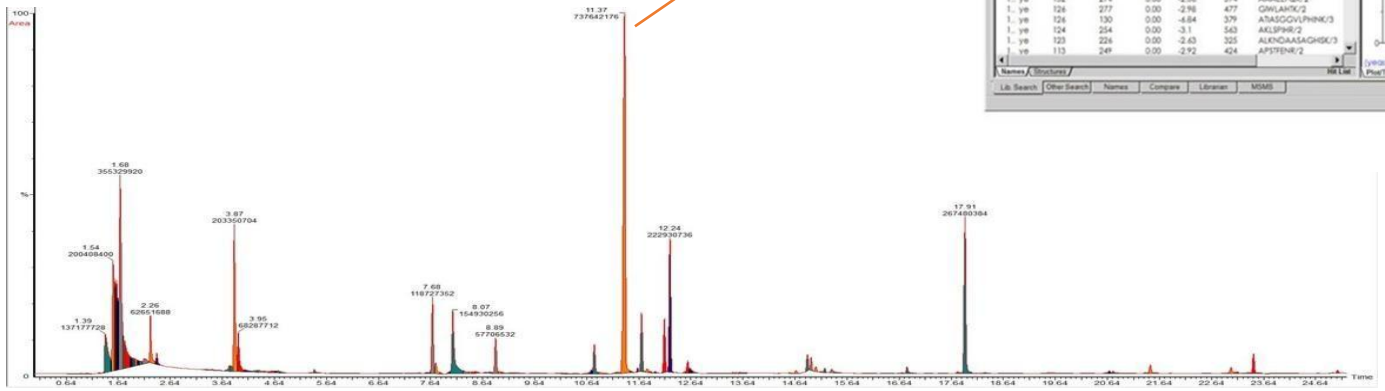
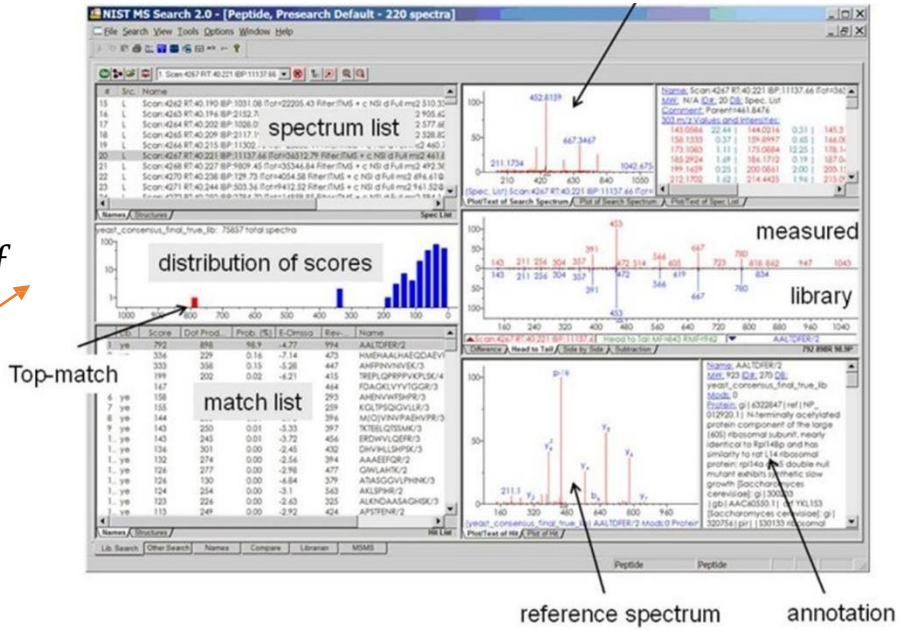
The identification of compounds was carried out using the NIST library of the GC

"The main use of the library is for the identification of compounds by matching fragmentation "fingerprints" (mass spectra) of their ions as generated by the ionization of molecules





….Confirmed by Retention index

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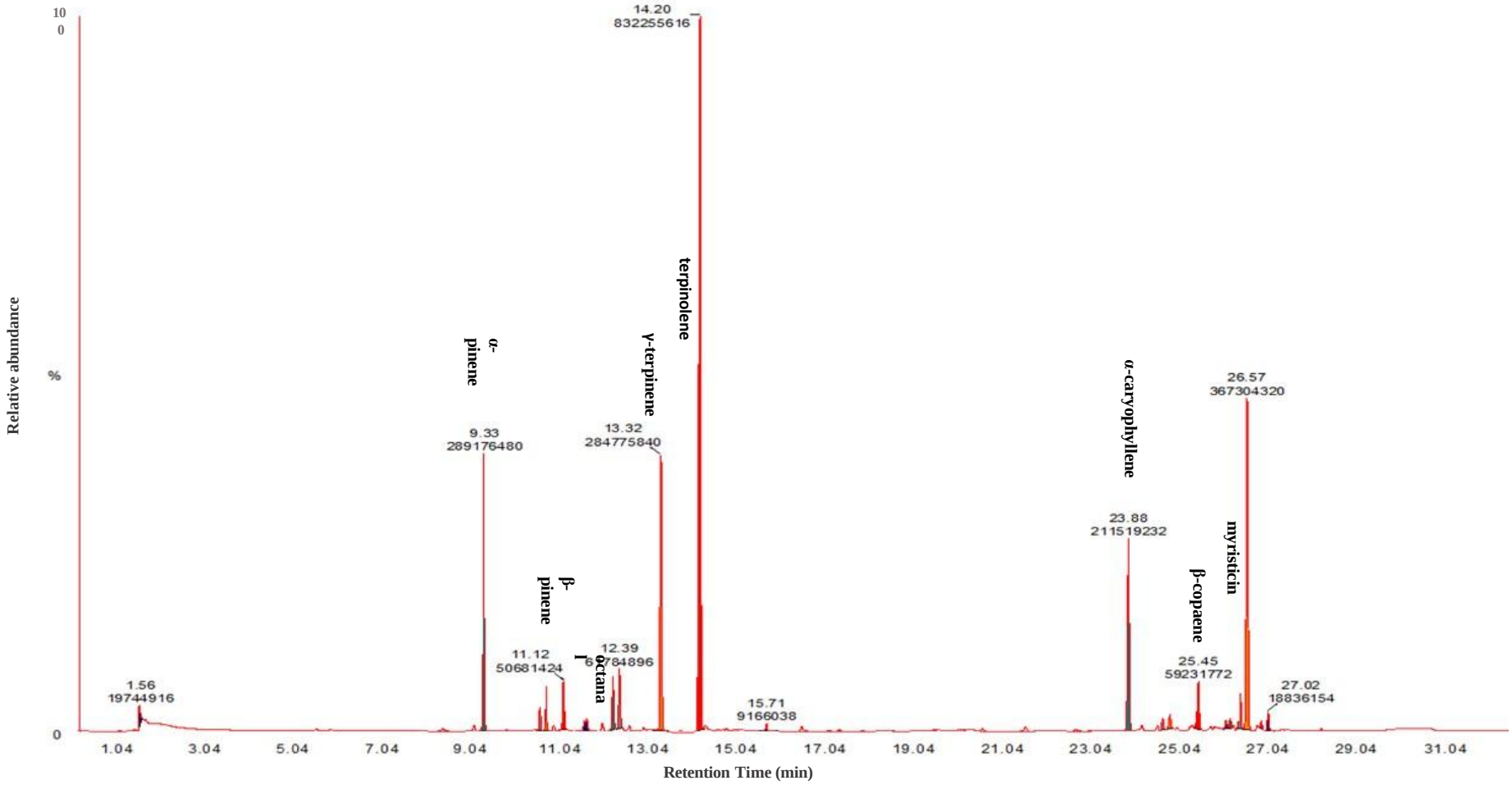
Example of Kovats retention indices

