Hort Innovation

Final Report

International Olive Council – Technical Committee

Project leader:

Claudia Guillaume

Delivery partner:

Modern Olives

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Project:

International Olive Council – Technical Committee OL15002

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Level 7
141 Walker Street
North Sydney NSW 2060

Telephone: (02) 8295 2300

www.horticulture.com.au

Content

Summary

The International Olive Council (IOC) is an intergovernmental organisation which brings together olive oil and table olive producing and consuming stakeholders. The IOC provides a forum for authoritative discussion on issues of interest to the olive industry. Australia is not an IOC member and by consequence attended the meetings in the status of Observer. Following each meeting, the Australian representative attending produced a report highlighting the most important points having an implication for the Australian olive industry and then distributed it to the industry representatives.

The Australian Olive Industry participated in the International Olive Council Chemists' meetings that were conducted twice yearly (April and October) in Madrid, Spain.

These meetings were highly specific technical meetings of olive oil chemists. The main topics of discussion and debate were

1. existing and new analytical methods to analyse the different grades of olive oil for quality and authenticity,
2. identify appropriate limits for the analysed parameters,
3. analysis of other analytical methods and regulations from around the world (i.e., Codex),
4. examine nutritional guidelines in relationship to olive oil,
5. share research results related to olive oil chemistry, etc.

Public summary

The International Olive Council (IOC) is an intergovernmental organisation which brings together olive oil and table olive producing and consuming stakeholders. This is a forum for authoritative discussion on issues of interest to the olive industry. Australia is not an IOC member and by consequence attended the meetings in the status of Observer. Following each meeting, the Australian representative attending produced a report highlighting the most important points having an implication for the Australian olive industry and then distributed it to the industry representatives. The Australian Olive Industry participated in the International Olive Council Chemists' meetings that were conducted twice yearly (April and October) in Madrid, Spain.

Keywords

International Olive Council, Olive Oil, technical committee.

Introduction

The International Olive Council (IOC) is an intergovernmental organisation which brings together olive oil and table olive producing and consuming stakeholders. This provides a unique forum for authoritative discussion on issues of interest to the olive industry.

The structure of the IOC revolves around the key figures of the Council of Members and its committees, the chairperson of the Organisation and the Executive Secretariat.

The committee of interest to the Australian Olive industry is the Technical committee, which is described below:

TECHNICAL COMMITTEE

The brief of this committee is to review and discuss the programs and activities proposed by the Executive Secretariat in the areas of olive oil chemistry and standardisation, research and development, technology transfer, training and customised technical assistance for the member countries.

Australia is not an IOC member and by consequence attends the meetings in the status of Observer.

For Australia to be represented at these meetings, the following needs to occur:

1) The IOC needs to invite Australia to be an observer (which means that the Technical committee has accepted Australia as part of the group).

2) The Australian government needs to endorse the chemists that will be attending the meeting.

These steps have been completed and Australian representatives are in the position to attend the Chemists' meeting for the next years.

Methodology

Modern Olives Laboratory Service (Claudia Guillaume) and NSW DPI (Jamie Ayton) alternately attended the International Olive Council Chemists' meetings that are held twice a year in Madrid, Spain.

Outputs

Following each meeting, the Australian representative who attended produced a report highlighting the most important points that were considered to have an implication for the Australian olive industry

These reports were then distributed to the industry representatives through the Australia Olive conference, Technical meetings, and Australia olive meetings.

Outcomes

The main benefits from participating in this meeting for the Australian olive industry are summarised:

i. Access to new proposed analytical methods and/or limits before they become part of international regulations. This will allow chemists to analyse Australian oils to ensure that the changes do not constitute Technical Barriers to Trade for Australian oils.

ii. Australia's accredited laboratories will be better prepared to continue achieving international accreditations (i.e., IOC), which will benefit Australian growers using them to certify their products.

iii. The Australian industry will have inside information about new research, trends in nutritional guidelines, etc. that may be suitable to promote olive oil in Australia.

iv. Local olive growers are expanding and strengthening Australia's influence as a producer on the world stage, through funding an accredited chemist to speak on their behalf their industry at the International Olive Council's chemists' meetings.

Monitoring and evaluation

Following each meeting, the Australian representative attending produced and circulated a report highlighting the most important points having an implication for the Australian olive industry.

The industry reflected on the value of being associated with this project with the following feedback.

