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Is profiling of volatile compounds from virgin rapeseed oil a promising tool for the assessment of the sensory quality?

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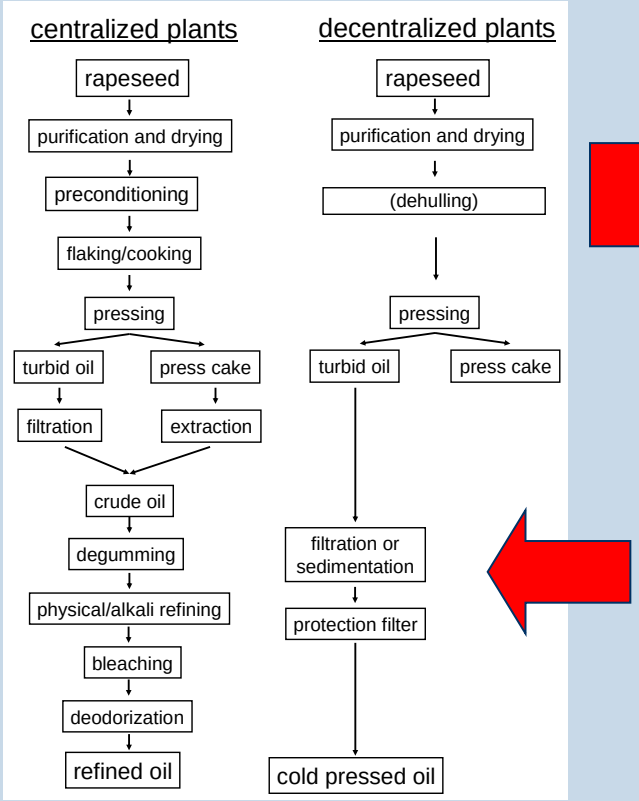
Definition for cold-pressed oils

Cold pressed oils are obtained, **without altering the oil**, by mechanical procedures only, e.g. expelling or pressing, **without the application of heat**. They may have been purified by washing with water, settling, filtering and centrifuging only (Codex Alimentarius for Named Vegetable oils).

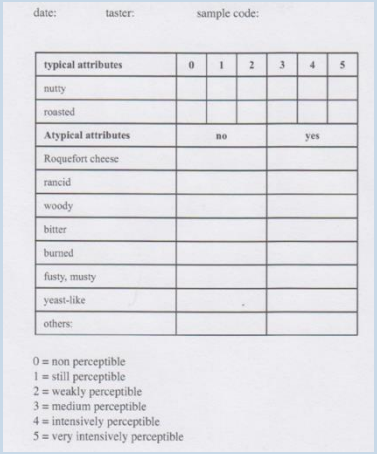


- Intense aroma and taste
- Only slightly processed product
- Original composition of the oil
- Production is highly environmental friendly

Sensory evaluation of virgin oils is one of the most important quality features!

Sensory assessment of edible oils

- Standardized test method **DGF C-II 1 (14)**
- Descriptive and evaluative method
- Test panel with 3 - 5 trained tasters


date: taster: sample code:

typical attributes	0	1	2	3	4	5
nutty						
roasted						

Atypical attributes	no	yes
Roquefort cheese		
rancid		
woody		
bitter		
burned		
fusty, musty		
yeast-like		
others:		

0 = non perceptible
1 = still perceptible
2 = weakly perceptible
3 = medium perceptible
4 = intensively perceptible
5 = very intensively perceptible

Typical attributes	0	1	2	3	4	5
seed-like						
nutty						
wood-like/strawy						
astringent						

