IAEA 60 Years
Atoms for Peace and Development

Organic and Halal food authentication

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ISO Fd Food ERA Chair

Exploratory workshop:
Isotopic techniques in food characterisation

December 7-9, 2016 | Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia

Joint FAO/IAEA Programme in Food and Agriculture

IAEA 60 Years Atoms for Peace and Development

Atomic energy for peace and development

Sustainable agricultural development, improved nutrition and food security

the peaceful application of Nuclear techniques in Food and Agriculture (NAFA)

What we do in the FEPL

Vet. drug residues

Pesticide residues

Traceability Authenticity

Applied research

Participate EU Projects

Inter-comparison Exercises

Collaborate w Research Institu Equip. Manufact

Coordinate Proj

Develop standards (CODEX)

Committees (CODEX)

Advice

Technical cooperation

Raising Awareness

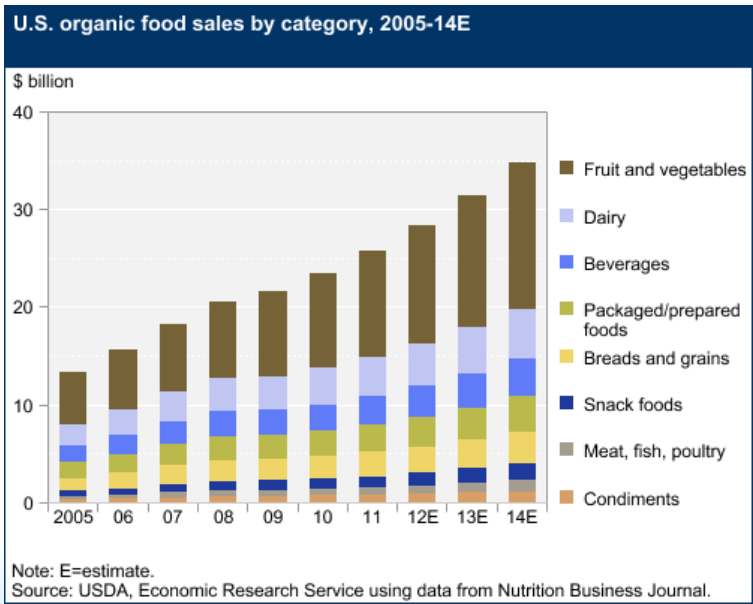
Training (National & Regional)

Organsing Fellowships

ternships

The World of Organic Agriculture 2016 Figures

- **Growth continues: Global organic market at 80 billion US Dollars per year with 43.7 million hectares of organic agricultural land worldwide.**
- **Organic agriculture is practiced in 172 countries by approximately 2.3 million farmers.**



The role of the FAO in Organic agriculture in developing countries

STUDIES

FAO helps developing countries gain access to international markets by providing technical information on production requirements and trade information on market opportunities.

Guidelines in progress include:

- Organic Agriculture in Senegal;
- Organic Pastures and Feed;
- Comparative Study of Different Organic Fertilizers, Bio-Fertilizers and Water Retaining Products; and
- Technical Guidelines on Conservation and Processing of Organic Fruits and Vegetables; Guidelines on Organic Horticulture Production.

Organic Food Fraud?

- Economic motivation is clear…
- Price differentials vary between a few percent for some fruit and vegetable to 100s of percent for processed foods such as peanut butter and drinks such as wine and beer. USDA ERS figures derived an average 30% premium for 37 fruits & veg.
- The potential breadth of fraud is large from ‘farmers-markets’ to ‘organised crime’

Mail Online

Company director jailed for re-selling cheap supermarket food as expensive organic produce

By Daily Mail Reporter
Last updated at 6:27 PM on 23rd September 2009

The director of a food company which provided pies to an upmarket
jailed for re-packaging cheap supermarket goods as organic.

Neil Stansfield, 51, bought food from non-organic sources including
netting £500,000 in his six-year fraud operation.

His firm Onefood - which stood for 'Organic, Natural and Ethical'- sold a £20 Waitrose sal
bought from a local butcher for £1.30 were sold for £2.50.

Non-organic: Food from Onefood - who repackaged supermarket food and sold it as organic - could have ended up on the dinner table at Buckingham Palace

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Relatively few reported cases...

a) Not much fraud going on?

b) Not detecting it?

Organised Organic food crime

Malta placed at centre of huge organic products fraud

Tuesday, 12 April 2016, 14:30 Last update: about 9 months ago

"investigation being carried out by the prosecutor of Verona, which it says is the largest investigation in Italy. Farmers, businessmen and certification companies were passing non-organic products into the market as organic. This amounts to fraud of around…"

Adulterated Organic fertiliser

THE FBI FEDERAL BUREAU OF INVESTIGATION

San Francisco Division

Home • San Francisco • Press Releases • 2012 • Former President of Organic Fertilizer Company Pleads Guilty to Fraud in Conn

Former President of Organic Fertilizer Company Pleads Guilty to Fraud in Connection with Selling Synthetic Fertilizer to Organic Farms

Company Grossed Millions Based on Six Years of Fraud

U.S. Attorney's Office
February 28, 2012

Northern District of California
(415) 436-7200

"Company grossed millions based on 6 years of fraud"

THE FBI FEDERAL BUREAU OF INVESTIGATION

Sacramento Division

Home • Sacramento • Press Releases • 2012 • Owner of Kern County Fertilizer Business Sentenced for Organic Fertilizer Fraud

Owner of Kern County Fertilizer Business Sentenced for Organic Fertilizer Fraud

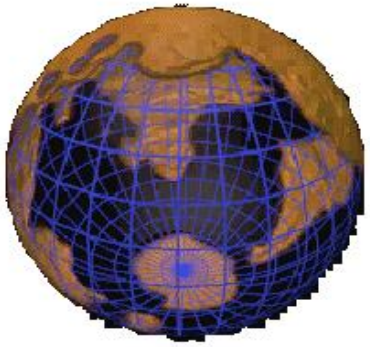
U.S. Attorney's Office
November 19, 2012

Eastern District of California
(916) 554-2700

Is it Organic?

"You may not feel any healthier right away, but you'll definitely feel more smug."

‘Points’ of concern…



Equivalency

IFOAM
ACOS (UK)
NOP (USDA-US)
(EC) No 834/2007
COR (Canadian)
JAS (Japan)

Non-segregated processing and packaging lines. Commingling, substitution (economic fraud)

Fertilisers, pesticides, herbicides, seeds

Production Farm

Cooperative storage

Wholesaler

Distribution Centre

Retail outlet

Consumer

Organic Farming

- Organic farming severely restricts the use of artificial chemical fertilisers and pesticides.
- Organic farmers rely on developing a healthy, fertile soil and growing a mixture of crops.
- Management practices which sustain soil health and fertility.
- The use of natural methods of pest, disease and weed control.
- High standards of animal welfare.
- Low levels of environmental pollution.
- Enhancement of the landscape, wildlife and wildlife habitat.
- The prohibition of all genetically engineered food and products.

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Stable isotope analysis

“Can nitrogen stable isotope analysis help us detect conventional crops that have been labelled as Organic?”

The natural abundance of Nitrogen stable isotopes

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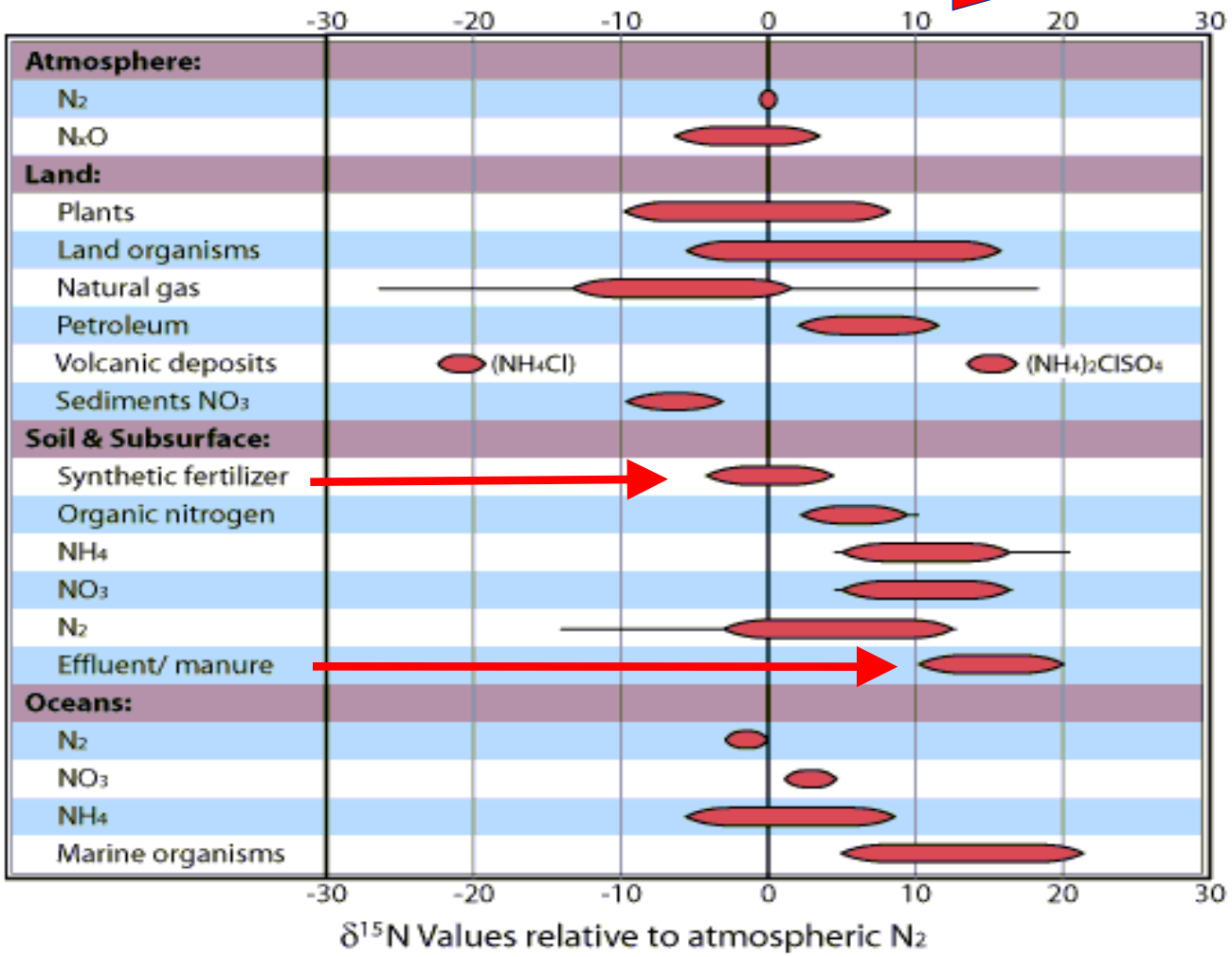
^{14}N	^{15}N
14.00307	15.0001
99.63%	0.37%
Stable	Stable

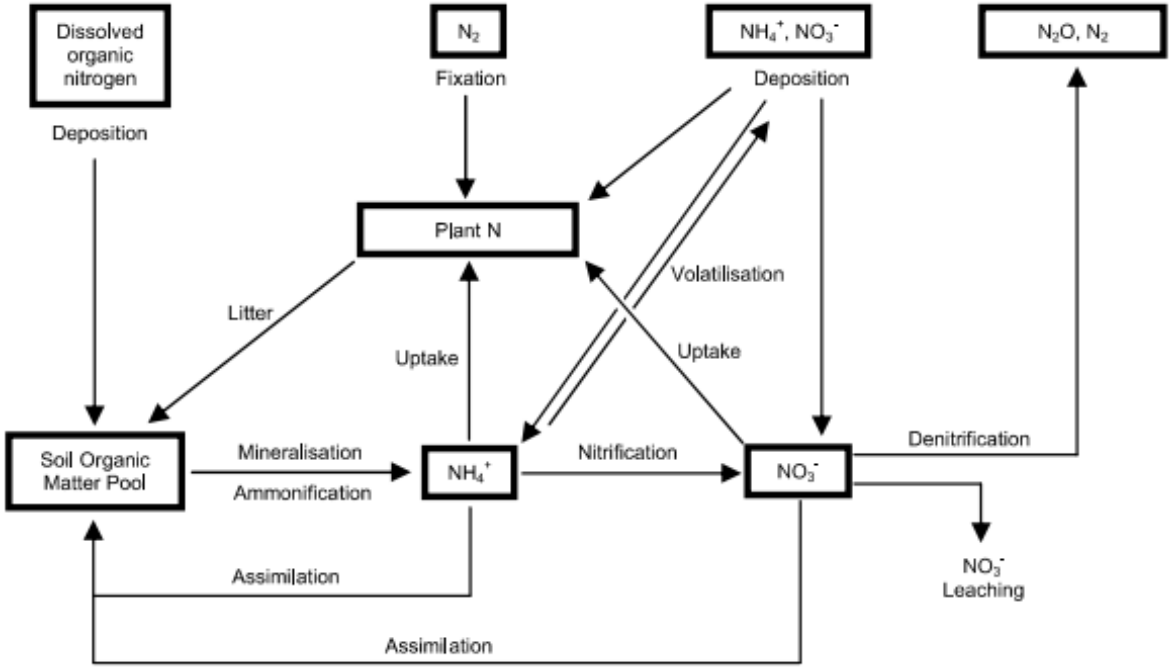
Increasing ^{15}N content



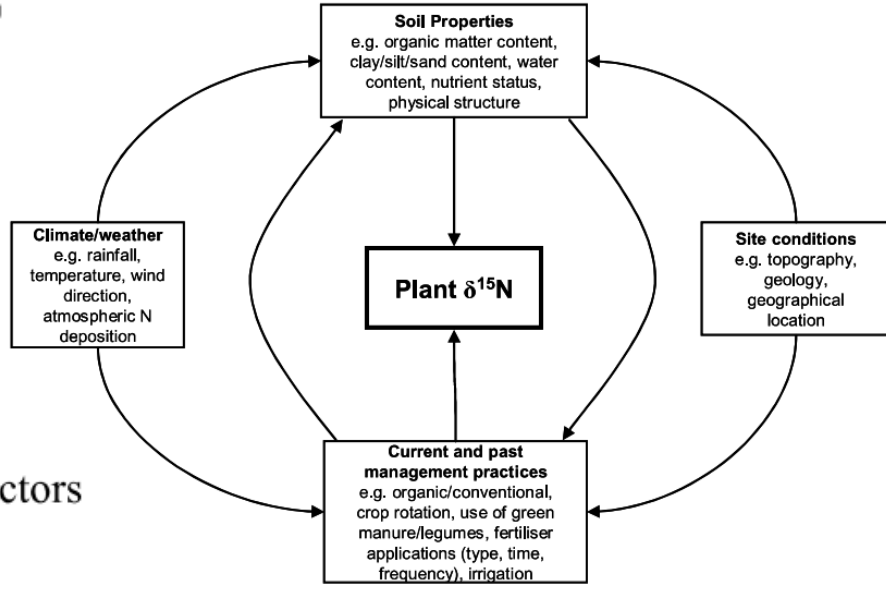
FROM: SAHRA

http://www.sahra.arizona.edu/programs/isotopes/nitrogen.html

Nitrogen cycles are not simple…

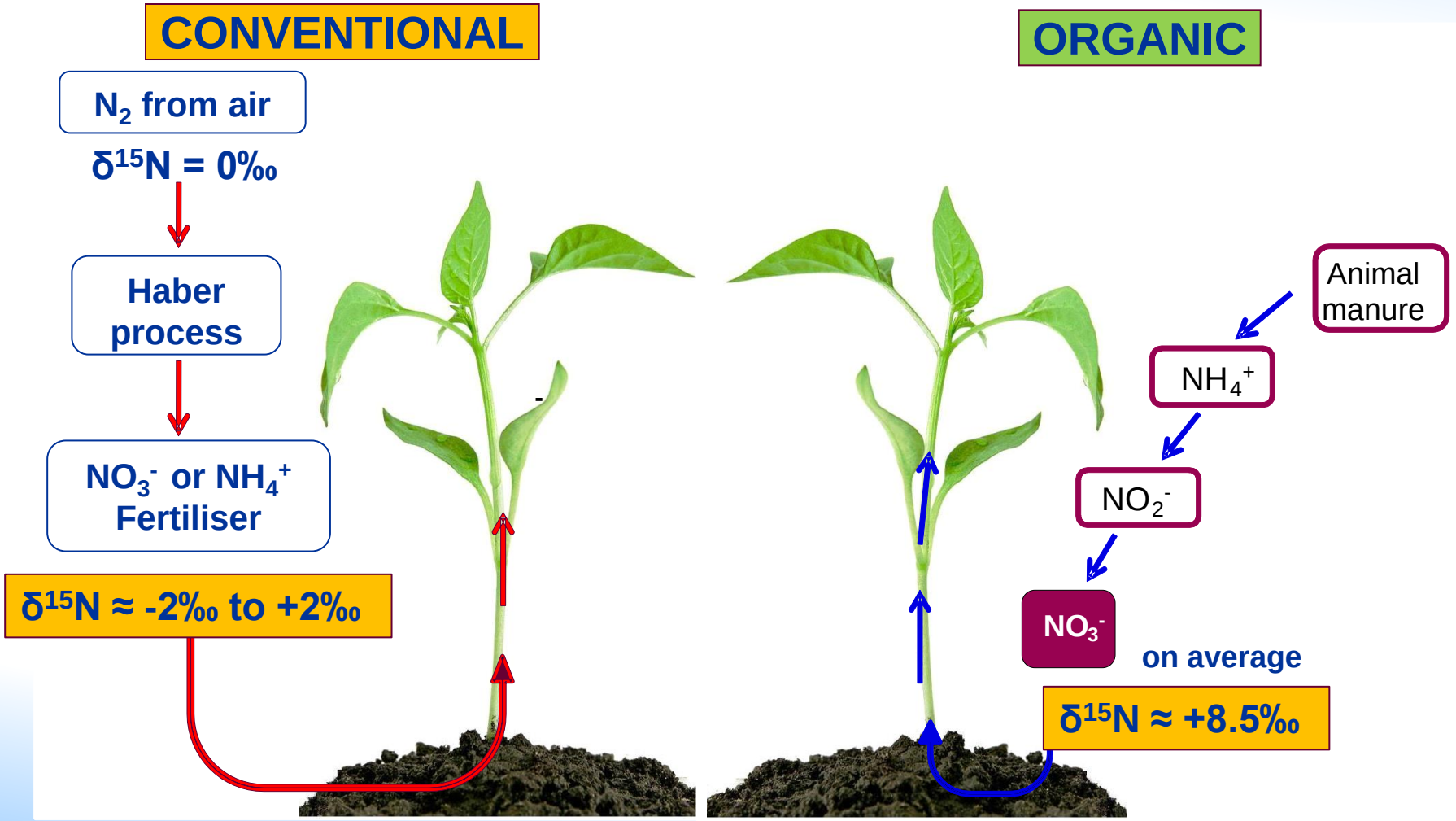


Schematic diagram of the nitrogen cycle showing principle reservoirs and pathways of nitrogen (adapted from Kendall, 1998)

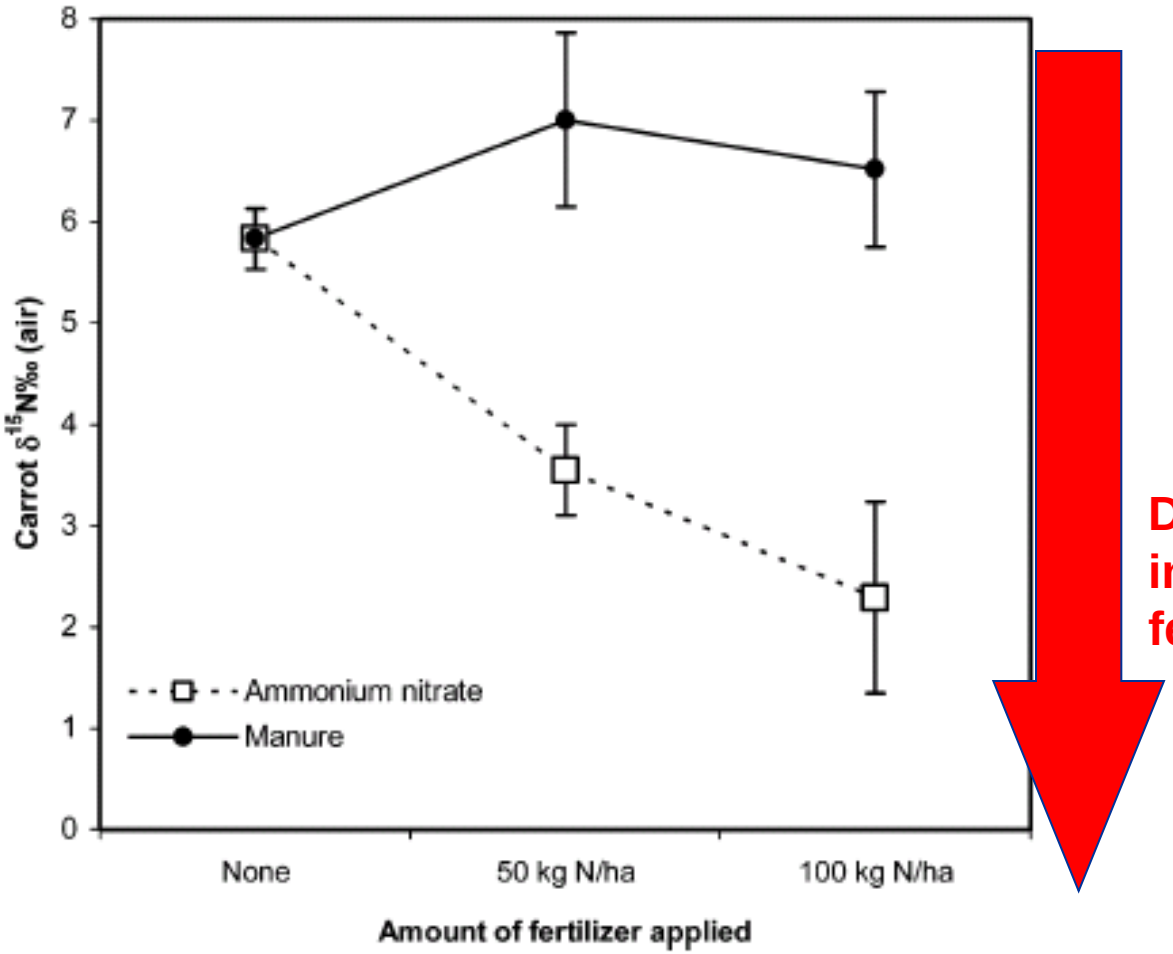


Simplified schematic diagram showing the principal factors which may influence plant nitrogen isotope values

Rationale for measuring nitrogen isotopes in plants to confirm Organic production



