



Techniques for Detection of Food Aroma Compounds

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Food flavors

**Volatiles
(aroma active)**

**Nonvolatiles
(taste active)**

predominant contributors

acids, neutral compounds, sulfur and nitrogen compounds, alcohols, aldehydes, ketones, hydrocarbons, esters...

❖ >6000 compounds have been identified in volatile fraction of foods.

Classes of Aroma Compounds in Coffee

Chemical class	Number of compounds
Hydrocarbons	74
Alcohols	20
Aldehydes	30
Ketones	73
Acids	25
Esters	31
Lactones	3
Phenols (and ethers)	48
Furans	127
Thiophenes	26
Pyrroles	71
Oxazoles	35
Thiazoles	27
Pyridines	19
Pyrazines	86
Amines and miscellaneous nitrogen compounds	32
Sulfur compounds	47
Miscellaneous	17
<i>Total</i>	791



Monitoring the flavor quality of foods

- ❖ Raw materials: typical flavor quality, off-flavor chemicals
- ❖ Packaging materials: should be checked for residual solvents
- ❖ Processing steps: flavor/off-flavor development
- ❖ Finished products: typical flavor quality, detection of adulteration, off-flavor development during storage

The goal of the flavor research in food industry;
identify and classify aroma chemicals that contribute to
the characteristic flavor of foods

Limitations in Sample Preparation

Concentration level, matrix, complexities of aromas, reactivity and instability, separation&quantification

Detection/Analysis of Food Aroma

Instrumental techniques

Sensory analysis

Combined techniques

Instrumental / Combined methods

Separation/ Isolation

- Distillation
- Solvent extraction
- Headspace sampling
- Solid phase microextraction (SPME)

Identification/ Quantification

- Gas chromatography (GC)
- Gas chromatography-mass spectrometry (GC-MS)
- Gas chromatography-olfactometry (GC-O)
- Electronic nose (E-Nose)



Separation / Isolation Methods

Distillation

➤ Direct Distillation

(scorching of the sample, bumping, foaming problems)

➤ Indirect Steam Distillation

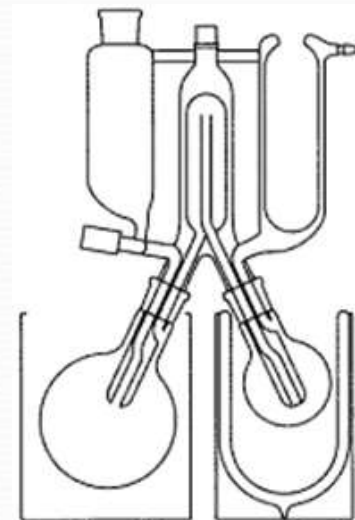
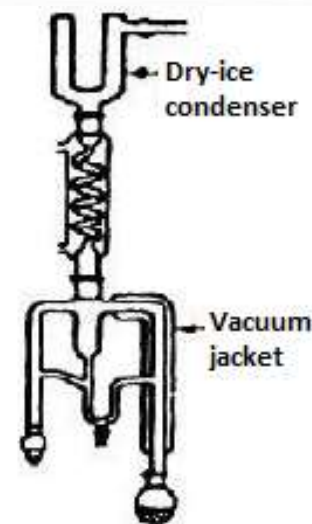
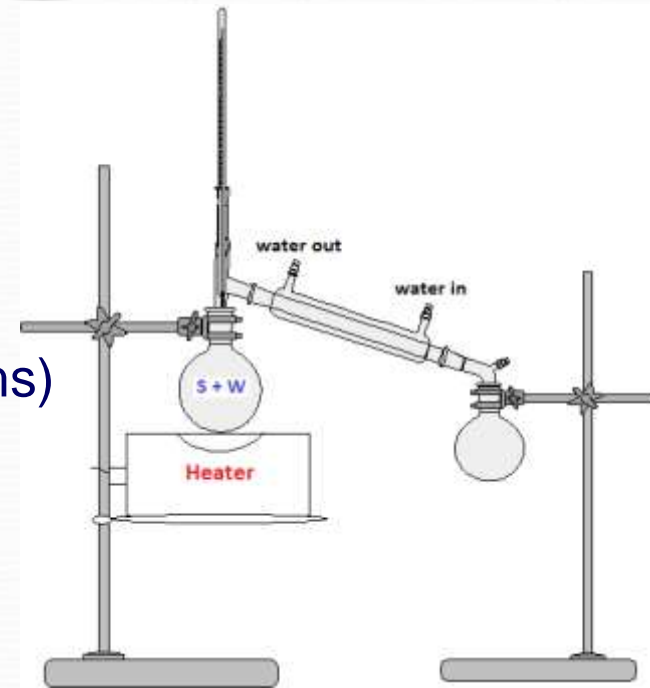
(rapid&easy, less decomposition of the sample)

➤ Vacuum Steam Distillation

(less decomposition of the sample)

➤ Simultaneous Steam Distillation /Ext.

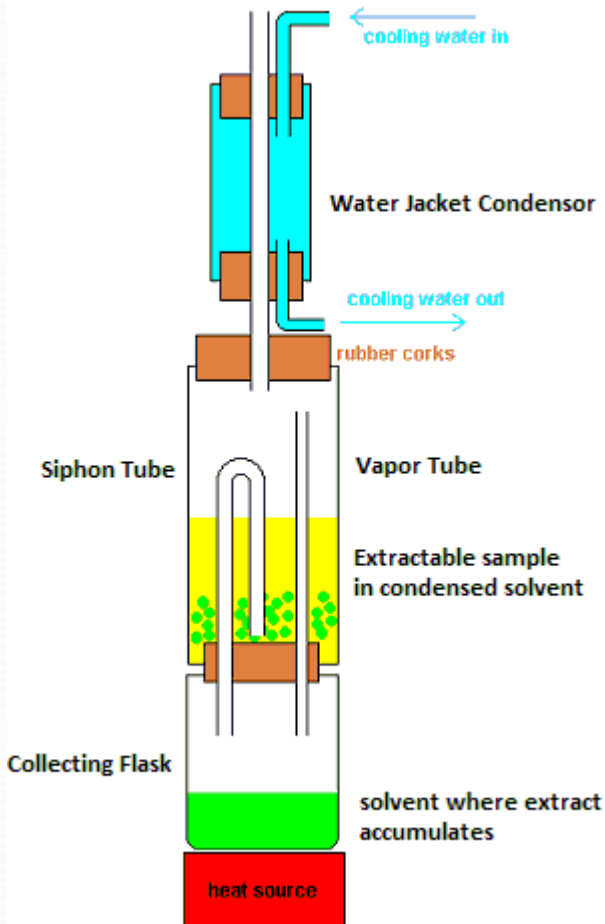
(removes and concentrates in a single step, small volume of solvents, high recovery)



