Honey authenticity: determination of exogenous sugars by NMR

Seminar (2019) Report

Department for Environment Food & Rural Affairs

Food Standards Scotland
For safe food and healthy eating

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Honey authenticity: determination of exogenous sugars by NMR

Seminar Report

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Contents

Note: Text recorded in sections relating to the workshop elements of the seminar are those of the individual workshop participants and do not necessarily represent the views of all participants, their organisations, or the organisers.

1. Executive Summary

The Government Chemist, the Department for Environment Food and Rural Affairs (Defra), the Food Standards Agency (FSA) and Food Standards Scotland (FSS) held a UK seminar on honey authenticity: determination of exogenous sugars by nuclear magnetic resonance (NMR) on 13 November 2019, which was attended by 57 people representing stakeholder organisations.

The aim of the seminar was to bring together stakeholders involved in honey production and analysis to discuss this topic and ideally come to an agreed position. It was anticipated that the output of this seminar would help inform future UK government policy on the use of NMR for honey authenticity.

The seminar consisted of a series of presentations from invited experts that set the scene for the workshop part of the day, which involved participants splitting into four representative groups to discuss the suitability of NMR for enforcement purposes and to identify gaps and priorities to assessing the use of NMR for the appraisal of honey authenticity.

There was consensus support for NMR as a tool in verifying the authenticity of foods but that, based on the available evidence, NMR methods are not yet suitable for the detection of exogenous sugars in honey for enforcement purposes. In order to address this, the groups made a number of suggestions centred on the creation of a forum for continuing dialogue between all parties, provision of training, education and guidance on the production and analysis of honey, and standardisation of the application and interpretation of NMR approaches for the determination of exogenous sugars in honey.

The UK honey seminar provided a valuable forum for stakeholders to come together to discuss the use of NMR for the determination of exogenous sugars in honey and has produced some constructive ideas on how the UK could address some of the current issues faced.

Defra, FSA, FSS and the Government Chemist will continue to work together, along with interested stakeholder groups, to consider how to take forward the suggestions and themes arising from the seminar, facilitating continued dialogue and information exchange in this important area.

2. Introduction

Honey is a high value commodity and in order to protect the consumer and legitimate businesses it is important to be able to detect misleading practices such as adding sugars. Detecting exogenous sugars is challenging and though reasonably effective methods to detect cane sugar adulteration are available, this is not the case for commodities such as beet sugar, where the problem is much more challenging and existing methods are complex and time-consuming. In recent years, scientists have begun to employ nuclear magnetic resonance (NMR) based methods to combat such adulteration. The UK honey industry has raised some concerns about the robustness of NMR methods, particularly in relation to the databases which underpin the interpretation of results.

The power of NMR to elucidate molecular structure is well known but it is a relative newcomer in the analytical investigation of food authenticity and particularly honey. Its applicability to food authenticity stems from the ease of sample preparation, high reproducibility and the ability to identify and quantify molecular markers in complex mixtures.

Following the concerns expressed by industry, the FSA issued advice[1] in December 2015 that no enforcement action on alleged added sugar in honey should be taken on the basis of NMR results alone.

The results of a European Commission (EC) Co-ordinated control plan on honey authenticity were published in 2016[2]. Of the 147 samples taken from the UK in the EC Co-ordinated control plan, 93% were found to be compliant with only 5% classed as non-compliant[3]. The non-compliances were mostly related to the incorrect botanical source (4%) and or for the presence of exogenous sugars or sugar products (1%). In addition, and in accordance with the EU protocol, 2% of samples were classified as being in "suspicion of non-compliance" with regards to geographical region (1%) and possible presence of exogenous sugars or sugar products (1%). The European Commission Co-ordinated control plan used state-of-the-art methods for detection of added sugars in honey but these did not include NMR methods.