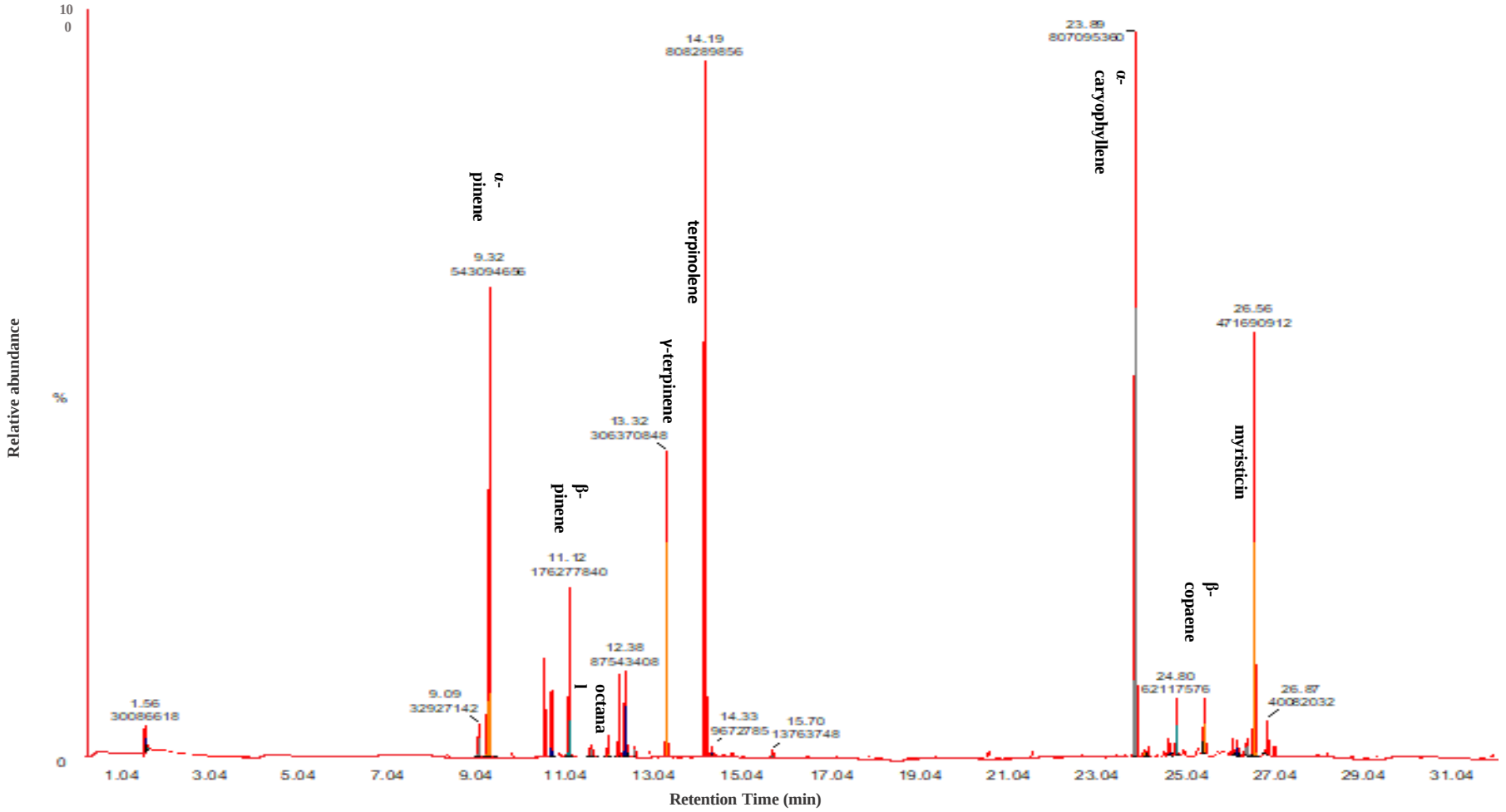
No.	Name	t_R	RI	HD	MAHD	SFME
1	α-Pinene	13.05	932	0.72	0.67	0.83
2	Camphene	13.91	948	0.13	0.01	0.02
3	Sabinene	15.19	972	1.05	1.10	1.37
4	β-Pinene	15.40	976	1.39	1.32	1.74
5	Myrcene	16.16	990	0.64	0.67	0.94
6	α-Terpinene	17.55	1017	0.03	0.03	0.09
7	D-Limonene	18.21	1030	1.53	1.52	2.15
8	1,8-Cineol	18.38	1033	17.14	17.56	19.85
9	γ-Terpinene	19.76	1059	0.05	0.08	0.18
10	Sabinene hydrate	20.44	1072	0.31	0.37	0.38
11	Terpinolene	21.15	1085	0.06	0.06	0.11
12	β-Linalool	22.03	1102	0.10	0.09	0.14
13	*cis*-Sabinol	23.99	1142	0.30	0.23	0.29
14	*trans*-Verbenol	24.30	1148	0.20	0.16	0.16
15	Menthone	24.77	1158	1.22	1.28	1.25
16	*iso*-Menthone	25.21	1167	0.65	1.06	1.02
17	Borneol	25.52	1173	1.25	0.83	0.92
18	*trans*-Isopulegone	25.75	1177	1.37	1.04	0.89
19	Terpinen-4-ol	25.98	1182	0.27	0.27	0.44
20	α-Terpineol	26.73	1197	2.14	2.28	2.51
21	Verbenone	27.40	1211	0.14	0.16	0.26
22	Pulegone	28.85	1244	61.66	54.88	38.93
23	Carvone	29.02	1247	-	0.11	0.29
24	Piperitone oxide	29.50	1256	0.08	0.22	0.41
25	*cis*-Carvone oxide	29.99	1262	0.06	0.06	0.12
26	*iso*-Piperitenone	30.33	1272	0.05	0.17	0.30
27	Piperitenone	33.35	1341	1.31	1.73	2.14
28	Piperitenone oxide	34.32	1364	3.88	9.77	17.75
29	β-Caryophyllene	36.72	1420	0.64	0.66	1.59
30	Germacrene D	39.29	1481	0.25	0.31	0.73
31	γ-Gurjunene	39.89	1496	0.07	0.12	0.35
32	γ -Cadinene	40.60	1514	0.08	0.08	0.22
33	τ-Cadinol	45.64	1644	0.45	0.52	0.75
34	Prasterone acetate	60.02	-	0.12	0.16	0.31
35	Sclareol	61.56	-	0.63	0.40	0.57
	Total monoterpenes (1-28)			97.48	97.53	95.48
	Total sesquiterpenes (29-33)			1.77	1.91	3.64
	Other classes (34, 35)			0.75	0.56	0.88
	Extraction time (min)			150	40	40
	Yield %			0.56	0.60	0.55