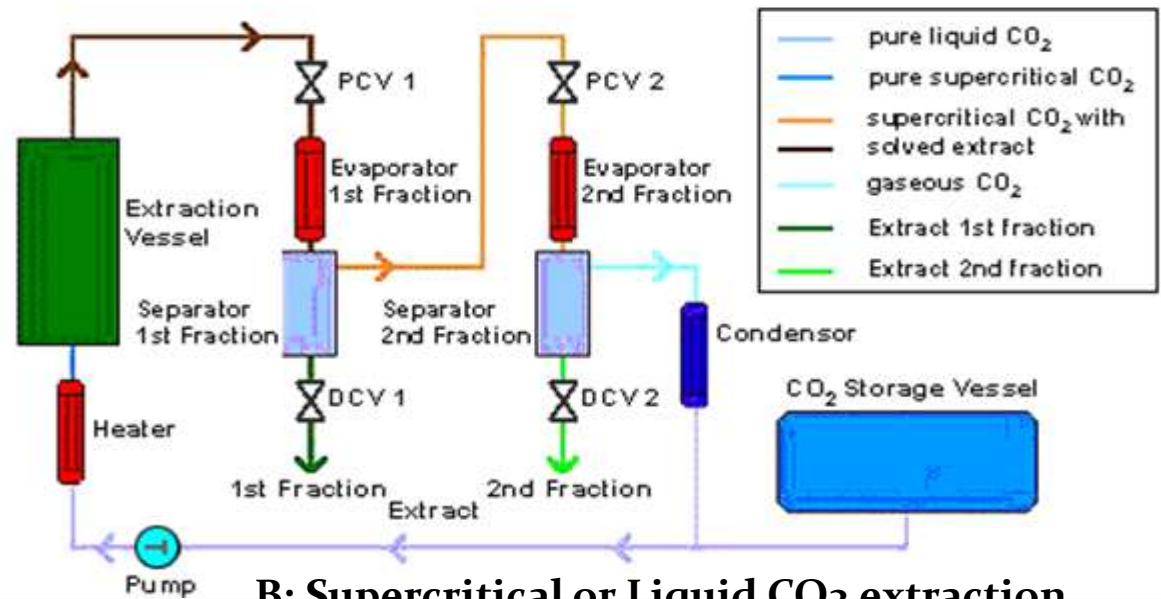
Solvent extraction

(diethyl ether, diethyl ether/ pentane mixtures, hydrocarbons, Freons, methylene chloride and supercritical carbon dioxide)

- **Direct Solvent Extraction of Aqueous Samples**
- **Direct Solvent Extraction of Solid Samples**
- **Co-distillation of Sample with Solvent**



A: Tube/Cylinder Extractor

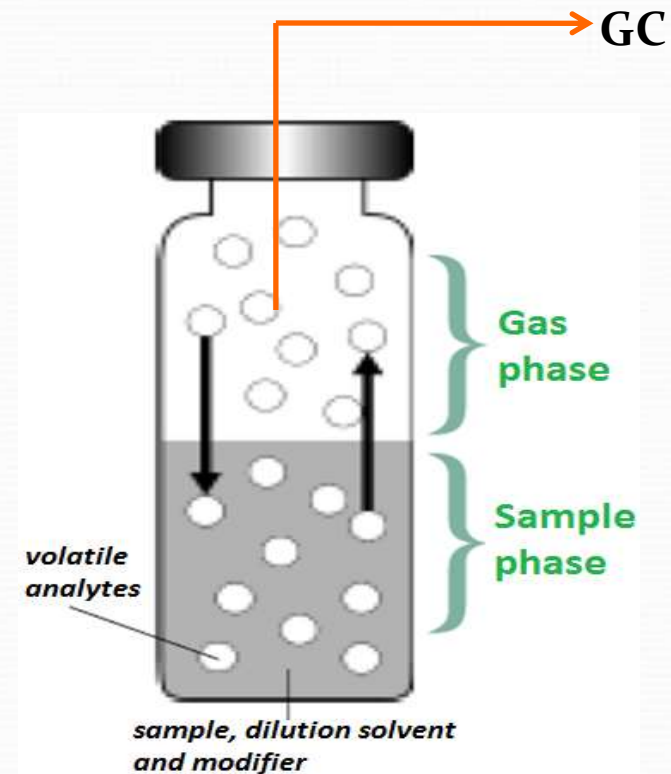


B: Supercritical or Liquid CO₂ extraction

Headspace sampling

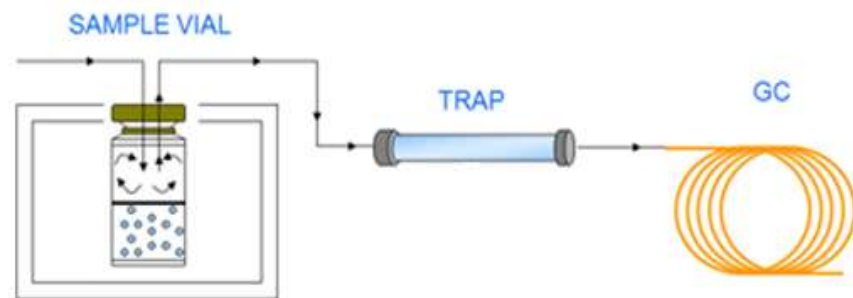
➤ Static headspace

- developed in early 60s
- measures the components in the gas present in the space above the sample
- 👍 eliminates solvent peak, attractive for sample screening, low cost
- 👎 equilibrium required, volatility and concentration limitations (head-space contains volatiles at 10^{-4} to 10^{-10} g/L)