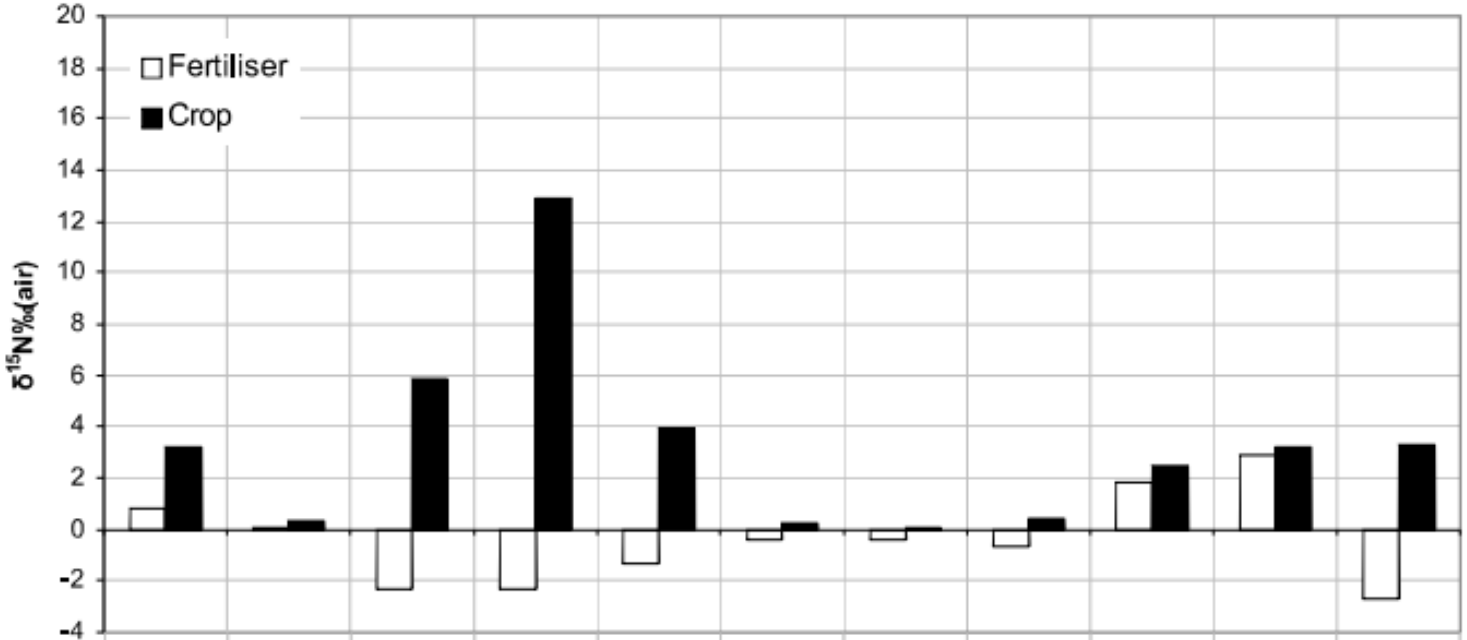
Controlled cultivation ‘proof of concept’ experiments



A S Bateman, S D Kelly & T D Jickells (2005) *“Nitrogen Isotope Relationships between Crops and Fertilizer: Implications for Using Nitrogen Isotope Analysis as an Indicator of Agricultural Regime”* ***Journal of Agricultural and Food Chemistry, 53(14); 5760-5765.***

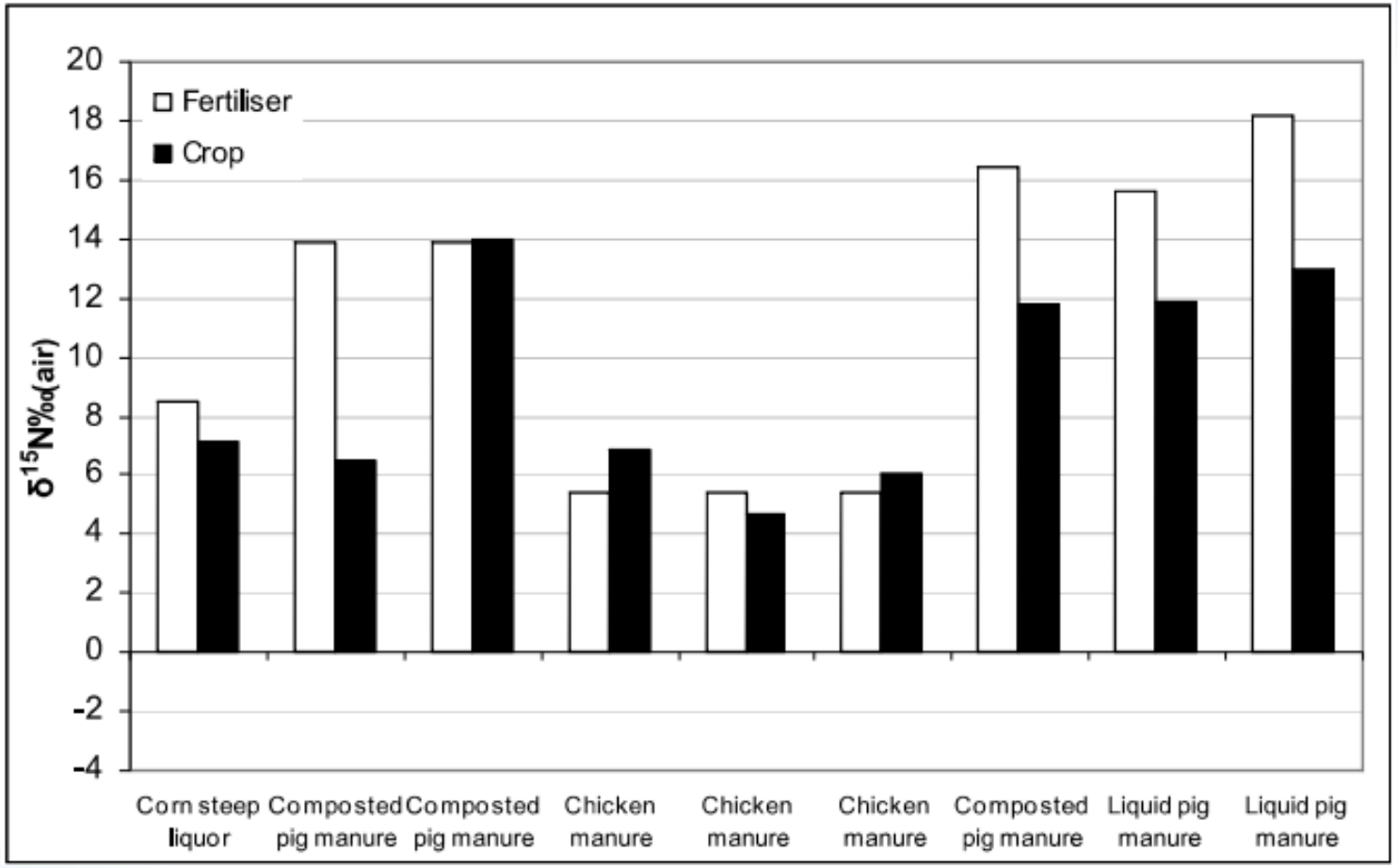
Decreasing ^{15}N content with increasing synthetic fertiliser application

‘pot or glass house experiments’ – synthetic fertiliser

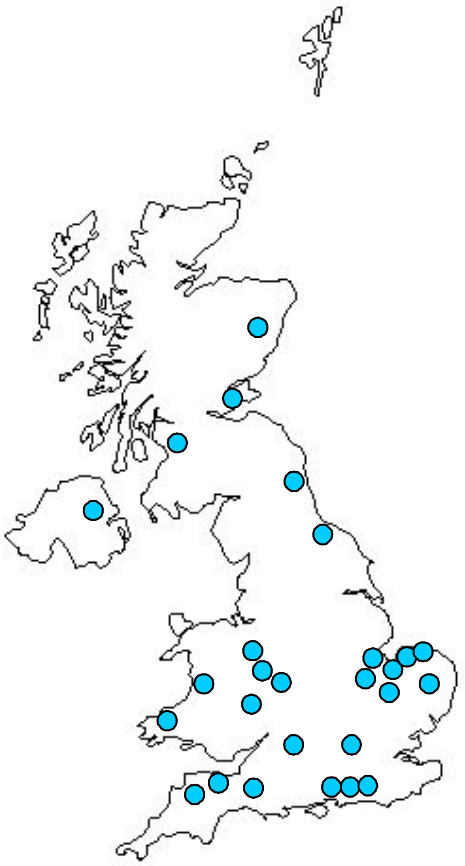


Fertiliser	Ammonium nitrate	n/a	Urea	Urea	Ammonium nitrate	Urea + ammonium phosphate	Urea + ammonium phosphate	Urea	Potassium nitrate	Ammonium chloride	Urea
Timing of application	Most applied at start, some at 50 days	Continuous	Single application at start	Single application at start	Single application at 30 days	5 application throughout growing period	3 application throughout growing period	Single application	Continuous	Continuous	Basal dressing at 10 days and 2nd application at 40 days
Type of application	Basal dressing, granular	Drip feed	Solution	Solution	Solution	Solution	Solution	Basal dressing	Solution	Solution	Basal dressing and then solution at 40 days
Crop	Tomato	Tomato	Maize	Maize	Carrot	Tomato	Lettuce	Chinese cabbage	Tomato plant	Tomato plant	Chinese cabbage
Reference	Nakano et al., 2003	Nakano et al., 2003	Choi et al., 2001	Choi et al., 2002	Bateman et al., 2005	Bateman et al., 2005	Bateman et al., 2005	Yun et al., 2006	Evans et al., 1996	Evans et al., 1996	Lim et al., 2007

‘pot experiments’ – manure



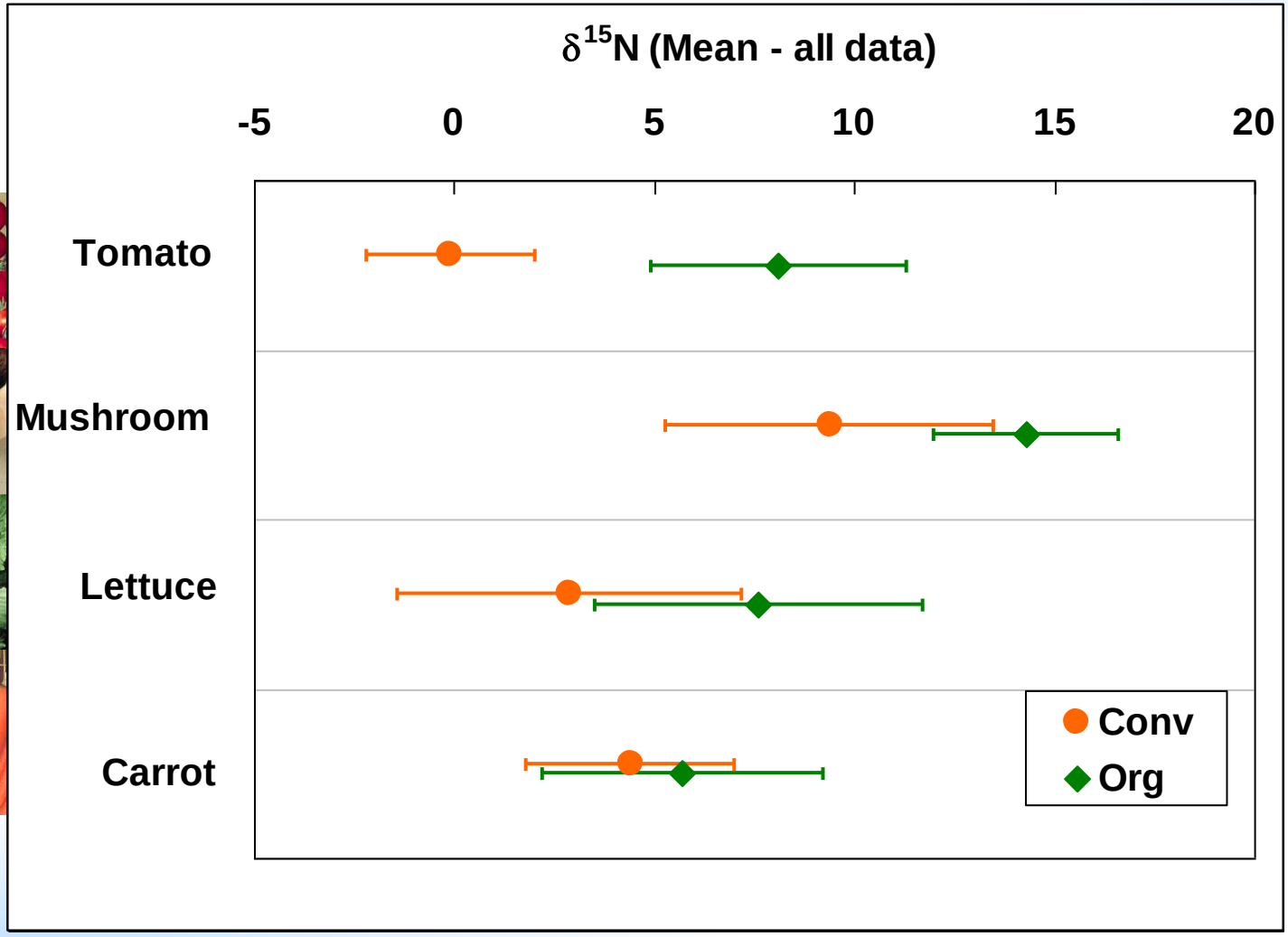
UK Baseline ‘shopping basket’ survey from demo. farms



- Soil type
- Weather
- Time of harvest
- Type and $\delta^{15}N$ of synthetic fertiliser
- Type and $\delta^{15}N$ of fertiliser applied by organic grower

+ some samples from Italy, Germany, Spain & Holland

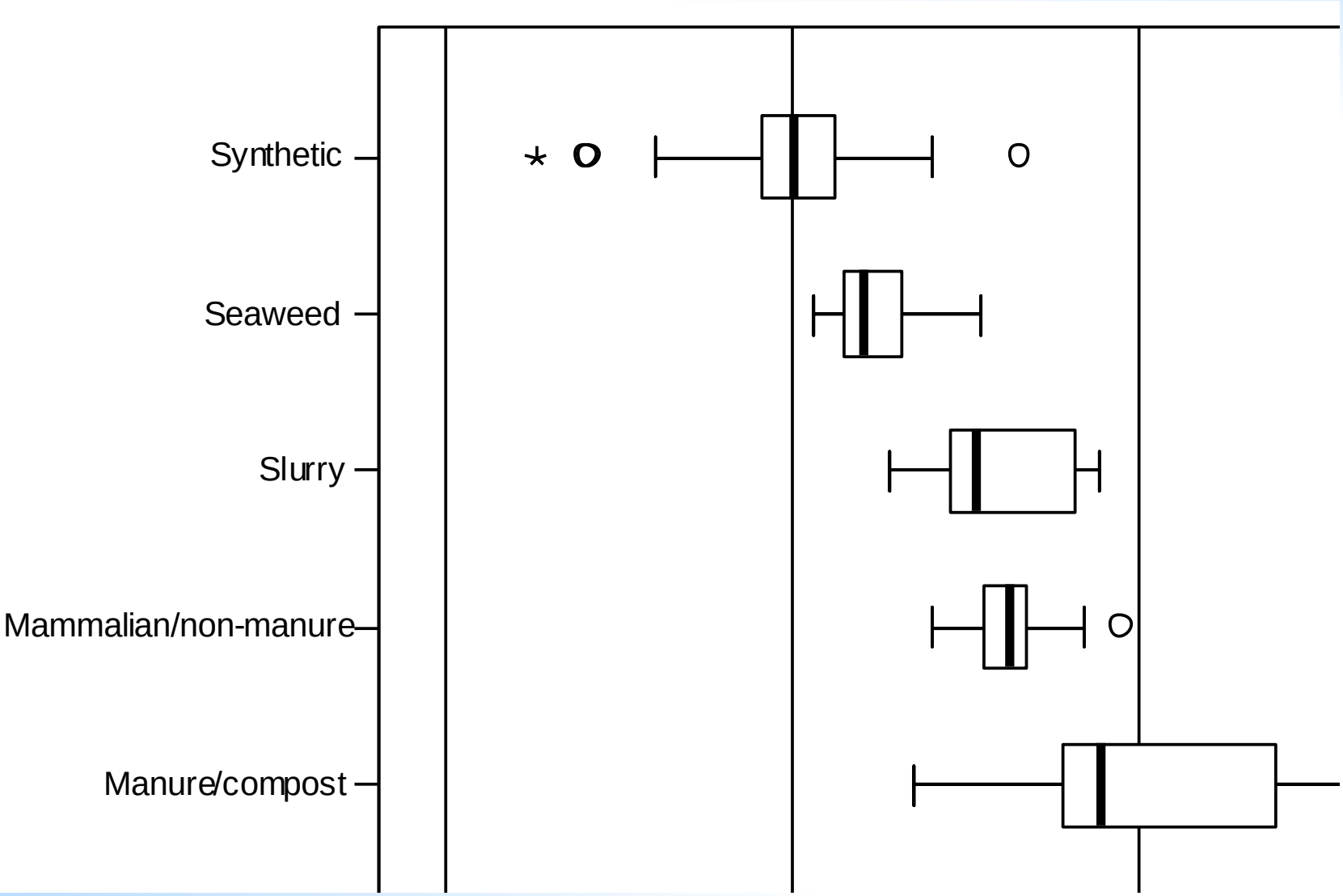
Baseline ‘shopping basket’ survey - Summary data



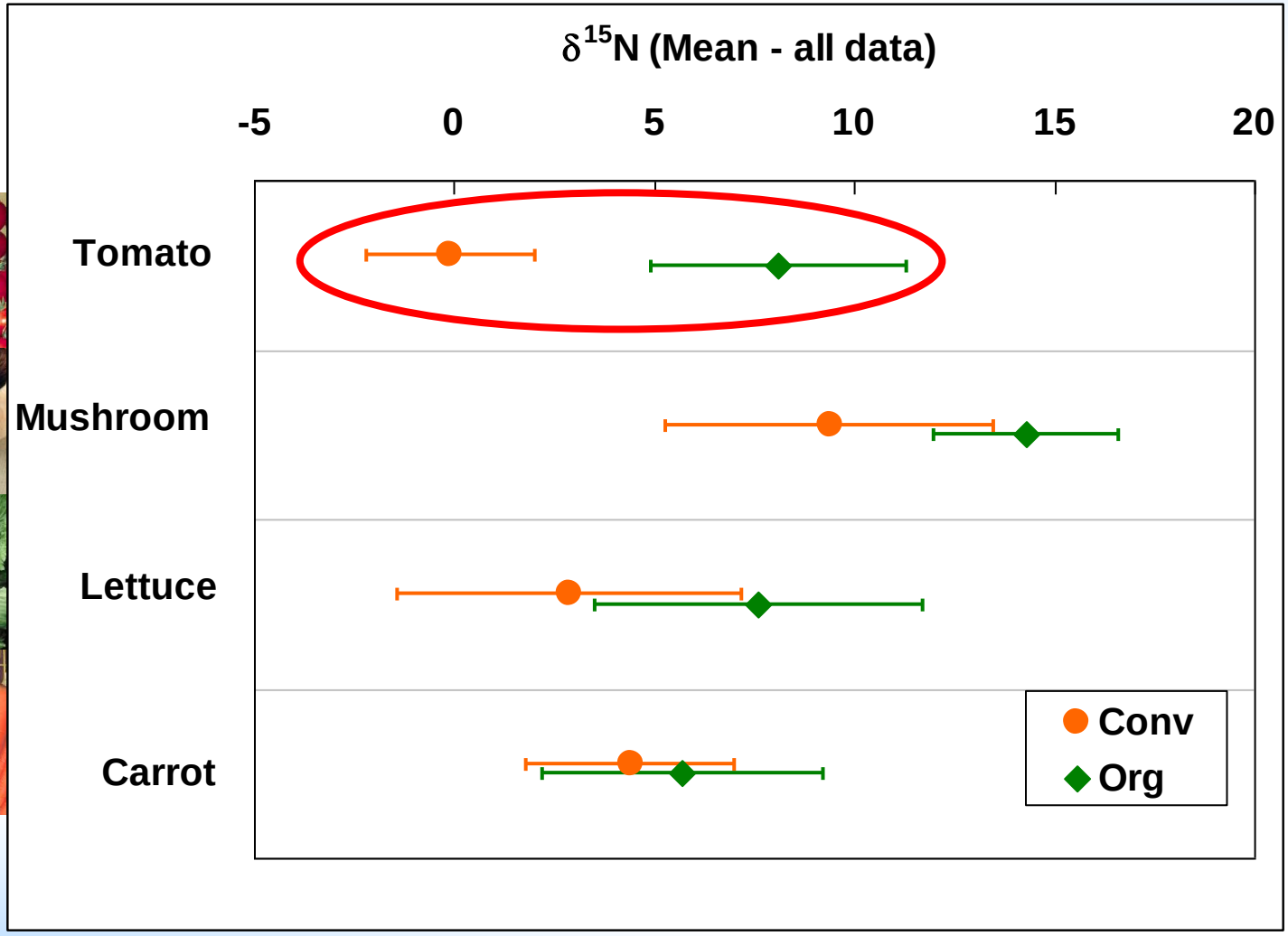
n	
43	
61	
14	
11	
55	
49	
18	
13	

Nitrogen stable isotopes in Fertilisers

Bateman and Kelly (2007) Fertilizer nitrogen isotope signatures, *Isotopes in Environmental and Health Studies,* Vol. 43, No. 3, September 2007, 237–247

