
بسم الله الرحمن الرحیم

بِسْمِ اللّٰهِ الرَّحْمنِ الرَّحِيمِ

الحمدلله المتجلي بجماله

المحتجب بجلاله

و الصلوة علي محمد و آله

Introduction to "OIC/SMIIC 35:2020 and Challenges with Halal Authenticity

Mohammad Hossein Shojaee AliAbadi DVM,PhD

INTERNATIONAL CONFERENCE ON
"GLOBALISING THE TRUST IN HALAL CERTIFICATION"
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Senior Scientific Adviser of Iran National Standards Organization (INSO)
Faroogh Life Sciences Research Laboratory, Iran

THE STANDARDS AND METROLOGY INSTITUTE FOR ISLAMIC COUNTRIES

Organisation of Islamic Cooperation

OIC Global Halal Quality Infrastructure

ELEMENTS OF OIC GLOBAL HALAL QUALITY INFRASTRUCTURE

DESIGNATED STRUCTURE

Peer Evaluation

- Develop additional documents regarding MLA/MRA and peer evaluation to implement the requirements of OIC/SMIIC standards;
- Perform peer-assessments of ABs and regional organizations;
- Coordinate with legislators to approve the results of conformity assessments.

OIC Member States

Issue notification based on registration and accreditation

Accreditation Bodies

Perform accreditation assessment according requirements of OIC/SMIIC standards

Conformity Assessment Bodies

Apply for notification and accreditation from Member States wishing to operate in their markets.

SMIIC

Develop OIC/SMIIC standards

Requirements for Halal Accreditation Bodies Accrediting Halal Conformity Assessment Bodies

Specifies requirements for the competence, consistent operation and impartiality of accreditation bodies

SMIIC/CCA

OIC/SMIIC 3:2019

Requirements for Bodies Providing Halal Certification

Specifies the rules that the halal certification bodies shall satisfy and the requirements for the execution of halal certification activities.

SMIIC/CCA

OIC/SMIIC 2:2019

Producers, Manufacturers and Service Providers

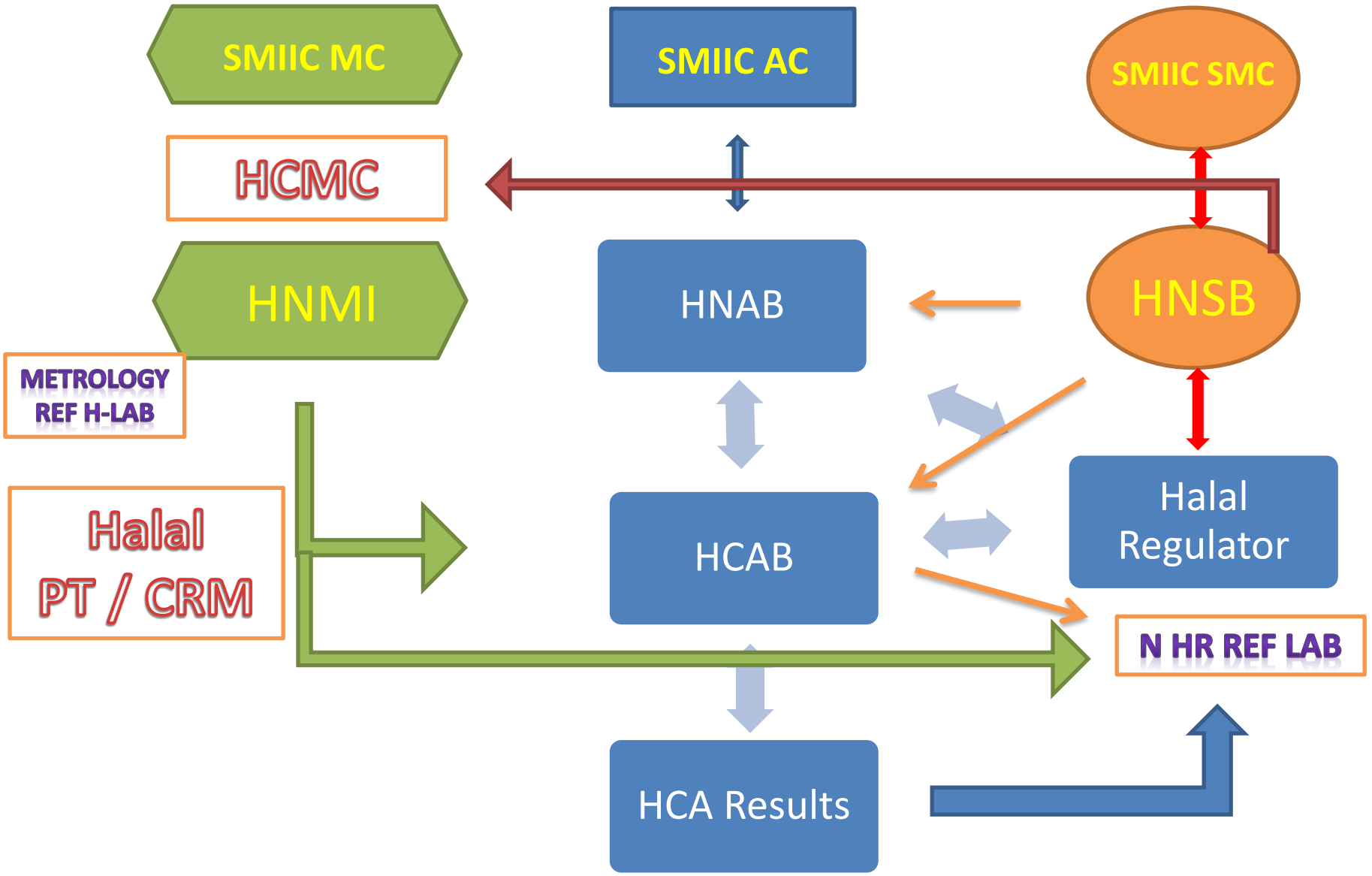
OIC/SMIIC standards halal related to products (product/service/process)

SMIIC TCs

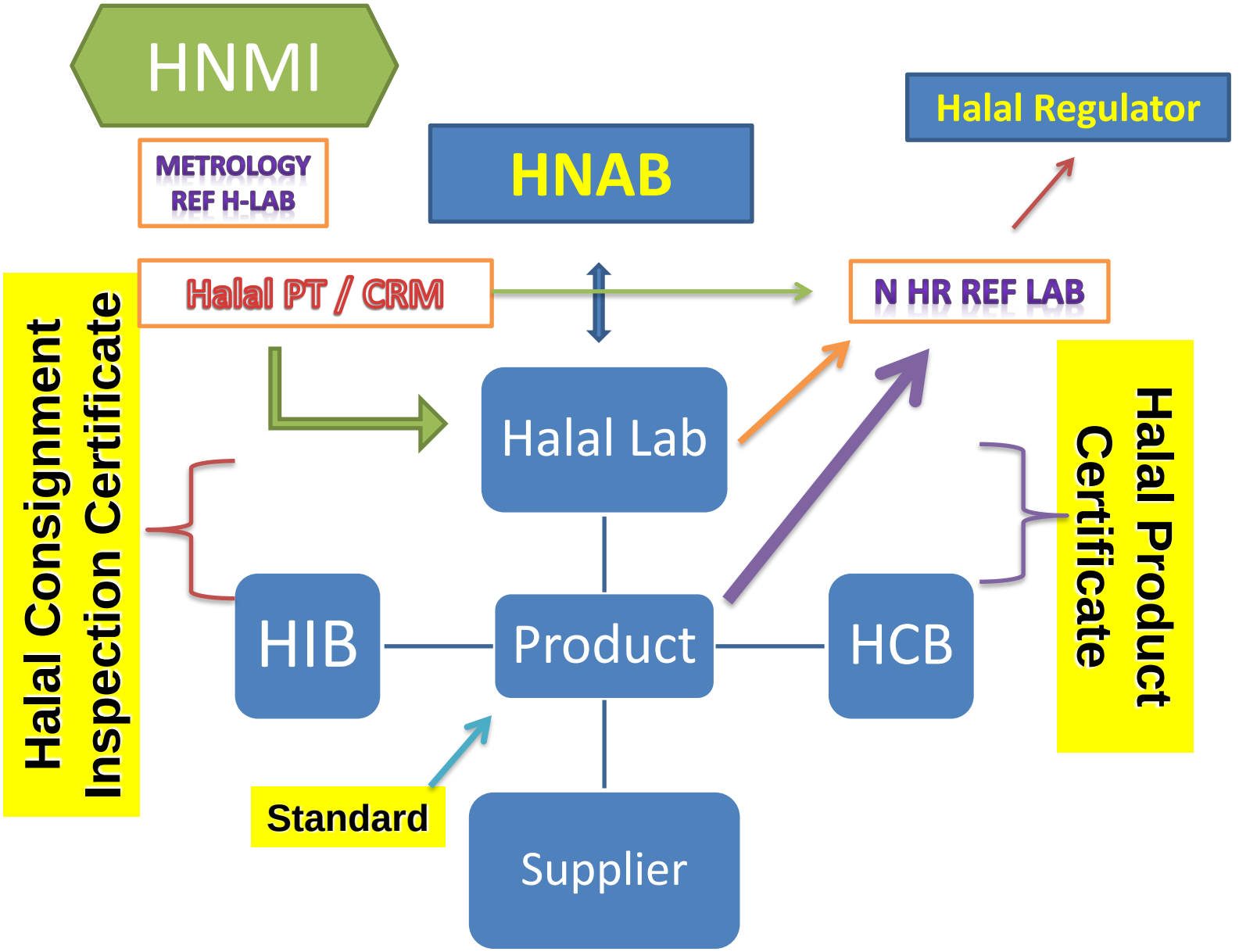
OIC/SMIIC 1:2019

OIC/SMIIC 4:2018

Other OIC/SMIIC standards



OIC Global Halal Quality Infrastructure



Tangible Products

OIC/SMIIC 35: 2020

OIC/SMIIC 35: 2020

OIC/SMIIC 35: 2020

First Edition
20-02-2020

Conformity Assessment - General Requirements for the Competence of Laboratories Performing Halal Testing

The Standards and Metrology Institute for Islamic Countries
l'Institut de Normalisation et de Métrologie pour les Pays Islamiques
معهد المواصفات والمقاييس للدول الإسلامية

OIC/SMIIC 35 : 2020

FOREWORD **(SMIIC + SMIIC CCA)**

INTRODUCTION **(17025)**

1 SCOPE

2 NORMATIVE REFERENCES **(17025+OIC/SMIIC 1)**

3 TERMS AND DEFINITIONS **(17025+OIC/SMIIC 1)**

4 GENERAL REQUIREMENTS REQUIREMENTS

5 STRUCTURAL REQUIREMENTS **(Muslim + Islamic values)**

6 RESOURCE REQUIREMENTS **(Halal training + Muslim + DNA/Halal/Najis Contamination)**

7 PROCESS REQUIREMENTS **(Islamic Cleansing + Halal/Najis Contamination + No halal mark on report)**

8 MANAGEMENT SYSTEM REQUIREMENTS

Bibliography **(17025 + OIC/SMIIC 1)**

(Muslim+Non Halal/Najis Contamination)

OIC/SMIIC 35: 2020

OIC/SMIIC 35: 2020

1 SCOPE

This document specifies the **general requirements** for Laboratories performing Halal Testing.

All the organizations performing laboratory activities are included to the scope of this document.

Compliance to this document does not in any way exempt laboratories from or diminish their responsibilities in observing/complying with **existing national laws and regulations/guidelines currently enforced in the country**.

OIC/SMIIC 35: 2020

2 NORMATIVE REFERENCES

- **ISO/IEC 17025,** General requirements for the competence of testing and calibration laboratories
- **ISO/IEC 17000,** Conformity assessment — Vocabulary and general principles
- **OIC/SMIIC 1**, General requirements for halal food

OIC/SMIIC 35: 2020

3 TERMS AND DEFINITIONS

- For the purposes of this document, the terms and definitions given in
  - **ISO/IEC 17000,**
  - **ISO/IEC 17025 and**
  - **OIC/SMIIC 1**
- shall apply.

OIC/SMIIC 35: 2020

4 GENERAL REQUIREMENTS

- For the purpose of this document, all requirements given in Clause 4 of ISO/IEC 17025:2017 shall apply.

- 4.1-Impartiality
  - All the requirements given in clause 4.1 of ISO/IEC 17025:2017 shall apply.

- 4.2-Confidentiality
  - All the requirements given in clause 4.2 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

5 STRUCTURAL REQUIREMENTS

All the requirements given in clause 5 of ISO/IEC 17025:2017 and the following shall apply.

5.1 The laboratory shall appoint a member of staff who shall be a **competent Muslim**, irrespective of other duties and responsibilities, shall have **defined responsibility and authority for ensuring that activities of halal testing is implemented** and followed at all times; he/she shall also have **direct access to the highest level of management** at which decisions are made on laboratory policy or resource.

OIC/SMIIC 35: 2020

5 STRUCTURAL REQUIREMENTS

5.2 Laboratories Performing Halal Testing staff and all of its **employees shall be committed to all Islamic values** especially to those related to halal.

OIC SMIIC 2:2019

3.13 Islamic Entity

the legal entity which is wholly **owned** by Muslims, and at the same time **managed** and **operated** by Muslims.

OIC/SMIIC 35: 2020

6 RESOURCE REQUIREMENTS

6.1 General

The requirement given in clause 6.1 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

6 RESOURCE REQUIREMENTS

6.2 Personnel

All the requirements given in clause 6.2 of ISO/IEC 17025:2017 and the following shall apply.

6.2.1 The laboratory shall have **suitable trainings** planned for laboratory staff in the **relevant area/topics of halal**, provided that record of such trainings shall be kept.

6.2.2 The laboratory shall ensure that technical **personnel assigned to perform halal testing should be Muslim** and shall undergo appropriate **halal related training**.

6.2.3 Competent **personnel authorized for the review and authorization of halal testing** result shall be **a Muslim**.

OIC/SMIIC 35: 2020

6 RESOURCE REQUIREMENTS

6.3 Facilities and environmental conditions

All the requirements given in clause 6.3 of ISO/IEC 17025:2017 and the following shall apply.

6.3.1 For laboratories performing molecular techniques involving in-vitro **nucleic acid amplification**, **separate rooms/spaces** for nucleic acid extraction, amplification, and detection shall be provided to **minimize the risk of cross-contamination**.

OIC/SMIIC 35: 2020

6 RESOURCE REQUIREMENTS

6.4 Equipment

All the requirements given in clause 6.4 of ISO/IEC 17025:2017 and the following shall apply.

6.4.1 The **equipment** used for laboratory purposes **shall not be made of or contain any materials** that are decreed as **non-halal or Najis** by the laws of Islam.

6.4.2 **Oils/grease** used in the maintenance of equipment and devices, that may come into **contact with the product**, shall be food grade oil and shall not contain any ingredients that are **non-halal or Najis.**

OIC/SMIIC 35: 2020

6 RESOURCE REQUIREMENTS

6.5 Metrological traceability

All the requirements given in clause 6.5 of ISO/IEC 17025:2017 shall apply.

6.6 Externally provided products and services

All the requirements given in clause 6.6 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.1 Review of requests, tenders and contracts

All the requirements given in clause 7.1 of ISO/IEC 17025:2017 shall apply.

7.2 Selection, verification and validation of methods

7.2.1 Selection and verification of methods

All the requirements given in clause 7.2.1 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.2.2 Validation of methods

All the requirements given in clause 7.2.2 of ISO/IEC 17025:2017 shall apply.

7.3 Sampling

All the requirements given in clause 7.3 of ISO/IEC 17025:2017 shall apply

OIC/SMIIC 1: 2019

General Requirements for Halal Food

10 VALIDATION AND VERIFICATION

10.1 Validation and verification of methods

Halal Authenticity Confirmatory test methods of analysis used for Halal food control purposes shall be prove beyond reasonable doubt are:

a) Objectively identified from the Halal source of food,

b) It is free from any non-Halal and Najis components according on risk based approach,

c) The requirements of slaughtering according to Islamic Rules are fulfilled (wherever is possible)

OIC/SMIIC FDS 22

Halal Edible Gelatin – Requirements And Test Methods

Table 1 – Characteristics, requirements and methods of test of gelatine

No	Characteristics	Requirements	Methods of Test (Ref. to Annex)
HALAL AUTHENTICITY			
xx)	Authenticity Test of Raw Materials	Positively Identified	-
xxi)	Authenticity Test for Halal Animal Origin (e.g., Bovine)	Positively Identified	-
xxii)	Authenticity Test for non Halal Animal Origin (e.g., Porcine)	Negative (LOD of Validated Method)	-
xxiii)	Positive Identification of Halal Slaughtering provisions (only from certified sources / or any validated test)	Positively Identified	-
xxiv)	confirmation of Traceability of raw material	Positively confirmation of (bone, skin, acid / lime)	-

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.4 Handling of test or calibration items

All the requirements given in clause 7.4 of ISO/IEC 17025:2017 and the following shall apply.

7.4.1 The laboratory shall document measures to be taken to **ensure no cross contamination between samples**. Measures shall include **method of cleaning according to Islamic Rules** for **equipment, area, laboratory staff which have been in contact with non-halal source**. There shall be a **proper sample segregation and storage** prior to testing and during examination process to ensure no contamination.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.5 Technical records

All the requirements given in clause 7.5 of ISO/IEC 17025:2017 shall apply.

7.6 Evaluation of measurement uncertainty

All the requirements given in clause 7.6 of ISO/IEC 17025:2017 shall apply.

7.7 Ensuring the validity of results

All the requirements given in clause 7.7 of ISO/IEC 17025:2017 shall apply.

7.8 Reporting of results

7.8.1 General

All the requirements given in clause 7.8.1 of ISO/IEC 17025:2017 shall apply.

7.8.2 Common requirements for reports (test, calibration or sampling)

All the requirements given in clause 7.8.2 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.8.3 Specific requirements for test reports

All the requirements given in clause 7.8.3 of ISO/IEC 17025:2017 and the following shall apply.

7.8.3.1 The laboratory **shall not issue a halal mark on report** about the suitability of the product in question as halal. The laboratory shall **only provide technical/scientific test results and interpretation** of its analysis in their reports. Islamic interpretation of results are beyond the competency of the laboratory and shall be avoided.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.8.4 Specific requirements for calibration certificates

All the requirements given in clause 7.8.4 of ISO/IEC 17025:2017 shall apply.

7.8.5 Reporting sampling – specific requirements

All the requirements given in clause 7.8.5 of ISO/IEC 17025:2017 shall apply.

7.8.6 Reporting statements of conformity

All the requirements given in clause 7.8.6 of ISO/IEC 17025:2017 shall apply.

7.8.7 Reporting opinions and interpretations

All the requirements given in clause 7.8.7 of ISO/IEC 17025:2017 shall apply.

7.8.8. Amendments to reports

All the requirements given in clause 7.8.8 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35: 2020

7 PROCESS REQUIREMENTS

7.9 Complaints

All the requirements given in clause 7.9 of ISO/IEC 17025:2017 shall apply.

7.10 Nonconforming work

All the requirements given in clause 7.10 of ISO/IEC 17025:2017 shall apply.

7.11 Control of data and information management

All the requirements given in clause 7.11 of ISO/IEC 17025:2017 shall apply.

8 MANAGEMENT SYSTEM REQUIREMENTS

All the requirements given in clause 8 of ISO/IEC 17025:2017 shall apply.

OIC/SMIIC 35 : 2020

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OIC/SMIIC 35: 2020

Islam & Science

What we measure?

- **Measurand =** A quantity or property intended to be measured

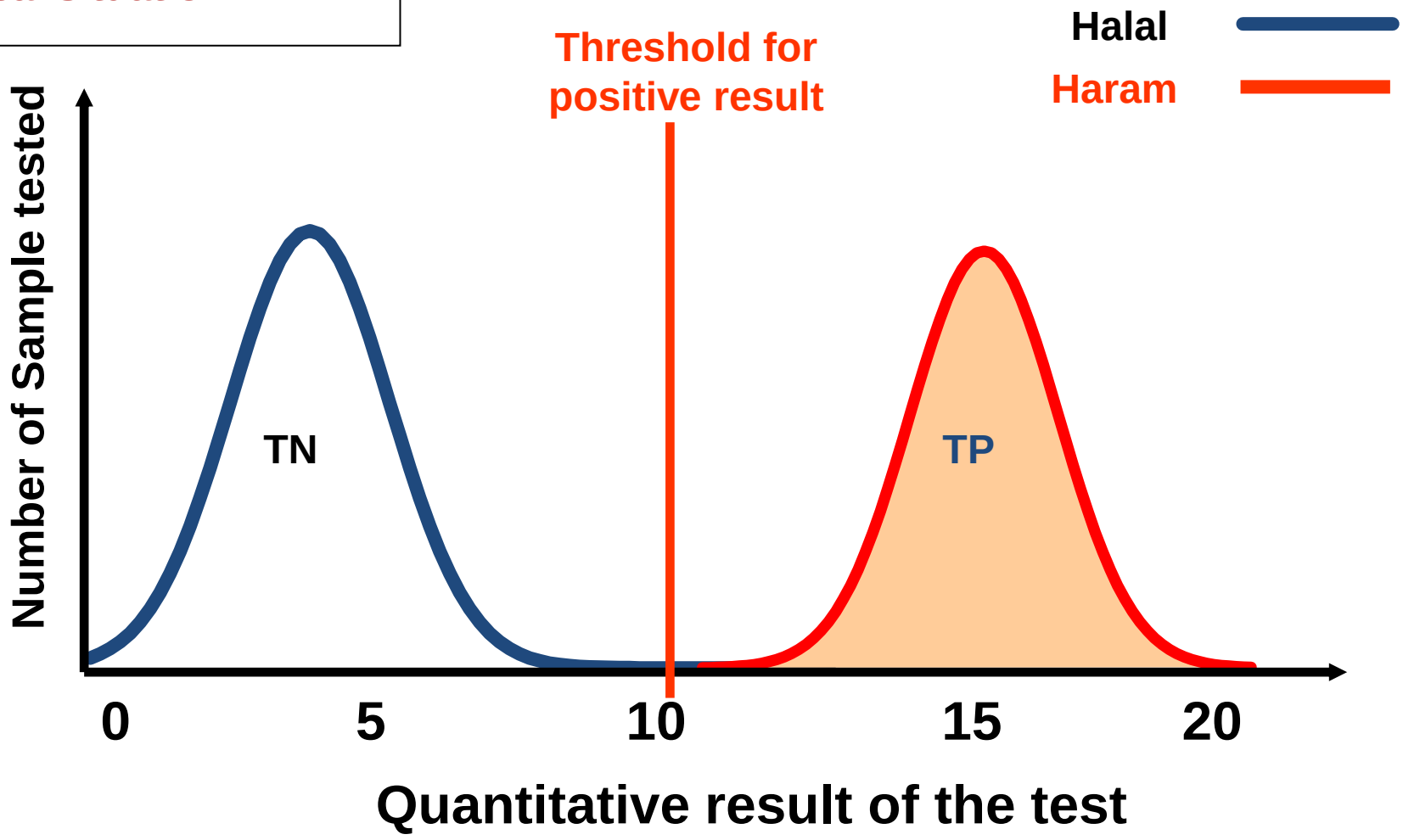
- Surrogate marker vs Target Marker
  - How Specific is?
  - Could be destroyed?
  - Could be Masked?
- In Speciation test:
  - DNA in meat is a Target Marker
  - DNA in Gelatine is a Surrogate Marker
  - Segments of Bovine alpha 1 Collagen tryptic Peptides are a Target Marker

Two-way table

	Test Results	
True Condition Status	Halal (+)	Haram (-)
Halal (+)	**True Positive**	**False Negative**
Haram(-)	**False Positive**	**True Negative**

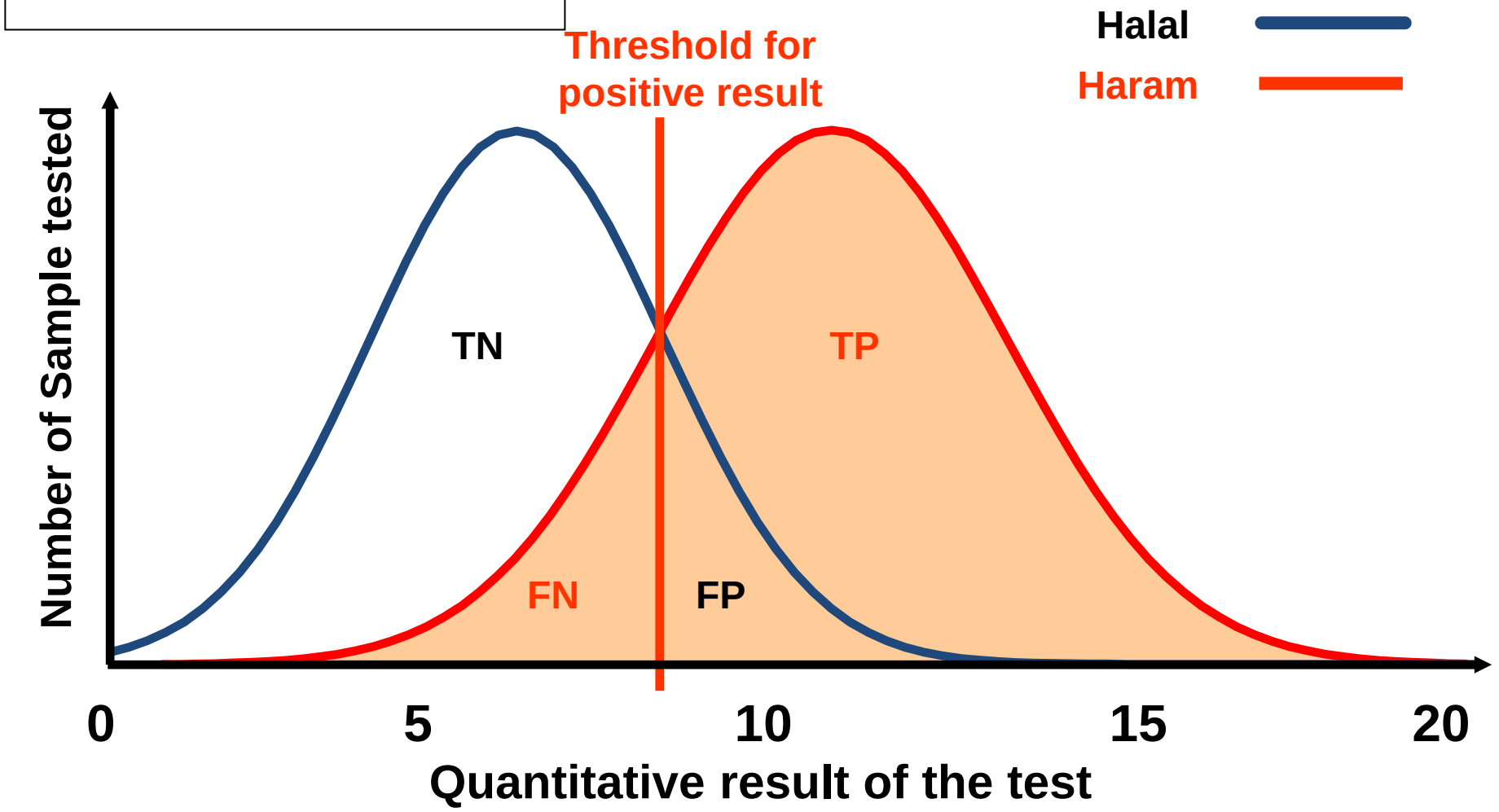
Distribution of quantitative test results among Halal and Haram

