BUNDESINSTITUT FÜR RISIKOBEWERTUNG

BfR

Risiken erkennen – Gesundheit schützen

Analytical Traceability of Food and Feed

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Definition: Traceability

Codex Alimentarius:

„***Traceability/product tracing:*** the **ability** to follow the movement of a food through specified stage(s) of production, processing and distribution.“

Traceability - Approaches

Labeling Documentation Database

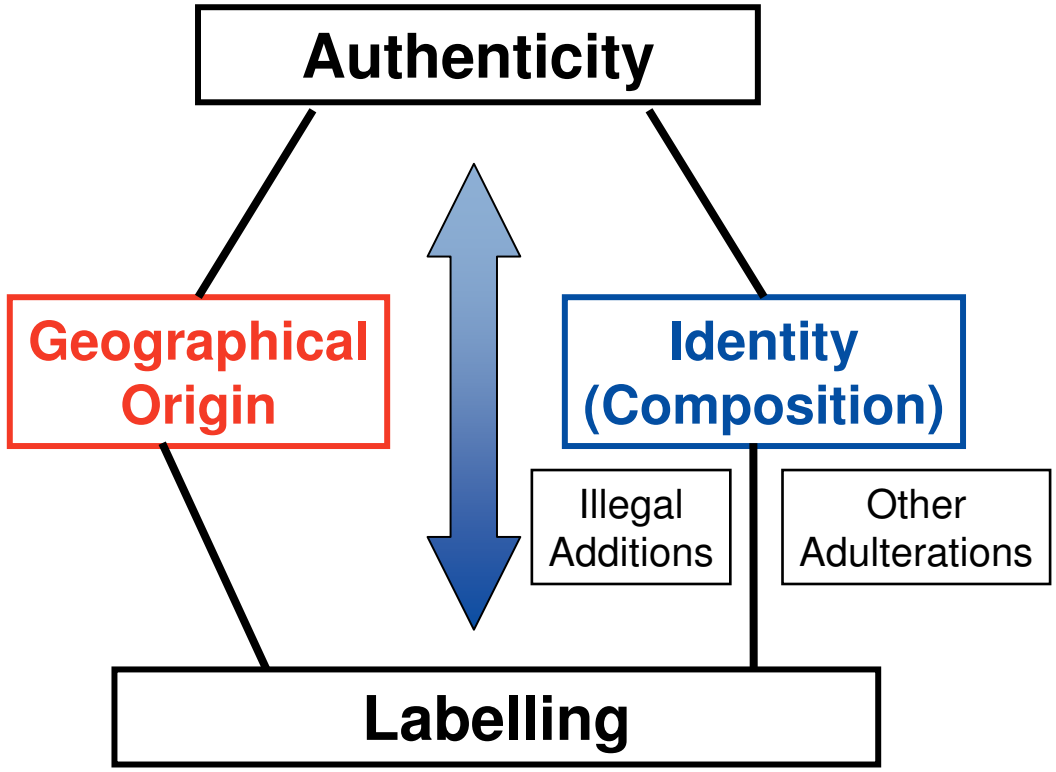
Traceability systems trace and track “food packaging”

- **Verification** with analytical methods

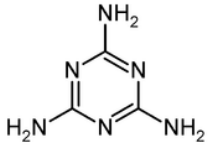
Starting points for analytical methods

BSE
Beef UK

Dioxin in
Irish pork



Melamine



Dyes

Methanol

- Substitution by cheaper but similar ingredient
- Extend food using adulterant, e.g. water, starch
- Undeclared process, e.g. irradiation, freezing
- Incorrect origin, e.g. geographic, species or method of production

Analytical methods for authentication

Analysis of composition

Classical analysis, wet chemistry, chromatography, **spectroscopy**,
Detection of non-natural food constitutes

