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EU Coordinated action to deter certain fraudulent practices in the honey sector

Analytical testing results of imported honey

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Abstract

In the frame of the EU coordinated action 15 EU Member States (BE, BG, CZ, DE, DK, EL, ES, FR, HU, IE, IT, LT, PL, RO, SE) plus CH and NO randomly sampled 320 honey consignments originating from 20 exporting countries, which were sent to JRC for analysis to detect the presence of exogenous sugar syrup in honey.

Several methods (EA/LC-IRMS, HPAEC-PAD, ^{1}H-NMR) were used by the JRC to detect markers indicative for the presence of exogenous sugar syrups.

Of the 320 samples received from the competent authorities of the participating countries, 147 (46 %) were suspicious of being non-compliant with the provisions of the EU Honey Directive 2001/110/EC.

The suspicion rate was considerably high in comparison to an earlier EU-wide coordinated control plan conducted in 2015-17, where 14 % of the analysed samples did not comply with established benchmark criteria to assess honey authenticity. However, a different set of methods with improved detection capability was used in the present study, which may explain this difference.

Stable carbon isotope ratio analysis by EA-IRMS (AOAC method 991.41), a method that has frequently been used in the past to detect sugar syrups made of maize starch or sugarcane, was not effective in detecting honey suspicious of being adulterated. This is a clear indication that such sugar syrups are no longer used to extend honey and have been replaced by syrups made mostly from rice, wheat or sugar beet.

The highest absolute number of suspicious consignments originated from China (66 out of 89, 74 %), although honey originating from Turkey (14 out of 15, 93 %) had the highest relative proportion of suspicious samples. Honey imported from the United Kingdom had an even higher suspicion rate (10 out of 10, 100 %). However, the available traceability information suggests that this could be the result of honey produced in other countries and further processed in the United Kingdom before its re-export to the EU.

Although a substantially high number of honey consignments imported into the internal market was tested, the obtained results represent the situation during the sampling period (10/2021 to 02/2022) and shall not be generalised or extrapolated to other situations.

1 Context

Honey is a natural product which has been valued for its sweetening properties since ancient times. The provisions of the EU Honey Directive[1] aim at preserving the purity of honey as an unprocessed raw agricultural product, excluding modifications to its chemical composition. In the EU, market demand for honey is higher than domestic production and a substantial amount of honey is imported. Unfortunately, not all honey placed on the market is genuine. The results of a Coordinated Control Plan organised in 2015-17 at the EU level plus Norway and Switzerland, showed that at least 14 % of the checked samples did not conform to purity benchmarks[2]. As a follow-up the JRC organised a Round Table Discussion with stakeholders of the honey production chain and representatives of the competent authorities in the Member States as well as from European Commission services to identify gaps in knowledge related to authenticity testing of honey and possibilities to address them[3]. Extension of honey with sugar syrups was seen as the most widely observed malpractice.

Producer organisations[4] as well as consumer protection associations[5] have repeatedly raised concern over the presence on the market of honey not complying with regulatory requirements. These concerns are supported by the number of notifications regarding adulterated honey in the Administrative Assistance and Cooperation – Food Fraud system in 2020[6].

The EU average unit value for imported honey was 2.32 €/kg (excluding honey from New Zealand) in 2021[7], whereas sugar syrups made from rice are available at around 0.40 – 0.60 €/kg, depending on the purity of the syrup. The price difference between authentic honey and sugar syrups and the difficulty of detecting extension of honey with syrups provides attractive fraud opportunities for dishonest business operators. Various sources indicated that particularly imported honey does not comply with provisions of the EU Honey Directive 2001/110/EC, specifically with the requirement that honey shall not have added to it any food ingredient, including food additives, nor shall any other additions be made other than honey (Annex II, Composition criteria for honey).

Therefore, an action at EU level was organised to gather intelligence on the incidence of non-conforming honey imported into the Union through sampling and analysis that served to target further investigations within the EU. The action was initiated and coordinated by the Directorate-General for Health and Food Safety (DG SANTE) of the European Commission and implemented by the members of the EU Food Fraud Network who received the technical assistance of the Joint Research Centre and the support of the European Anti-Fraud Office (OLAF).

2 Scope of the EU coordinated action

The EU Food Fraud Network agreed to focus in the first phase of the coordinated action on imported honey through sampling at border control posts (BCPs) and analysis of the samples by the JRC. In case that a consignment presented to BCP control consisted of more than one production lot, only one lot was subjected to sampling. Several aliquots from a lot had to be taken to form an aggregate sample which had to be sent to JRC for analysis. Each aggregated sample had to be identified with the corresponding Common Health Entry Document (CHED)[8] reference number of the sampled consignment. This allowed retrieving all necessary

[1] Council Directive 2001/110/EC of 20 December 2001 relating to honey. https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32001L0110&from=EN

[2] https://ec.europa.eu/food/safety/agri-food-fraud/eu-coordinated-actions/coordinated-control-plans/honey-2015-17_en (accessed 24/06/2022)

[3] Technical Round Table on Honey Authentication, JRC-Geel, Belgium, 25 January 2018 Ares(2018)1677606 - 27/03/2018

[4] Copa-Cogeca Position Paper on the European Honey Market. Action Plan to Rectify the Alarming Situation. Brussels, February 2020 https://www.copa-cogeca.eu/downloadThread.aspx?threadID=2153212 (accessed 24/06/2022)

