



The Second Annual Meeting of the Arab Section of AOAC INTERNATIONAL

# Food Fraud and Latest Technologies for Accredited Detection

Dr. Mohammad Hijaz

Hijaz Scientific Supplies Est

---



# Food Fraud and Latest Technologies for Accredited Detection

---

01 Introduction to EMA – Economically Motivated Adulteration

02 History and Coverage

03 Fraud Detection

04 NMR: a Powerful Technique for Food Authenticity Control

05 FoodScreener™ Solution

06 Honey Profiling 3.0

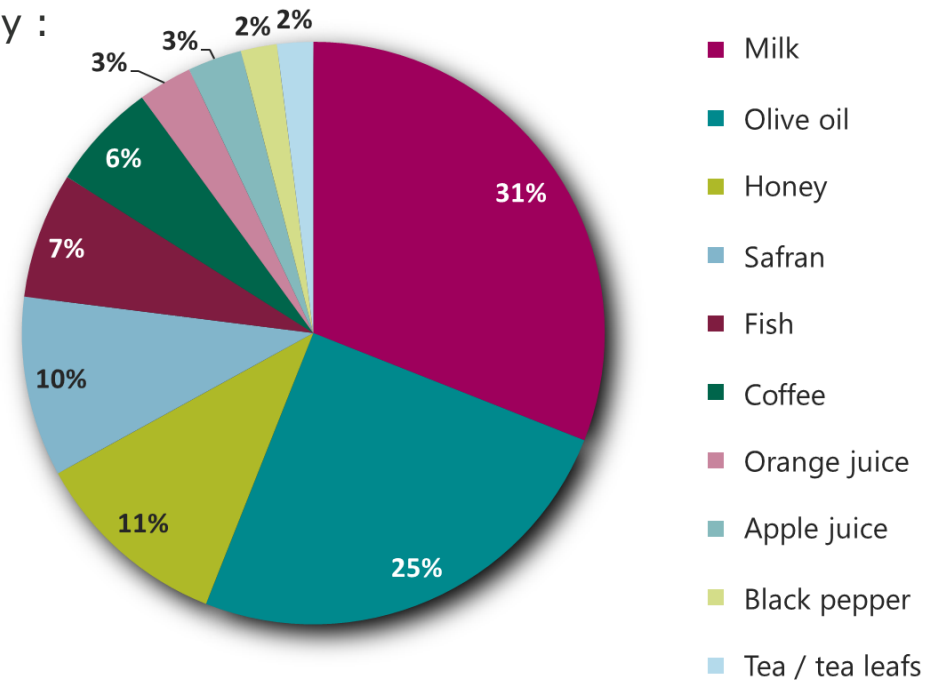
07 Q&A

08 Summary

# What is it?

## What are the causes?

- It occurs when products are deliberately/intentionally :
  - Diluted, mislabeled or misrepresented
  - Tampered/substituted with another product
- Incentives/Causes for food adulteration:
  - Profit
  - Unsatisfied market demand
  - Cost pressure at industry



Source: USP (United States Pharmacopeial Convention, Rockville, USA, 2013)

=

FOOD FRAUD AND LATEST TECHNOLOGIES FOR ACCREDITED DETECTION

# History and Coverage

---

# Highlights in Recent Years

**Food fraud: Ten states unable to ensure food security; 15% of all samples sub-standard**

By PE Online | Updated: Jun 03, 2020 5:09 PM

India is facing a food fraud problem with about ten states unable to ensure food safety and 15% of food samples failing to pass quality tests.

**Police seize 1,305 bottles of adulterated olive oil in Greater Victoria**

Five establishments were inspected and three brands are being investigated by the Specialized Consumer Protection Department (Decon)

**Nas of Catania seizes seven tons of honey sold without traceability**

THE TRADER DENOUNCED

STUDIO CON APEW

La tua SCUOLA DI LINGUE

Contattaci su WhatsApp +39 377 4368137

WENN'S SCHNELL BEHEN SOLL

**NEWS**

Home US Election Coronavirus Video World UK Business More

Business Market Data Global Trade Companies More

**US beekeepers fear for their future**

By Pamela Parker  
Business reporter, BBC News, California

26 June 2020

**THE COURIER.CO.UK**

NEWS POLITICS SPORT BUSINESS LIFESTYLE OPINION SUBSCRIBE

CORONAVIRUS Dundee Angus & The Mearns Perth & Kinross Fife Send us a story Supplements Home delivery

**LIFESTYLE / FOOD & DRINK**

**The sticky issue of 'honey laundering' and how a new Kitemark aims to protect local produce**

Rebecca Shearer lifts the lid on the global problem of fraudulent honey-making and hears how a new initiative aims to give consumers confidence in the products they're buying.

July 13 2020, 6:00pm by Rebecca Shearer

**EL ESPAÑOL**

REPORTS

**The war between Félix Solís and García Carrión over Valdepeñas wine, on its crucial day due to fraud**

Both wineries are fined by the Castilla-La Mancha Ministry of Agriculture for alleged fraud in the labeling of wine.