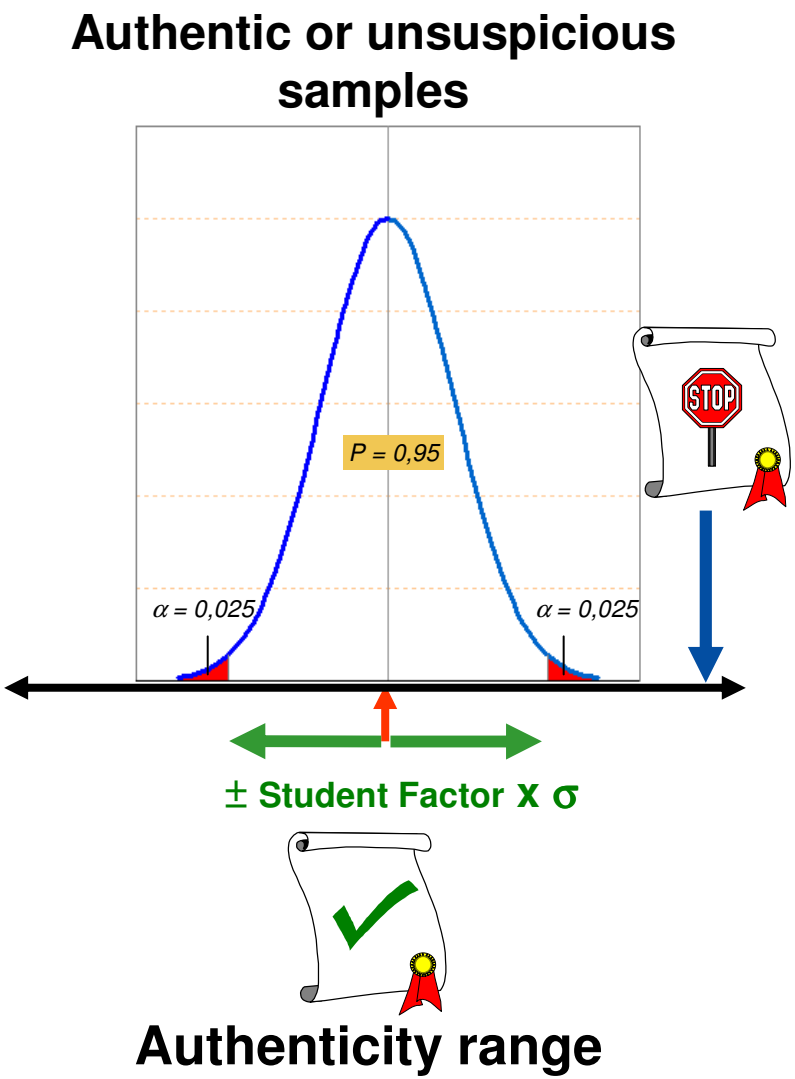
Analysis of stable isotopes

(D/H, $^{13}C/^{12}C$, $^{18}O/^{16}O$, $^{15}N/^{14}N$)

Enantioselective Analysis

Molecular biological Methods

Classical approach
Reference Data (bases)



„Classical approach“ Grape Variety (Shikimc acid)

HPLC

- Wines of the Burgundy Group show a low SA content

Natürliche Shikimisäuregehalt von Weinen der Burgundergruppe (alle Werte in mg/l)

	Burgunder-gruppe gesamt	Burgunder-gruppe (authentisch)	Burgunder-gruppe (Handelsware)	Pinot blanc	Pinot noir	Pinot grigio	Pinot Precoce Noir	Pinot Meunier
n	420	132	288	170	158	83	5	4
Mittelwert	15,06	14,58	15,28	15,26	15,91	13,42	11,56	11,50
s	±5,93	±5,28	±6,20	±5,48	±6,25	±6,03	±3,15	±4,20
min.	n.n.	1,3	n.n.	2	1,3	n.n.	6,4	6
max.	31	30,5	31	30	31	29	15	16
Median	14,0	14,0	14,0	14,0	15,6	13,0		
VB-95=(s·t)	±11,66	±10,46	±12,22	±10,83	±12,37	±12,02		
Minimal- und Maximalwerte des VB-95	3.4<15.1<26.7	4,1<14,6<25,0	3,1<15,3<27,5	4,4<15,3<26,1	3,5<15,9<28,3	1,4<13,4<25,4		