Ideal situation

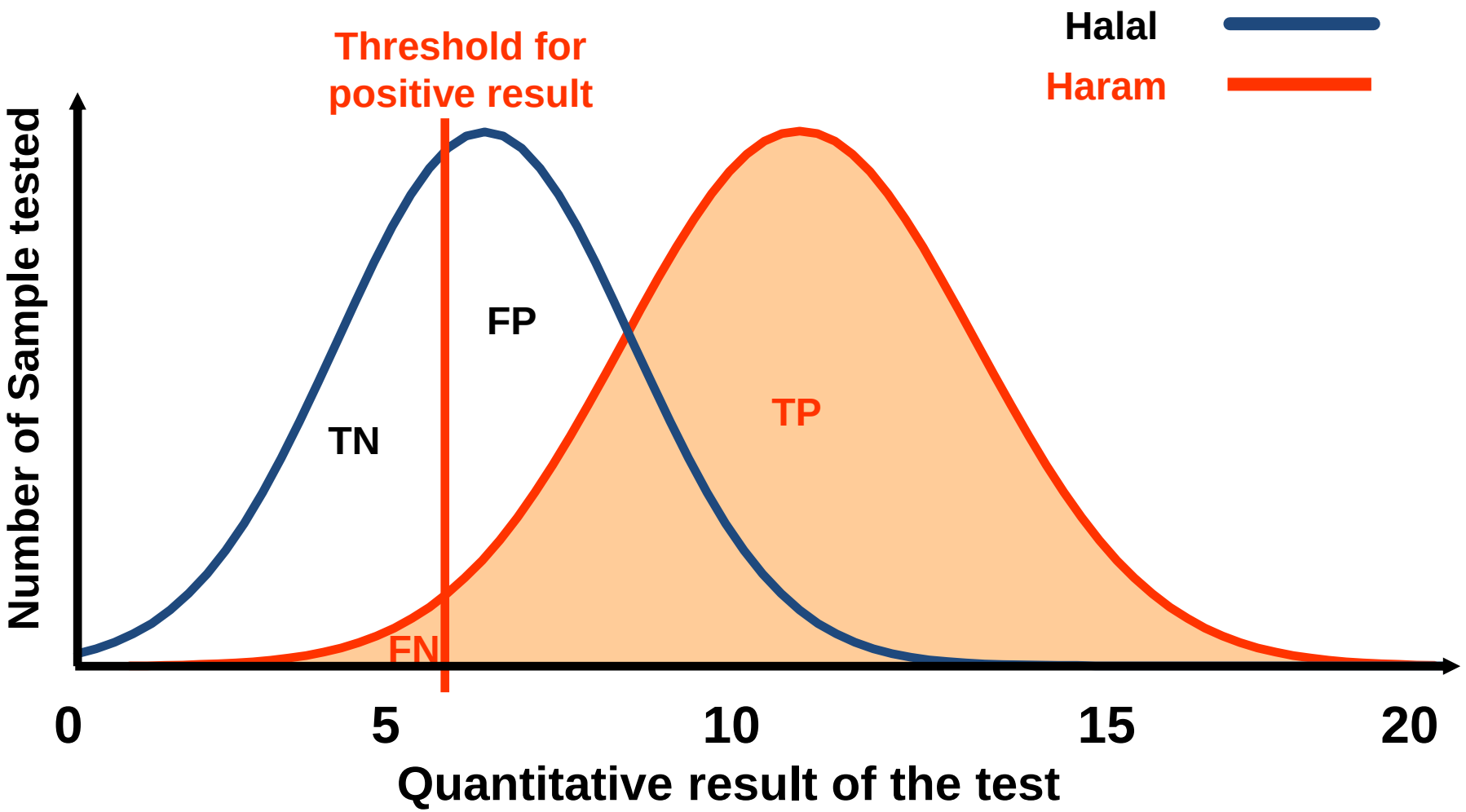


Distribution of quantitative results among affected and non-affected people

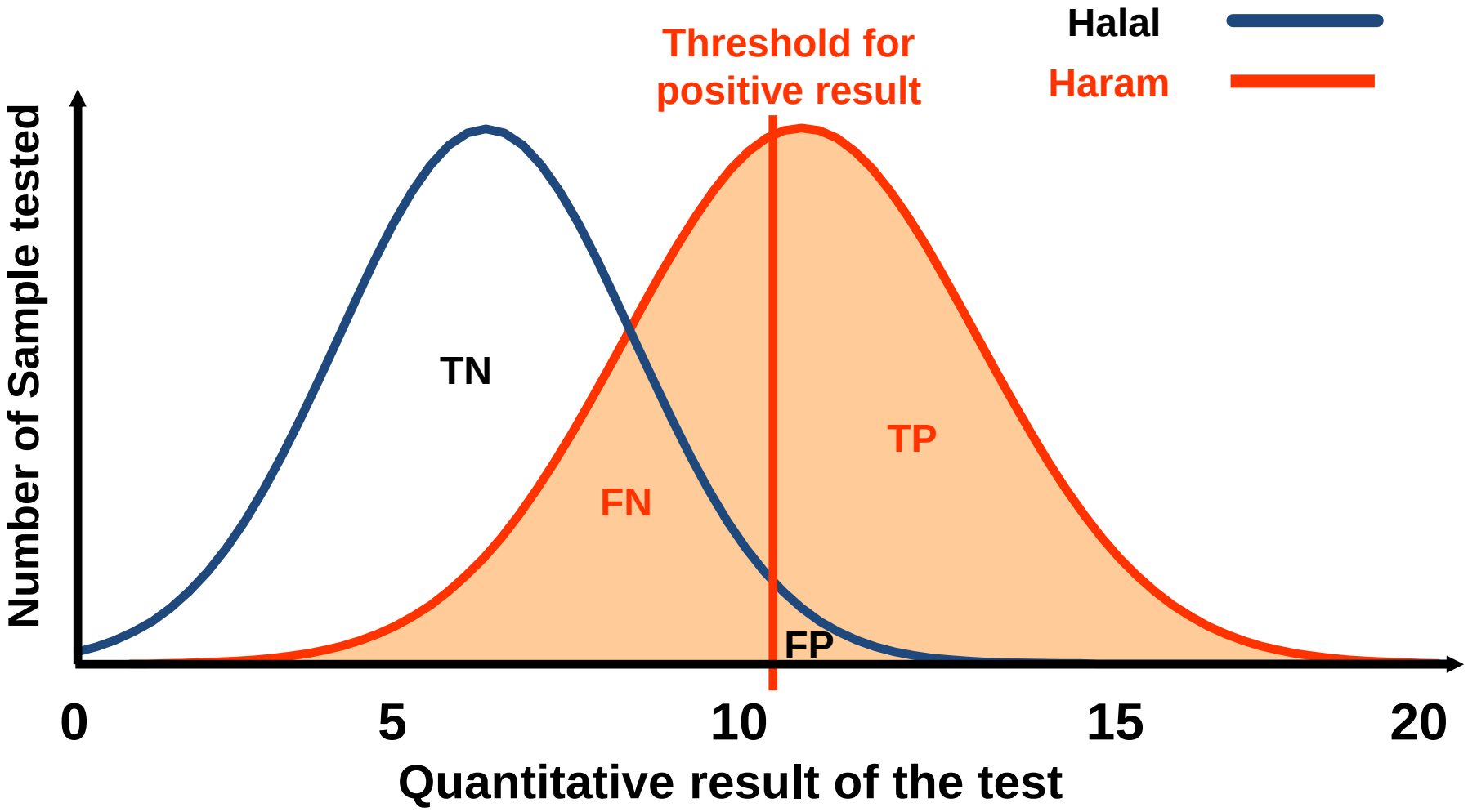
Realistic situation



Effect of Decreasing the Threshold



Effect of Increasing the Threshold

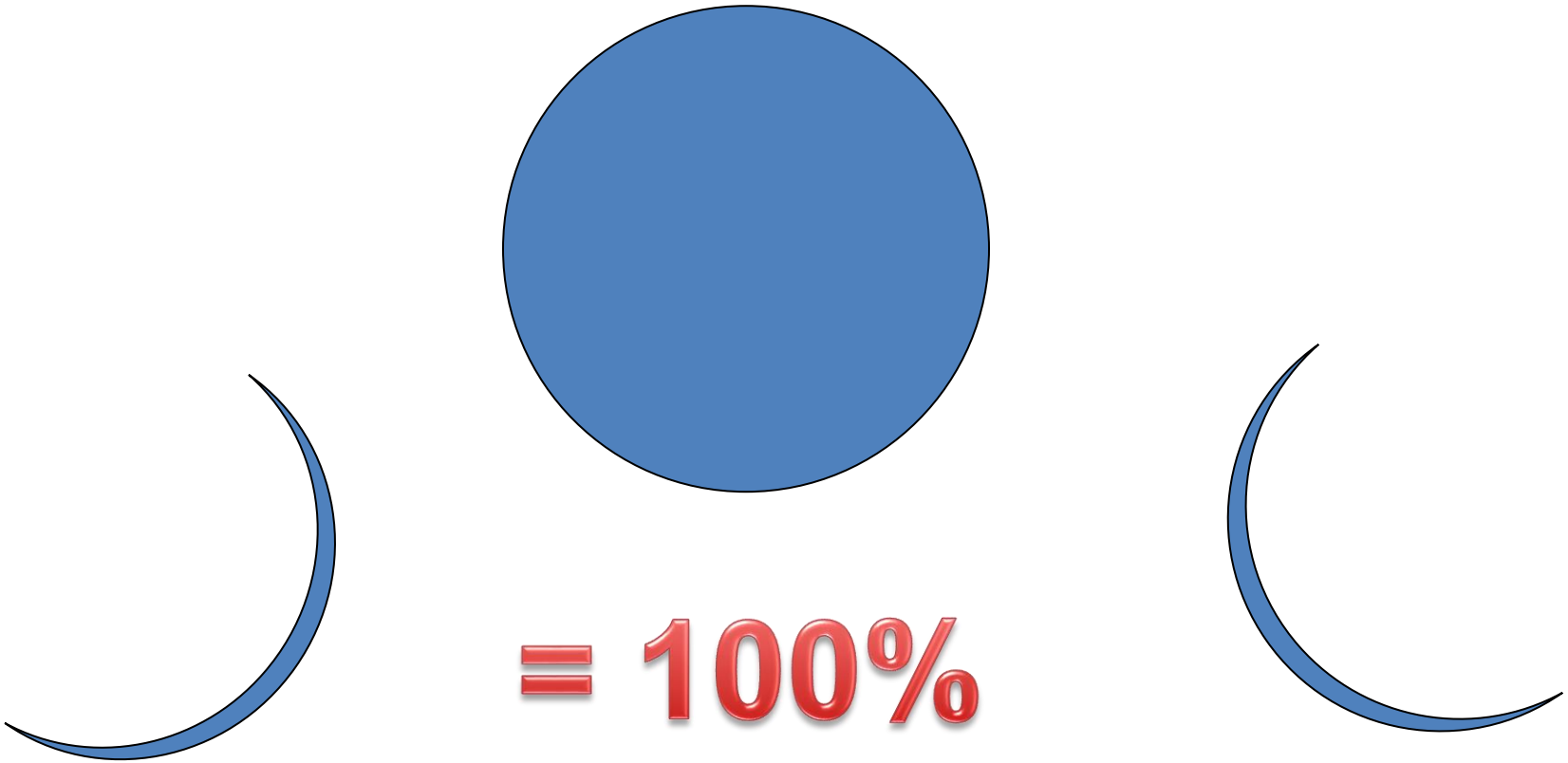


Definition – Halal Food Integrity

- Constant threat to Integrity of Halal Foods
- 1) Misunderstanding the Halal
  - Not a clear technical definition
  - Limited awareness in different sections
- 2) Fraudulently labelled imitations
  - to exploit its added value.

Islam & Science

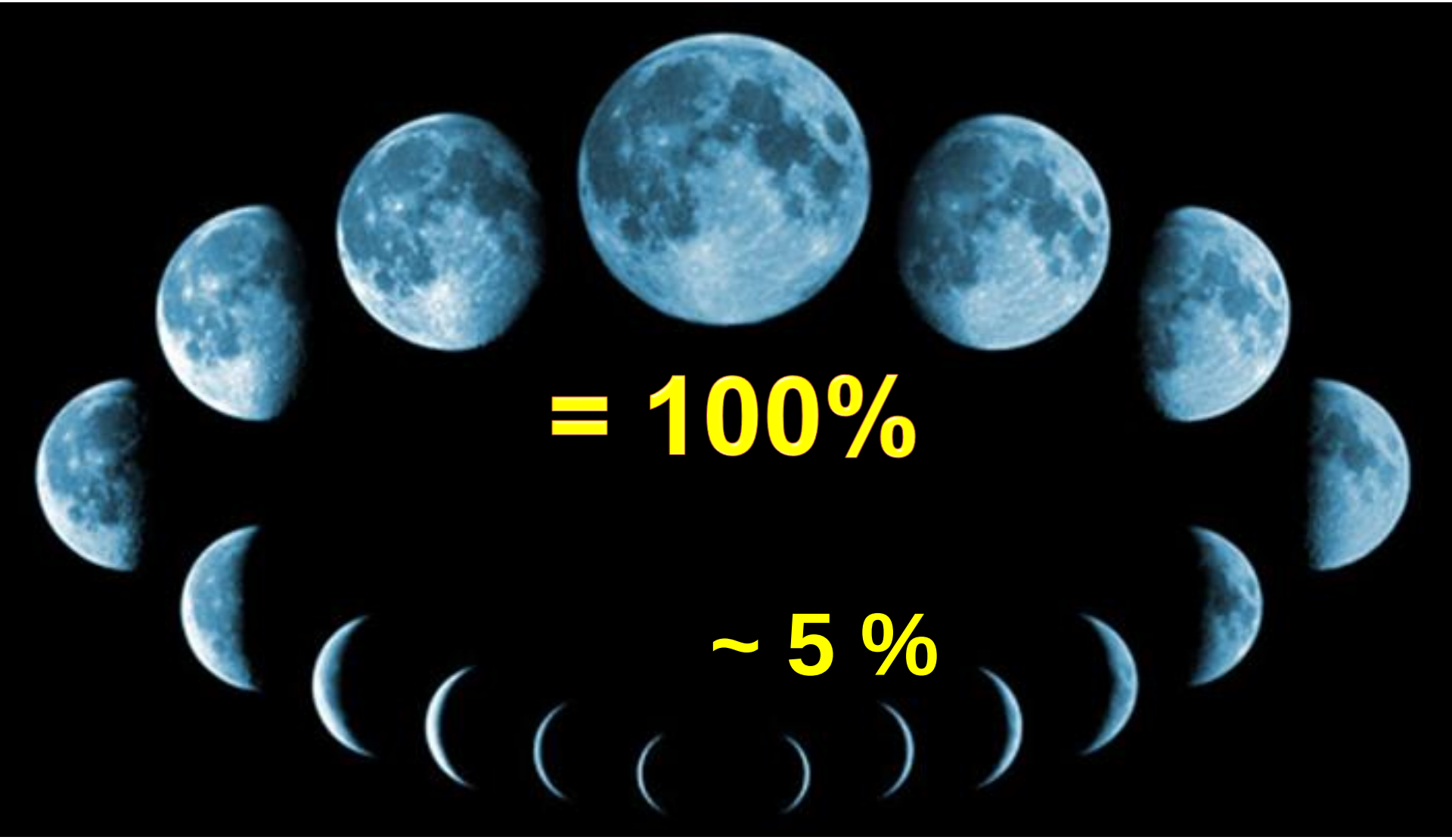
Lunar Calender



= 100%

~ 5 %

Lunar Calendar

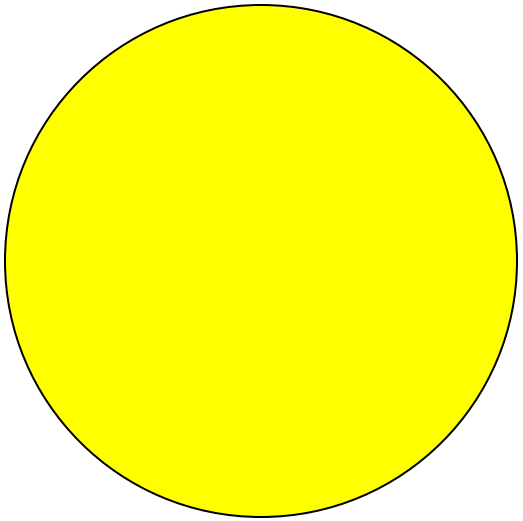


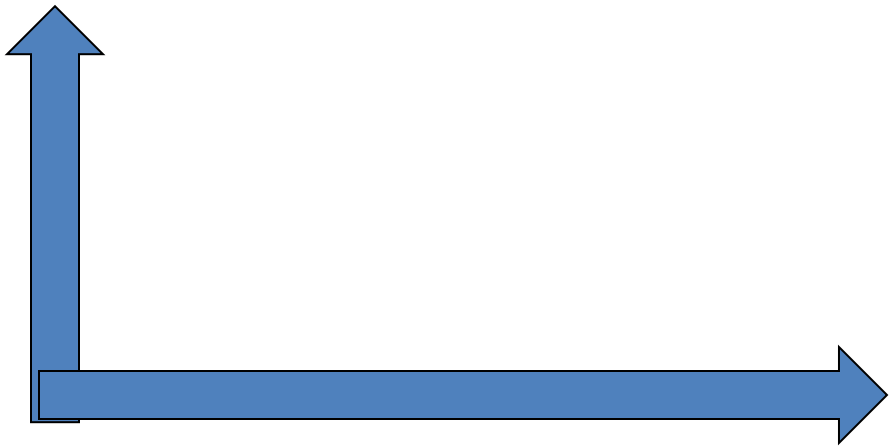
أَقِمِ الصَّلاَةَ لِدُلُوكِ الشَّمْسِ
إِلَى غَسَقِ اللَّيْلِ وَقُرْآنَ
الْفَجْرِ إِنَّ قُرْآنَ الْفَجْرِ كَانَ
مَشْهُودًا

Salat Zuhr

East

West

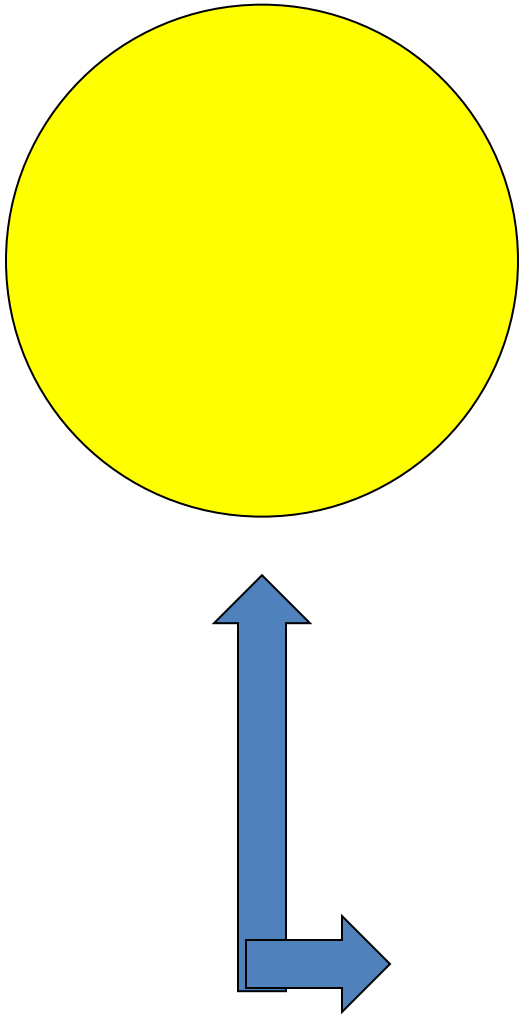




Salat Zuhr

East

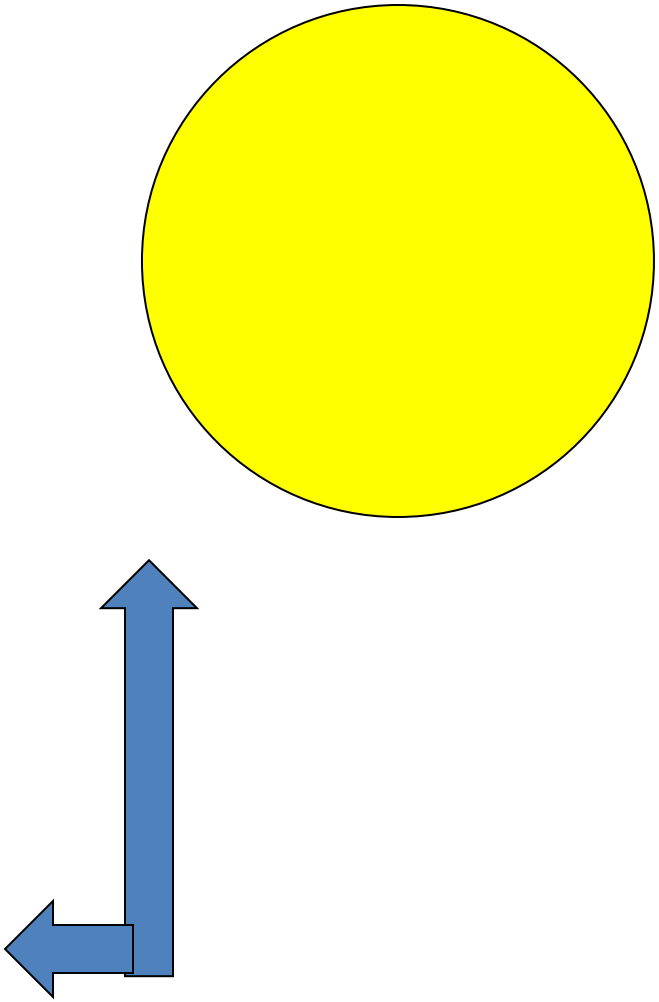
West



Salat Zuhr

East

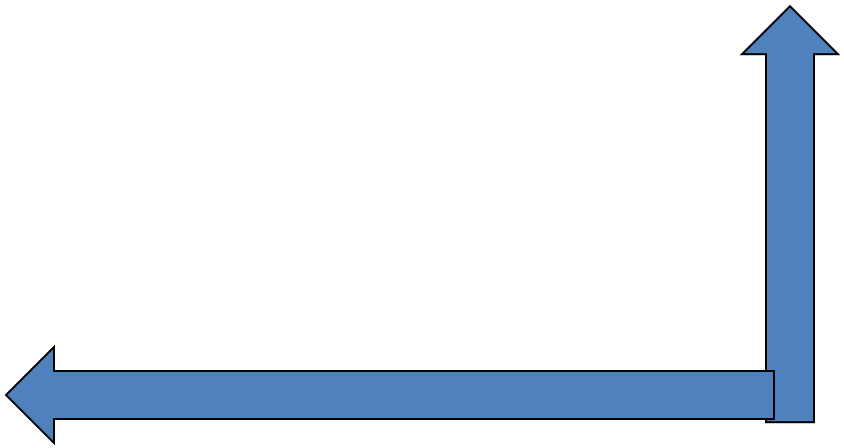
West



Salat Zuhr

East

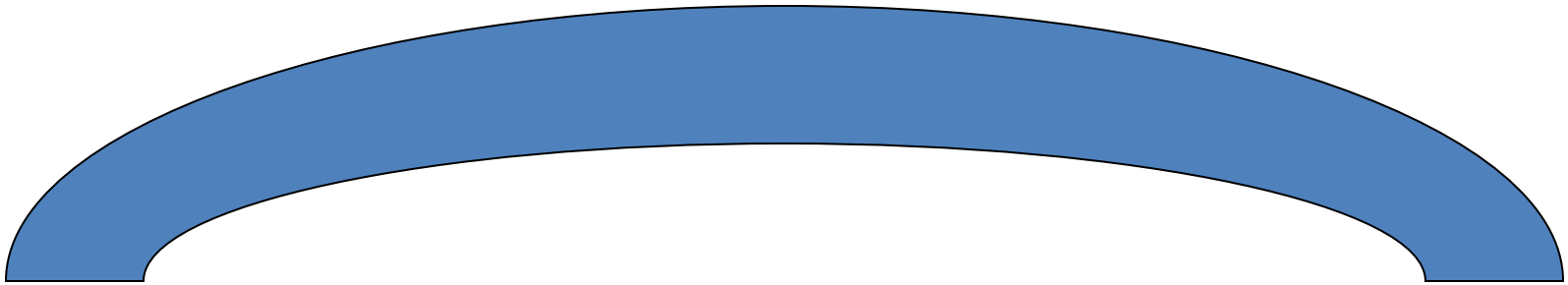
West

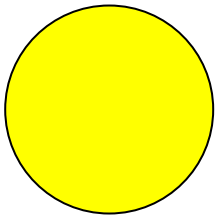


Salat Fajr

East

West





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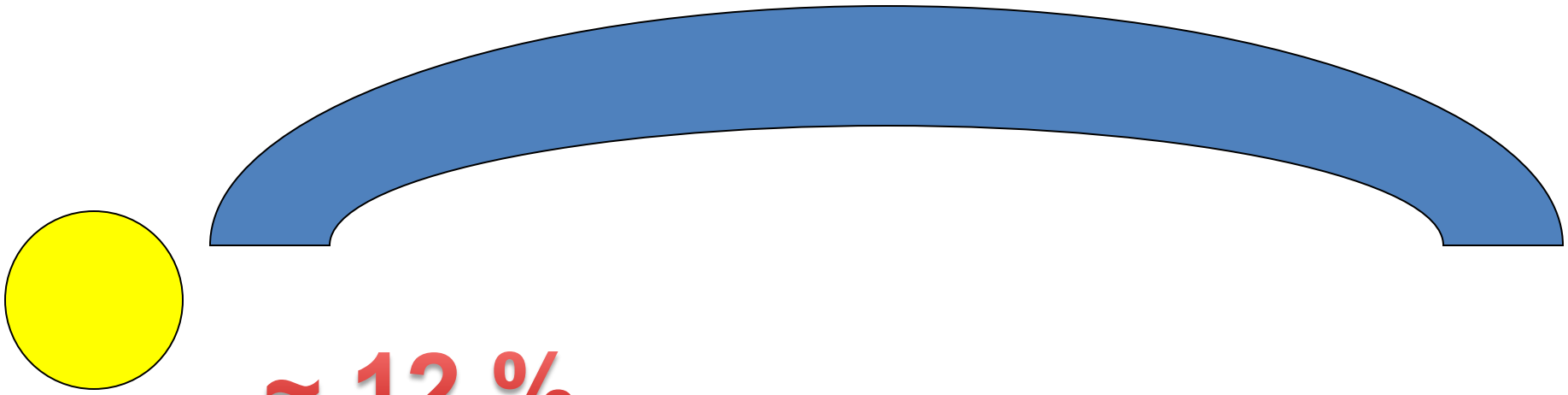
Salat Fajr

East

West



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Conditions for Tazkieh

- **By Default All Meats are Haram unless they have been became Halal through a process called Tazkieh**
  - ***Halal Meat Animal,***
  - ***after performing Tazkieh,***
- ***Body parts*** ***(except some forbidden parts up to 15 parts)***
- ***will be Halal for eating***

Halal Test Fundation

- **By Default**

- **All Plant are Halal**
  - **=> 100% Olive Oil => Halal**
- **All Meats are Haram**
  - **=> only establish the Tazkieh => Halal**

  - ***Halal Meat Animal,***
  - ***after performing Tazkieh,***

Validation of Halal Analytical Method Steps

1. Providing the proof of concept both
   - theoretically &
   - Practically
2. Performing in house Validation of analytical methods based on an International guideline
3. Conducting an inter-laboratory study to determine Validation of analytical methods based on an International guideline

To confirm the Halal status of a Food product we need to confirm 3 main aspects (OIC/SMIIC 1: 2019)

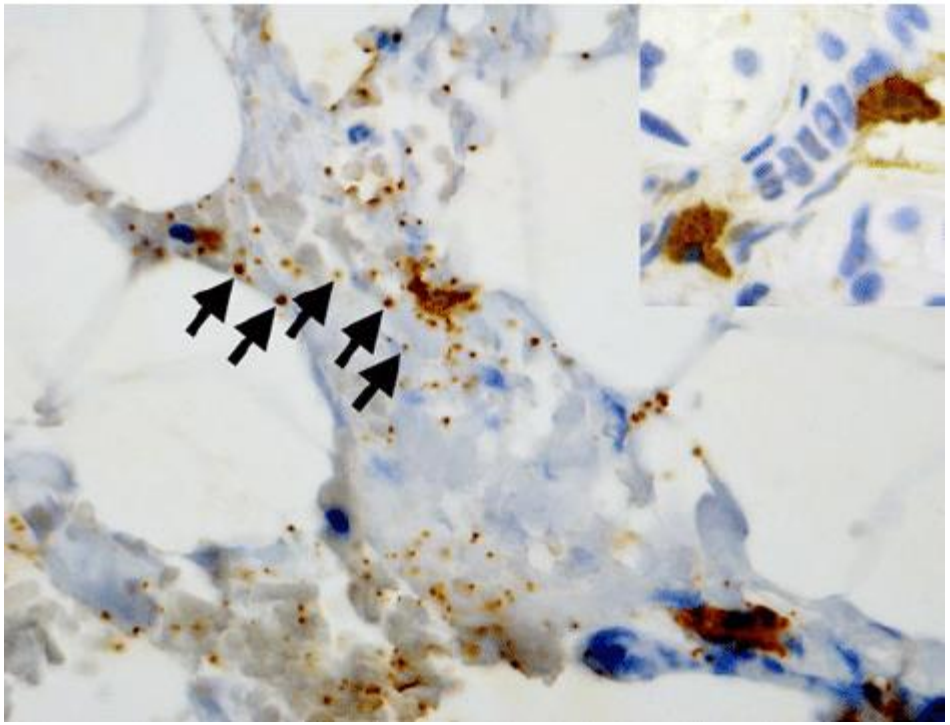
a) Objectively identified from the Halal source of food,
   - e.g. Halal Animal like Sheep

b) non-Halal and Najis components Free(risk based)
   - e.g. Haram Animal like Pig or byproducts of Haram sources such as Porcine Gelatin or Non Shariah Slaughtered Bovine Gelatin, or Non Shariah Slaughtered Halal animal byproducts such as E471, E472 Mono- and diglycerides of fatty acids o at least to distinguished between Animal based product and plant based
   - Being free from any Najis components (e.g. blood, urine, faeces etc)

c) Fulfillment of Islamic slaughtering requirements (wherever is possible)
   - Positively passed all steps of **Tazkieh** process

(Most difficult one less exploed)

To confirm the Halal status of a Food product we need to confirm 3 main aspects (OIC/SMIIC 1: 2019)



Mast cell degranulation within surgical wound margins identified by punctiform signals (arrows) adjacent to positive mast cells (immunohistochemistry, ×400).

Insert Absence of significant degranulation in the internal control (original magnification, ×400)

Top 10 Adulterated Products at EU

1- Olive Oil	2- Fish	3- Organics	4- Milk	5- Grains
				
6- Honey & Maple Syrup	**7- Coffee & Tea**	**8- Spices**	**9- Wine**	**10- Fruit Juices**
				

Root of the Problem

Non Specific Methods

Assay without Identification

Identification through components Measurement

Root of the Problem

Pharmaceutical Test Methods	Food Test Methods
Identification Assay (Direct Proof)	Authenticity Test
Quality Assays	Quality Assays (Proof by Contradiction)
Safety Assays	Safety Assays

Identification and Assay Nelfinavir

Identification:	
^{1}H NMR Spectrum	consistent with literature description
Melting Point	observed: 192-195°C (uncorrected); Literature value 192°C
Assays:	
Residual Organic Solvents	EtOH (0.4 wt%) and Et_2O (1.0 wt%) observed by ^{1}H NMR
Total Water and Other Inorganic Substances	0.3 wt% inferred from total carbon analysis (67.69% carbon calculated for $C_{32}H_{45}N_3O_4S$; 67.39% found)
Nelfinavir-related Substances	0.0 area % by HPLC; ^{1}H NMR consistent with HPLC
Optical Rotation (c = 1, MeOH)	observed: $[\alpha]_D$ = -125°, literature $[\alpha]_D$ = -124°

Total organic nitrogen - Kjeldahl method

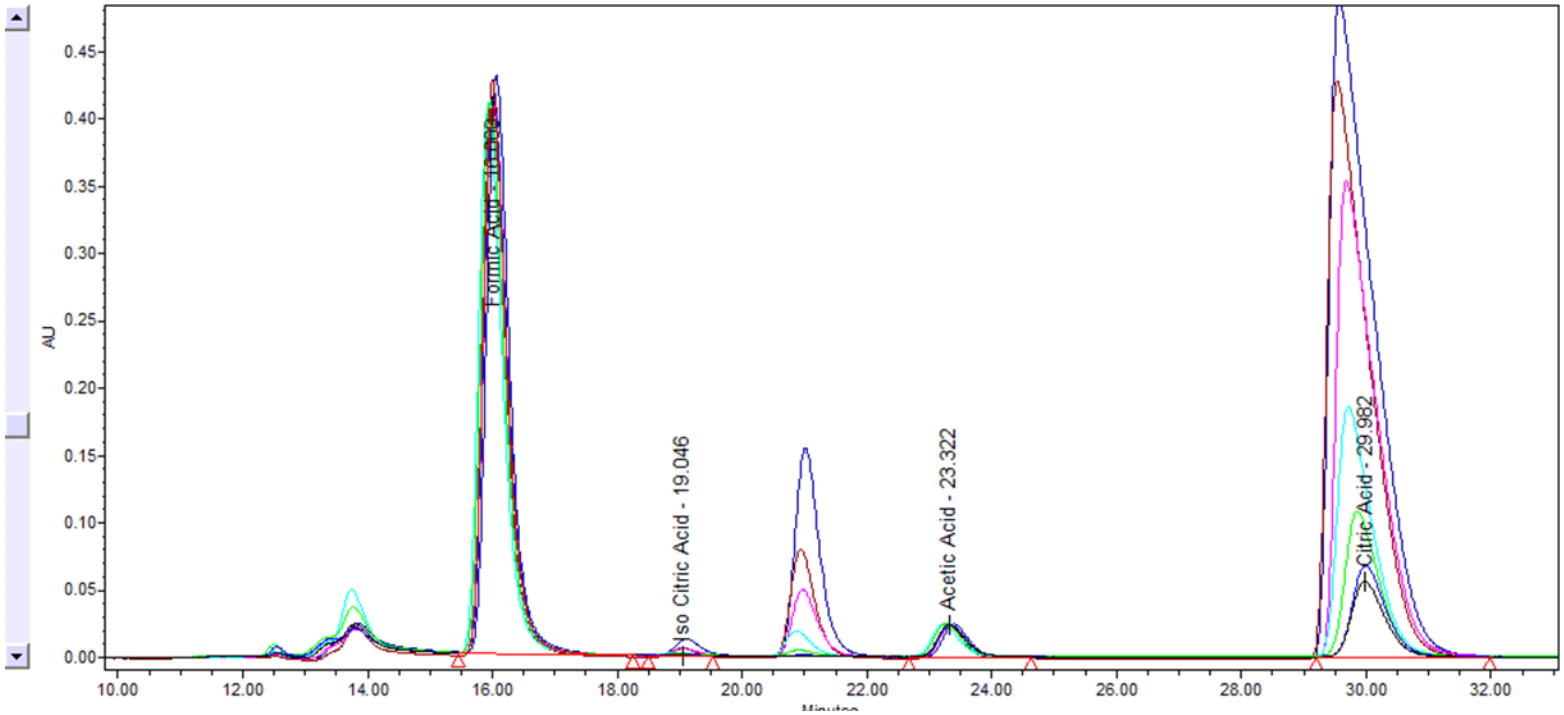
- Crude protein content
- Johan Kjeldahl (1883) developed the basic process
- Principle: total organic N released from sample and absorbed by acid
  - Digestion: sulfuric acid + catalyst
  - Neutralization and distillation; Sodium hydroxide
  - Titration; Hydrochloric acid

Melamine contamination in Milk

Persian lime - Citrus × latifolia, seedless lime, Bearss lime, and Tahiti lime,

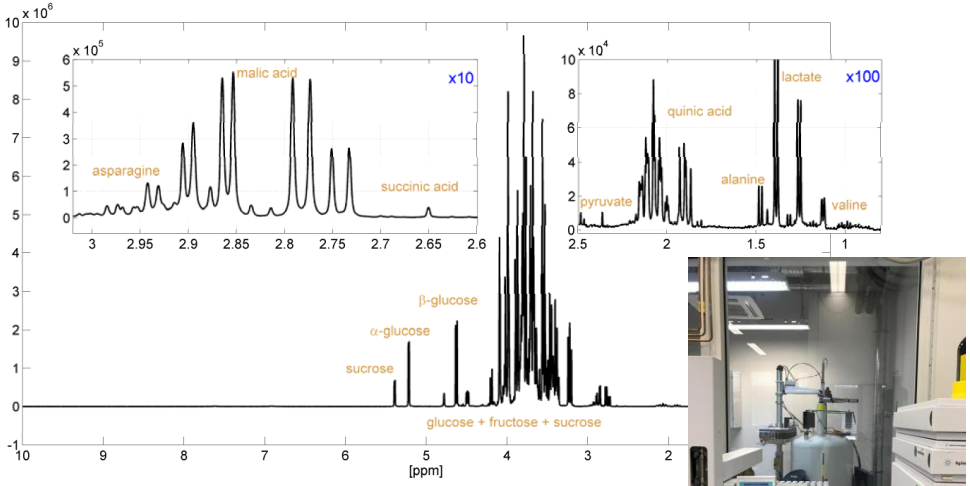
is a citrus fruit species of hybrid origin [most likely from a cross between key lime (Citrus aurantiifolia) and lemon], known only in cultivation.