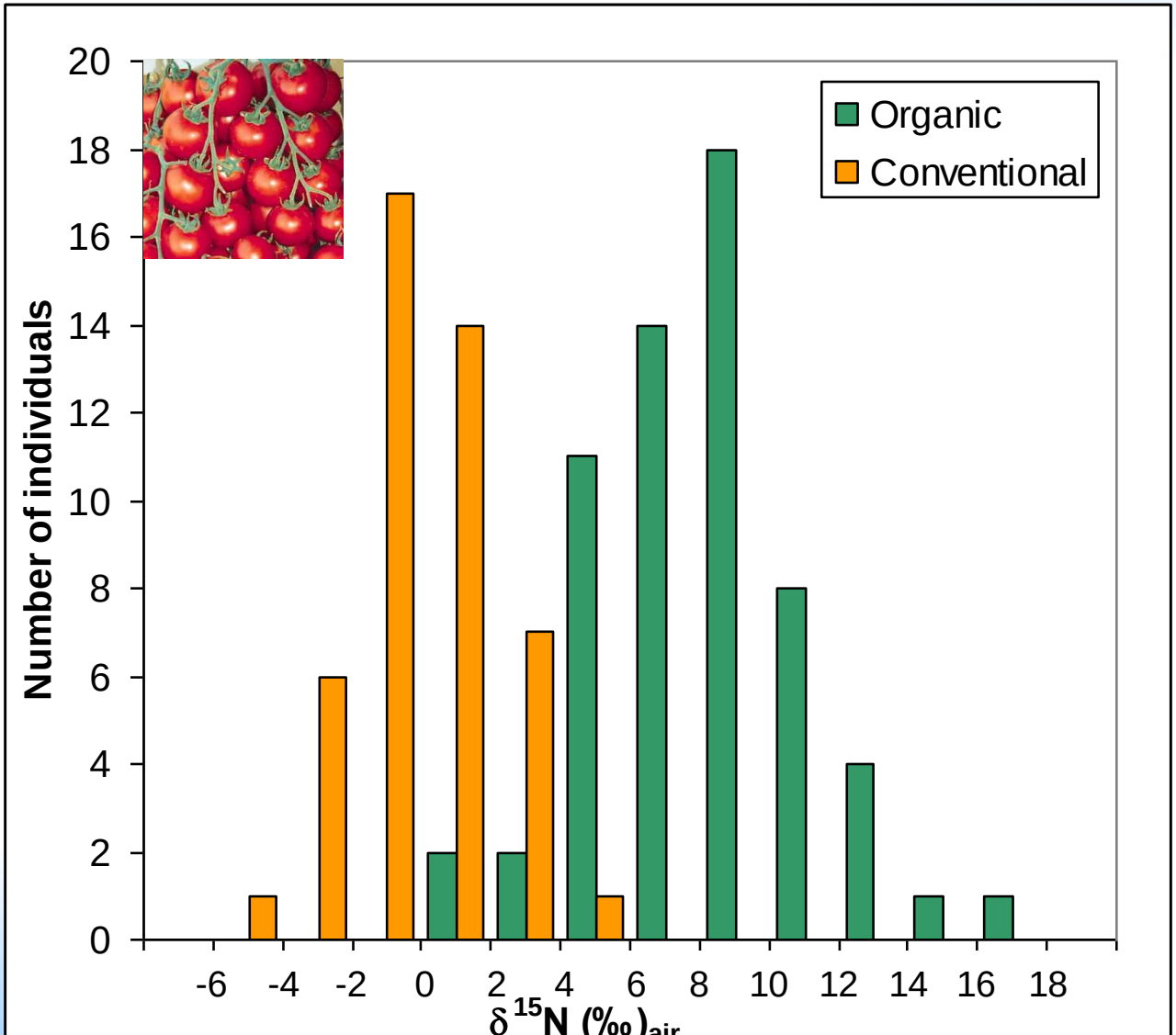


Baseline ‘shopping basket’ survey - Summary data

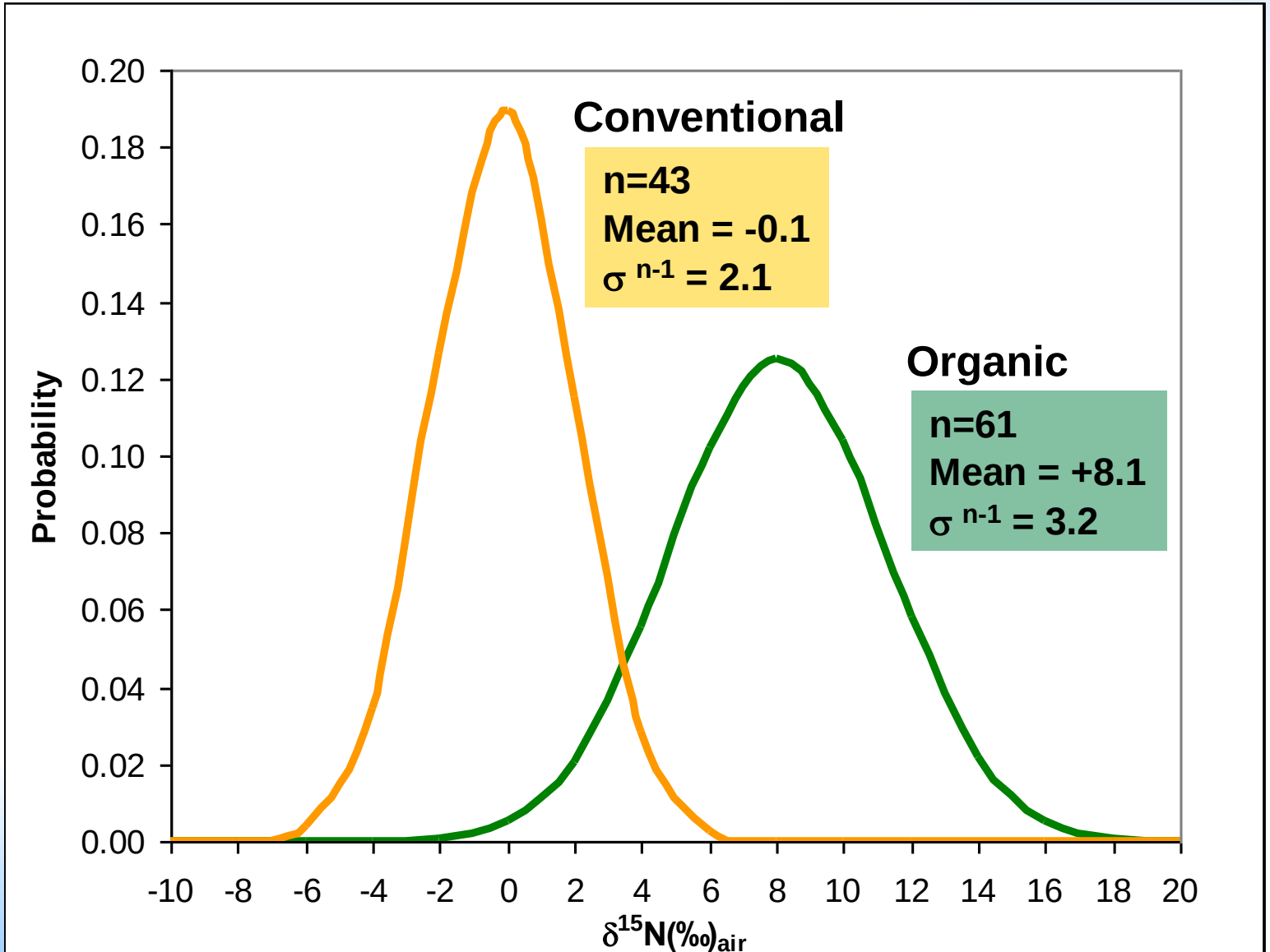


n	
43	
61	
14	
11	
55	
49	
18	
13	

Tomato total nitrogen isotope data



Tomatoes ^{15}N normal distribution



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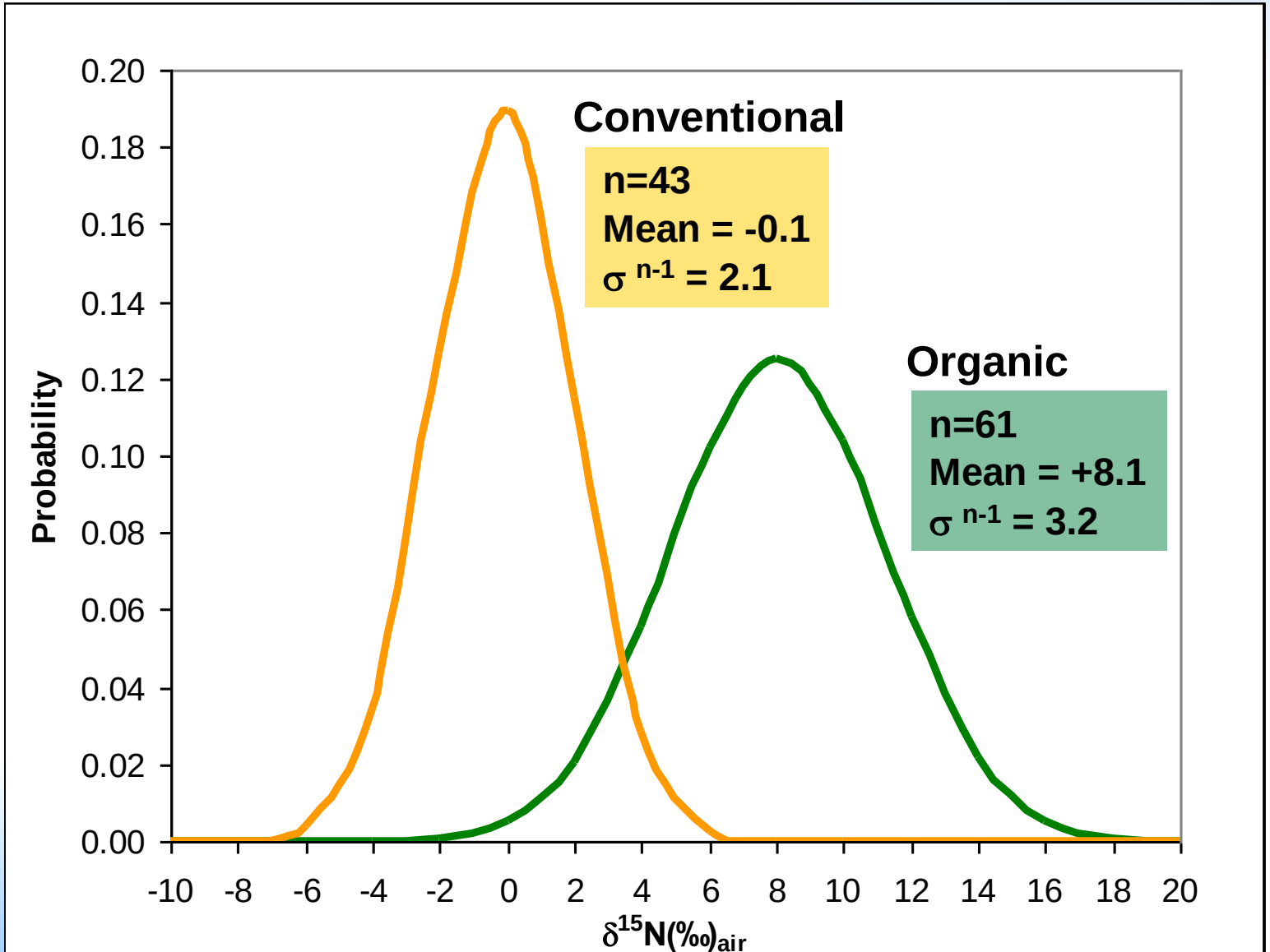

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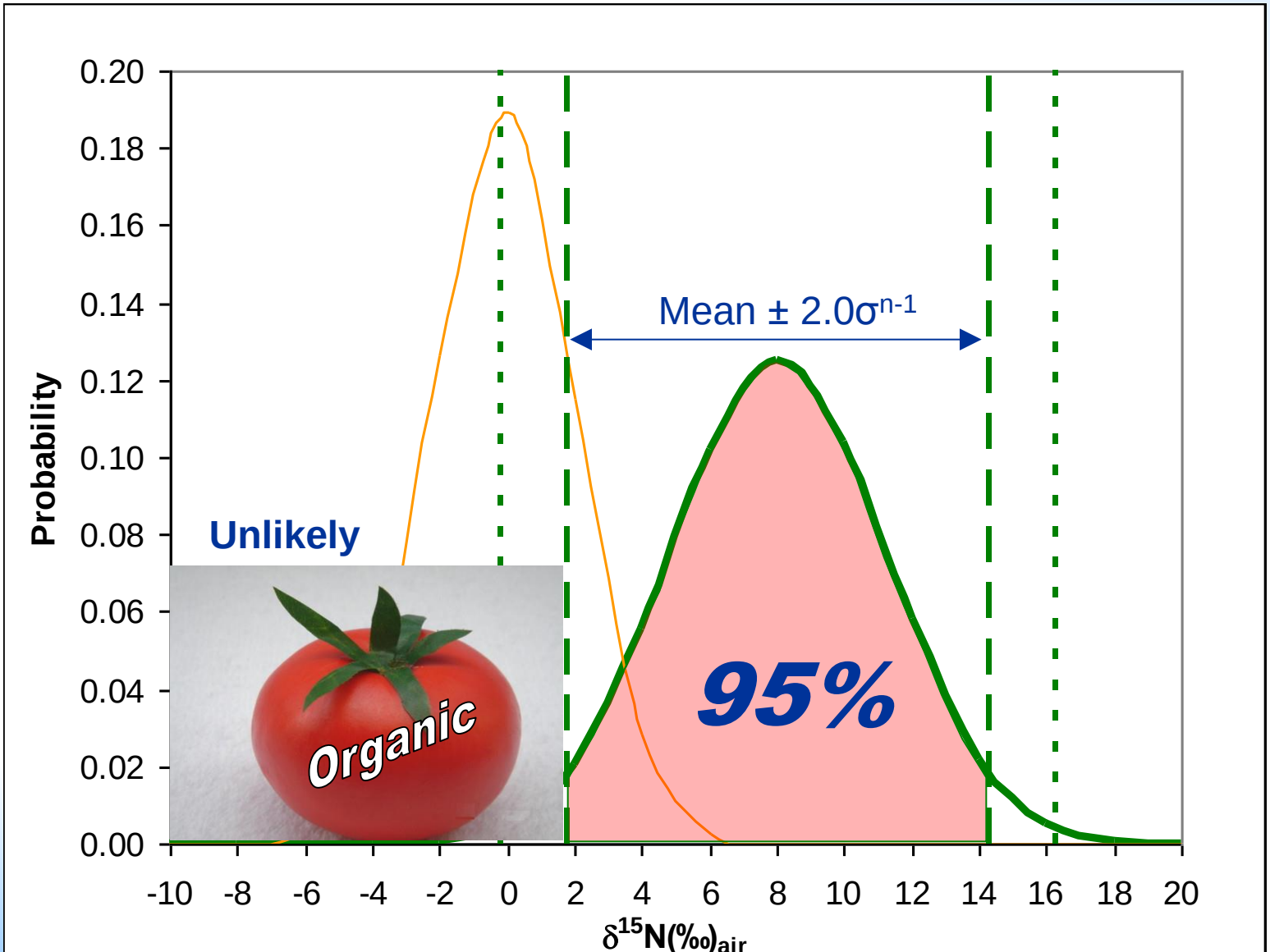
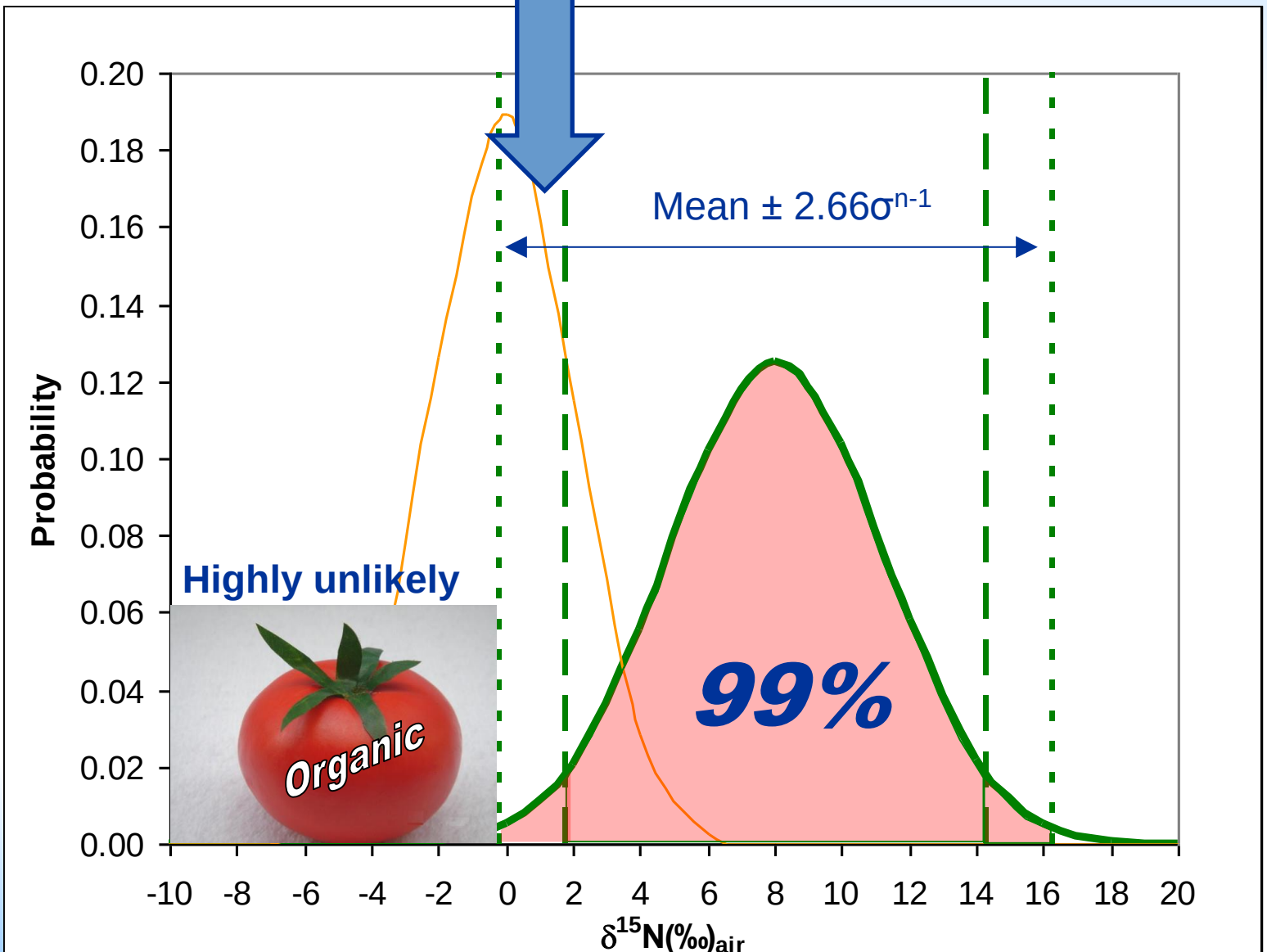


Tomat normal distribution

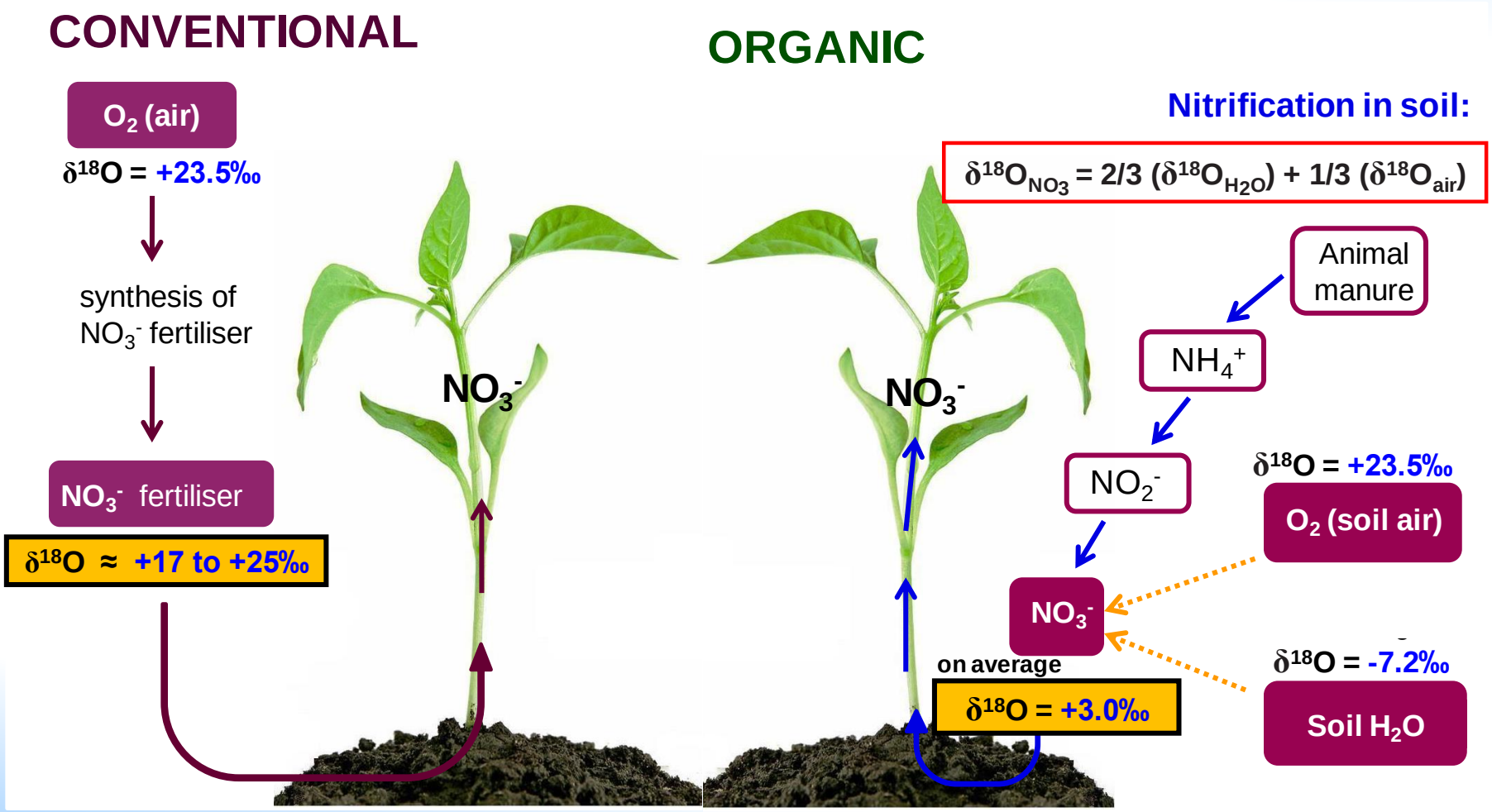
Cut-off value of 0‰ includes 50% of conventional Tomatoes leading to 'false negatives'.