"There is no doubt that the information garnered at these meetings has helped Australian growers stay abreast of any new proposed testing methods and limits that may affect how we trade outside of Australia and helped to ensure that Australia's accredited laboratories can continue to achieve important and necessary international accreditations that help facilitate exports. The caliber of the proposed attendees as observers will ensure that Australia's position on technical issues is appropriately considered".

"Australia is a new olive industry and that gives us a different view to some of the more traditional countries – they don't see us as a competitor because we're less than 1 per cent of the world market, but we have a lot of expertise and good resources in terms of technology and research, so they respect us".

"Participation also helps to ensure Australian olive growers and other industry stakeholders are prepared for any changes to international regulations that may affect trade, and that our accredited laboratories that certify domestic products can continue achieving international accreditation".

Recommendations

The recommendations from the outcomes of this project are:

1- Keep participating in those international meetings.

   a. They are were very productive and provided a range of benefits for the Australian accredited laboratories and the Australia Olive Industry in general not only for the information gathered, but also for the networking and promotion of high-quality olive oil outside Australia.

   b. It is challenging and demanding to achieve significant changes in the regulations or methodologies as the transition is very slow, but we must be respectful of other countries and opinions and continue to participate, discuss, and debate the importance of the chemical and sensory issues that are critical to our industry.

2- Keep participating in ring tests or investigations to improve methods and expand the knowledge of new methodologies/technologies

   a. Whilst Australia is a minor olive producer, it has been beneficial in being able to understand other views in relation to olive oil quality and processing from larger producing countries.

   b. It does provide a wider/more informed range of views to participate in research for method improvements. The issues/concerns normally are related to challenges that the local industry is facing.

Refereed scientific publications

NA

References

NA

Intellectual property, commercialisation and confidentiality

No project IP, project outputs, commercialisation or confidentiality issues to report

Appendices

1- October 2014 – IOC meeting minutes report

2- April 2015 – IOC meeting minutes report

3- April 2016 – Claudia Guillaume (CG) meeting minutes report

4- September 2017 – IOC meeting minutes report

5- March 2018 – CG meeting minutes report

6- October 2018 – Jamie Ayton (JA) meeting minutes report

7- April 2019 – IOC meeting minutes report

8- October 2020 – CG meeting minutes report

INTERNATIONAL OLIVE COUNCIL

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Príncipe de Vergara, 154 – 28002 Madrid – España Telef.: +34 915 903 638 Fax: +34 915 631 263 - e-mail: iooc@internationaloliveoil.org – http://www.internationaloliveoil.org/

MEETING OF CHEMISTS ON METHODS OF ANLAYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

Madrid, 2–3 October 2014

MEETING REPORT

The Executive Director welcomed participants, particularly the observer representative sent by the Brazilian authorities. After conveying the apologies of Ms Fernández who was on sick leave, he announced that he would chair discussions.

ITEM 1: The draft amended agenda was approved.

ITEM 2: Proposals for the revision of methods or for the adoption of new methods or limits

1. Amendment of the colorimetric method for the determination of phenolic compounds (**F. Lacoste –** document 2.8 included in document 2.13) and Possible use of SPE for HPLC separation of the phenolic fraction for subsequent HPLC determination (**W. Moreda** - document 2.13)

This proposal is prompted by the current situation where on the one hand there is an IOC method based on high performance liquid chromatography (HPLC) and liquid/liquid extraction and on the other there is interest among laboratories and industry stakeholders in developing a faster colorimetric method. It has emerged in a context where the EU has adopted a health claim for extra virgin olive oils containing more than 5 mg/kg of hydroxytyrosol and derived products which cannot be used because there is no method for measuring this content.

After lengthy discussion, the experts agreed to set up a collaborative test to compare the two methods for the extraction of the compounds intended for measurement (solid-phase extraction (SPE) and liquid/liquid extraction specified in the current method) and two methods for measuring the extracts (HPLC and colorimetry). They also pointed out that the wording of the health claim needed to be reviewed to express the content in millimoles/kg.

It was agreed that W. Moreda would propose an updated version of document 2.13 for circulation by the Executive Secretariat, with the assistance of M. Servili and F. Lacoste. The list of participant laboratories in the collaborative test is given in Annex 1.

2. Combination of three methods (methyl esters, fatty acids and trans fatty acids) into one (**W. Moreda** - document 2.7)

The rationale for combining these three methods is that the IOC standard currently refers to an ISO standard and an EU regulation is currently under review with a view to possibly referring to IOC methods.