July 9, 2020 - 02:55

**Olive Oil Times**

Business

**Brazil Reveals Widespread Olive Oil Fraud**

Brazil's Ministry of Agriculture, Livestock and Farming reported that sixty-four percent of olive oil analyzed in the last two years did not meet the quality standards required by their labeling.

Apr. 20, 2017 By Julie Al-Zoubi

7.6k

**The Region**

Murcia Cartagena Lorca Molina Sewer Mazarron Eagles Yecla Pacheco

**Extra virgin olive oil leads food fraud cases**

15% of the samples analyzed by inspectors in shops belonged to a product of lower quality than advertised

**food fraud costs 49\$ billion/year!**

# Highlights in Recent Years

THE TIMES OF INDIA

## Govt wants NMR-testing mandatory for honey exported from India

Nov 24, 2019, 09:45 PM IST

New Delhi, Nov 24 () The government has asked the Export Inspection Council (EIC) that comes under the Commerce Ministry to make NMR-testing mandatory for honey exported from India to ensure quality as part of its efforts to boost outward shipments.

The Sydney Morning Herald

BUSINESS CONSUMER AFFAIRS FOOD SAFETY

## Fake honey scandal widens to Australian-sourced brands

By Adele Ferguson & Chris Gillett  
October 3, 2018 – 12:00am

153 View all comments

One in five samples of local honey sourced along the eastern seaboard of Australia, including boutique brands, has been found to be fake, deepening a global scandal over the impurity of honey.

## Riddle of how 1,700 tons of manuka honey are made... but 10,000 are sold

23 Aug, 2016 1:30pm

3 minutes to read



Research by the main honey producers' organisation in New Zealand has revealed that 1,700 tonnes of manuka are produced there each year. Photo / Brett Phibbs

## Tesco pulls honey off shelves amid purity concerns

25 November 2019

Share



Tesco has temporarily withdrawn pots of its own-brand honey amid concerns that it contains adulterated ingredients.

## Tesco agrees to withdraw 'fake' honey



Tests suggested that the honey was made from plant sugars.



## Fake honey still pouring into Canada, and local beekeepers are feeling the sting

Share

Canadian Food Inspection Agency found loads of fake honey cut with cheap syrup imported as 'pure honey'

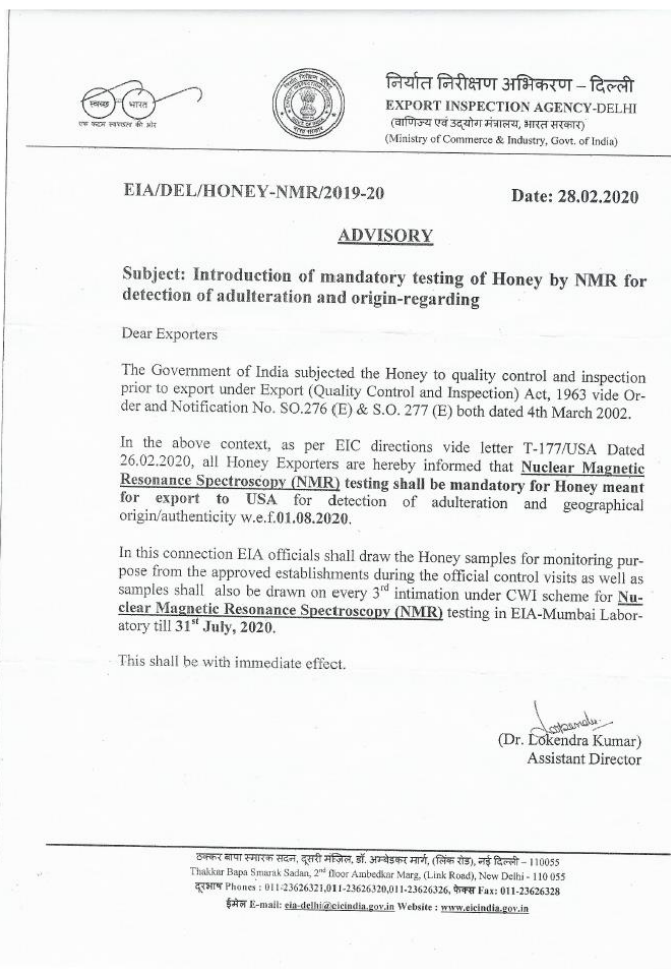
Ali Chakson · CBC News · Posted: Jul 25, 2019 4:00 AM ET | Last Updated: July 25, 2019







# Highlights in Recent Years Push Back by Governmental Agencies



**Newsnotes**

## U.S. CUSTOMS AGENCY TO PURCHASE NMR EQUIPMENT FOR TESTING OF HONEY IMPORTS

- June 1, 2020 - (excerpt)

**AHPA officials meet with Customs and Border Protection (CBP) and Congressional staff on new testing to prevent imported honey fraud**

April 20, American Honey Producers Association – Last month, two senior Customs and Border Protection officials provided details for its new program for stopping fraudulent honey imports to senior staff of the Senate Appropriations Committee and AHPA President Kelvin Adee and Legislative Co-Chair Mark Jensen. This program is designed to prevent country-of-origin (COO) fraud, whereby dishonest importers evade the steep antidumping (AD) duties owed on honey imports from China by falsely claiming what is actually Chinese honey was produced in another country. Led by Senators Tester (D-MT) and Hoeven (R-ND), Congress last year included funding for this program in CBP’s FY 2020 budget.

