

New and Advanced Analytical Developments in Oil Processing

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- ② The use of standard methods for the quality assessment of oils and fats helps suppliers and users to find a common base for discussions.



Standard Methods

Standard methods are used to measure quality criteria:

- ③ applied worldwide
- ③ tested by collaborative study
- ③ generally accepted by authorities



AOCS / AOAC / IUPAC / ISO / CEN







Crude Palm Kernel Oil

RBD Palm Kernel Oil

Crude Palm Kernel Olein

RBD Palm Kernel Olein

Crude Palm Kernel Stearin

RBD Palm Kernel Stearin



Characteristics of Olive Oil Types

	Acidity (%)	Peroxide value (mEq O ₂ /kg)	Waxes (mg/kg)	Saturated acids in 2-position of the triglyceride (%)	Stigmastadienes (mg/kg) ⁽¹⁾	Difference between HPCL and theoretical ECN42	Organoleptic assessment Median of defects (Md)
1. Extra virgin olive oil	≤ 0.8	≤ 20	≤ 250	≤ 1.5	≤ 0.15	≤ 0.2	Md = 0
2. Virgin olive oil	≤ 2.0	≤ 20	≤ 250	≤ 1.5	≤ 0.15	≤ 0.2	Md ≤ 2.5
3. Lampante olive oil	> 2.0	-	≤ 300 ⁽³⁾	≤ 1.5	> 0.50	≤ 0.3	Md > 2.5 ⁽²⁾
4. Refined olive oil	≤ 0.3	≤ 5	≤ 350	≤ 1.8	-	≤ 0.3	-
5. Blended olive oil composed of refined & virgin olive oils	≤ 1.0	≤ 15	≤ 350	≤ 1.8	-	≤ 0.3	-
6. Crude olive-pomace oil	-	-	> 350 ⁽⁴⁾	≤ 2.2	-	≤ 0.6	-
7. Refined olive-pomace oil	≤ 0.3	≤ 5	> 350	≤ 2.2	-	≤ 0.5	-
8. Olive-pomace oil	≤ 1.0	≤ 15	> 350	≤ 2.2	-	≤ 0.5	-

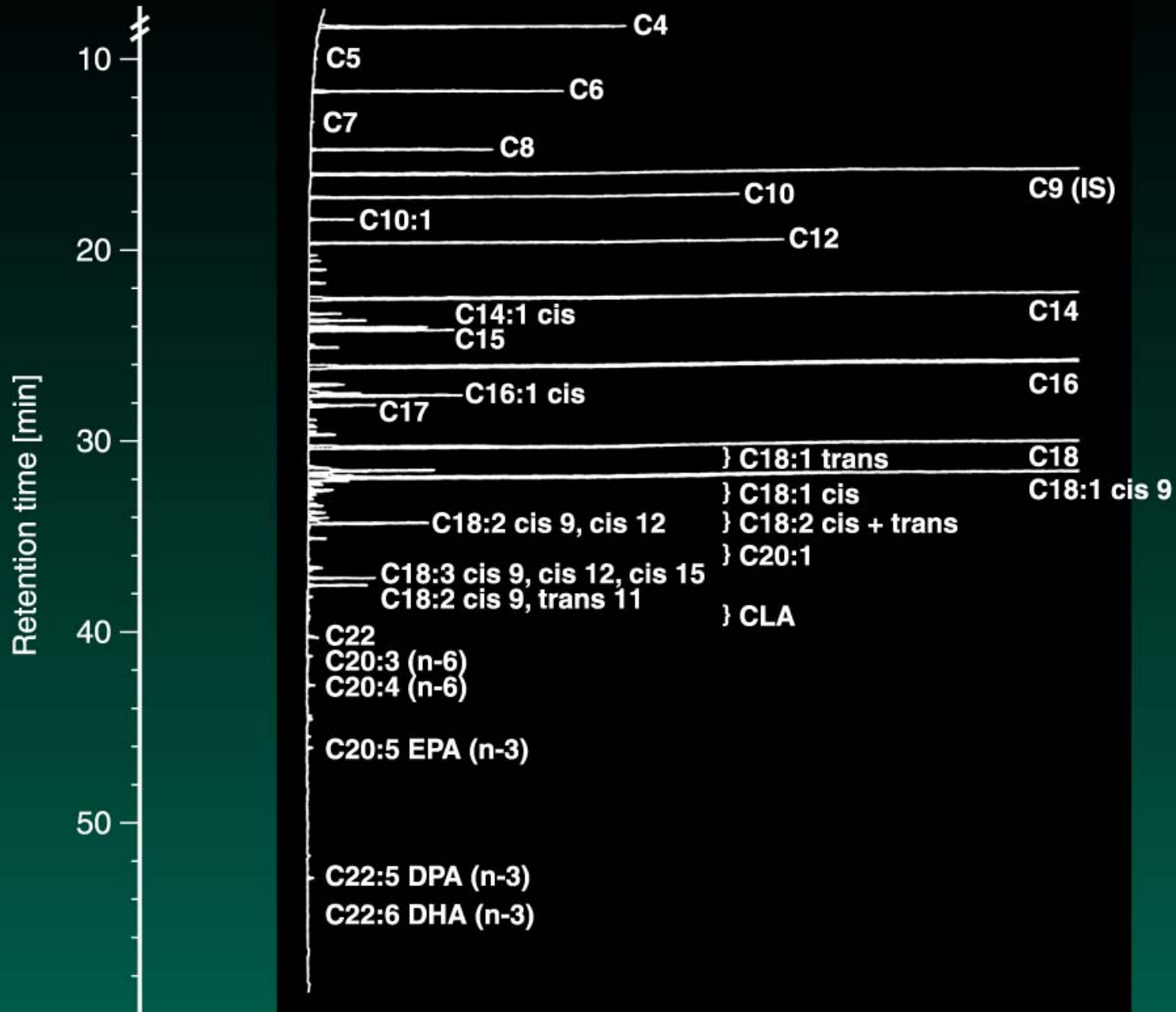
(1) Sum of isomers that could (or could not) be separated by capillary column.

(2) Or if the median of defects is ≤ 2.5 and the median of fruity is 0.

(3) Oils with a wax content of between 300 and 350 mg/kg are considered to be lampante olive oil if the total aliphatic alcohol content is ≤ 350 mg/kg or if the erythrodiol and uvaol content is ≤ 3.5 %.

(4) Oils with a wax content between 300 and 350 mg/kg are considered to be crude olive-pomace oil if the total aliphatic alcohol content is > 350 mg/kg and if the erythrodiol and uvaol content is > 3.5 %.





Fatty Acids⁽¹⁾ (%)

Peanut

Butyric (<i>Butanoic</i>)	$C_4H_8O_2$	
Caproic (<i>Hexanoic</i>)	$C_6H_{12}O_2$	
Caprylic (<i>Octanoic</i>)	$C_8H_{16}O_2$	
Capric (<i>Decanoic</i>)	$C_{10}H_{20}O_2$	
Lauric (<i>Dodecanoic</i>)	$C_{12}H_{24}O_2$	
Lauroleic (<i>cis-9-Dodecenoic</i>)	$C_{12}H_{22}O_2$	
Myristic (<i>Tetradecanoic</i>)	$C_{14}H_{28}O_2$	X
Myristoleic (<i>cis-9-Tetradecenoic</i>)	$C_{14}H_{26}O_2$	
Palmitic (<i>Hexadecanoic</i>)	$C_{16}H_{32}O_2$	6
Palmitoleic (<i>cis-9-Hexadecenoic</i>)	$C_{16}H_{30}O_2$	X



Fatty Acids⁽²⁾ (%)

Peanut

Stearic (<i>Octadecanoic</i>)	$C_{18}H_{36}O_2$	5
Oleic (<i>cis-9-Octadecenoic</i>)	$C_{18}H_{34}O_2$	61
Ricinoleic (<i>12-Hydroxy-cis-9-Octadecenoic</i>)	$C_{18}H_{34}O_3$	
Linoleic (<i>cis-9, cis-12-Octadecadienoic</i>)	$C_{18}H_{32}O_2$	22
Linolenic (<i>cis-9, cis-12, cis-15-Octadecatrienoic</i>)	$C_{18}H_{30}O_2$	X
Eleostearic (<i>cis-9, trans-11, trans-13-Octadecatrienoic</i>)	$C_{18}H_{30}O_2$	
Licanic (<i>4-Keto-9, 11, 13-Octadecatrienoic</i>)	$C_{18}H_{28}O_3$	



Fatty Acids⁽³⁾ (%)