- *The Kovats retention index or simply Kovats index is a value that identifies the relative elution times of the various compounds in gas chromatography*
- *Retention indices are retention times normalized to adjacently eluting n-alkanes.*
- *The range of the employed n-alkanes must cover the expected retention time range of all possible target compounds.*

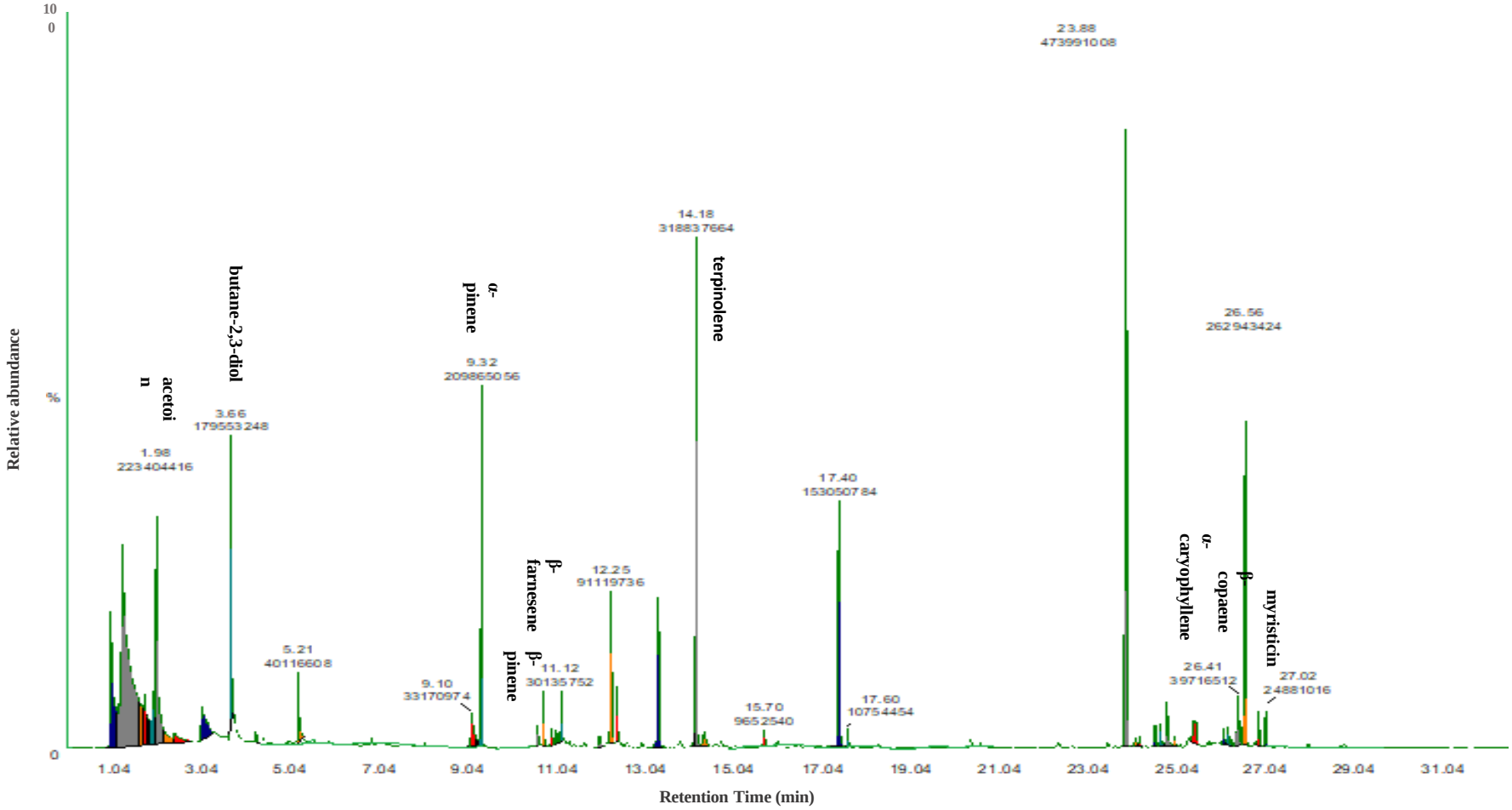
Total ion current (TIC)
Chromatogram of Carrots at 25 °C (day 1)



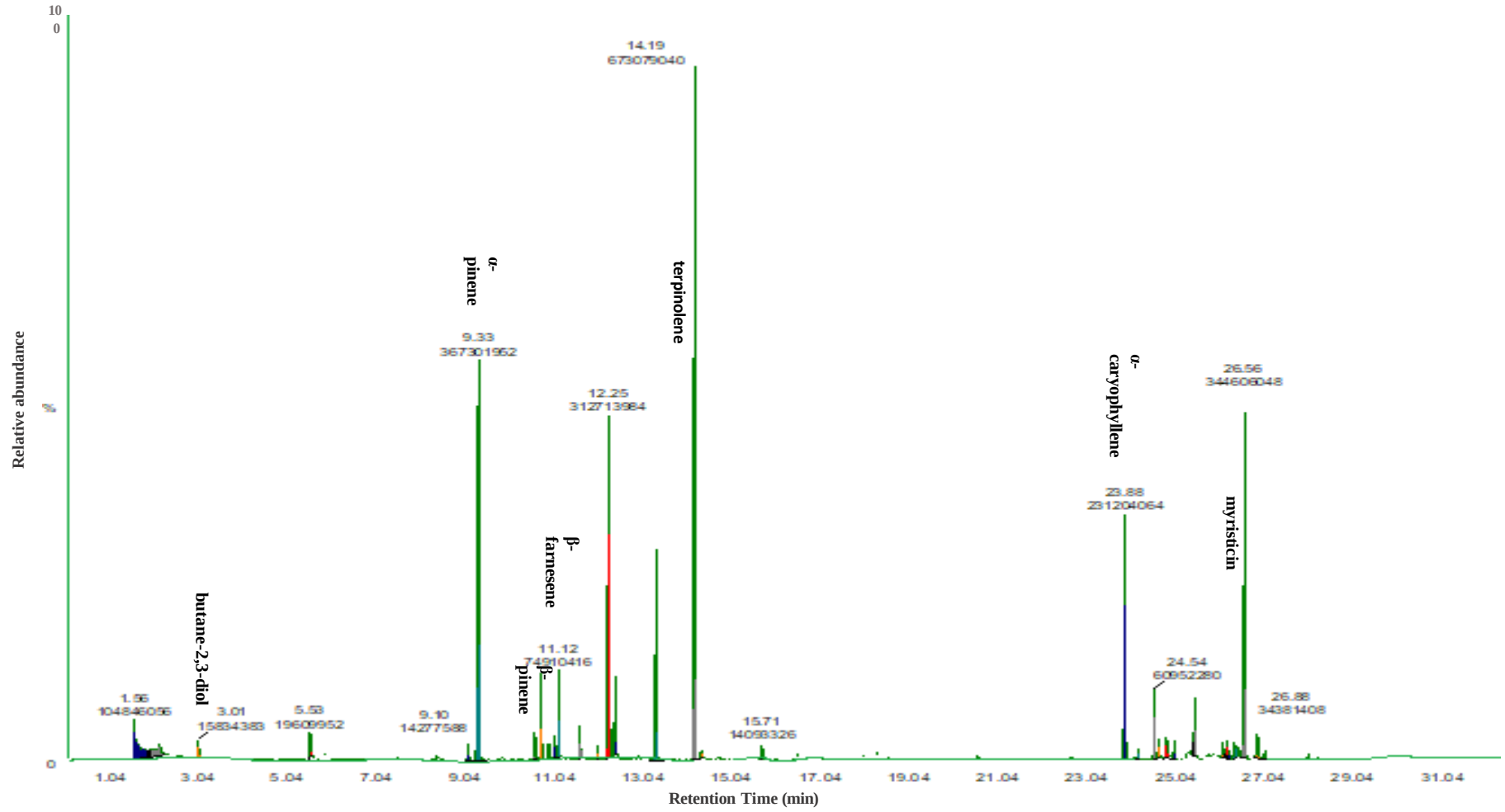
Total ion current (TIC) Chromatogram of Carrots at 4 °C (day 1)