CBP’s Larry D. Fluty, Executive Director for the agency’s Laboratories & Scientific Services, reported CBP is purchasing a nuclear magnetic resonance (NMR) testing machine specially equipped for testing honey imports, and is developing the required honey testing protocols with Pacific Northwest National Laboratory. He also noted CBP would use both NMR and existing trace-mineral detection technologies in testing for false

FOOD FRAUD AND LATEST TECHNOLOGIES FOR ACCREDITED DETECTION

# Fraud Detection

---



# What are the most common Frauds in EMA's?

## Example: Honey

**Deliberate addition of sugar syrups**



**False declaration of origin (geographical & botanical)**



**Unripe honey**



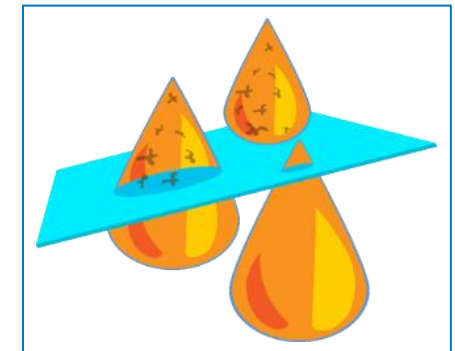
**Inappropriate Bee Feeding**



**Transshipment**



**Ultrafiltration**



# What Are The Conventional Techniques of Fraud

## Example: Honey

### False declaration of origin

- Hiding real country of origin or variety.
- Intend: Avoid higher tariff rules, avoid further testing, reach higher market value
- Transshipment = sending the honey to another country where it is relabeled before being exported.
- Pollen grains manipulation to deceive detection by pollen analysis
- Filtration of endogeneous pollen grains and addition of pollen grains from another country

### Floral source (e.g. Manuka)

Riddle of how 1,700 tons of manuka honey are made... but 10,000 are sold

23 Aug, 2016 1:30pm

3 minutes to read



Research by the main honey producers' organisation in New Zealand has revealed that 1,700 tonnes of manuka are produced there each year. Photo / Brett Phibbs

### Geo. origin (e.g. Transshipment)



# What Are The Conventional Techniques of Detection

## Example: Honey

### Presence of Foreign Sugars



- $^{13}\text{C}$ -IRMS &  $^{13}\text{C}$ -LC-IRMS
- HPLC (sugar composition)
- LC-MS (markers)
- Foreign Enzymes

### ■ Limitation:

- **markers can be removed**
- **Syrups can be purified.**
- **$^{13}\text{C}$ -IRMS can only detect C4 sugars (sugarcane, corn).**

### Country of Origin & Botanical Origin



- Microscopy (Pollen Analysis)

### ■ Limitation:

- **Pollen grains can be removed or added to the honey**

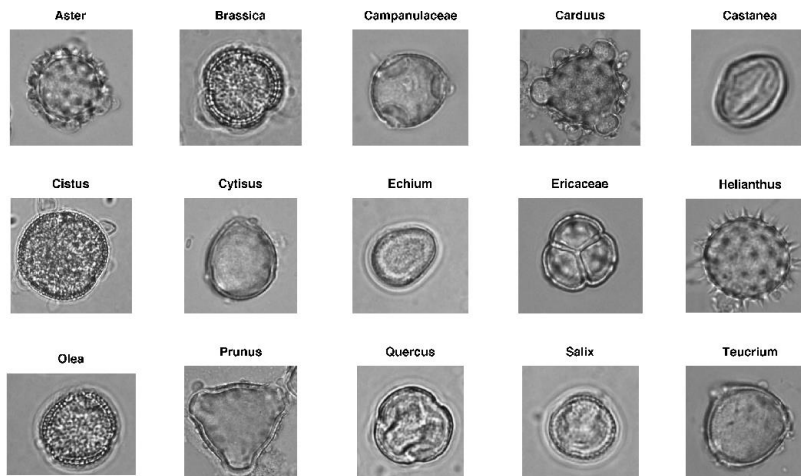
# What Are The Conventional Techniques of Detection

## Example: Honey

### Detection of false declaration of origin

#### Pollen analysis

- Identification and counting pollen grains by microscopy
- Requires a trained expert and is time consuming
- Not working on pollen-filtered honeys.
- Not able to differentiate the real pollens from exogeneous pollen grains.