[5] Un miel correct pour moins de 6€ Test Achats - avr. 2021 - N° 662 https://www.test-achats.be/sante/alimentation-et-nutrition/aliments-et-complements-alimentaires/news/miel-authenticite (accessed 24/06/2022)

[6] 2020 Annual Report – The EU Agri-Food Fraud Network and the Administrative Assistance and Cooperation System. https://ec.europa.eu/food/system/files/2021-09/ff_ffn_annual-report_2020_1.pdf

[7] https://ec.europa.eu/info/sites/default/files/food-farming-fisheries/animals_and_animal_products/documents/market-presentation-honey_en.pdf

[8] The Common Health Entry Document (CHED) is a mandatory document that must be presented at border control post in order to carry out security checks when these goods enter the EU market: animals, products of animal origin, products of plant origin, feed and food products.

control and traceability data, including the place of first destination of the consignment within the EU, from the TRACES[9] system.

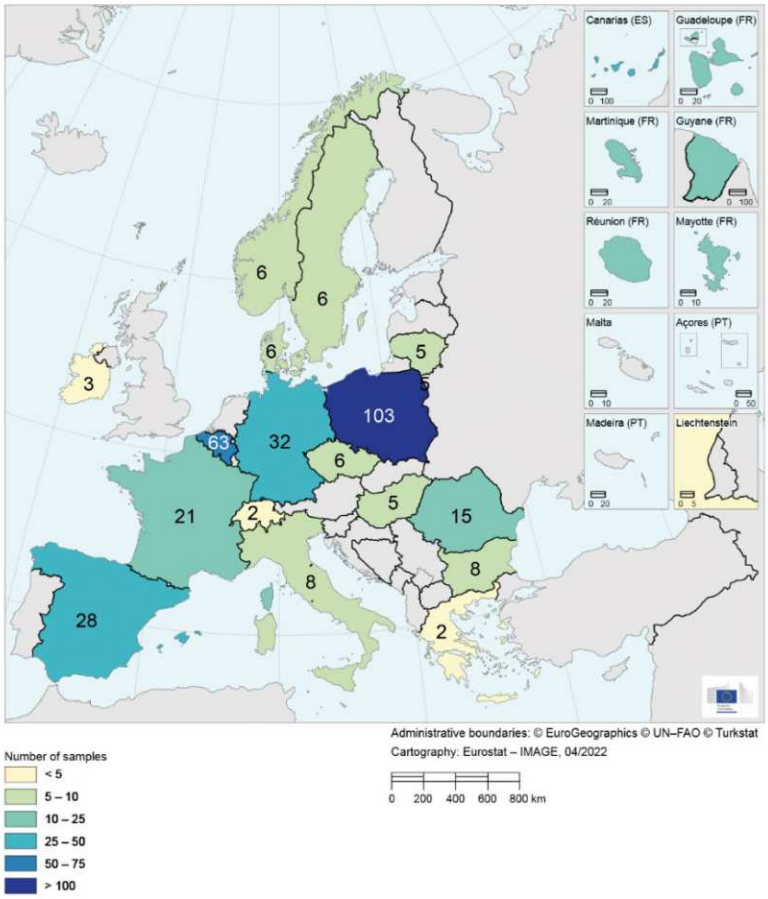
Detection of extension of honey with sugar syrup(s) and related labelling issues was the primary aim of the coordinated action. Other means of adulterating or misdescribing the true nature of honey, e.g. related to geographic or botanical origin, were outside the scope of the action.

The JRC had to analyse the honey samples sent by the participating countries in the coordinated action using stable carbon isotope ratio isotope analysis, which would allow detecting adulteration with sugar syrups produced from maize or sugarcane at levels more than 1 %, and at levels of more than 10 % for syrups produced from rice, wheat or sugar beet. In addition, for the detection of addition of sugar syrups obtained from rice or wheat a profiling method targeting the presence of exogenous oligo- and polysaccharides and other adulteration markers in honey had to be employed.

3 Participation

Fifteen EU Member States (BE, BG, CZ, DE, DK, EL, ES, FR, HU, IE, IT, LT, PL, RO, SE) plus CH and NO participated in the coordinated action. They sampled in total 320 consignments of honey (**Figure 1**). Based on the information reported by the competent authorities the majority of samples was taken at BCPs (84 %); the rest either at the premises of honey packers/distributors or unknown. JRC received the samples between 28/10/2021 and 11/02/2022 for laboratory analysis.

Figure 1. Participation in the EU coordinated action to control imported honey.



[9] TRACES is the European Commission's online platform for sanitary and phytosanitary certification required for the importation of animals, animal products, food and feed of non-animal origin and plants into the European Union, and the intra-EU trade and EU exports of animals and certain animal products.

4 Origin of honey consignments and trade flow

The majority of sampled honey consignments originated from China (89), Ukraine (74), Argentina (34), Mexico (22), Brazil (18) and Turkey (15) (**Figure 2**) and most of them were declared as 'polyfloral' (77 %) or 'monofloral' honey (11 %); the rest was of unknown botanical origin. The consignments were randomly sampled and offered a good representation of the trade flows during the sampling campaign. **Figure 3** informs about the complex trade flows of honey from exporting countries to the place of first destination of the sampled consignments in the EU. Flow patterns vary to a large degree; whereas honey imported by Poland stayed in the country, a larger part of honey imported by Germany was re-exported to other EU Member States.

Figure 2. Geographical origin of sampled honey consignments.