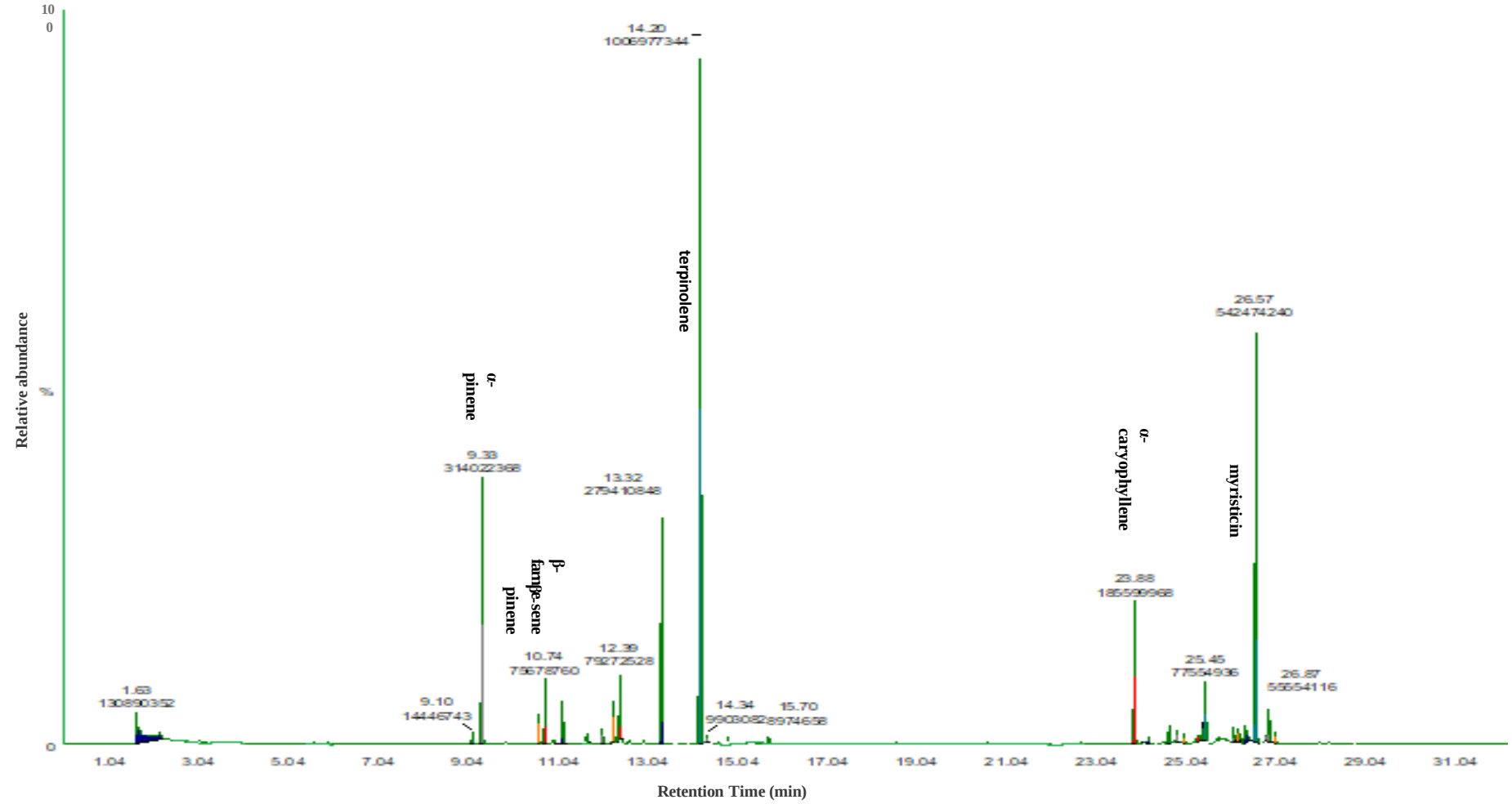
Chromatogram of Carrots at 25 °C (day 26)



Chromatogram of Carrots at 4 °C (day 26)



Chromatogram of Carrots at -18 °C (day 19)



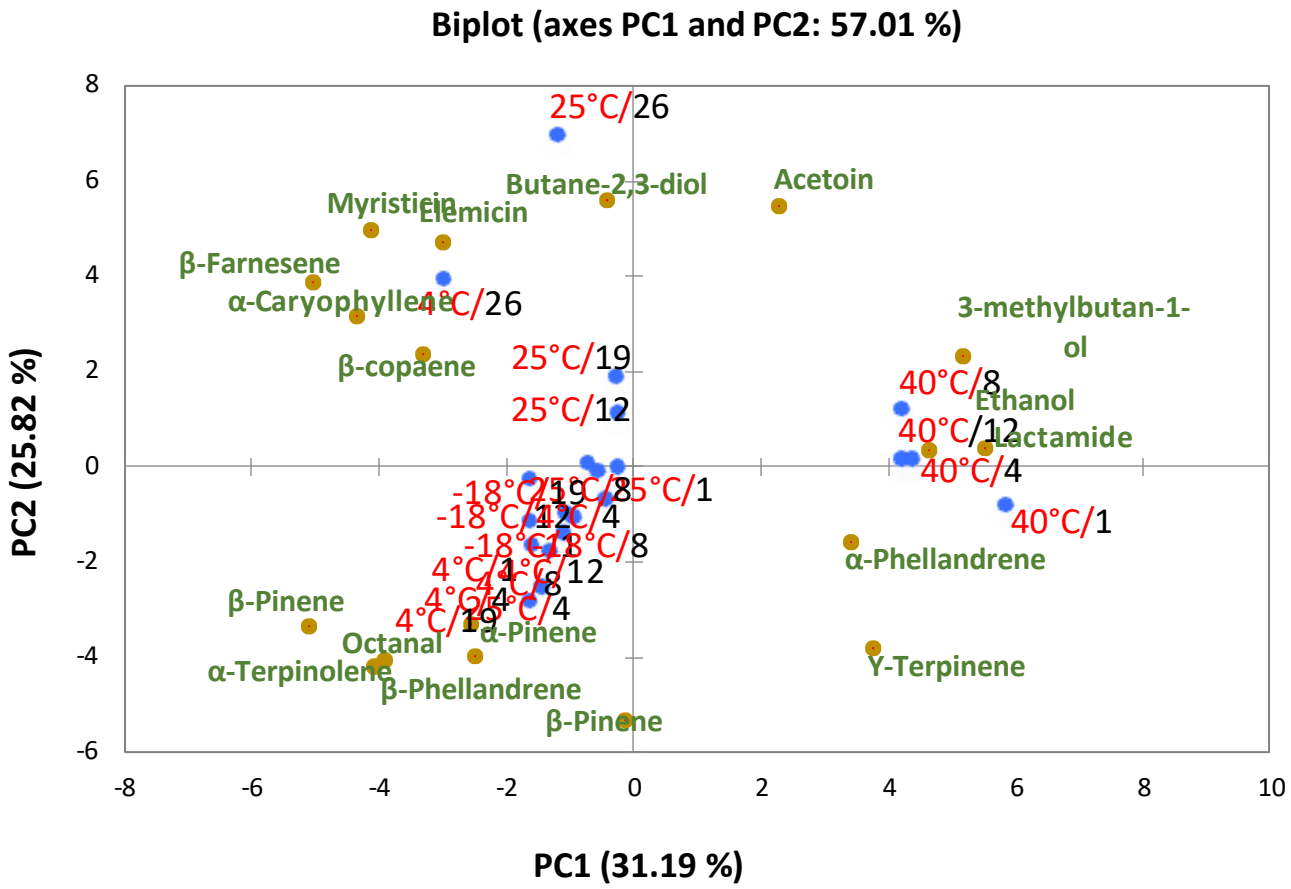
GC-MS analysis performed on the carrot samples whole set; data are expressed as % of the total GC area

