The European Commission's science and knowledge service

Joint Research Centre

European Commission

Results of honey authenticity testing by liquid chromatography-isotope ratio mass spectrometry

JRC-Directorate F:

Health, Consumers and Reference Materials

Alain MAQUET

CDG Animal Products – Beekeeping sector, Bruxelles, BE– 21/11/2017

Fraud cases

TOP STORY

INTELLECTUAL PROPERTY RIGHTS

06/30/2016

HSI Chicago seizes nearly 6 imported from China

CHICAGO — Special agents with U.S. Immigra Security Investigations (HSI) seized nearly 60 t was destined for U.S. consumers.

The smuggled honey was contained in 195 55-g from Vietnam to evade anti-dumping duties app

Le miel : u colossale... e n'arrive p

Par lefigaro.fr, AFP agence | Mis à jo

LE FIGARO PREMIUM 1€ le premier mois 101 commenta

Pour pouvoir répondre à la demande, le produits frauduleux : faux étiquetage, o sucre... L'exportation de miel dans le m

«Le miel adultéré (modifié ou falsifié), c apicole, plus que les pesticides ou les p professeur argentin Norberto Garcia, invité au 21e Congrès de l'Union nationale de l'apiculture française (Unaf), à Clermont-Ferrand. M. Garcia, qui est également le président de l'Organisation internationale des exportateurs de miels, a constaté que les exportations de miel ne cessent de croître: alors que le nombre de ruches a augmenté dans le monde de 8% de 2007 à 2013, l'exportation de miel dans le monde a augmenté de 61%.

R.it FIRENZE

Home | Cronaca | Sport | Tempo Libero | Foto | Ristoranti | Annunci Lo

Miele »

Caccia al miele degli dei: l'ultima corsa all'oro della Nuova Zelanda

Via la stanchezza di fine inverno, con i "fantastici 4" che danno

In Toscana aumentano le frodi sul miele

di Maurizio Melani

Lo leggo dopo | 03 febbraio 2016

Il settore agroalimentare italiano, per l'importanza strategica che ricopre e l'appeal sul mercato estero, è sottoposto a continui tentativi di frode. La palma d'oro va naturalmente al vitivinicolo e al settore oli e grassi che staccano tutti gli altri. Miele compreso. Qualche dato? Nel 2015 l'ICQRF, Ispettorato Centrale della tutela della Qualità e Repressione Frodi, organismo con attività ispettiva, analitica e sanzionatoria, ha effettuato quasi 34.000 controlli analizzando circa 9.000 campioni. Il tasso di irregolarità si attesta sul 9,5%. Leggermente più bassa la media nel comparto miele col 9,2%. Dato rispecchiato anche in Toscana.