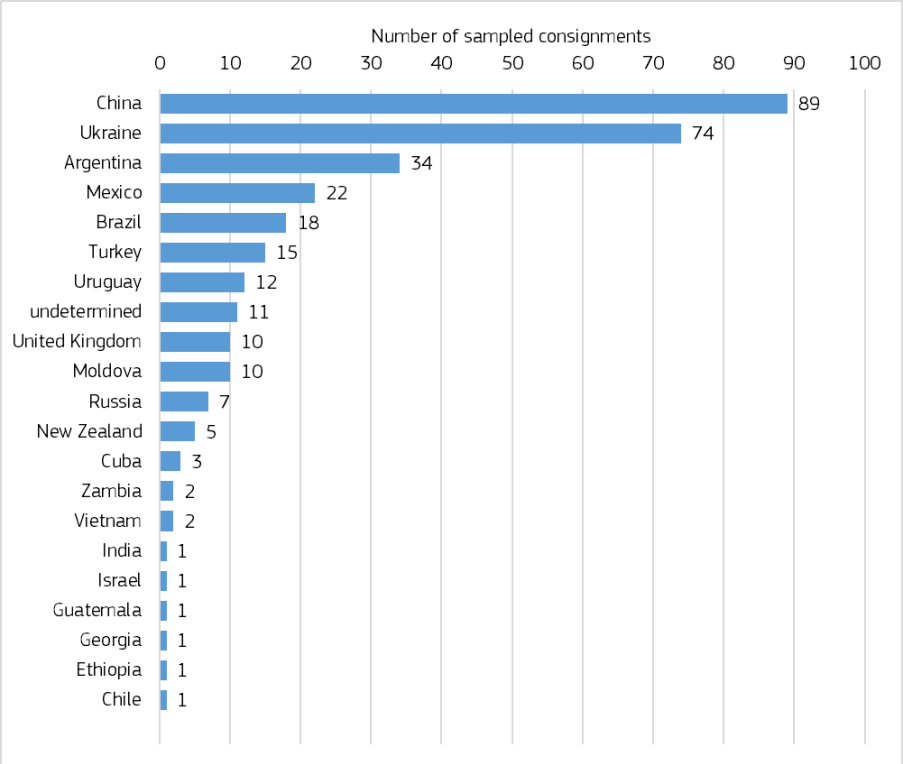
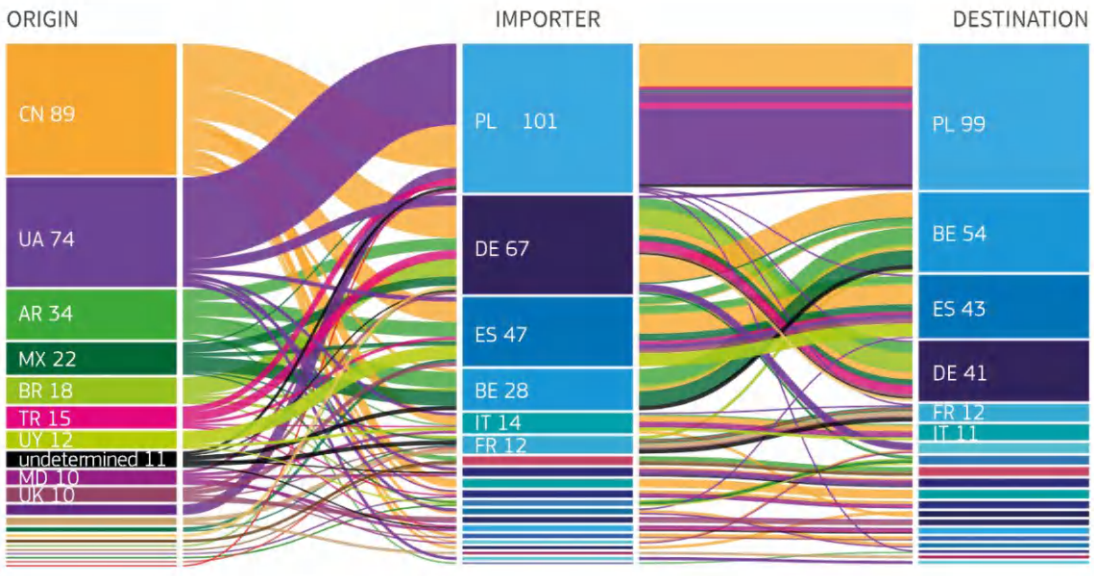


Figure 3. Flow of sampled honey consignments from exporting countries to importing EU Member States and place of first destination in EU Member States. Origin, importer and destination information according to the CHED documentation.



5 Laboratory testing

5.1 Test methods

The JRC used several methods to detect honey suspicious of containing added sugar syrup(s) (**Table 1**). Method details are given in Annex I.

Table 1. Detection methods for sugar syrup(s) added to honey.

Method principle	Benchmark values / Markers of adulteration
Elemental Analyser/Liquid Chromatography – Isotope Ratio Mass Spectrometry (EA/LC-IRMS)	Benchmark values for differences between $^{13}C/^{12}C$ stable carbon isotope ratios of protein and sugar compounds as proposed by Elflein and Raezke[10]
High-Performance Anion Exchange Chromatography - Pulsed Amperometric Detector (HPAEC-PAD)	Polysaccharides with a degree of polymerisation (DP) equal or larger than ten (DP≥10)[11]
Liquid Chromatography - High Resolution Mass Spectrometry (LC-HRMS)*	Oligosaccharides with DP≥6 and <10[12] 2-Acetylfuran-3-glucopyranoside (AFGP)[12] Difructose anhydride (DFA)[12]
Proton Nuclear Magnetic Resonance (^{1}H-NMR) Spectroscopy	Mannose[13] Honey-Profiling™[14]

* Insufficient sensitivity of the method to detect polysaccharides with DP≥10.