Headspace sampling

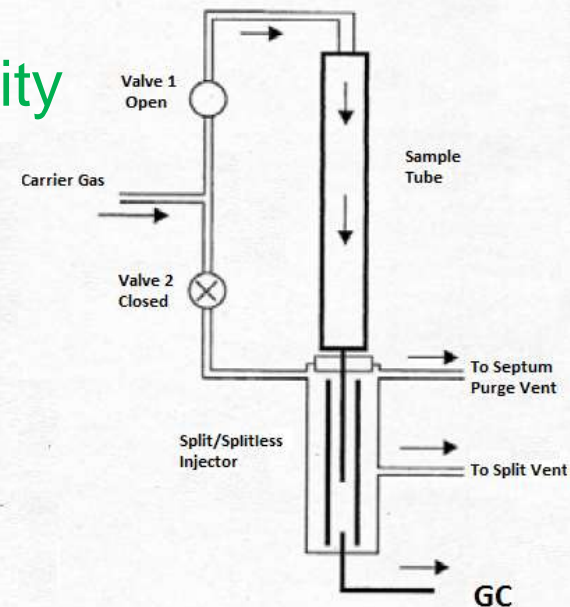
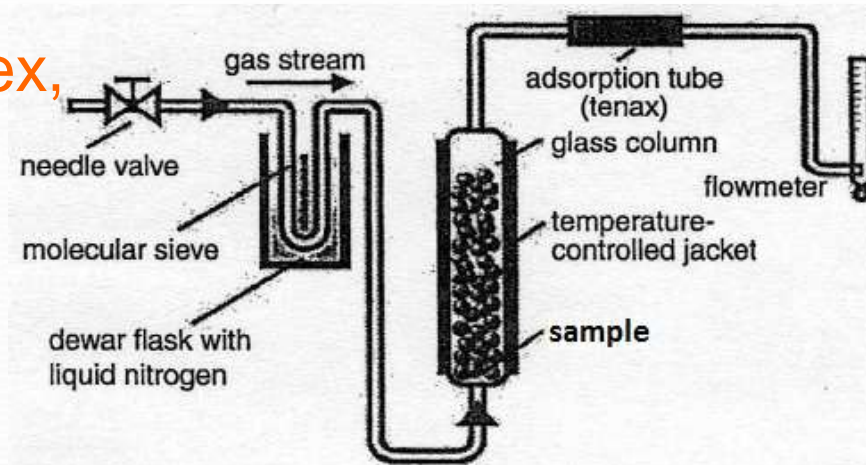
➤ Dynamic headspace (purge&trap)



- developed in early 70s, purging gas strips out the volatiles which are concentrated on a sorbent trap that is then rapidly heated for desorption

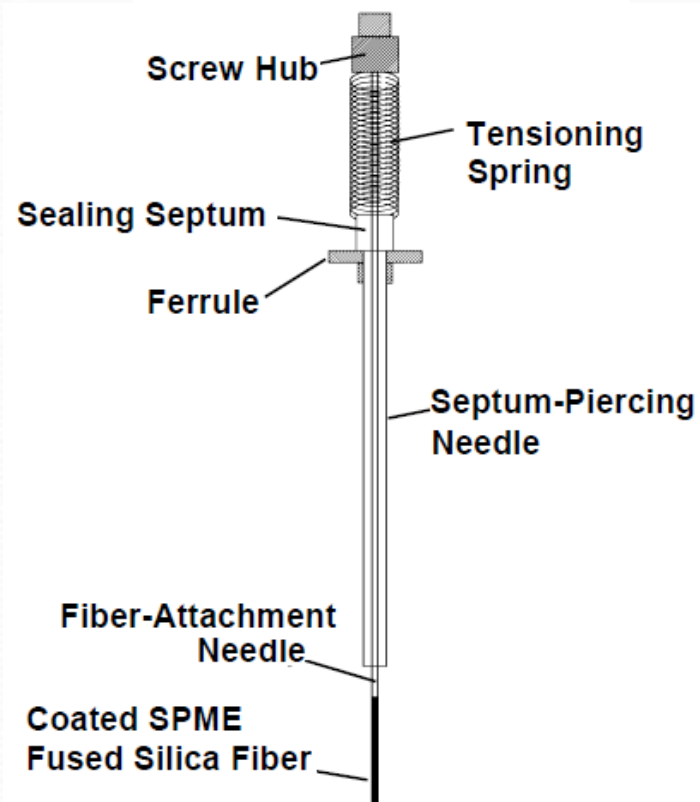
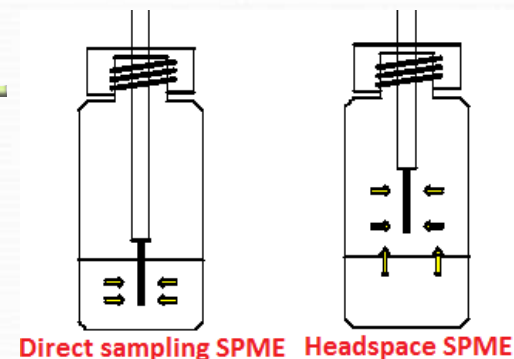
👍 eliminates solvent peak, sensitivity, selectivity