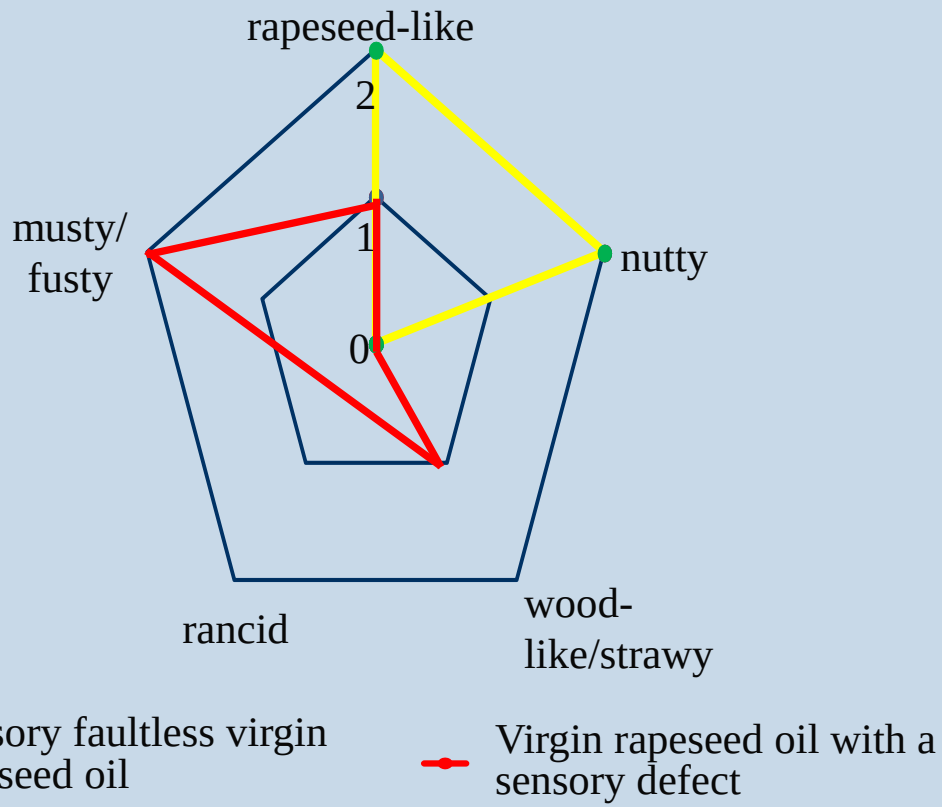
Tasting glass

Atypical attributes	No	Yes
roasty/burnt		
bitter		
rancid		
fusty/musty		
others		

But

- Time consuming
- Personnel intensive
- With an uncertainty due to peoples subjectivity

Aroma profile of different virgin rapeseed oil qualities



Prerequisites for the development of an analytical method for the evaluation of the sensory quality

Drawbacks of the sensory evaluation of cold-pressed oils

- Time consuming
- Personnel intensive
- With an uncertainty due to peoples subjectivity

Does an analytical method solves the problem?

- Identification of compounds responsible for the typical smell or the off-flavour.
- Identification of compounds which differentiate sensory good and bad oils.
- Development of a fast and robust analytical method.
- Development of a statistical model for the classification of the oils into different qualities.

Is it possible to characterize the sensory quality of cold-pressed oil on basis of the distribution of the volatile/aroma-active compounds?

Profiling of cold-pressed rapeseed oils from the market

Profiling: Screening of a large number of cold-pressed rapeseed oils

Sensory good quality

Sensory evaluation

Sensory bad quality

GC-Olfactometry

Dyn. HS-GC-MS

Information about the sensory quality

Statistical correlations??

Information about volatile and aroma-active compounds

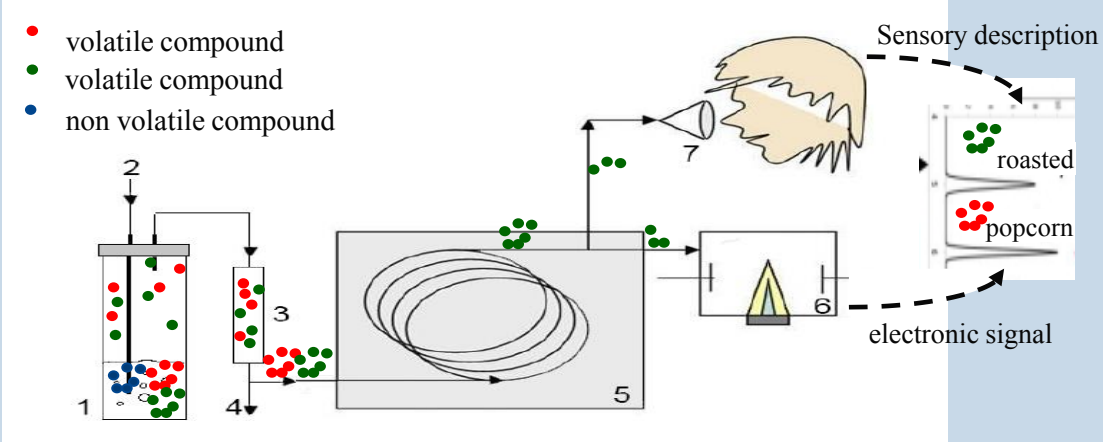
Dyn-HS: Dynamic Headspace **FID**: Flame Ionisation Detector **GC-MS**: Gas Chromatography-Mass Spectrometry