European Commission

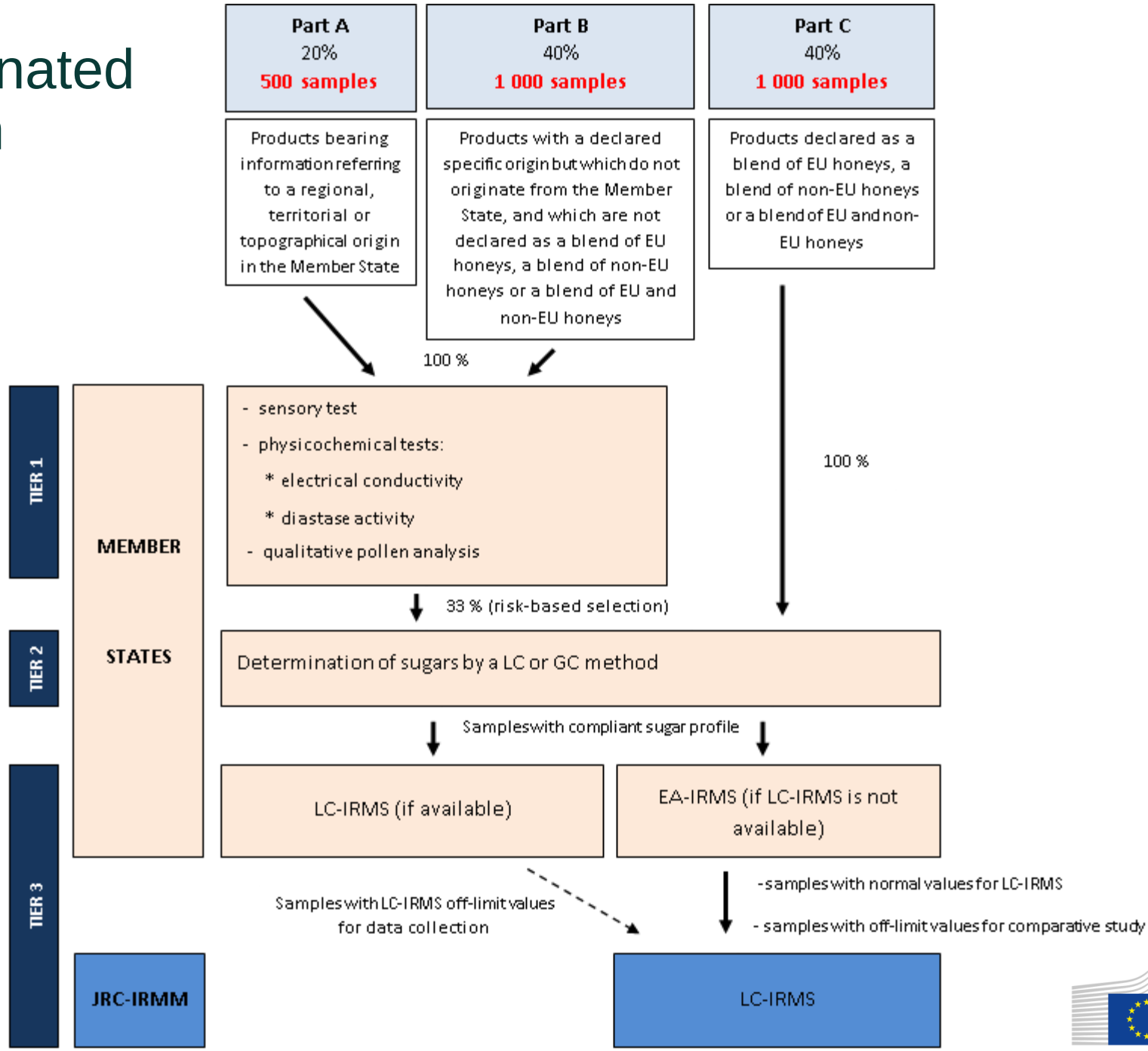
Coordinated control plan on authenticity of honey to detect fraudulent practices

- honey mislabelled with regard to its geographical and/or botanical origin;

- products declared or presented as honey although containing **exogenous sugars or sugar products**

European Commission

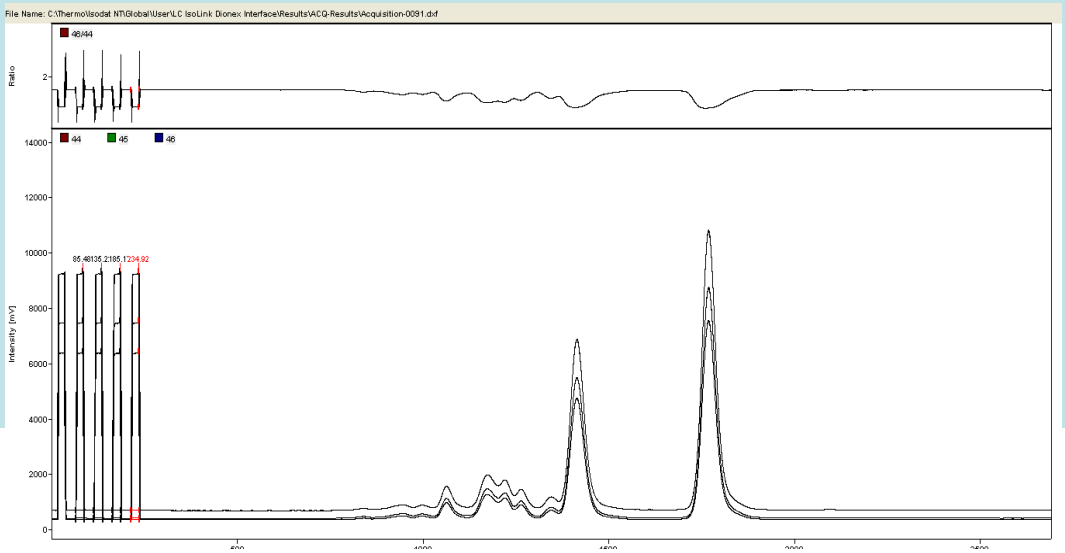
Proposed coordinated control plan



European Commission

JRC's contribution

- Analyse the samples received from the Member States by the in-house validated Liquid Chromatography – Isotope Ratio Mass Spectrometry (LC-IRMS) method and interpret the results on the basis of published authenticity criteria.