# What Are The Types of Fraud

## Example: Honey

### Sugar syrups in honey

- According to EU-Directive of 2001 and Codex Alimentarius, honey should be exempt of foreign sugars.
- Foreign sugars can be:
  - a deliberate addition to honey in order to stretch the product.
  - a consequence of **bee feeding** with sugar syrups.
- Feeding bees with industrial sugar syrups:  $\approx$  40 times less expensive than feeding with honey.
- Sugar syrups need to be handled with care and be given outside of production periods.
- Famille Michaud, the first reseller of honey in France tests all the honeys they buy with their NMR instrument. **They find sugar syrups in 10% of the French honeys tested.**



[https://www.francetvinfo.fr/replay-jt/france-2/20-heures/video-les-abeilles-nourries-au-sirop-de-betterave\\_3521387.html](https://www.francetvinfo.fr/replay-jt/france-2/20-heures/video-les-abeilles-nourries-au-sirop-de-betterave_3521387.html)

# What Are The Conventional Techniques of Detection

## Example: Honey

---

- Pollen Analysis + Organoleptic Analysis → *botanical & geographical origin*
- $^{13}\text{C}$ -IRMS → *addition of syrup from  $C_4$ -plants (corn, cane sugar)*
- $^{13}\text{C}$ -LC-IRMS → *addition of syrup from  $C_3$ - and  $C_4$ -plants*
- Sugar Spectrum (HPLC) → *general sugar composition*
- SM-B & SM-R (LC-MS) → *marker for beet and rice sugar addition*
- Oligosaccharides (HPLC) → *residues of syrup production from starch*
- Foreign Enzymes (e.g. *amylase*) → *residues of syrup production from starch*



~ 1000 \$

~ 3 days

FOOD FRAUD AND LATEST TECHNOLOGIES FOR ACCREDITED DETECTION

# NMR: a Powerful Technique for Food Authenticity Control

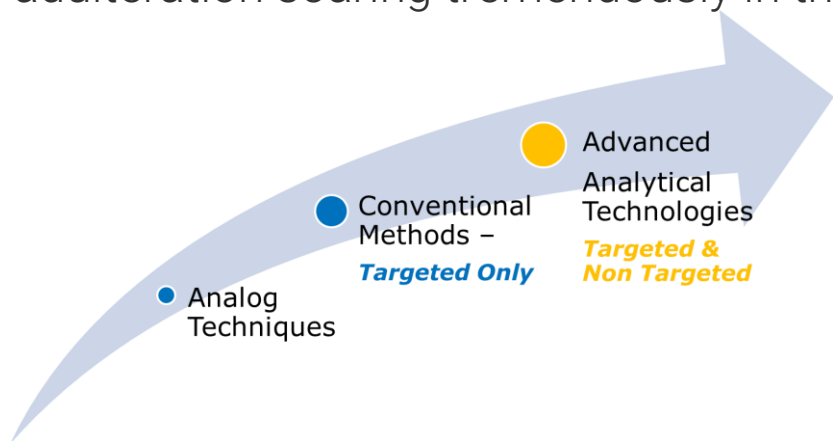
---



# Why is NMR Needed? How is NMR Different

## The need for multi-marker and non-targeted methods to tackle Dynamic Food Fraud

- Standard targeted methods more and more fail to detect fraud, as the fraudsters become more sophisticated and learn quickly how to deceive these tests.
- Costs of looking for specific markers of adulteration and the inability to keep up with the fraudsters has resulted in the amount of adulteration soaring tremendously in the last decade.





# Example

SAMPLES NUMBER	DETECTED ADULTERATED USING C3/C4 (LC-IRMS)	DETECTED ADULTERATED BY NMR
5035	80	508

SAMPLES NUMBER	DETECTED ADULTERATED USING FOREIGN OLIGOSACCHARIDES ELSD	DETECTED ADULTERATED BY NMR
2659	50	515

With the courtesy of





FOOD FRAUD AND LATEST TECHNOLOGIES FOR ACCREDITED DETECTION

# FoodScreener by Bruker

---

# FoodScreener™

## What is it?

---

**The Bruker FoodScreener is a completely automated, push-button, nuclear magnetic resonance (NMR) solution that includes analysis and reporting based on 400MHz.**

It is capable of simultaneous identification and complete quantification of compounds with minimum sample preparation at a reduced cost per sample. It is a dependable screening technique for targeted and non-targeted multi-marker analyses.

The FoodScreener enables the detection of unpredicted and even unknown fraud. Complex statistical models allow the testing of origin authenticity, production process control, false labeling, sample similarity, and species purity. The results are lab-, user- and system independent. Different NMR solutions for juice, wine and honey profiling are available



# FoodScreener™

## Solution for adulteration, authenticity and quality control of food

---

### Features

- Speed of Analysis
  - 20 minutes vs. 2 days
- Ease of Use:
  - SOP's / Fully automated / No need for an NMR expert
- Reduced Costs:
  - Ex. Honey: divided by 3 compared to usage of many conventional methods
- ISO 17025 Accreditation
  - :Ensuring a deeply validated method, relying on a huge Database of 18000 well-characterized samples