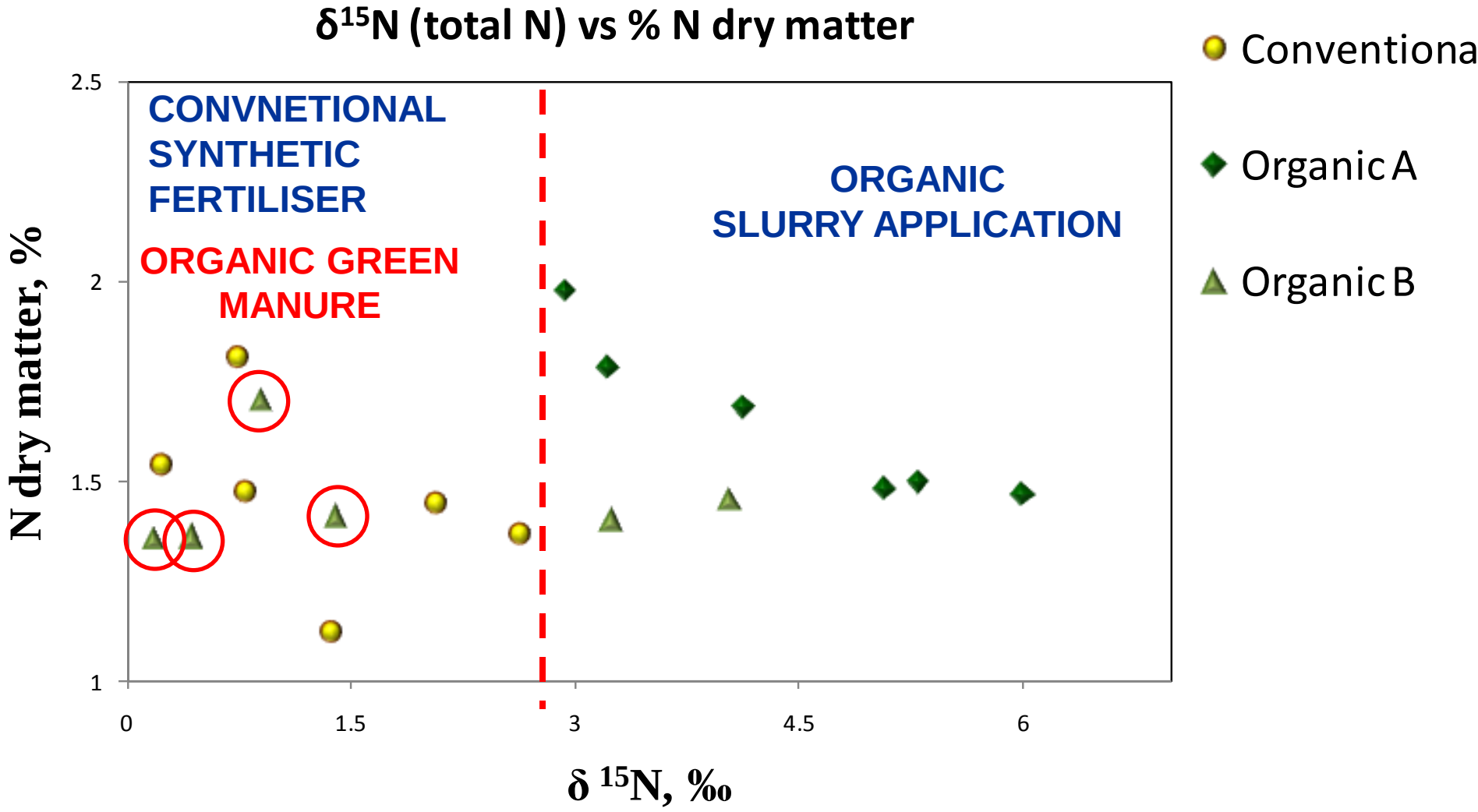
Stable isotope analysis

“Can oxygen compound-specific isotope analysis help us detect conventional crops that have been labelled as Organic?”

Rationale for measuring oxygen isotopes in extracted plant nitrate to confirm Organic production



Potatoes grown under conventional and Organic systems (OrgTrace)



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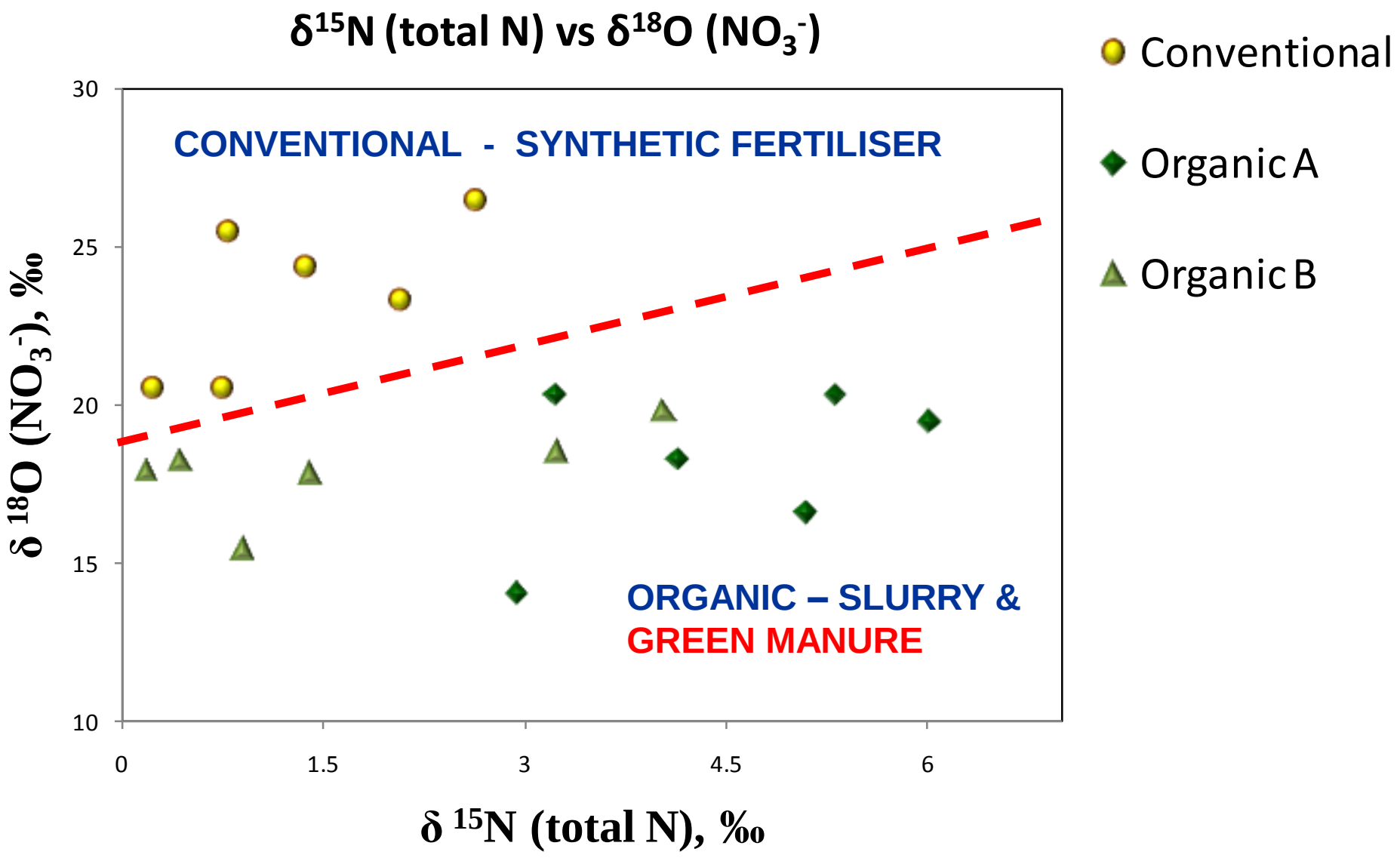

Organic Farming

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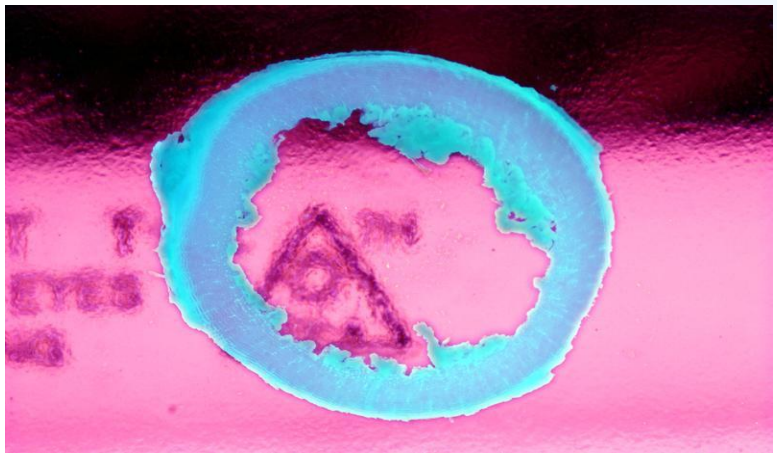
Fluorescence microscopy to detect the frequency of veterinary drug administration

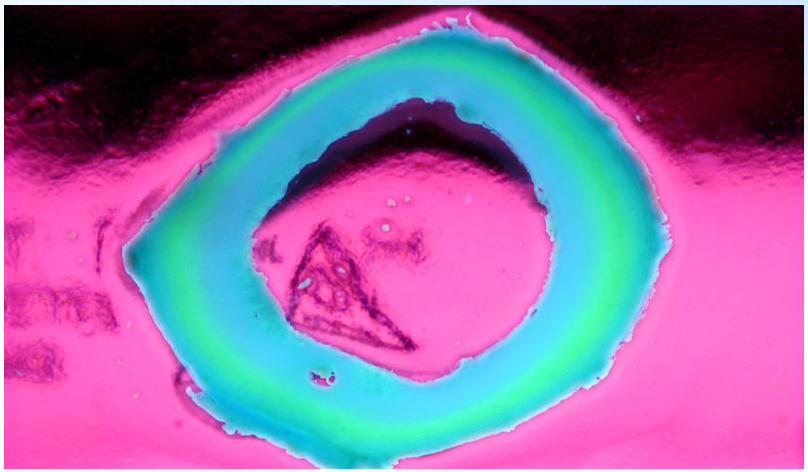
Fluorescence microscopy to detect the frequency of veterinary drug administration

- If animals receive more than three courses of treatment with antibiotics within one year (or more than one course of treatment if their productive lifecycle is less than 1 year) the livestock concerned, or produce derived from them, may not be sold as Organic
- Aim to determine deposition patterns for:
  - Tetracyclines in poultry and porcine bone
- Produce validated method(s) for verification that correct veterinary medicinal procedures have been used in Organic herds.

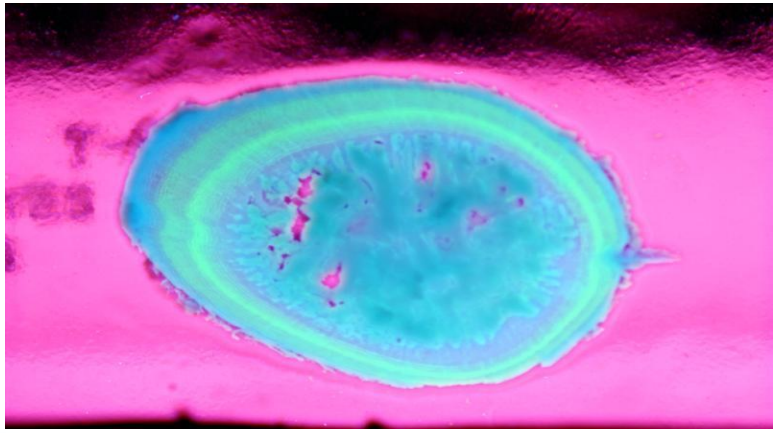
Tetracycline – Pig



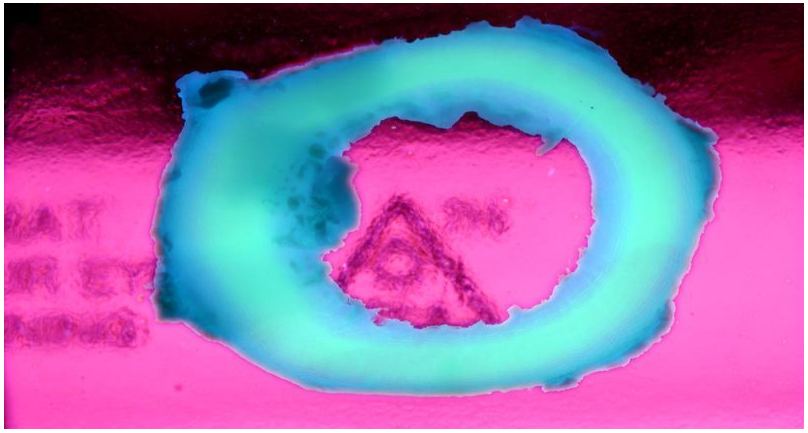
control



single dose

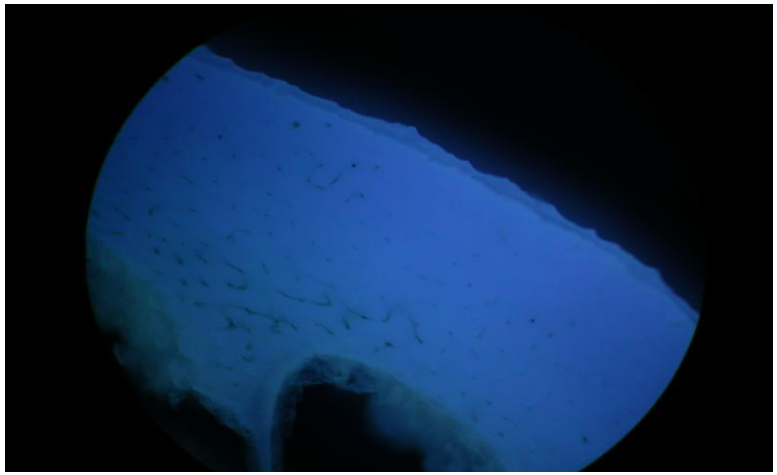


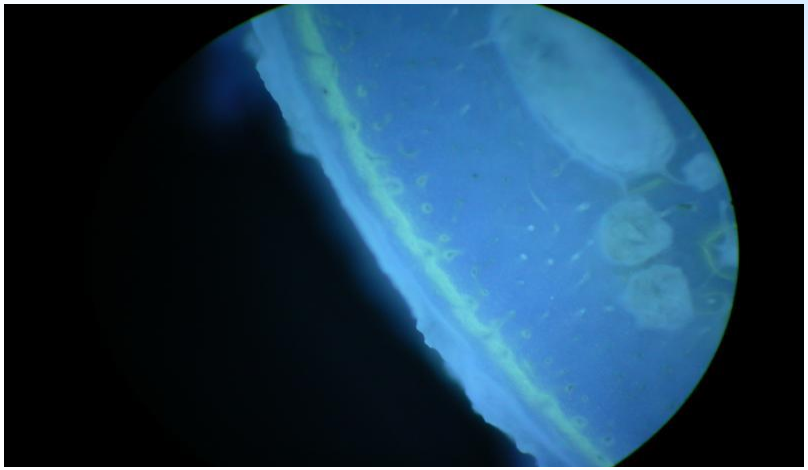
double dose



prophylactic dose

Tetracycline - Chicken





JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

J. Agric. Food Chem. **2006**, *54*, 1523–1529

Verification of Compliance with Organic Meat Production Standards by Detection of Permitted and Nonpermitted Uses of Veterinary Medicines (Tetracycline Antibiotics)

MITCHELL KELLY,* JONATHAN A. TARBIN, HELEN ASHWIN, AND MATTHEW SHARMAN

Central Science Laboratory, Sand Hutton, North Yorkshire YO41 1LZ, United Kingdom

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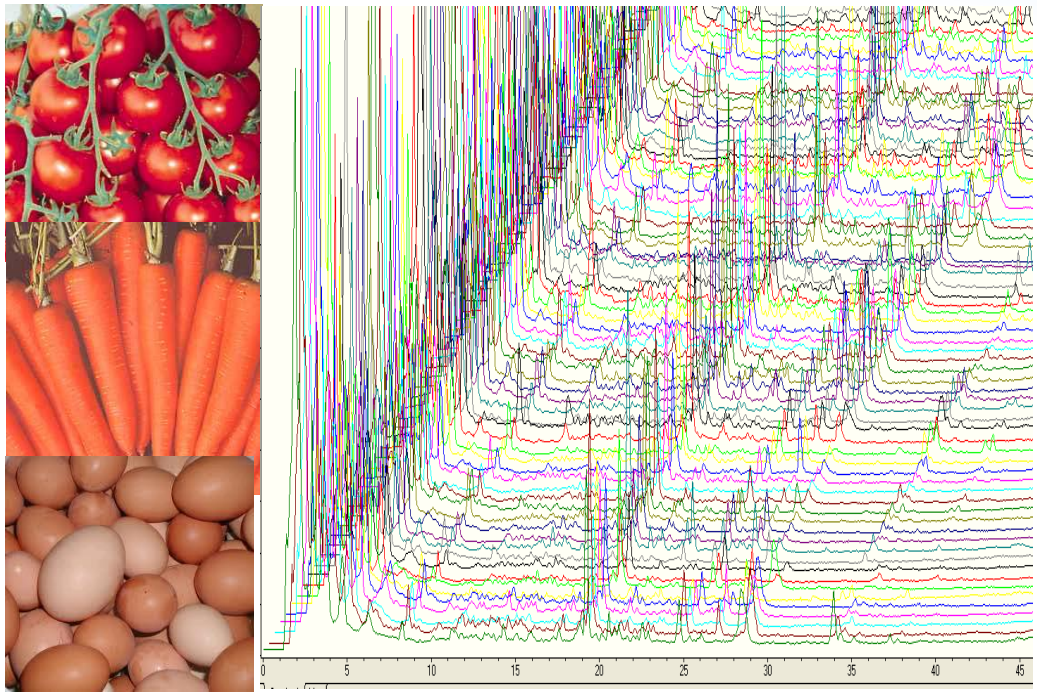
Are there real and consistent intrinsic differences in the chemical and mineral composition of organic and conventional crops? Can they be exploited to determine authenticity?

Nutritional quality of organic foods: a systematic review[1–4]

Alan D Dangour, Sakhi K Dodhia, Arabella Hayter, Elizabeth Allen, Karen Lock, and Ricardo Uauy

Conclusions: On the basis of a systematic review of studies of satisfactory quality, there is no evidence of a difference in nutrient quality between organically and conventionally produced foodstuffs. The small differences in nutrient content detected are biologically plausible and mostly relate to differences in production methods. *Am J Clin Nutr* 2009;90:680–5.

Metabolomics



Non-targeted LC/MS protocol

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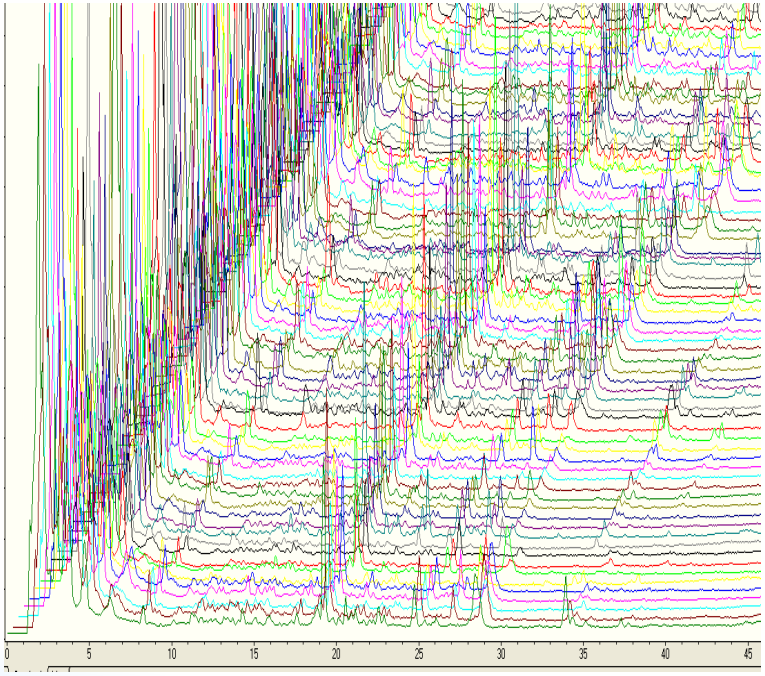
VG Quattro LC/MS Study-

TIC of methanolic extract of freeze-dried material



XCMS processing

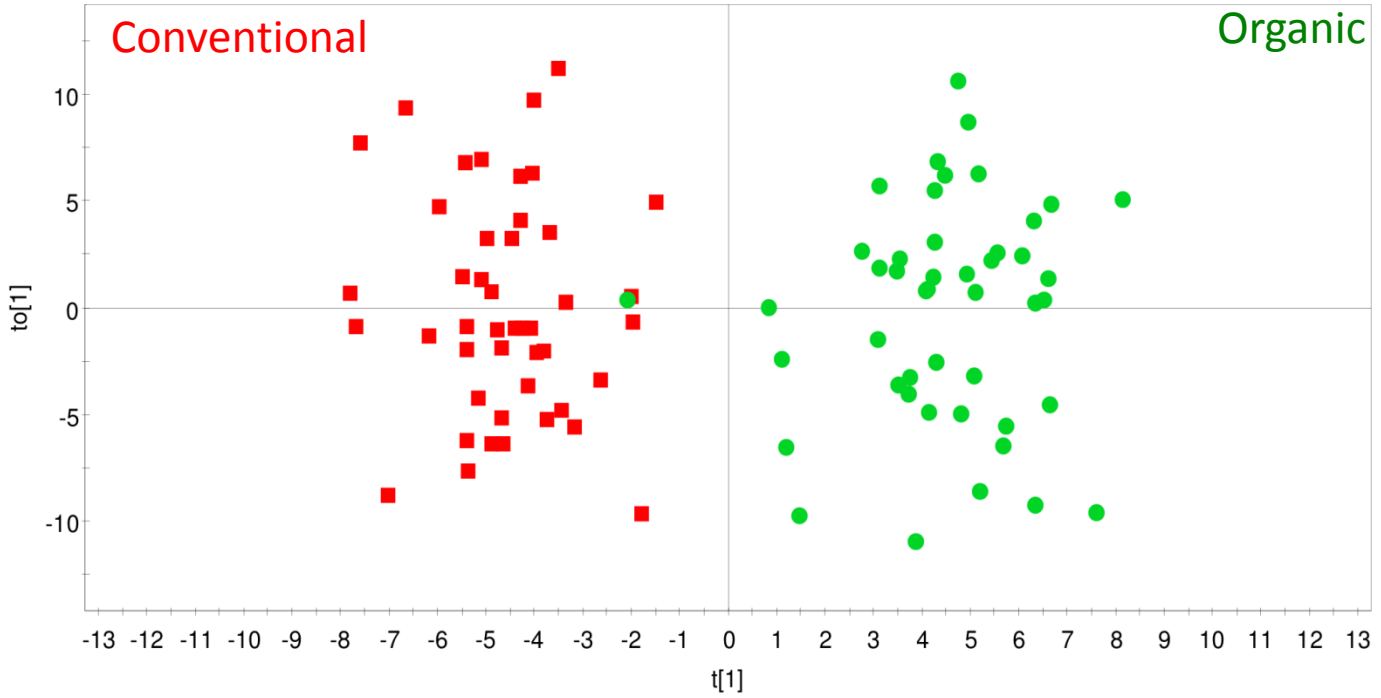
	name	tstat	pvalue	A17	Col01	C01	C08
1	M415T2852	-7.096437	2.12E-10	120.004708	2575.881696	3698.98387	2721.077107
2	M393T2932	-7.342163	5.37E-10	420.148201	6298.221588	490.003052	6909.045181
3	M171T119	-6.733164	1.12E-09	89933.4068	53878.66347	66538.1422	37236.45911
4	M135T85	-6.680006	1.46E-09	15813.4125	32997.22995	69140.509	0
5	M94T86	-7.188859	1.54E-09	0	517993.0997	293271.703	118577.2021
6	M291T205	-6.916524	2.55E-09	29472.6902	23526.6226	22296.4784	12165.22768
7	M420T798	-6.483831	4.10E-09	7803.08558	0	0	1633.277029
8	M140T190	-6.303088	1.19E-08	38695.6609	44293.41504	32856.5431	29282.65393
9	M103T83	-6.202845	1.67E-08	36166.71	57319.29039	104722.064	1608.29657
10	M177T172	-5.998163	3.43E-08	41216.5574	105577.9759	89461.9054	47582.90405
11	M430T732	-5.913502	5.01E-08	3704.86807	87.67103952	7824.08581	760.7770386
12	M453T204	-5.8784	1.23E-07	7644.16129	6285.166329	913.410479	7692.327354
13	M476T128	-5.67226	1.40E-07	28406.746	13236.46073	32329.8545	6119.925376
14	M177T134	-5.763137	1.80E-07	23331.7088	29276.8288	22538.8515	18570.5241