European Commission

Sampling by the Member States (plus Norway & Switzerland)

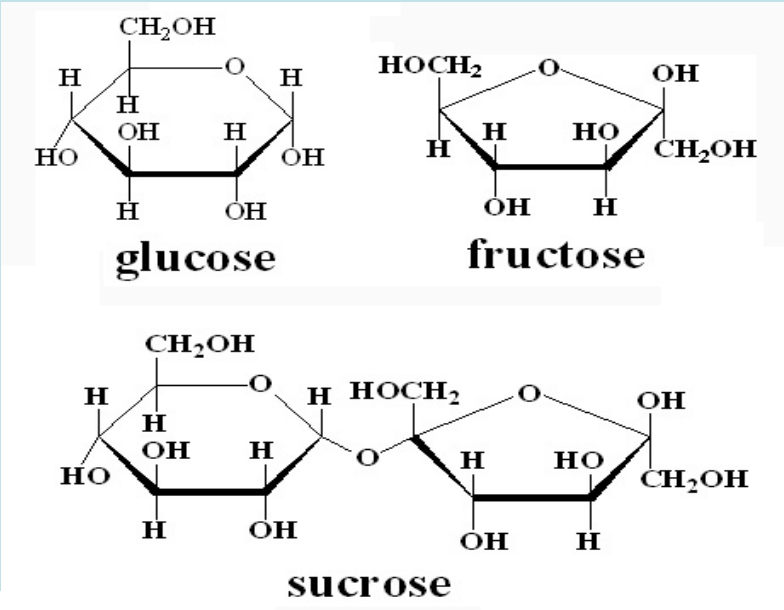
Source type	Samples collected	
Border inspection	35	1.5%
Distributor	157	6.9%
Importer	63	2.8%
Packaging companies	134	5.9%
Processor	81	3.6%
Producer	152	6.7%
Retailer	1010	44.6%
Storage companies	60	2.7%
Wholesaler	81	3.6%
Unknown	491	21.7%
Total	**2264**	**100.0%**

Sampling by the Member States (plus Norway & Switzerland)

Samples	Number
Collected by Member States (plus Norway and Switzerland)	2264
Sent to JRC and analysed by LC-IRMS	1069
-) of which without meta-data	38
-) non-compliant by applying the tests of Tiers 1 and 2 and EA-IRMS in the Member States	138
-) compliant by applying the tests of Tiers 1 and 2 and EA-IRMS in the Member States	**893**