5.2 Compliance assessment

Honey offered on the internal market needs to comply with Council Directive 2001/110/EC, particularly with the composition criteria given in Annex II. If extended with appropriate sugar syrups imitating the sugar composition of genuine honey, the adulterated products will in most cases comply with the provisions. Therefore, the presence of an adulteration marker in honey or a $^{13}C/^{12}C$ isotope ratio not respecting the benchmark values of **Table 1** led to the conclusion that the honey was suspicious of not being in compliance with the requirement that honey shall not have added to it any food ingredient, including food additives, nor shall any other additions be made other than honey.

6 Analytical testing results

Of the 320 samples received from the competent authorities of the EU Member States, 147 (46 %) were suspicious of being non-compliant with the provisions of the EU Honey Directive 2001/110/EC because at least one marker of extraneous sugar sources was detected (**Figure *4***). However, the used techniques provided qualitative information (presence/absence of markers) and, therefore, it was not possible to estimate the level of exogenous syrups present in honey.

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10 Lutz Elflein, Kurt-Peter Raezke: Improved detection of honey adulteration by measuring differences between $^{13}C/^{12}C$ stable carbon isotope ratios of protein and sugar compounds with a combination of elemental analyzer - isotope ratio mass spectrometry and liquid chromatography - isotope ratio massspectrometry (δ^{13}C-EA/LC-IRMS). Apidologie 39 (2008) 574–587

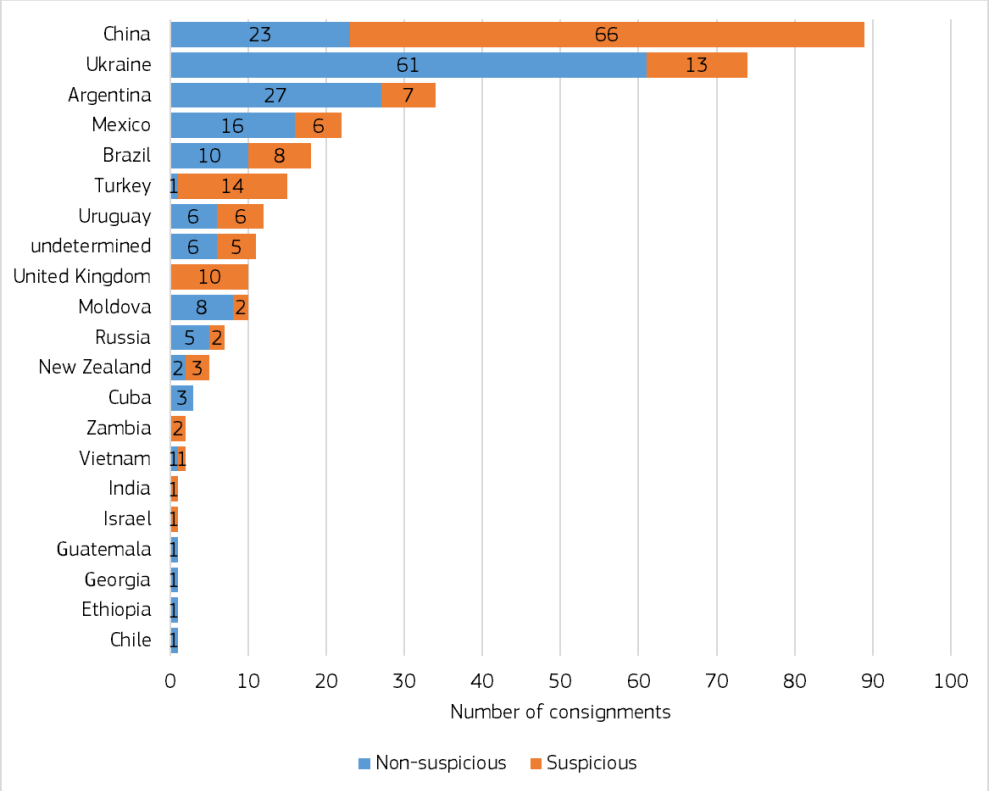
11 V. Morales, N. Corzo, M.L. Sanz: HPAEC-PAD oligosaccharide analysis to detect adulterations of honey with sugar syrups. Food Chemistry 107 (2008) 922–928

12 Bing Du, Liming Wu, Xiaofeng Xue, Lanzhen Chen, Yi Li, Jing Zhao, Wei Cao: Rapid Screening of Multiclass Syrup Adulterants in Honey by Ultrahigh-Performance Liquid Chromatography/Quadrupole Time of Flight Mass Spectrometry. Journal of Agricultural and Food Chemistry 63 (2015) 6614−6623

13 J. Missler, T. Wiezorek, G. Beckh: Mannose: a marker for adulteration with syrup or resin treatment of blossom honey. Proceedings of the XIII International Conference on the Applications of Magnetic Resonance in Food Science, 2016

14 https://www.bruker.com/en/products-and-solutions/mr/nmr-food-solutions/honey-profiling.html (accessed 05/07/2022)

Figure 4. Geographical origin of suspicious honey consignments



Oligo/polysaccharides and mannose were the markers which most frequently flagged a suspicious honey, either in combination or as single marker. In 44 % of suspicious cases, even two to five marker substances were present.