# FoodScreener™

## Solution for adulteration, authenticity and quality control of food

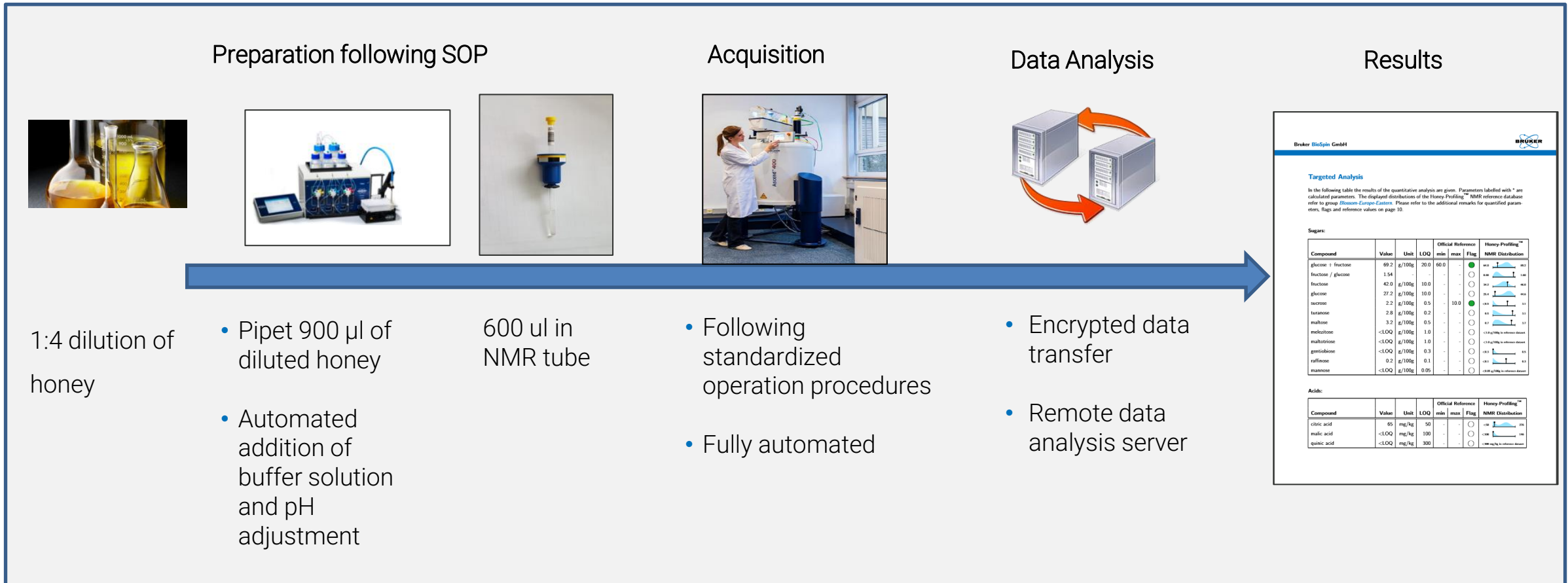
### Benefits:

- Centralized Database with worldwide network including governmental laboratories. *Goal: have a database of authentic material covering all its natural variations and to define common/harmonized purity criteria*
- Transparency about parameters used and related thresholds (purity criteria)  
*= No Black Box*
- Round tables have been kicked-off to discuss and harmonize purity criteria and interpretation, amongst honey experts
- A method which is continuously expanded and updated

# FoodScreener™

## Solution for adulteration, authenticity and quality control of food

### Workflow:





## Available Modules



### Honey-Profiling (NMR)

Enables honey producers to screen their samples for adulteration and authenticity, in order to ensure their brand's reputation.



### Olive Oil Profiling (NMR)

Strengthen your brand's image and ensure your product's quality and authenticity with NMR-based Olive Oil-Profiling



### Juice-Profiling (NMR)

For each fruit juice a multitude of parameters related to quality and authenticity is evaluated simultaneously from a single data set acquired within a few minutes.

And others upcoming such as Spices.



### Wine-Profiling (NMR)

This comprehensive solution offers a quick, fully automated and cost-efficient tool to ensure wine quality and authenticity, protecting brand integrity and consumer confidence

FOOD SCREENER

# Honey-Profiling 3.0 Database

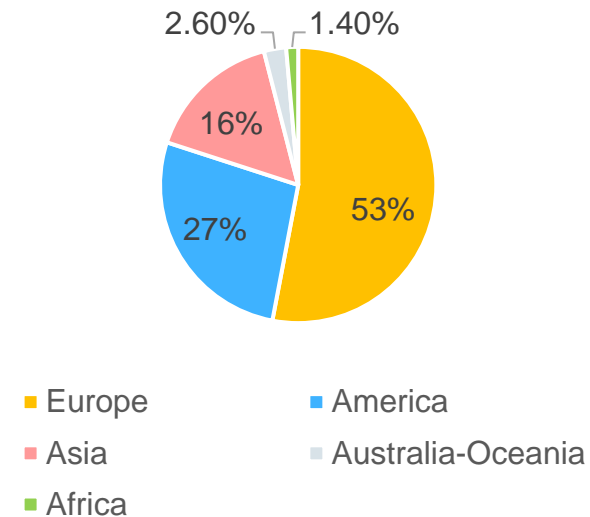
---

## Honey-Profiling 3.0 Database

- 28,500 authentic samples / >2000 adulterated samples
- > 50 countries of origin & 100 botanical varieties.
- Monofloral and polyfloral honeys from a single country source.
- Blends of honeys from different countries.
- 2.000 honey dew
- 1.500 industrial honeys (or “baker honey”)

### Samples per country in the Data Base:

- > 800: USA, Guatemala, Austria, El Salvador, Serbia, Tanzania...
- > 200: New Zealand, Brazil, Vietnam, Thailand, India, Turkey...
  - > 700: Spain, Germany, Mexico, Cuba, Romania, Chile...
  - > 1.000: China, Ukraine, Argentina, France...