👎 more complex, expensive



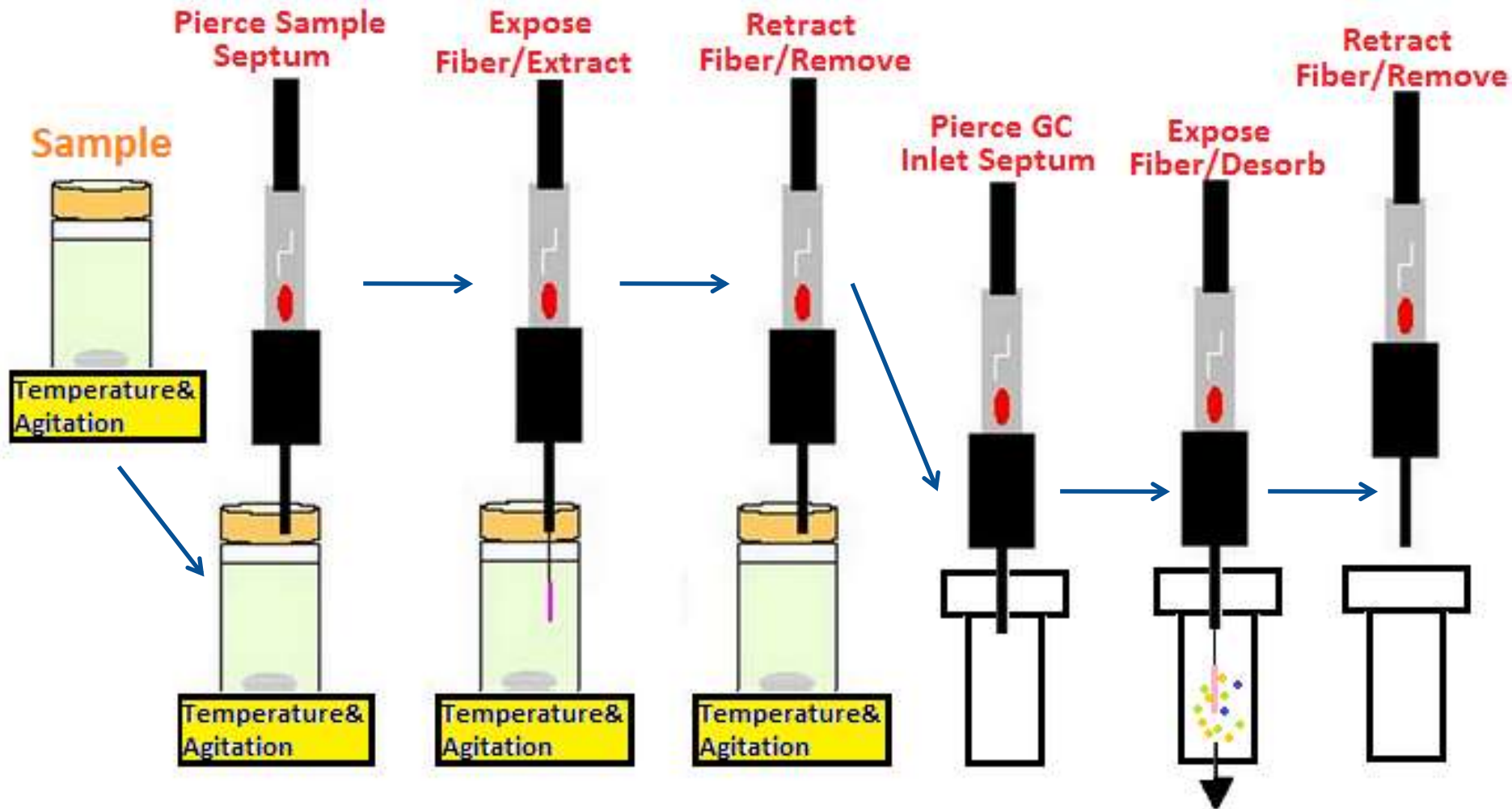
Solid-Phase Microextraction

- Equilibrium method
- Solvent-free sample preparation
- Fast, precise, relatively simple
- Strict control of parameters such as temperature, sample amounts, vessel volume, pressure
- Coating dependent-solubility
- Capacity of fiber