XCMS *software (http://metlin.scripps.edu/download/)*

OPLS-DA on tomato samples

Orthogonal Projections to Latent Structures Discriminant Analysis

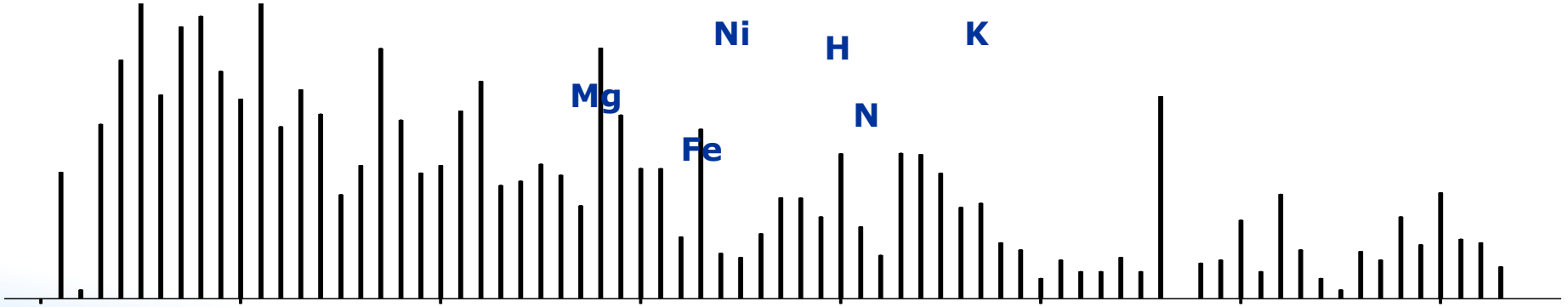
Model with 1 predictive LV and 3 orthogonal LVS (Q2 = 0.39, CV-ANOVA = 10^{-6})



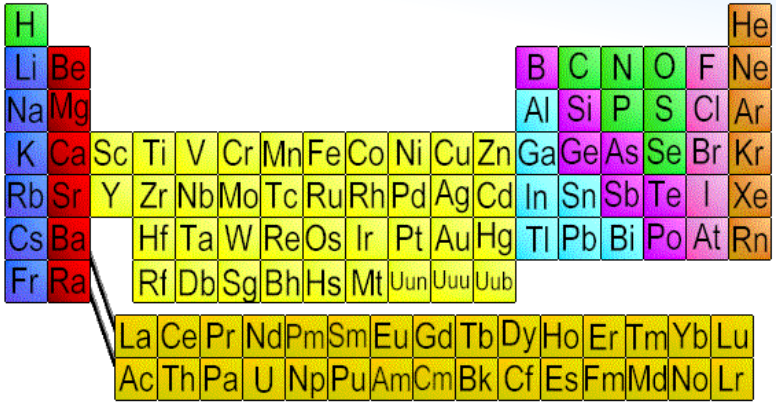
Organic tomatoes – elevated concentrations of:

+ Anti-oxidants : Chlorogenic acid, Coumaric and caffeic acid.
+ Natural pesticide: Tryptamine
+ AA: Gly, Ala, Leu, Tyr
+ Alpha-tomatidine

Elemental fingerprinting by ICP-MS

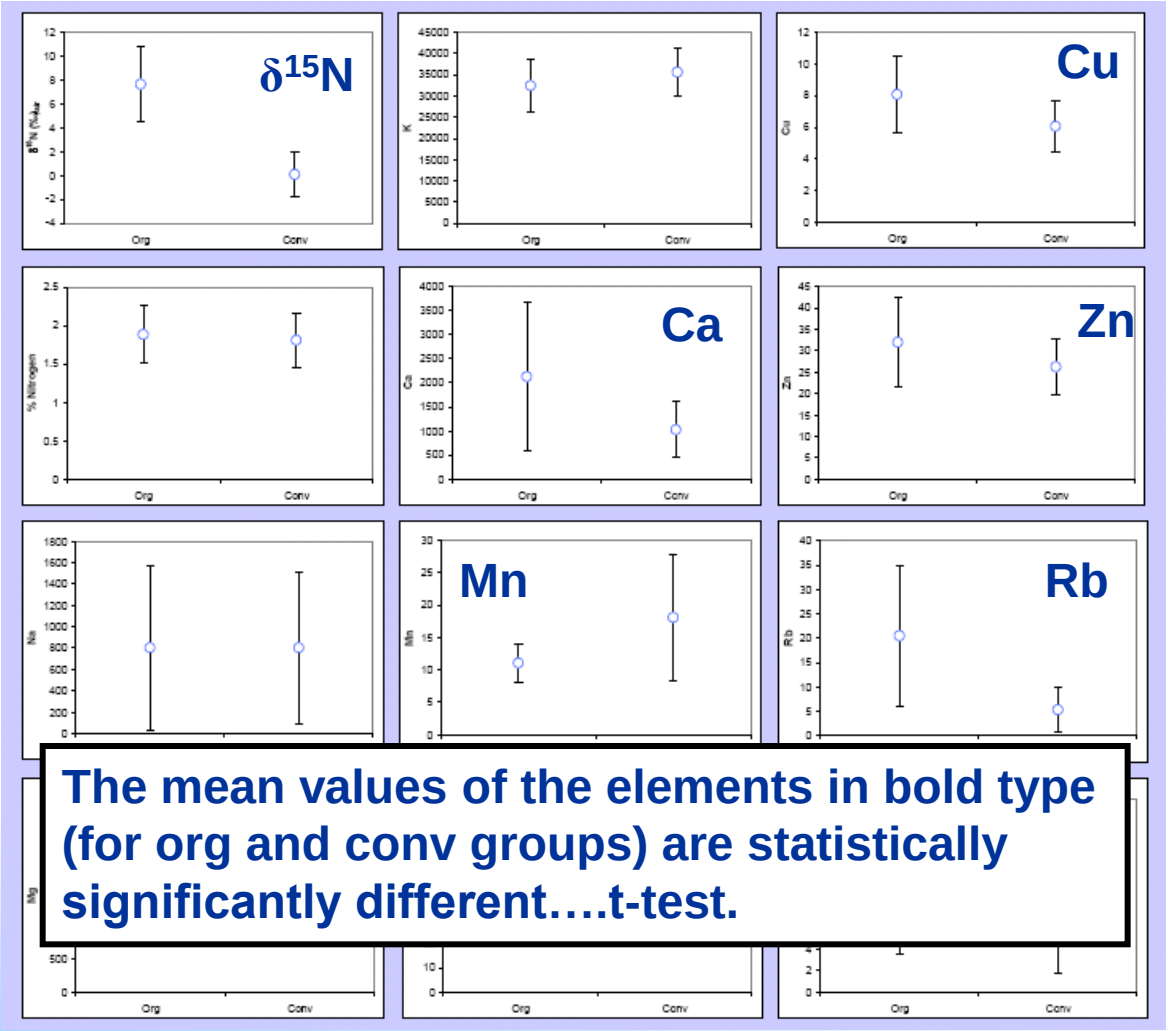


Multi-element signatures by ICP



- 55 Element scan - measure macro, micro and trace elements simultaneously by ICP-MS (Na, Mg, K, Ca, Mn, Fe, Cu, Zn, Rb, Sr, Cd, Ba)
- Identify key variables that separate organic and conventional crops using supervised pattern recognition
- We did not have '*a priori*' hypothesis so if we see differences, we need to try and explain them.

Multi-element signatures organic/conventional tomatoes



The mean values of the elements in bold type (for org and conv groups) are statistically significantly different….t-test.

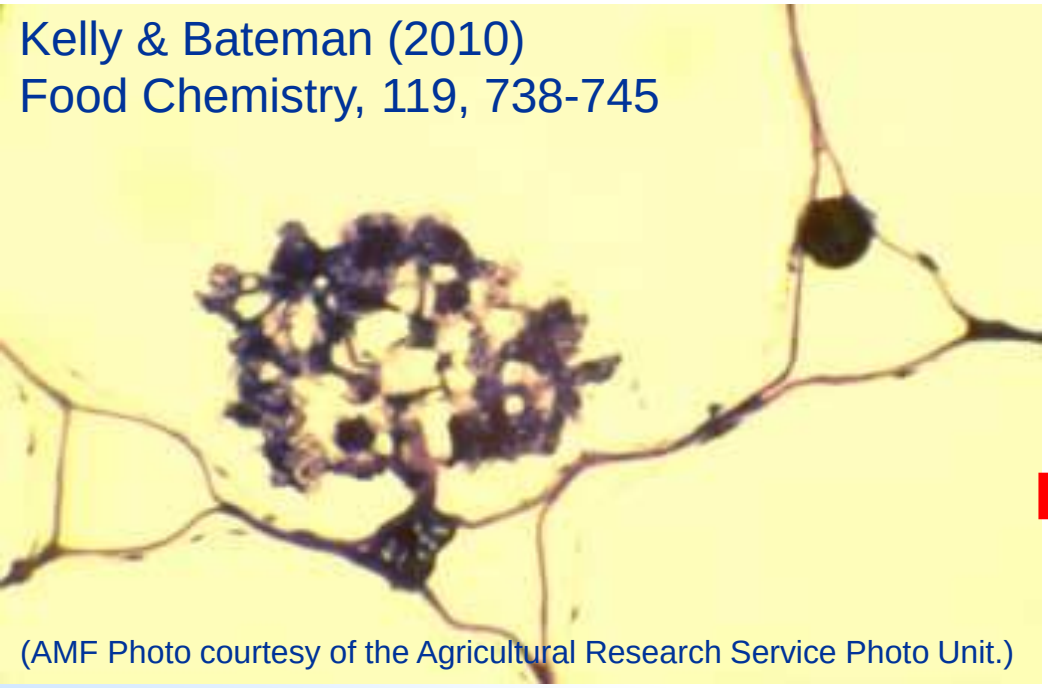
Ca	2x higher in org
Cu + Zn	slightly higher in org
Mn	2x lower in org
Rb	4x higher in org

Possible explanations

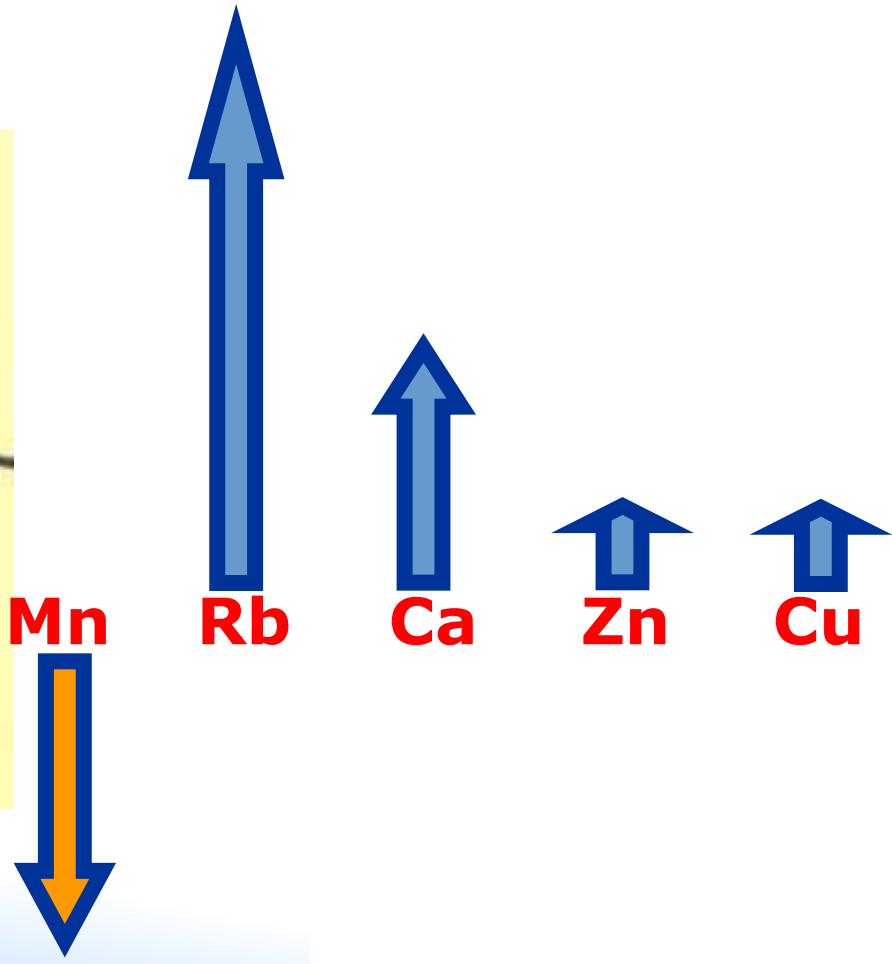
- Cu/Zn used in animal feed supplements, use of manure in org. cultivation?
- Rb usually geographical indicator. Soil grown vs hydroponic?
- More Mn available in hydroponic solution than naturally in soil?
- Mycorrhizal associations?

AMF association in organic Soils (Arbuscular Mycorrhizal Fungi)

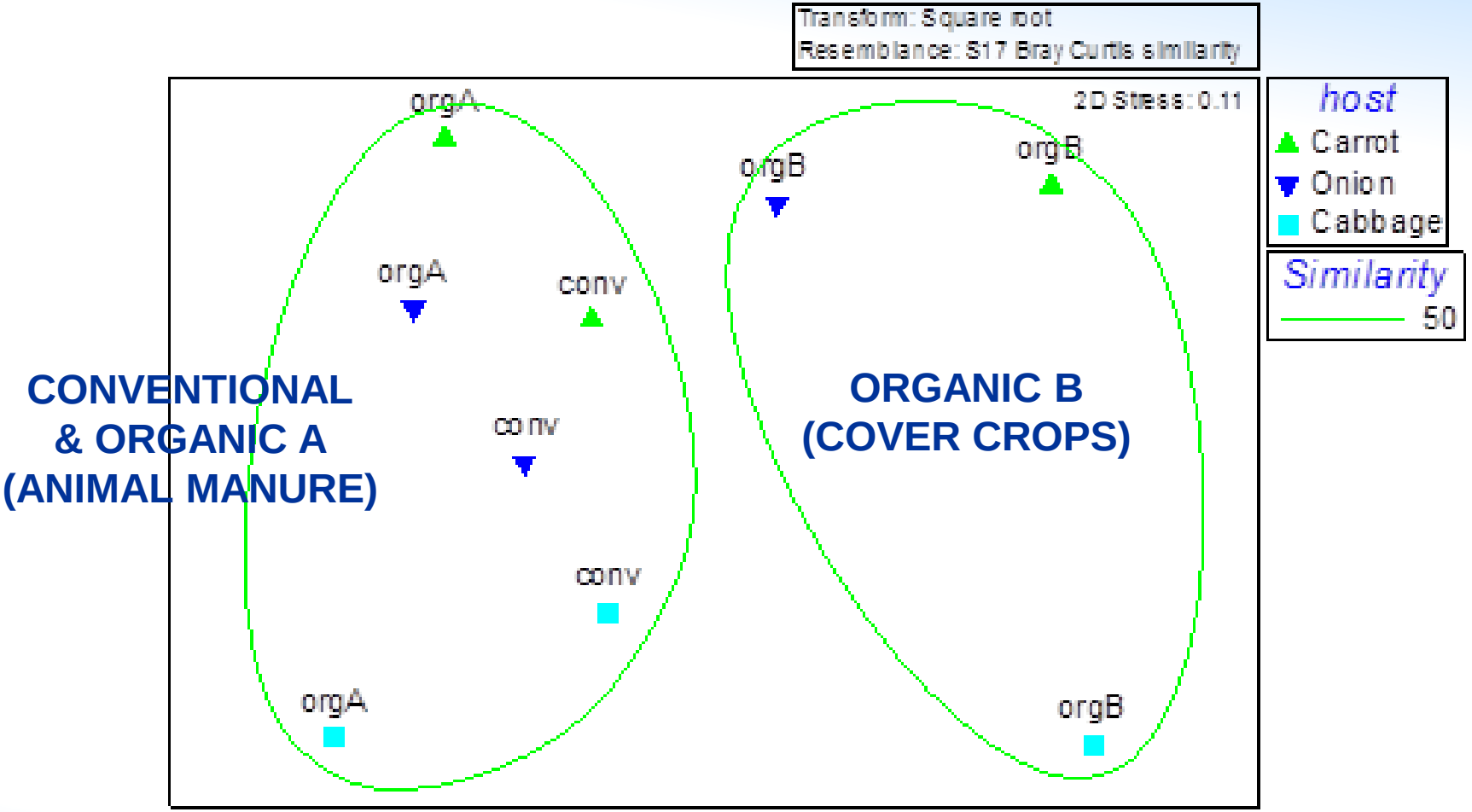
Kelly & Bateman (2010)
Food Chemistry, 119, 738-745



(AMF Photo courtesy of the Agricultural Research Service Photo Unit.)



Soil fungal community analysis for discrimination between organic/conventional cultivation



analysis of similarities (ANOSIM)

Halal food ‘authenticity’ or compliance with sharia law

- Approximately one quarter of the world’s population is Islamic.
- The halal market is currently worth 16% of the entire global food industry and is predicted to rise to 20% in the near future.
- The halal market is estimated to contribute between 40 and 100 billion EURO to the European Economy

Halal food ‘authenticity’ or compliance with sharia law

- Question of slaughtering of animals which are allowed to be eaten following the Islamic ritual and techniques.
- Alcoholic drinks and food containing ethyl alcohol.
- Vinegars derived from ethanol from the spirit industry.
- Food containing porcine meats and derivatives.
- Food additives and their origin

Dr. Md. Sani Ibrahim is currently JAKIM’s consultant on technical issues in Halal requirements.

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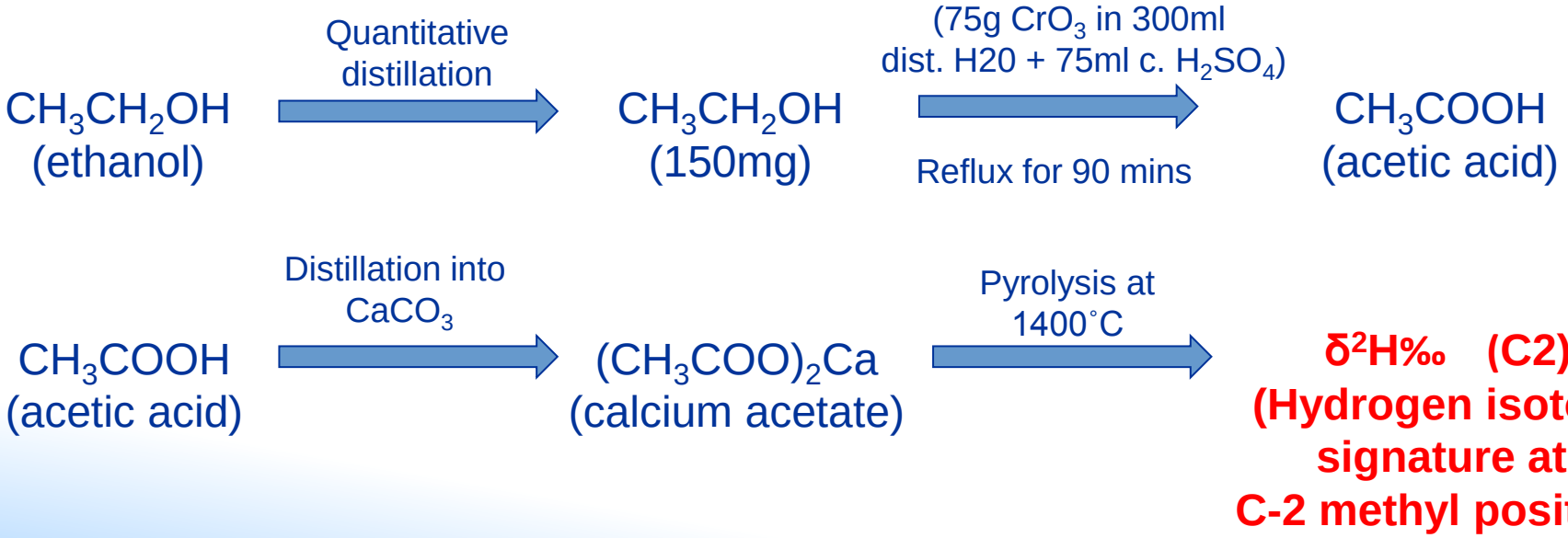
Conversion of ethanol to calcium acetate to obtain methyl ($\delta^2H‰$)$_{C2}$

Z Lebensm Unters Forsch (1989) 188:434–438

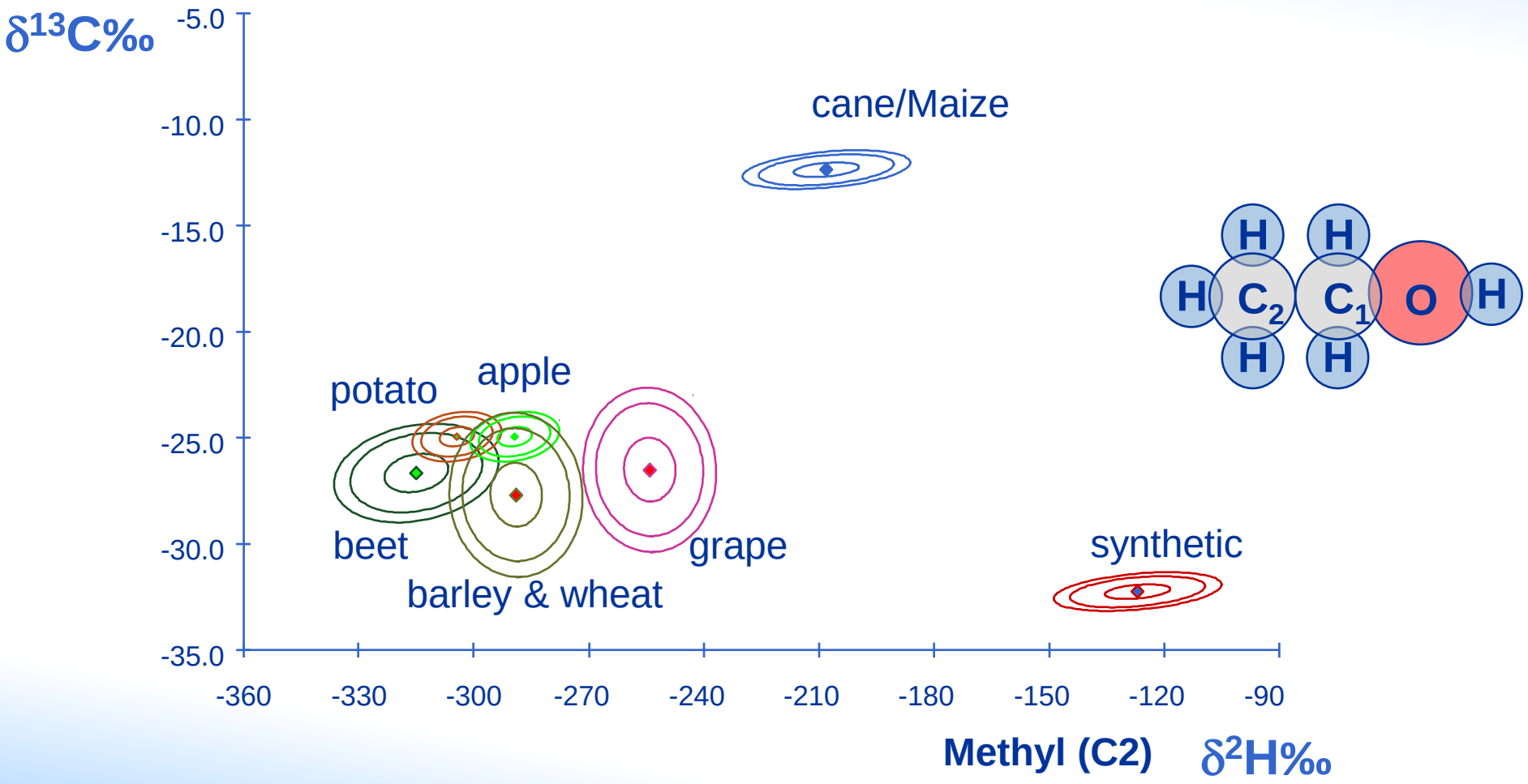
Proof of origin of ethanol and sugar from wine through hydrogen and carbon position-specific stable isotope measurements

Andreas Roßmann und Hanns-Ludwig Schmidt

Lehrstuhl für Allgemeine Chemie und Biochemie, Technische Universität München, D-8050 Freising-Weihenstephan, Bundesrepublik Deutschland



What is the source of ethanol?