Peanut

Arachidic (<i>Eicosanoic</i>)	$C_{20}H_{40}O_2$	2
Gadoleic (<i>cis-9-Eicosenoic</i>)	$C_{20}H_{38}O_2$	
Arachidonic (<i>5, 8, 11, 14-Eicosatetraenoic</i>)	$C_{20}H_{32}O_2$	
Behenic (<i>Docosanoic</i>)	$C_{22}H_{44}O_2$	3
Erucic (<i>cis-13-Docosenoic</i>)	$C_{22}H_{42}O_2$	
Clupanodonic (<i>4, 8, 12, 15, 19-Docosapentaenoic</i>)	$C_{22}H_{36}O_2$	
Lignoceric (<i>Tetracosanoic</i>)	$C_{24}H_{48}O_2$	1
Nisinic (<i>4, 8, 12, 15, 18, 21-Tetracosahexaenoic</i>)	$C_{24}H_{38}O_2$	
Cerotic (<i>Hexacosanoic</i>)	$C_{26}H_{52}O_2$	
Montanic (<i>Octacosanoic</i>)	$C_nH_{2n}O_2$	





CRUDE PALM OIL

CRUDE PALM OLEIN

CRUDE PALM STEARIN

REFINED, BLEACHED AND
DEODORISED PALM OLEIN

REFINED, BLEACHED AND
DEODORISED PALM STEARIN

REFINED, BLEACHED AND
DEODORISED PALM OIL



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Characteristics of Olive Oil Types

	Fatty acids content ⁽¹⁾						Total sterols (mg/kg)	Erythrodiol and uvaol (%) (**)
	Myristic (%)	Linolenic (%)	Arachidic (%)	Eicosenoic (%)	Behenic (%)	Lignoceric (%)		
1. Extra virgin olive oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.2	≤ 0.2	≥ 1,000	≤ 4.5
2. Virgin olive oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.2	≤ 0.2	≥ 1,000	≤ 4.5
3. Lampante olive oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.2	≤ 0.2	≥ 1,000	≤ 4.5 ⁽²⁾
4. Refined olive oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.2	≤ 0.2	≥ 1,000	≤ 4.5
5. Blended olive oil composed of refined & virgin olive oils	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.2	≤ 0.2	≥ 1,000	≤ 4.5
6. Crude olive-pomace oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.3	≤ 0.2	≥ 2,500	> 4.5 ⁽³⁾
7. Refined olive-pomace oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.3	≤ 0.2	≥ 1,800	> 4.5
8. Olive-pomace oil	≤ 0.05	≤ 1.0	≤ 0.6	≤ 0.4	≤ 0.3	≤ 0.2	≥ 1,600	> 4.5

(1) Other fatty acids present (%): palmitic: 7.5 to 20.0; palmitoleic: 0.3 to 3.5; heptadecanoic: ≤ 0.3; stearic: 0.5 to 5.0; oleic: 55.0 to 83.0; linoleic: 3.5 to 21.0.

(2) Oils with a wax content of between 300 and 350 mg/kg are considered to be lampante olive oil if the total aliphatic alcohol content is ≤ 350 mg/kg or if the erythrodiol and uvaol content is ≤ 3.5 %.

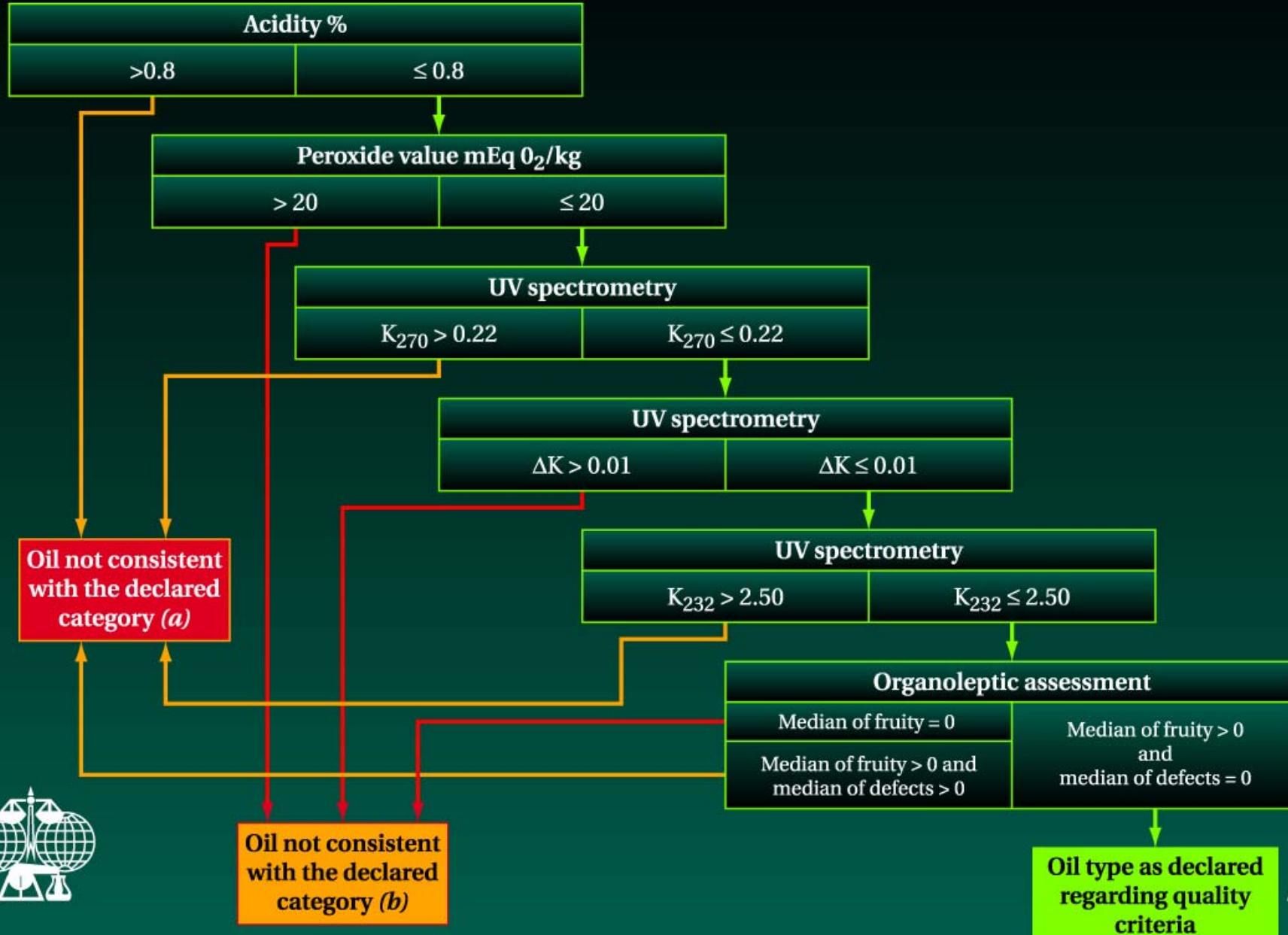
(3) Oils with a wax content of between 300 and 350 mg/kg are considered to be crude olive-pomace oil if the total aliphatic alcohol content is above 350 mg/kg and if the erythrodiol and uvaol content is > 3.5 %.

Notes:

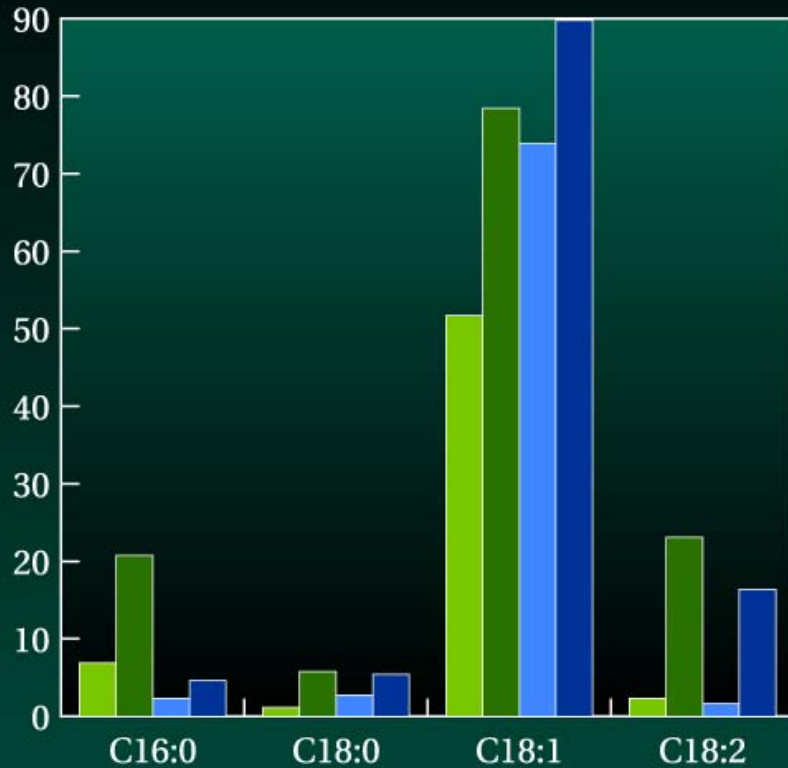
- The results of the analyses must be expressed to the same number of decimal places as used for each characteristic. The last digit must be increased by one unit if the following digit is > 4.
- If just a single characteristic does not match the values stated, the category of an oil can be changed or the oil declared impure for the purposes of this Regulation.
- If a characteristic is marked with an asterisk (*), referring to the quality of the oil, this means the following:
 - for lampante olive oil, it is possible for both the relevant limits to be different from the stated values at the same time
 - for virgin olive oils, if at least one of these limits is different from the stated values, the category of the oil will be changed, although they will still be classified in one of the categories of virgin olive oil.
- If a characteristic is marked with two asterisks (**) this means that for all types of olive-pomace oil, it is possible for both the relevant limits to be different from the stated values at the same time.