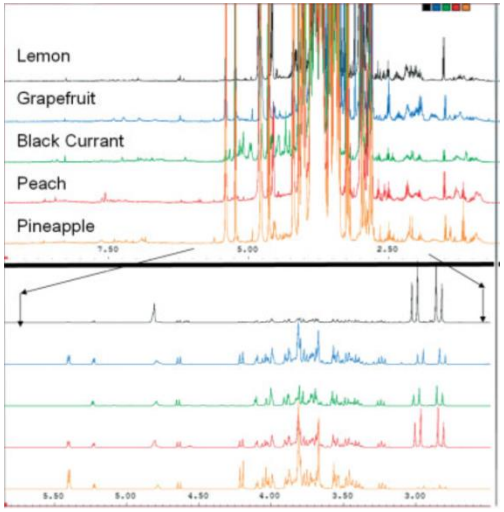
How Can Science Help Prevent Food Fraud?

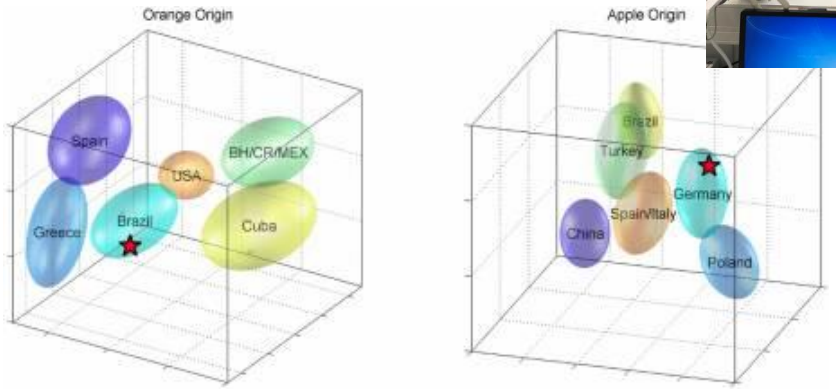


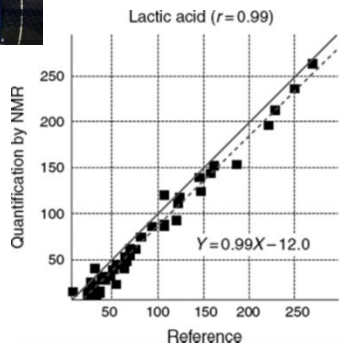
Citric acid-Isocitric acid ratio		max.		200	The concentrations of citric acid correlates with the isocitric acid concentration to a certain extent. Therefore, the ratio can be used to detect an acidification with citric acid. The ratio normally lies between 100 and 200 but values up to 240 can be observed in Argentinean lemon. Juices.

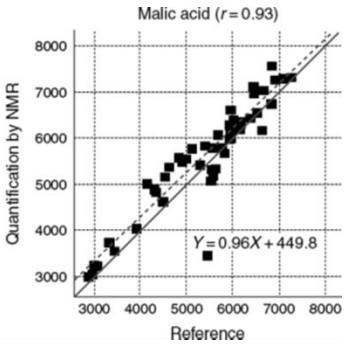
NMR Method


malic acid
x10
asparagine
succinic acid
quinic acid
lactate
x100
pyruvate
alanine
valine
β-glucose
α-glucose
sucrose
glucose + fructose + sucrose
[ppm]


Lemon
Grapefruit
Black Currant
Peach
Pineapple


Orange Origin
Spain
USA
BH/CR/MEX
Greece
Brazil
Cuba
Apple Origin
Brazil
Turkey
Germany
Spain/Italy
China
Poland

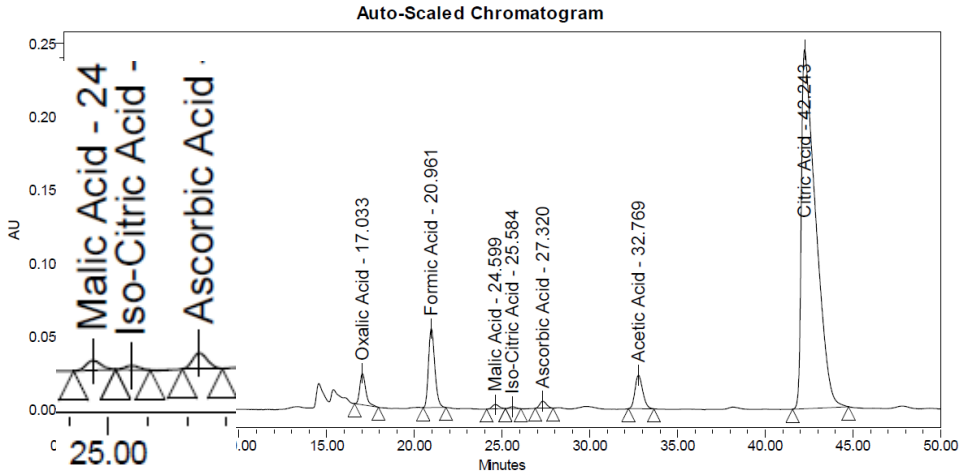

Lactic acid (r = 0.99)
Quantification by NMR
Reference
Y = 0.99X − 12.0


Malic acid (r = 0.93)
Quantification by NMR
Reference
Y = 0.96X + 449.8

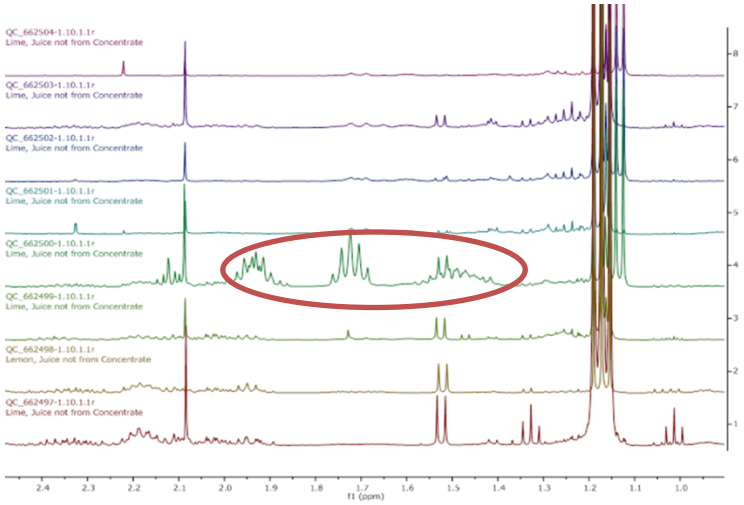
NMR Method

Compound	Conc. [mg/ml]	Flag		Compound	Conc. [mg/ml]	Flag
Ethanol	< 10	○		Sucrose	31,000	●
Methanol	N/Q	○		a-D-Glucose	10,800	○
Malic acid	1,500	●		b-D-Glucose	12,500	○
Citric acid	10,700	●		D-Glucose	23,400	●
Quinic acid	N/Q	○		D-Fructose	27.300	●
Citramalic acid	N/Q	○		Alanine	66	●
Lactic acid	< 5	●		Proline	956	●
Fumaric acid	< 5	●		5-HMF	18	○
Succinic acid	21	○		Phlorin	23	○
Benzoic acid	< 10	●		a-D-Galacturonic acid	52	○
Formic acid	< 5	●		Chlorogenic Acid	< 5	○
D-glucon acid	N/Q	○		Acetaldehyde	N/Q	○

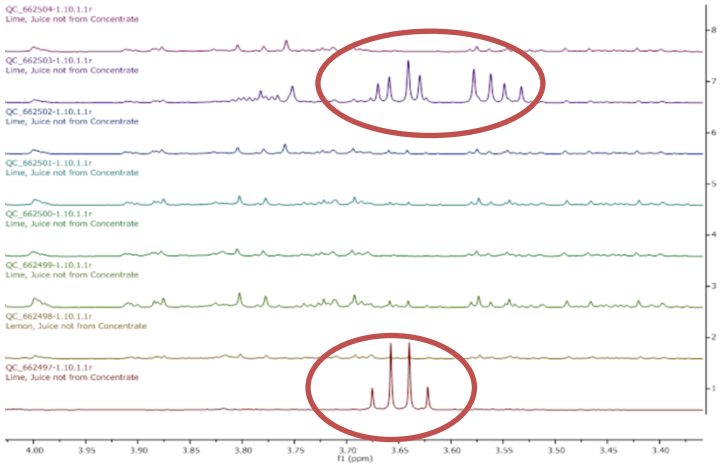
Targeted v non-targeted analytical approaches



high glycerol

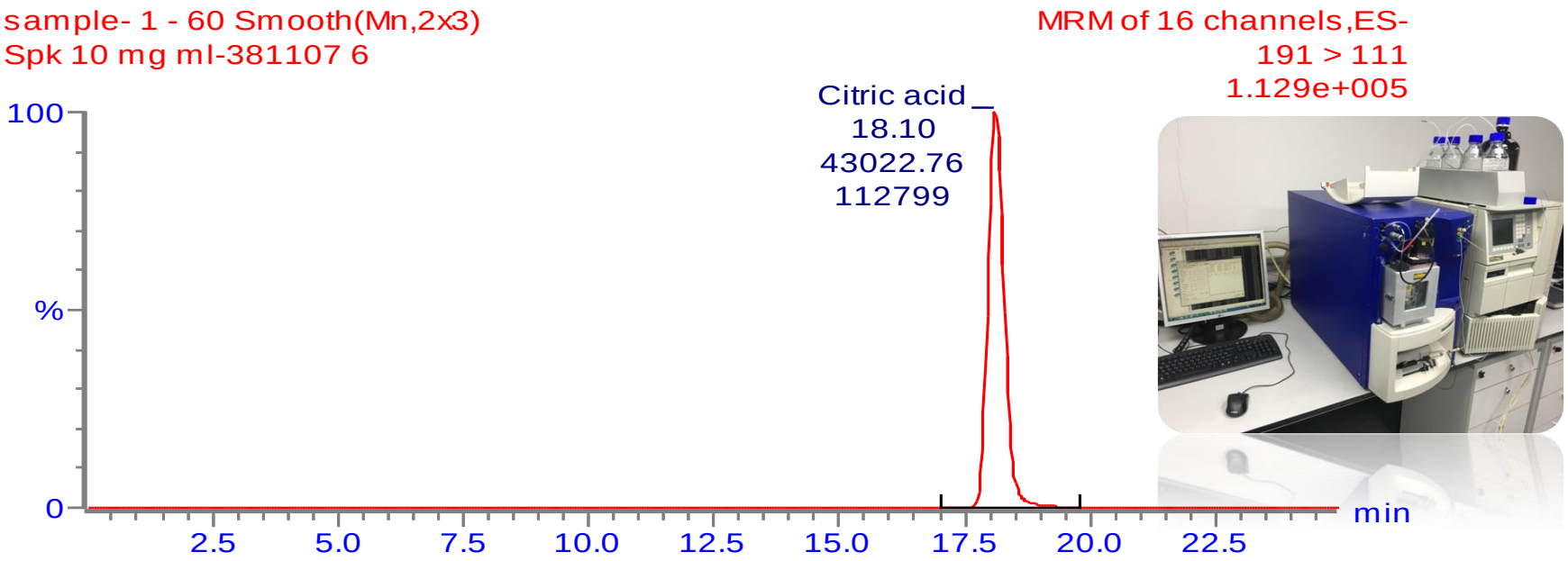


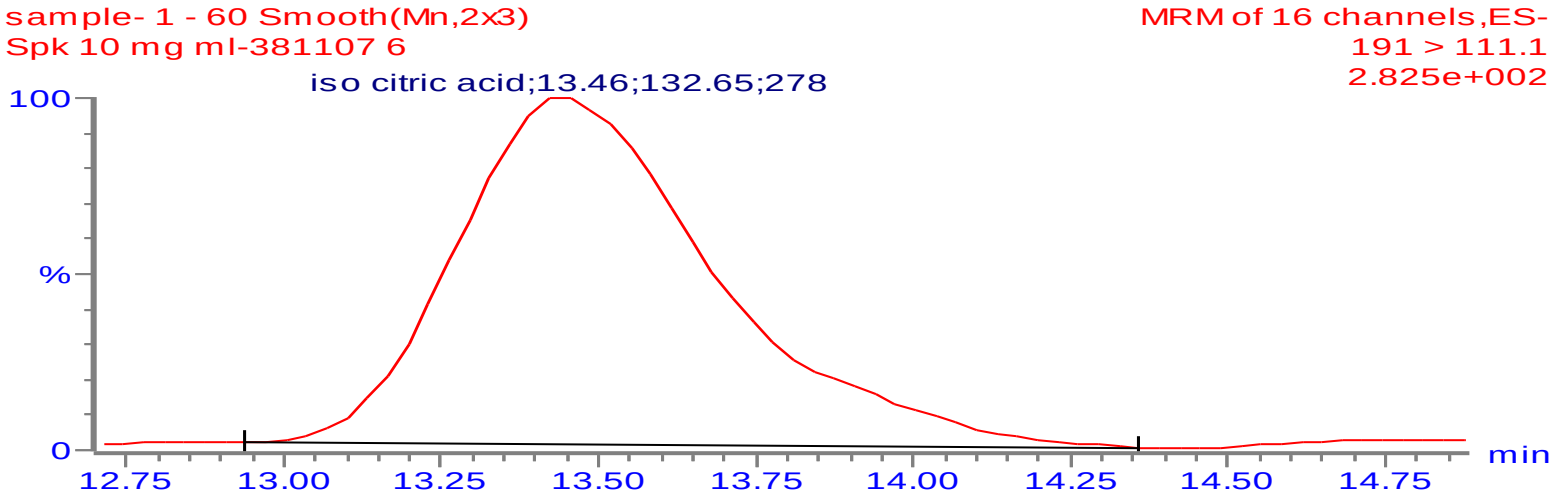
abnormal NMR signals



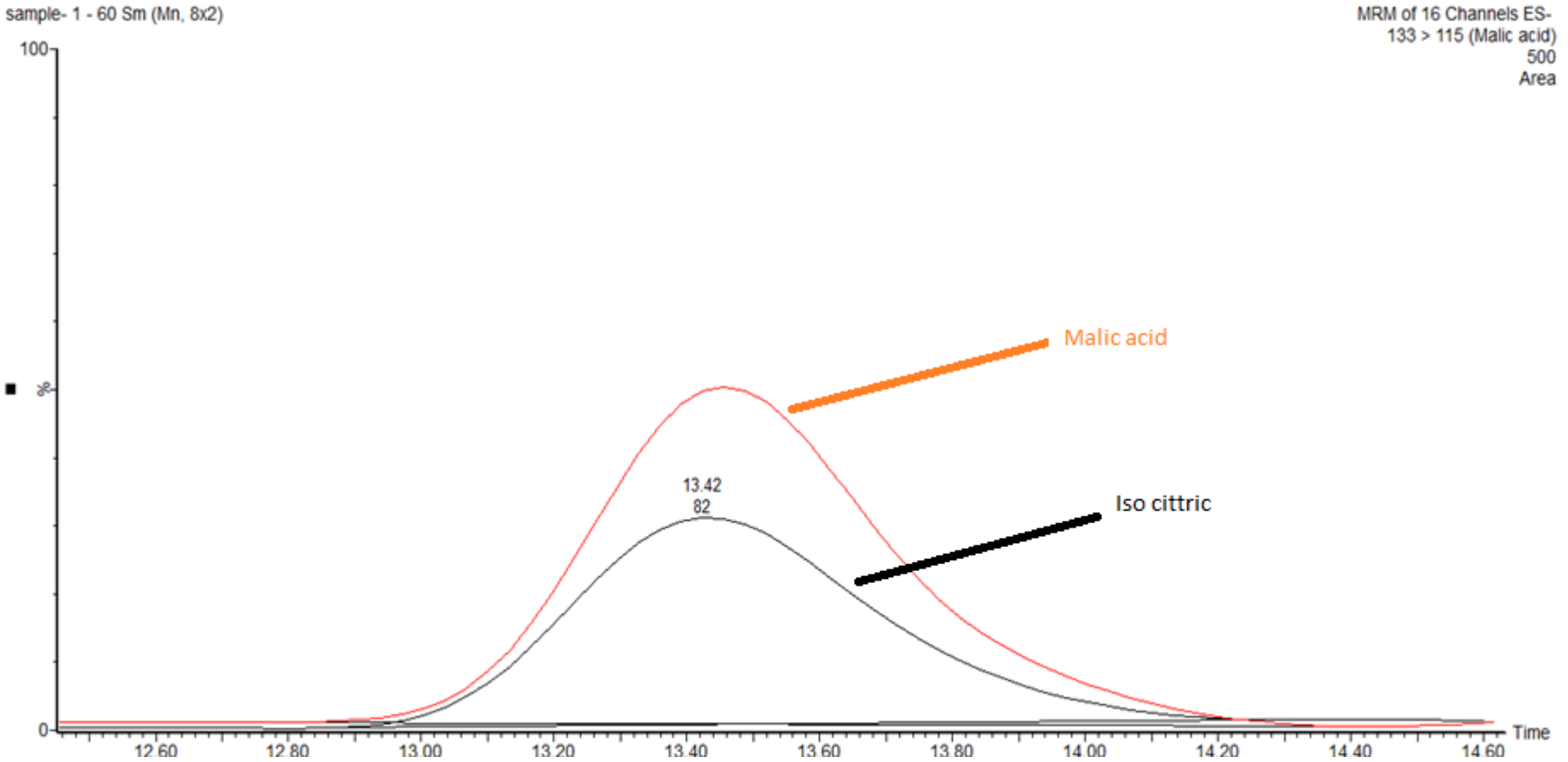
high ethanol

ISO Citric acvid v Citric acid

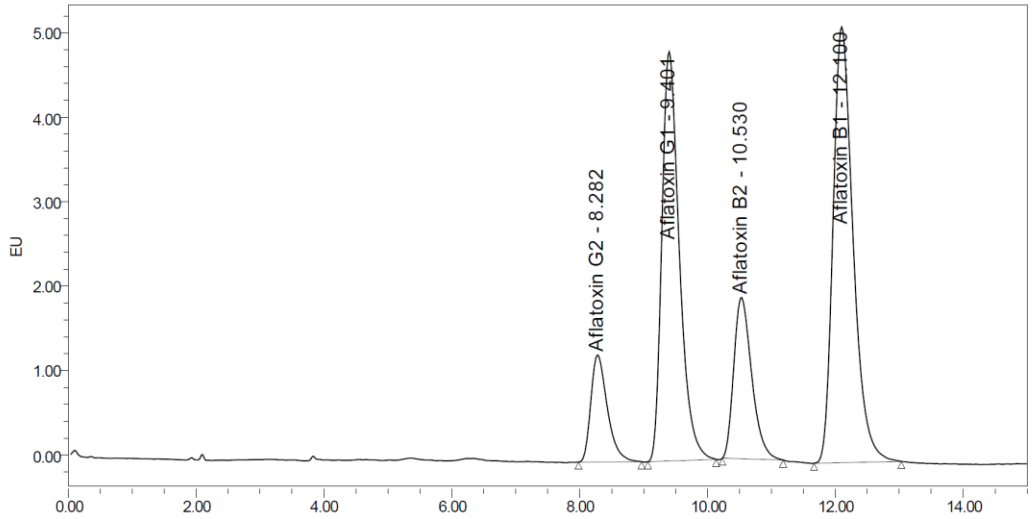


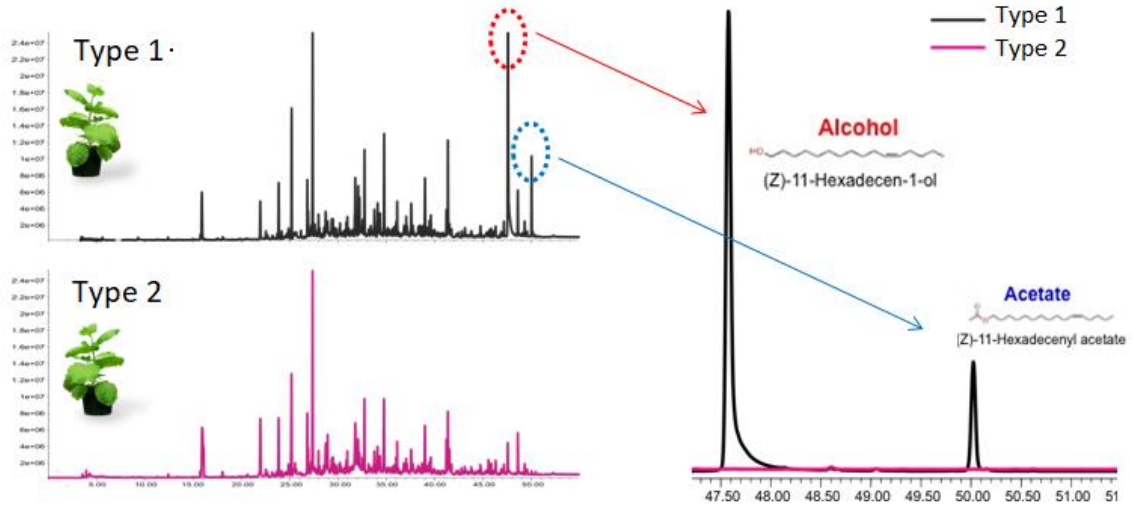


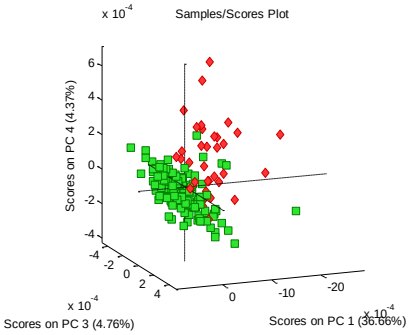
Malic & Isocitric overlay



Targeted v non-targeted analytical approaches



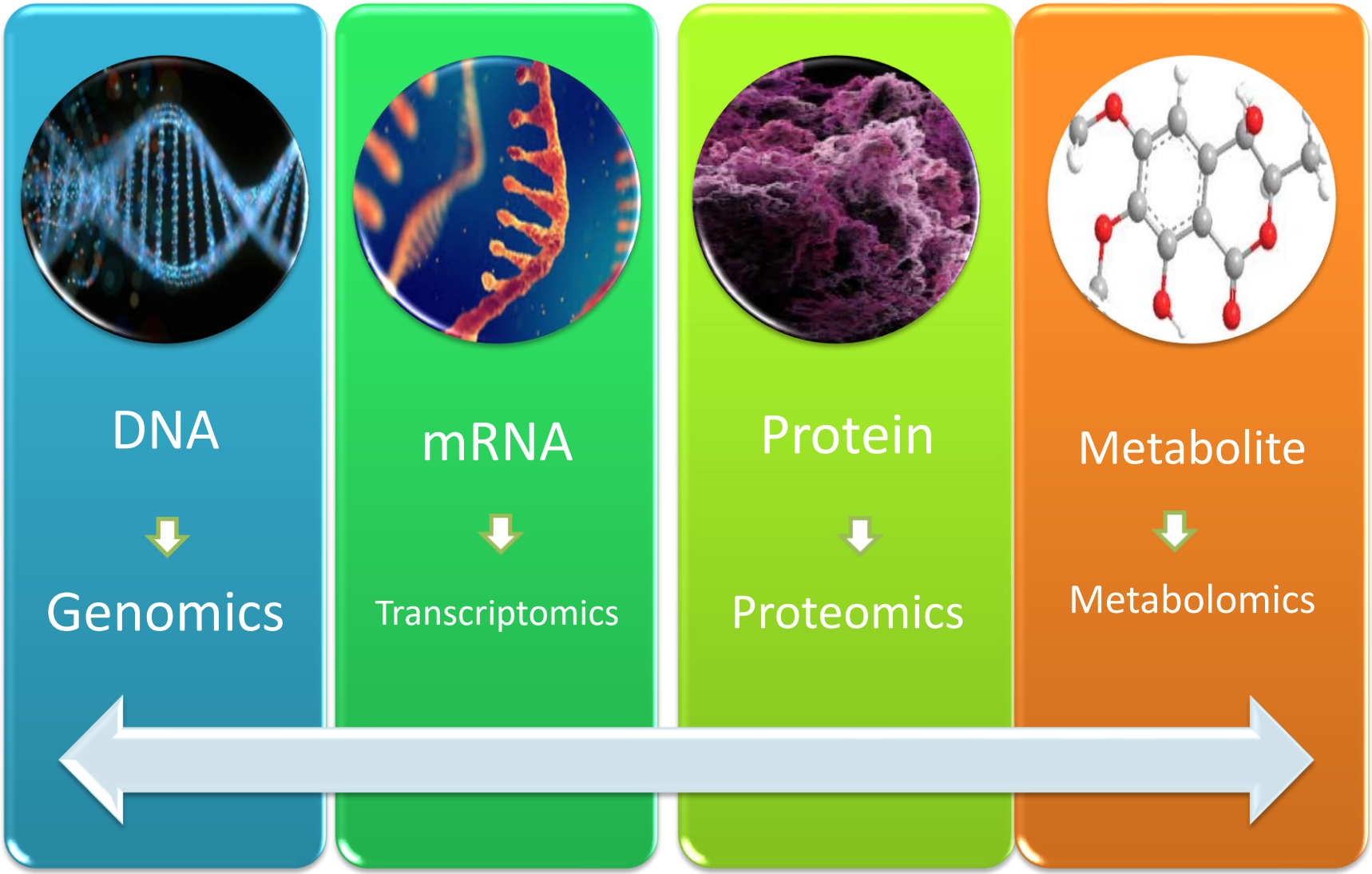




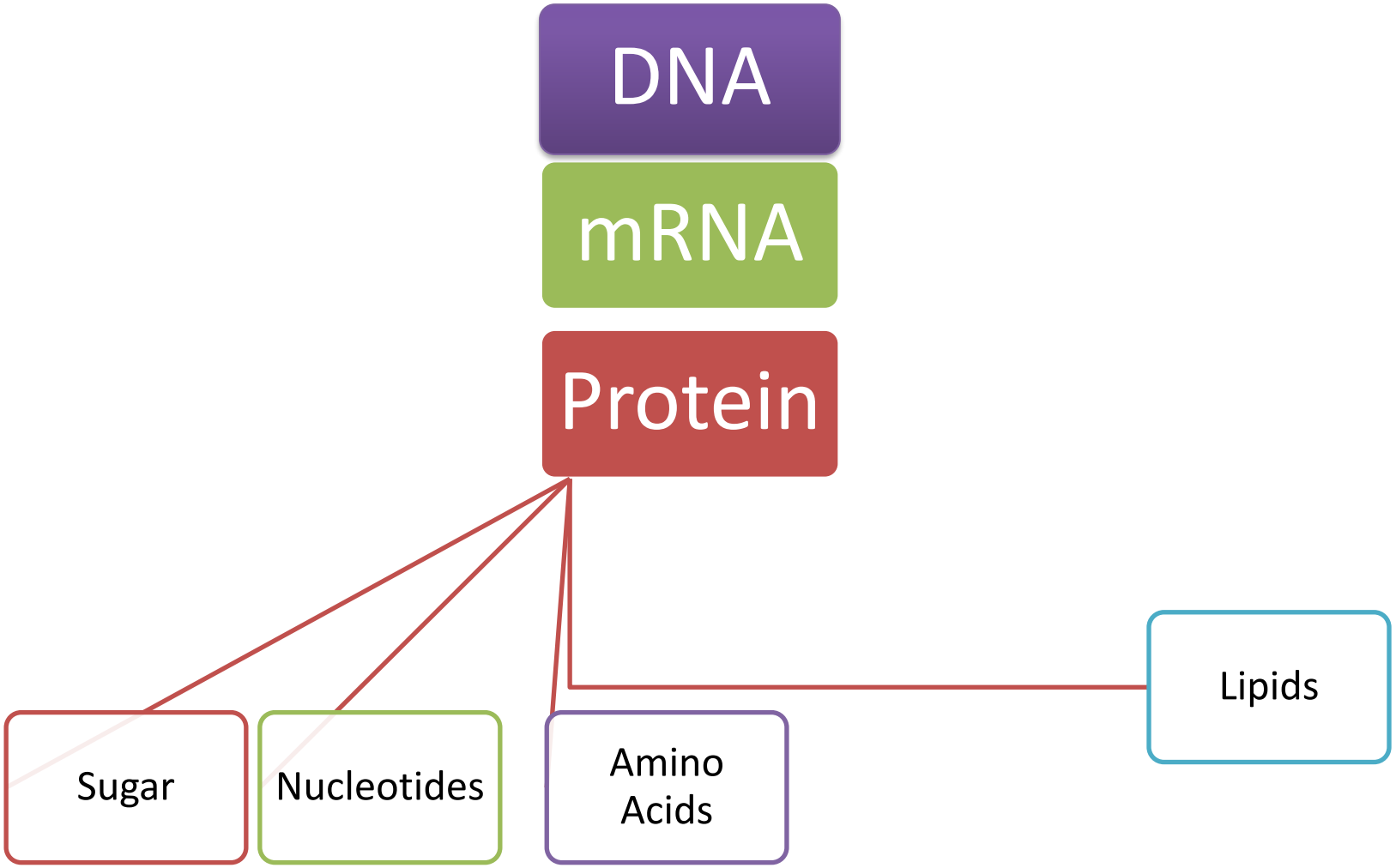
Targeted v non-targeted analytical approaches

Subject	Targeted Analyses	Non-Targeted Analyses
Approach	Bottom-up	Top-down
Analytes	Targeted compounds	Fingerprint
Sensitivity	High sensitivity	High throughput
Sample preparation	Selective	Unselective/minimum
Data analysis	Univariate/Calibration	Multivariate/Modeling (Chemometrics' software)
Control limits	Regulators	Specific Databases
Consistency	Simple Sample representativeness	Complex Experimental design

Omics [genome, transcriptome, proteome, & metabolome (lipidome)]



Metabolome



Genomics

	
Real Time PCR	**NGS**
Targeted Approach Only for the targeted DNA Maximum only detect what you are looked for TAT of 4-6h	**Non-Targeted Approach** Expected & Unexpected DNA Detect any kind of species, (unexpected species) TAT of 24-36h

Test Report : Sheep Meatball

Flag	Alerts	Ingredients	DNA Presence	Organism Group	Species Found
	Found	Sheep	Expected	Sheep	Ovis aries
	Found	Onion	Expected	Onion	Allium capa Yellow Pearl Onion (Allium ampeloprasum var. sectivum)
	Found	Potato	Expected	Potato	Solanaceae (tomato, potato, eggplant)
	Missing	Lovage	Expected	Lovage	******
	Missing	Pepper	Expected	Black Peppercorn	******
	Adulterant	Beef	Un-expected	Beef	Bos taurus
	Adulterant	Pork	Un-expected	Pork	Sus scrofa
	Allergen	Soybean	Un-expected	Soybean	Glycine max

Pathogen, Hygienic, Toxigenic Fungi

Targeted v non-targeted analytical

Targeted Analyses (TA)

- Targeted Analyses (TA) are used:
  - If the adulterating materials are known or
  - if the authentic food contains specific marker compounds that can be used to assess its purity.
- In the TA approach, the known compounds can be analytically targeted to either determine
  - if the food has a previously identified adulterant or
  - if the food had been diluted or replaced
  - by looking for compounds known to be at certain levels in the authentic product.
- Wide range of technologies (traditional wet chemistry to high-end liquid chromatographic mass spectrometry methods).