Solid-Phase Microextraction

Extraction and Desorption

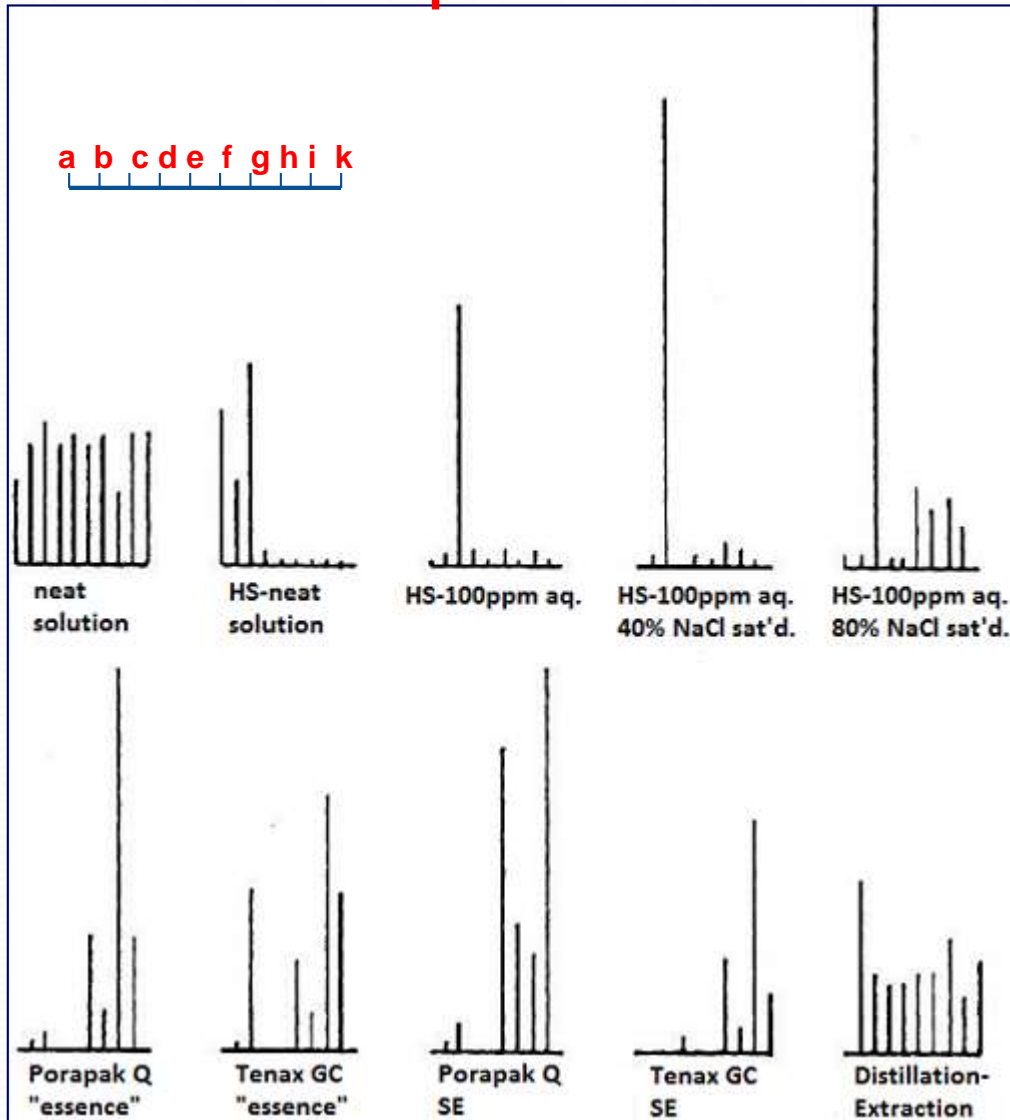


Solid-Phase Microextraction

Types of Solid Phases for SPME

- ❖ Polydimethylsiloxane (PDMS)
- ❖ Polyacrylate (PA)
- ❖ Polyethyleneglycol (PEG)
- ❖ Carboxen-polydimethylsiloxane (CAR-PDMS)
- ❖ Polydimethylsiloxane-divinylbenzene (PDMS-DVB)
- ❖ Divinylbenzene/Carboxen- Polydimethylsiloxane (DVB-CAR-PDMS)

Comparison of some methods used for aroma compound isolation



Detection Limits and Precision of Organic Volatile in Water

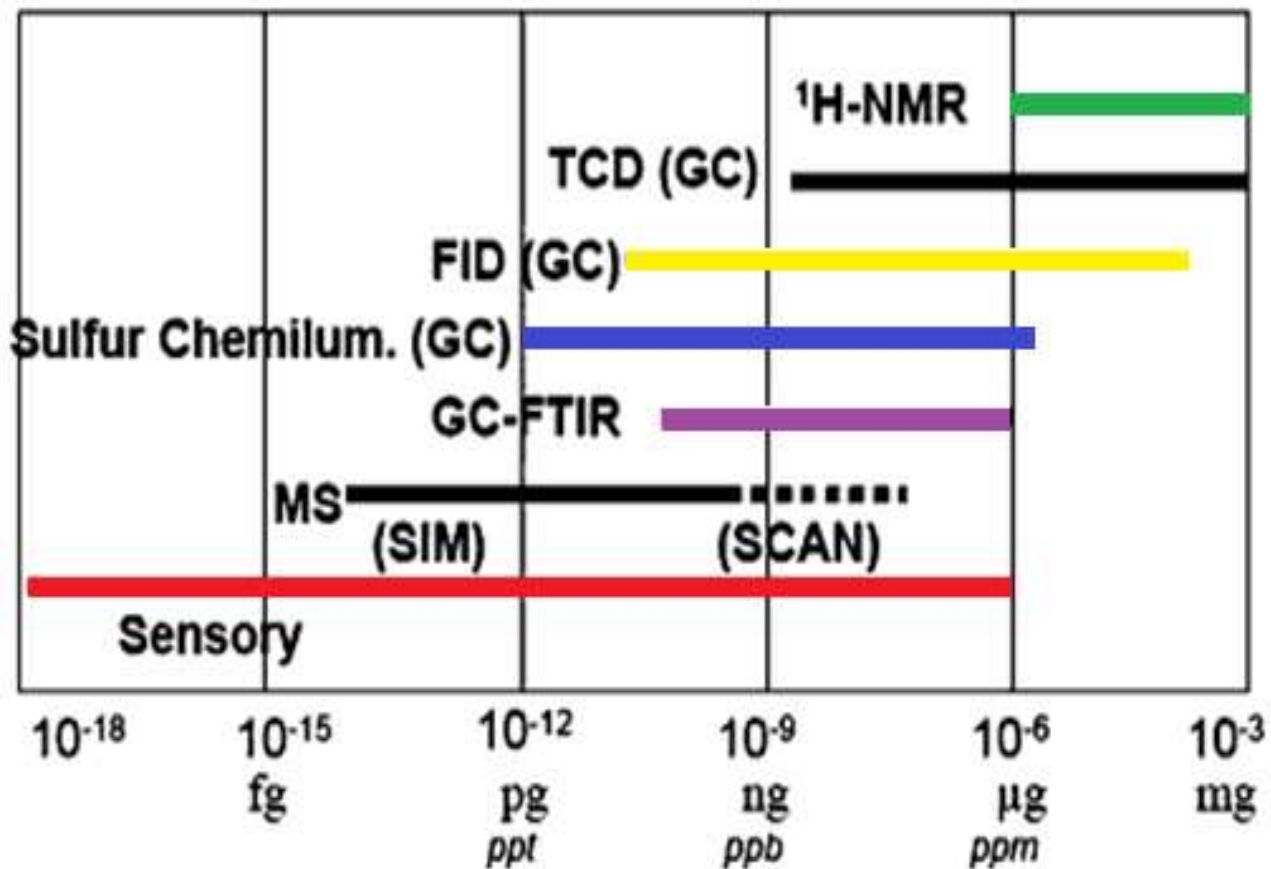
Technique	Detection limit with FID (ppb)	Precision (%rsd)
SPME	0.05-0.3	1-3
Static Headspace	1-2	1-3
Dynamic Headspace	0.003-0.005	1-8
Direct Injection	17-240	2-3

Identification/Quantification

GC-FID, GC-TCD,
GC-FTIR, GC-SCD
GC-MS, GC-MS-MS
GCxGC...