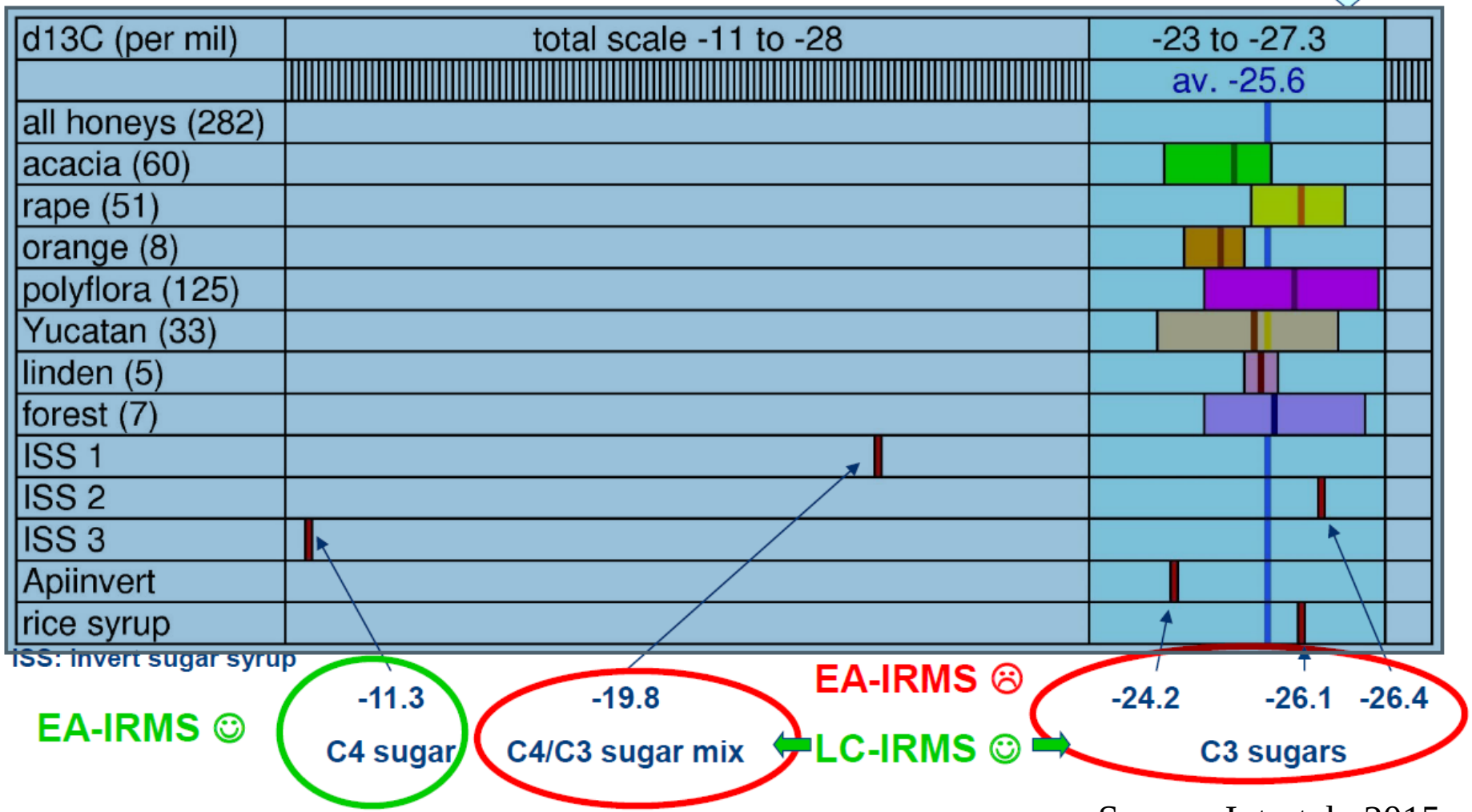
Honey composition

- Water ~ 18%
- Fructose ~ 31 - 49%
- Glucose ~ 23 - 41%
- Disaccharides (i.e. sucrose) ~ 0.2 - 10%
- Oligosaccharides 3-5% including:
  - trisaccharides (melezitose, raffinose, erlose, etc.)
  - traces of oligosaccharides with higher DP