Dynamic Purge and Trap gas chromatographic analysis with mass spectrometric detection

Method:

- 400 mg rapeseed oil
- extraction of the volatile compounds by purge-and – trap technique
- Column: CP-Sil 19 CB

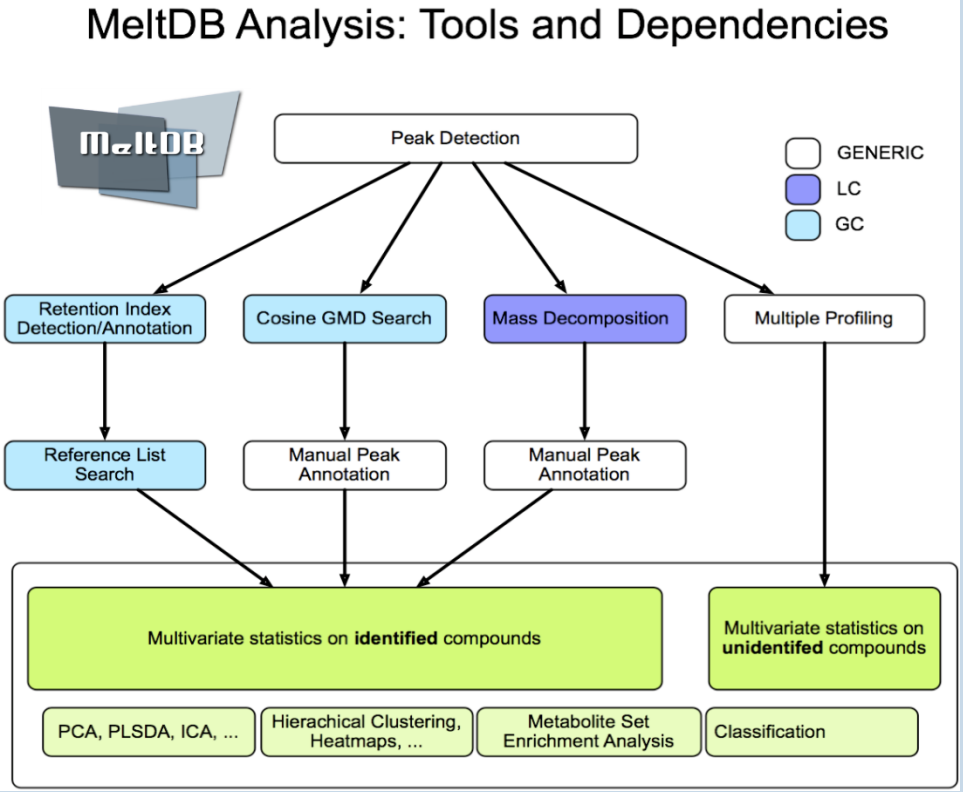
Olfactometry



1. headspace-vail with argan oil; 2. gas supply; 3. trap (Tenax); 4. gas supply; 5. gas chromatograph; 6. detector; 7 sniffing port