GC-O
GC-MS-O
e-nose

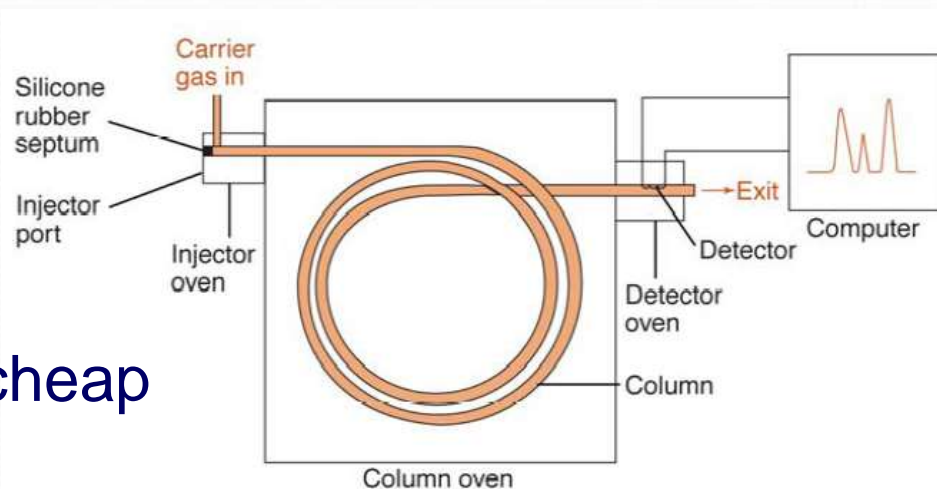
MS based e-nose...



GC

GC is premier technique for separation and analysis of volatile compounds

- Fast analysis
- Small samples (μl or μg)
- High resolution
- Reliable, relatively simple and cheap
- Non-destructive
- Allows on-line coupling, e.g. to MS
- Sensitive detectors (easy ppm, often ppb)
- Highly accurate quantification (1-5% RSD)

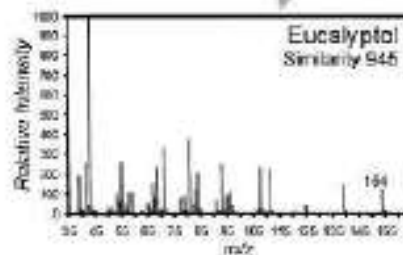
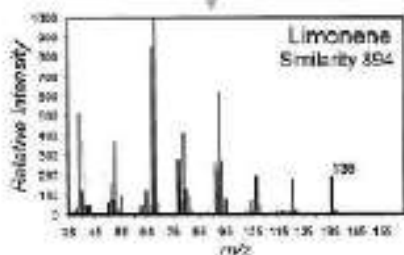
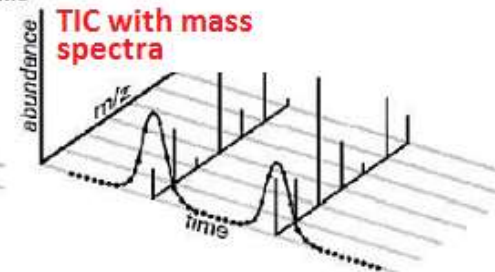
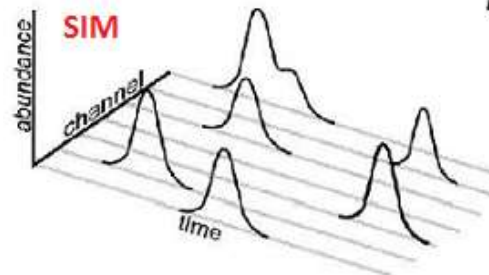
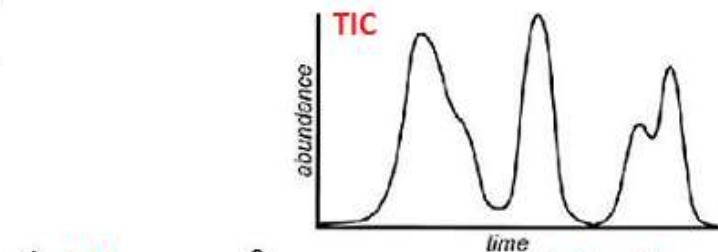
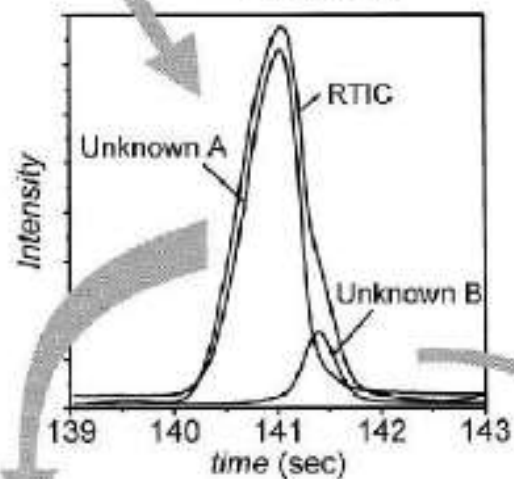
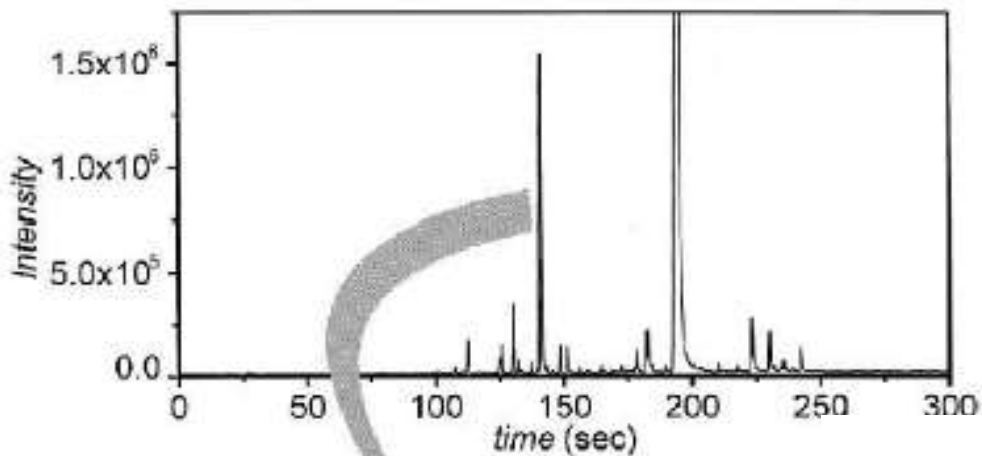