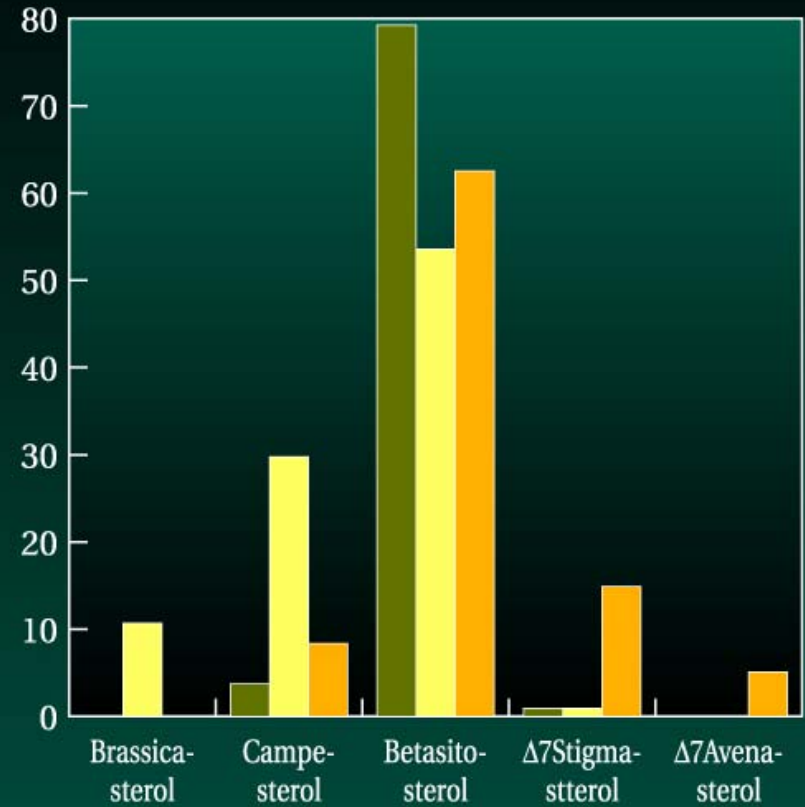
Quality Criteria of Extra Virgin Oil



Fatty Acids and Sterol Composition (%) of Different Oils



- Olive mini
- Olive maxi
- Sunflower oleic mini
- Sunflower oleic maxi

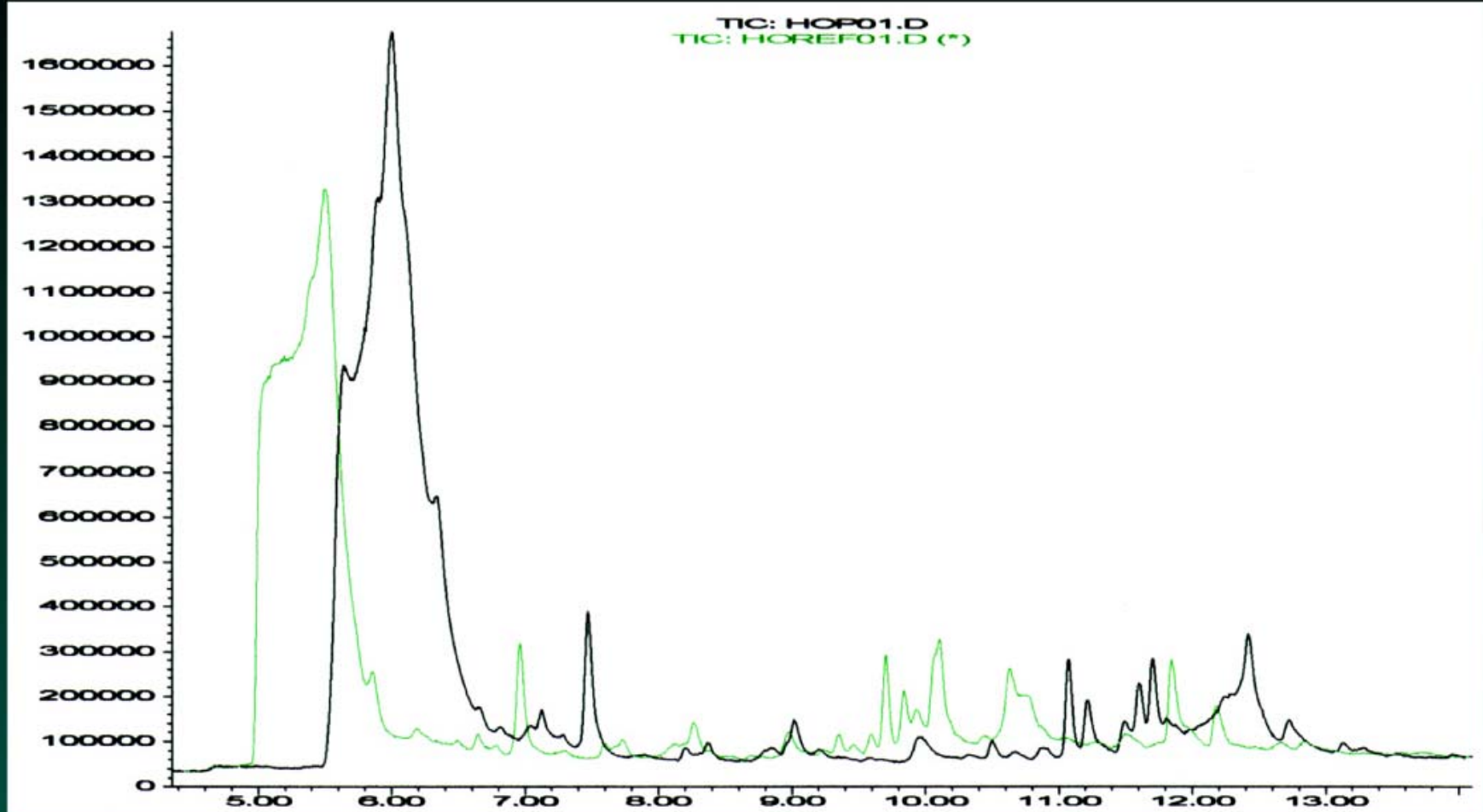


- Olive
- Colza
- Sunflower