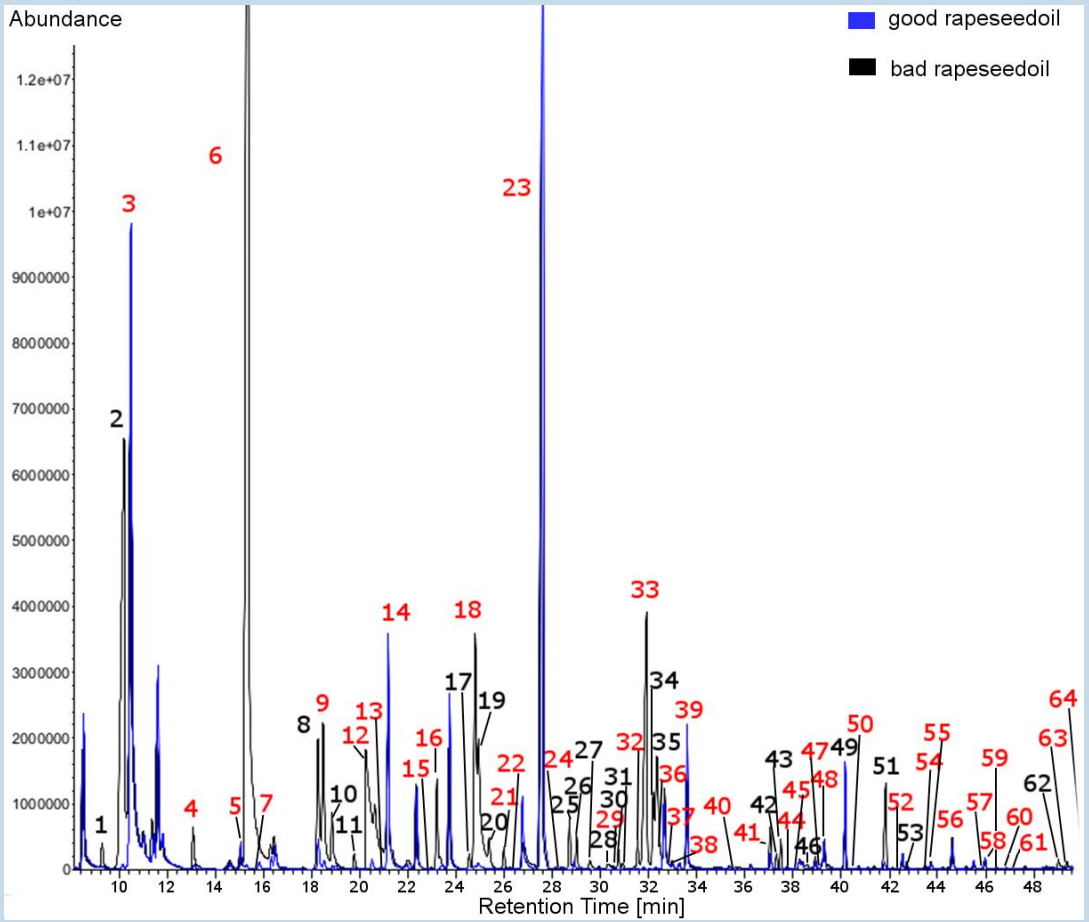
Data analysis

MeltDB - Software platform for the storage, visualization and analysis of large data sets



- Identification of differences and mutual interferences of compounds from samples with sensorial good and bad quality by means of multivariate methods (PCA, ANOVA, Heatmap...)
- Identification of marker compounds for the differentiation of sensory good and bad cold-pressed rapeseed oils

GC-Olfactometry of a sensory good and bad rapeseed oil



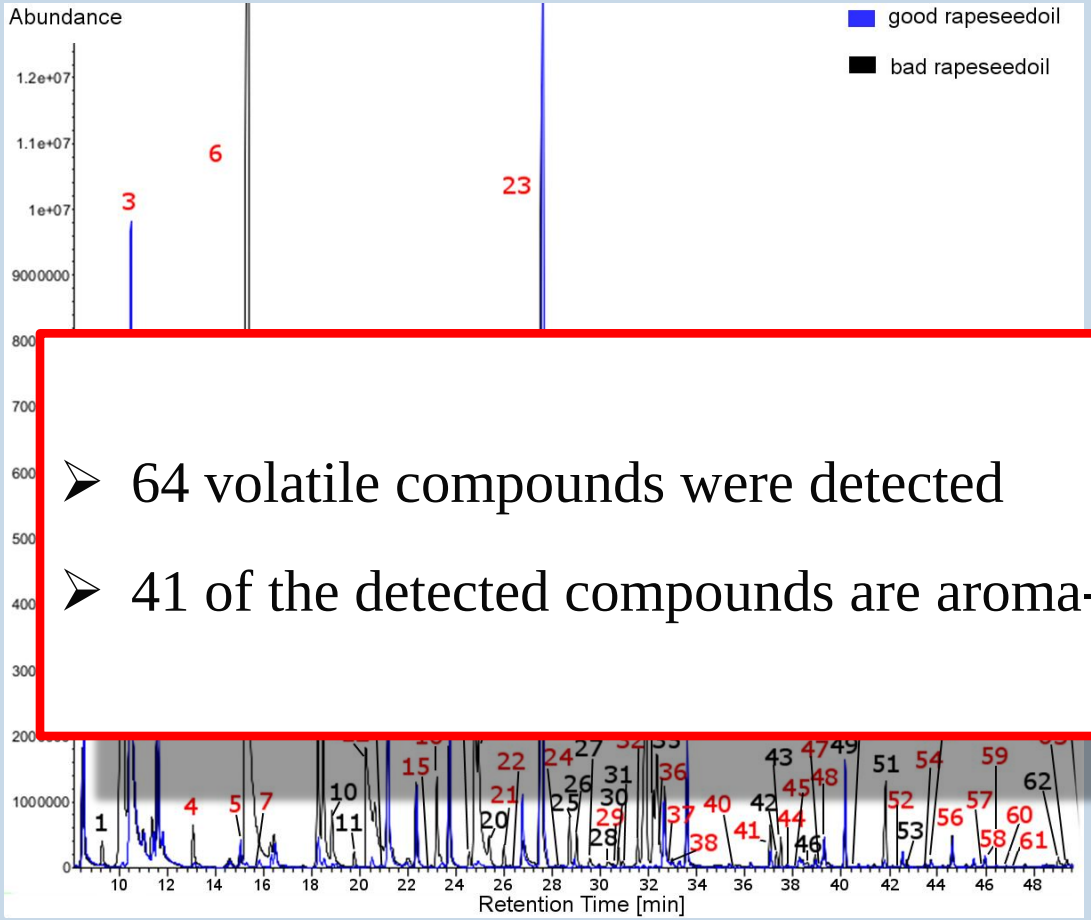
Black numbers:
Volatile compounds (not aroma-active) detected exclusively or significantly increased in sensory bad oils.