GC-MS

MS provides information that aids in the structural identification of each component. Gas-phase ions are separated according to mass/charge ratio and sequentially detected.

Parts of MS

- Sample introduction
- Source (ion formation, EI)
 - Mass analyzer - high vac (Quadrupole, ion trap, TOF....)
- Detector (electron multiplier tube)

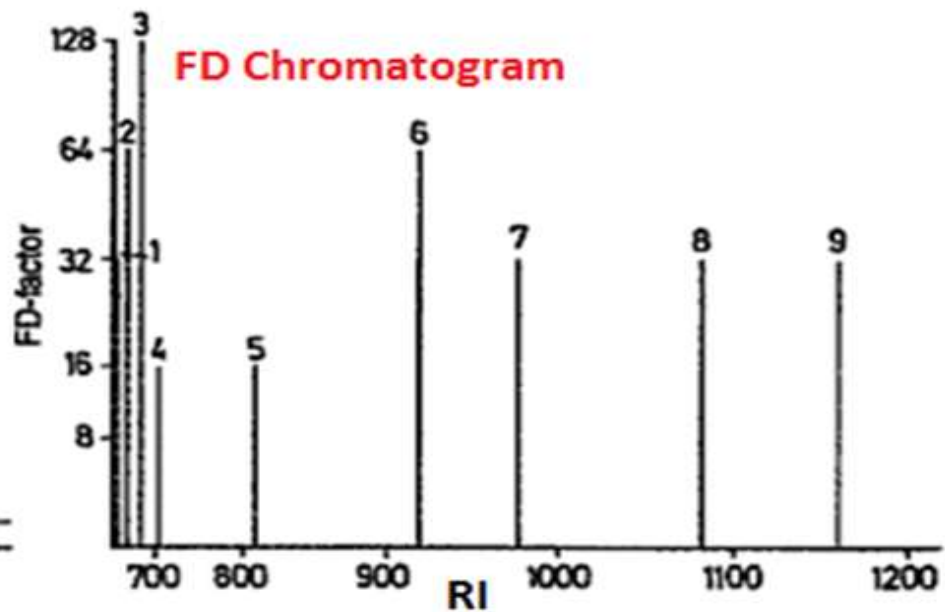
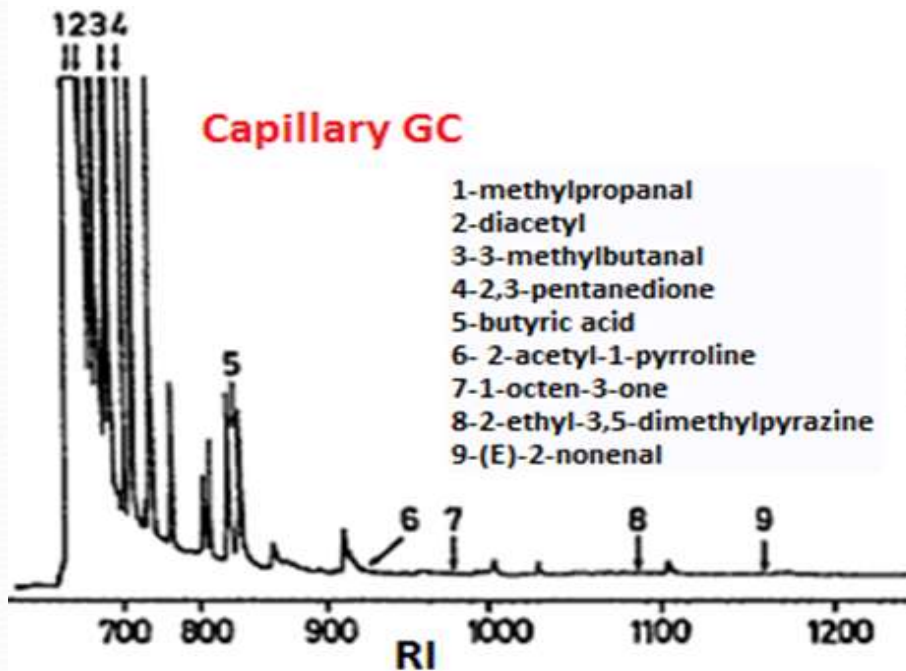
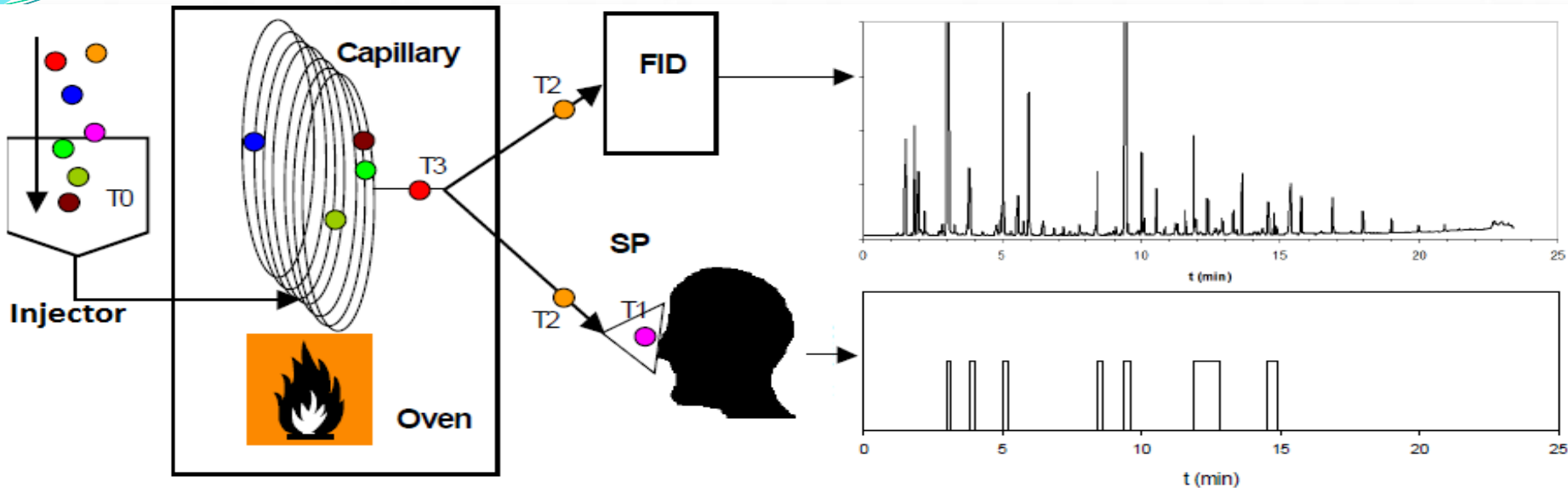