- Other ingredients up to 6% including anti-oxidants (flavonoids), organic acids, minerals, proteins and amino-acids



European Commission

not all sugar syrups are outside the naturally occurring range of honey



Source: Intertek, 2015

Benchmark purity criteria – EA/LC-IRMS

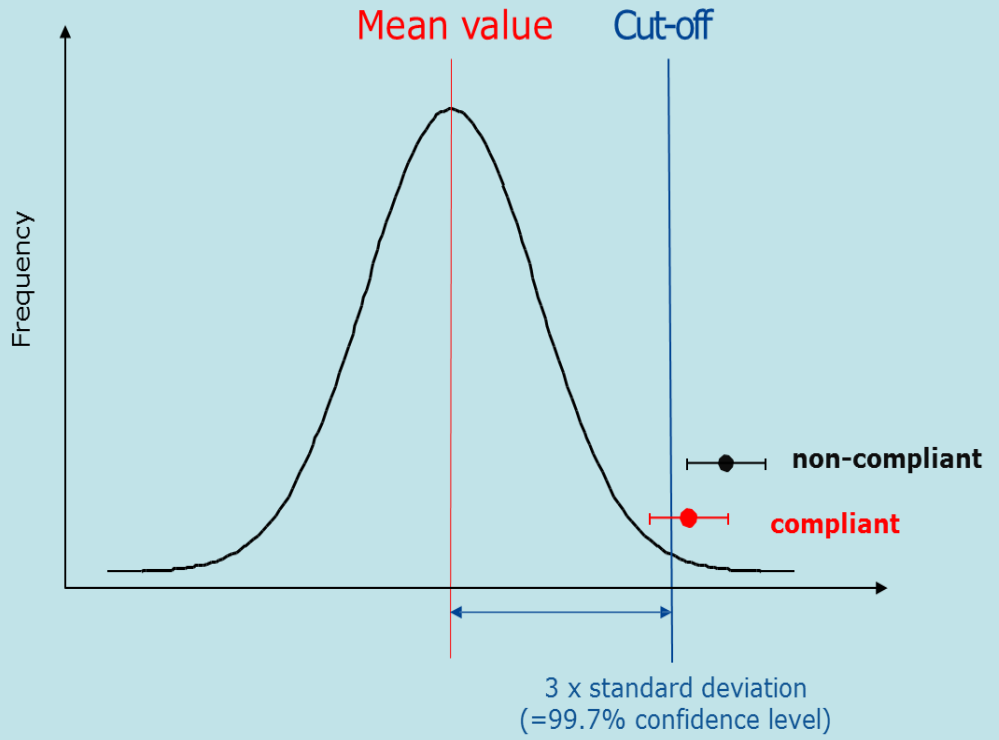
Raezke (Intertek, 2015) & Elflein method (eFood Lab, 3/2015)