Red numbers:
Aroma-active volatile compounds detected by at least 3 from 5 trained tasters.

Main component classes

- esters (13)
- aldehydes (11)
- alcohols (10)
- others (12)
- not identified (18)

GC-Olfactometry of a sensory good and bad rapeseed oil



Black numbers:
Volatile compounds (not aroma-active) detected exclusively or significantly increased in sensory bad oils.

- 64 volatile compounds were detected
- 41 of the detected compounds are aroma-active

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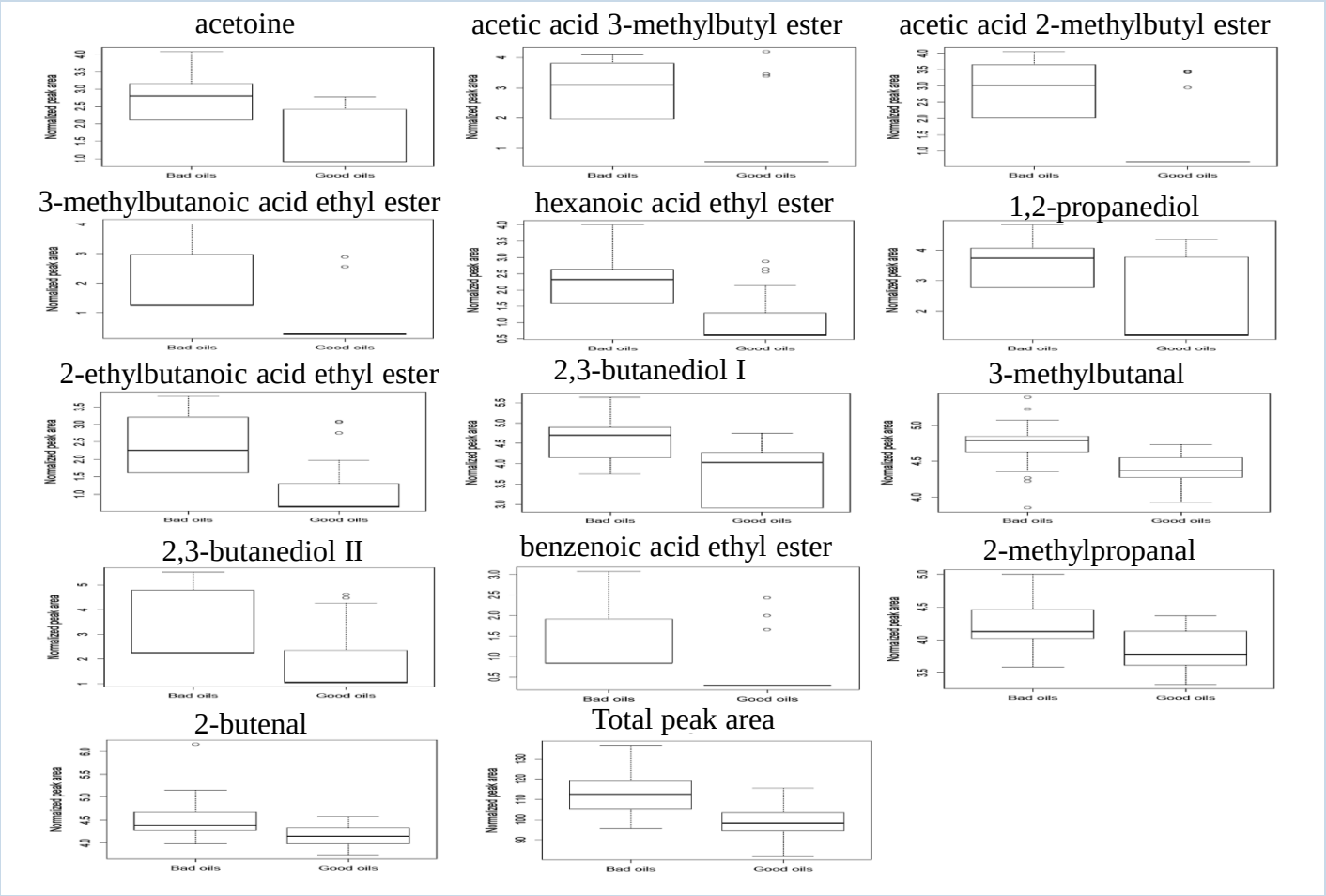
- alcohols (10)
- others (12)
- not identified (18)