# Honey-Profiling 3.0 Database

---

## Capabilities:

- **Detection of sugar syrups**
  - Better performance vs. conventional methods (10 x more adulteration detected)
  - 20% of adulterated samples pass all conventional methods, but are detected with NMR (Famille Michaud)
- **Verification of labeling of origin**
  - Only method that combines detection of sugar syrups and verification of origin
  - Method which is very difficult to counterfeit (compared to pollen analysis or single markers)
  - Method that can be applied to pollen-filtered honey
- **Detection of Atypical Samples**
- **Analysis of constituents and Regulated Parameters**



**Honey-Profiling (NMR)**

# Honey-Profiling 3.0 Database

## Detection of sugar syrups

- Quantitative analysis of many parameters: up to 60 parameters per sample
- The values are compared to the reference thresholds
- Reference thresholds have been determined and validated thanks to a large database of authentic and known adulterated honeys from various countries and botanical varieties

## Detection of Sugar Syrups

(Analysis-ID: HO-2000-02/0167)

Following tests have been applied in order to detect sugar syrups:

Nr	Type	Description	Result	Value	Limit	Out
1	Intensity/Ratio	3.263 (absolute quantitative)	passed	475	<1279	-
2	Intensity/Ratio	5.077 (absolute quantitative)	passed	154	>39	-
3	Intensity/Ratio	3.636 (absolute quantitative)	passed	2604	<4674	-
4	Intensity/Ratio	4.262 (absolute quantitative)	passed	74	>29	-
5	Intensity/Ratio	4.195 (absolute quantitative)	passed	128	<1200	-
6	Intensity/Ratio	5.271 (absolute quantitative)	passed	30.6	>5.6	-
7	Intensity/Ratio	4.280 (absolute quantitative)	passed	58	>20	-
8	Intensity/Ratio	5.113/(3.270-3.310)	passed	0.005	<0.036	-
9	Intensity/Ratio	4.496/(3.270-3.310)	passed	0.039	>0.012	-
10	Intensity/Ratio	5.334/(5.270-5.300)	passed	0.08	<0.13	-
11	Intensity/Ratio	3.546/(5.270-5.300)	passed	1.15	>0.62	-
12	Intensity/Ratio	3.740/(5.270-5.300)	passed	3.2	>1.2	-
13	Intensity/Ratio	3.857/(5.200-5.260)	passed	0.0145	>0.0037	-
14	Intensity/Ratio	4.150 (absolute quantitative)	passed	258	>115	-
15	Intensity/Ratio	5.181 (absolute quantitative)	passed	50	>24	-
16	Intensity/Ratio	4.055/(5.030-5.070)	passed	3	<46	-
17	Intensity/Ratio	1.809/(5.030-5.070)	passed	0.2	<1.0	-
18	Intensity/Ratio	3.708/(5.030-5.070)	passed	312	<872	-
19	Intensity/Ratio	6.765/(5.250-5.270)	passed	0.009	<0.046	-
20	Intensity/Ratio	2.200/(5.305-5.315)	passed	0.236	>0.019	-
21	Intensity/Ratio	3.326/(3.270-3.310)	passed	0.415	>0.034	-
22	Intensity/Ratio	4.037/(3.270-3.310)	passed	1.56	>0.73	-
23	Intensity/Ratio	4.006/(5.270-5.300)	passed	1.10	>0.70	-
24	Intensity/Ratio	3.564/(5.270-5.300)	passed	20.2	>10.0	-
25	Intensity/Ratio	5.388/(5.370-5.400)	passed	0.19	>0.13	-
26	Intensity/Ratio	3.524/(4.075-4.110)	passed	0.064	<0.070	-
27	Intensity/Ratio	3.182/(4.075-4.110)	passed	0.0024	<0.0045	-
28	Intensity/Ratio	3.785/(4.075-4.110)	passed	0.060	>0.036	-
29	Intensity/Ratio	3.857/(4.075-4.110)	passed	0.0093	>0.0021	-
30	Intensity/Ratio	4.267/(4.970-4.990)	passed	1.5	<4.7	-
31	Intensity/Ratio	4.276/(4.970-4.990)	passed	0.3	<5.4	-
32	Intensity/Ratio	4.204/(5.090-5.110)	passed	1.1	<5.7	-
49	Quantification	Fructose/Glucose	passed	1.12	>0.85 and <1.95	-
50	Quantification	Fructose+Glucose	passed	69.4	>40	-
51	Quantification	Turanose	passed	1.43	>0.3	-
52	Quantification	DHA(D) and Mannose(M)	passed	3 / 0.000	D<30 or M<0.05	-
53	Quantification	Sucrose	passed	0.2	<15	-



# Honey-Profiling 3.0 Database

## Quantitative analysis

### Regulated parameters in EU-directive / Codex Alimentarius

- Absolute quantification
- HMF, glucose+fructose, sucrose
- Conclusion according to directive

### Codex Alimentarius and EU-Directive 2001/110/EC:

Following parameters are required according to Codex Alimentarius and EU-Directive 2001/110/EC. The concentrations are obtained by direct quantification. Parameters labelled with \* are calculated parameters.