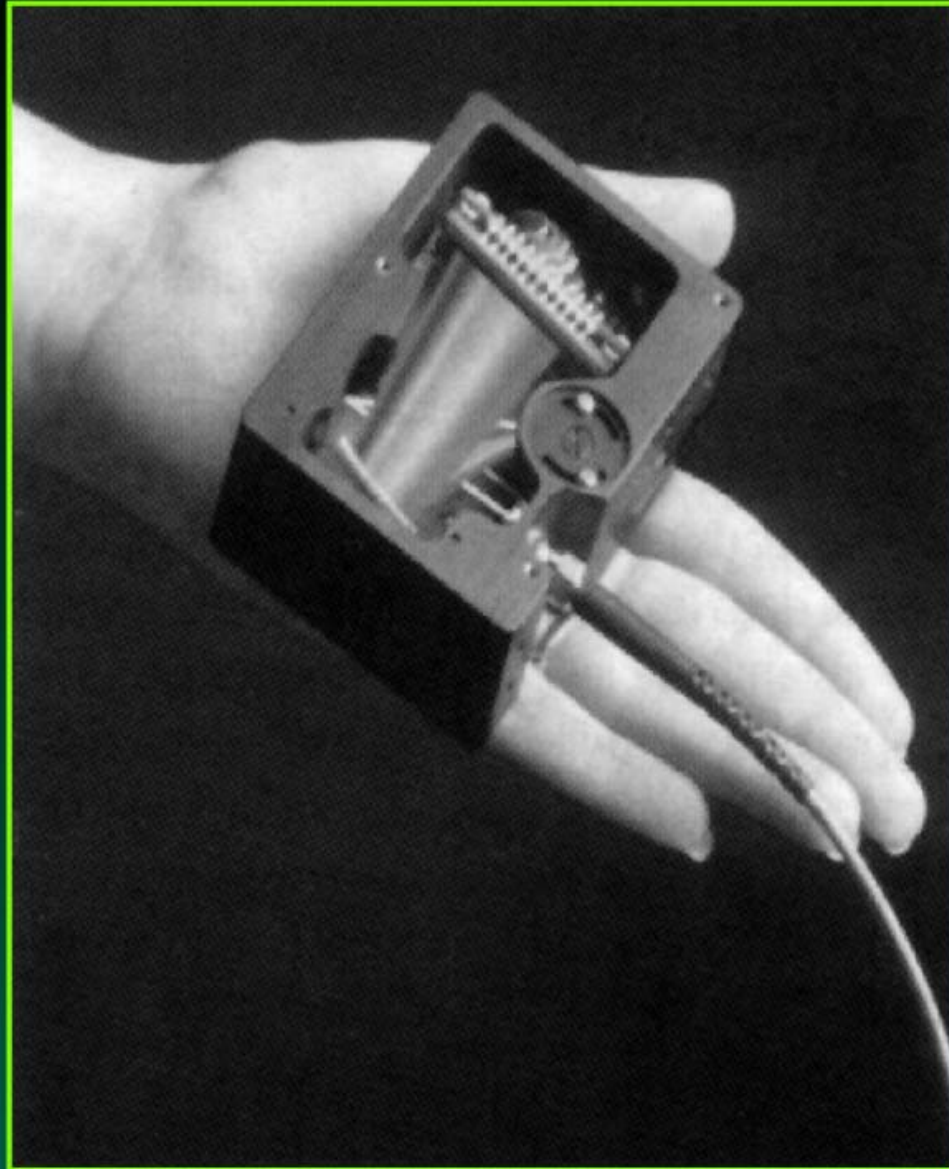




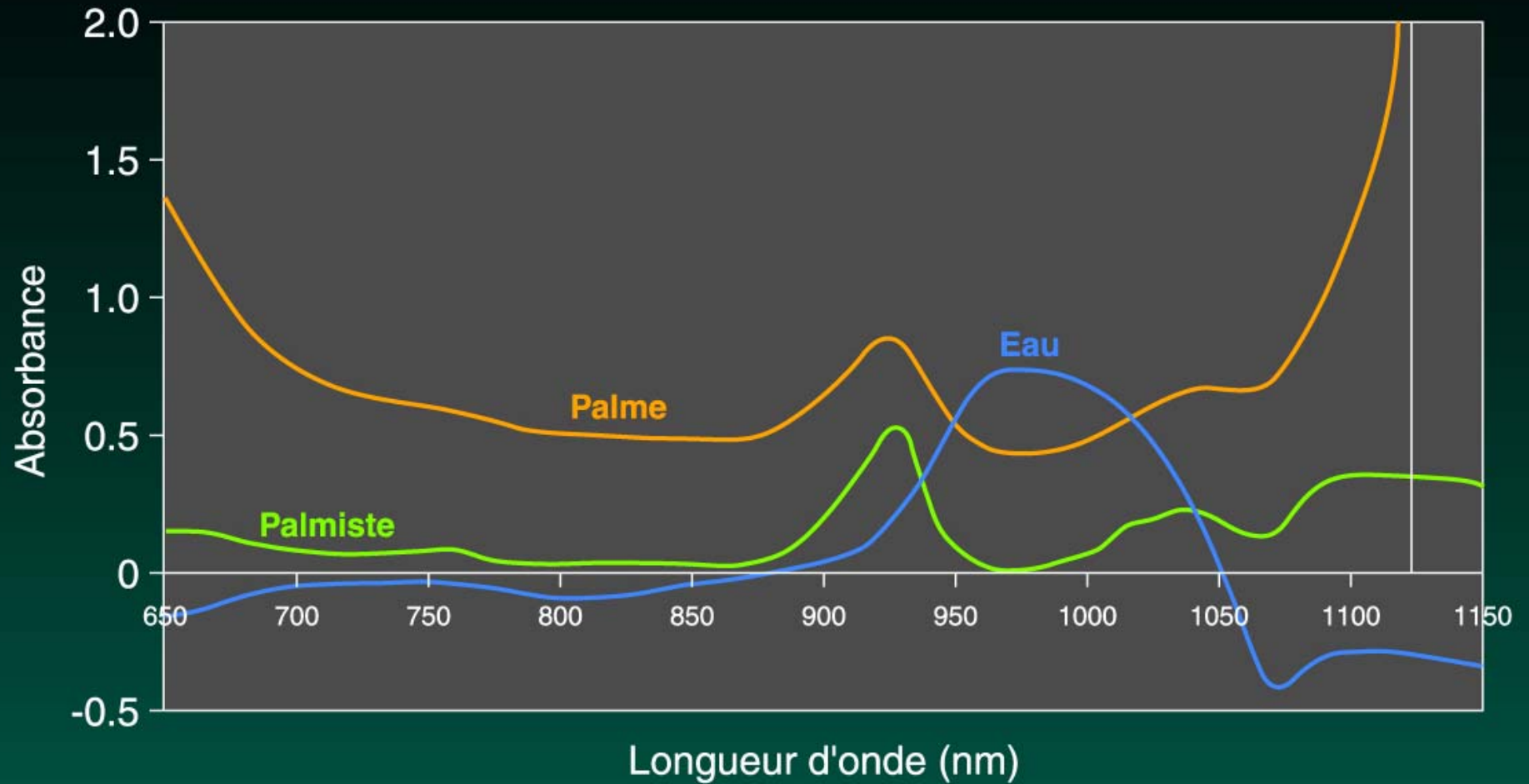
GC/MS Analysis



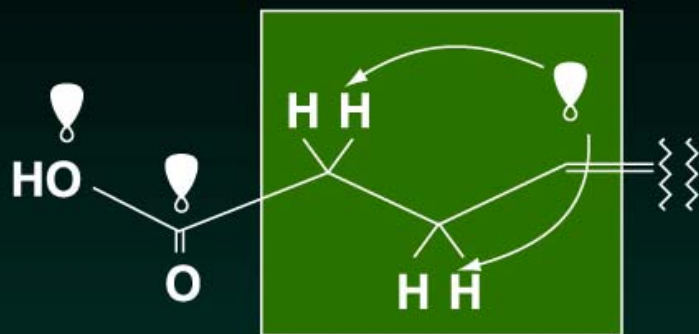
NIR On-line Spectrophotometer



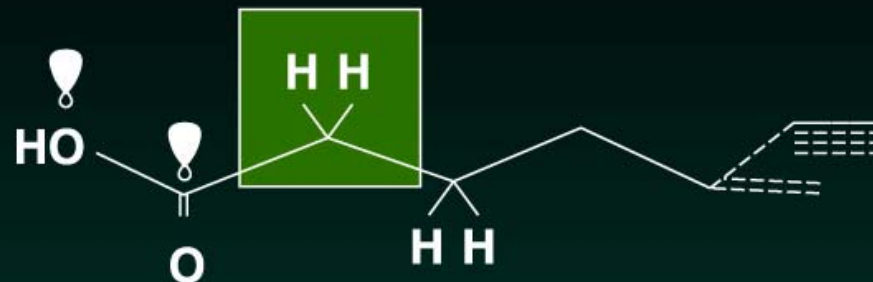
NIR Spectrum



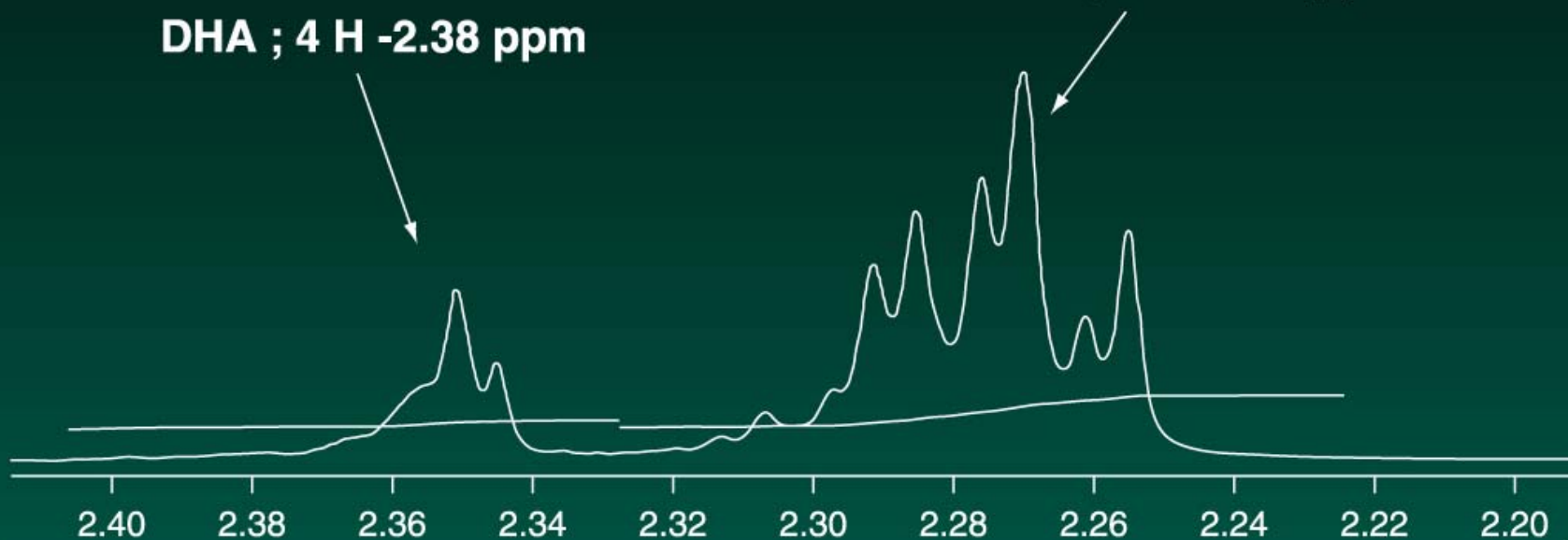