TA Examples

- Adulterated extra virgin olive oil with cheaper vegetable oils.
  - unique fatty acid profile (11 olive oil fatty acids)
  - to measure levels of expected
    - sterols,
    - equivalent carbon number 42
    - stigmastadiene
- verifying a meat or fish species
  - using PCR and DNA sequencing.
- Assessment of Fish freshness using simple chromatography to measure:
  - volatile nitrogen or
  - biogenic amines

Non-Targeted Analyses

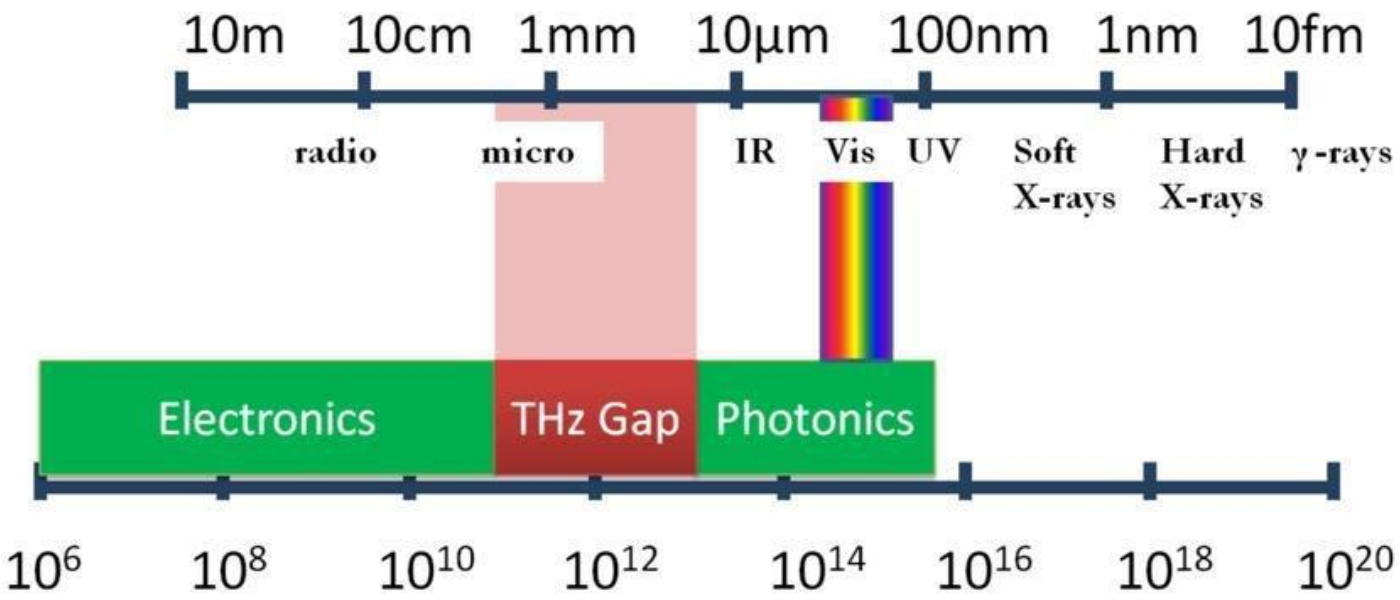
- Fingerprints
  - Human fingerprints to identify potential lawbreakers
  - DNA fingerprinting
  - Chemical fingerprints of Food ingredients
- Non-Targeted Analyses (NTA) consisted of:
  - measuring technologies and
  - data processing software (big data)

Non-Targeted Analyses

- NTA fingerprint (molecular profiles):
  - Large molecules, => MALDI-TOF
  - Small molecules => NMR, spectroscopic (IR, NIR, Raman)
- NTA => Profiling by Chemometrics Technique
- LC/MS (high resolution)
- LC/MS/MS
- Low amount adulterant => noise > failed NTA approach

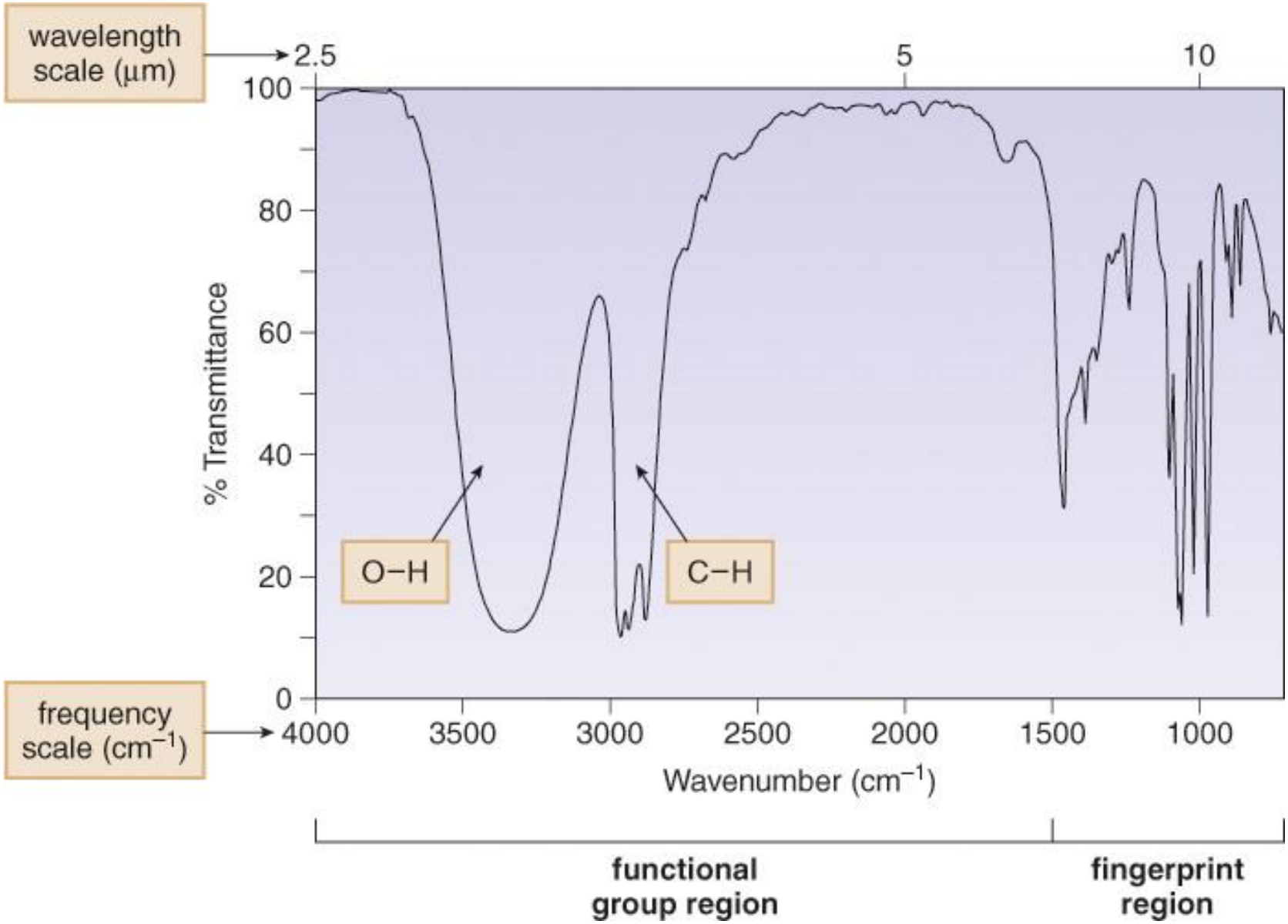
Gelatin Speciation FTIR Method November 2019

Electromagnetic Spectrum



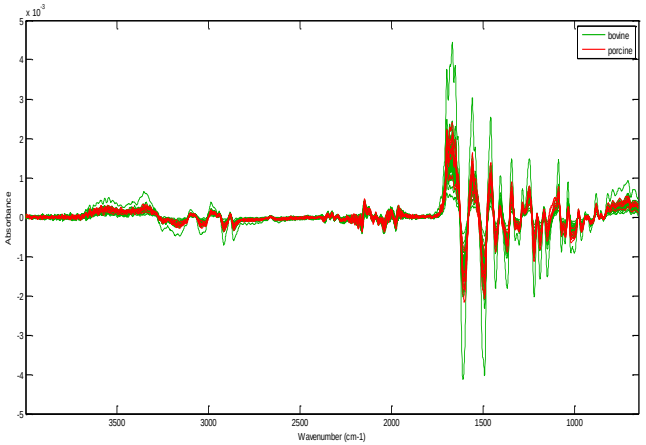
The IR spectrum is divided into two regions: the functional group region (at > 1500 cm-1), and the fingerprint region (at < 1500 cm-1).

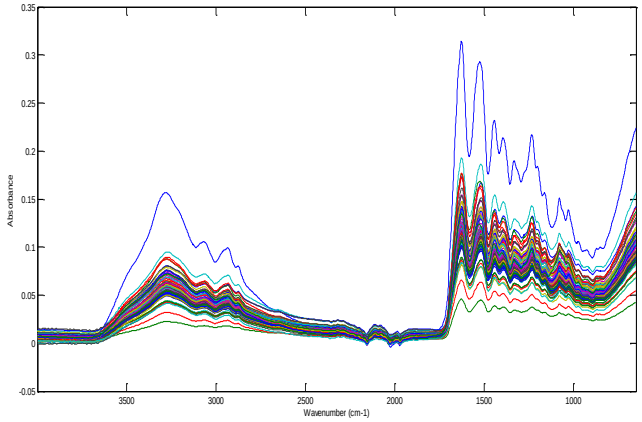
Characteristics of an IR Spectrum—1-Propanol



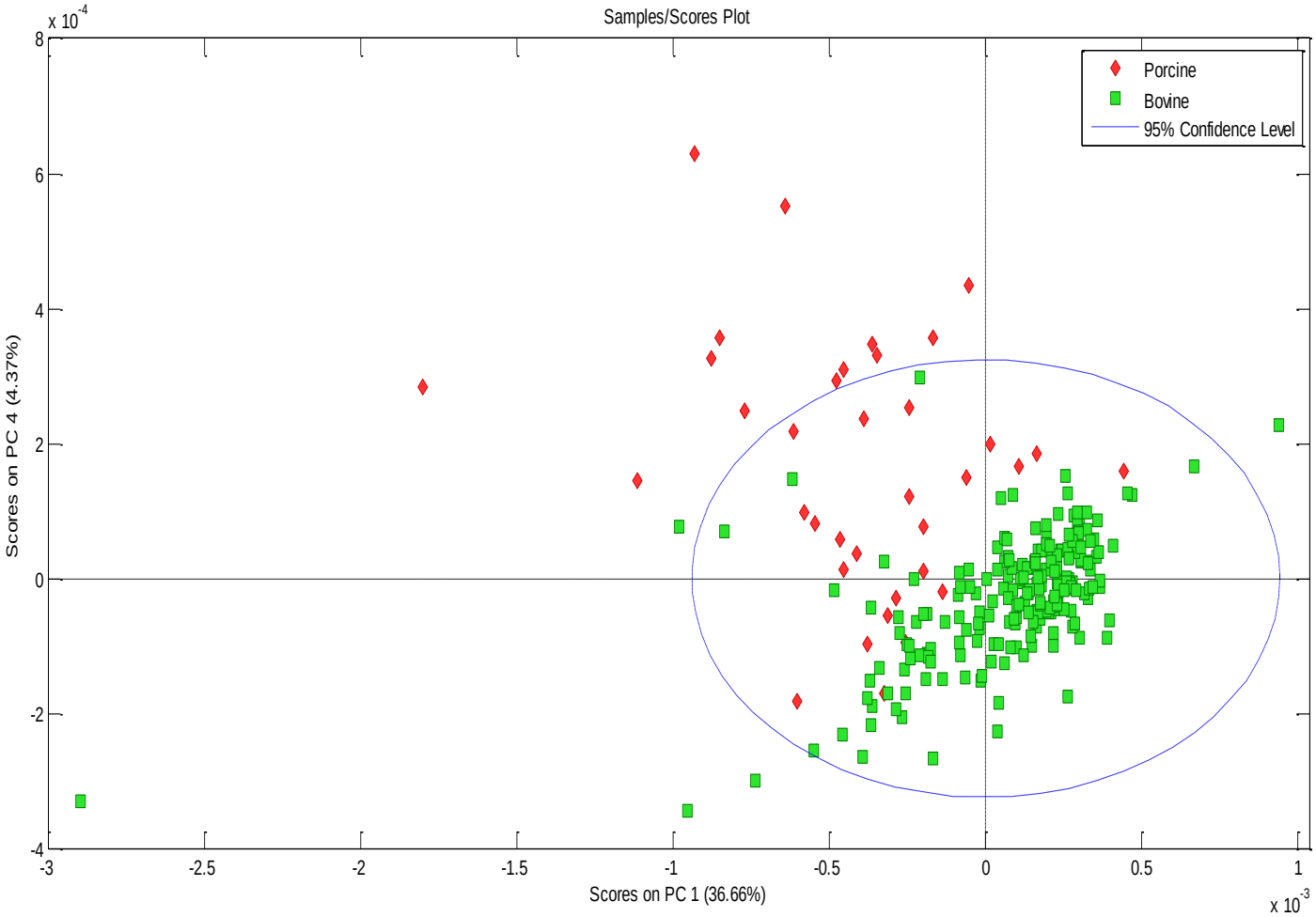
Gelatin Speciation

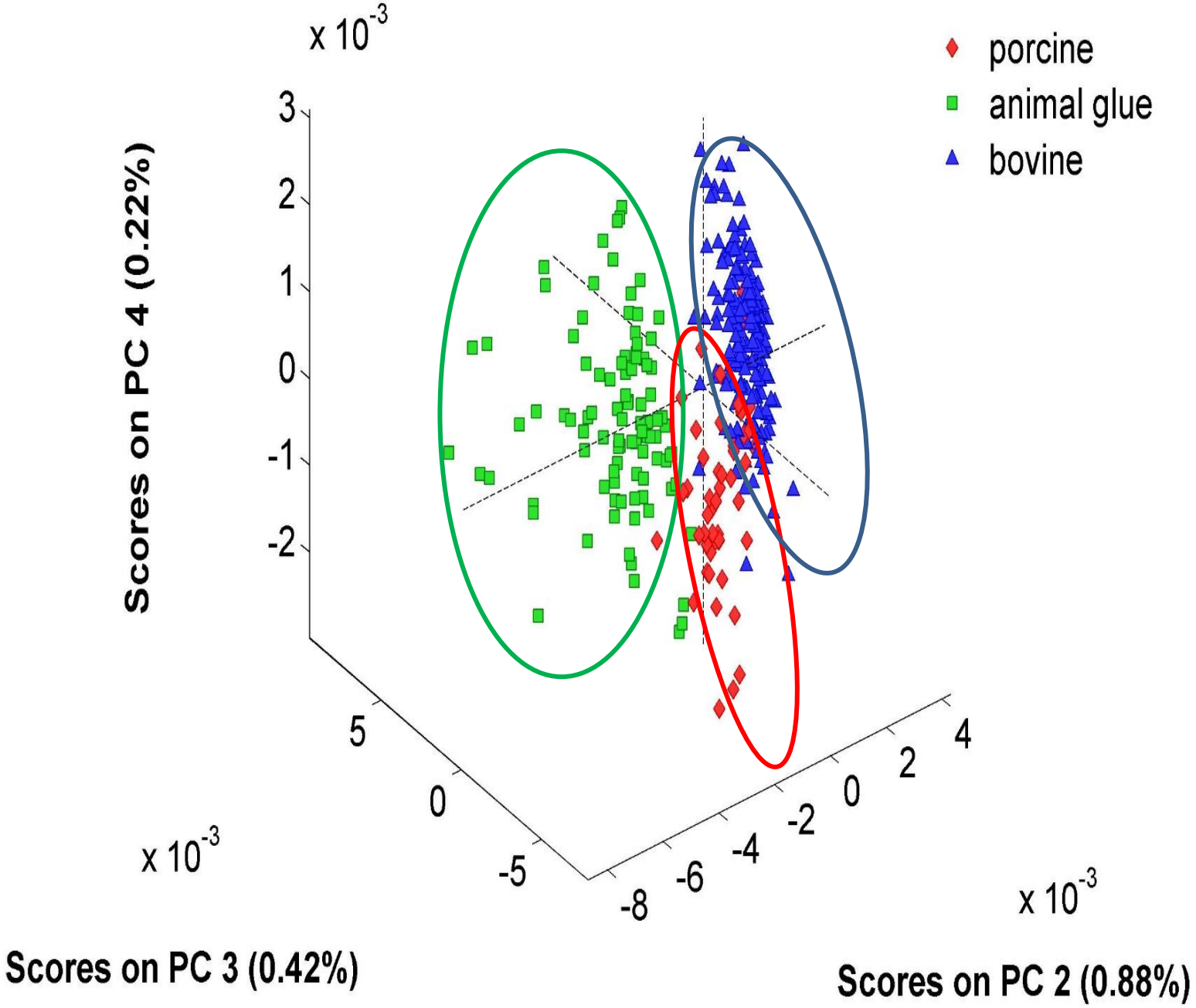
Diamond Attenuated Total Reflectance (ATR)





PCA score plot (2D)




porcine
animal glue
bovine
x 10^-3
Scores on PC 4 (0.22%)
3
2
1
0
-1
-2
x 10^-3
5
0
-5
Scores on PC 3 (0.42%)
-8
-6
-4
-2
0
2
4
x 10^-3
Scores on PC 2 (0.88%)

SIMCA [Preprocessed With Extended Multiplicative Signal Correction (EMSC)] & Variable Selection (1730-1477 cm-1) (3 PC)

Prediction set (validation set)	True positive (sensitivity %)	True negative (specificity %)
Bovine	**91**	**100**

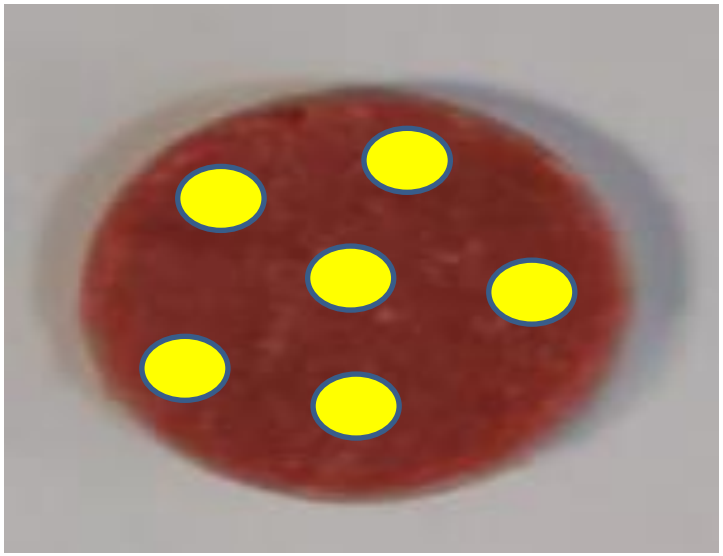
SIMCA (preprocessed with 2nd derivative (Sav-Gol)) and variable selection (1730-1477 cm-1) (4 PCs)

Prediction set (validation set)	True positive (sensitivity %)	True negative (specificity %)
Porcine	**100**	**98.9**

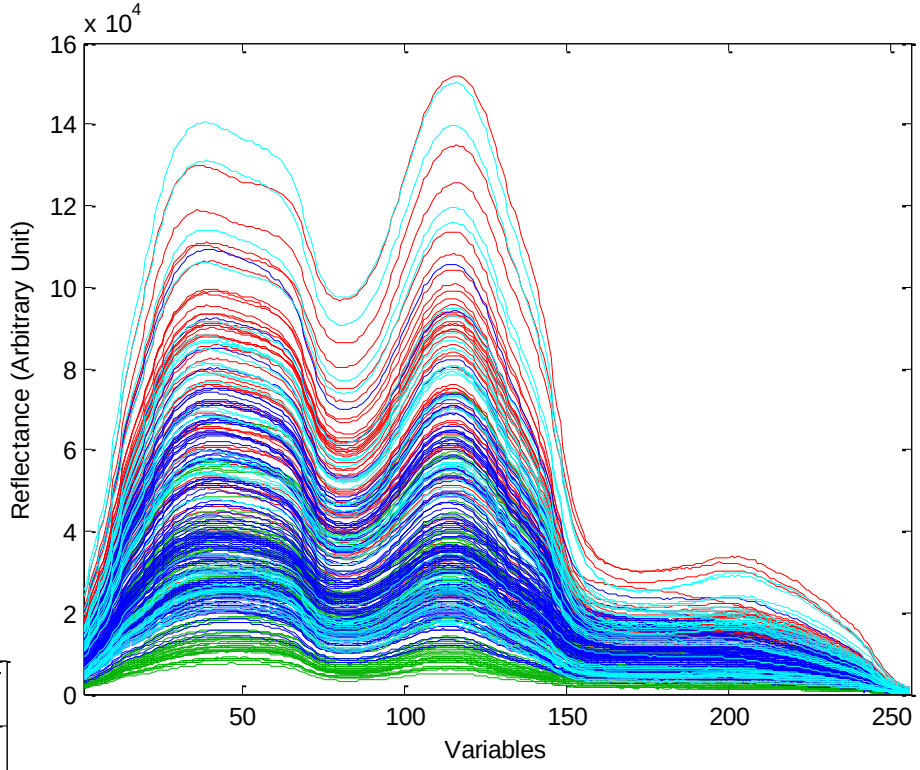
Meat Speciation

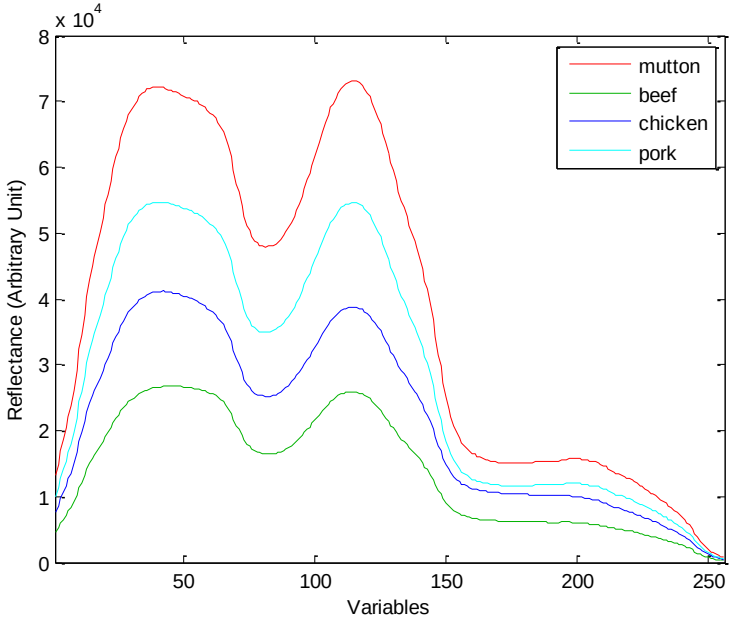
Spectral acquisitions were performed on six points of non-minced and minced samples

In this study, datasets were divided into calibration (70%) and validation (30%) sets with duplex algorithm



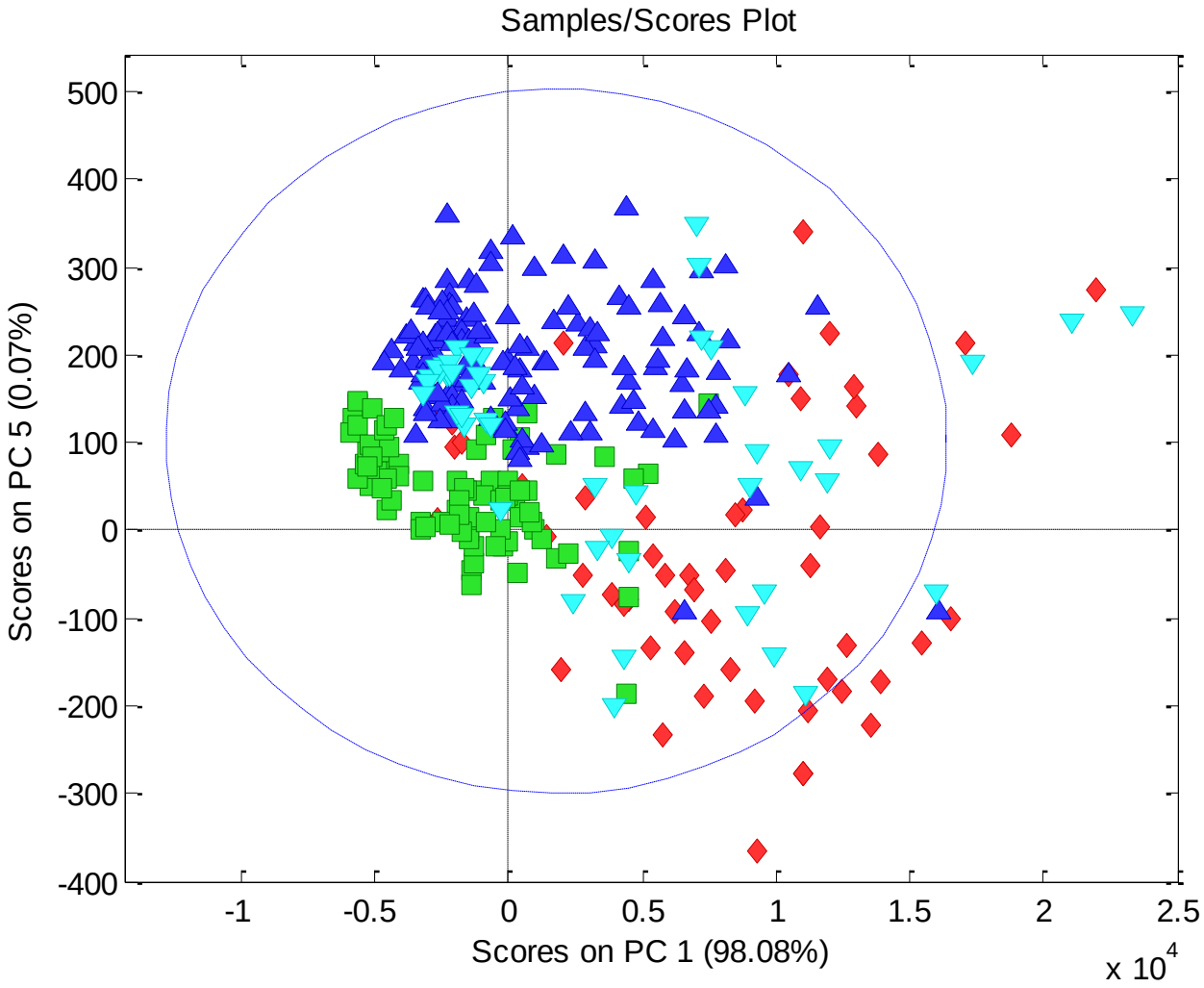
SPECTRUM OF NIR



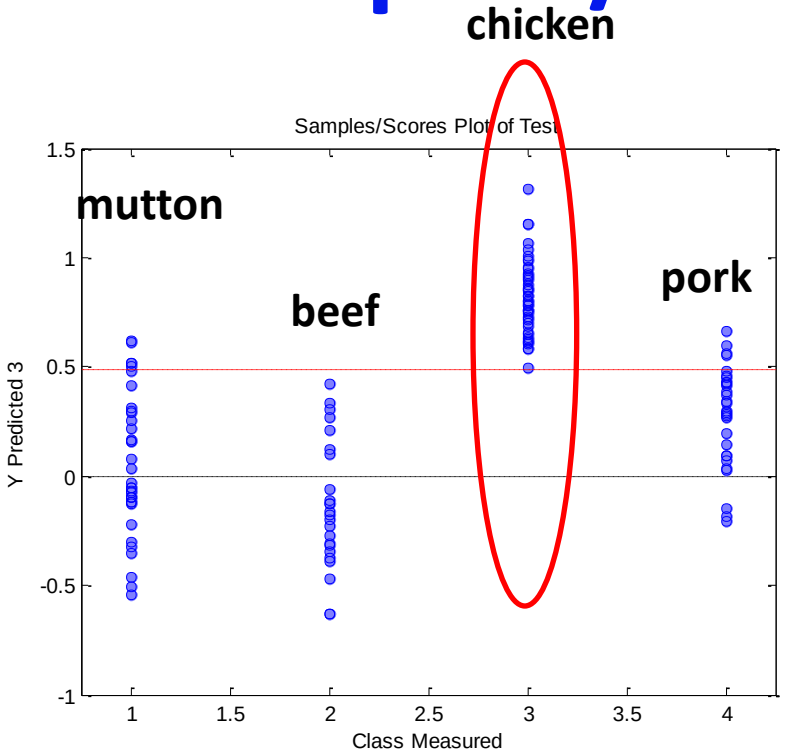


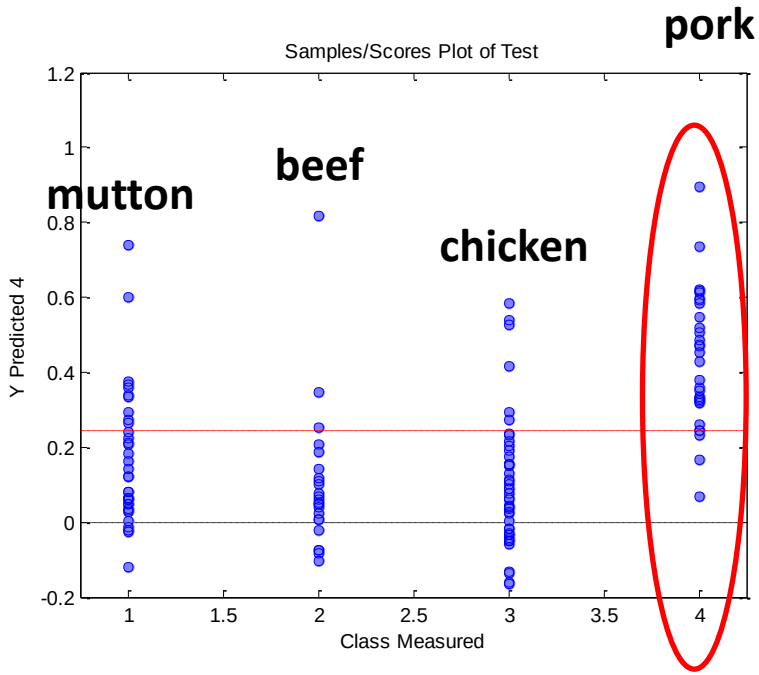
Averaged spectra

PCA Samples/Scores Plot (4 species)