Subsequently, in January 2018, the EC Joint Research Centre (JRC) held a technical round table on honey authentication. The purpose of this meeting was to collect the opinion of a broad representation of stakeholders on the challenges of authenticating honey, to identify the gaps in available tools and knowledge and identify ways of filling those gaps. The most common forms of honey fraud were discussed and needs for addressing them in an effective manner identified. The meeting report was published in March 2018[4].

[1] FSA letter 22nd December 2015, ENF Ref: ENF/E/15/041: https://webarchive.nationalarchives.gov.uk/20180411191034/https://www.food.gov.uk/sites/default/files/enf-e-15-041.pdf

[2] https://ec.europa.eu/food/sites/food/files/safety/docs/oc_control-progs_honey_jrc-tech-report_2016.pdf

[3] https://acss.food.gov.uk/sites/default/files/uk-honey-report130416.pdf

[4] https://ec.europa.eu/jrc/sites/jrcsh/files/ares181569074-1_technical_round_table_on_honey_adulteration_report.pdf

In January 2018, the FSA and Defra asked the Government Chemist for independent advice on the use of NMR to determine exogenous sugars in honey. Following input from the Government Chemist, the FSA updated the enforcement advice[5] to state that a 'weight of evidence' approach should be applied, including traceability checks and follow up discussions, before enforcement action is considered for alleged added sugar in honey.

The Government Chemist, Defra, FSA and FSS also agreed to hold a UK seminar on honey authenticity: determination of exogenous sugars by NMR, in order to share information on relevant scientific developments and perspectives to try to come to a consensus on a way forward.

3. Aim

The aim of this seminar was to bring together representatives from mainly UK stakeholders involved in honey production and analysis to discuss the determination of exogenous sugars in honey by NMR and, ideally, come to an agreed position on a way forward. It was anticipated that the output of this seminar would help inform future UK government policy on the use of NMR for honey authenticity.

4. Seminar

The seminar took place at Defra (Nobel House) on 13th November 2019 and was attended by 57 people representing stakeholder organisations. A list of participants can be found at Appendix 1.

The seminar programme was as follows:

[5] FSA letter 8th January 2018, URN PLGEN18001 ENF/E/18/002 , https://smartercommunications.food.gov.uk/communications/files/16?scrollPos=11347.3330078125 (an account must be requested and authorised to assess this link)

Programme

10:00 Registration

10:30 Chair's introduction: Michael Walker, Head of the Office of the Government Chemist, LGC

10:35 Opening Remarks: Karen Lepper, Deputy Director for Food Standards and Consumers, Defra

10:40 Introduction - Legislative and policy overview: Michelle McQuillan, Defra

11:00 UK enforcement perspective: David Pickering, Buckinghamshire & Surrey Trading Standards

11:20 Industry perspective: Cathal Henigan, British Honey Importers and Packers Association (BHIPA).

11:40 Break

11:55 Food integrity scientific opinions: James Donarski, Fera Science Ltd

12:15 Use of NMR in Germany: Sandra Weber, CVUA-Karlsruhe

12:35 NMR methods for exogenous sugars (1): Thomas Spengler & Lea Heintz, Bruker

12:55 Lunch

13:50 NMR methods for exogenous sugars (2): Eric Jamin, Eurofins

14:10 NMR methods for exogenous sugars (3): Adrian Charlton, Fera Science Ltd

14:30 Introduction to workshop: Selvarani Elahi, Deputy Government Chemist, LGC

14:40 Workshop

15:40 Break

15:55 Feedback on workshop

16:40 Summary

17:00 Close of seminar

5. Introductory Speakers

Chair's introduction: Michael Walker, Head of the Office of the Government Chemist

After welcoming everyone to the event, Michael said that in recent years an increasing number of studies have appeared in the peer reviewed literature advocating the use of NMR in food safety and authenticity applications. There are several laboratories offering an analytical service in this respect.

The seminar participants were asked to:

- Draw some conclusions (either way) on the evidence about the applicability of NMR to honey authenticity (and in particular the issue of exogenous sugars)
- Articulate any gaps
- Make suggestions for next steps that could be taken.