After discussion, the experts agreed to delete method B from document 2.7, to change the trigger limit for oil purification from the current proposed value of 3.3% to 2% (which is a grade limit in the IOC standard) and to leave the reference to a minimum 60-m column. E. Billa and K. Ben Ammar did not agree with purifying the oil at an acidity below 2% on the grounds that this is only required in the global method, which is not yet compulsory, and for ECN_{42}.

W. Moreda will make these changes to document 2.7, which will be placed before the IOC Council of Members for adoption in November 2014.

3. Quantification of erythrodiol in absolute terms as opposed to as a % of the sterols (**L. Conte)**

The proposed change is of interest because it will help to provide a more reliable determination of real erythrodiol content in the light of the changes in the concentration of the sterol fraction of the oil, which depends on refining processes. As a result, it will be easier to identify the addition of olive pomace oil or second- centrifugation oil to virgin olive oil.

After discussion, the experts agreed that the change proposed by L. Conte was worthwhile. Before a proposal is framed to the IOC Council of Members, a collaborative test will be carried out using oils with known erythrodiol content to check the robustness of the measurements. The list of participant laboratories is given in Annex 1.

A discussion on a last-minute document regarding HPLC separation of the sterols was deferred until the next day after a brief exchange of views between its author (W. Moreda) and C. Mariani. The outcome of the discussions held the next day was that this is a method used routinely by companies and some public laboratories instead of the thin-layer chromatography (TLC) method used by the IOC. W. Moreda suggested testing the two methods for the fractionation of the unsaponifiable matter – the official IOC method (TLC) and the HPLC method for sterols – during the collaborative test proposed by L. Conte for measuring

erythrodiol content. The experts agreed to modify the collaborative test consequentially. A. Faberi asked for future thought to be given to the choice and volume of solvents in order to reduce the environmental impact of these methods.

L. Conte and W. Moreda will jointly coordinate this collaborative test, which will be open to any interested private IOC-recognised laboratories. The samples will therefore include an olive pomace oil and a sunflower oil with a very high oleic acid content.

4. Determination of free fatty acids (**F. Lacoste** - document 2.6)

The object of this proposal is to adopt a specific IOC method. F. Lacoste pointed out that the proposed method was very similar to that already included in the European regulations and that a collaborative test had already been conducted in September. After discussing the test results (document 6.2), the experts agreed that the method could be validated **once** a further collaborative test had been conducted (in the same laboratories as in the first test) on three samples containing a free acidity of around 0.5%, 0.8% and 2% respectively in order to coincide with the grade limits specified in the IOC standard.

5. Amendment of the K_{270} limit (**Turkey)**

The Turkish proposal was modified to propose solely amending the K_{270} limit for refined oil from 0.9 to 1.05.

This proposal was not discussed because the Turkish expert was absent. A number of experts (W. Moreda, L. Di Giacinto, L. Conte and E. Christopoulou) suggested asking for the following:

- Justification of the assumption that the measure of K_{270} is linear (they do not share this assumption) and of the reasons for proposing a higher limit, which will require stronger refining (higher temperature or the use of more bleaching earth);

- Test results for oils representative of Turkish production;

- Justification of the increase from 0.9 to 1.05 because this lies inside the testing margin of error;

- Other consequences of this change because it will not lead to better quality oils.

These remarks will be forwarded to the Turkish expert for consideration.

6. Amendment of the spectrophotometric method (**L. Conte** - document 2.11)

After discussion, the experts agreed to correct the value for cyclohexane in paragraph 2 to read 270 instead of 268. They also agreed to mention the possibility of referring to a wider range than 0.1–0.8 (section 5.3), the linearity of which has to be checked although not necessarily using reference material given the cost involved (section 3.1.2 should envisage the possibility of allowing laboratories to make the reference solution themselves for calibration purposes). It was also agreed to add a note to section 6.2.

The question of whether to express the measurement of delta K in absolute or real terms was discussed at length, particularly by E. Christopoulou on the one hand and A. Faberi and L. Di Giacinto on the other.

The experts finally agreed to forward their written comments to L. Conte, who will update document 2.11. This update will be circulated to the experts and, if there are no objections, will be submitted to the Council for adoption in November.

7. Amendment of olive oil definitions (**Turkey)**

The Executive Director outlined the Turkish proposal in the absence of the Turkish expert.