Stable carbon isotope ratio analysis by EA-IRMS (AOAC method 991.41), a method that has frequently been used in the past to detect sugar syrups made of maize starch or sugarcane , was not effective in detecting honey suspicious of being adulterated. This is a clear indication that sugar syrups made of maize starch or sugarcane are no longer used to extend honey.

The highest absolute number of suspicious consignments originated from China (66 out of 89, 74 %), although honey originating from Turkey (14 out of 15, 93 %) had the highest relative proportion of suspicious samples (**Figure 5**).

Honey imported from the United Kingdom had an even higher suspicion rate (10 out of 10, 100 %). However, the available traceability information suggests that this could be the result of honey produced in other countries but further processed and re-exported by the United Kingdom.

Honey consignments sampled in Romania had the lowest level of suspicion of non-compliance (3 out of 15, 20 %), while the highest rate was observed for France (17 out of 21, 81 %). Trade flows differed also to a certain degree reflecting the importance of major sea ports for trade routes; honey from The Americas entered the EU through ports in Belgium, Germany, France and Spain, while Ukrainian honey was mostly exported to neighbouring Poland and Moldavian honey to Romania (**Figure 6**). However, Chinese honey was sampled by nearly all EU Member States participating in the coordinated action.

Figure 5. Proportion (%) of suspicious honey consignments of major honey exporting countries (for statistical reasons, countries with less than 10 samples were not considered).