Quantification of DHA Concentration



DHA ; 4 H -2.38 ppm



Others ; 2 H -2.28 ppm

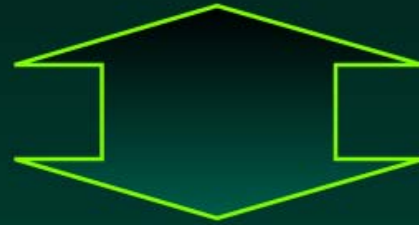






Analytical Evaluation of Freshness

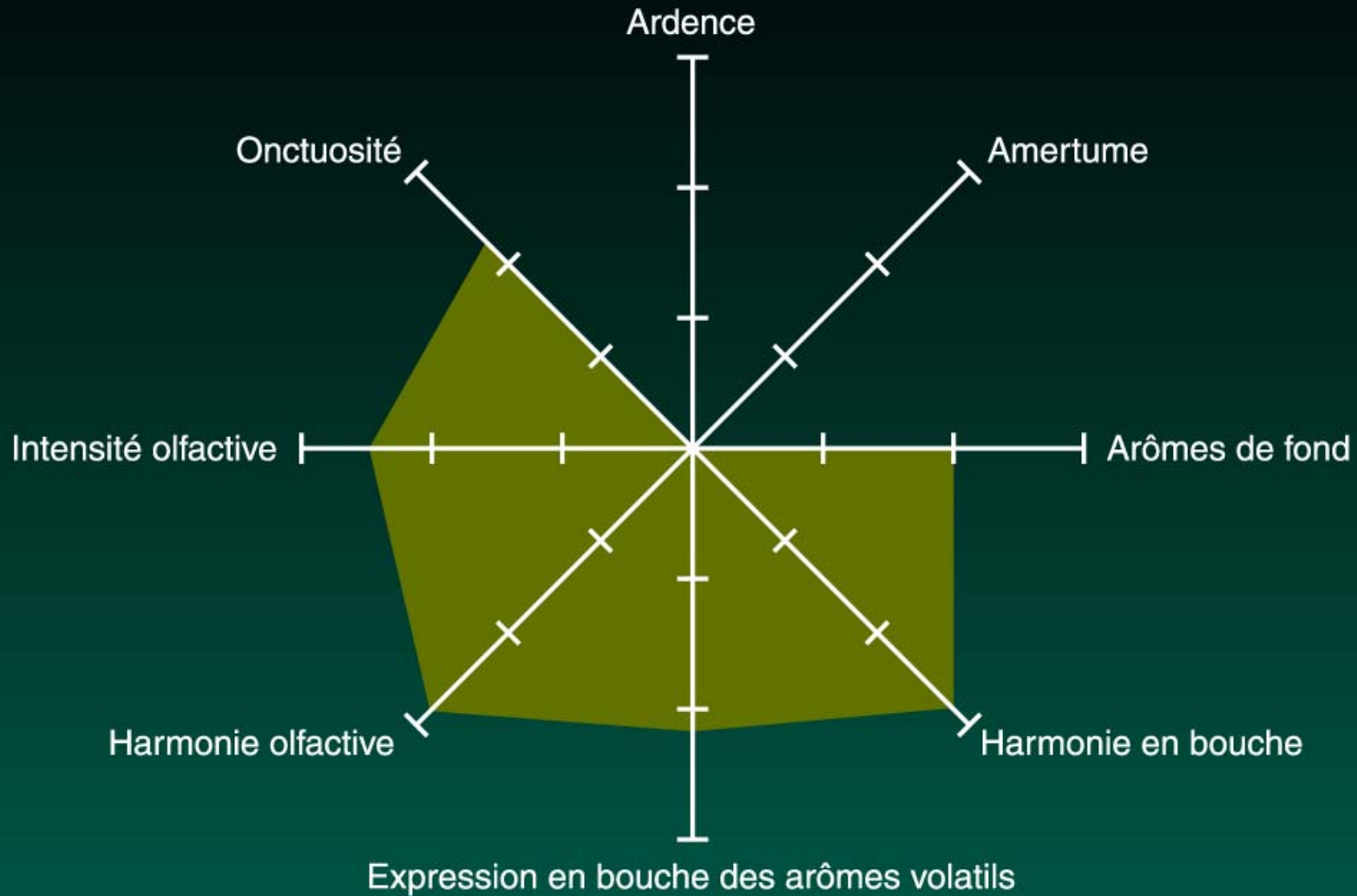
- ② Peroxyde value (PV)
- ② Anisidin value (AV)
- ② Thiobarbituric acid (TBA)