The EU representative asked for this item to be discussed in the framework of the debate on the future of the IOC.

Discussions on this matter could go no further because the Turkish expert was not present; it will be conveyed to the Council.

8. Method for the determination of methanol and ethanol (**W. Moreda** - document 2.9)

This subject falls within the framework of the research into the measurement of alkyl esters, of which these two alcohols are precursors.

After discussion, the experts concluded that additional work was needed to improve and better substantiate the proposal. This task was assigned to Drs Faberi, Lacoste, Moreda, Garcia, Izquierdo, Rovellini and Kerem.

9. New method of peroxide analysis (**F. Lacoste** - document 2.12)

This subject was on the conference table because there is currently no IOC method for determining the peroxide value. The ISO method uses iso-octane and the EU method uses chloroform. The idea was to propose two alternatives for laboratories in the same IOC method.

For this reason, a collaborative test had been conducted to compare the two methods, the results for which were presentd by F. Lacoste (documents 6.1 and 6.2). There was a good correlation between the two methods except for sample PA4 for which the reproducibility and mean peroxide value were different. The experts therefore requested Dr Lacoste to contact the laboratories that had had problems with this sample and then to conduct a further collaborative test (with the same participant laboratories) including a sample of refined olive oil containing approximately 1 meqO_2/kg peroxides with a view to framing a proposal to the Council.

10. Amendment of the method for HPLC determination of polyphenols (**P. Rovellini, M. Servili, L. Conte** and **W. Moreda)**

It was agreed that this item could be considered to have been dealt with under item 1.

11. Method for the determination of C_{13}/C_{12} and O_{18}/O_{16} and H_2/H_1 according to the isotope ratio (**A. Faberi** – old item 7)

After hearing about research conducted in Italy as well as about the ongoing European FoodIntegrity project led by the Seville Fats & Oils Institute, the experts agreed that it was too early for provisional IOC adoption of the method proposed by A. Faberi, which aims to trace the geographical origin of Italian olive oils within the framework of protected designations of origin.

This item will be inserted for appraisal in the agenda of the next chemistry expert meeting. The Advisory Committee will be asked for its opinion on the desired level of traceability (PDO, regional national, European, Mediterranean, etc.).

12. Amendment of the alkyl esters and waxes method in order to allow determination of minor compounds (**C. Mariani)**

After discussion, the experts agreed to run a collaborative test before taking a decision of the amendments. The list of participant laboratories is given in Annex 1.

13. Errors in document T.20/Doc No 26 (**C. Mariani)**

The experts agreed to the proposed corrections. The **corrected text will be put forward for Council adoption at the session in November.**

15. Amendment of the method for the determination of aliphatic alcohols (**C. Mariani)**

The experts agreed on using the current method for the determination of aliphatic alcohols also for the determination of triterpene dialcohols (markers indicating the presence of olive pomace oil or second-centrifugation virgin olive oil). The question of whether to propose limits will be broached at a later date.

The change in the title of the method will be proposed to the Council for adoption in November.

16. Global method (letter from **R. Aparicio**)

The Executive Director informed the experts that R. Aparicio had addressed a letter to the IOC, which he had asked to be circulated to participants, in which he asked two questions, namely whether the method validated for the detection of hazelnut oil had also been validated for the detection of adulteration with other vegetable oils and whether the mathematical formulas validated for adulteration with hazelnut oil had subsequently been modified.

D. Garcia, who is a work colleague of Dr Aparicio, gave a historical overview of the development of the global method and proposed conducting a new validation test.

W. Moreda confirmed that the mathematical formulas had not been modified and that a collaborative test organised by the Seville Fats & Oils Institute was underway. Twenty private Spanish laboratories and 20 private Italian laboratories are participating in the test which is meant to determine the addition of new oils in olive oil, particularly high-oleic sunflower oil.

It emerged from discussions that blends of two oils with very different fatty acid compositions might be at the root of the false positives. The reason is that on the basis of the analyses, the model reconstructs the theoretical profile of an oil that does not exist and concludes that the blend is adulterated.