GC-O

Experiments based on human subjects sniffing GC effluents are described as GC-O.

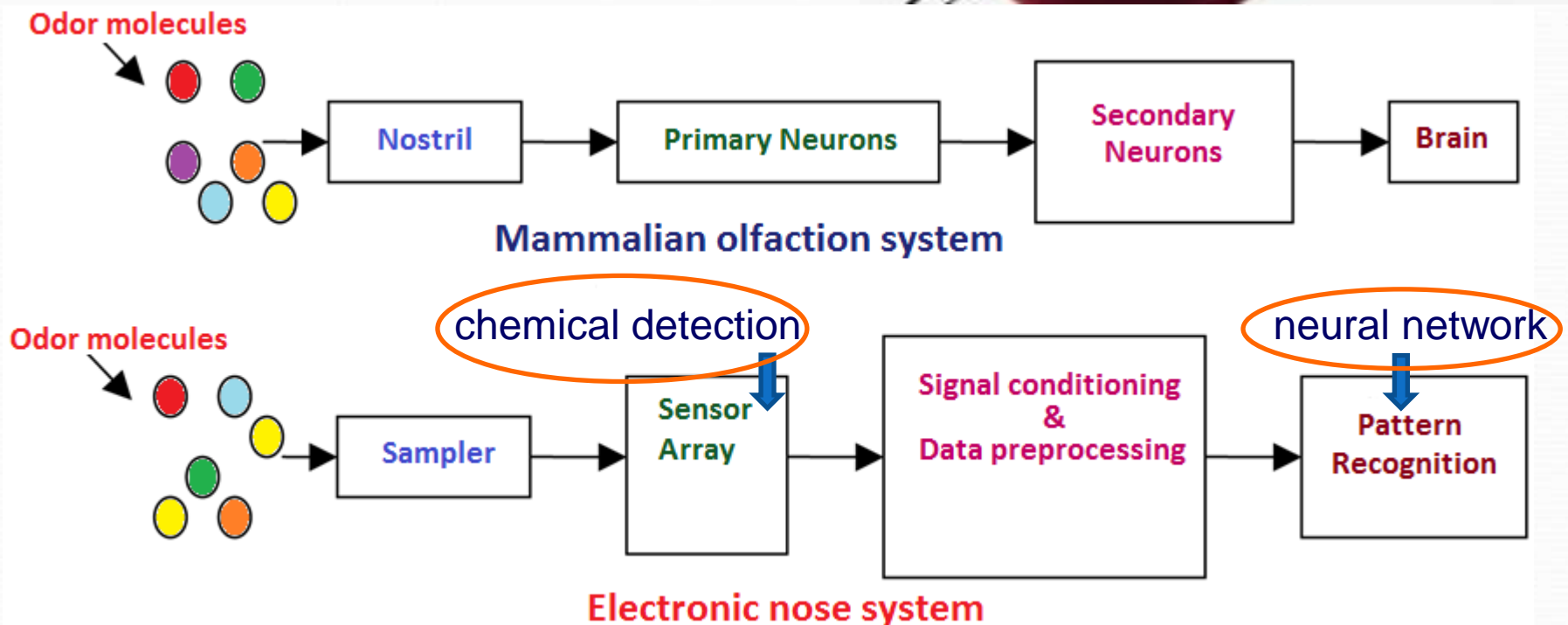
Dilution techniques (Charm Analysis and AEDA) and **time-intensity measurements** (Osme) are the two main GC-O methods.





E-Nose

- ✓ Principal chemical components
- ✓ Olfactory image analysis
- ✓ Trace analysis of impurities
- ✓ Sensory panel biofeedback



Remarks

- ❖ Isolation and separation of flavor components (sample preparation) is critical stage in flavor analysis. Different methods can be used, depending on the quality and character of the particular food (SPME, HP-extraction by supercrit.-CO₂, SAFE, SBSE are commonly used modern methods).
- ❖ MS based-GC instrumentations are still commonly used systems for identification and quantification of flavor compounds (SPME-GC-MS, GC-MS-O, MS-based e-nose and SIDA are lately preferred approaches).



Thank You!