Compound	Value	Unit	LOQ	Official Reference		
				min	max	Flag
glucose + fructose *	69.4	g/100g	20.0	45	-	●
sucrose	<LOQ	g/100g	0.5	-	10	●
5-hydroxymethylfurfural (HMF)	<LOQ	mg/kg	5	-	80	●

Following flags are used according to Codex Alimentarius and EU-Directive 2001/110/EC:


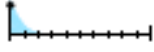

Compound	Flag	Concentration	Declaration	Interpretation
glucose + fructose	●	< 45 g/100g	All	Not compliant
	●	< 60 g/100g	Blossom	Not compliant for blossom honey
		≥ 60 g/100g	All	Compliant
		≥ 45 g/100g	Honeydew	Compliant for honeydew honey
●	≥ 45 g/100g, < 60 g/100g	Unknown	Compliant for honeydew honey and blends of honeydew honey with blossom honey. Not compliant for blossom honey.	





# Honey-Profiling 3.0 Database

## Quantification - Targeted Analysis

- Absolute quantification
- Sugars, organic acids, amino acids, freshness and quality criteria
- Comparison to reference values (according to Database samples)

Compound	Value	Unit	LOQ	Reference Range	Flag
alanine	12	mg/kg	5	<5  24	●
aspartic acid	<LOQ	mg/kg	150	<150 mg/kg in reference dataset	●
glutamine	<LOQ	mg/kg	200	<200 mg/kg in reference dataset	●
leucine	<LOQ	mg/kg	40	<40  132	●
proline	180	mg/kg	150	237  891	●
valine	<LOQ	mg/kg	10	...	●

Compound	Value	Unit	LOQ	Reference Range	Flag
citric acid	152	mg/kg	50	<50  370	●
malic acid	962	mg/kg	100	<100  114	●
quinic acid	<LOQ	mg/kg	300	<300 mg/kg in reference dataset	●

In the following table the results of the quantitative analysis are given. The concentrations are obtained by direct quantification. Parameters labelled with \* are calculated parameters. The reference range is derived from the *India Blossom* samples in the Honey-Profiling Database. The reference range bases on 105 samples.



# Honey-Profiling 3.0 Database

## Detection of false declaration of origin

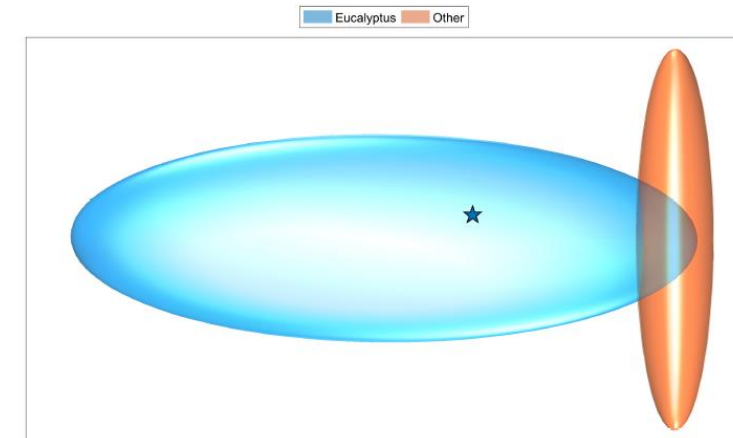
- Based on the **complete chemical composition of the honey** observed by  $^1\text{H-NMR}$
  - Statistical analysis of the NMR spectrum: untargeted buckets/variables.
  - Differentiation of the supposed/declared origin or variety with all other ones present in the Database (50 countries / 100 varieties).
  - Statistical models have been validated by Monte Carlo cross validation (**Criteria: TP > 98%**)
- *Very difficult/ impossible to deceive*
  - *No need for an expert and fast*
  - *Applicable to pollen-filtered honey*

**Statistical Model:** Variety Eucalyptus

(Analysis-ID: HO-1125-01/0025)

This model is based on 15825 samples, thereof 170 samples of reference group *Eucalyptus*.

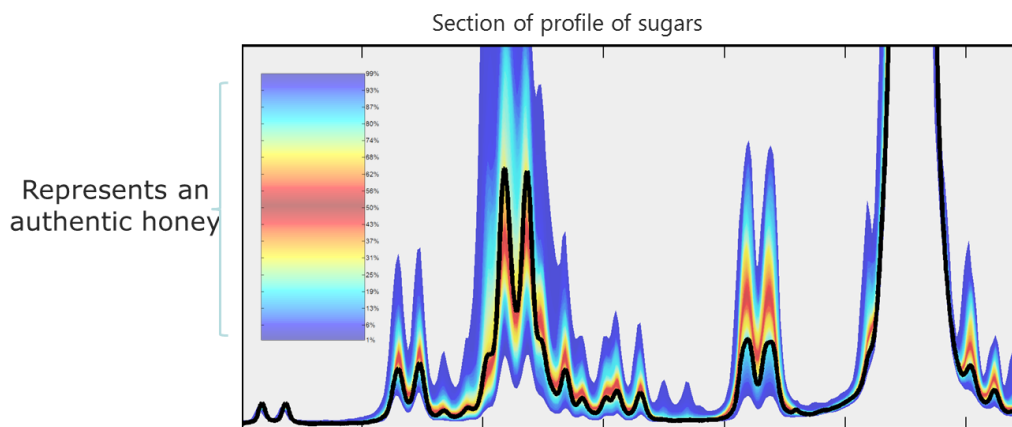
**Result:** Consistent with declared variety *Eucalyptus*. The probability of consistency is 100.0%.