In conclusion, it was agreed that laboratories of the chemistry experts (see Annex 1) would participate in a new collaborative test using 100% authentic oils supplied by A. Buedo, P. Rovellini and W. Moreda. It was also agreed to give the experts the details on the mathematical formulas used to conclude whether or not a sample is adulterated and to take into account the triacylglycerol composition

results (global method) obtained by the laboratories that participated in the IOC recognition proficiency test for 2014/2015. Lastly, the mathematical model containined in the Italian EXEMPLA method will also be used in tandem to check whether it is relevant for the determination of the authenticity of the samples tested.

Consequently, given the work time table, it will not be possible to frame a proposal to the Council in November to make the application of the global method compulsory in the IOC standard.

Old ITEM 5: Study on the determination of copper pyropheophytin (**F. Lacoste)**

F. Lacoste recalled that the Taiwan FDA had proposed two methods for the quantification of copper pyropheophytin, which the Taiwan authorities consider to be a marker for adulteration with colouring agents, in addition to a purification method and two measurement methods (by UV-visible HPLC or mass/mass detection HPLC). These methods were revised in the summer because the matrix was found to affect the calibration of the HPLC mass/mass method. All the texts are being finalised.

F. Ulberth from the Joint Research Council announced that he would shortly be able to send the IOC the HPLC calibration standard, which had been purchased from the sole world suppliers. W. Moreda confirmed that he too would shortly be able to send the IOC the five samples, plus the training sample and accompanying chromatogram and value.

L. Di Giacinto also proposed participating in the collaborative test.

The Executive Director confirmed that as soon as the Executive Secretariat received the samples and the standard, it would prepare the material for shipment to the 17 laboratories that had registered for this collaborative test (2 from Taiwan).

After an exchange of views between the EU representative and the IOC Executive Director, it was agreed that the results would be presented at a meeting held at IOC headquarters in January 2015. As previously, the Taipei Economic and Cultural Office in Madrid would be invited to attend this meeting along with the test leaders (F. Lacoste, W. Moreda and F. Ulberth). At the meeting, it would be advisable to broach the question of the wording of the certificates that importers are currently requested to produce by the Taiwan authorities in order to bring it into line with the work accomplished.

Old ITEM 9: Relations with standards agencies

1. ISO (document 9.1 - **F. Lacoste)**

F. Lacoste commented on Table 9.1 and pointed out that it was the IOC that had requested ISO to register various IOC methods under its name.

L. Conte stressed that the objective was for there to be standards in non-IOC member countries that did not necessarily want to rely on IOC standards or to resolve problems regarding the interpretation of results (tolerance around the limit).

F. Lacoste said that ISO wondered about the advantages for the IOC of having such ISO methods if IOC methods were maintained and ISO methods were not cited in the IOC trade standard.

After discussion, it was agreed that the Council should indicate whether it wishes for the IOC to continue strengthening its cooperative relations with ISO. The experts have no opinion on this matter other than that the ISO methods could be included in the IOC standard when they are ISO-referenced.

2. CODEX: draft campesterol document, feedback from Argentina, opinion of composition working group (**E. Christopoulou)**

The Executive Director gave the floor to L. Conte who had chaired the meeting of the composition working group held on 1 October. He recalled that some years ago the CODEX had requested the IOC to conduct a study on the possible modification of the campesterol limit for olive oils, following a request from Australia. The IOC had requested samples three years in a row but Australia only supplied samples in the first year; moreover, they were samples that had been produced in laboratories as opposed to real samples and were not therefore representative of the realities of Australian agriculture and processing. The IOC nevertheless went ahead with the study and in 2013 the Council adopted a decision tree for oils containing between 4 and 4.5% campesterol. As agreed, a report will be addressed to CODEX requesting them to insert this decision tree in the Codex standard. The draft of this report, co-authored by E. Christopoulou and L. Conte, had been presented to the composition working group, which made comments on the wording. The amended version was distributed to the chemistry experts during the meeting. Dr Christopoulou pointed out the sections of the report that were new and differed from the preceding draft (notably Table 10 and the conclusion at the top of page 14) and thanked all the IOC-recognised laboratories for their contribution to this task.

After an exchange of views, all the experts agreed to relay the report to the IOC Council of Members for adoption and transmittal to CODEX for the February 2015 meeting after some editorial amendments had been made.

These discussions led to a spin-off subject being raised, namely the question of delta-7-stigmastenol.

The Syrian experts present at the meeting requested the IOC to also send the part of the initial report dealing with delta-7-stigmastenol, which had served as the basis for the inclusion of two decision trees in the IOC standard for this parameter in 2013 (one for virgin oils and the other for olive pomace oils).