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What is the source of gelatine?
What is gelatine used in?

fera
The Food and Environment Research Agency









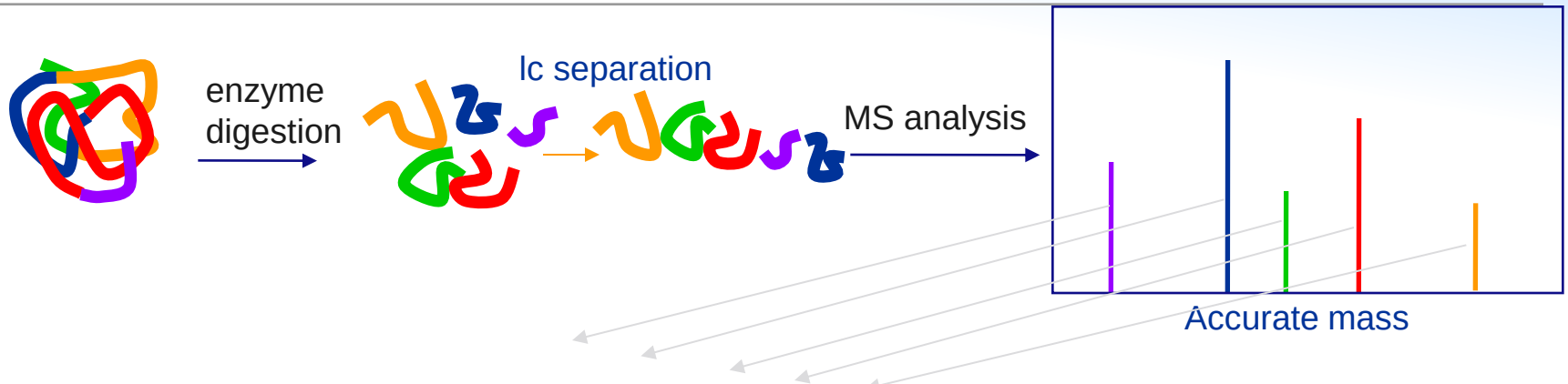




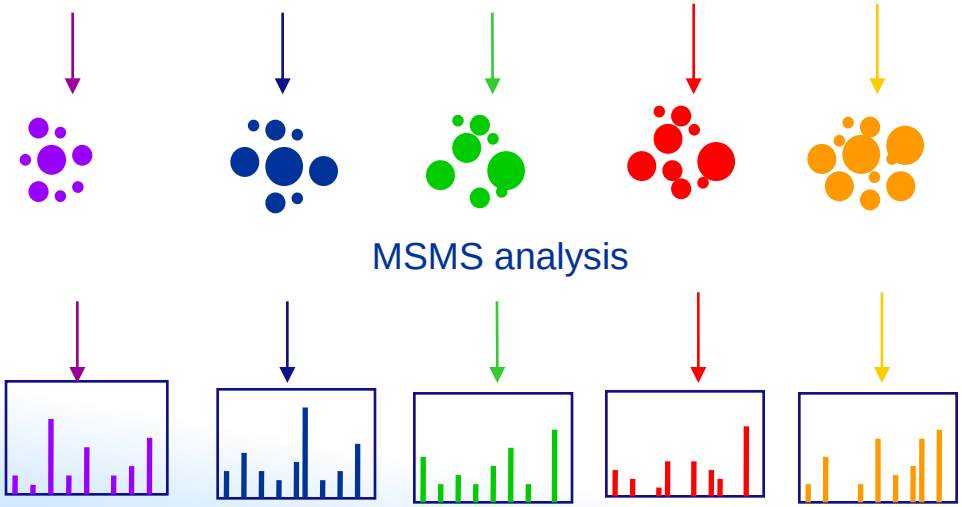


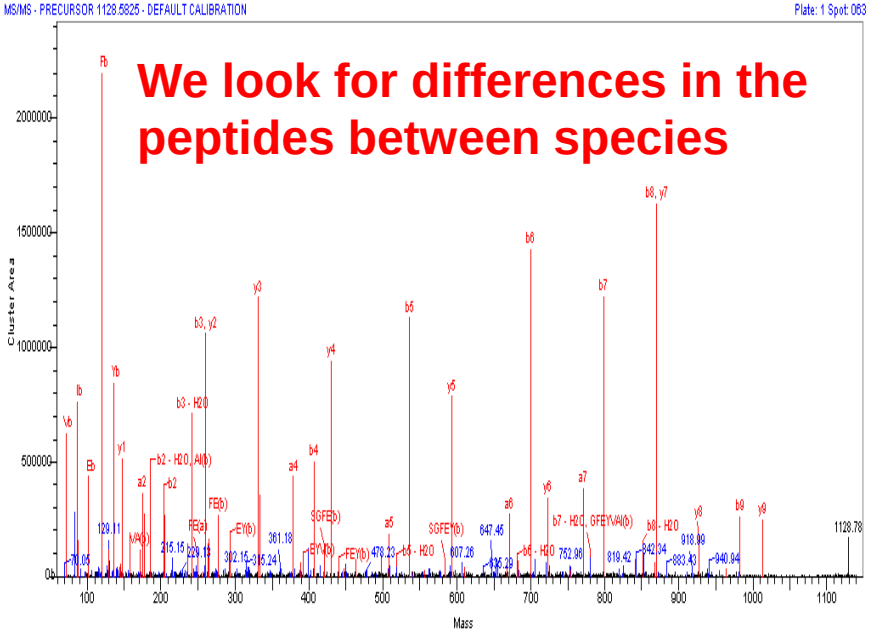


LC QTOF MSMS



Fragmentation of tryptic peptides in MSMS mode





Small UK FSA studies (2009)

- Chicken injection powders (tumbling)

Labelled as ‘free from pork and beef’

identified 4 cow-specific peptides

‘Chicken’, ‘Halal slaughtered’

ID’d cow-specific peptides in exudate

Halal food ‘authenticity’ or compliance with sharia law

- Question of slaughtering of animals which are allowed to be eaten following the Islamic ritual and techniques.
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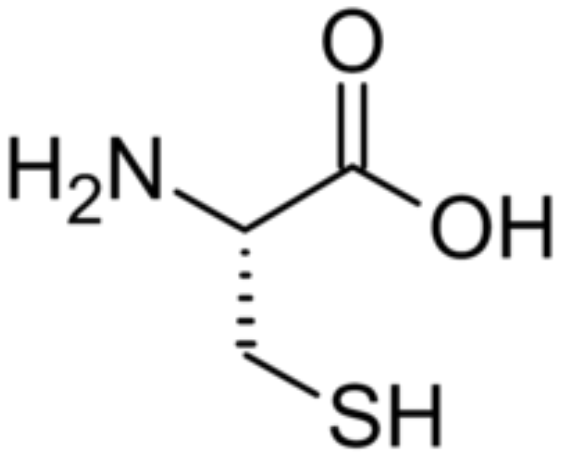
E920 Food additive in bread

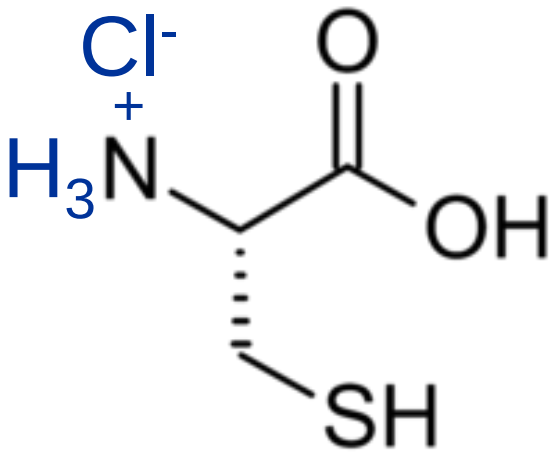
The Food and Environment Research Agency

L-Cysteine



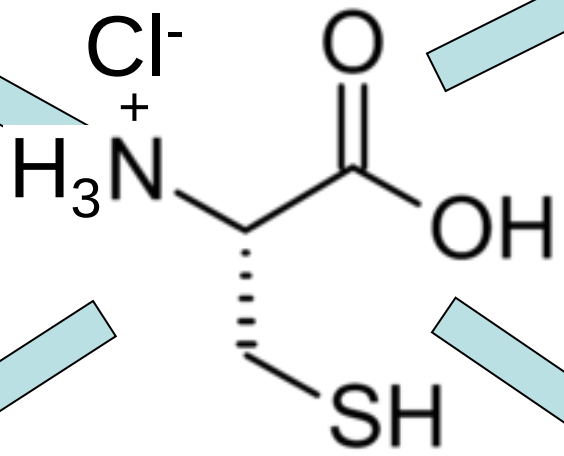
L-Cysteine

- L-Cysteine hydrochloride (E920)



L-Cysteine Industrial uses

Expectorant
(Acetylcysteine)



Flour treatment agent
"Improver"

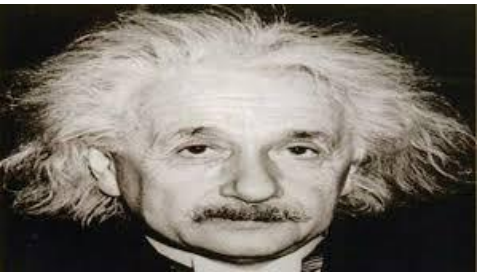
Dietary supplement
(Anti-oxidant)

Meat flavour enhancer

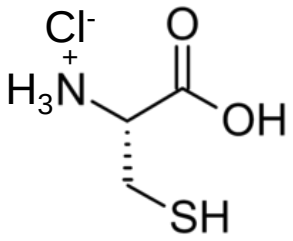
L-Cysteine
Industrial sources

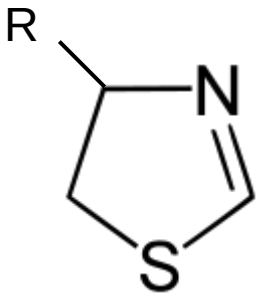
Chicken feathers ~10%w/w

Duck feathers ~10%w/w



Human hair ~ 20w/w

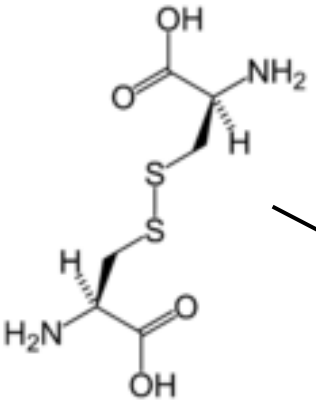




Substituted thiazolines

Joint FAO/IAEA Programme
Nuclear Techniques in Food and Agriculture

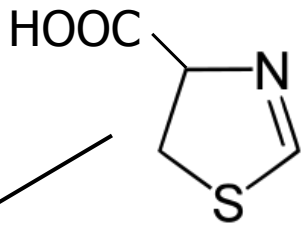
L-Cysteine production



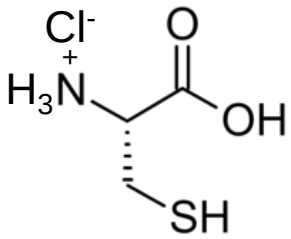
Human hair (keratin)
contains approximately
10-14%w/w L-cystine

hydrolysis
HCl

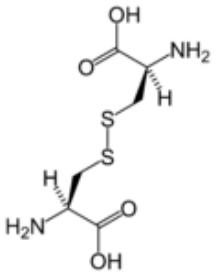
Assymetric enzyme
Hydrolysis by
Pseudomonas
thiazolinophilum



DL 2-amino-Δ^2-thiazoline
-4-carboxylic acid (DL-ATC)

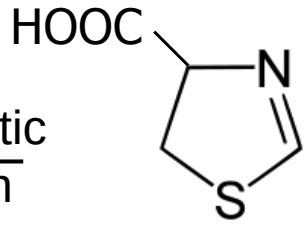


Racemisation



DL-cystine

Electrolytic
reduction



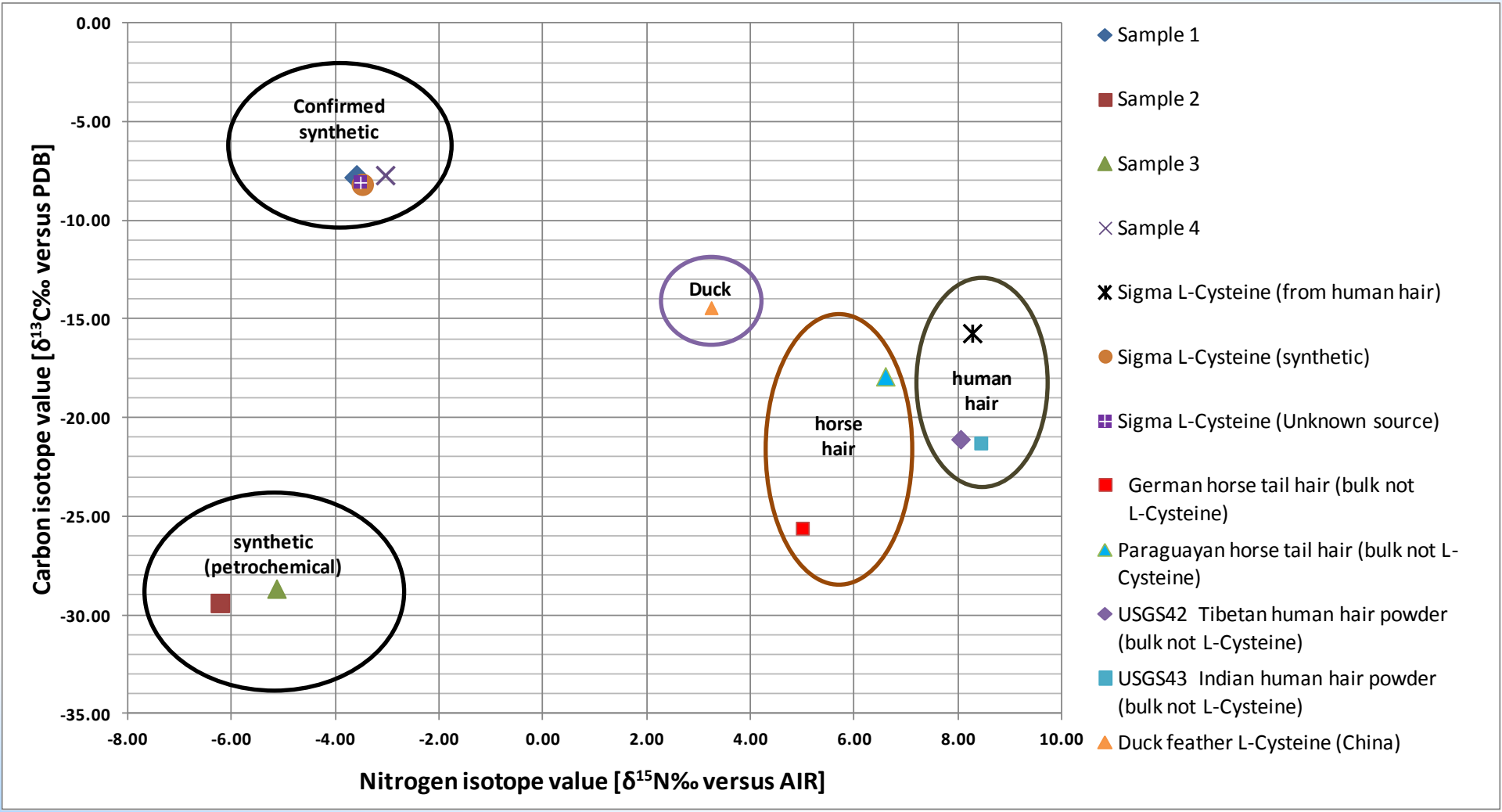
DL 2-amino-Δ^2-thiazoline
-4-carboxylic acid (DL-ATC)

E920 Ethical issues

- Ingredients from the human body are Haram
- Haram is opposite of Halal and means unlawful
- L-Cysteine from human hair is Haram*
- Some companies choose to label food ingredients derived from animal sources as “non-vegetarian” including E920 from poultry feathers
- Some bread manufacturers have been challenged over the source of their E920 in bread
- **Can we tell if E920 is from a non-animal source using stable isotope analysis?**

*Muslim consumer group
http://www.muslimconsumergroup.com/ingredient_types.html

Carbon versus nitrogen stable isotope results



Summary E920 (Cysteine HCL) sources

Identity	δ13C‰	δ15N‰	δ34S‰	14C [% modern]	
Industrial E920 for flour improvers					
Sample #1	-7.8	-3.6	5.6	90.20	Synthetic fermentation
Sample #2	-29.4	-6.2	25.8	< 2	Synthetic petrochemical
Sample#3	-28.7	-5.1	11.4	<2	Synthetic petrochemical
Sample #4	-7.7	-3.0	0.2	98.00	Synthetic fermentation
Sigma-Aldrich					
Sigma-Aldrich C7755 L-Cysteine (from human hair)	-15.7	8.3	7.0		
Sigma-Aldrich W326305 L-Cysteine (Confirmed synthetic)	-8.2	-3.5	4.1		
Sigma-Aldrich C7352 L-Cysteine (Unknown source)	-8.1	-3.5	23.0		
Duck feather L-Cysteine (China)	-14.4	3.2	8		
Bulk hair samples (not extracted L-Cysteine)					
German horse tail hair	-25.6	5.0	6.8		
Paraguayan horse tail hair	-17.9	6.6	10.0		
USGS42 Tibetan human hair powder	-21.1	8.1	7.8		
USGS43 Indian human hair powder	-21.3	8.4	10.5		

TEST SAMPLES 1 TO 4 WERE ACCEPTABLE FOR HALAL LABELLING

Summary Conclusions (1)

1. **Stable nitrogen isotope** (^{15}N) analysis is more suitable for some crop types than others e.g. horticultural, hydroponic conventional versus Organic soil grown. Oxygen isotope analysis of nitrate offers the potential for a more secure classification and more research is required. N & O Isotopes can provide evidence to corroborate whether chemical fertiliser has been applied to a crop *& authenticate Organic fertiliser*.
2. **Fluorescence microscopy** can reliably determine the frequency of tetracycline dosing in meat with the *limitation of requiring bone to conduct the test.*
3. Preliminary **metabolomic analyses** have shown promise as a reliable screening technique to distinguish between Organic and conventional crops but requires accurate mass LC-MS for peak assignment. Other spectroscopic profiling techniques may also show potential e.g. Phosphorus NMR.

Summary Conclusions (2)

4. **Multi-element analysis** improves classification rates for Organic crops, when combined with ^{15}N analysis alone. However, Further research is required to understand the full potential and assess the reliability of this technique.

6. **Existing food authenticity techniques** do have a great deal to offer in the Organic arena if they are accepted as being part of the overall certification process *NOT a replacement*.

7. **The way forward is to work with certification bodies** to apply the tests when there is suspicion of fraud on the farm and with Enforcement Agencies and retailers when their is suspicion of fraud further along the supply chain.

Acknowledgements

- CRP D52040 “Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food” – OPEN NOW

http://cra.iaea.org/cra/explore-crps/all-opened-for-proposals.html

- “Confirmation of the production origin of food using isotopic techniques” – OPEN END 2017

IAEA Labs Seibersdorf at the opening of Frieden strasse

IAEA 60 Years
Atoms for Peace and Development

Thank you and merry Christmas!