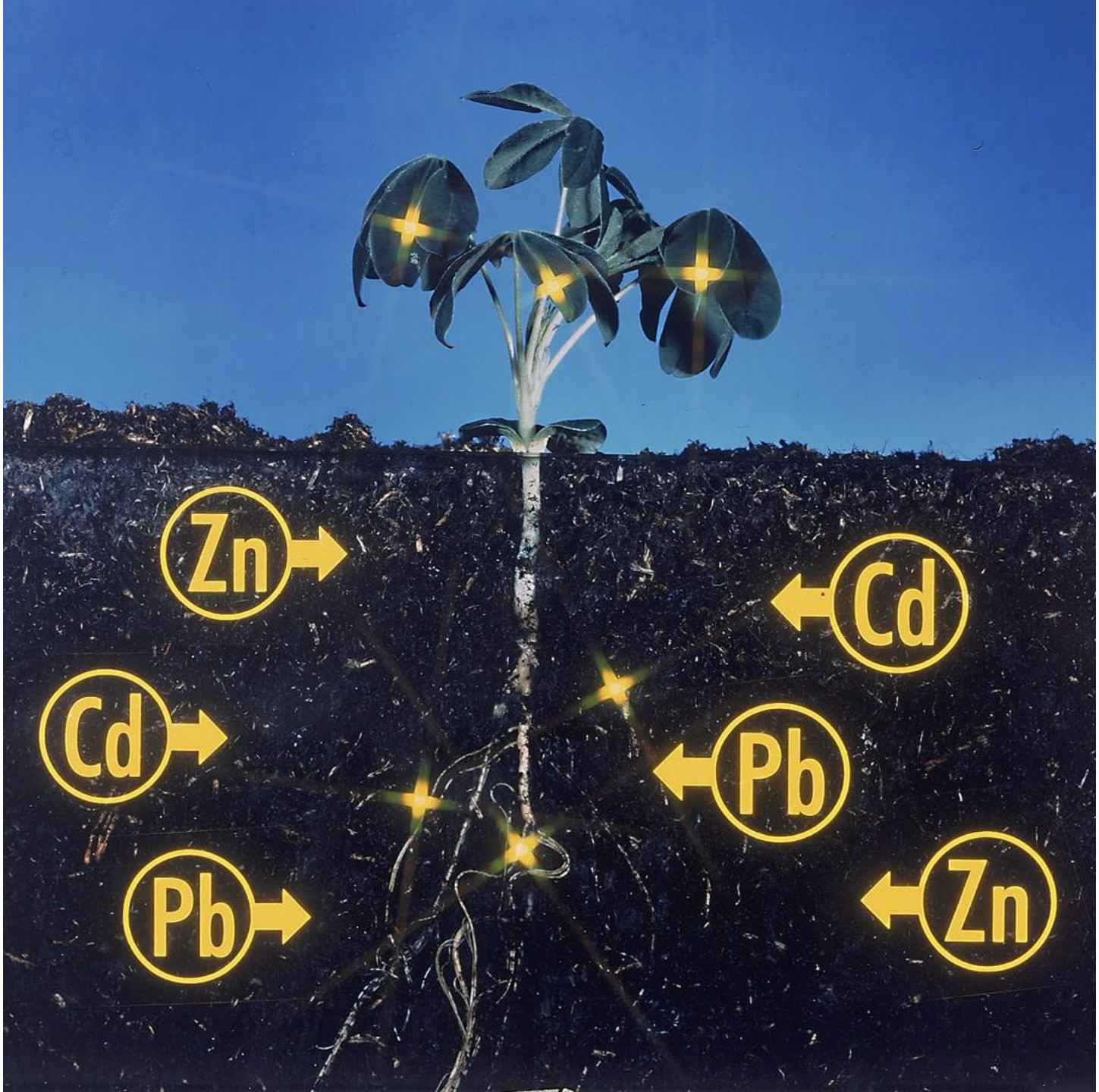
Samples /scores plot of test





Novel Analytical Techniques

- The 2 classes of techniques:
- Inorganic Analysis
  - Stable Isotope Analysis
  - Trace Element Analysis
- Metabolomics
  - small molecules that are produced through the biochemical processes as food developed or produced


Zn
Cd
Cd
Pb
Pb
Zn

بِسْمِ اللَّهِ الرَّحْمنِ الرَّحِيمِ

وَالْبَلَدُ الطَّيِّبُ يَخْرُجُ نَبَاتُهُ بِإِذْنِ رَبِّهِ

وَالَّذِي خَبُثَ لَا يَخْرُجُ إِلَّا نَكِدًا

كَذَٰلِكَ نُصَرِّفُ الْآيَاتِ لِقَوْمٍ يَشْكُرُونَ

[سورة الأعراف:58]

بِسْمِ اللّٰهِ الرَّحْمٰنِ الرَّحِيْمِ

1. And the good land - its vegetation emerges by permission of its Lord;
2. but that which is bad - nothing emerges except sparsely, with difficulty.
3. Thus do We diversify the signs for a people who are grateful.

Photo : A Amin khah

Arsenic in Rice

http://www.abdn.ac.uk/arsenic/De%20ganga%202008.html

- **De ganga, India, 2008**
- Location: latitude 22º57' and longitude 88º56'
- Growing season = Boro
- No. of cultivars = 80
- Soil arsenic = 17.4 mg/kg
- Grown under flooded conditions

	Arsenic levels (ppb)
Mean	384
SD	167
CV	433
Max	840
Min	110
No of Cultivar	80

Arsenic in Rice

http://www.abdn.ac.uk/arsenic/Nonaghata%202008.html

- **Nonaghata, India, 2008**
- Location: latitude 23º42' and longitude 88º44'
- Growing season = Boro
- No. of cultivars = 80
- Soil arsenic = 6.2 mg/kg
- Grown under flooded conditions

	Arsenic levels (ppb)
Mean	291
SD	117
CV	403
Max	730
Min	50 (x1)
No of Cultivar	80


VIIB
8
9
25
Mn
manganese
54.938
26
Fe
iron
55.845
27
Co
cobalt
58.933
28
44
45

Natural Iron isotopic composition of blood is an indicator of Speciation

Mixing proportions of stable iron isotopes in all living and nonliving matter are relatively constant in nature

4 Fe Stable Isotopes	54Fe	56Fe	57Fe	58Fe
Abundance in living matter	5.8 %	91.8 %	2.1 %	0.3 %
Abundance in nonliving matter	5.8 %	91.8 %	2.1 %	0.3 %

Natural Iron isotopic composition of blood is an indicator of Speciation

J Biol Inorg Chem (2013) 18:1–7
DOI 10.1007/s00775-012-0943-7

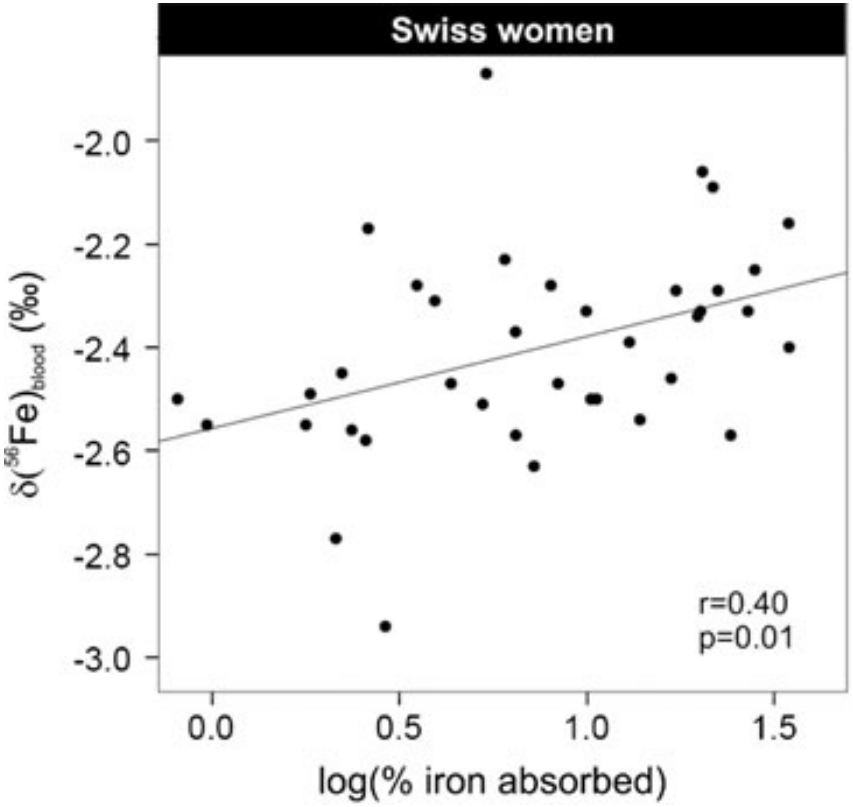
ORIGINAL PAPER

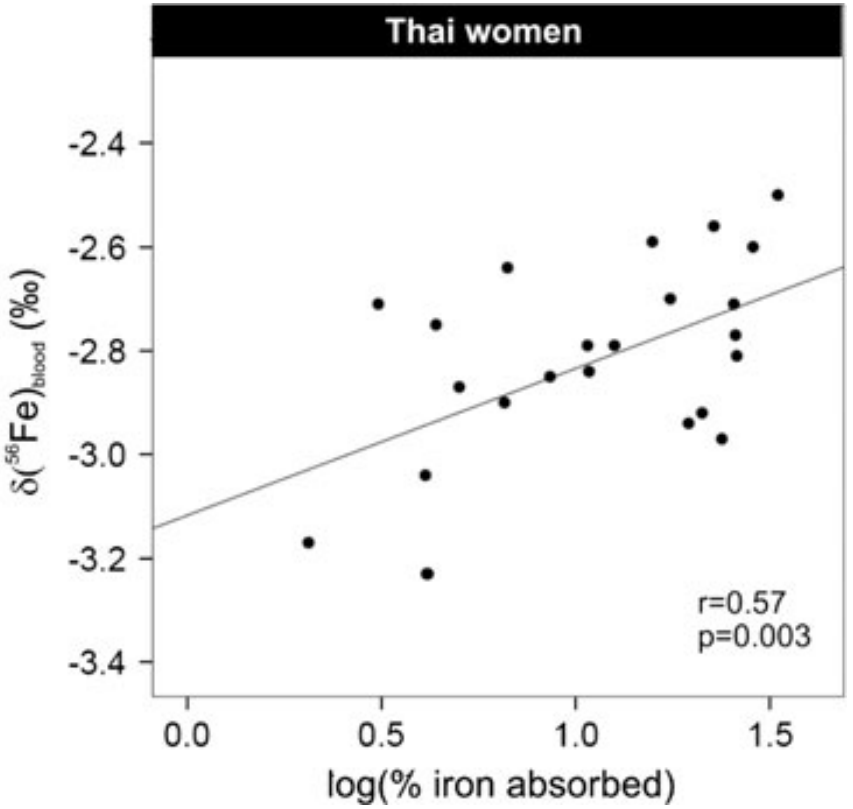
Natural iron isotopic composition of blood is an indicator of dietary iron absorption efficiency in humans

Karin Hotz · Thomas Walczyk

- **Food Authenticity Concept**

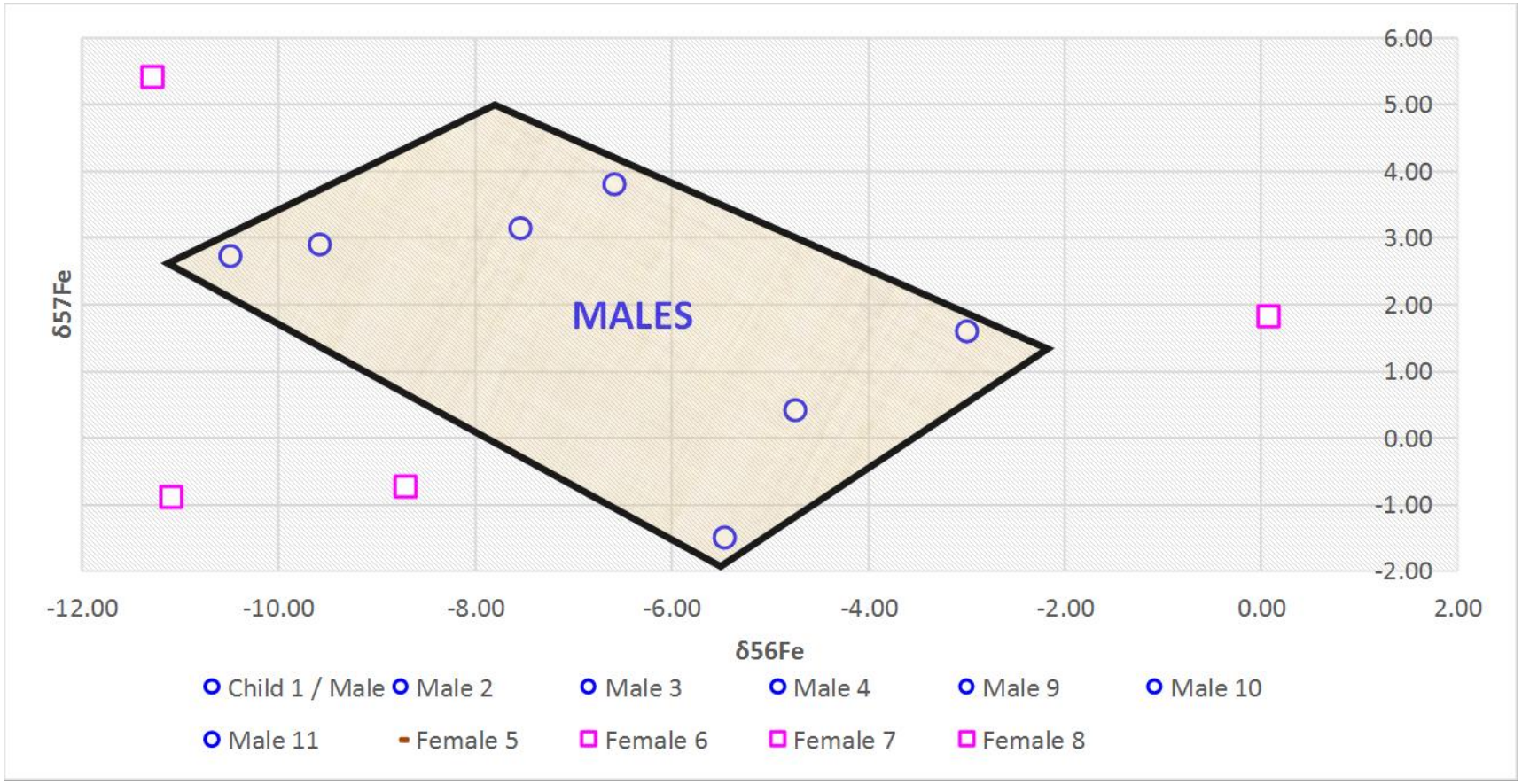
Natural Iron isotopic composition of blood is an indicator of Speciation



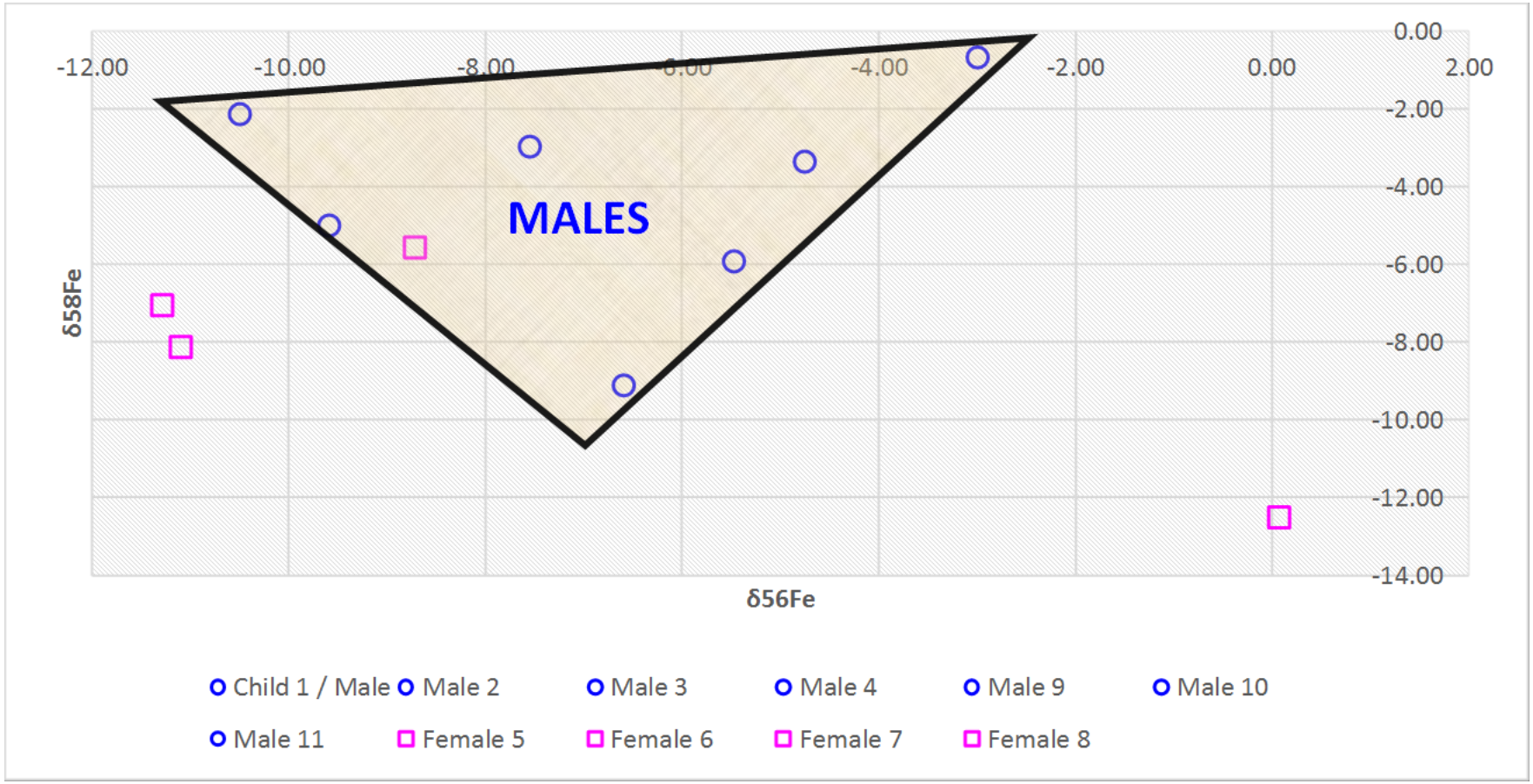


Preliminary Study 2017

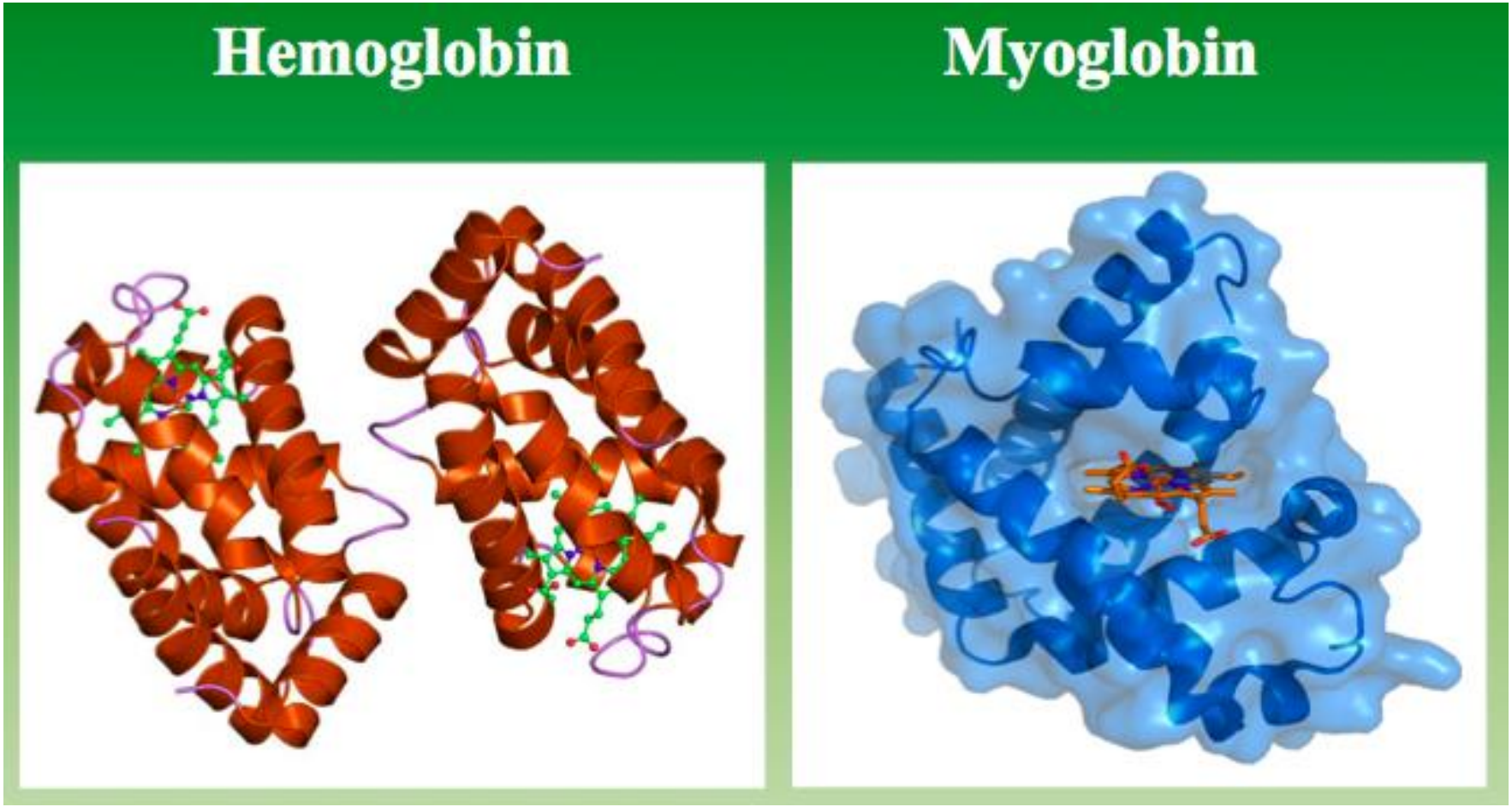
Fe isotopes ratios differences between Female and Male in Human Blood



Fe isotopes ratios differences between Female and Male in Human Blood



Natural Iron isotopic composition of blood is an indicator of Speciation



Differences of $4^{th}/5^{th}$ Decimal Places

Element	Minor Isotope	Natural Abundance [%]
Hydrogen	^{2}H	0.01557
Carbon	^{13}C	1.11140
Nitrogen	^{15}N	0.36630
Oxygen	^{18}O	0.20004
Sulfur	^{34}S	4.21500

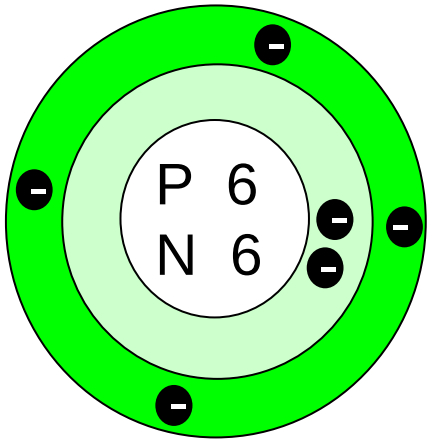
This is where the information is

Isotopic Fingerprinting of Solids and Liquids

Stable Isotopes of Carbon

'light' isotope

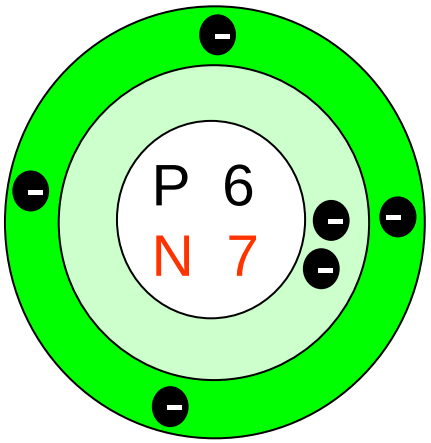
$^{12}_{6}C$



98.93 %

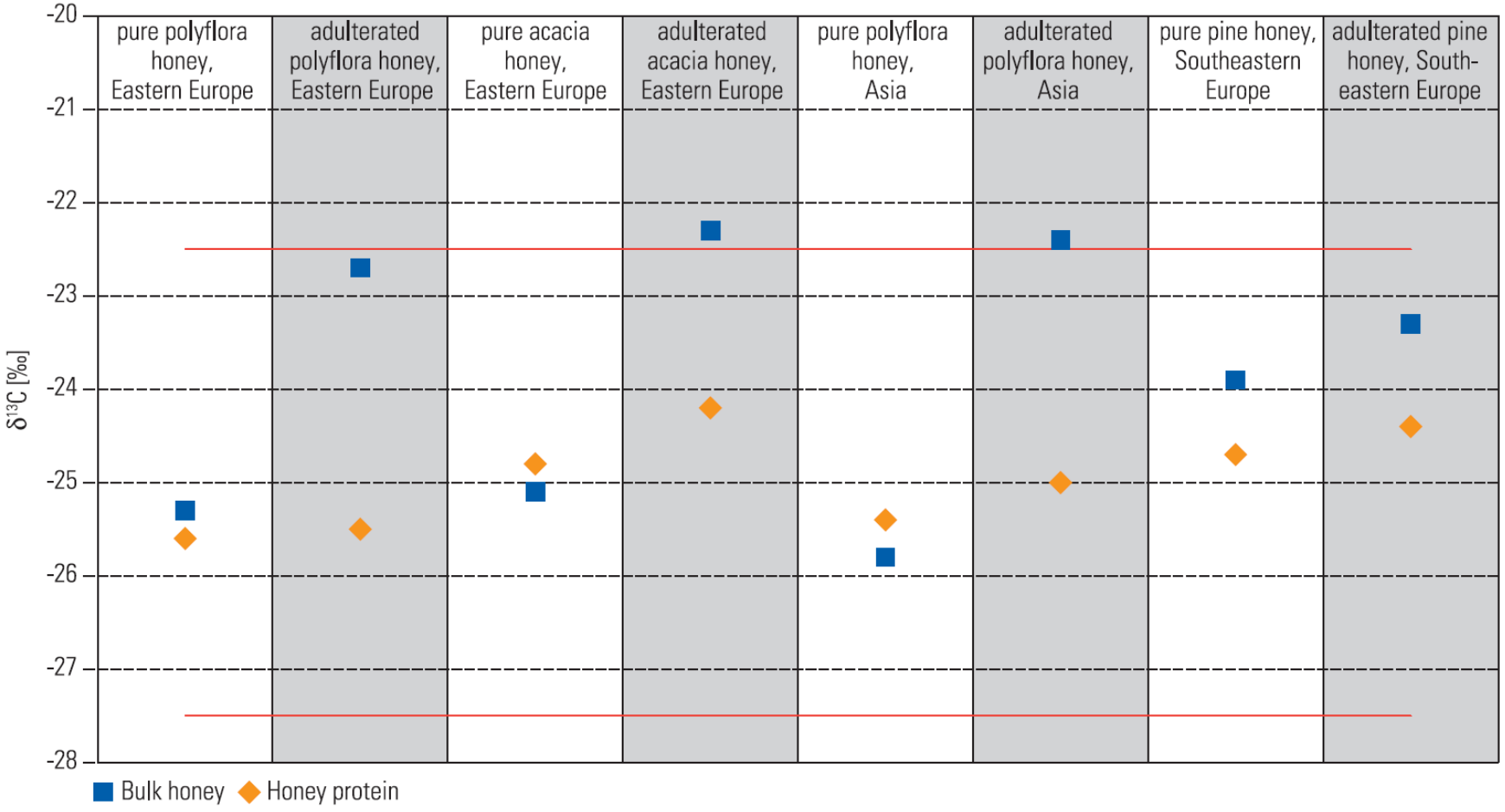
'heavy' isotope

$^{13}_{6}C$

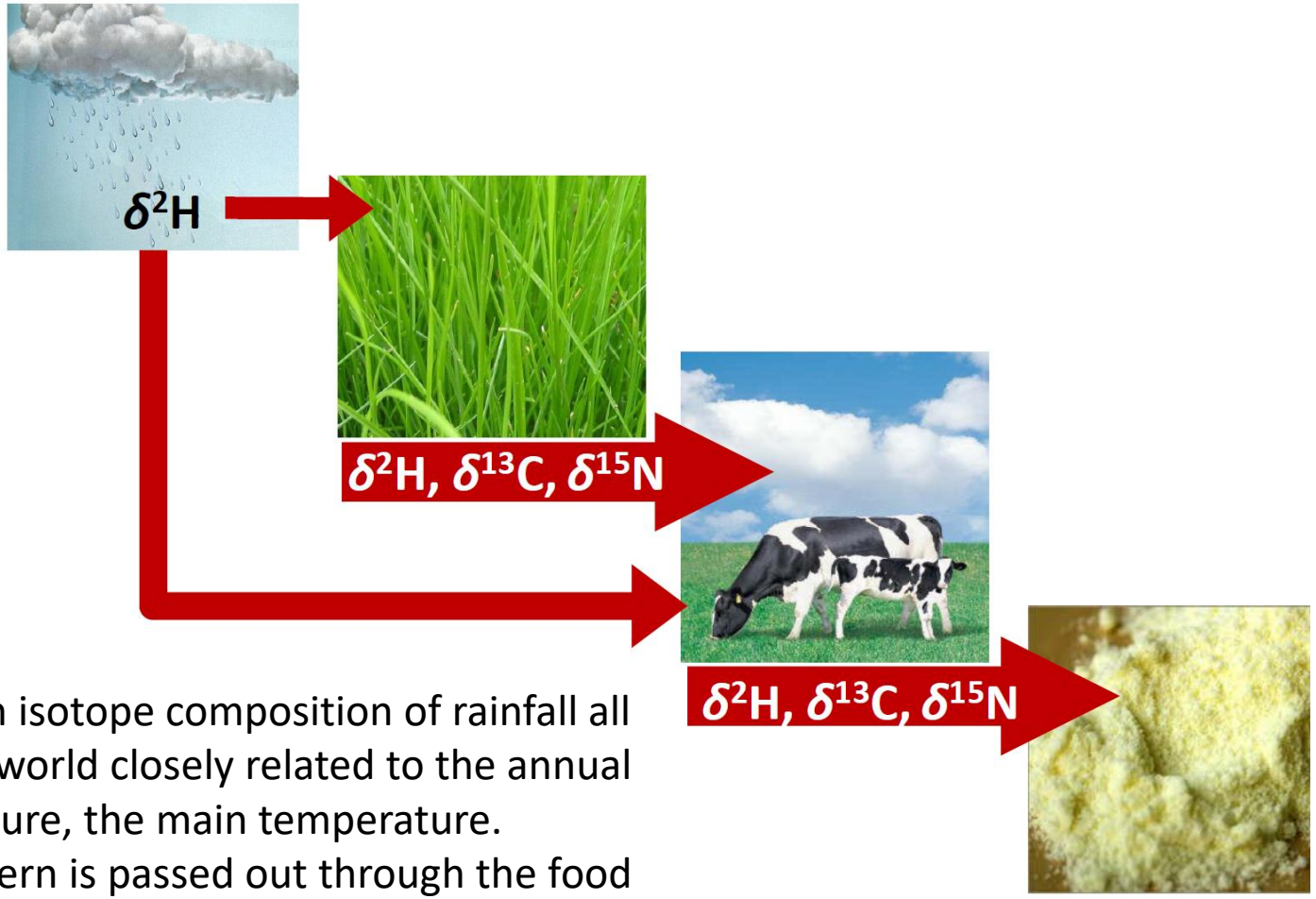


1.07 %

$\delta^{13}C$ values of honey and related proteins Natural variation of difference (honey protein - bulk honey): max. 1 ‰