L. Conte confirmed the validity of the initial report in which nothing would have to be changed before addressing it to CODEX for the February 2015 meeting. On a wider note, the Executive Director wondered whether the February meeting might not be an appropriate time to suggest that the CODEX harmonise its standard with the IOC standard (as regards the limits for stigmastadiene, myristic acid and waxes and the insertion of the parameters and limits for 2 glyceryl monopalmitate and ethyl esters).

The experts agreed to these two approaches. The Council will therefore be presented a report for transmittal to CODEX regarding delta-7-stigmastenol and an overall request for harmonisation of the Codex standard with the IOC standard.

3. California Olive Oil Standard: opinion of the experts

The experts expressed deep concern over this compulsory trade standard for handlers who produce and bottle more than 5 000 gallons of oil per year in California.

The EU representative informed the experts that the European Union intended to write to the U.S. authorities to express its concern on the basis of the comments forwarded by the EU Member States.

After discussion, it was agreed that the chemistry experts would send their comments to the IOC Executive Secretariat, which would then draw up a draft letter for submission to the Technical Committee in November.

Old ITEM 3: Research on the changes in quality parameters during storage

3.1. Ongoing research

L. Conte presented document 3.2 reporting the changes that occur in alkyl esters over time in stored oils, as well as in methanol and ethanol content. This is a complex subject. The only thing that is clear so far is that ethanol (the precursor of ethyl ester) forms through fermentation in the olive fruits and that oil filtration appears to play a role.

J.R. Izquierdo reported on the progress in an ongoing study entailing monthly analysis of several parameters (including methanol and ethanol as of this year) in samples collected from 124 storage vats containing oils produced according to different methods (two or three-phase, horizontal or vertical system, filtered or unfiltered). The results are expected to be available shortly but already show changes that will need to be interpreted.

L. Di Giacinto reported on research underway in Italy to measure the changes over time in all the quality parameters by collecting seven samples every two months. So far, there had been five sample collections. She also reported on a research project conducted by the Italian Ministry of Agriculture on 401 samples showing that the ethyl ester content of extra virgin olive oils is in the vicinity of 9 mg/kg; hence, the IOC limit, which would be fixed at 30 mg/kg in 2014/15, would give leeway for this parameter. The same project has also shown that a two-year shelf life is not reasonable for virgin olive oil because numerous samples did not pass the sensory test.

E. Billa informed the experts that a study was also underway in Greece to track the changes in the content of ethyl esters, tocopherols and polyphenols in samples. The study would be completed by the end of the year but it could already be announced that setting the limit at 30 mg/kg as of 2015 would not pose a problem.

C. Mariani was of the opinion that the presence of ethanol was more important than acidity in encouraging ethyl ester formation and might drive some processors to perform mild deodorisation. He added that on analysing an excellent virgin olive oil stored for 10 years he detected the quasi-absence of ethyl esters but a large amount of methyl esters and winey-like sensory defects.

Several Spanish experts urged against reaching overhasty conclusions because tests showed the influence of extraction type and various other factors on ethyl ester formation.

The experts did not examine the document presented by M. Servili because he was not present.

3.2. The experts did not consider it expedient to comment on the document published by UC Davis on the influence of packaging on oil quality.

Old ITEM 4: Ongoing research on the determination of phenolic compounds (**L. Conte** and **J.R. Izquierdo)**

E. Billa reported that ongoing research on this subject revealed changes in the polyphenol profile but not its total content, which remained stable for the time being.

L. Di Giacinto will also provide results showing a decrease in the more active phenolic compounds.

F. Lacoste informed participants that France's Technical Olive Committee was about to complete a study on this subject, the results of which would be released in 2015. The research was conducted on bottles of oil stored for 11 months in a box in darkness and then one month in the light (to emulate real marketing conditions).

Old **ITEM 6:** Other ring tests held in 2014 and tests for 2015 (**F. Lacoste)**

F. Lacoste reported that she had led an ISO collaborative test on the determination of mineral oils in samples of vegetable oils, including an olive pomace oil and a virgin olive oil adulterated with mineral oil. After the test, the method will be proposed for ISO endorsement in April 2015. A relevant document was sent after the meeting and is attached in Annex 3.