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- Wines of the Burg

Natürliche Shikimisäuregeh

	Burgunder-gruppe gesamt
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Mittelwert	15,06
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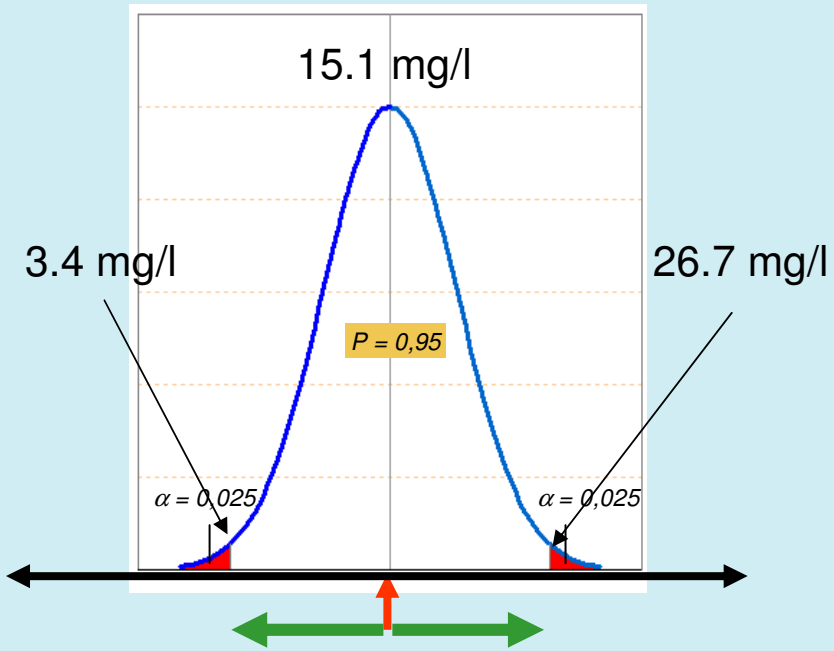
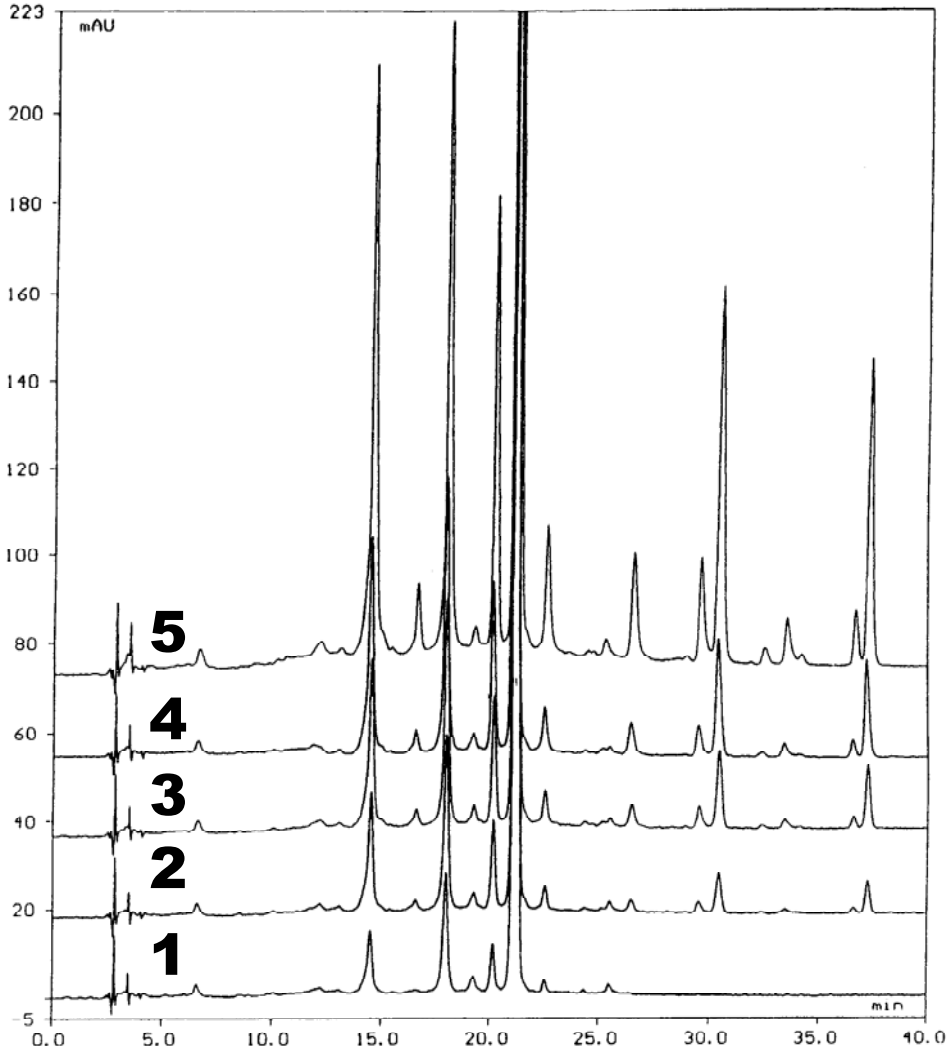


± Student Factor x σ

±1.96 x 5.93

„Pinot Noir“ characterisation



100 % Dornfelder

30 % Dornfelder + 70 % Pinot Noir

20 % Dornfelder + 80 % Pinot Noir

10 % Dornfelder + 90 % Pinot Noir

100 % Pinot Noir

Multivariate Statistical Approaches (Chemometrics)