Whilst there are broader issues in this area that require international action, seminar participants were asked to focus on gaps and make suggestions on aspects that impact specifically on the UK and may potentially be addressed within the UK.

Opening Remarks: Karen Lepper, Deputy Director for Food Standards and Consumers, Defra

Karen explained that one of her responsibilities is to head up policy in England on food labelling and food compositional standards. Honey is one such food that is covered by specific legislation in its own right, defining its composition and labelling by law[6].

Karen acknowledged the vast amount of knowledge and experience in the room, either as a honey business, honey analysts or food enforcers. She emphasised that the workshop was about information sharing, encouraging debate, and listening to each other's perspectives.

Karen said she hoped that the workshop would help Defra to gain a better understanding of the issues, identify any existing gaps in knowledge and provide a mechanism for participants to give feedback on how they think things might move forward in the UK.

She said that consumers want to be assured that what they are buying is genuine and they are not being deceived; that is especially true of something like honey where the rules are very clear that "nothing should be added or taken away". Karen highlighted that the focus of everyone attending the workshop – legislators, enforcers, industry, food analysts – should be to protect UK consumers and see our UK honey market prosper.

6. Expert Presentations

Detailed presentations (see 'programme' above) were given by invited experts. All the presentations are available on the Government Chemist website.

The questions and answers that followed some of the presentations are given in Appendix 2.

7. Introduction to the Workshop

Selvarani Elahi, Deputy Government Chemist, explained that the approach being followed for the workshop part of the seminar was adapted from the Government Office for Science (GO-Science) Futures Toolkit. Participants were split into four representative groups and asked to consider the following questions:

[6] Council Directive 2001/110/EC (honey directive)

1. What is the suitability of NMR for enforcement purposes? (Groups 1 & 2)
2. What should the next steps be to resolve difficulties in the use of NMR to assess honey authenticity? (Groups 1 & 2)
3. What are the gaps to progress on the use of NMR to assess honey authenticity for exogenous sugars? (Groups 3 & 4)
4. What can be done on a UK basis to address the above gaps and by whom? (Groups 3 & 4).

Group work

Facilitators

Each group was assigned a facilitator to:

- Ensure the group selected a rapporteur and scribe
- Keep the discussion focussed on the questions
- Allow sufficient time to discuss both questions
- Ensure each group reaches a consensus on their top 3 points for the rapporteur to feed back to the meeting

The facilitators were asked to remain impartial and avoid making technical contributions to the discussion.

Each group was also provided with a flipchart and Post-it® notes and were asked to:

- Consider each question individually and write down their ideas on Post-it® notes. (10 minutes)
- Collate and discuss ideas together as a group, sorting and grouping Post-it® notes thematically on the flipchart. (20 minutes)
- Identify their top 3 ideas to feed back to meeting.

Each group appointed a rapporteur and scribe. Rapporteurs were given 10 minutes to give feedback on the two questions that their group addressed, encompassing a general account of how the session went and the top 3 ideas for each question.

8. Summary of Workshop Results

Having listened to the presentations from the experts, the four groups considered the questions posed; groups 1 and 2 considered questions 1 and 2 and groups 3 and 4 considered questions 3 and 4.

The results of the workshop[7] are given in Appendix 3 and summarised below.

[7] The workshop results reflect the views of the participants; they are not agreed government policy.

- **Question 1: “What is the suitability of NMR for enforcement purposes?”**

There was positive support for NMR as a tool in verifying the authenticity of foods. However, there was agreement that based on the available evidence, NMR methods were not yet suitable for the detection of exogenous sugars in honey for enforcement purposes. The reasons given for this included a lack of information on the databases underpinning interpretation of the method outputs, particularly around the origin of ‘authentic’ samples and representation of the UK honey market. In addition, participants felt that there was insufficient information on the results of inter-laboratory comparisons of the methods and on the scope of laboratory accreditation.