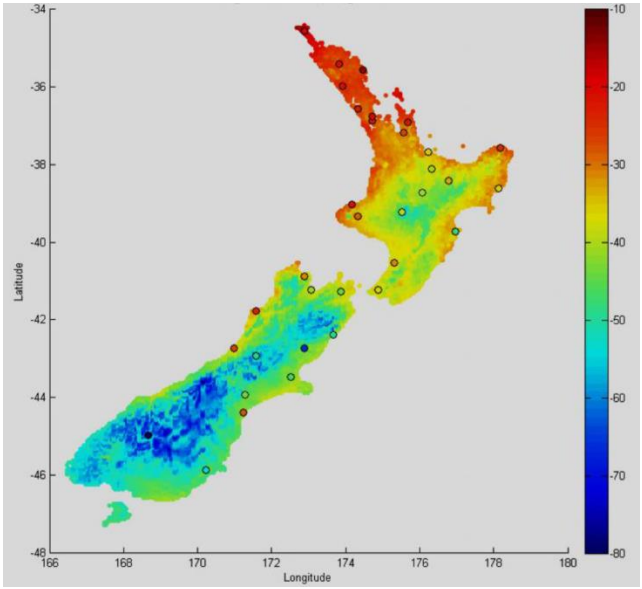


Hydrogen Isotope Composition of Rainfall

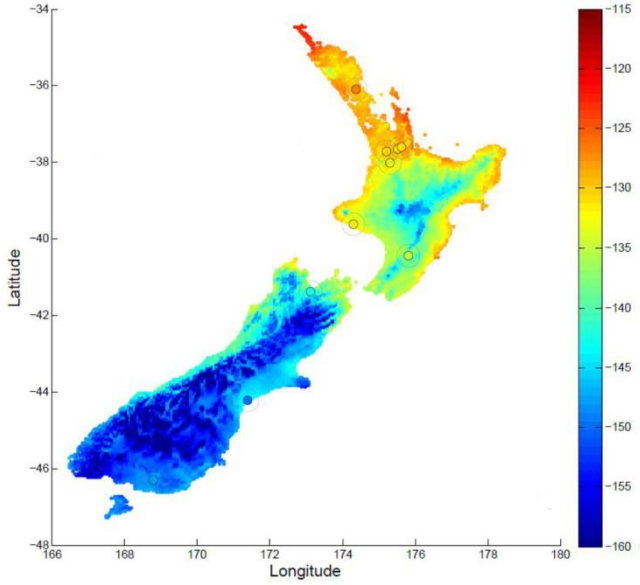


Hydrogen isotope composition of rainfall all over the world closely related to the annual temperature, the main temperature.
That pattern is passed out through the food

New Zealand Rainfall δ^2H

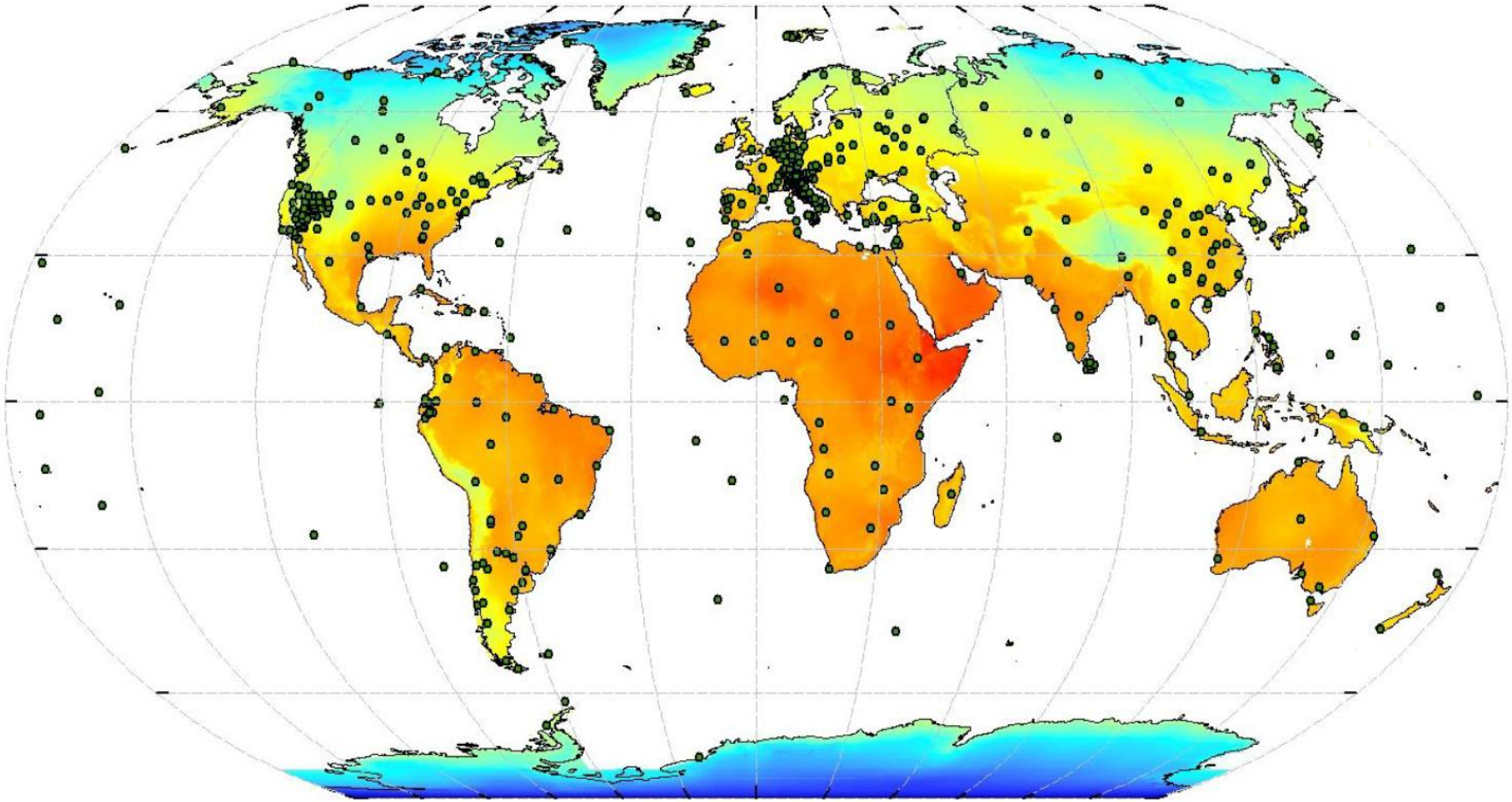


New Zealand Milk Powder δ^2H



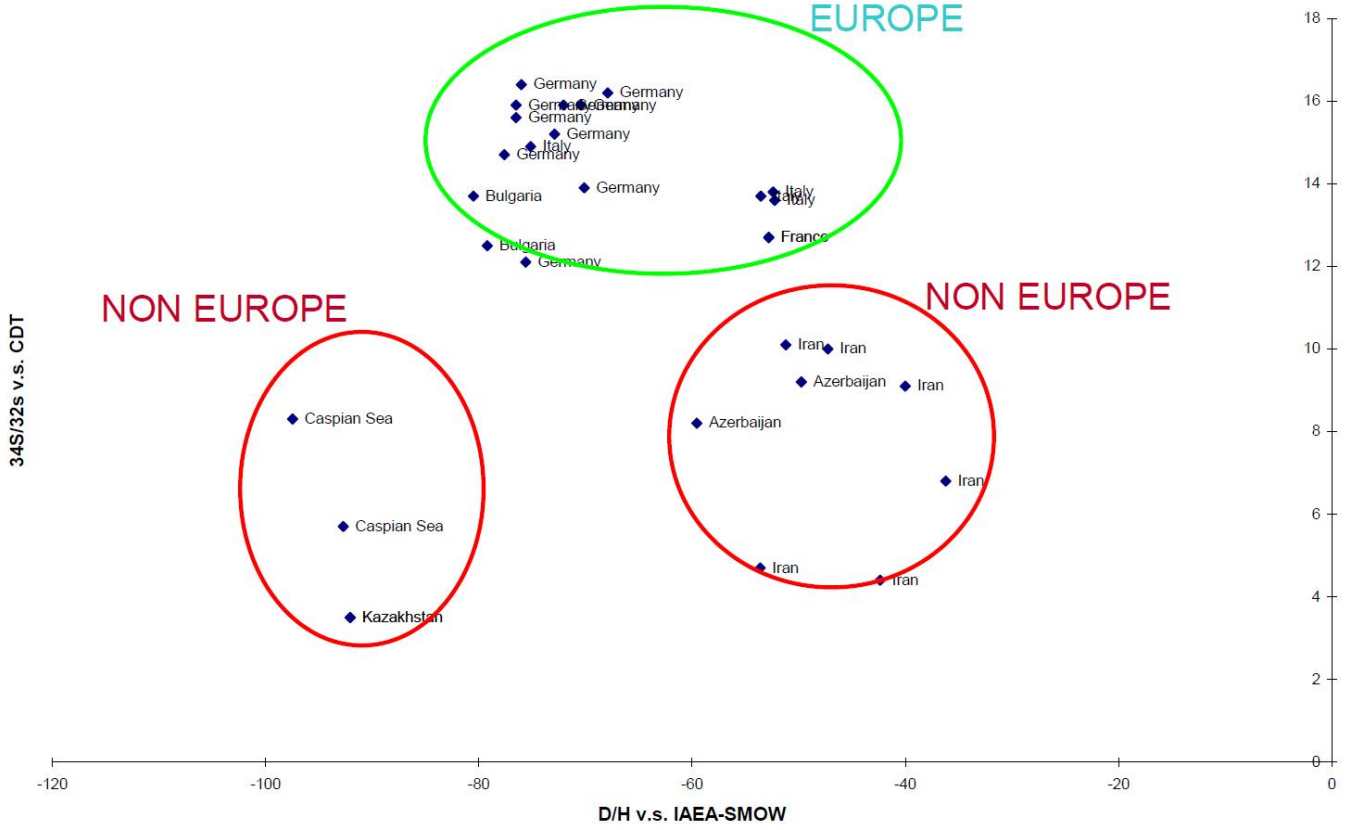
- Ehteshamrad E., et al., 2013. *Journal of Agricultural and Food Chemistry*,

Global Pattern

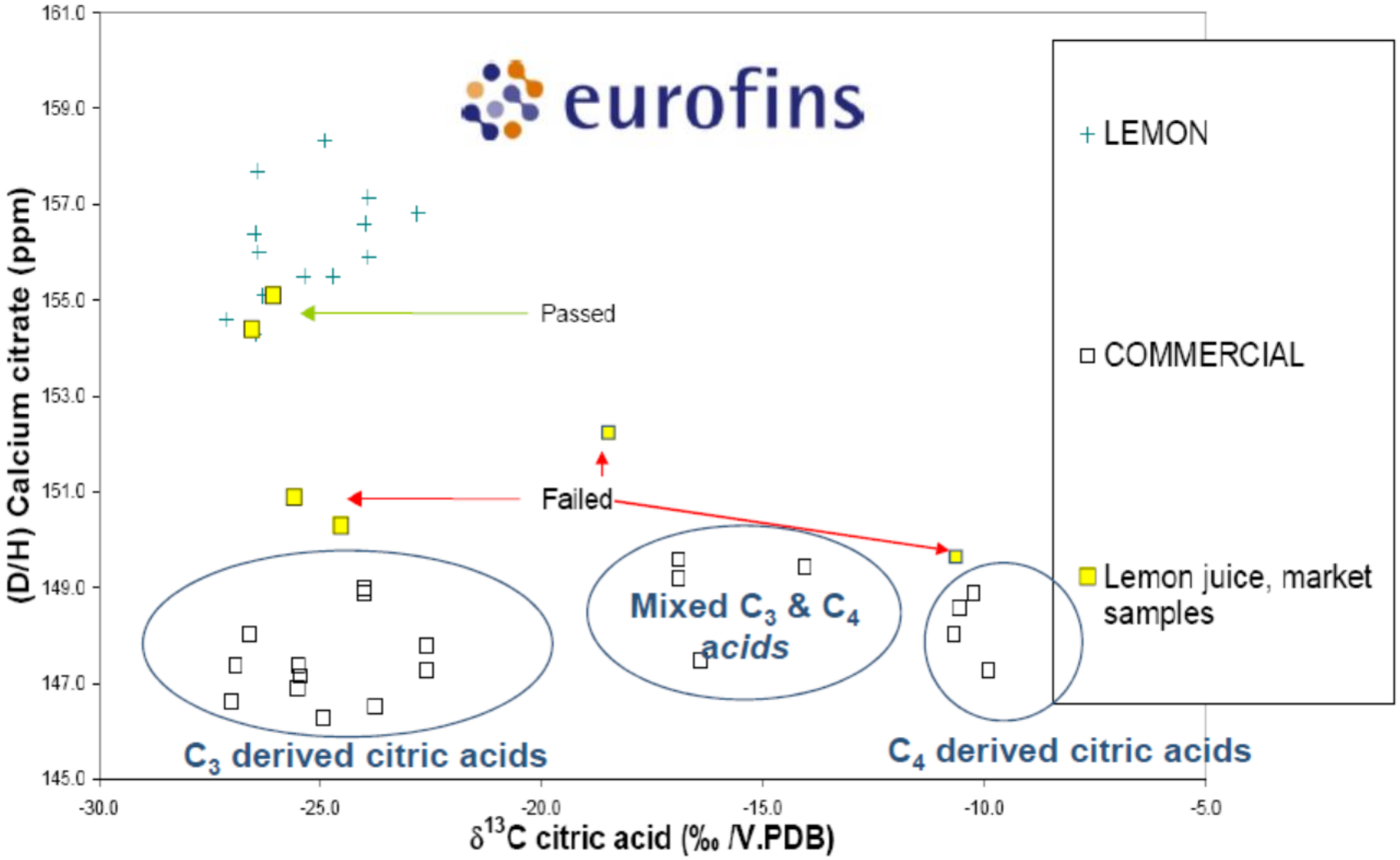


Stable Isotopes

Differentiation of caviar using hydrogen (D/H) and sulfur (34S)



Detection of added C_3 and C_4 derived citric acid to lemon juice



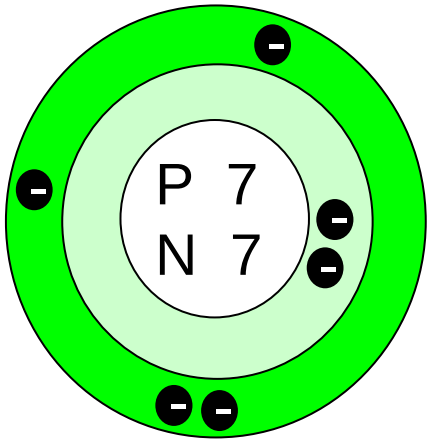
Stable Isotopes of Nitrogen

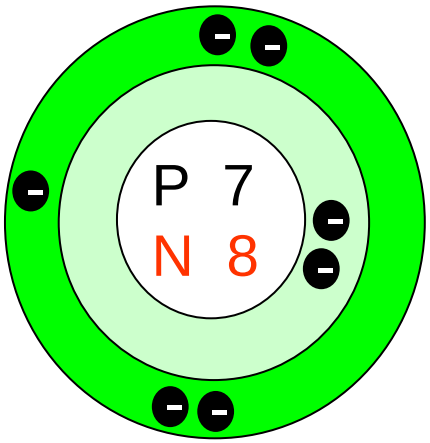
'light' isotope

'heavy' isotope

$^{14}_{7}N$

$^{15}_{7}N$

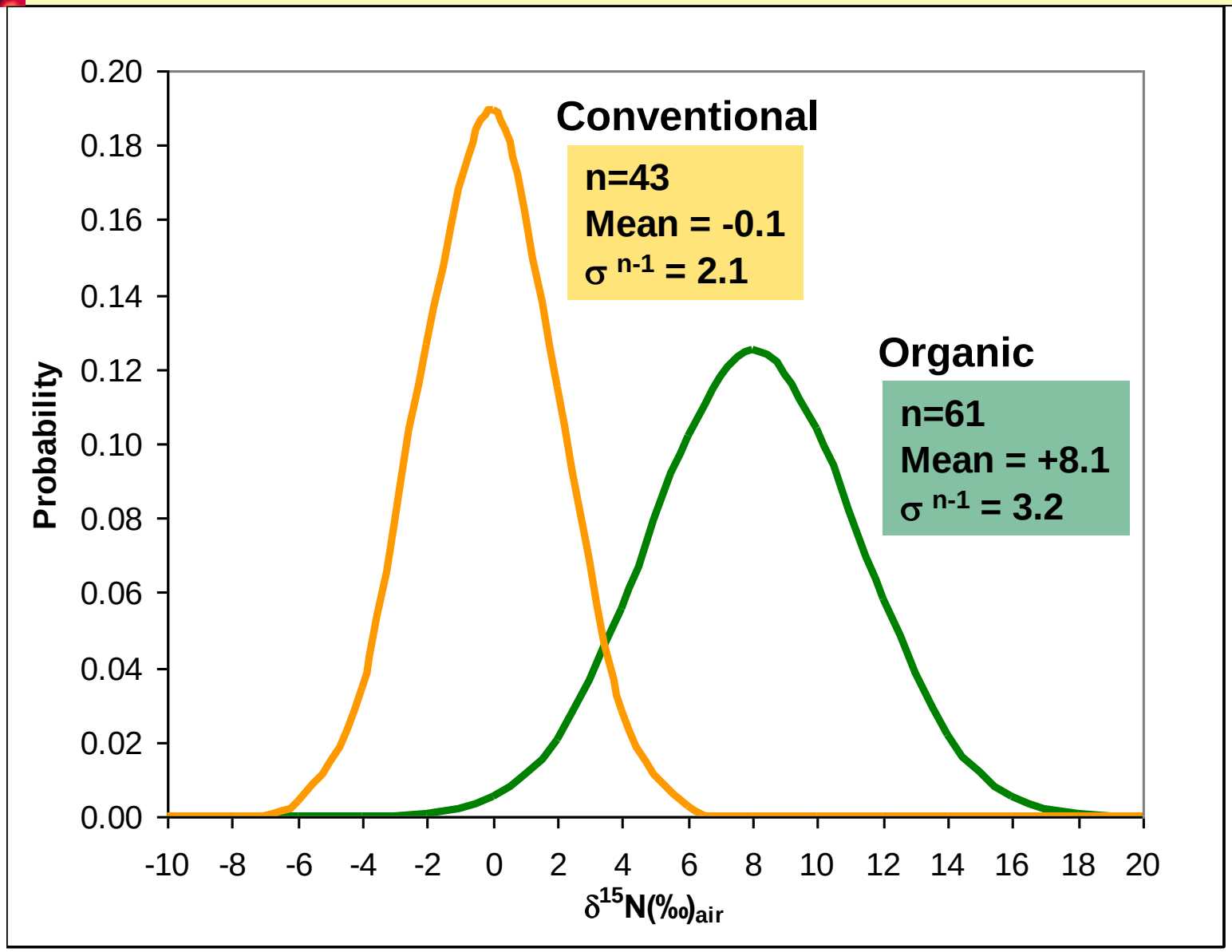




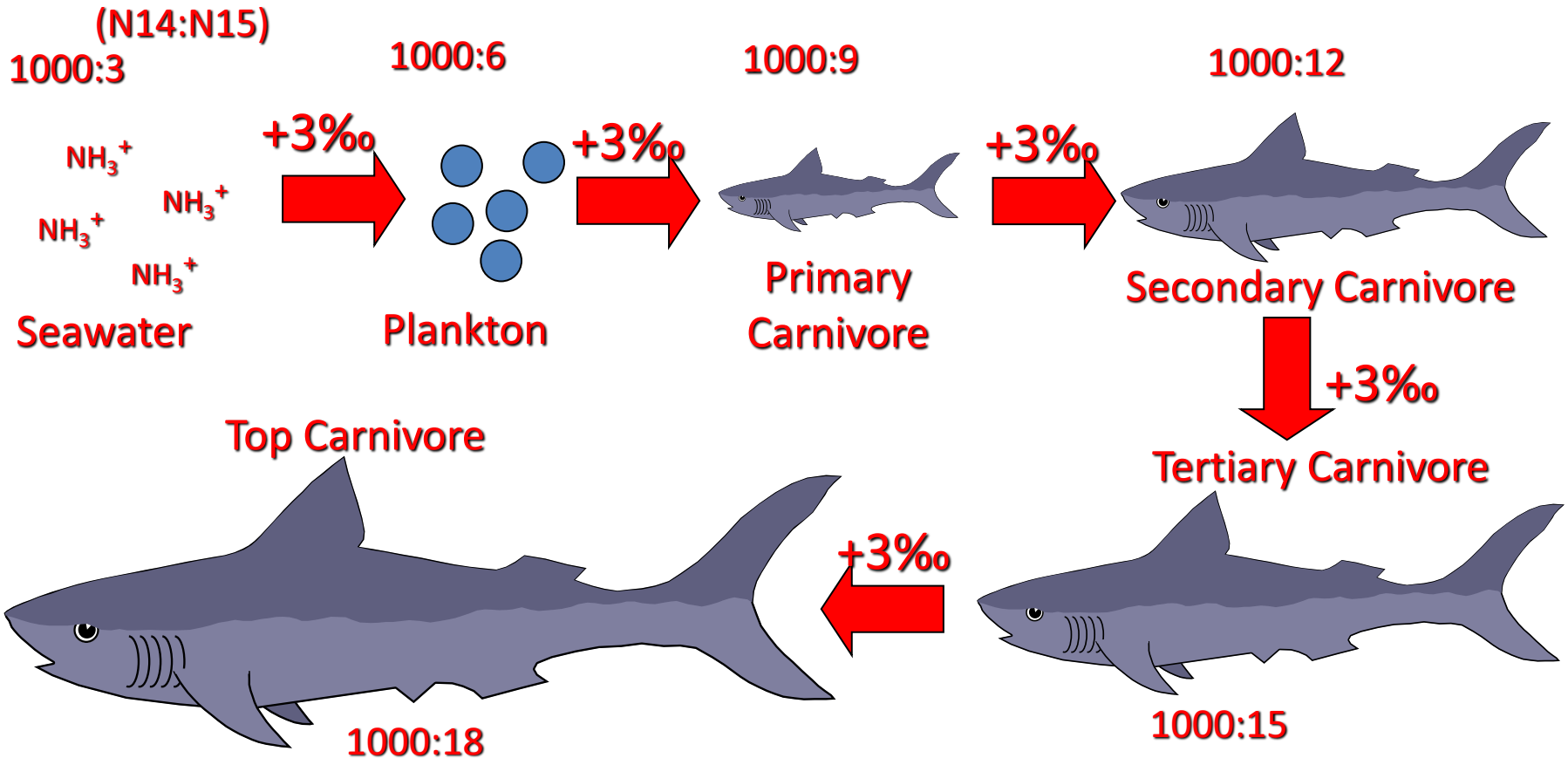
99.63 %

0.37 %

Tomatoes – normal dist^{n}



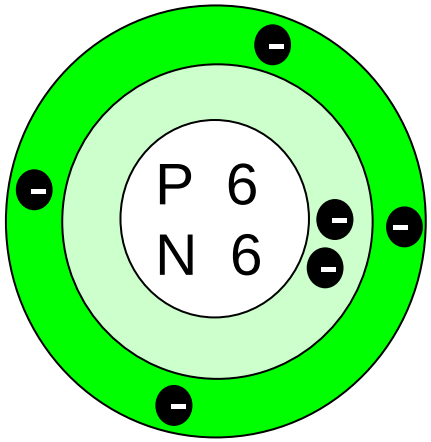
Using Stable Isotopes - What Happens in Nature ?



Stable Isotopes of Carbon

'light' isotope

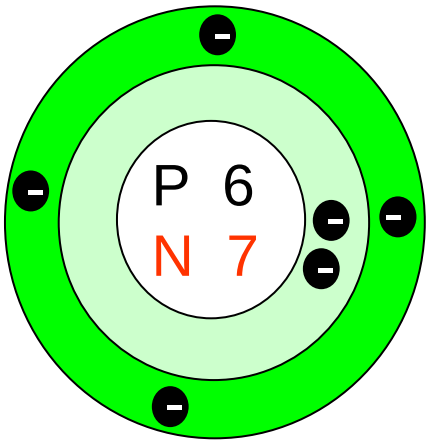
$^{12}_{6}C$



98.93 %

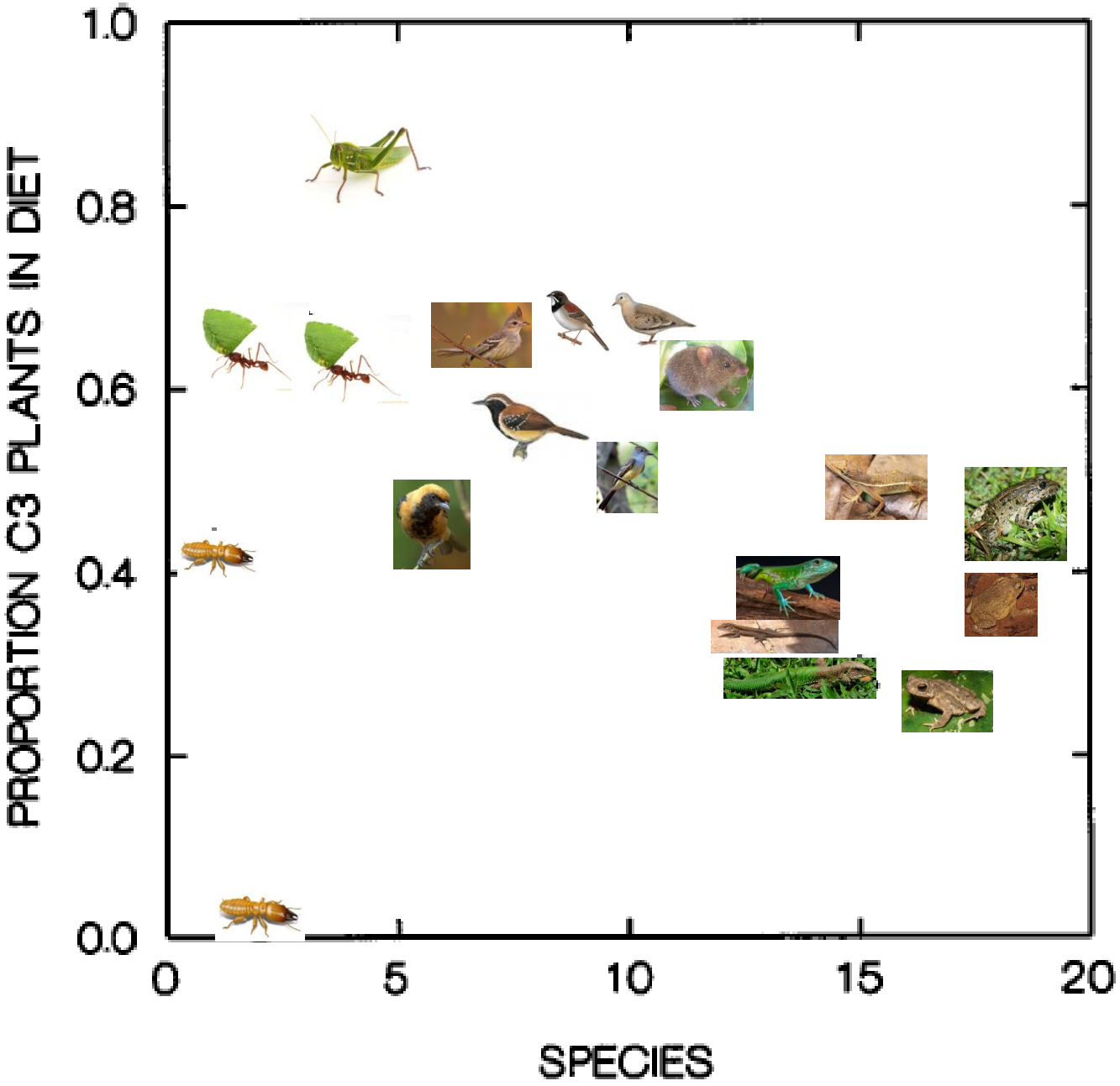
'heavy' isotope

$^{13}_{6}C$



1.07 %

Oecologia (1999) 119:91±96



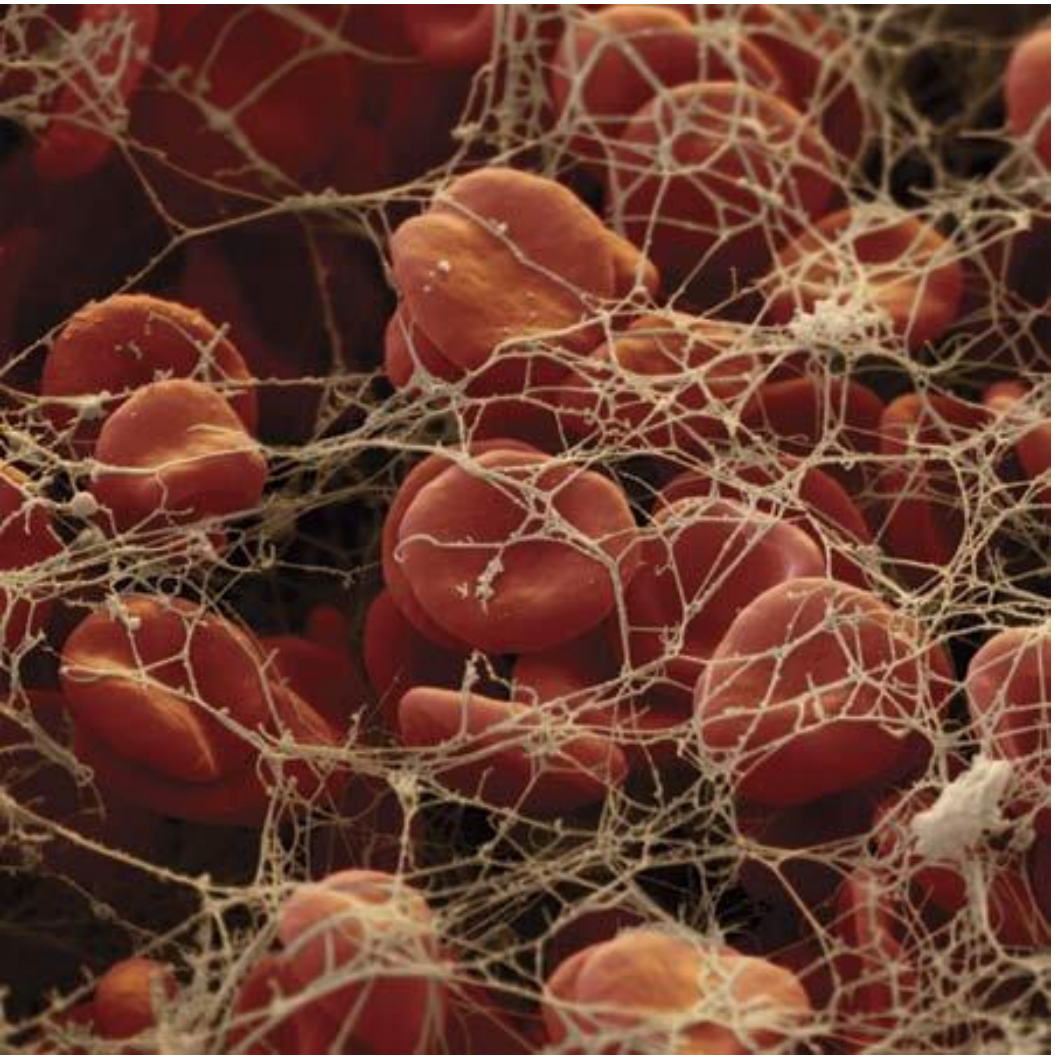
Stable Isotope mass specs are gas-source