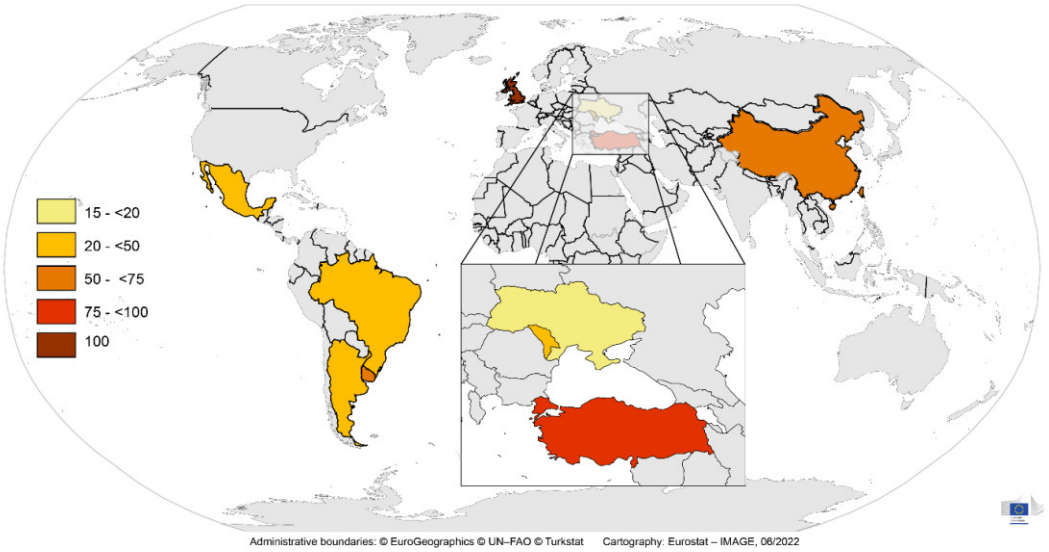
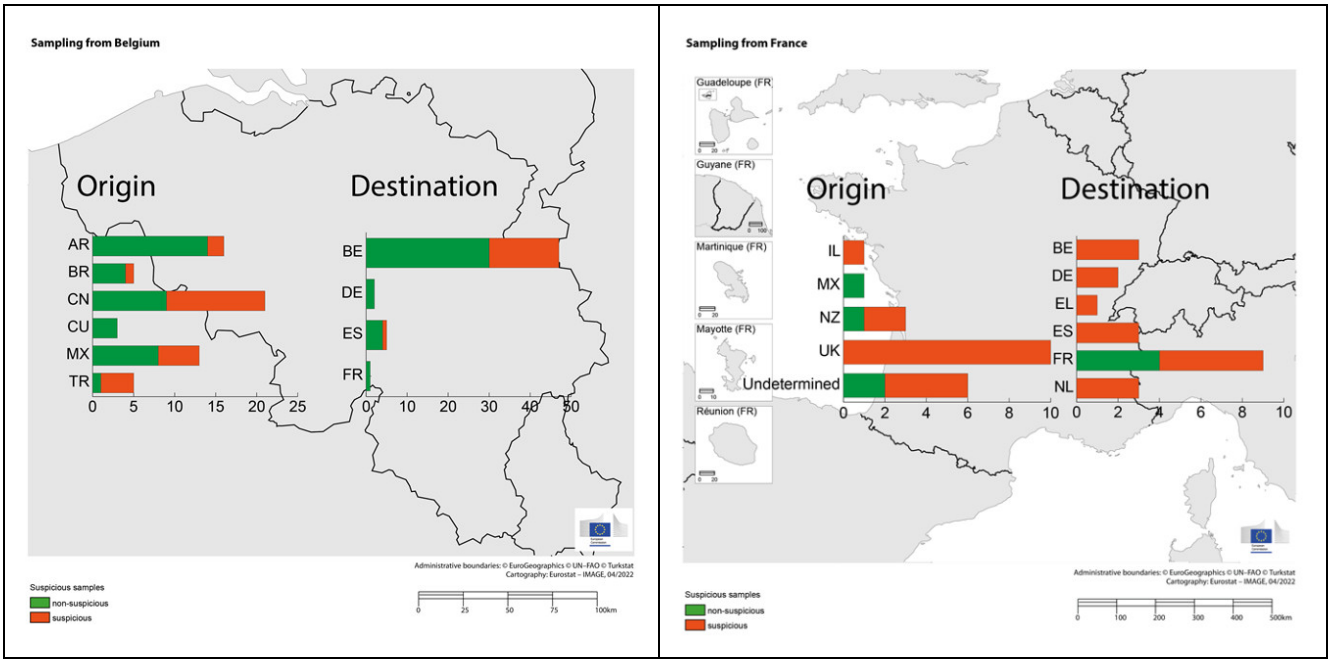
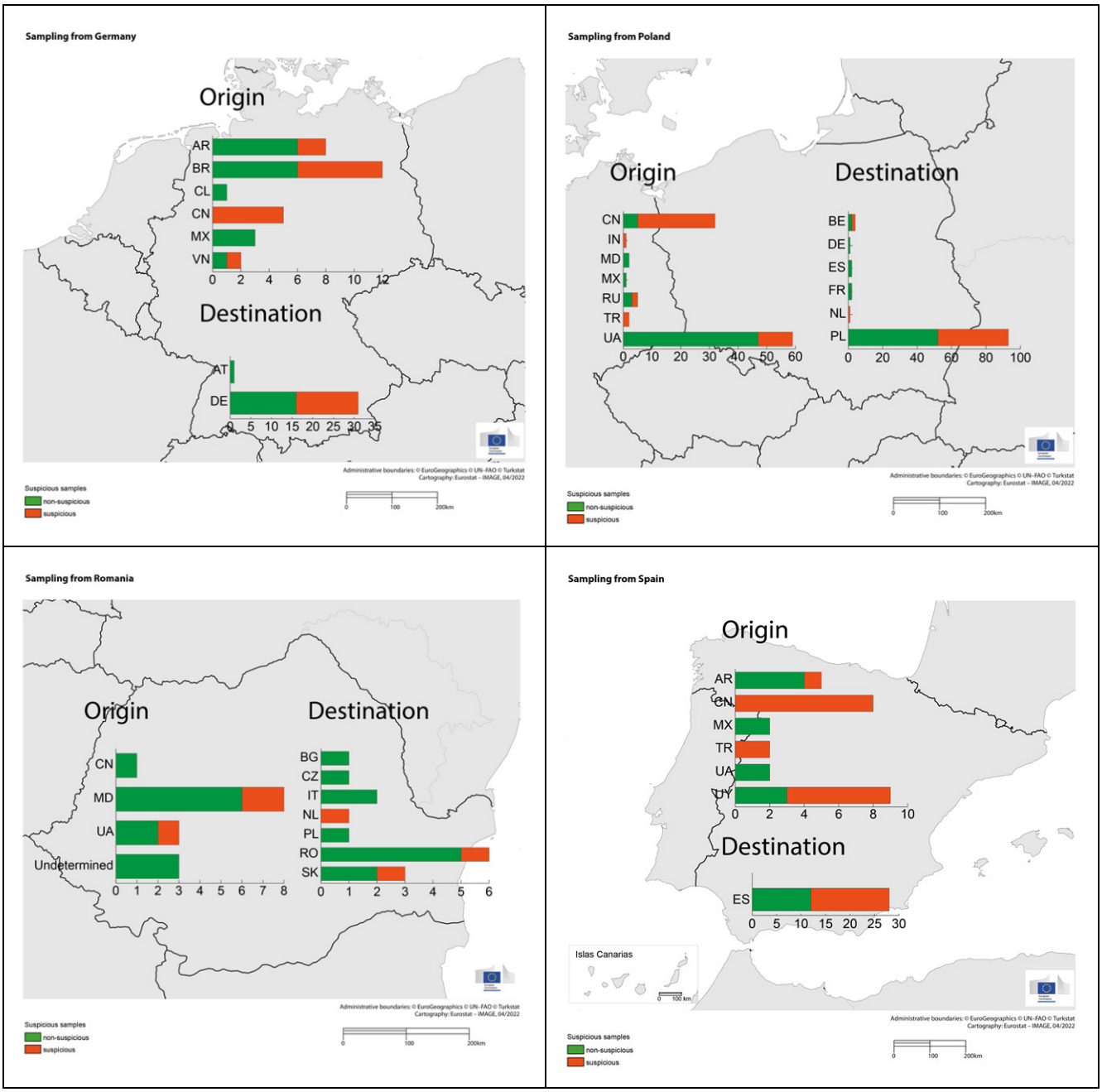


Figure 6. Geographical origin of honey consignments entering the EU (for statistical reasons, Member States with less than 10 samples were not considered; bars in orange indicate number of suspicious samples, bars in green number of non-suspicious samples).