- **Question 2: “What should the next steps be to resolve difficulties in the use of NMR to assess honey authenticity?”**

There was consensus from both groups that addressed this question that the robustness of databases used for interpretation of results for exogenous sugars needs to be improved; a number of next steps were suggested in relation to the validation of databases, which included development of a protocol for sample collection, independently or by government, curation of authentic samples, and external validation and scrutiny of the databases. Other suggestions included further clarification on the enforcement advice and the definition of honey and an examination of databases for other commodities, as a potential model for success. Participants also felt that better communication between all parties involved should be encouraged.

- **Question 3: “What are the gaps to progress on the use of NMR to assess honey authenticity for exogenous sugars?”**

Gaps to progress suggested by the groups included development of acceptance criteria for adding samples to databases, the development of standardised testing methodology and reporting protocols. A lack of trust between the industry and laboratories was also identified as a gap.

- **Question 4: “What can be done on a UK basis to address the above gaps and by whom?”**

Participants suggested the establishment of working groups involving government, industry, laboratories and retailers for continued dialogue and the provision of training, education and guidance on the production and analysis of honey. The establishment of centres of excellence was referred to and independent scrutiny of databases was discussed.

Seminar Report

A draft report on the seminar was prepared and circulated to all attendees. Two comments were received. It was suggested that the data for the UK samples in the European Commission (EC) Co-ordinated control plan on honey authenticity should be mentioned and some technical detail in the Q&A section (Appendix 2) was felt to be ambiguous and should be deleted. Both these comments were actioned.

9. Conclusions and Next Steps

The UK honey seminar provided a valuable forum for stakeholders to come together to discuss the use of NMR for the determination of exogenous sugars in honey and has produced some constructive ideas on how the UK could address some of the current issues faced.

There was consensus among participants that the seminar had provided a useful forum for constructive discussion. Consensus also appeared to have been reached on positive support for NMR as an instrumental technique in itself however the stumbling block lay in the current databases relied upon for the interpretation of NMR outputs. Opinion was divided on whether extant databases should be independently examined to assess their fitness for purpose for the determination of exogenous sugars in honeys sold in the UK. There was agreement on all other points and that much work remains to be done. It was agreed that continuing dialogue is essential and will serve to make the UK a more hostile environment for any who might wish to perpetrate fraud in the honey supply chain.

Defra, FSA, FSS and the Government Chemist will continue to work together, along with interested stakeholder groups, to consider the suggestions made and decide how to address the priority themes emerging from the seminar, facilitating continued dialogue and information exchange in this important area.

10. Acknowledgments

The UK seminar on honey authenticity: determination of exogenous sugars by NMR was organised by LGC with funding from the Government Chemist programme (funded by the Department for Business, Energy and Industrial Strategy) with financial input from Defra, FSA and FSS.

Appendix 1: Seminar Participants

Name	Affiliation
Margaret Ginman	Bee Farmers Association
Elizabeth Andoh Kesson	British Retail Consortium
Lea Heintz	Bruker
Thomas Spengler	Bruker
David Pickering	Buckinghamshire & Surrey Trading Standards
Peter Martin	Consultant
Sterling Crew	Consultant
Poppy Owen	Co-op
Sandra Weber	CVUA-Karlsruhe
Karen Lepper	Defra
Michelle McQuillan	Defra
Miguel Arranz	Defra
Paul Ndede	Defra
Sophie Rollinson	Defra
Julie Fallows	Duerr's
Eric Jamin	Eurofins
Adrian Charlton	Fera Science Ltd
James Donarski	Fera Science Ltd
Keneth Chinyama	Food and Drink Federation
Michelle Young	Food Standards Agency
Valerie McFarlane	Food Standards Agency
Nuala Meehan	Food Standards Agency in NI
Faith Chung	Food Standards Scotland
Lynsey Scullion	Food Standards Scotland
Jane White	Glasgow Scientific Services
Joel Scott	Hain Daniels