D/H	→	H_2
$^{18}O/^{16}O$	→	CO_2, CO, O_2
$^{13}C/^{12}C$	→	CO_2, CO
$^{15}N/^{14}N$	→	N_2
$^{34}S/^{32}S$	→	SO_2, SO, SF_6
$^{37}Cl/^{35}Cl$	→	CH_3Cl

Identifying the species of blood

- based meat binders by LC-MS

Erythrocytes trapped in a mesh of fibrin threads

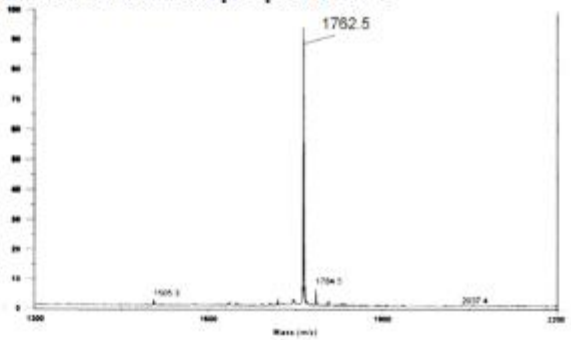


- Fibrin,
  - a tough,
  - insoluble protein
- formed after injury to the blood vessels

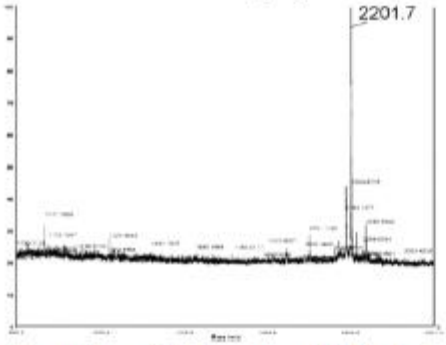
MALDI-TOF MS of synthetic standards

The Food and Environment Research Agency

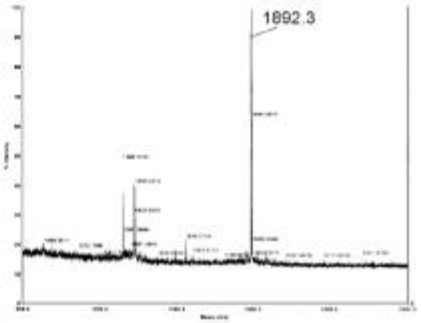
Porcine fibrinopeptide A



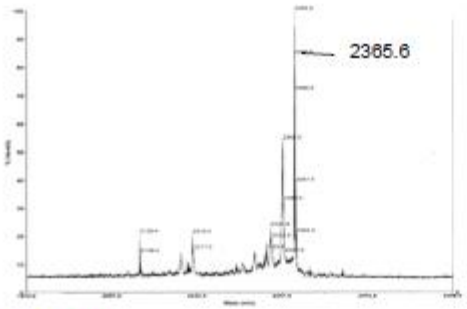
Porcine fibrinopeptide B



Bovine fibrinopeptide A



Bovine fibrinopeptide B



Porcine Fpeptide A:	AEV QDK GEF LAE GGG VR	1762.9
Porcine Fpeptide B:	AID YDE DED GRP KVH VDA R	2201.3
Bovine Fpeptide A:	EDG SDP PSG DFL TEG GGV R	1891.9
Bovine Fpeptide B:	QFP TDY DEG QDD RPK VGL GAR	2364.1 Da

Matrix effects

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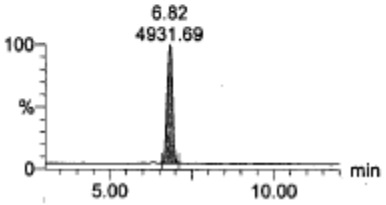
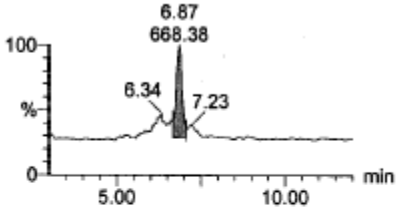
Detection of 5% bovine Fibrimex™ in pork

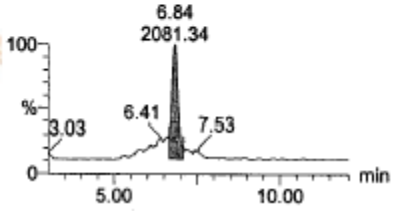
A947>685
(z=2)



B780>296
(z=3)

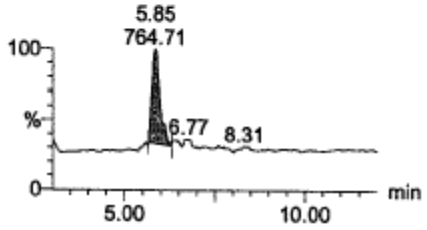


B780>171
(z=3)

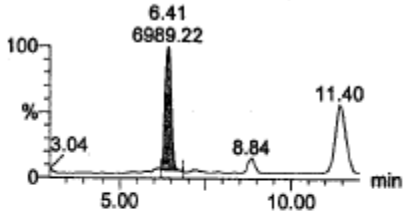


Detection of 5% porcine plasma in beef mince

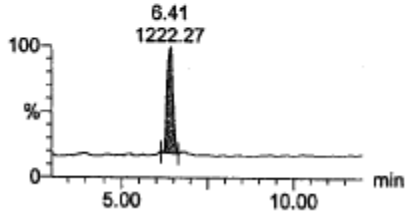
B551>251
(z=4)



A588>201
(*z=3*)

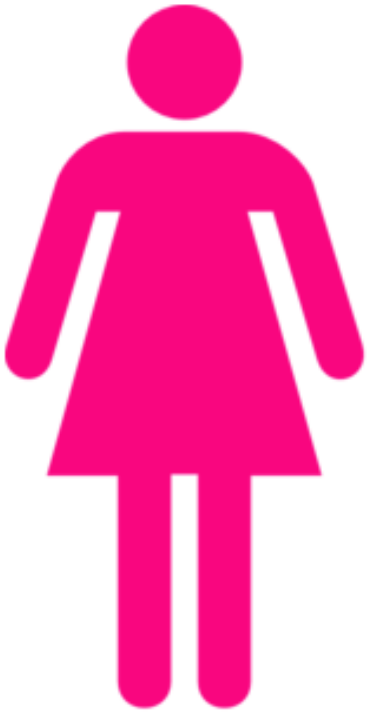


A588>446
(*z=3*)



1 OUT OF 2 MEN

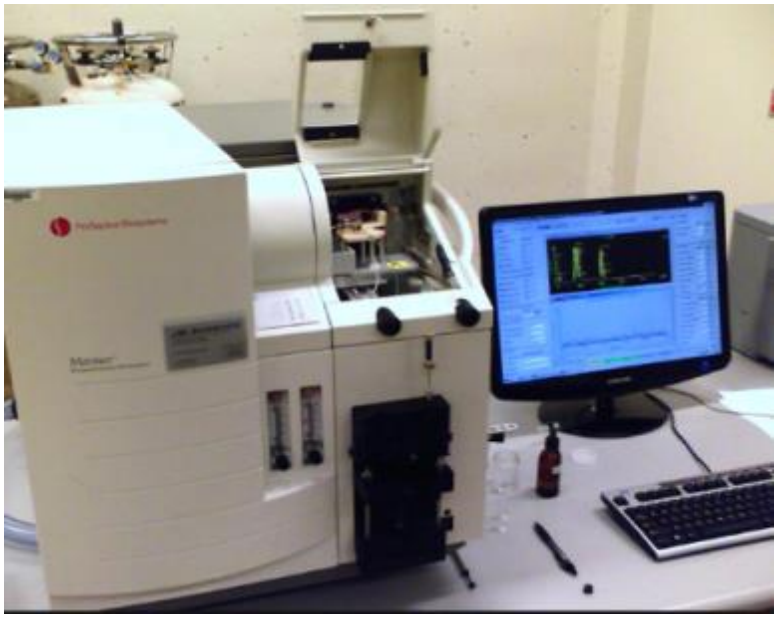
1 OUT OF 3 WOMEN



RISK DEVELOPING CANCER DURING THEIR LIFETIME

THE TWO TECHNOLOGIES

ELECTROSURGERY

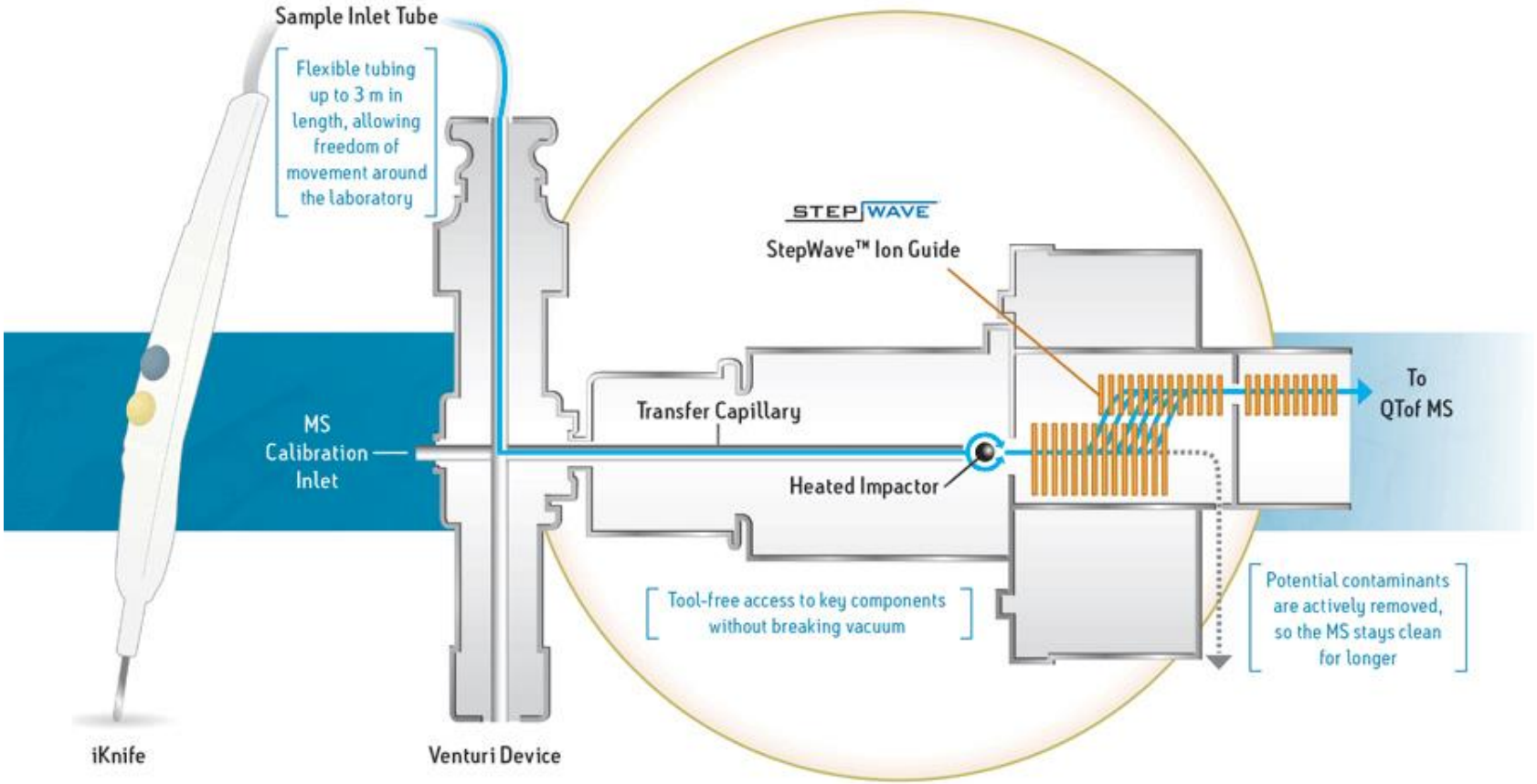


MASS SPECTROMETRY

CLINICAL TRIALS

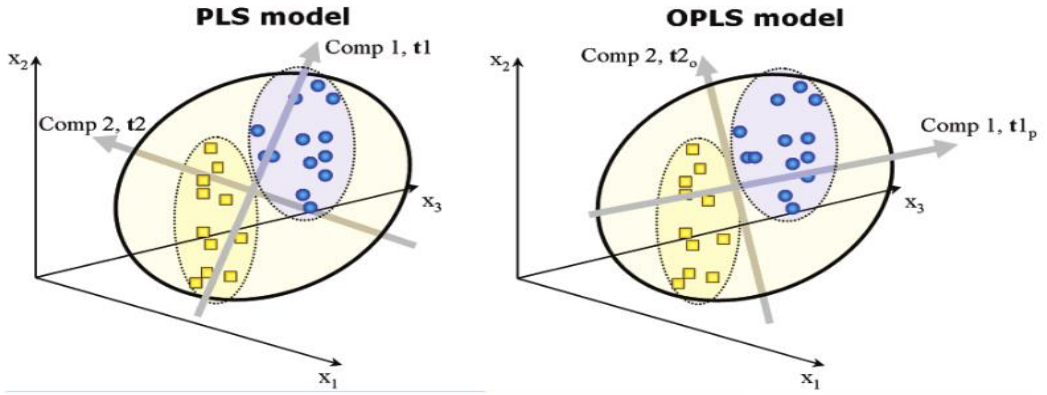
ALL 81 PATIENTS HAD TUMORS REMOVED WITH 100% ACCURACY

REIMS Research System with iKnife for Direct Sampling



Rapid Evaporative Ionization Mass Spectrometry (REIMS)

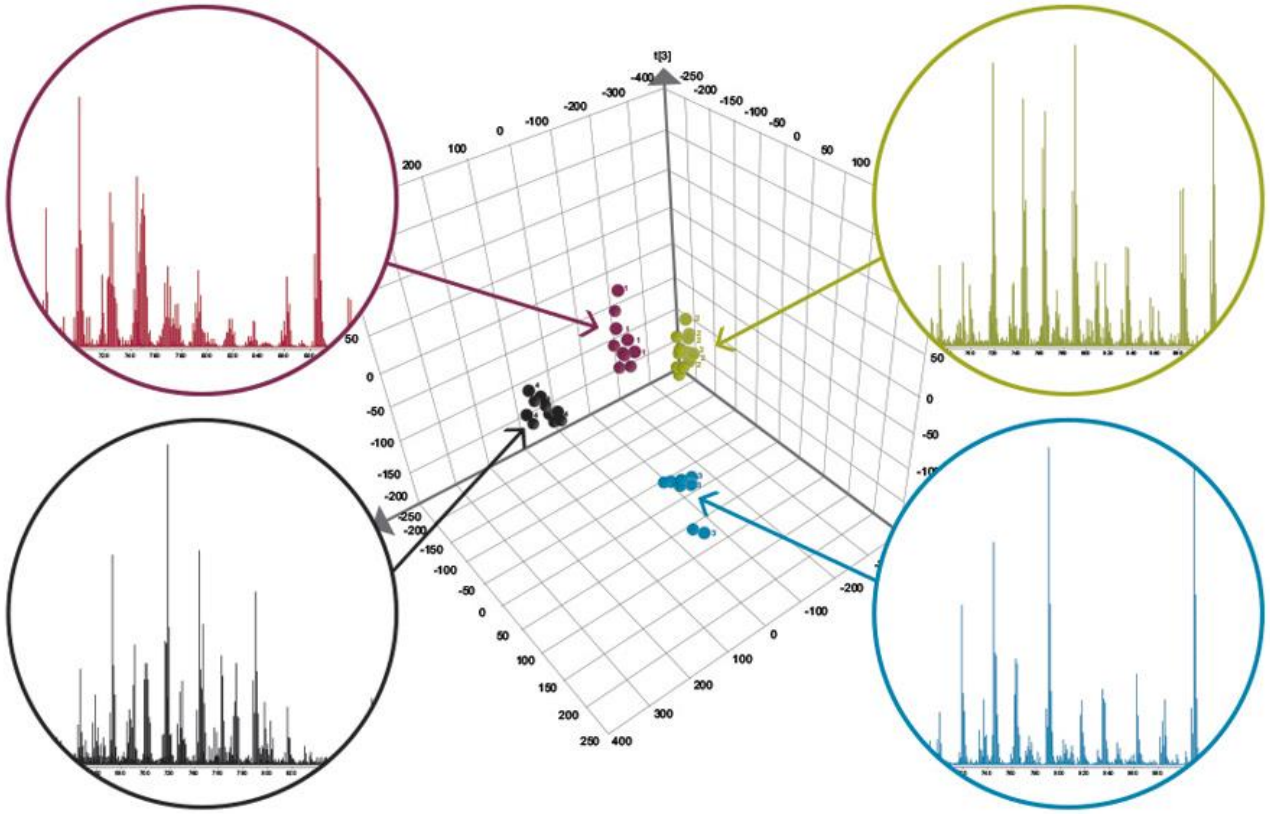
REIMS Research System with iKnife for Direct Sampling



A geometrical illustration of the difference between the PLS-DA and OPLS-DA models. In the left panel, the PLS components cannot separate the between-class variation from the within-class variation, and the resulting PLS component loadings mixes both types of variations. In the right panel, the OPLS components are able to separate these two different variations. Component 1 ($t1_p$) is the predictive component and displays the between-class ([blue circles], [yellow squares]) variation of the samples. The corresponding loading profile can be used for identifying variables important for the class separation. Component 2 ($t2_o$) is the **Y**-orthogonal component and models the within group (within-class) variation.

- OPLS method is a recent modification of the PLS method to help overcome pitfalls
- Main idea to seperate systematic variation in X into two parts, one linearly related to Y and one unrelated (orthogonal).
- Comprises two modeled variations, the Y-predictive ($T_pP_p^T$) and the Y-orthogonal ($T_oP_o^T$) compononents.
- Only Y-predictive variation used for modeling of Y.
- $X = T_pP_p^T + T_oP_o^T + E$
- $Y = T_pC_p^T + F$
- E and F are the residual matrices of X and Y
- OPLS-DA compared to PLS-DA

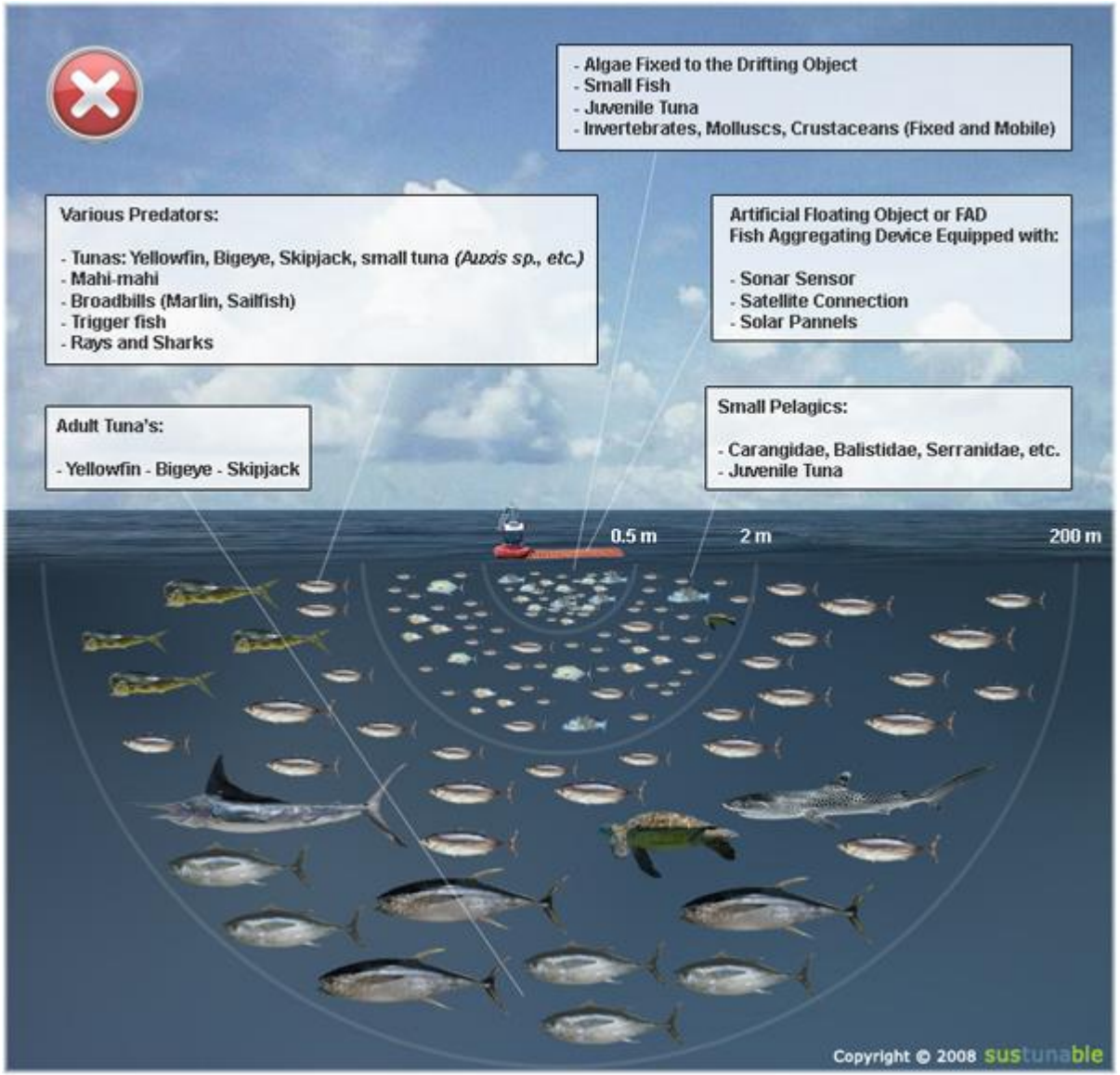
REIMS Research System with iKnife for Direct Sampling



OPLS-DA Plot showing 4 distinct sample groups, with an example mass spectrum from each group.

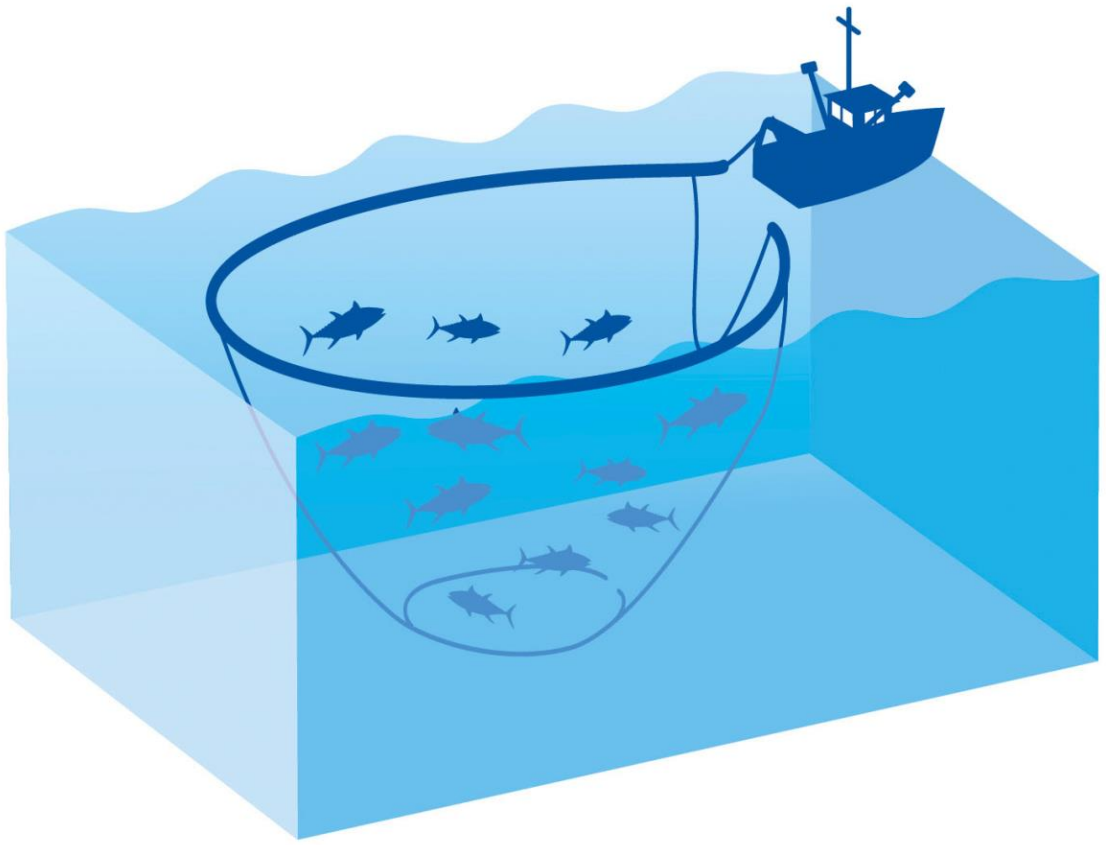
Sustainable Tuna Fishing

Fish Aggregating Devices (FAD's)

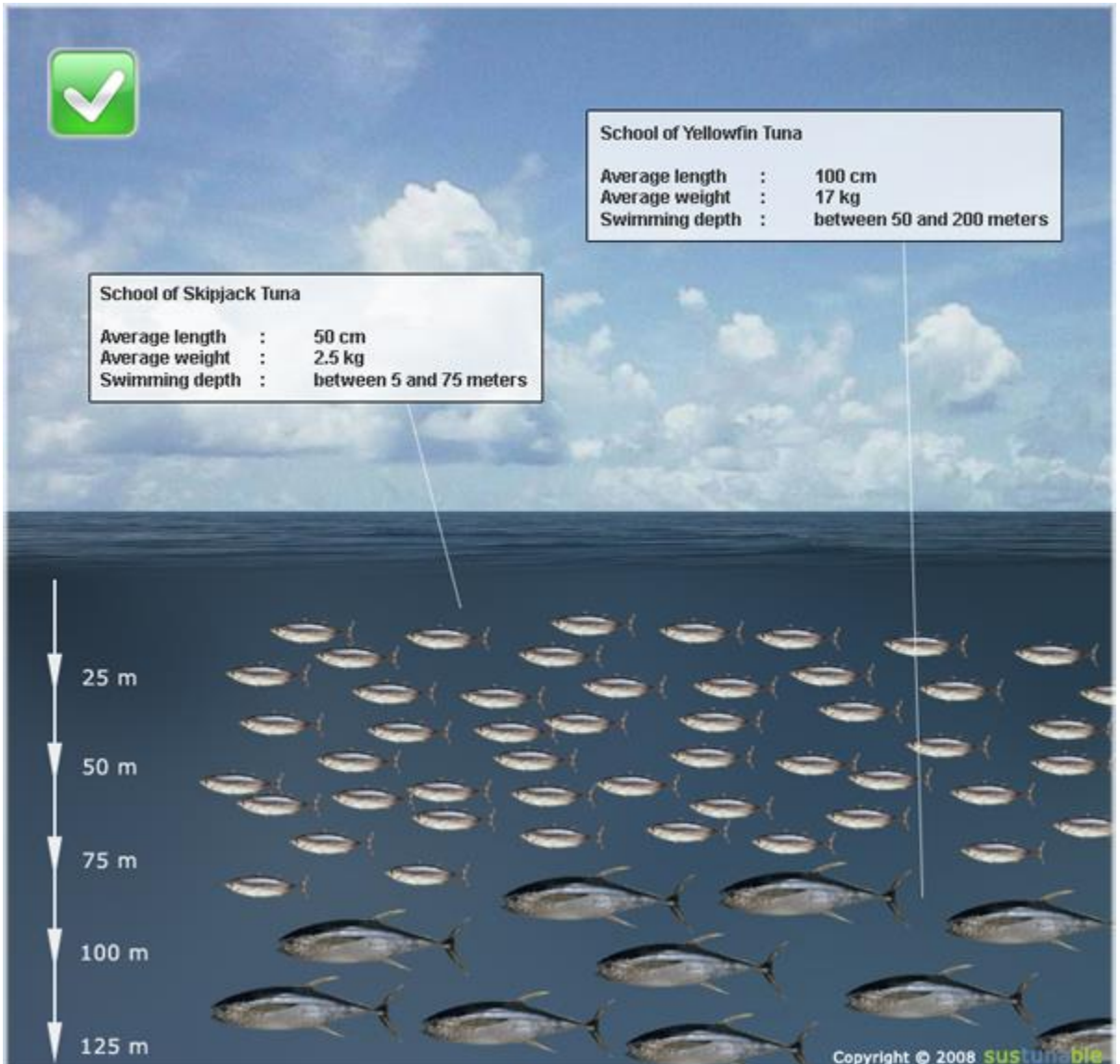


Sustainable Tuna Fishing

Sustainable Tuna Fishing



Sustainable Tuna Fishing



Targeted profiling

- Targeted metabolomic profiling is fundamentally different than most chemometric approaches.
- In targeted metabolomic profiling the compounds in a given biofluid or tissue extract identified and quantified by comparing the spectrum of interest to a library of reference spectra of pure compounds.
- Key advantage: Does not require collection of identical sets = More amenable to human studies or studies that require less day-to-day monitoring.
- Disadvantage: Relatively limited size of most current spectral libraries = bias metabolite identification and interpretation.
- A growing trend towards combining the best features of both chemometric and targeted methods.