**Organoleptic
Deterioration (meat and dairy products)**

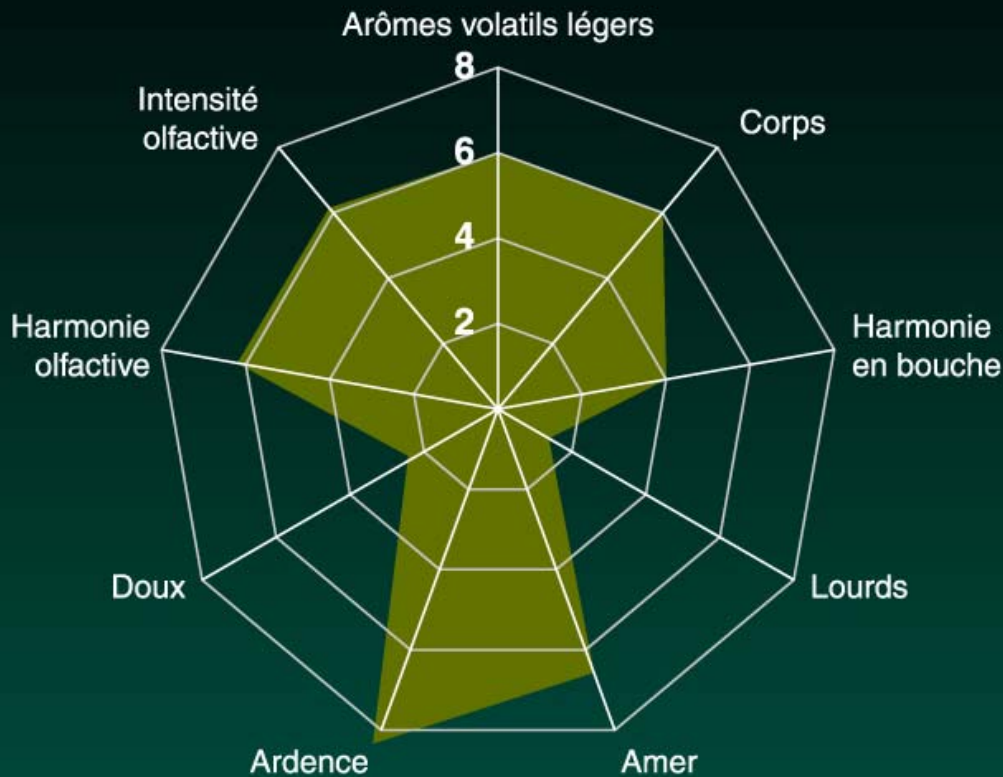


Profile of an Olive Oil

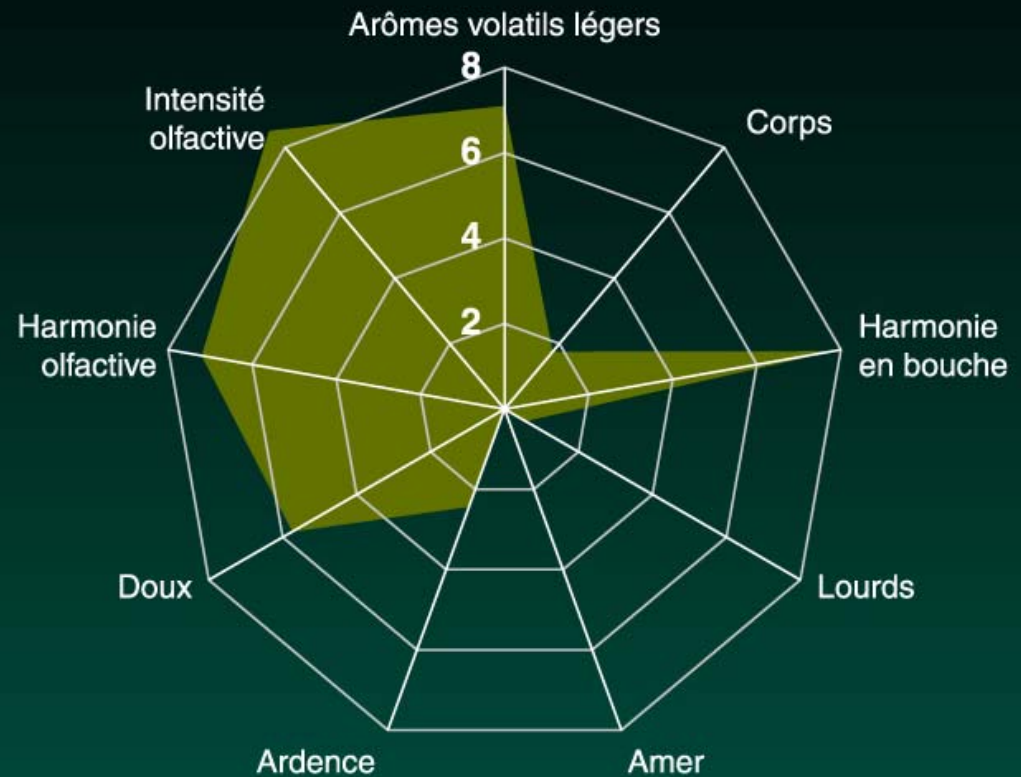


Profile of an Olive Oil

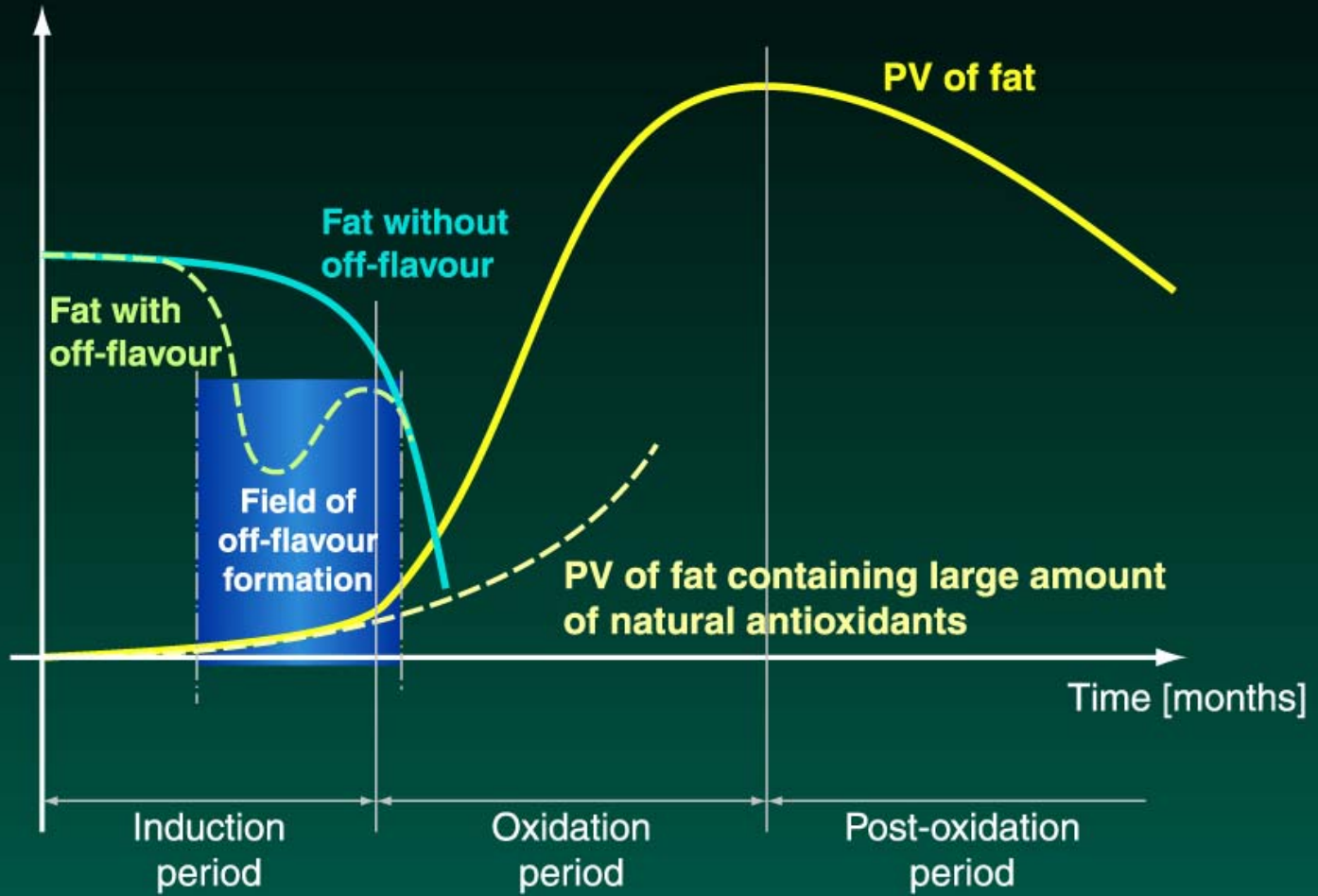
Huile obtenue à partir d'olives
insuffisamment mûres



Huile obtenue à partir d'olives
très mûres



Analytical value or organoleptic scores



Melting

Solid fat contents versus temperature
melting points

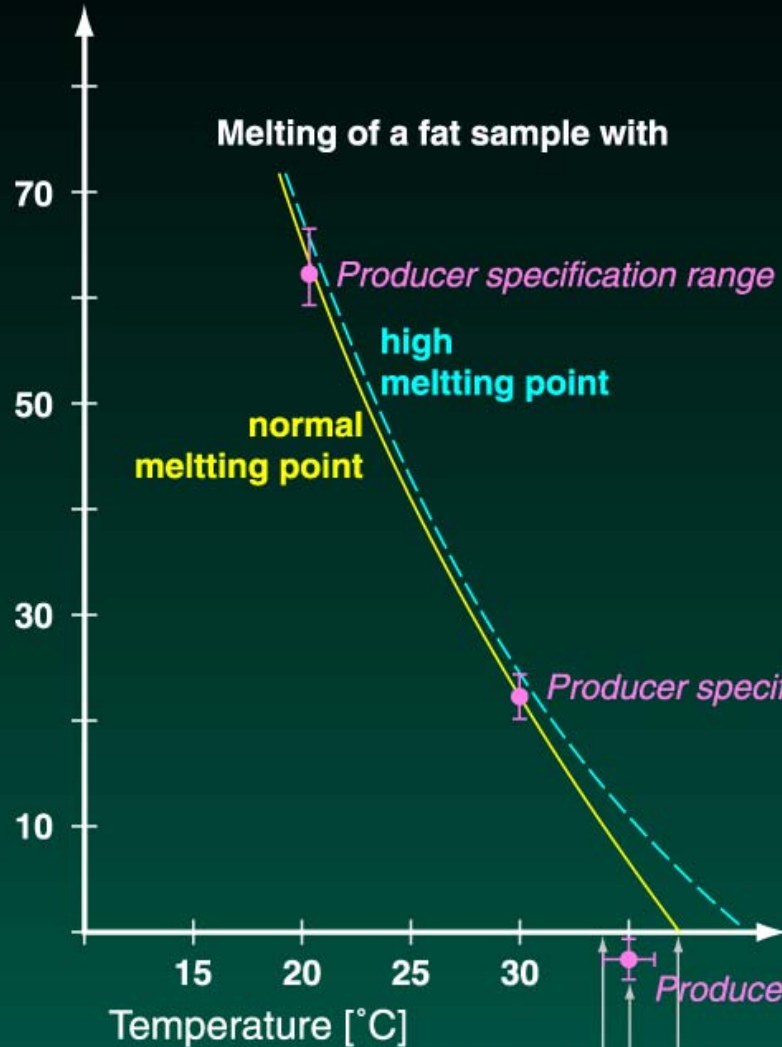
Solid ← → Liquid

Solidification

Cooling curves



Solid fat content (SFI)