Jon Griffin	Kent Scientific Services
James Absolon	Lamex Foods
Sally Clarke	Lamex Foods
Bob Oswald	LGC
Cailean Clarkson	LGC
Kirstin Gray	LGC
Mark Woolfe	LGC
Michael Walker	LGC
Selvarani Elahi	LGC
Vicki Barwick	LGC
Steve Higgon	London Port Health Authority
James Mitchell	Marks and Spencer
David Hoyland	Minerva Labs
Jon Roe	Morrisons
Sean Daly	National Food Crime Unit / Food Standards Agency
David McHattie	Portal Veterinarian Stansted
Liz Moran	Public Analyst Scientific Services Ltd
Steve Batchford	Sainsbury's
Thomas Heck	Sarant Ltd
Simon Rowell	Suffolk Coastal Port Health Authority
Alison Lord	Tesco
Nick Laverty	Tesco
Gary Gould	The Association of Port Health Authorities
Andy Wilson	Trading Standards Officer Bristol
Paul Maylunn	Trading Standards Officer Cambridgeshire
Emily James	Trading Standards Officer Oxfordshire
Kelly Edwards	Trading Standards Officer Powys
Elizabeth Benson	Uren
Cathal Henigan	Valeo Foods

Patrick Robinson	Valeo Foods
Laura Jackson	Waitrose

Appendix 2: Questions and Answers following presentations

Questions were asked following some of the presentations; the questions asked and answers provided were noted.

1. Food integrity scientific opinions *(James Donarski, Fera Science Ltd)*

Question 1: Issue of NMR field strength and transferability of data?

Answer 1: Most commercial providers use 400MHz instruments for their screening methods[8].

2. Use of NMR in Germany *(Sandra Weber, CVUA-Karlsruhe)*

Question 2: Are there any proficiency testing schemes on using NMR in honey authenticity?

Answer 2: Not for honey but there is a scheme for fruit juices.

Comment: Since 2017, there is regular proficiency testing for honey authenticity by NMR organised by Eurofins, with 20 participants each using their own SOPs.

3. NMR methods for exogenous sugars [1] *(Thomas Spengler & Lea Heintz, Bruker)*

Question 3: How do you know that database honey samples are authentic?

Answer 3: Bruker use 3 main partners in Germany to collect samples according to a protocol. The samples are examined using all the information on traceability, analyses using conventional tests, and NMR analyses. Outliers are removed (around 10% of samples), and although Bruker are not 100% certain that samples in the database are authentic, it is certain that there will not be any false positives. Sample collection has now been changed so that samples of honey are collected directly from the beehive.

4. NMR methods for exogenous sugars [2] *(Eric Jamin, Eurofins)*

Question 4: Why did honeydew honey have such a high non-compliance?

Answer 4: The high non-compliance rate was because much of honeydew honey was not declared properly (as honeydew honey).

Question 5: Was there much difference in compliance with imported honey?

Answer 5: Eurofins did not find much difference in rates of adulterated honey from China compared to other tested honey.

5. NMR methods for exogenous sugars [3] *(Adrian Charlton, Fera Science Ltd)*

[8] NMR instruments are available with a range of magnetic field strengths from 300 to over 1000 expressed in megahertz (MHz). Higher field strengths give improved sensitivity and resolution (separation) of peaks in the NMR spectrum, an advantage when complex spectra are encountered.

Question 6: How big is Fera's honey database?

Answer 6: Fera is building up its database each season at a rate of about 100 samples/year.

Question 7: Does Fera talk to Bruker about its database?

Answer 7: Yes, but it was some time ago.

Appendix 3: Workshop Results

Two groups considered each question; groups 1 and 2 considered questions 1 and 2 and groups 3 and 4 considered questions 3 and 4. The outcome of the discussions and points made by the groups during feedback to the meeting are captured below.