Volatile compounds	GC area (%)																				
	Storage time (days)																				
	25°C						4°C						-18					40 °C			
days	1	4	8	12	19	26	1	4	8	12	19	26	1	4	8	12	19	1	4	8	12
α-phellandrene	n.d	1	n.d	n.d	1	n.d	1	1	1	1	1	n.d	n.d	n.d	n.d	n.d	n.d	7	1	n.d	n.d
β-phellandrene	n.d	3	1	1	2	n.d	3	3	2	1	4	1	2	1	2	1	1	n.d	n.d	n.d	n.d
terpinolene	n.d	1	n.d	n.d	1	n.d	1	1	1	n.d	1	n.d	n.d	n.d	1	1	0	n.d	n.d	n.d	n.d
α-pinene	12	7	12	11	15	2	14	12	14	14	18	8	14	9	12	12	10	n.d	10	15	5
(-) -β-pinene	2	3	2	2	3	1	3	3	3	3	3	2	3	2	3	3	3	n.d	n.d	n.d	2
β-pinene	2	6	1	1	4	n.d	5	6	3	3	5	1	3	2	3	2	6	5	3	3	2
octanal	1	n.d	n.d	n.d	n.d	n.d	1	1	1	1	n.d	n.d	1	1	n.d	n.d	n.d	n.d	n.d	n.d	n.d
γ-terpinene	12	7	6	5	7	n.d	8	9	7	8	4	1	7	8	9	10	7	21	9	9	5
β-farnesene	1	1	1	1	1	2	1	1	1	1	n.d	1	1	1	1	1	1	n.d	n.d	n.d	n.d
α-caryophyllene	1	2	1	2	1	2	2	1	1	1	1	5	1	1	1	1	2	n.d	n.d	n.d	n.d
β-copaene	2	1	2	2	2	5	2	2	3	n.d	n.d	n.d	2	2	2	n.d	4	n.d	n.d	n.d	n.d
myristicin	2	1	2	n.d	1	4	1	1	1	1	1	5	1	1	1	1	2	n.d	n.d	n.d	n.d
elemicin	1	n.d	1	1	n.d	2	n.d	n.d	1	n.d	n.d	4	n.d	1	n.d	n.d	n.d	n.d	n.d	n.d	n.d
butane-2,3-diol	n.d	n.d	2	2	5	13	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	3
acetoin	n.d	n.d	9	3	6	15	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	5	3	5	6
ethanol	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	5	n.d	n.d	6
lactamide	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	4	11	16	7
3-methylbutan-1-ol	n.d	n.d	n.d	n.d	1	2	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	1	3	3	3

Principal Component Analysis GC-MS

Score plot: samples at 25°C/12, 25°C/19, 25°C/26, 4°C/26 and all 40°C samples are discriminated

Loading plot: Major contribution of the VOCs to the relative sample (i.e. 40° samples produce different molecules from day 1)



UNIVERSITA DEGLI STUDI DI TERAMO

Facoltà di Bioscienze e tecnologie agroalimentari e ambientali

Quality of Pasta samples

- Drying is the most important unit operation in pasta;
- high-temperature (HT) drying technology has been widely applied by pasta manufacturers;
- HT drying has a positive influence on the mechanical properties of pasta;
- To avoid Maillard's reaction as much as possible, it is necessary to monitor volatile compounds, which may be markers of the quality of the finished product.

The samples were inserted into a 20 mL vial hermetically closed with a screw cap and a silicone septum. A Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber 50/30 µm (Supelco, Bellefonte, PA) was used for the extraction; exposition of the fiber in the headspace for 40 min at 40 °C was used for samples.

The fiber was inserted in the injection port of the GC (operating in splitless mode). GC separation of compounds was carried out initially at 40 °C for 10 min, then raising to 230 °C at 4 °C/min.

UNIVERSITÀ DEGLI STUDI DI TERAMO

Pasta samples

Facoltà di Bioscienze e tecnologie agroalimentari e ambientali

High price

Volatile compounds																		
	A	A	A	B	B	B	C	C	C	D	D	D	E	E	E	F	F	F
3-methy l-f uran	-	-	-	-	-	-	-	-	1±1.15	-	-	-	-	-	-	-	-	-
pentanal	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
lattamide	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1,3-butanediol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2-f uranmethanol	-	-	-	-	-	-	-	-	-	1±0.58	-	-	-	7±0.6	-	-	-	-
f urf ury l alcohol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
hexanal	24±1.5	30±2.65	23±1.53	15±2.08	41±4.04	7±2.00	-	2±2.08	-	-	2±2.31	11±1.00	3±2.52	-	1±1.53	3±1.15	1±0.58	1±0.58
2-hexenal	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
benzaldehy de	1±1.15	3±1.00	-	2±2.08	7±2.00	2±1.52	-	-	-	-	-	-	-	-	-	-	-	-
4-methy lbenzaldehy de	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
3-methy lbenzaldehy de	-	-	-	-	-	-	-	-	-	3±1.00	-	-	-	10±1.52	6±1.00	4±0.57	5±1.15	6±1.00
2-penty lf uran	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
nonanal	6±1.00	3±1.52	13±1.53	15±4.50	10±1.00	5±4.00	2±1.52	2±1.15	4±2.00	4±1.52	1±1.15	5±2.00	4±1.52	-	-	-	-	-
2-nonenal	10±2.00	7±0.57	4±3.51	5±3.00	1±1.00	-	-	-	-	-	-	1±1.52	-	-	-	-	-	-
decanal	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	7±4.5
3,7-dimethy l-1-octanol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2,2,4,4-tetramethy ltetrahy drofuran	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1±1.53	1±2.08	1±1.33	15±3.51
2-isopropy l-5-methy l-1-heptanol	-	-	-	-	-	-	-	-	-	-	-	-	3±3.00	6±0.57	6±1.18	9±3.00	10±3.08	14±3.05
2-buty l-1-octanol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1,1-dodecanediol	-	1±0.59	-	1±1.53	2±3.05	-	-	-	-	-	-	1±1.15	-	-	-	-	-	-