parameter	average	range	purity limit
δ ^{13}C (‰) protein (p)	-25.2 ± 1.0	-22.3 to -28.9	none (depending on honey type)
δ ^{13}C (‰) honey (h)	-25.3 ± 1.0	-22.4 to -29.3	none (depending on honey type)
Δ p-h (‰)	0.2 ± 0.6	-1.2 to 2.1	none (refer to C4 sugar limit)
C4 sugar (%) *	1.0 ± 1.7	0 to 6.9	value < 7% (AOAC)
δ ^{13}C (‰) fructose (fru)	-25.2 ± 1.0	-22.2 to -29.1	none (depending on honey type)
δ ^{13}C (‰) glucose (glu)	-25.4 ± 1,0	-22.4 to -29.7	none (depending on honey type)
δ ^{13}C (‰) disaccharides (ds)	-25.8 ± 1.2	-21.7 to -29.9	none (depending on honey type)
δ ^{13}C (‰) trisaccharides (ts)	-24.8 ± 1.3	-21.2 to -29.4	none (depending on honey type)
Delta δ ^{13}C (‰) (fru – glu)	0.2 ± 0.3	-0.99 to +0.99	-1.0 < value < +1.0
Delta δ ^{13}C (‰) (max.)	0.3 ± 1.3	-2.10 to +2.10	-2.1 < value < +2.1
fru/glu ratio	1.21 ± 0.17	0.80 to 1.84	none (depending on honey type)
ds (area %)	5.3 ± 2.4	0.7 to 18.3	none (depending on honey type)
ts (area %)	1.1 ± 1.9	0.7 to 30.2	none (depending on honey type)
oligosaccharides (area %)	< 0.7	-	value < 0.7 %

N= > 20,000 honeys (Intertek's database)

Measurement uncertainty
in dubio pro reo

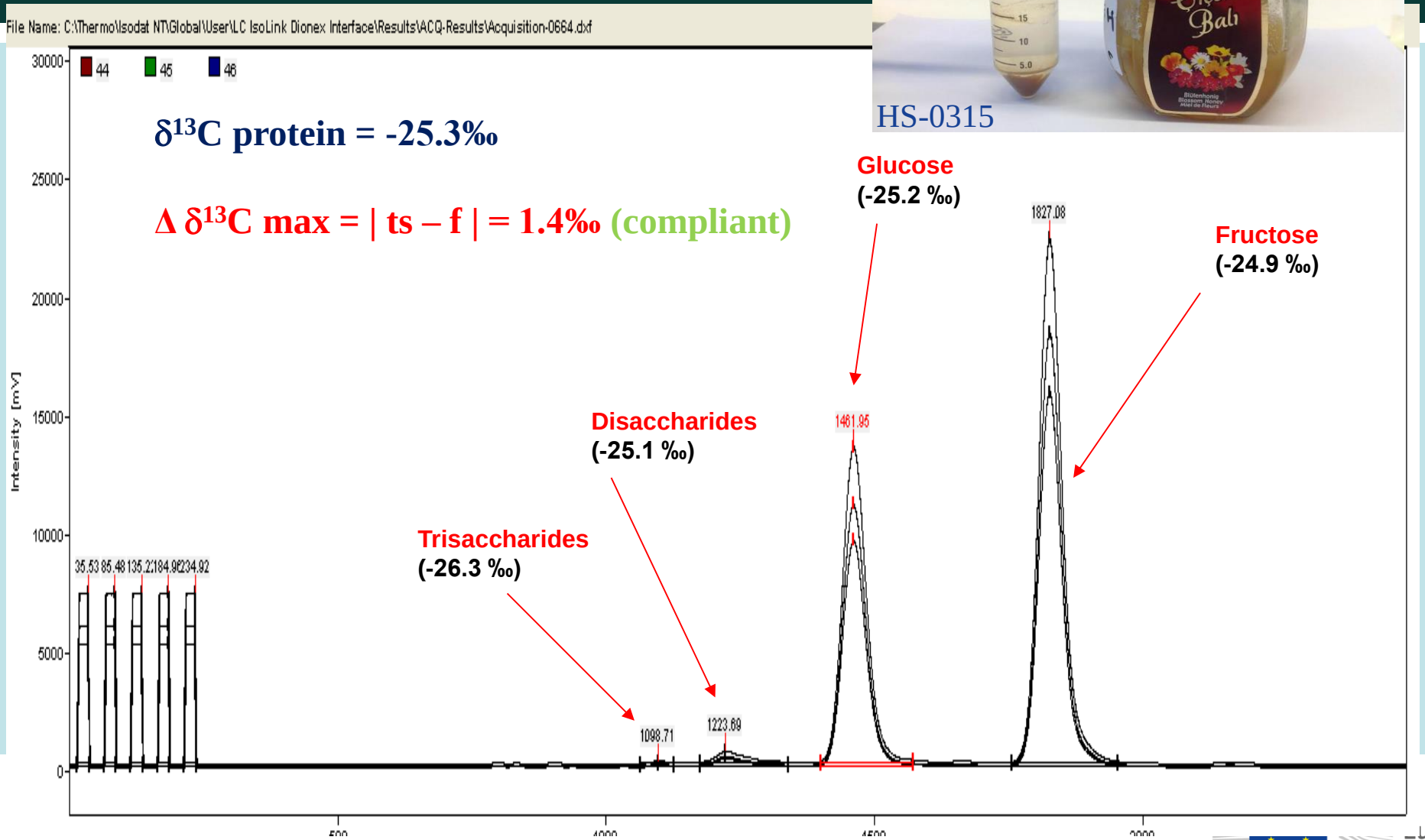
Parameter	Std dev.
$\delta^{13}C_{protein}$	0.16‰
$\delta^{13}C_{fructose}$	0.11‰
$\delta^{13}C_{glucose}$	0.12‰
$\delta^{13}C_{disaccharides}$	0.18‰
$\delta^{13}C_{trisaccharides}$	0.23‰
Percent peak area oligosaccharides	0.66%