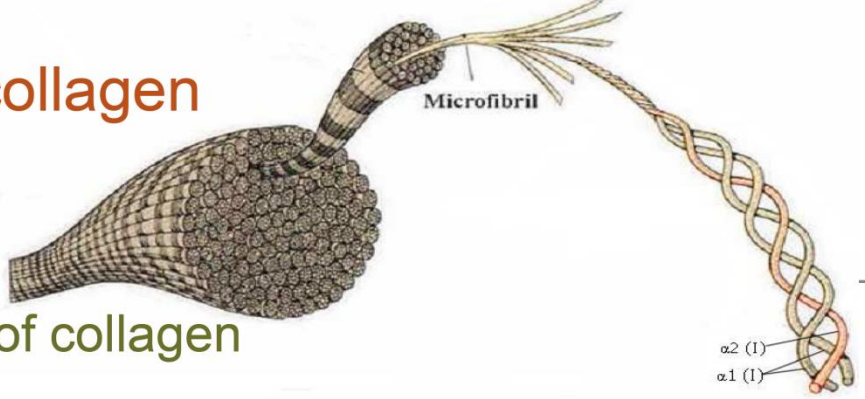
Working with archaeologists ?

fera
The Food and Environment Research Agency

- Derived from collagen
- Bone protein
- Useful species-specific marker
- Collagen more robust than DNA
- Forensic / archaeology / environmental applications

BIOARCH
THE UNIVERSITY of York

Bovine type 1α1 collagen



The Food and Environment Research Agency

More than 27 types of collagen

Type can determine tissue of origin

Fibrous collagens are comprised of two identical alpha1 (I) chains and one genetically different alpha 2 (I)

Large number of repeating amino acid triplets , G-z-x,
where z or x is frequently Proline which can be hydroxylated.

Hydroxyproline determination

Bovine collagen α chain: 138,938 Da protein (1463 amino acids)
156 TRYPTIC PEPTIDES

Bovine alpha 1 (1) Collagen tryptic peptides (Uniprot)

The Food and Environment Research Agency

SEQUENCE	MASS
GAR	302.333
GIK	316.401
GDK	318.33
GPR	328.371
GDR	346.343
TTK	348.4
NPK	357.41
GER	360.37
TSR	362.386
GHR	368.396
DLK	374.437
TCR	378.447
EGSK	419.435
AGER	431.449
SGDR	433.421
DGVR	445.476
NPAR	456.502
YYR	500.555
GGPGSR	529.553
GDAGPK	543.577
DVWK	546.624
GDTGAK	547.566
GAPGDR	571.591
GPAGER	585.618
YHDR	589.608
GDTGPR	601.617
AHDGGR	611.615
GLPGER	627.698
DGSPGAK	630.655
SPEGSR	631.643
AEGNSR	632.631
DCPNAK	646.716
GFPGER	661.715
GADGAPGK	671.708
GAAGEPGK	685.735
GPPGPPGK	705.812

SEQUENCE	MASS
GPAGPPGR	707.787
GAPGPAGPK	750.853
TVIEYK	751.878
GAAGLPGPK	766.895
GDAGPAGPK	768.825
GEPGDAGAK	800.823
NWYISK	809.92
GPAGPQGPR	835.918
GFSGLDGAK	850.927
GEGGPQGPR	853.89
ADDANVVR	858.907
GVVGLPGQR	882.03
GSEGPQGVR	885.932
MCHSDWK	906.042
GPPGSAGSPGK	910.982
QGPSGASGER	944.957
GETGEQGDR	947.914
MFSFVDLR	1014.207
GANGAPGNDGAK	1028.046
DLEVDTTLK	1033.144
GNSGEPGAPGSK	1057.085
GRPGAPGPAGAR	1063.184
GFPGADGVAGPK	1072.186
EGAPGAEGSPGR	1084.111
GVQGPPGPAGPR	1089.219
GLPGTAGLPGMK	1098.326
GQAGVMGFPGPK	1145.342
GVPGPPGAVGPAGK	1160.338
SLSQQIENIR	1187.318
GLTGSPGSPGPDGK	1226.308
GEAGPSGPAGPTGAR	1281.348
GFPGLPGPSGEPGK	1296.445
GPSGPQGPSGPPGPK	1316.436
GEPGPAGLPGPPGER	1387.515
GSAGPPGATGFPGAAGR	1427.539
ALLLQGSNEIEIR	1455.674

SEQUENCE	MASS
STGISVPGPMGPSGPR	1496.701
DGLNGLPGPIGPPGPR	1513.716
GANGAPGIAGAPGFPGAR	1537.698
GLTGPIGPPGPAGAPGDK	1558.754
GETGPAGPAGPIGPVGAR	1560.73
NSVAYMDQQTGNLK	1568.722
GSPGEAGRPGEAGLPGAK	1607.743
NGDDGEAGKPGRPGER	1611.648
DGEAGAQGPPGPAGPAGER	1690.746
GEPGSPGENGAPGQMGPR	1694.796
LMSTEASQNITYHCK	1725.952
VGPPGPSGNAGPPGPPGPAGK	1764.958
GPPGPMGPPGLAGPPGESGR	1785.007
GEPGPTGIQGPPGPAGEEGK	1831.957
TGPPGPAGQDGRPGPPGPPGAR	1993.169
SGEYWIDPNQGCNLDAIK	2023.204
GAPGADGPAGAPGTPGPQGIAGQR	2057.21
FTYSVTYDGCTSHTGAWGK	2081.244
GEPGPPGPAGFAGPPGADGQPGAK	2086.248
GETGPAGPPGAPGAPGAPGPVGPAGK	2121.337
GDAGAPGAPGSQGAPGLQGMPGER	2135.296
GETGPAGRPGEVGPPGPPGPAGEK	2168.351
GEPGPPGPAGAAGPAGNPGADGQPGAK	2252.385
GDAGPPGPAGPAGPPGPIGNVGAPGPK	2259.506
VFCNMETGETCVYPTQPSVAQK	2432.764
GNDGATGAAGPPGPTGPAGPPGFPGAVGAK	2500.71
GFSGLQGPPGPPGSPGEQGPSGASGPAGPR	2657.839
PVPCQICVCDNGNVLCDDVICDELK	2708.145
LPIIDVAPLDVGAPDQEFGFDVGPACFL	2916.338
GLPGPPGAPGPQGFQGPPGEPGEPGASGPMGPR	3005.314
VPTDECCPVCPEGQESPTDQETTGVEGPK	3033.257
TGDAGPAGPPGPPGPPGPPGPPSGGYDLSFLPQPPQEK	3588.935
DGIPGQPGLPGPPGPPGPPGPPGLGGNFAPQLSYGYDEK	3753.142
HVWYGESMTGGFQFEYGGQGSDPADVAIQLTFLR	3765.127
GEQGPAGSPGFQGLPGPAGPPGEAGKPGEQGVPGDLGAPGPSGAR	4018.328
LLLLLAATALLTHGQEEGQEEGQEEDIPPVTCVQNGLR	4086.583

Theory

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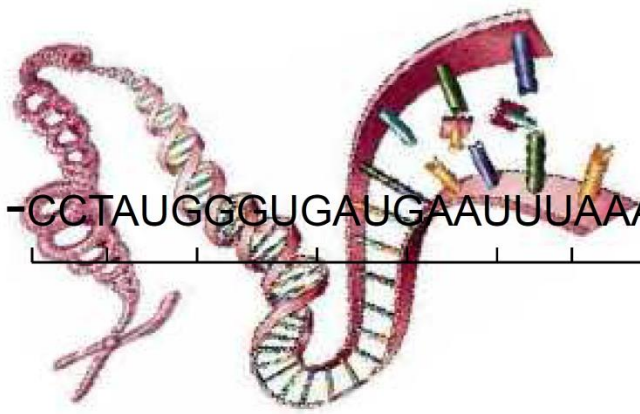
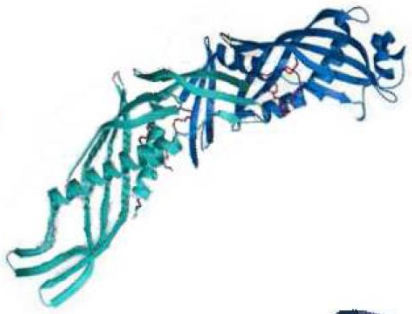


-CCTAUGGCUGAUGAAUUUAAACGAAAUUUAGCUAUUACUGUUUCUCCUCAACAUUAUUGU

Protein sequence database (*www.**uniprot**.org*)



A = Alanine	M=Methionine
C = Cysteine	N=Asparagine
D= Aspartic acid	P=Proline
E=glutamic acid	Q=glutamine
F= Phenylalanine	R =Arginine
G=glycine	S=Serine
H=Histidine	T=Threonine
I=Isoleucine	V= Valine
K = Lysine	W = Tryptophan
L=Leucine	Y=Tyrosine

LC QTOF MSMS Mass Spectrometry

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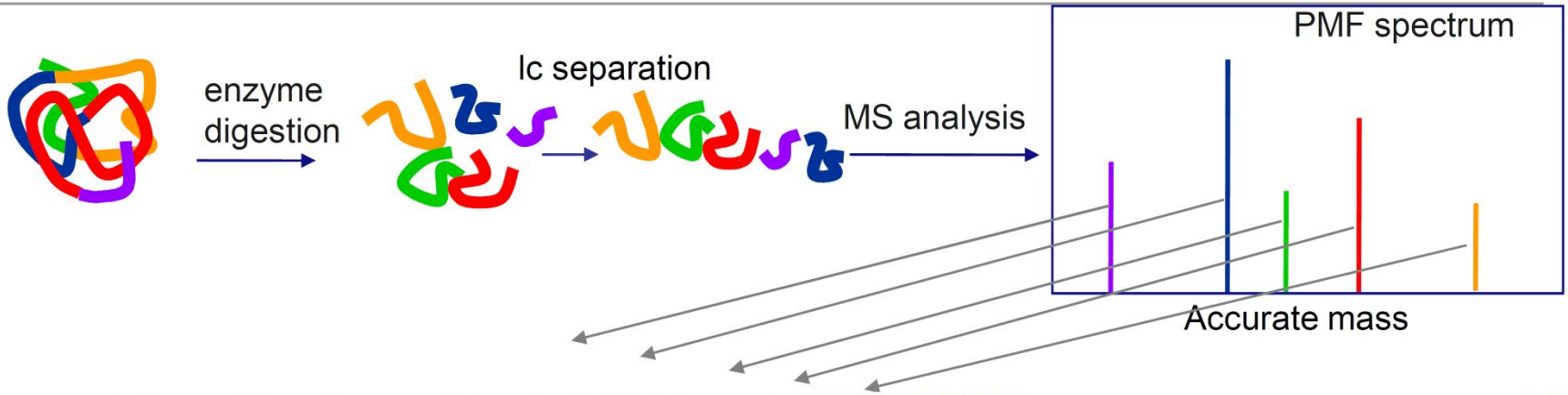
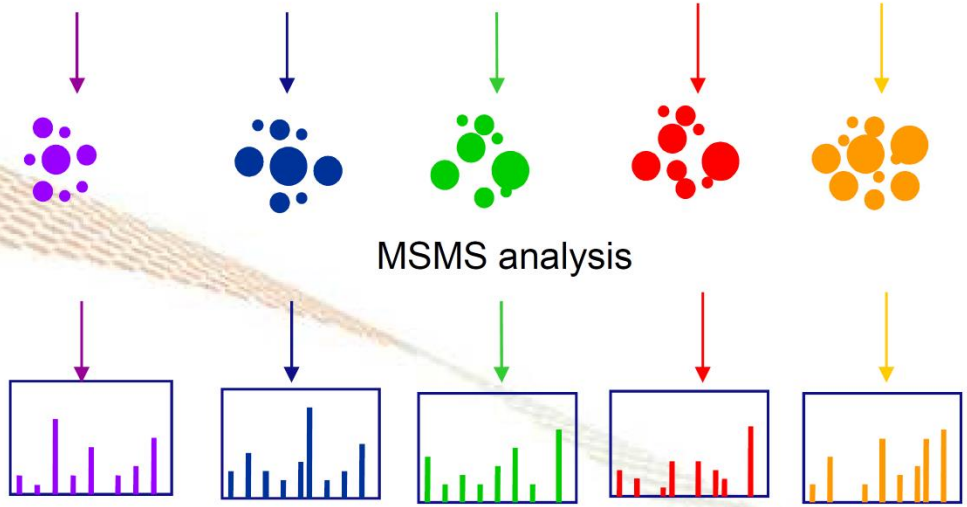
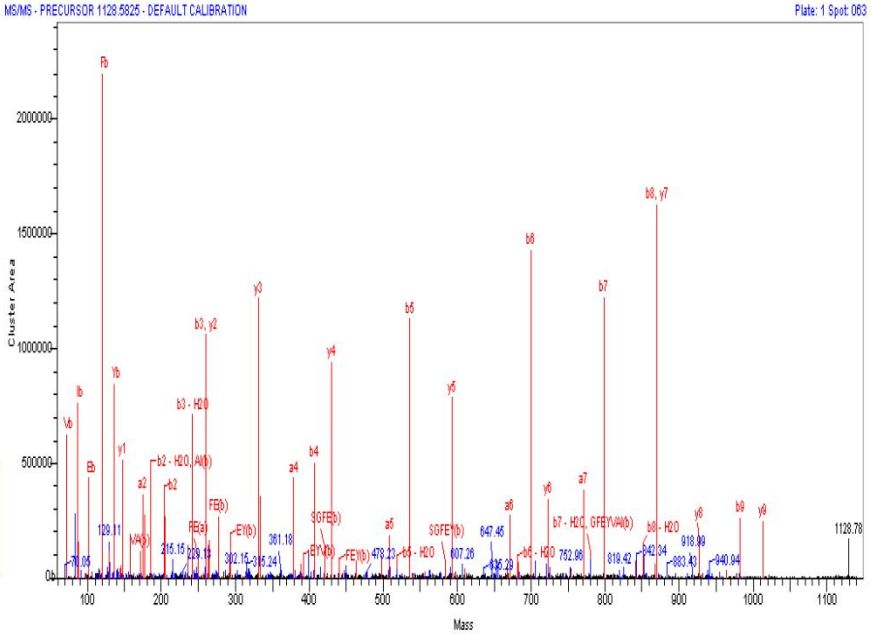
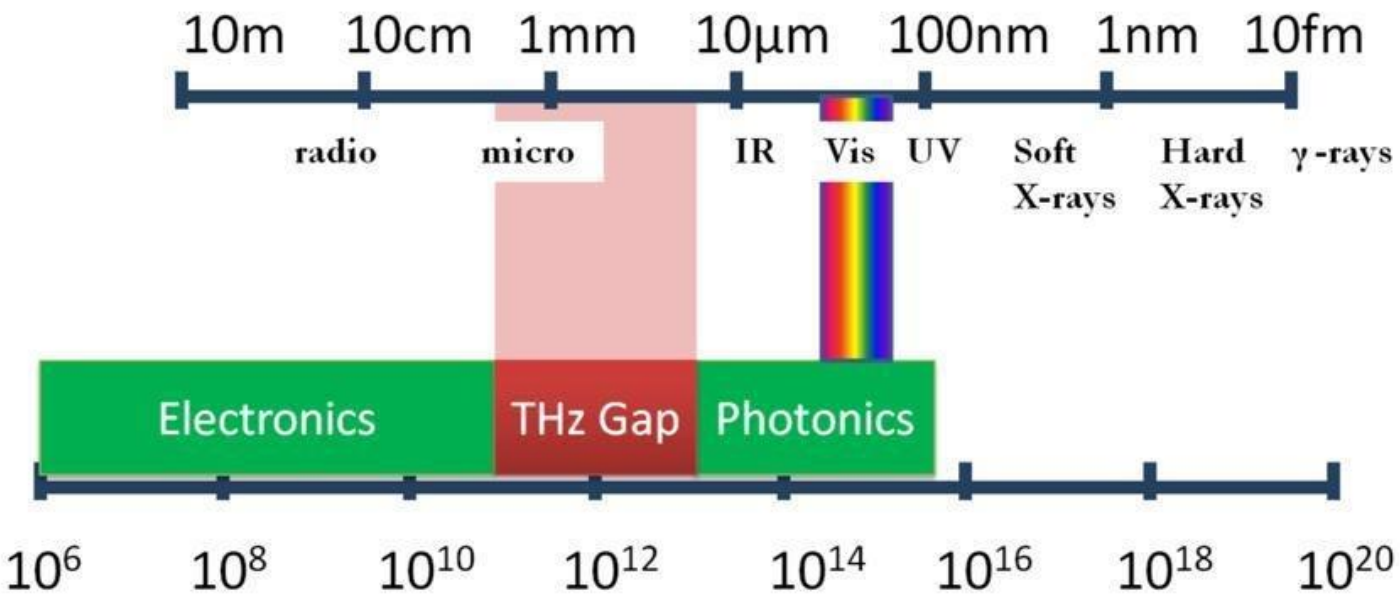


Fragmentation of tryptic peptides in MSMS mode

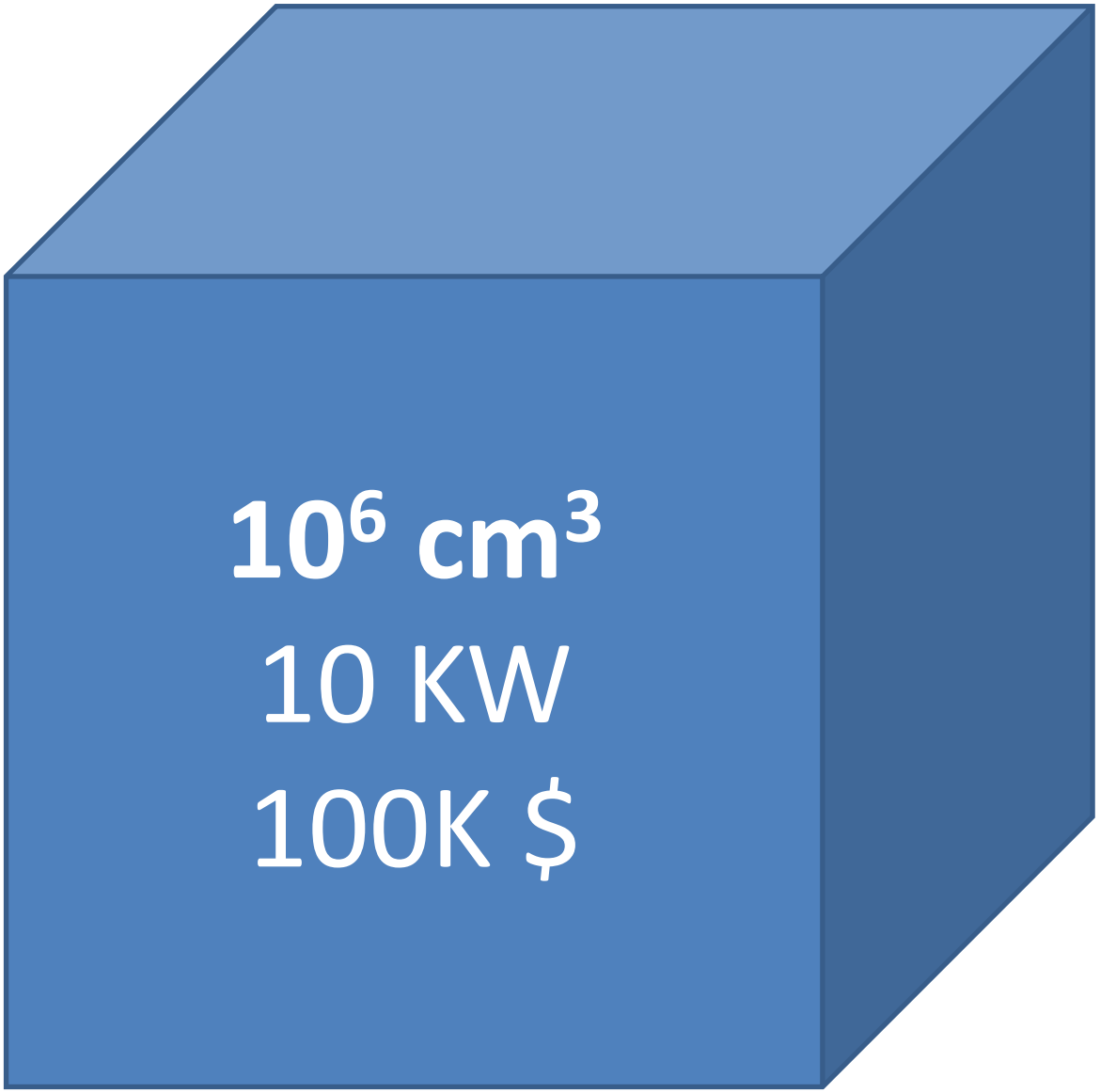




Electromagnetic Spectrum



Terahertz



10 cm3

1 W

10 $

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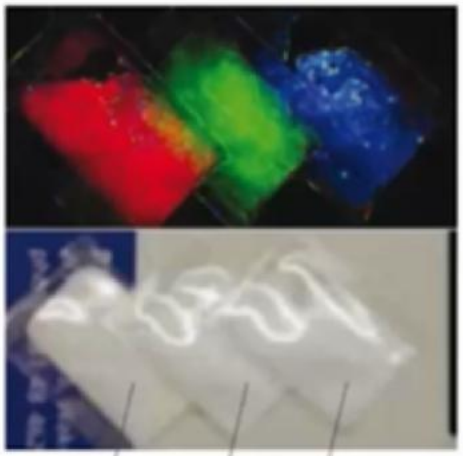
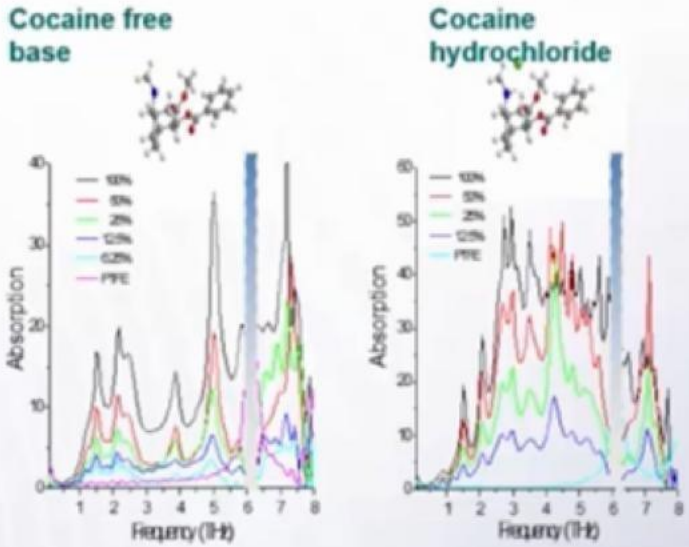
Application areas

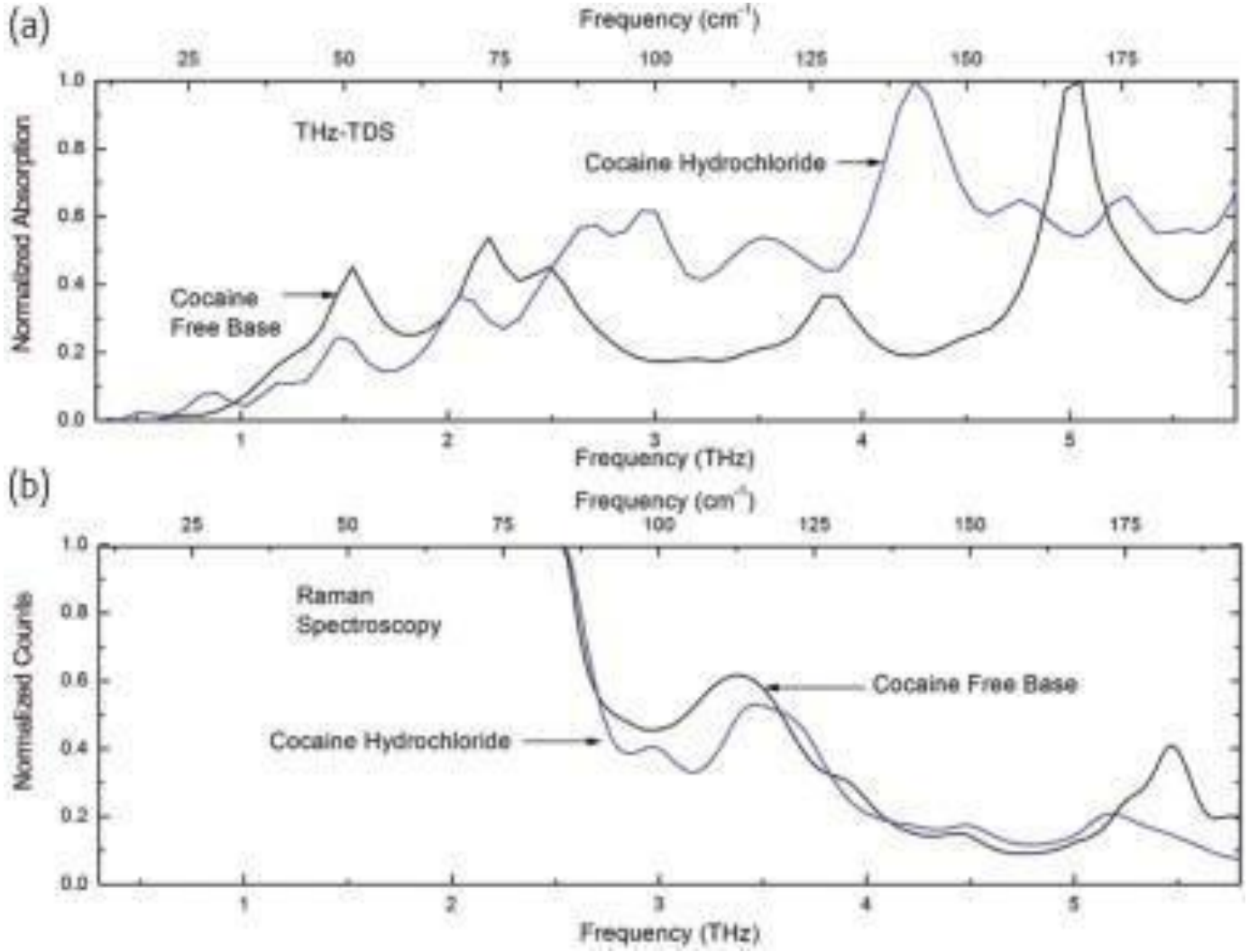


Nagoya group data



Leeds group data

Comparison between the far-IR spectra of polycrystalline cocaine free base and cocaine hydrochloride obtained using (a) THz-TDS and (b) Raman spectroscopy



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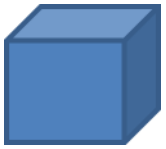


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Conclusion

There is no magic Solution

- Test Method
- Solution

- New Methods
- Smartphone spectrometer
- PhasmaFood
- Big Data and Modeling
- Direct Analysis by Consumers

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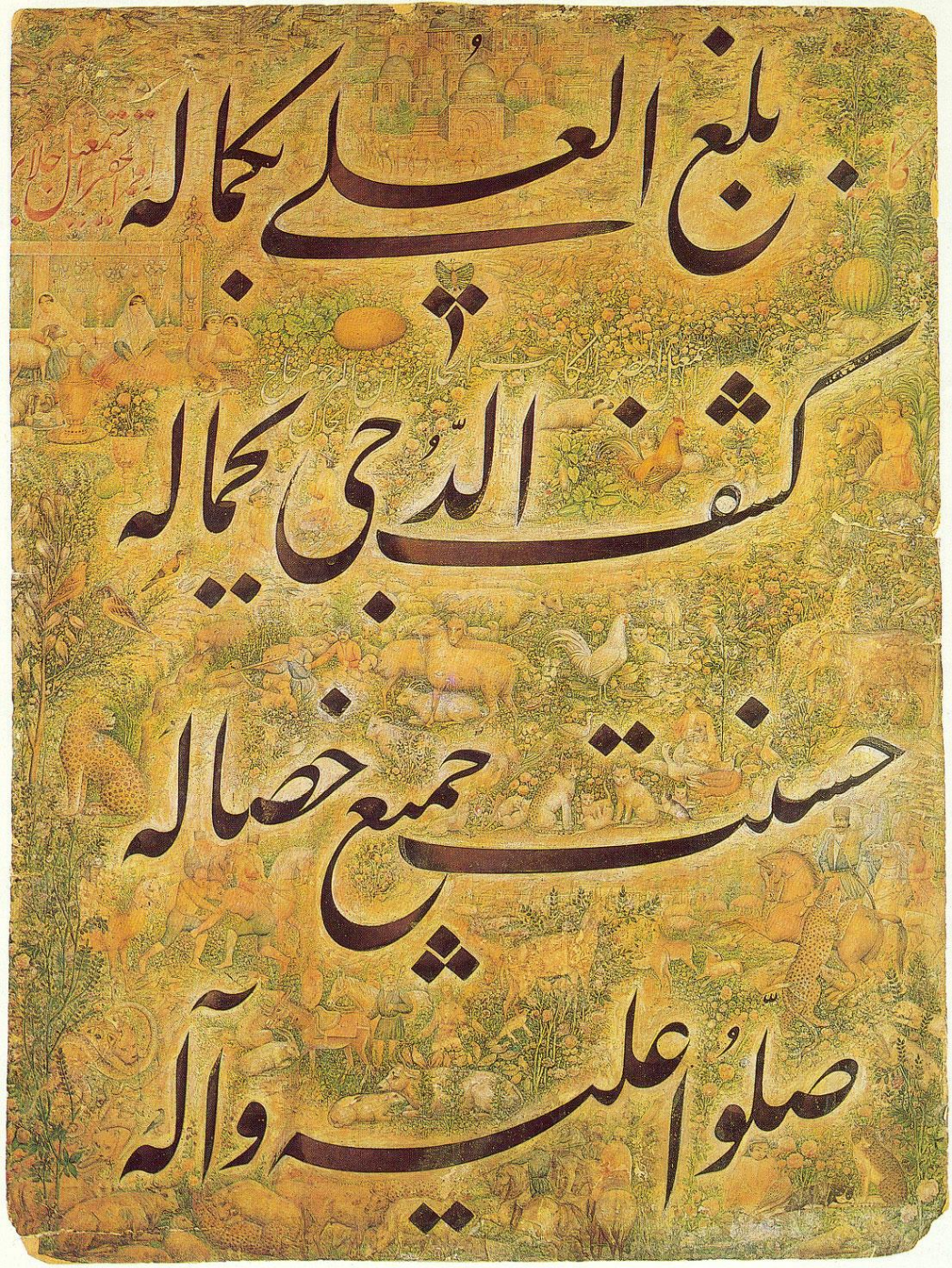
- His perfection procured exaltation
- His beauty dispelled the darkness
- All his attributes were good ones
- Pray for him, and for his family



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Allah Knows Best

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