S.Kelly@iaea.org

Summary Conclusions (3)

8. Food control systems require analytical techniques for verification and investigation when the ‘system’ fails
9. There is no “magic” method
10. A combination of techniques is required to produce a toolbox for regulators and industry
11. Combination of conventional analytical techniques, emerging techniques and chemometrics show promise in this field but present challenges for enforcement work
12. Open access databases from authentic samples are crucial and the biggest single barrier to deployment and wider use of authentication methods in Member States

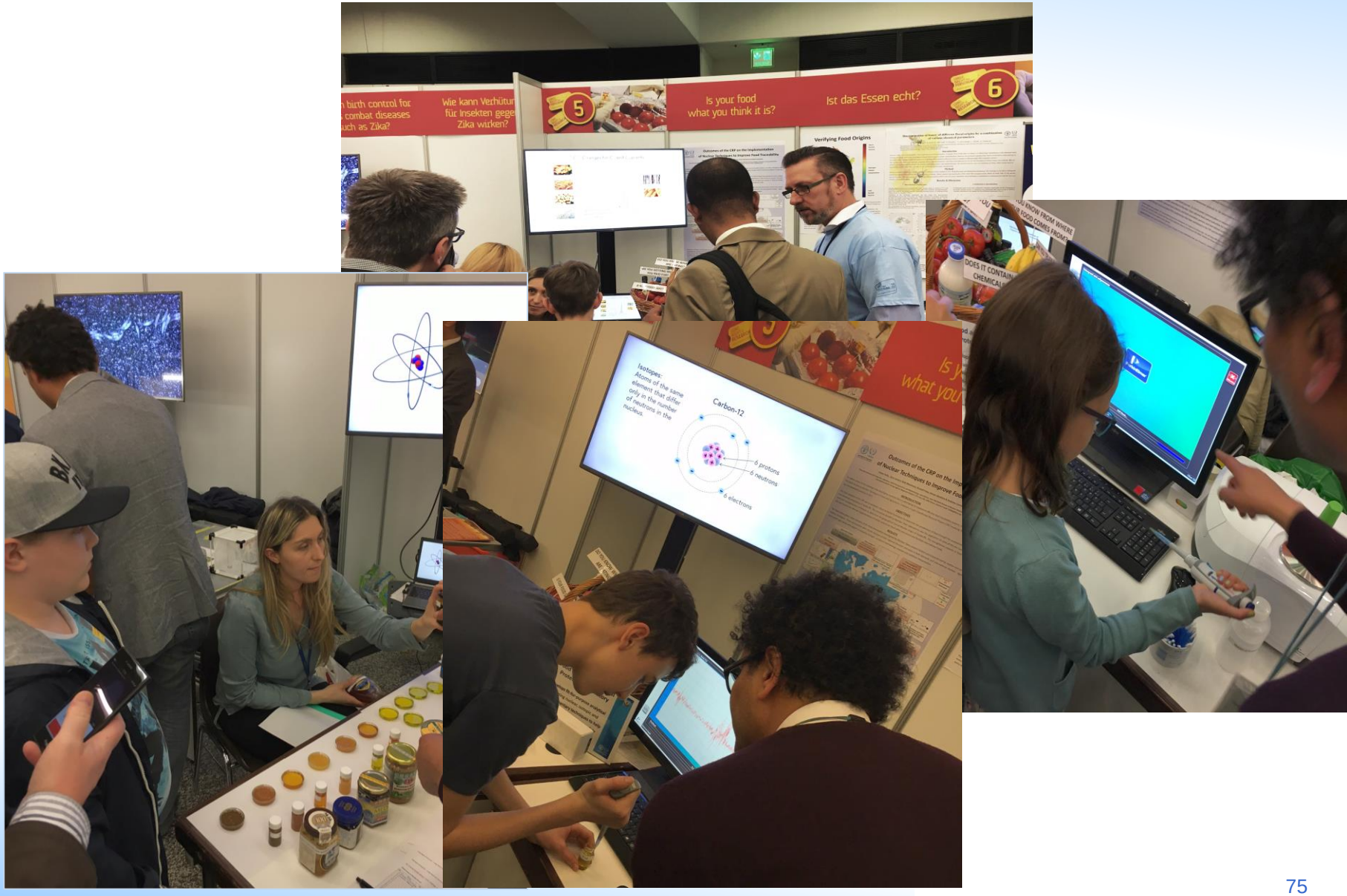
Technical cooperation: Training and capacity building

Stable isotope measurements to establish food origin

Using radioisotopes to investigate the transfer of natural toxins from the environment to food

Analysis of trace-level chemical contaminants in food

Outreach & education

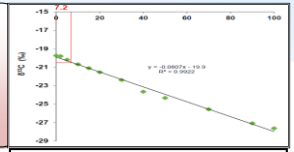


Coordinated Research Projects

IAEA 60 Years Atoms for Peace and Development

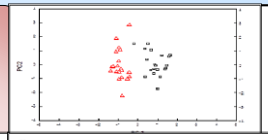
Agreement Holders
Austria, Portugal, Sweden, UK & USA
Advice and support

China partner 1
Milk adulteration with Soya milk, determined by carbon isotope analysis

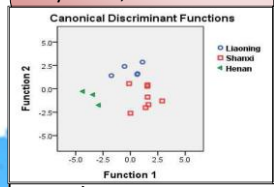


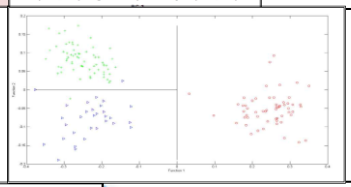
Calibration curve— bovine milk mixed with soya milk

China partner 2
Pork, honey and tea
production & geo-origin by IRMS, ICP & FAMEs



Organic v's Conventional Pork meat



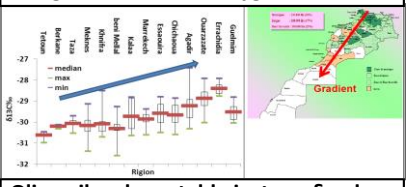


Organic v's Convention Tea Shangdong & Hanghzou

Acacia honey from Liaoning, Shanxi and Henan province

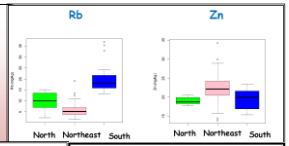
Morocco
Olive & Argan oils
IRMS, NMR

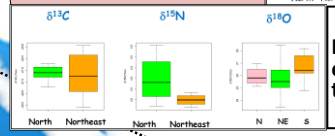
Argan oil carbon & oxygen δ-values



Olive oil carbon stable isotope δ-values

Thailand
Jasmine rice
Geo-origin
INAA, ICP-AES, IRMS





Isotopes and trace elements in authentic Jasmine rice

Lebanon
Wine and olive oil
ICP/MS-CRI, CRDS

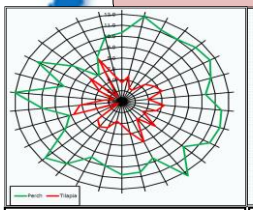
Uganda
Fish, coffee and peanuts
NIR, IRMS, ICP-MS

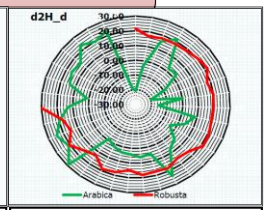
Pakistan
Basmati rice
Geo-origin by PCR, IRMS and ICP-MS

India
Basmati rice, wheat and fruit juice
Geo-origin by PCR, SIA &

Singapore
Poultry and eggs
IRMS, TIMS, SNIF-NMR

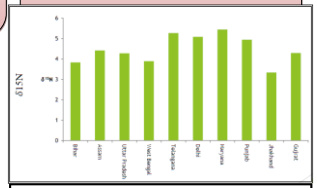
Chile
Carmenere wine
NAA, AAS, LC-MS/MS





Perch and Tilapia nitrogen stable Isotope δ-values

Arabica and Robusta coffee hydrogen stable Isotope δ-values



Authentic basmati rice nitrogen stable Isotope δ-values

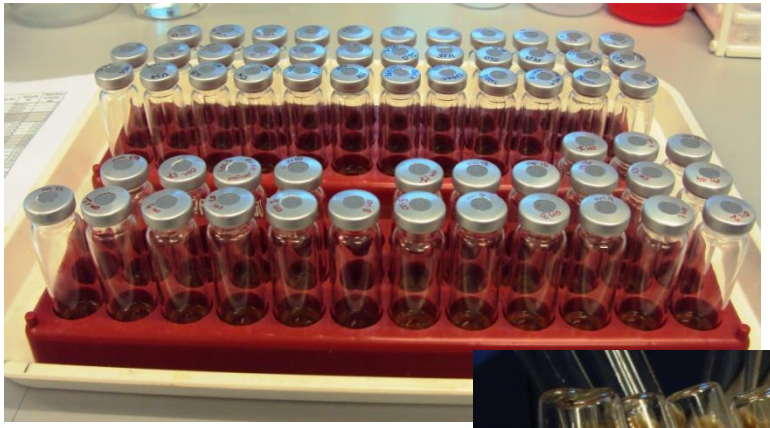
METHODOLOGY

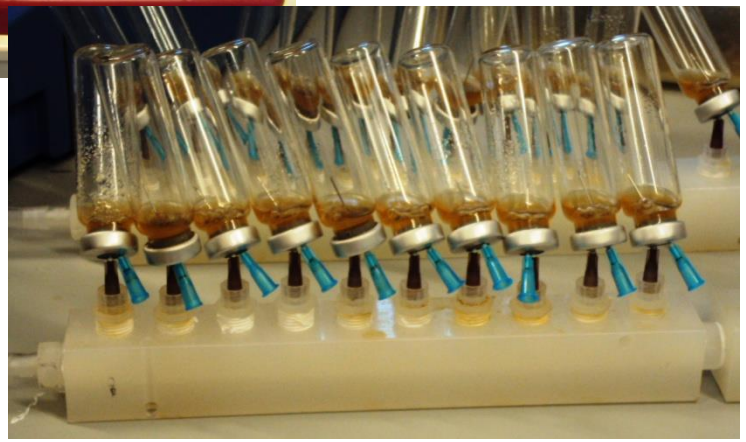
1. Crop samples are freeze-dried and milled to a fine powder
2. Nitrate extraction (in water, 90°C)
3. Measurement of nitrate concentration (ion chromatography)
4. The denitrifier method

METHODOLOGY

4 The denitrifier method *

using denitrifying bacteria *Pseudomonas aureofaciens* for conversion of plant NO_3^- (liquid extract) to N_2O (gas)



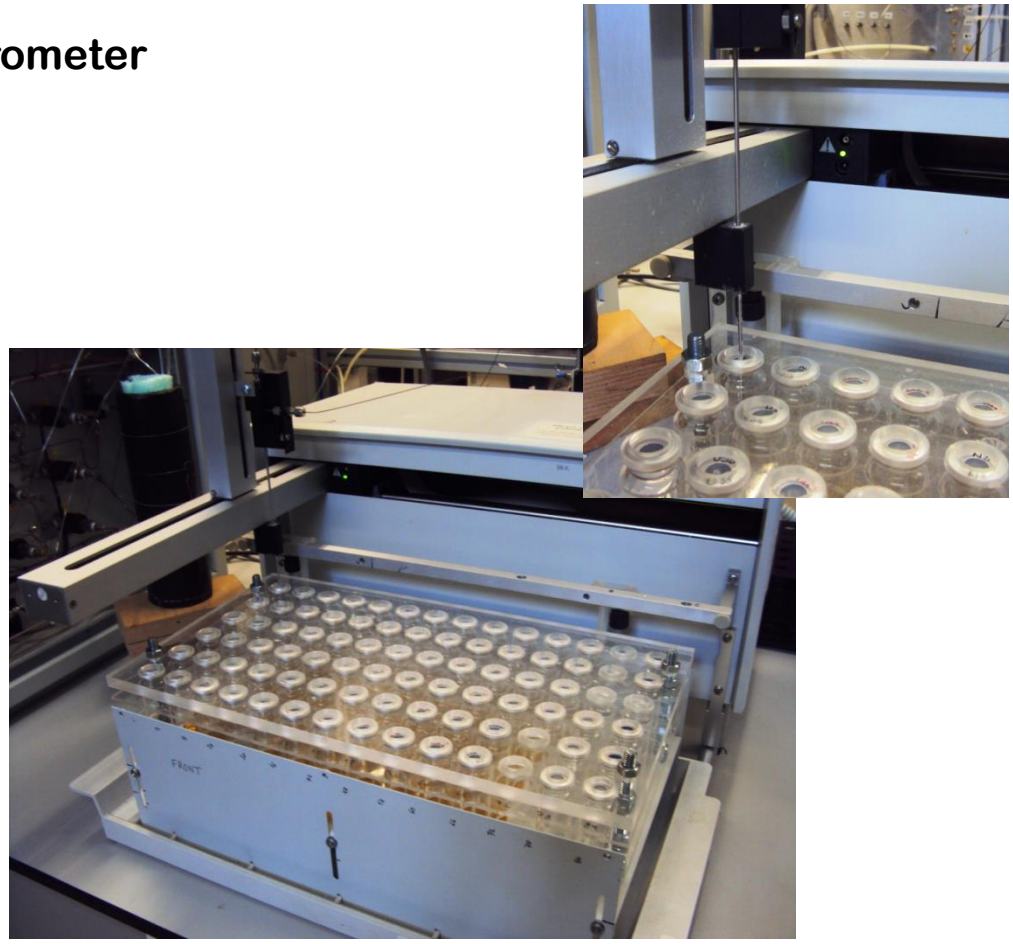


* Sigman et al., 2001; Casciotti et al., 2002

METHODOLOGY

5 Stable N and O analysis

ANCA TG II system
Gilson head-space autosampler
PDZ Europa GEO 20-20 mass spectrometer



METHODOLOGY

5 Stable N and O analysis

ANCA TG II system
Gilson head-space autosampler
PDZ Europa GEO 20-20 mass spectrometer

Organic Farming

- **Organic farming severely restricts the use of artificial chemical fertilisers and pesticides.**

- Organic farmers rely on developing a healthy, fertile soil and growing a mixture of crops.

- Management practices which sustain soil health and fertility.

- The use of natural methods of pest, disease and weed control.

- High standards of animal welfare.

- Low levels of environmental pollution.

- Enhancement of the landscape, wildlife and wildlife habitat.

- The prohibition of all genetically engineered food and products.

Apart from non-allowed usage, a number of external sources may cause contamination of organic crops, including the following:

- leaching of pesticides from conventional to organic crops
- use of contaminated water for irrigation
- atmospheric deposition of volatile compounds used in the surrounding areas (overspray)
- plant uptake from soils contaminated by persistent pesticides or herbicides (e.g. Dieldrin, DDE, DDT)
- Cross-contamination from conventional crops during manufacturing, transport or storage
- Consequently, is there a justification for using pesticide analysis for ‘intelligence’ in cases of suspected fraud?
- USDA 2012 survey found that over 40% of organic crops tested exceeded MRLs

Pesticide residue analysis

IAEA 60 Years Atoms for Peace and Development

Pesticide residue analysis of Organic foods: UK (2009)

- In 2009, out of 3835 samples, 246 (6.4%) were labelled as Organic.
- None of the residues gave concerns for the health of any group of people who might have eaten the foods. None of the residues were over the relevant MRLs

Food	Where the sample was from	Pesticide residues found	Amount of residue found (mg/kg)	MRL
Beetroot	Spain	chlorpyrifos	0.04	0.05
Bread (2 samples)	UK	chlormequat pirimiphos-methyl	0.09 0.02, 0.05	No MRL No MRL
Eggs (2 samples)	UK	dieldrin	0.002, 0.004	0.02
Infant Food	Spain	diphenylamine	0.03	No MRL
Limes	Dominican Republic	carbendazim	0.04	0.5
Peppers (2 samples)	UK	pyrethrins	0.02, 0.06	1

Organic Farming

- **Organic farming severely restricts the use of artificial chemical fertilisers and pesticides.**

- Organic farmers rely on developing a healthy, fertile soil and growing a mixture of crops.

- Management practices which sustain soil health and fertility.

- The use of natural methods of pest, disease and weed control.

- High standards of animal welfare.

- Low levels of environmental pollution.

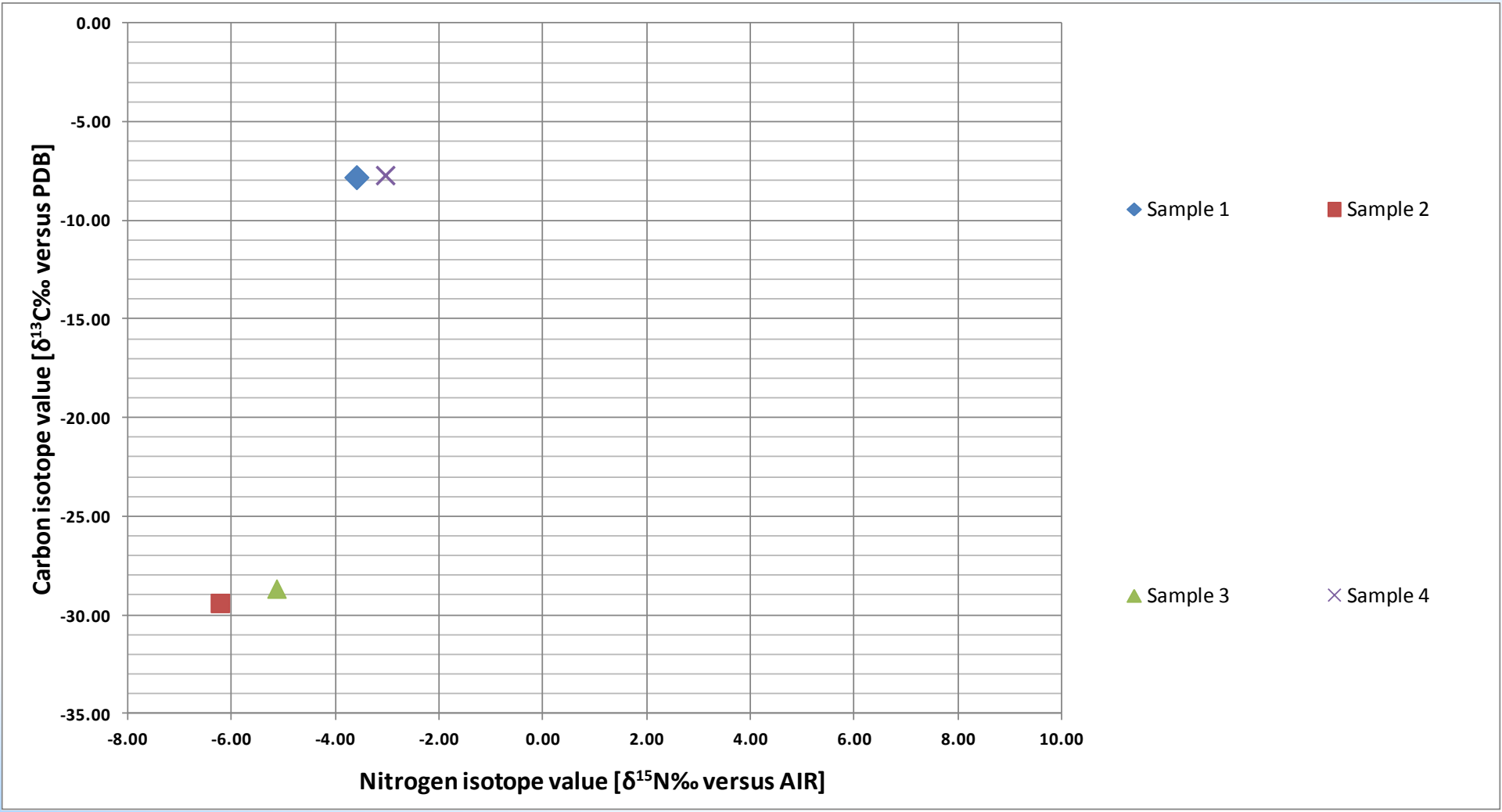
- Enhancement of the landscape, wildlife and wildlife habitat.

- The prohibition of all genetically engineered food and products.

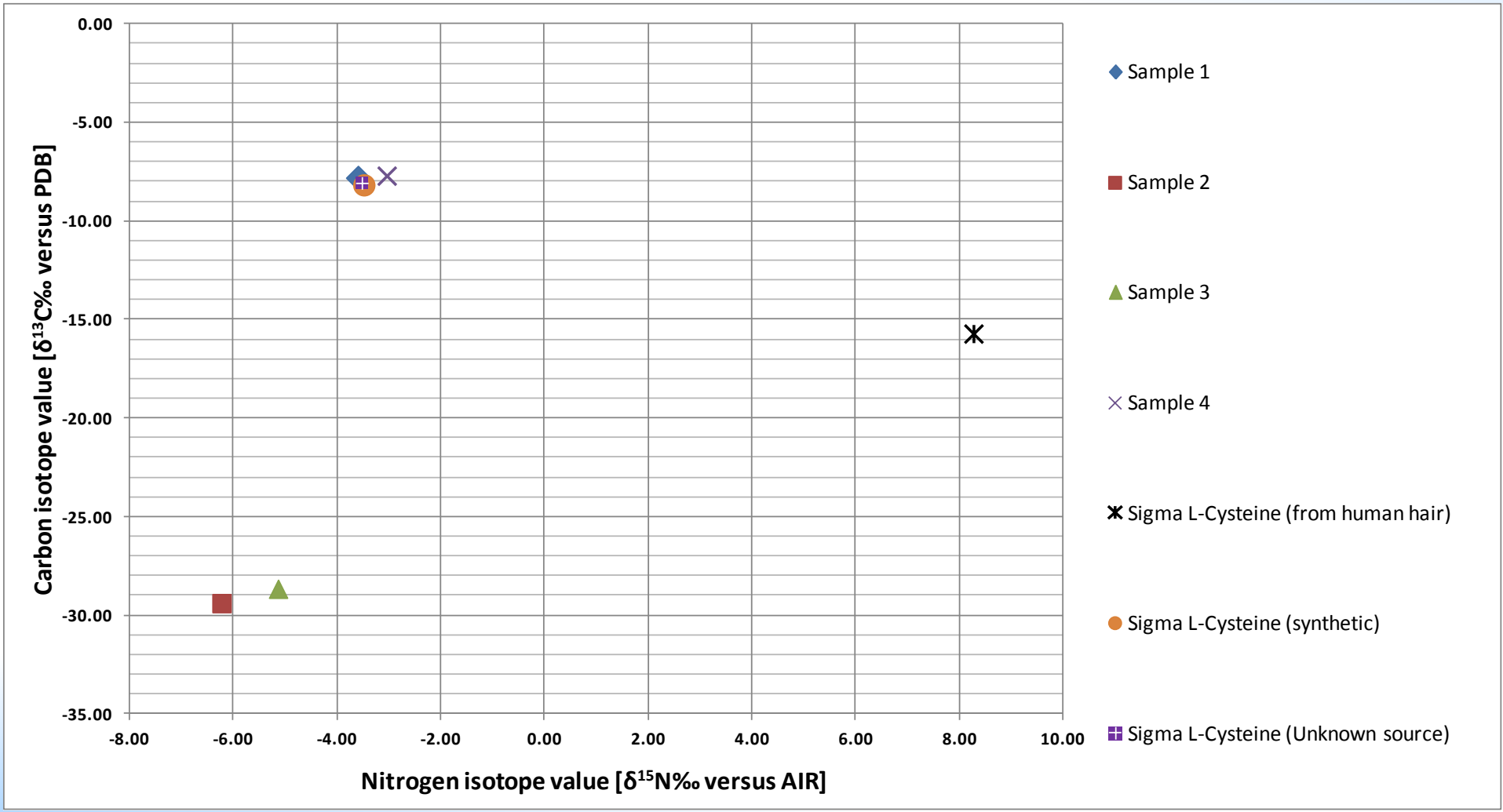
Metabolomics

- Technology aimed to give us a complete overview of the biochemical make-up of a biological product
- Non-targeted analyses
  - Metabolite ‘fingerprinting’ is pattern recognition without chemical structure assignment
  - Metabolite ‘profiling’ involves identifying the metabolites e.g. Accurate mass LC-MS
- Targeted analyses
  - Analyses of selected compound classes