7 Conclusions

The EU coordinated action launched by the European Commission confirmed the assumption that a significant part of honey imported into the internal market is suspicious of not complying with the provisions of the EU Honey Directive 2001/110/EC. Of the 320 samples received from the competent authorities of the EU Member States, Norway and Switzerland, 147 (46 %) were suspicious of being non-compliant. The suspicion rate was considerably high in comparison to an earlier EU wide coordinated control plan conducted in 2015-17, where 14 % of the analysed samples did not comply with established benchmark criteria to assess honey authenticity. However, a different set of methods with improved detection capability was used in the present study, which may explain this difference.

Stable carbon isotope ratio analysis by EA-IRMS (AOAC method 991.41), a method that has frequently been used in the past to detect sugar syrups made of maize starch or sugarcane, was not effective in detecting honey suspicious of being adulterated. This is a clear indication that sugar syrups made of maize starch or

sugarcane are no longer used to extend honey and that they have been replaced by syrups made mostly from rice, wheat or sugar beet. Consequently, improved, harmonised and generally accepted analytical methods are still needed to increase the capability of official control laboratories to detect honey adulterated with tailor-made sugar syrups that imitate to a large extent the characteristic sugar profile of genuine honey. Once available, such improved detection methods will constitute an effective deterrent to reduce fraud opportunities in international honey trade.

List of abbreviations and definitions

DP	Degree of polymerisation
EA/LC-IRMS	Elemental Analyser/Liquid Chromatography – Isotope Ratio Mass Spectrometry
^{1}H-NMR	Proton Nuclear Magnetic Resonance Spectroscopy
HPAEC-PAD	High-Performance Anion Exchange Chromatography - Pulsed Amperometric Detector
LC-HRMS	Liquid Chromatography - High Resolution Mass Spectrometry

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Annex 1. Analytical methods and decision mechanism applied to identify suspicious samples of honey in the frame of the Coordinated Control Plan for Honey

JRC's quality system is ISO 9001 certified and certain testing activities of JRC Geel are ISO 17025:2017 accredited. However, the methods of analysis used for generating the reported data are outside the scope of accreditation. This fact has to be considered in case a competent authority wishes to initiate regulatory action and might bring a requirement for retesting for regulatory or enforcement action at Member State level.

A1. Elemental Analyser/Liquid Chromatography – Isotope Ratio Mass Spectrometry (EA/LC-IRMS)

The combination of elemental analyser with an isotope ratio mass spectrometer (EA-IRMS) to determine the $\delta^{13}C$ values of protein isolated from honey together with liquid chromatography coupled to an isotope ratio mass spectrometer (LC-IRMS) to determine the $\delta^{13}C$ values of fructose, glucose, disaccharides and trisaccharides was used to detect addition of sugar syrups made from C4 plants, notably from maize, and from C3 plants, notably from rice, wheat or potato.

The AOAC 998.12 method was used to determine $\delta^{13}C$ values of protein isolated from honey using a NCS 2500 Elemental Analyser (Thermoquest Italia S.p.A., Milan, Italy) coupled to a Thermo Fisher Scientific Delta Plus XP IRMS operated using ISODAT NT 2.0 software..

LC-IRMS analyses were performed using a Dionex Ultimate 3000 HPLC system equipped with a pump, an auto-sampler and a column compartment for temperature control. The HPLC was linked to a Thermo Fisher Scientific LC-ISOLINK interface and analyses of CO2 isotopic ratios were carried out using a Thermo Fisher Scientific Delta V Advantage IRMS operated using ISODAT 3.0 software.

Honey samples were prepared by diluting honey with Ultrapure water (Fluka Analytical) to a concentration of 4 mg/mL. The HPLC column was a 300 x 7.8 mm Phenomenex Rezex RCM Monosaccharide (Phenomenex, Utrecht, NL). A flow rate of 0.3 mL/min and a temperature of 70 °C were used during analysis. Ultrapure water from VWR Chemicals was used as an eluent. The IRMS was calibrated using two-point linear normalisation with two certified reference standards IAEA-601 benzoic acid and IAEA-CH6 sucrose.

The benchmark values proposed by Elflein and Raezke[15] were used to assess honey purity based on the EA/LC-IRMS results. The differences in the $\delta^{13}C$ values ($\Delta\delta^{13}C_{max}$) of the individual sugars of authentic honey are distributed around a value close to zero. Based on the analysis of 451 authentic honey samples Elflein and Raezke calculated purity criteria (confidence level 99.7%) that which flag a honey sample as non-compliant if one of the $\Delta\delta^{13}C$ falls outside the cut-off limit. Measurement uncertainty of the LC-IRMS method was estimated using the intra-laboratory reproducibility standard deviation to estimate the expanded uncertainty (U) of the $\Delta\delta^{13}C$ values used for compliance testing (**Table A1**) was calculated according to:

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

$SD^2(a)$ and $SD^2(b)$ being the intra-laboratory reproducibility variances of the sugars used for calculating the differences. Only if the obtained $\Delta\delta^{13}C$ value exceeded the decision threshold, a sample was flagged as being suspicious.

[15] Elflein, L. and K.-P. Raezke, Improved detection of honey adulteration by measuring differences between $^{13}C/^{12}C$ stable carbon isotope ratios of protein and sugar compounds with a combination of elemental analyzer - isotope ratio mass spectrometry and liquid chromatography - isotope ratio mass spectrometry ($\delta^{13}C$-EA/LC-IRMS). Apidologie, 2008. 39(5): p. 574-587.

Table A1. Benchmark criteria and decision thresholds for assessing honey purity on the basis of EA/LC-IRMS measurements.