Volatile compounds of low-price pasta samples. Data were expressed as % of the total GC area

UNIVERSITÀ DEGLI STUDI DI TERAMO

Pasta samples

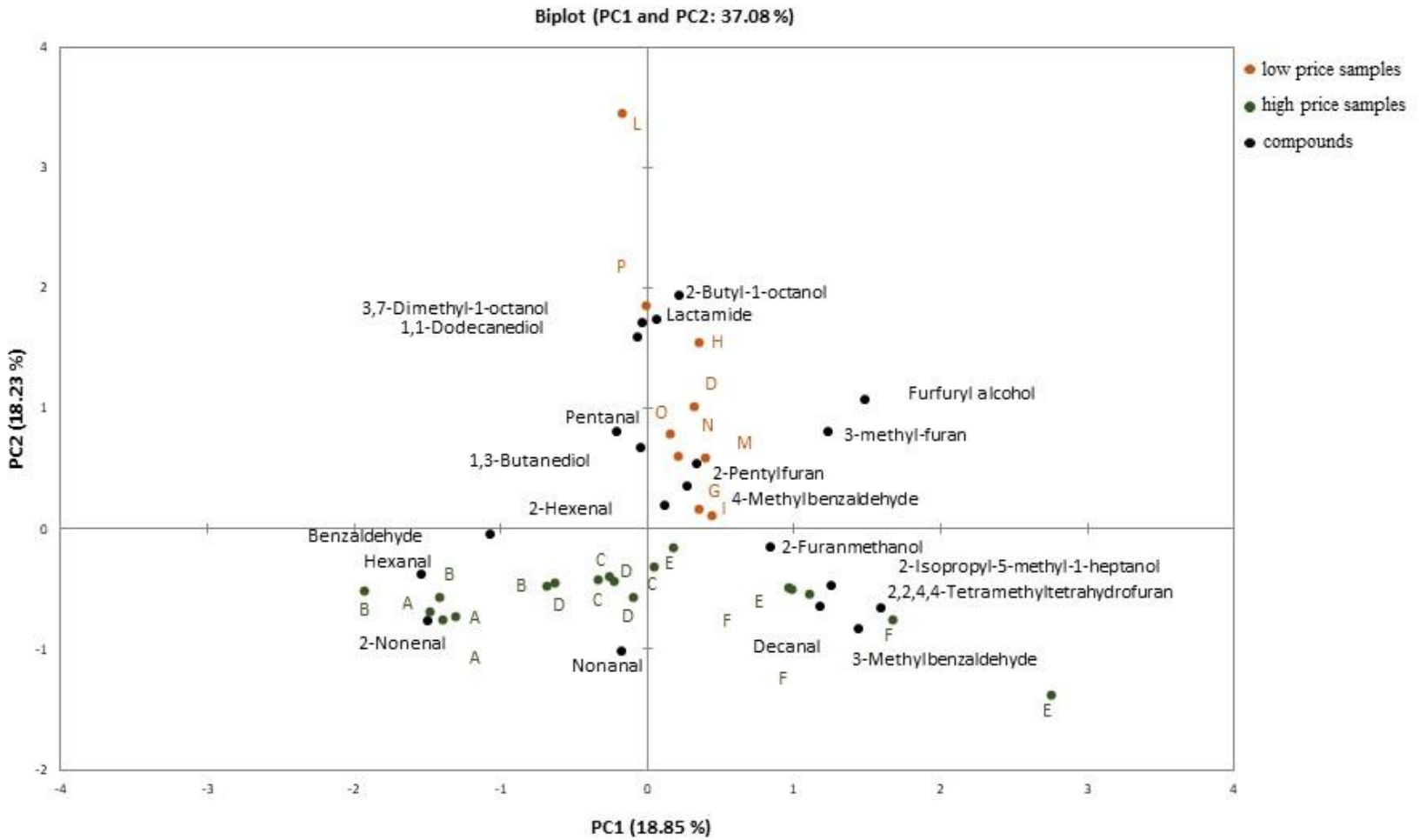
Low price

Facoltà di Bioscienze e tecnologie agroalimentari e ambientali

Volatile compounds	G	H	I	L	M	N	O	P	Q
3-methyl-furan	-	-	-	-	-	-	-	-	-
Pentanal	2±1.00	-	-	-	-	-	-	-	-
lattamide	8±3.00	20±4.51	-	-	-	-	-	14±2.00	-
1,3-butanediol	5±4.04	15±3.51	-	-	-	-	-	-	-
2-furanmethanol	-	-	1±1.53	-	-	-	-	-	-
furfuryl alcohol	-	-	-	-	-	-	1±1.58	1±1.00	-
hexanal	10±2.52	6±0.58	2±2.08	6±3.60	4±1.00	4±1.00	1±0.58	2±2.08	3±2.08
2-hexenal	-	-	1±0.58	-	-	2±1.53	-	1±1.15	-
benzaldehyde	-	1±1.53	1±1.53	1±1.53	1±2.08	1±1.15	1±1.15	1±1.00	-
4-methylbenzaldehyde	4±0.58	-	-	-	7±1.53	15±1.53	11±1.00	7±1.53	-
3-methylbenzaldehyde	-	5±1.00	8±2.00	4±2.00	-	-	-	-	-
2-pentylfuran	2±2.52	-	1±2.08	-	2±2.08	-	-	-	-
nonanal	-	1±0.58	1±0.58	-	2±0.58	-	-	1±1.15	1±0.58
2-nonenal	-	-	-	-	-	-	-	-	-
decanal	-	-	-	-	-	-	-	-	-
3,7-dimethyl-1-octanol	-	5±1.73	5±2.52	4±2.65	-	-	-	-	-
2,2,4,4-tetramethyltetrahydrofuran	-	-	-	-	-	-	-	-	-
2-isopropyl-5-methyl-1-heptanol	4±1.00	-	-	1±1.00	-	-	-	-	-
2-butyl-1-octanol	3±2.08	5±1.15	5±0.58	4±3.61	4±2.65	4±1.57	4±4.16	3±2.08	-
1,1-dodecanediol	-	-	8±1.00	-	-	-	-	1±1.53	-

Volatile compounds of high-price pasta samples. Data were expressed as % of the total GC area.

Principal Component Analysis GC-MS



PCA of the GC-MS response for low price samples (orange) and high price samples (green). The biplot (Score and loading) of the first two principal components show ed 37.08% of the cumulative variance. Data w ere expressed in (R.A. %) before PCA

GC x GC

GCxGC, or two-dimensional gas chromatography (2D-GC), is an advanced chromatographic technique used primarily for separating and analyzing complex mixtures of compounds, particularly those with many chemically similar components. GCxGC improves on traditional gas chromatography (GC) by adding a second dimension of separation, which helps resolve compounds that might overlap in single-dimensional GC.