Slip point

Drop point (Ubbelohde)

Clear melting point



Tubing Connections of the HPLC System

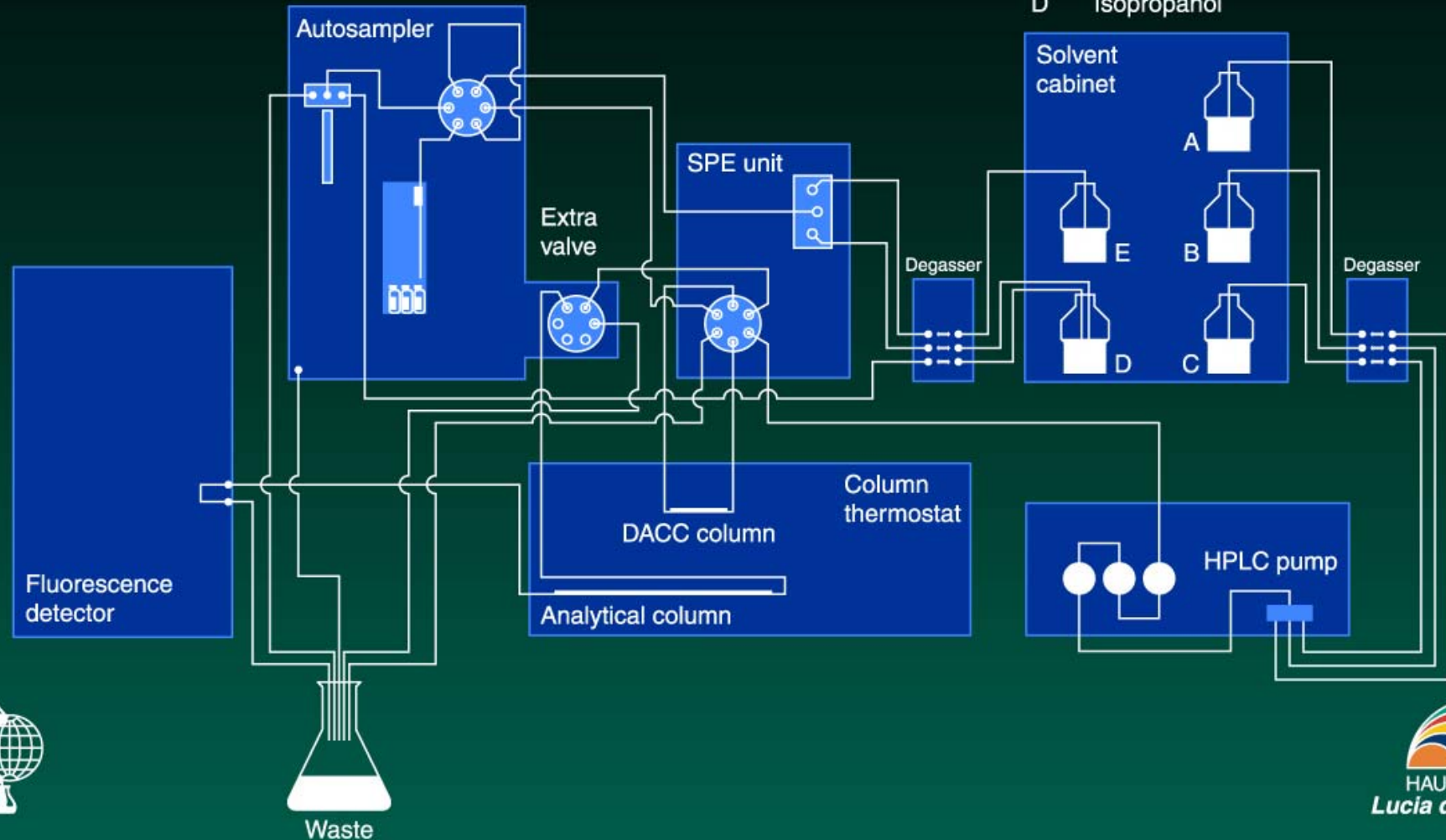
Mobile phases:

A Acetonitrile / Water 85/15

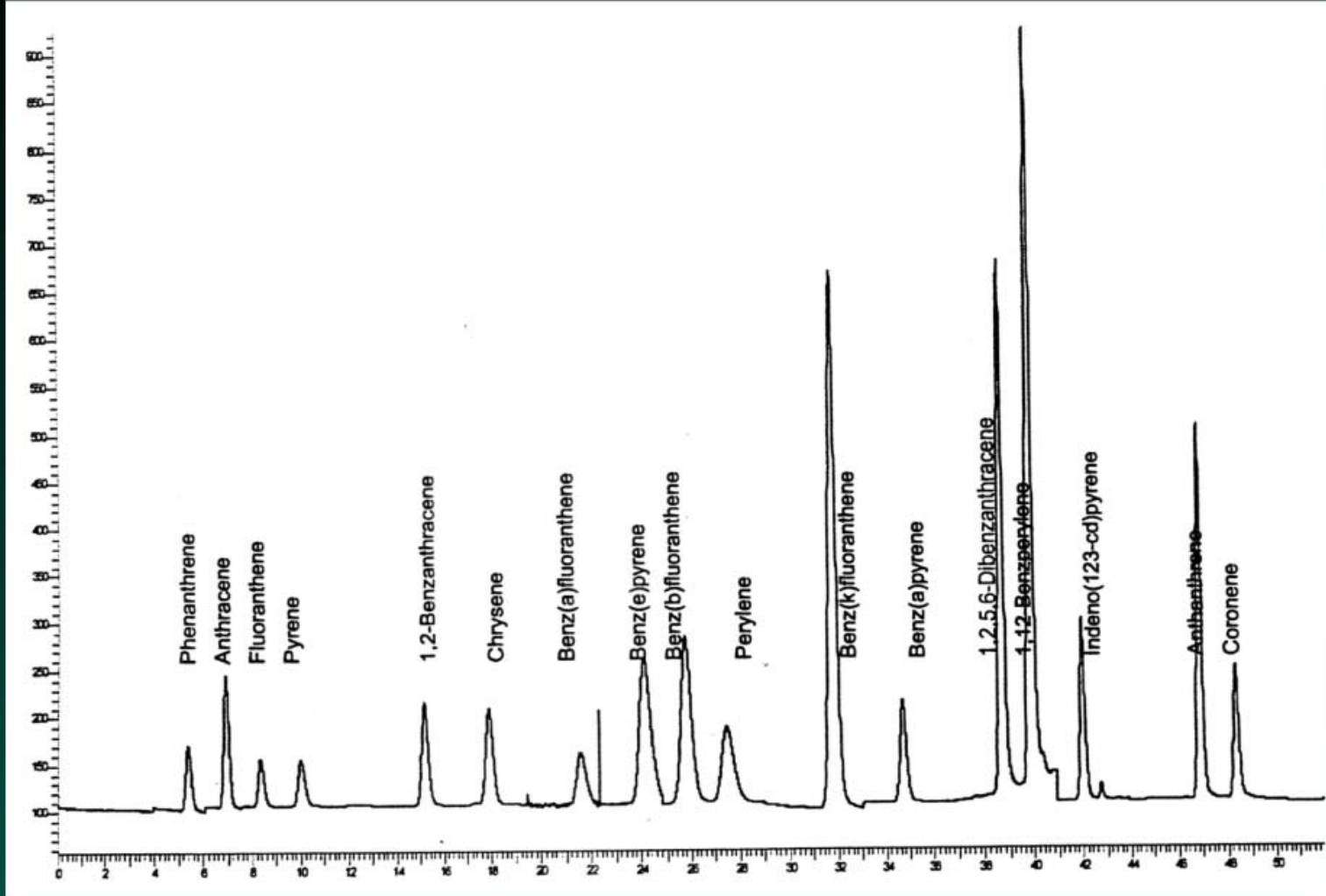
B Acetonitrile

C/E Acetonitrile / Ethylacetate 30/70

D Isopropanol



Chromatogram of a Calibration Sample



Fire Extinguishing

Class of Fire	Extinguishing agent	Do not use	Extinguishing agent	Do not use
Class A (solids)	Water	Water	Water	Water
Class B (liquids)	Water	Water	Water	Water
Class C (gases)	Water	Water	Water	Water
Class D (metals)	Water	Water	Water	Water
Class E (electrical)	Water	Water	Water	Water

Chemical waste

All chemical waste produced at the University of Exeter shall be collected in accordance with the following guidelines:

1. All chemical waste must be collected in a suitable container.
2. The container must be clearly labeled with the name of the waste.
3. The container must be sealed and stored in a suitable area.
4. The container must be stored in a suitable area.
5. The container must be stored in a suitable area.
6. The container must be stored in a suitable area.
7. The container must be stored in a suitable area.
8. The container must be stored in a suitable area.
9. The container must be stored in a suitable area.
10. The container must be stored in a suitable area.