•Matrix

Variable (analytical parameter, spectroscopic information..) →

Sample n

(possibly different groups) ↓

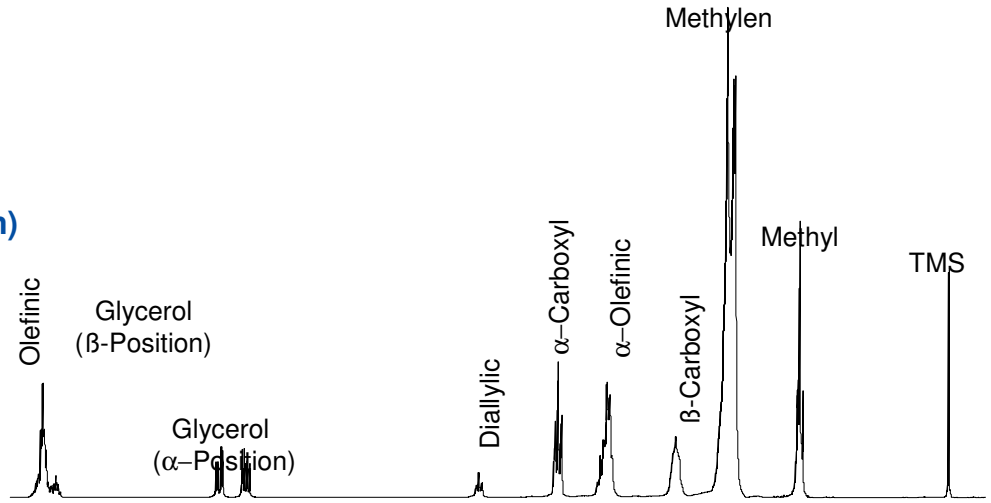
		Cluster	1	2	3	4	5	7
		ppm	4,36	4,35	4,34	4,33	4,32	4,22
Wein	Farbe	Land						
Wein_1379_1_	red	Hungary	0,031	0,054	0,024	0,074	0,100	0,464
Wein_1380_1_	red	Hungary	0,030	0,129	0,094	0,176	0,192	0,564
Wein_1381_1_	white	Hungary	0,317	0,267	0,287	0,273	0,179	0,208
Wein_1388_1_	red	Hungary	0,022	0,116	0,031	0,157	0,086	0,575
Wein_1389_1_	red	Hungary	0,275	0,180	0,273	0,159	0,184	0,113
Wein_1390_1_	red	Hungary	0,084	0,140	0,031	0,159	0,087	0,412
Wein_1391_1_	red	Hungary	0,610	0,419	0,413	0,436	0,398	0,397
Wein_1392_1_	red	Hungary	0,333	0,202	0,295	0,145	0,190	0,647
Wein_1395_1_	white	Hungary	0,528	0,275	0,247	0,354	0,270	0,314
Wein_1396_1_	white	Hungary	0,026	0,042	0,044	0,038	0,070	0,041
Wein_1397_1_	white	Hungary	0,462	0,426	0,361	0,185	0,243	0,035
Wein_1398_1_	white	Hungary	0,464	0,415	0,372	0,358	0,294	0,294

- **Unsupervised methods (strutuce discovery)**
  e.g. Cluster Analysis, Principal Component Analysis (PCA)
- **Supervised methods**
  Discriminant analysis (DA), Class moddelling (e.g. SIMCA)
- **Quantification**
  Partial Least Squares (PLS)

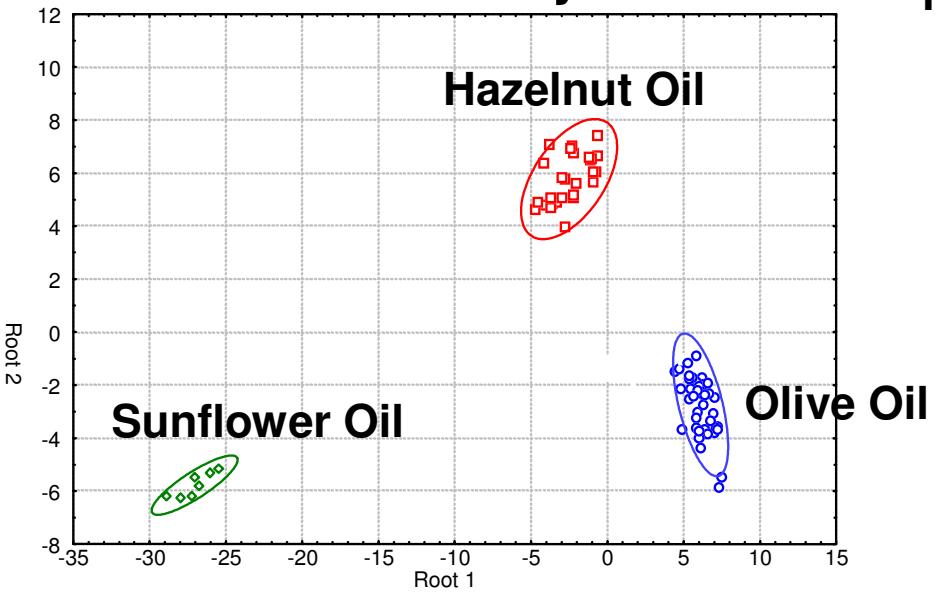
Olive oil

- **subject of falsification**
  1981 Toxic oil syndrome

(Rapeseed oil denaturated with 2% anillin)