A. Faberi recalled the collaborative test he had mentioned at the previous meeting of the chemistry experts concerning an alternative method for the determination of stigmastadiene. Although this method has the advantage that it calls for less silica and a smaller volume of sample, the results show that its repeatability and reproducibility are too high (double that of the existing method); the attendant report will be ready in March 2015. He added that the working group on method optimisation had proposed conducting a collaborative test. L. Di Giacinto emphasised that this method would perhaps be overtaken by the proposal of C. Mariani (item 2.2. on the agenda) and that it would be better to await the results of his collaborative test before starting that of Dr Faberi. It was eventually agreed to do so.

Old ITEM 7: Study of the isotope ratio method

This matter has already been dealt with under agenda item 2

Old ITEM 8: Information on the chromatogram interpretation workshop; organisation of the 2015 course

L. Conte reported on the workshop held at his laboratory in Udine with support from C. Mariani and W. Moreda; 30 participants attended, 20 with IOC funding. It was generally agreed that the IOC should repeat this training exercise in 2015, particularly on the global method. Seville was suggested as a possible venue (the funding for this course in 2015 has been entered in the budget that will be proposed to the Council in November)

ITEM 10: Summary of points for proposal to the Council in November

In short, draft Decisions will be placed before the Council for adoption in November 2014 on the following points:

- Combination of three methods (methyl esters, fatty acids and trans acids) into one single method (doc. 2.7);
- Amendment of the spectrophotometric method (doc. 2.11);
- Correction of errors in document T.20/Doc No 26;
- Application of the method for the determination of aliphatic alcohols also for the determination of triterpene alcohols;
- Submission to CODEX of the campesterol and delta-7-stigmastenol reports as well as of a more general request for harmonisation of the CODEX standard with the IOC standard;
- Proposal to the Council, if appropriate, of a draft letter addressed to the US authorities concerning the California Olive Oil Standard, based on the comments of the experts;
- Reduction of the lower limit for linoleic acid to 2.5% (discussed under *Other business* and accepted by all the experts present).

In addition, the Council will be asked:

Whether it wishes for the Council to continue building up its cooperative ties with ISO for the publication of joint standards?

In addition, it will be informed that:

- In the absence of Turkish representatives, the proposed amendments of olive oil definitions will be referred to the Council without any recommendations;

- The experts believe it would be very useful to hold another course in 2015 similar to the 2014 workshop held in Udine, particularly on the global method;

- They believe they do not possess results that would prevent the application of the planned reduction of ethyl ester content to 35 mg/kg in 2014/2015;

- They are not able to propose making the global method compulsory at this juncture;

- They believe the IOC will be able to hold a meeting in January 2015 to release the results on the methods proposed by the Taiwan Food and Drug Administration for the determination of copper pyropheophytin content in olive pomace oils, which is considered a marker for adulteration with colouring agents.

ITEM 11: Future work

A recap of all the scheduled tests was given under this item.

ITEM 12: Other business

12.1. The Executive Director reported that he had received an e-mail from Dr Grompone regarding the solvent used in the global method which she said was very toxic and highly inflammable. If she was obliged to use it, her University would impose draconian measures for its use (gloves, mask, alarm, etc.). The experts spoke about their experience in this respect (localised aspirators, etc.) and finally suggested that the IOC initiate a debate on the possibilities of replacing certain solvents in the IOC methods (notably hexane and cloroform) by less toxic products.

12.2. Dr Garcia presented the FoodIntegrity project questionnaire that would shortly be posted online and which the IOC could help to circulate. The questionnaire is for a survey of market sensitivity.

12.3. The experts were not able to examine a document that had arrived at the last minute concerning the adulteration of an Iranian olive oil because the Iranian expert was not present at the meeting.

12.4. The EU representative reported that following the call for research proposals on olive oil authenticity issued in early 2014, the name of the consortium that would carry out this project would soon be known. She also reported that a call for applications (closing date 31 October 2014) for the selection of EU experts appointed in a personal capacity for the EU group of chemistry experts had been published in the Official Journal on 17 September 2014.