# Honey-Profiling 3.0 Database

## Detection of atypical samples

- Statistical comparison of the NMR profile with “normal” profiles from the same floral type
- Potential to detect new frauds at early stage.



## Non-Targeted Verification Analysis

### Univariate Verification

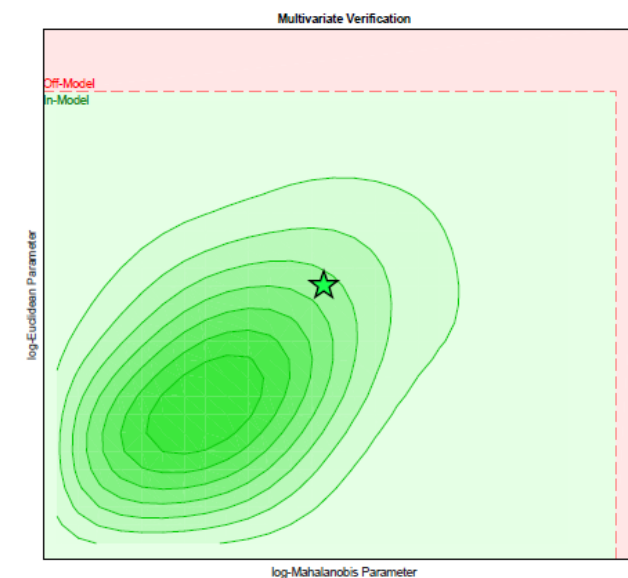
**Applied Model:** Eucalyptus

**Result:** No deviation was detected in univariate verification (In-Model).

### Multivariate Verification

**Applied Model:** Eucalyptus

**Result:** No deviation was detected in multivariate verification (In-Model).



FOOD SCREENER

# Summary

---

# Overview Techniques for Honey Testing– Sugar syrups

## Detection of sugar syrups varieties

	FOREIGN ENZYMES/ SYRUP MARKERS	EA-IRMS	EA/LC-IRMS	NMR	HR-MS
TYPE OF SUGAR SYRUPS DETECTED	1 specific syrup or enzyme	C4 only	C3 and C4	C3 and C4	C3 and C4
IDENTIFICATION OF SUGAR SYRUP USED	Yes	Not possible	Not possible	Not possible	Not possible
QUANTIFICATION of proportion of sugar syrup		Yes	No	No	No
AOAC OFFICIAL METHOD	No	Yes	No	No	No
HARMONIZATION	-	Yes	No	Yes	No
PERFORMANCE for syrups detection	Very low	~ 25% of adulterations detected**	~ 80% <b>less than NMR*</b>	~ 70% of adulterations detected**	Supposed to be <b>higher than NMR</b>
ORIGIN VERIFICATION	No	No	No	Yes	No

## Summary FoodScreener

---

An **all-in-one method**, comprehensive authenticity test: multi-marker and non-targeted

**deeply validated method**, relying on a huge Database of 18000 well-characterized samples

No NMR expertise required. Easy to operate, fully automated, no regular instrument cleaning

Fast: 20 – 25 min / sample

Difficult to deceive

Wide and worldwide network of partners and users

# Summary FoodScreener – Worldwide Adoption

- Exporters
- Importers
- Honey Packers
- Retailers
- Beekeepers Associations
- Commercial Service Providers
- Governmental Labs
- Hopefully the Arabic Branch of the AOAC

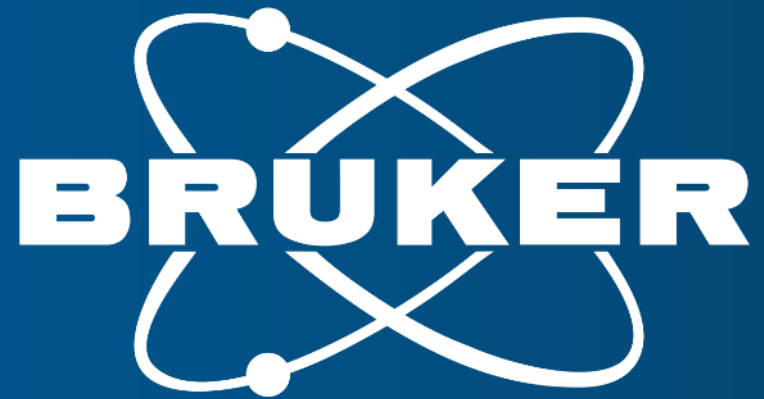


Famille Michaud  
APICULTEURS DEPUIS 1920



CallaghanInnovation  
New Zealand's Innovation Agency





Innovation with Integrity