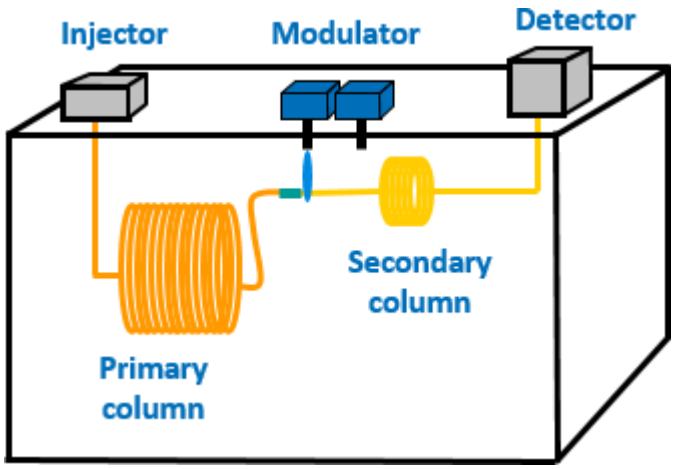
How GCxGC Works

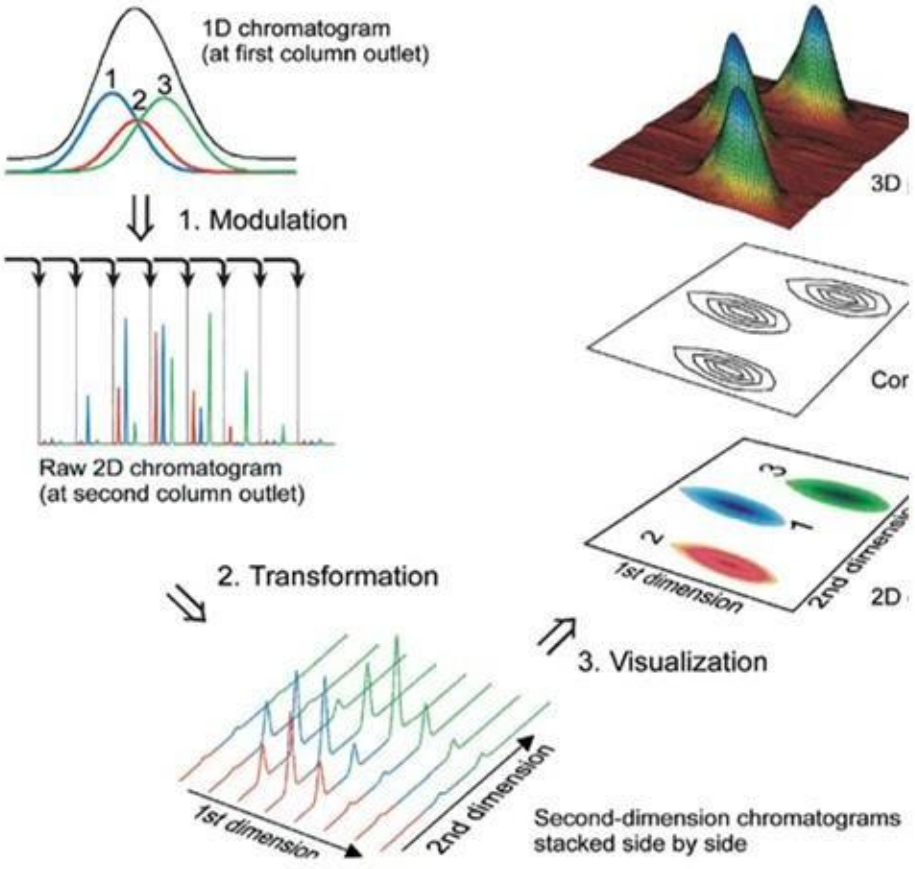
Two-Dimensional Separation: In GCxGC, the sample is first separated in a primary GC column, and then each fraction from this first column is further separated in a second GC column. The two columns have different stationary phases, which means they separate compounds based on different chemical properties. The first column typically separates based on boiling points, while the second column often separates by polarity or another property.

Modulator: Between the two columns, a device called a modulator captures the eluting fractions from the first column in a series of short pulses and releases them into the second column. This is essential for transforming the continuous stream from the first dimension into a time-structured input for the second.

Comprehensive Data: The two-dimensional separation produces a chromatogram with both retention times and signal intensity across two different axes, leading to more comprehensive data. The result is typically a contour plot or a 3D plot where peaks represent individual compounds.

GCxGC Chromatography





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Screening of volatile compounds composition of white truffle during storage by GCxGC-(FID/MS) and gas sensor array analyses

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White truffle (*Tuber magnatum Pico*) is one of the most valuable delicacies of the Italian and French cuisine. Previous works on truffle aroma report sulfur compounds and short-chain aldehydes as typical constituents which abundance is influenced by storage conditions. In this study, the head space of truffle aroma has been evaluated by two different (innovative) approaches: HS-SPME extraction and comprehensive two-dimensional gas chromatography (GCxGC), exploiting a dual-stage loop-type modulator

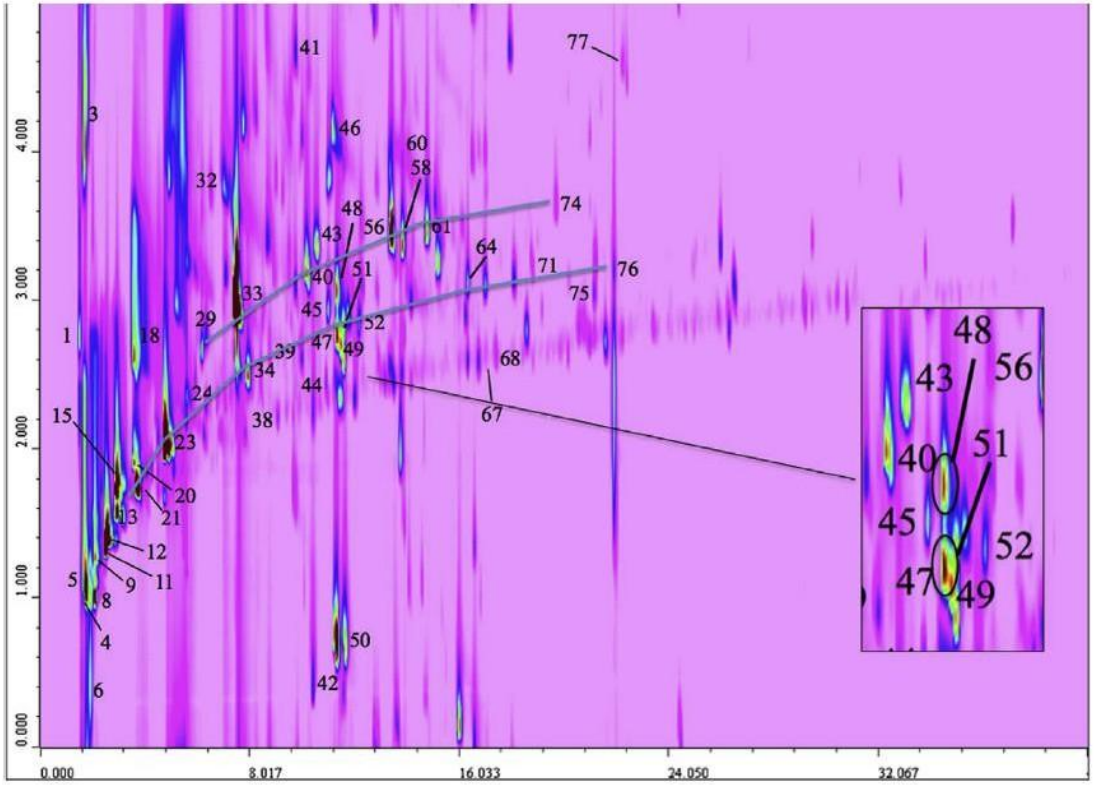

Fig. 1. HS-SPME-GCxGC chromatogram of Italian white truffle (*Tuber magnatum Pico*) sample. See Table 2 for peak identification. Top trend line: monounsaturated aldehydes. Bottom trend line: linear aliphatic aldehydes. The expansion shows the separation of peaks #47 and #48.