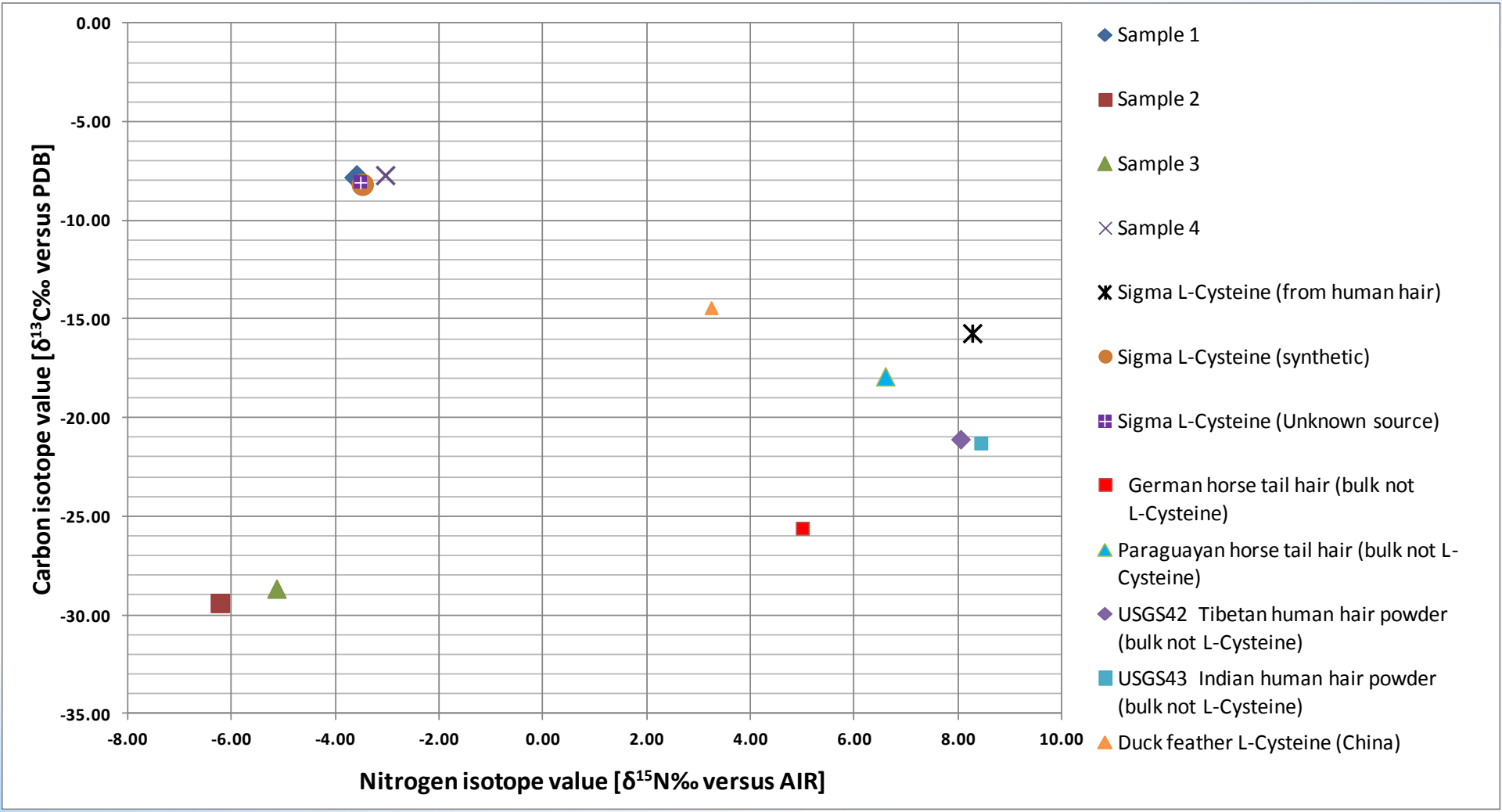
Carbon versus nitrogen stable isotope results



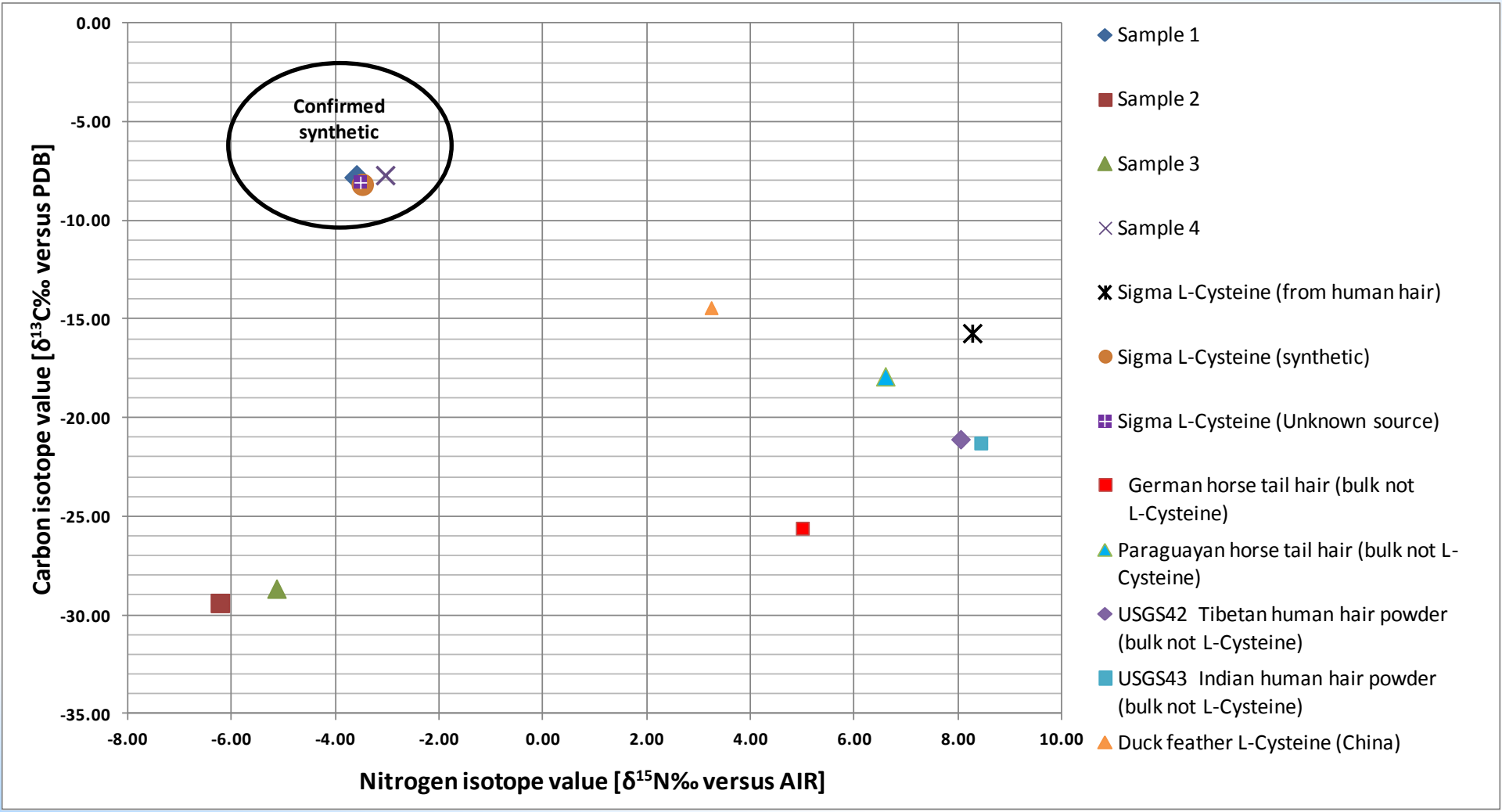
Carbon versus nitrogen stable isotope results



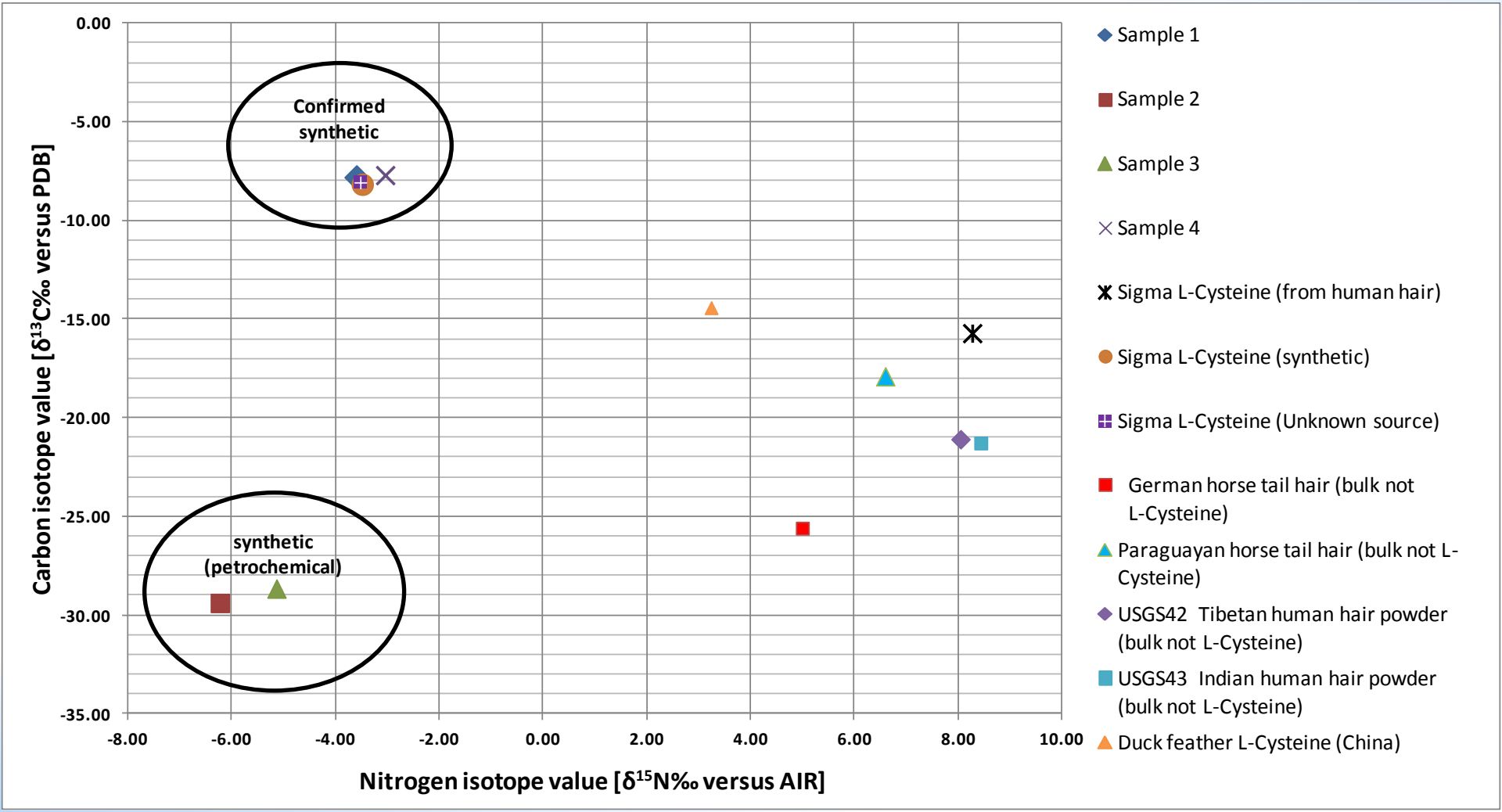
Carbon versus nitrogen stable isotope results



Carbon versus nitrogen stable isotope results



Carbon versus nitrogen stable isotope results



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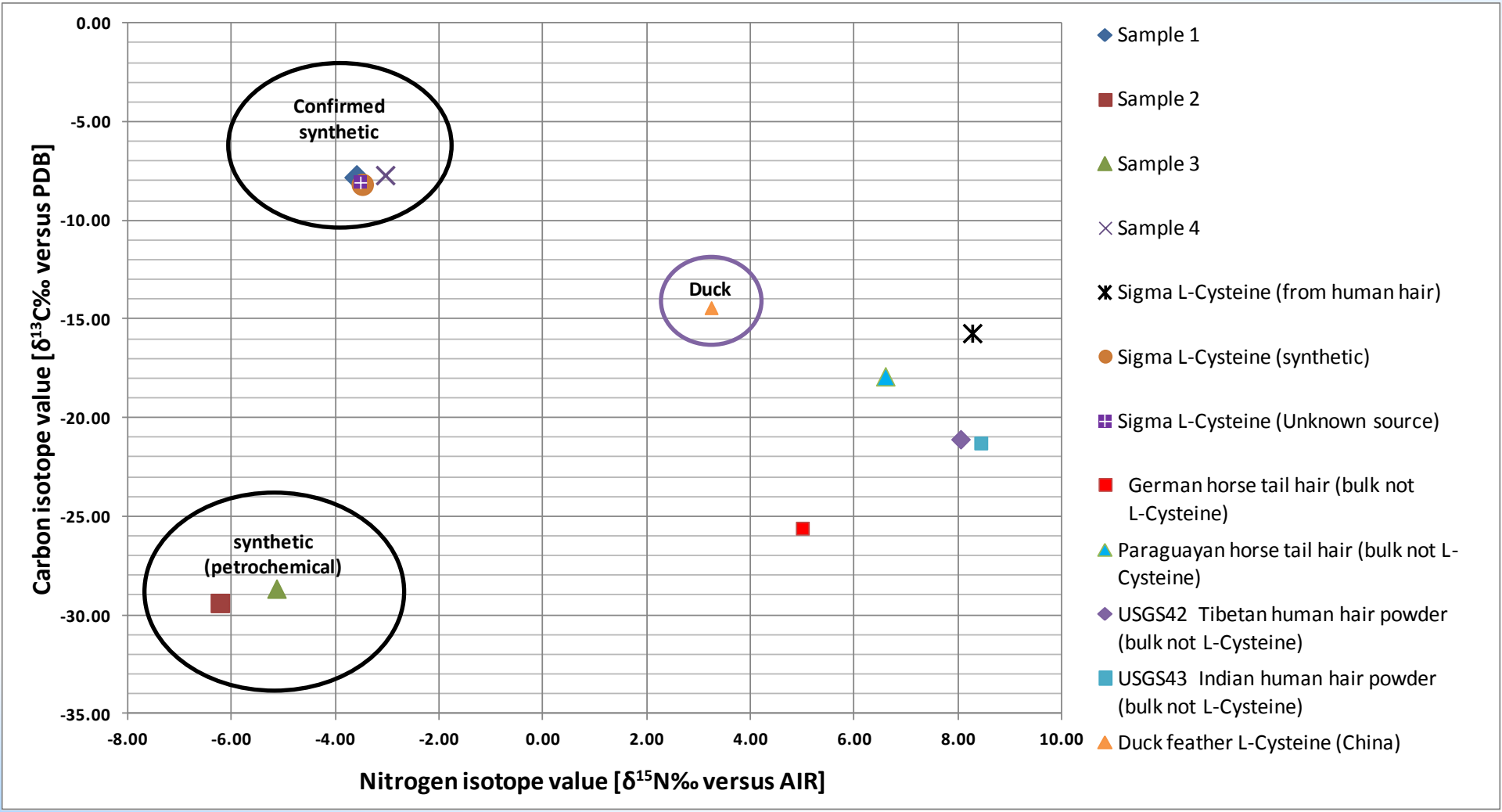
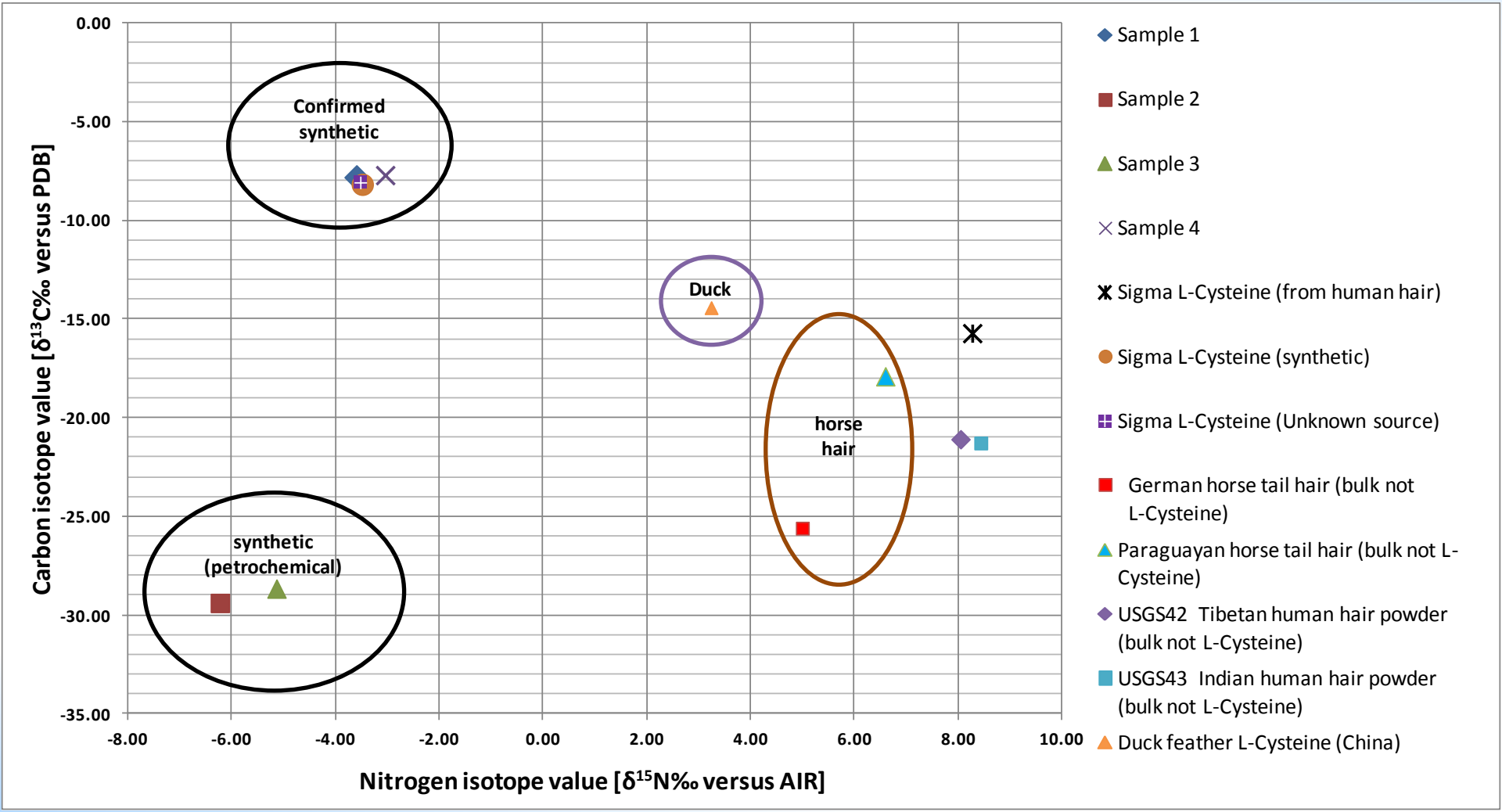


Carbon versus nitrogen stable isotope results



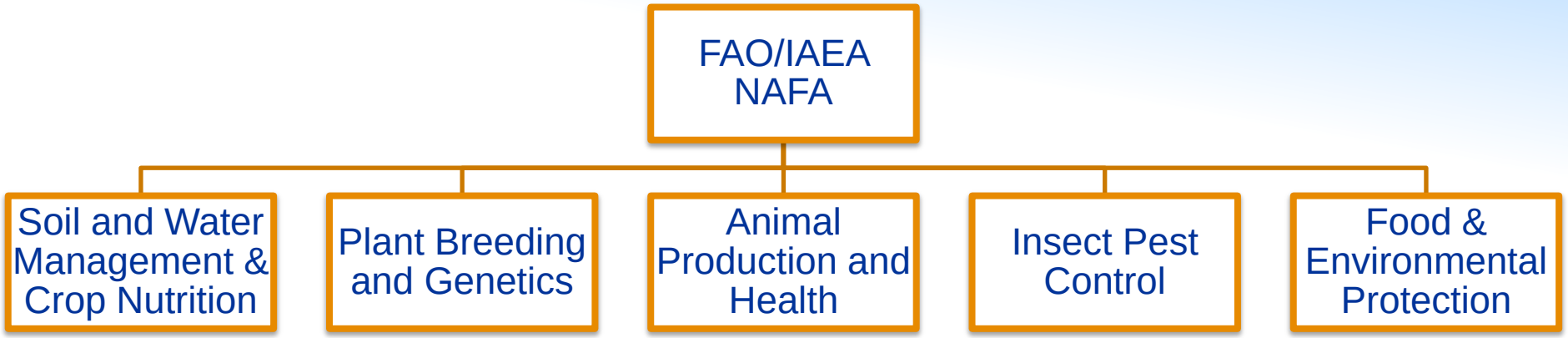
Coordinated Research Project calls

- CRP D52040 “Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food” – OPEN NOW

http://cra.iaea.org/cra/explore-crps/all-opened-for-proposals.html

- “Confirmation of the production origin of food using isotopic techniques” – OPEN END 2017

Activities of the FAO/IAEA Joint Division of Nuclear Techniques in Food & Agriculture



- Applied and adaptive R&D, method development…
- Coordinated Research Projects
    - Bring together researchers from both developing and developed MSs to coordinate research networks, facilitate applied research, and to foster the exchange of scientific and technical information
- Technical Cooperation Projects
    - Training to developing MSs implemented as individual or group fellowships, as scientific visits awarded to senior staff (programme managers and decision-makers), and as national, regional or interregional training courses, workshops or meetings.

Food & Environmental Protection Laboratory

- Assisting and supporting MS laboratories in their efforts to ensure their food is safe and authentic.
- Conducting applied and adaptive research in veterinary drug/pesticide residue analysis and food authenticity/origin determination.
- Actively participating in Codex committees
- Providing training to research Fellows
- Providing Scientific visitor training
- Undertaking expert missions
- Technical documents and guidelines
- Scientific communications and outputs

Food & Environmental Protection Laboratory

- Coordinated Research Projects
  - Development of Radiometric and Allied Analytical Methods to Strengthen National Residue Control Programs for Antibiotic and Anthelmintic Veterinary Drug Residues (D52036)
  - Implementation of Nuclear Techniques to Improve Food Traceability (D52037)
  - Accessible Technologies for the Verification of Origin of Dairy Products as an Example Control System to Enhance Global Trade and Food Safety (D52038)
  - Field-deployable analytical methods to assess the authenticity, safety and quality of food (Jan 2017)

Food & Environmental Protection Laboratory

- Technical Cooperation Projects
  - Inter-regional South East Asia
  - National – Malaysia, China and Iraq

- European Projects
  - partner in the EU FP7 Integrated Project ‘FoodIntegrity’
  - Partner in the EU Horizon 20-20 Project ‘Authent-Net’