1. **Question 1: "What is the suitability of NMR for enforcement purposes?"**

There was positive support for NMR as a tool in verifying the authenticity of foods. However, there was agreement that based on the available evidence, NMR methods were not yet suitable for the detection of exogenous sugars in honey for enforcement purposes. The reasons for this included a lack of information on the database underpinning the method, particularly around the origin of 'authentic' samples and representation of the UK honey market. In addition, there was insufficient information on the results of inter-laboratory comparisons of the method and the scope of laboratory accreditation.

Participants made the following specific points:

- Interpretation of results is not clear
- Robustness of supporting databases is unclear and with respect to representation of the UK honey market
- Composition and collection of 'authentic' honey samples is unknown / unclear
- Scope of laboratory accreditation is not clear
- Lack of information on the results of inter laboratory-comparisons
- Current legal definition of honey was said to leave the categorisation of mature/immature honey open to interpretation.

2. **Question 2: "What should the next steps be to resolve difficulties in the use of NMR to assess honey authenticity?"**

There was consensus from both groups that addressed this question that the robustness of databases used for interpretation of results for exogenous sugars needs to be improved; a number of next steps were suggested in relation to the validation of databases, which included development of a protocol for sample collection, independently or by government, curation of authentic samples, and external validation and scrutiny of the databases.

Participants made the following specific points:

- Agreement of a protocol for sample collection.
- Independent verification of sample collection.
- Some participants wanted governments in honey producing countries to be responsible for the collection and curation of samples for databases. Others suggested independent curation.
- Validation via, for example, an external proficiency scheme or inter-laboratory trial.

- Consideration to be given to seasonal changes, climate in the country of origin, permissible practices (such as blending) and changes in sample composition whilst in storage.
- Further consideration of the interpretation of the definition of honey
- Clarification of the current FSA enforcement letter.
- Examination of best practice models such as the Sure Global Fair (SGF) scheme for fruit juice to see how the NMR method gained acceptance in the fruit juice sector.
- Review of the robustness of current databases; participants felt that independent scrutiny was at least desirable and may be essential. They suggested that it should be established who would scrutinise database(s), e.g. UKAS, Government Chemist, BSI, CEN, ISO etc.
- Encourage better communication between laboratories, research bodies, producers, packers and enforcement agencies.

3. **Question 3: "What are the gaps to progress on the use of NMR to assess honey authenticity for exogenous sugars?"**

Gaps suggested by the groups included development of acceptance criteria for adding samples to databases, and development of standardised testing methodology and reporting protocols for the determination of exogenous sugars in honey by NMR.

A lack of trust between the industry and laboratories was also identified as a gap.

Participants made the following specific points:

- Acceptance criteria for adding authentic samples to database(s) are required, which should address questions such as:
  - When and how samples are collected?
  - What metadata is recorded?
  - What is the traceability?
- An independent, documented methodology to international standards is needed, which includes standardised reporting of results.

4. **Question 4: "What can be done on a UK basis to address the above gaps and by whom?"**

The following suggestions were made:

- Establishment of working groups involving government, industry, laboratories and retailers for continued dialogue.
- Provision of training, education, guidance on the production and analysis of honey.
- Consideration should be given to the potential of establishing centres of excellence.

In the sum-up of the day, when asked by the Chair for a view on extant databases for honey authenticity, opinion was divided on whether commercial databases might, with independent scrutiny, be useful rather than create a new database; participants who objected, said it was on

the basis that questions including transparency about the existing databases had not been addressed.

Representatives of commercial databases indicated openness to government and expert body access to their databases and it was noted that the UK honey industry is building its own database to represent honey sold in the UK based on samples collected directly from hives by bee keepers.

In one of the break-out sessions, there was some criticism of the manner in which HR-IRMS[9] results are being reported, which resembles the reporting of exogenous sugars about five years ago, and is causing problems in the industry. The commercial provider agreed to report this back to colleagues.

[9] High resolution isotope ratio mass spectrometry