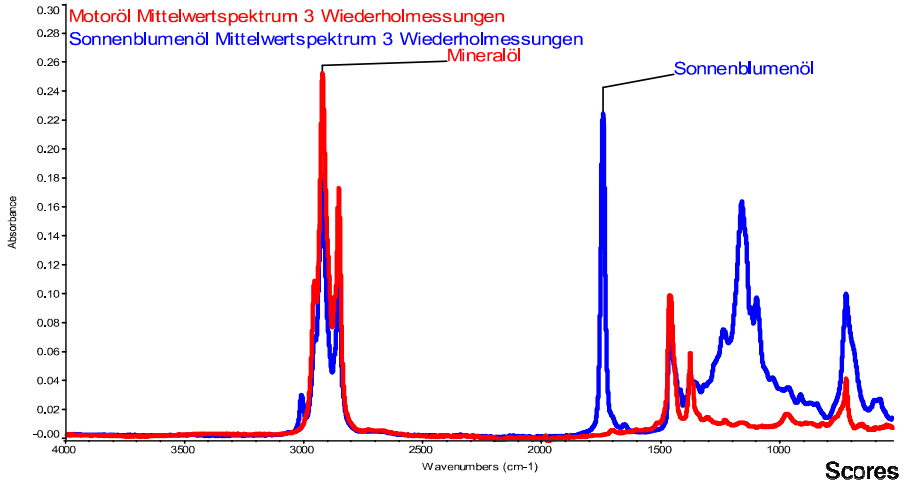


Discriminant Analysis

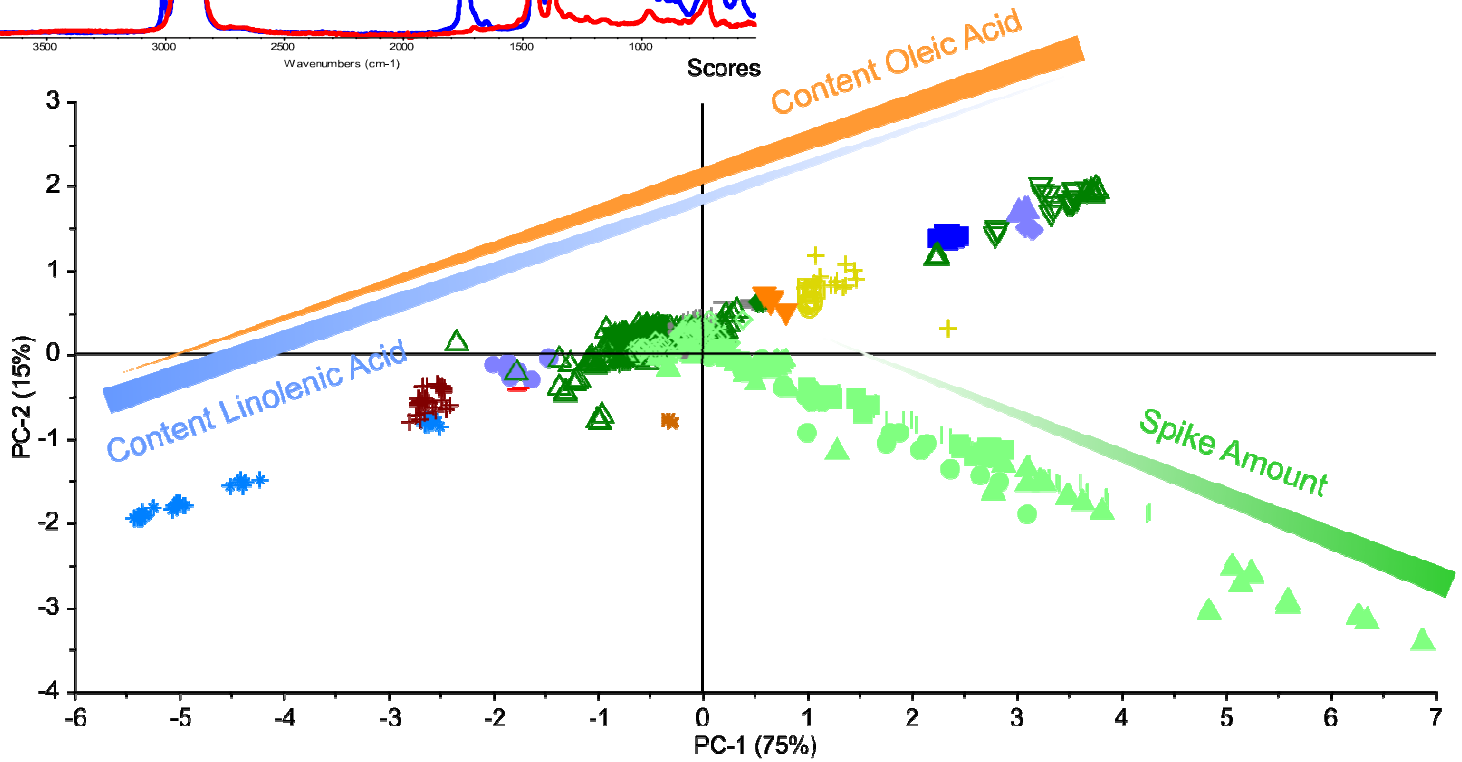