$$U = 2 * \sqrt{SD^2(a) + SD^2(b)}$$

European Commission

Compliant polyfloral honey



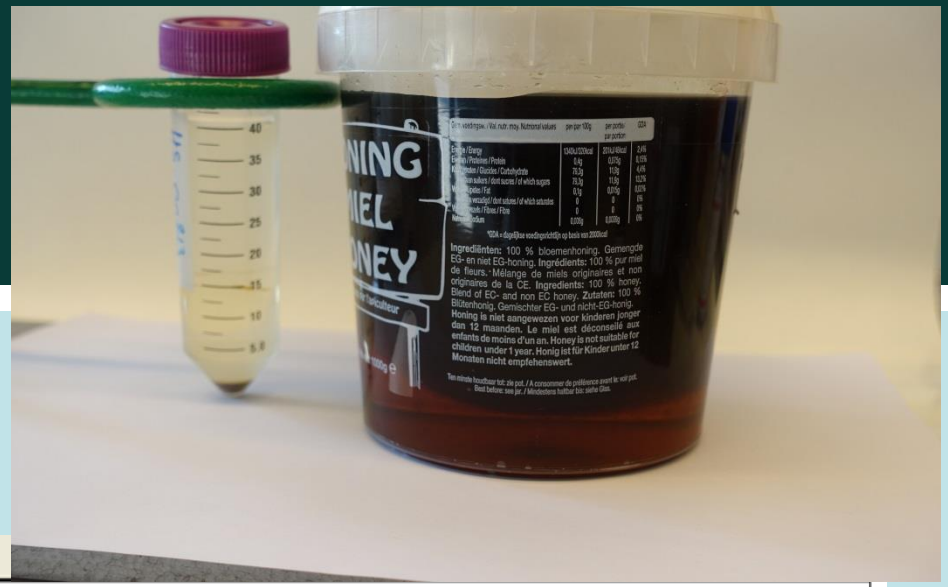
European Commission

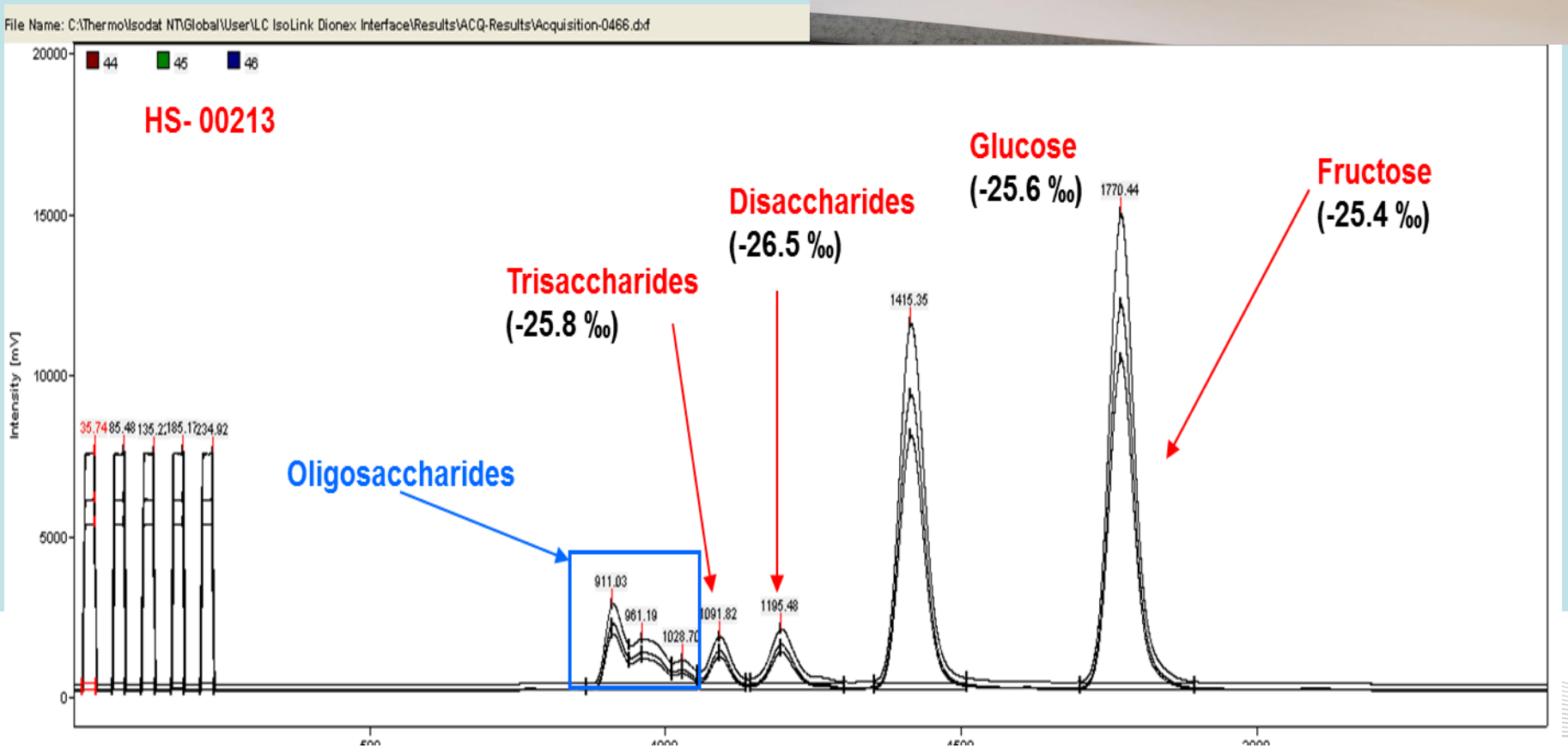
Suspicious honey

$\delta^{13}C$ protein = -29.3‰

$\Delta\ \delta^{13}C$ max = | f – p | = 3.9‰

Oligosaccharides peak area = 12% (suspicious)