Parameter	Benchmark	Decision threshold taking account of measurement uncertainty
$\Delta\delta^{13}C$ (fructose – glucose)	±1.0	± 1.33
$\Delta\delta^{13}C$ (fructose – disaccharides)	±2.1	± 2.52
$\Delta\delta^{13}C$ (fructose – trisaccharides)	±2.1	± 2.61
$\Delta\delta^{13}C$ (fructose – protein)	±2.1	± 2.49
$\Delta\delta^{13}C$ (glucose – disaccharides)	±2.1	± 2.53
$\Delta\delta^{13}C$ (glucose – trisaccharides)	±2.1	± 2.62
$\Delta\delta^{13}C$ (glucose – protein)	±2.1	± 2.50
$\Delta\delta^{13}C$ (disaccharides – trisaccharides)	±2.1	± 2.68
$\Delta\delta^{13}C$ (disaccharides – protein)	±2.1	± 2.58
$\Delta\delta^{13}C$ (trisaccharides – protein)	±2.1	± 2.66

A2. High Performance Anion Exchange Chromatography – Pulsed Amperometric Detector (HPAEC-PAD)

HPAEC-PAD was used to detect polysaccharides with a degree of polymerisation (DP) of 10 and higher in honey as an indicator for adulteration with sugar syrups. Such polysaccharides are the result of incomplete enzymatic breakdown of starch and/or incomplete removal of them during syrup purification.

A BioLC system ICS-2500 (Dionex, Sunnyvale, USA) including an ED50A electrochemical detector was used. Saccharides were separated with a CarboPac PA200, 3 × 250 mm column (Thermo Fisher Scientific, Merelbeke, Belgium). Separation of the oligo-/polysaccharides was achieved with a gradient program of 100 mM NaOH (eluent A) and 100 mM NaOH/1000 mM NaOAc (eluent B) at a flow of 0.45 ml/min. The temperature for the column was set to 30 °C.

A3. Liquid Chromatography – High Resolution Mass Spectrometry (LC-HRMS)

LC-HRMS was used to flag suspicious samples via the presence of marker compounds (mannose[16], difructose anhydride (DFA) and 2-acetylfuran-3-glucopyranoside (AFGP)[17]) indicating the presence of sugar syrups. It was also used to confirm the presence of oligosaccharides (DP 6 and higher).

Separation was afforded by a Waters BEH Amide 100 x 2.1 mm, 1.7 µm particle size, column in HILIC mode. Gradient conditions were optimized to obtain baseline separation of mannose. The high resolution mass spectrometric detection was done with an Orbitrap Elite (Thermo Scientific) in positive mode electro spray ionisation with single MS full scan range from m/z 50 to m/z 2000. Compounds were identified by comparison to reference materials of known purity.

[16] J. Missler, T. Wiezorek and G. Beckh: Mannose: a marker for adulteration with syrup or resin treatment of blossom honey. Magnetic Resonance in Food Science 2016 Proceedings. doi: 10.1255/mrfs.4

[17] Bing Du, Liming Wu, Xiaofeng Xue, Lanzhen Chen, Yi Li, Jing Zhao, and Wei Cao: Rapid Screening of Multiclass Syrup Adulterants in Honey by Ultrahigh-Performance Liquid Chromatography/Quadrupole Time of Flight Mass Spectrometry. J. Agric. Food Chem. 2015, 63, 6614−6623

A4. Proton Nuclear Magnetic Resonance Spectroscopy (^{1}H-NMR)

^{1}H-NMR was applied as a confirmatory analysis in case the marker compound mannose (indication for presence of sugar syrups) could be detected in honey samples by LC-HRMS.

The measurements were performed with a 400 MHz spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany). The specific parameters for the ^{1}H-NMR single experiment are listed in **Table A2**.

Table A2: Specific parameters for single experiment of analysis of honey.

Pulse programme	AQ [s]	Temperature	NS	DS [s]	TD	RG
noesygppr1d	3.985	300.00 K ± 0.1 K	32	4	65536	16

AQ [s] – acquisition time in s; NS – number of scans; DS – dummy sans; TD – time domain size; RG – receiver gain.

After the calculation of a free induction decay (FID) for each sample, the FID's were automatically pre-processed in TopSpin (Bruker BioSpin GmbH, Rheinstetten, Germany), which included a multiplication with an exponential line broadening (LB 0.3 Hz) function, Fourier transformation, phase and baseline correction. The chemical shift axis of the generated spectra was referred to the signal of 3-(trimethylsilyl)-propanoic-2,2,3,3-d_4 acid sodium salt (TSP) at δ 0.00 ppm.

On a case-by-case basis, NMR-based profiling in combination with a commercially available databank and software tool (Bruker Honey Profiling™) was used as supportive evidence before taking a final decision.

Decision rules for flagging suspicious samples

A sample was considered as being suspicious of adulteration with a sugar syrup if:

The results of the EA/LC-IRMS analysis fell outside the decision threshold(s) reported in **Table A1**, or;

Oligosaccharides with a degree of polymerisation (DP) between 6 and 9 were detected by LC-HRMS, or;

Polysaccharides with a DP between 10 and 19 were detect by HPAEC-PAD, or;

Mannose was detected by LC-HRMS and its presence confirmed by NMR, or;

Difructose anhydride (DFA) was detected by LC-HRMS, or;

2-Acetylfuran-3-glucopyranoside (AFGP) was detected by LC-HRMS.