^{1}H-NMR-Measurements

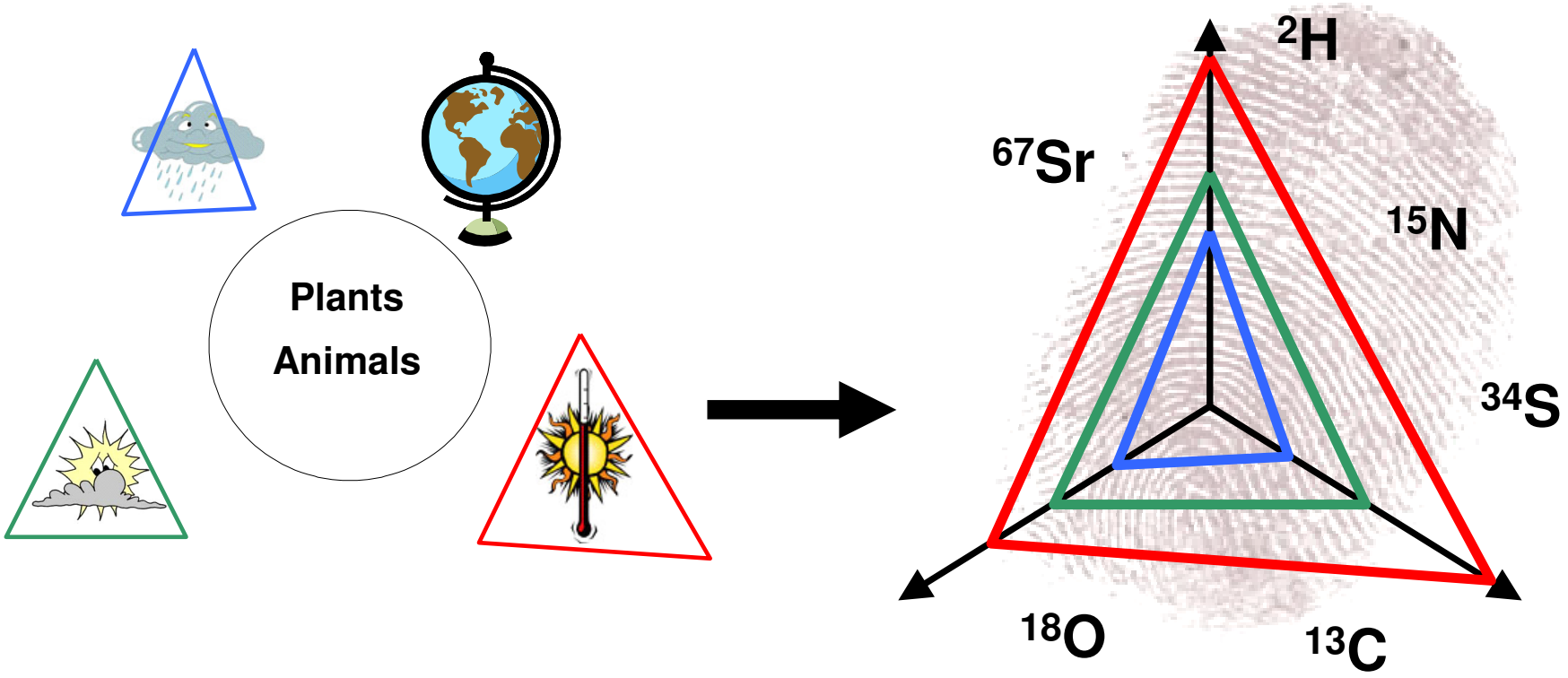
FT-IR analysis of edible oils: Addition of mineral oil