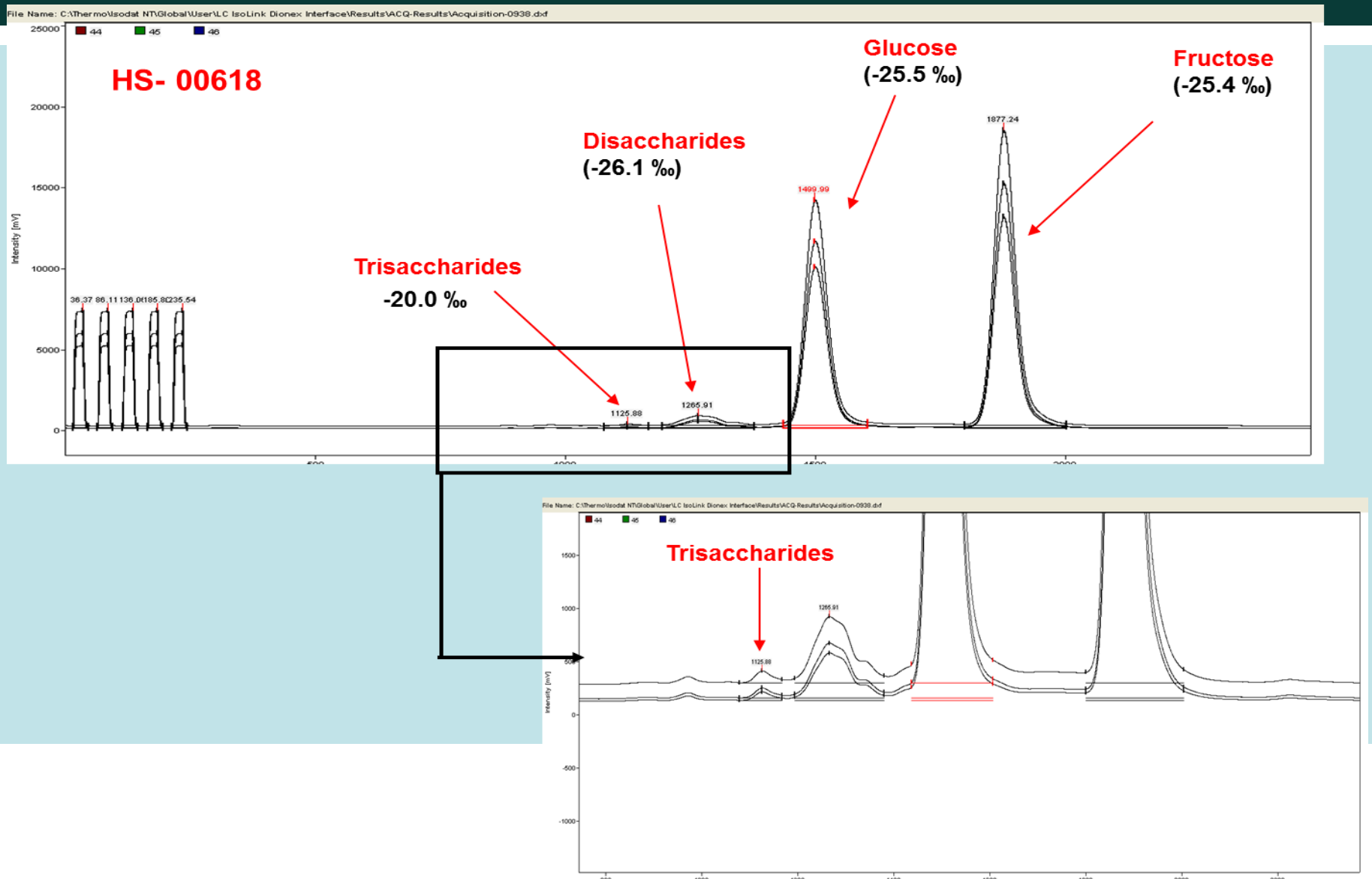




Preliminary conclusions on adulteration experiments using the LC-IRMS

Source adulterant	Potential detection limit	Pre-condition
C4 sugar	> 1%	
C3 sugar	> 10%	Oligosaccharides present

Issues with trisaccharide: peaks with low intensities



European Commission

Results

Origin	Samples (n)	Suspicion of non-compliance	
		(n)	(%)
Blend of EU honeys	96	19	19.8
Blend of EU and non-EU honeys	426	40	9.4
Blend of non-EU honeys	30	3	10.0
Single EU Member State	275	53	19.3
Single non-EU country	55	11	20.0
Unknown	11	1	9.1
TOTAL	**893**	**127**	**14.2**

Results

Category	Samples (n)	Suspicion of non-compliance	
		(n)	(%)
Border	4	0	0
Distributor	106	8	7.6
Importer	21	2	9.5
Packager	29	4	13.8
Processor	36	3	8.3
Producer	51	5	9.8
Retailer	563	92	16.3
Storage	22	3	13.6
Wholesaler	56	10	17.9
Unknown	5	0	0
TOTAL	893	127	14.2

Recommendations

- Harmonization of analytical methods
- Biobank of honeys, sugar syrups and bee feeding products
- European honey reference database
- Validation of emerging analytical methods

Ways forward

- Organisation of a technical meeting at JRC-Geel (BE) to collect ideas around gaps in knowledge and how to plug them (11th January 2018)

- Organisation of an interlaboratory comparison exercise on EA/LC-IRMS

→ Transfer the knowledge to the European Commission for further actions to reduce honey adulteration

Interested? Please contact alain.maquet@ec.europa.eu

Any questions?

European Commission

Stay in touch

EU Science Hub:
ec.europa.eu/jrc

Facebook:
EU Science Hub – Joint Research Centre

Twitter:
@EU_ScienceHub

LinkedIn:
Joint Research Centre

YouTube:
EU Science Hub

European Commission