Boxplots of the 13 compounds with significant differences in peak areas between sensory good and bad rapeseed oil samples



64 compounds detected

↓

41 aroma-active compounds identified

↓

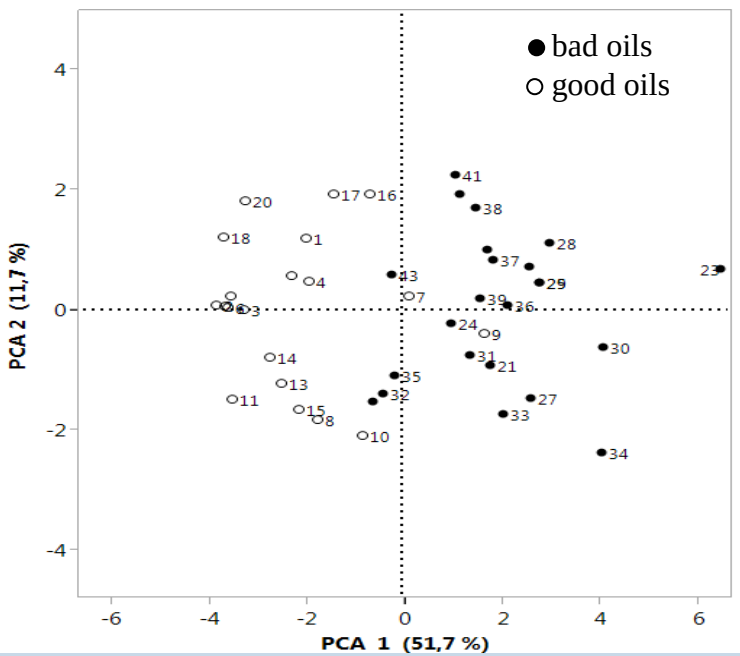
13 significant different compounds

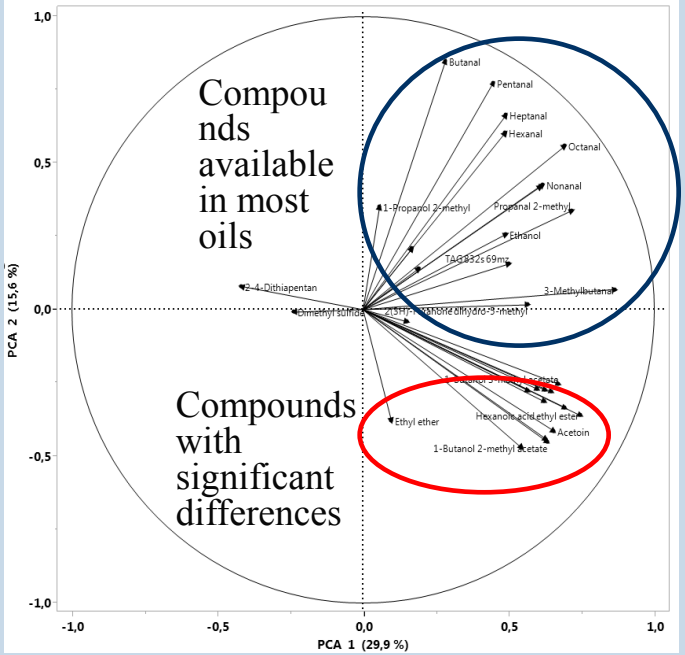
↓

statistical approach

Principle Component Analysis

- Data set: 20 sensory good and 23 sensory bad rapeseed oils
- 31 volatile compounds were identified as aroma active by GC-MS olfactometry
- 13 compounds showed significant differences





- Component 1 and 2 only represent 63 % of the total information.
- Separation mainly based on volatile compounds responsible for the off-flavor.

Classification parameters and results

Rapeseed oil quality = (0.445 * ***1,2-propanediol*** + 0.5911 * ***acetic acid 2-methylbutyl ester*** + 0.3006 * ***acetic acid 3-methylbutyl ester*** - 0.1288 * ***2,3-butanediol I*** + 0.2638 * ***2,3-butanediol II*** - 4.1364 * ***3-methyl butanal*** + 0.7139 * ***acetoin*** + 0.3369 * ***benzoic acid ethyl ester*** + 0.0885 * *3-methylbutanoic acid ethyl ester* + 0.2776 * ***2-methylbutanoic acid ethyl ester*** - 0.5574 * ***hexanoic acid methyl ester*** + 3.4439 * ***2-methylpropanal*** + 0.9923 * ***2-butenal***) - 4.1406.

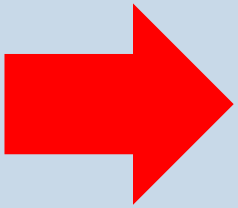
- **< 0 → good rapeseed oil quality**
- **> 0 → bad rapeseed oil quality**

Sensory classification	1,2-Propanediol	1-Butanol, 2-methyl acetate	1-Butanol, 3-methyl acetate	2,3-Butanediol I	2,3-Butanediol II	Butanal, 3-methyl	Acetoin	Benzenoic acid ethyl ester	Butanoic acid, 2-methyl, ethyl ester	Butanoic acid, 3-methyl, ethyl ester	Hexanoic acid ethyl ester	Propanal, 2-methyl	TAG: 763.3s 69mz (2-Butenal)	Formel