- Adddition of mineral as fraud
- Detection >1 %

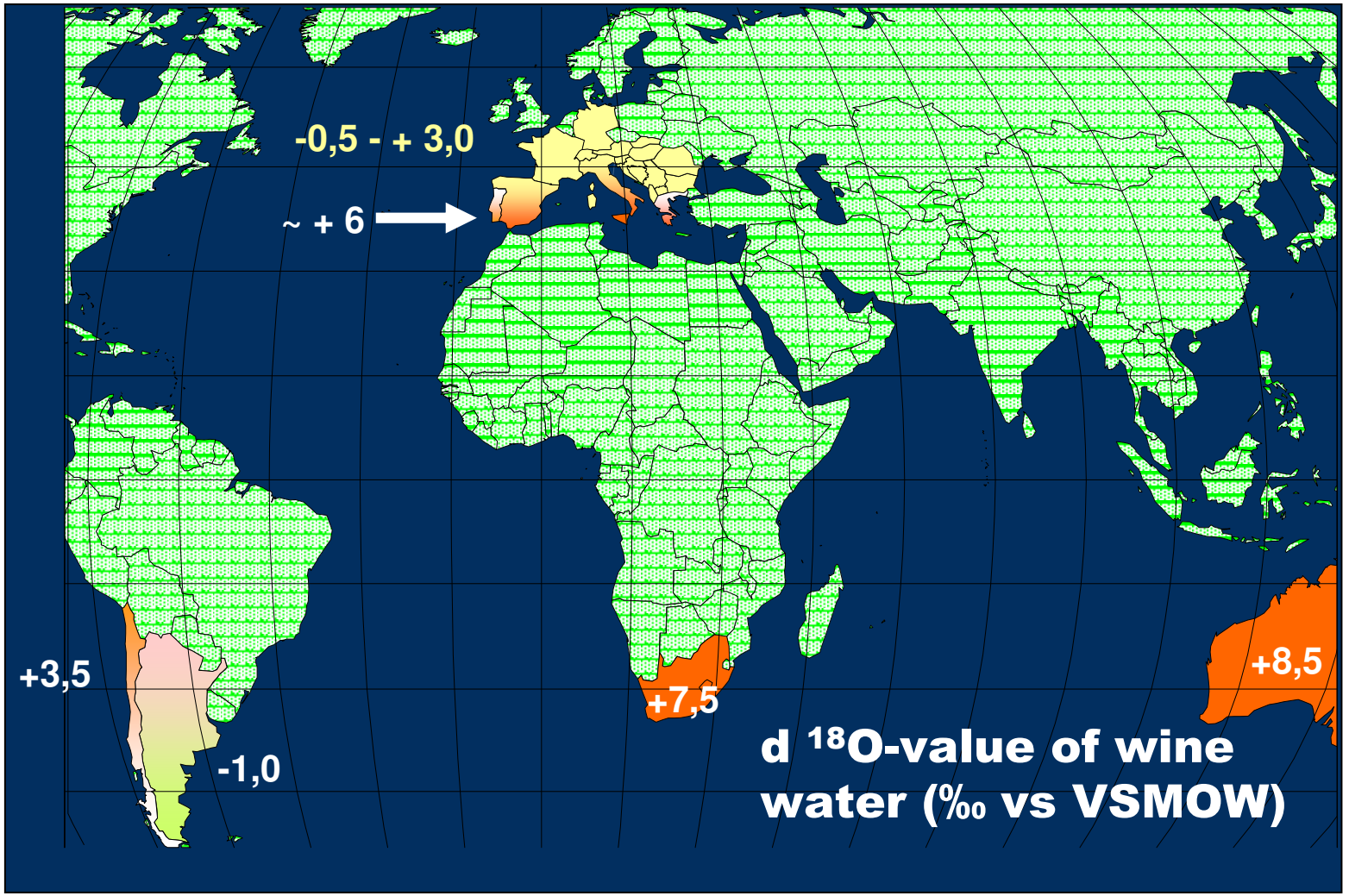


Stable Isotope Ratios “Fingerprint”