Model 12-10
Model 10-10

0 0 0 0
 0 0 0 0

Model 10-10
 0 0 0 0
 0 0 0 0

Model 10-10
Model 10-10

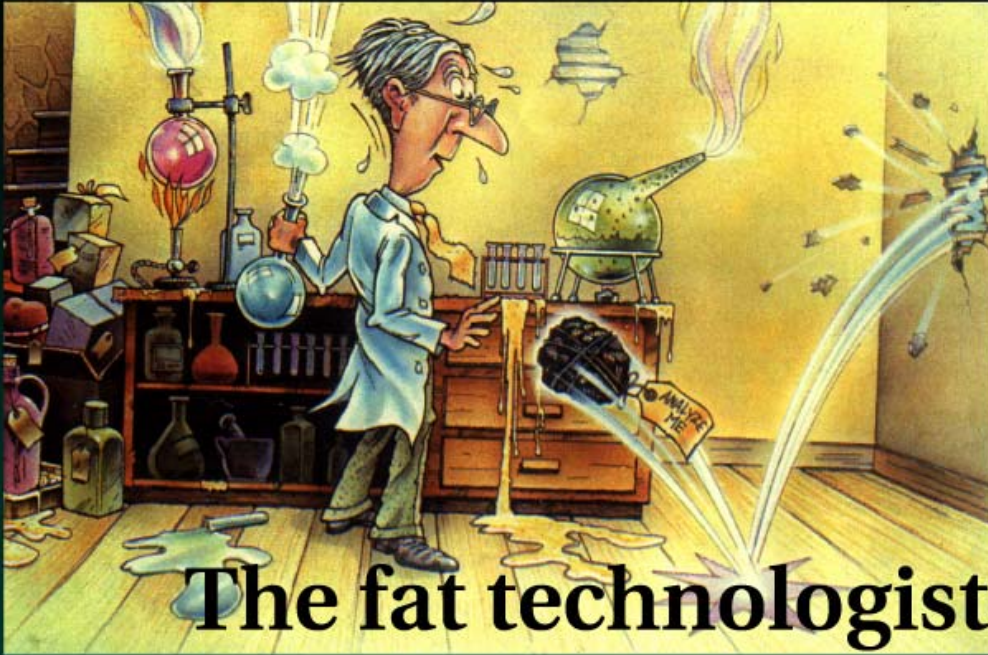
0 0 0 0
 0 0 0 0

Model 10-10
 0 0 0 0
 0 0 0 0

Laboratory glassware including a beaker, a flask, and a bottle on a tray.



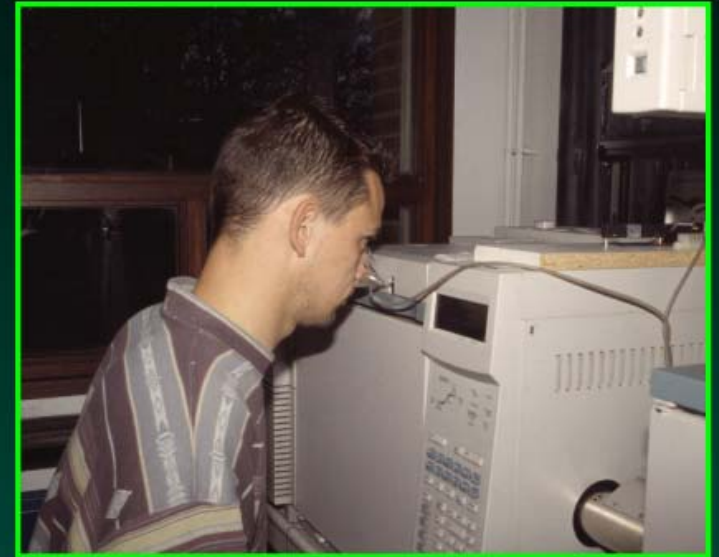
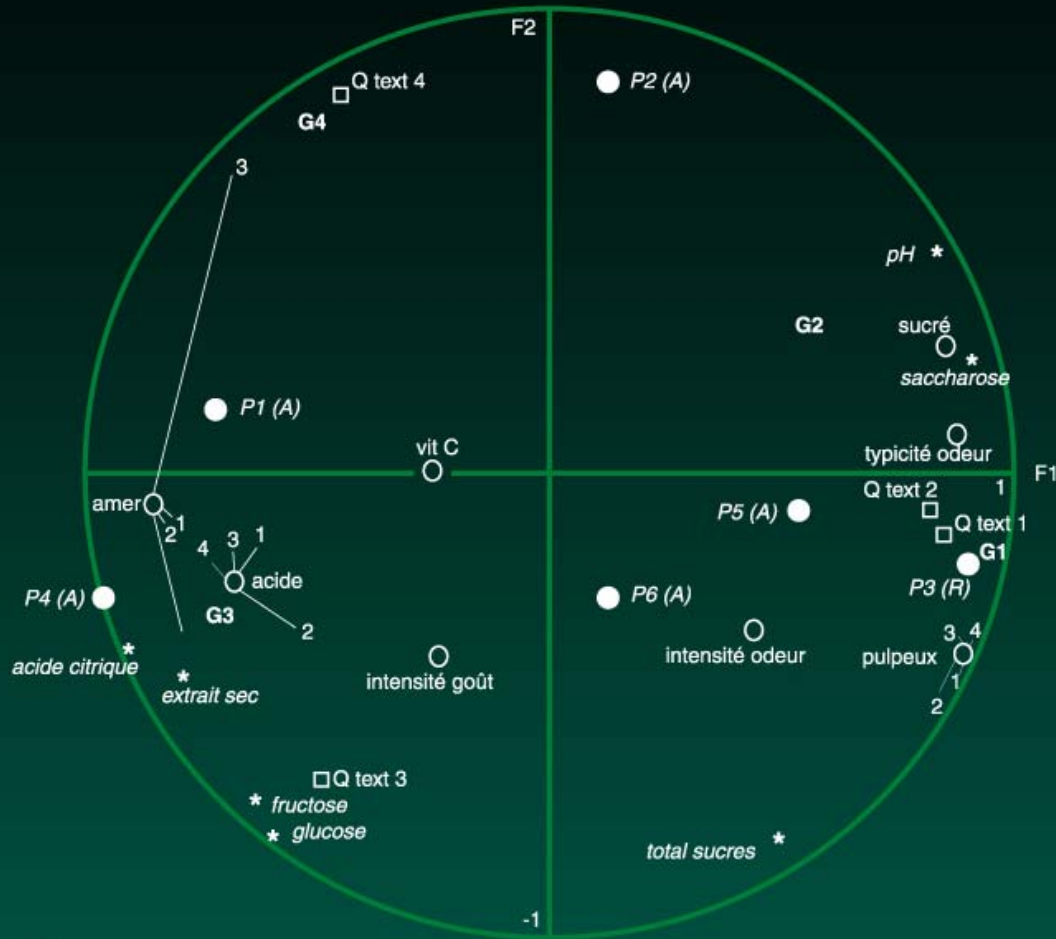
Conclusion



The fat technologist is not an analyst solving isolated analytical problems but rather a system chemist dealing with oils and fats for their optimal use in food.

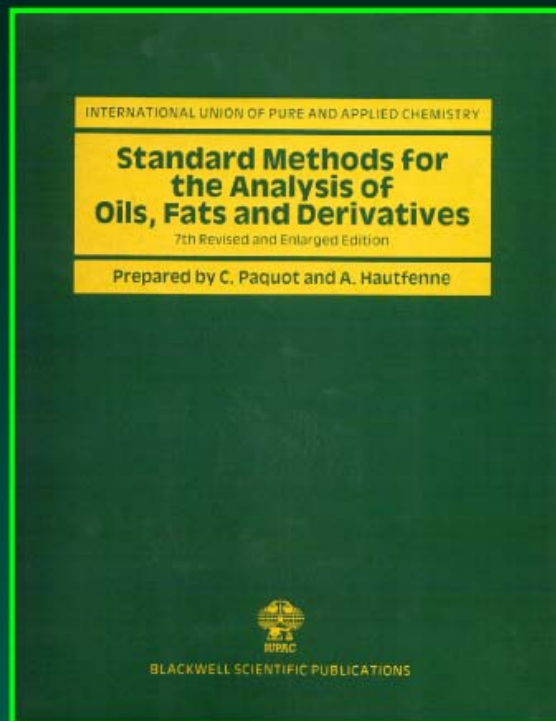


Multidimensional Analysis



IUPAC Standard Methods

- Today, more than 120 standard methods have been accepted after having been tested by international collaborative studies.



Official Methods AOCs (5th Edition)