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Comparison of fatty acid methyl esters of palm and palmist oils determined by GCxGC–ToF–MS and GC–MS/FID

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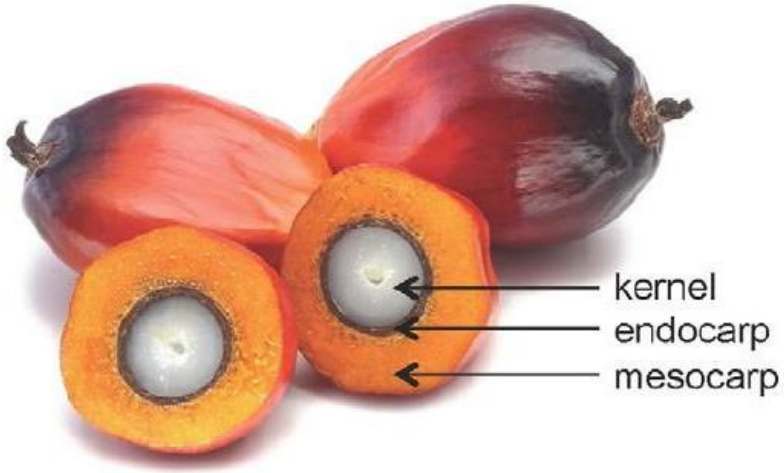

Fig. 1. The source of palm oil (mesocarp) and palmist oil (kernel).

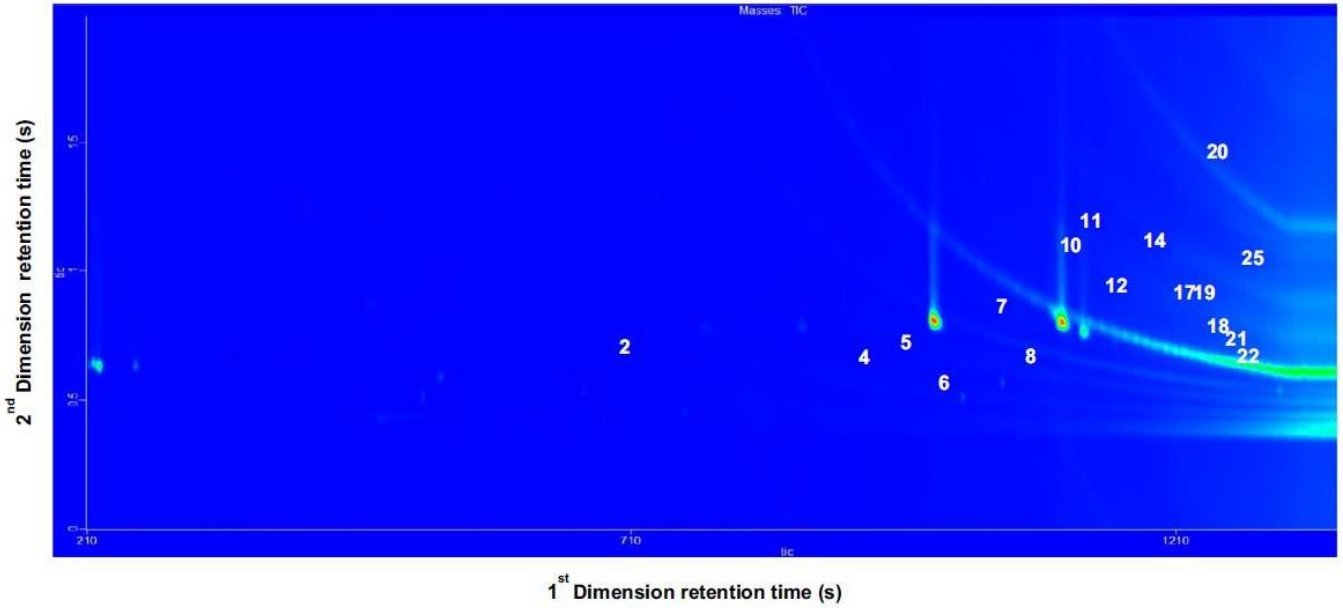

Fig. 2. Two dimensional contour plot of palm oil commercially available from Cameroon. For corresponding FAME, refer to peak number in Table 1.

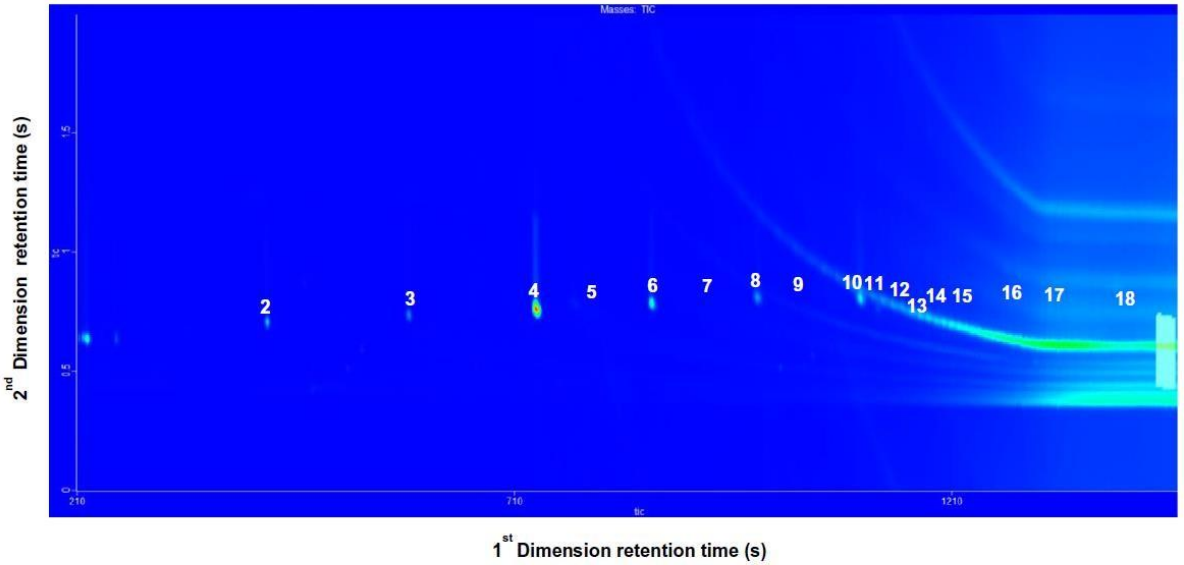

Fig. 3. Two dimensional contour plot of commercially available palmist oil. For corresponding FAME, refer to peak number in Table 2.