Geographical Origin

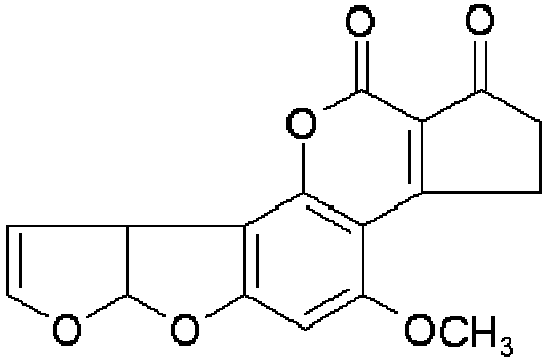
Isotopic analysis



Example - Authenticity control of pistachios

pistachios are popular snacks

Aflatoxine in Iran pistachios
1997 import-stop
strictly EU-import regulations



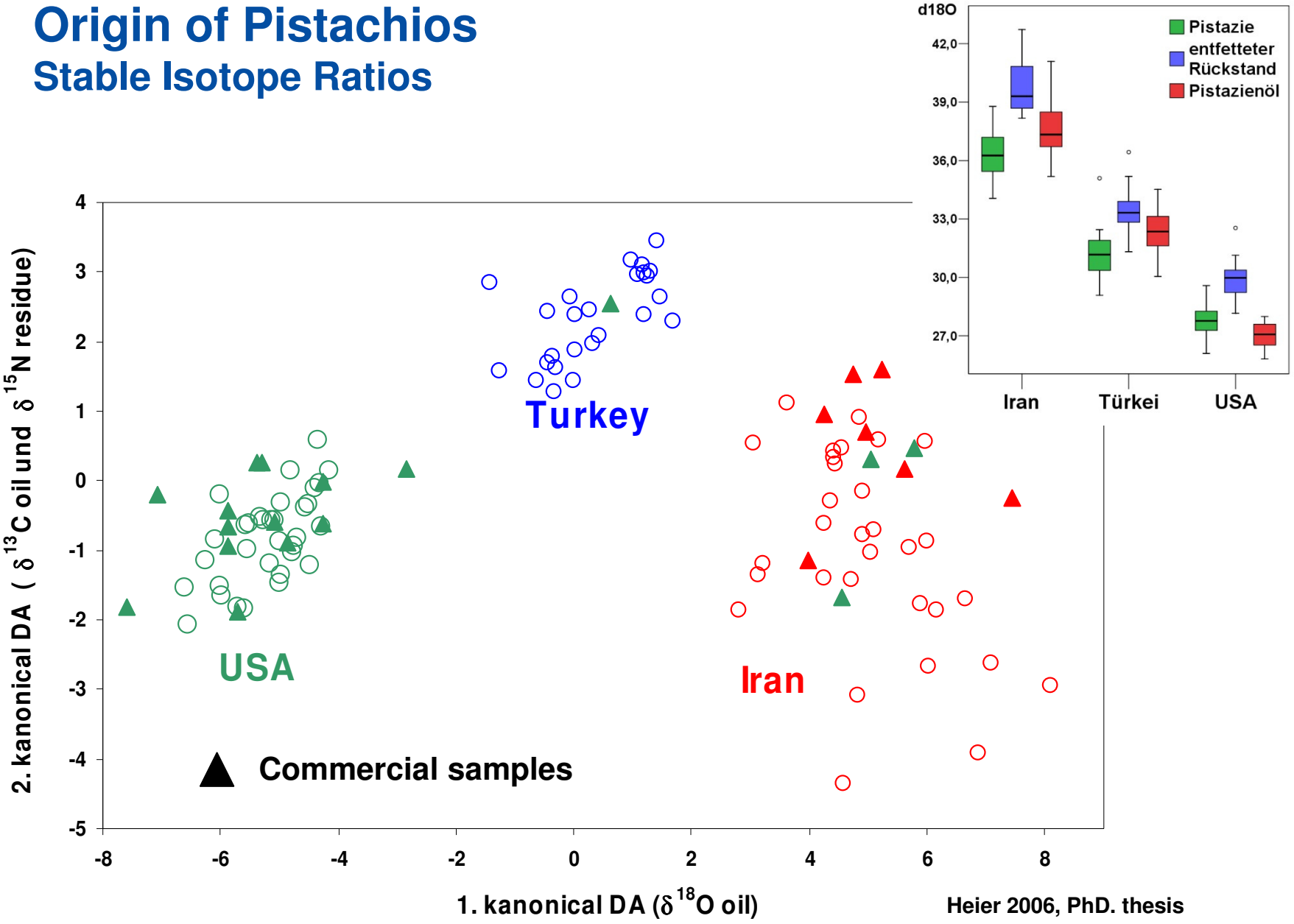
California

Iran

false declaration???

Authenticity control necessary

Origin of Pistachios
Stable Isotope Ratios



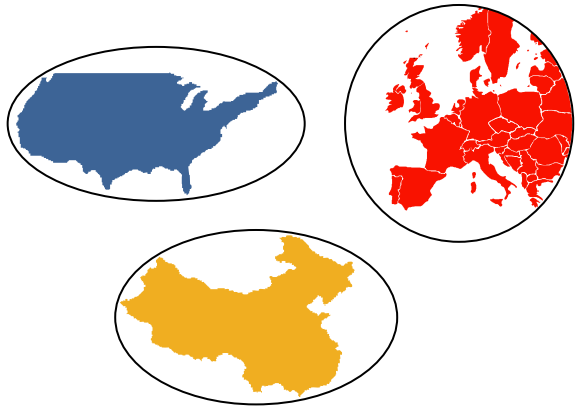
Heier 2006, PhD. thesis

Feed Origin EU project

DDGS

- co-product of ethanol production
- high nutrient content (protein, fat)

Geographical Origin

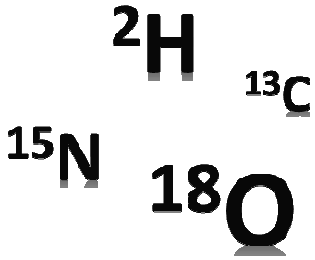


- DDGS are globally traded commodity
- crisis situations often associated with particular regions/countries

Stable Isotope Ratio Mass Spectrometry

- technique for food authentication
- geographical origin

Feed Origin Results



See Nietner, T., et al., Determination of geographical origin of distillers' dried grains and solubles using isotope ratio mass spectrometry, Food Research International (2013), http://dx.doi.org/10.1016/j.foodres.2013.11.002

Canonical discriminant analysis (CDA)

$$Y = b_0 + b_1\delta^{2}H + b_2\delta^{13}C + b_3\delta^{15}N + b_4\delta^{18}O$$

- **use of model for new samples**

Conclusions

- Different analytical approaches for authentication available
- Reference data (banks) needed !
  - *Application of unified methods of analysis*
  - *Recognition of authenticity ranges*
- Trend to spectroscopic methods/multivariate evaluations
  - *Often feasibility studies which have limited scope/questions*

Outlook

- Globalisation also in terms of fraud, „prediction“ difficult
- Health risks are „accepted“ by fraudsters
- Non-Targeted Analysis/Finger-Printing techniques will become more important
  - *Detection of „abnormalities“ will be the challenge*

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Thank you for your attention

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