good oil	0,0000	0,0000	0,0000	0,0000	0,0000	4,0246	2,1468	0,0000	0,0000	2,2713	0,0000	3,8142	3,3688	-2,0174
good oil	0,0000	0,0000	0,0000	0,0000	0,0000	3,9869	0,0000	0,0000	0,0000	0,0000	1,7729	3,8150	4,1156	-1,8178
bad oil	3,9646	4,0565	4,3563	4,8140	4,8436	5,0677	3,2853	2,4034	4,0353	4,0916	3,2455	4,5507	4,2882	2,2677
bad oil	4,2443	3,5943	3,9687	5,2370	5,1942	5,4089	3,0310	0,0000	0,0000	0,0000	3,2096	4,8257	4,0325	2,4905

	Groups	Predicted group membership		
training set		**bad oils**	**good oils**	**Total**
	bad oils	23 (100%)	0 (0%)	23
	good oils	1 (5%)	19 (95%)	20
validation set		**bad oils**	**good oils**	**Total**
	bad oils	4 (66.6%)	2 (33.4%)	6
	good oils	1 (7.7%)	12 (92.3%)	13

Summary

- An analytical method based on dynamic headspace GC-MS has been developed.
- 13 volatile compounds showed significant differences between sensory good and bad virgin rapeseed oils.
- Linear Discriminant Analysis was used for the prediction of sensory rapeseed oil quality on basis of 13 volatile compounds.
- Cross validation showed that the model defined by the discriminant function worked satisfactorily.
- For the first time it was shown that differences in sensory rapeseed oil quality mainly derived from volatile compounds with significant higher amounts in sensory bad virgin rapeseed oil while no compounds showed significant higher amounts in sensory good oils.



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Thank you for your attention

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