ANNEXES

Annex 1:

LISTS OF PARTICIPANT LABORATORIES IN COLLABORATIVE TESTS:

Collaborative test on the determination of phenolic compounds
12 laboratories

Collaborative test on the global method
16 laboratories

Collaborative test on the determination of erythrodiol content
23 laboratories (to be made open also to private, IOC-recognised laboratories)

Collaborative test on the determination of minor compounds using the method for the determination of alkyl esters and waxes
14 laboratories

Annex 2:

Final campesterol report

Annex 3:

Results of ISO collaborative test on mineral oils (F. Lacoste)

INTERNATIONAL OLIVE COUNCIL

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Príncipe de Vergara, 154 – 28002 Madrid – España Telef.: +34 915 903 638 Fax: +34 915 631 263 - e-mail: iooc@internationaloliveoil.org - http://www.internationaloliveoil.org/

MEETING OF CHEMISTS TO STUDY METHODS OF ANALYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

Date: Restricted group on the optimisation of COI-referenced testing methods
8 April 2015
(09.30–17.30 hours)

General group
9–10 April 2015
(09.30–17.00 hours)

Place: International Olive Council
Príncipe de Vergara, 154
Madrid

MEETING REPORT

The Executive Secretariat of the International Olive Council (IOC) convened the chemists officially designated by the IOC member countries to cooperate in studying methods of analysis for olive oils and olive pomace oils. The meetings took place at IOC headquarters in Madrid. Specifically, the restricted group met from 09.30 to 17.30 hours on 8 April 2015 while the general group met on 9 and 10 April 2015. Although scheduled to last from 09.30 to 17.00 hours, the meeting of the general group ran on until 17.30 on the 9th and until 18.00 on the 10th. Invitations to attend were also sent to observers from non-member countries, two of which sent representatives (Australia and Canada), as well as to representatives of standards agencies, none of whom attended on this occasion. Five representatives from the IOC Advisory Committee on Olive Oil and Table Olives also took part.

The Executive Secretariat was represented by the Executive Director, Jean-Louis Barjol, the Deputy Director, Ammar Assabah, and the Head of the Chemistry & Standardisation Unit, Mercedes Fernández Albaladejo.

The Executive Director emphasised the importance placed by the IOC on the work of the chemistry experts as well as on the harmonisation of international standards and national rules and regulations with the IOC trade standard, the aim of which is to promote trade, prevent fraud and protect consumers. He reported that the representatives of the Taipei delegation had again been invited to take part in the discussions on agenda item 2(c) concerning the study on copper pyropheophytins; however, they had notified that they would not be able to attend.

After running through the agenda business, the Executive Director urged Members to be proactive. He thanked the experts for their cooperation and the excellent job they were doing despite the delay in conducting the collaborative tests scheduled at the previous meeting of the working group in October 2014. Lastly, he stressed it was important to forward meeting documentation with more lead time – at least one week in advance – to allow it to be posted on the website and examined beforehand.

The list of attendees is given in document T.20/Doc. No 71-4.

The business listed on the provisional agenda (T.20/Doc. No 71-1) was discussed at the meeting and is detailed below:

1. Adoption of the report of the meeting held on 2 and 3 October 2014 (T.20/Doc. No 70-3)

The report on the previous meeting was adopted.

2. a. Update on the documents adopted at the 102nd regular session (November 2014) and 23rd extraordinary session (February 2015) of the Council of Members

The Executive Director gave an update on the documents proposed by the expert group, which had been presented to the Council of Members in November 2014 and adopted at the 23rd extraordinary session in February 2015, namely:

- Revised method for the determination of aliphatic and triterpenic alcohols by capillary column gas chromatography (COI/T.20/Doc. No 26)

After Dr Lacoste pointed out that no chromatograms had been included for triterpenic alcohols, Dr Mariani suggested including the chromatograms that he had sent for the last meeting; the rest of the experts agreed with this suggestion. They also agreed not to modify the internal standard for the time being because any change of this type would require fresh validation.

Dr Moreda proposed assembling the alcohol compound methods in a compendium together with the HPLC method.

At that point, Dr Faberi proposed modifying the internal standard. The modification could be validated in October and presented for adoption in November 2015. In the meantime, he proposed also including a photograph in the recently revised method.

After a wide-ranging discussion by several experts on a number of possibilities, it was unanimously agreed to move forward in two stages. First, Dr Lacoste would insert the missing chromatograms and photograph in the method (**Annex 1**). Then, Dr Moreda would organise the validation of the new method for the determination of alcohol compounds incorporating a new internal standard. This would be done before the next chemists' meeting in October 2015, with a view to proposing the method for adoption at the 103rd session of the Council.

- Combination of three methods of analysis (methyl esters, fatty acids and *trans* fatty acids) into one single method entitled *Determination of fatty acid methyl esters by gas chromatography* (COI/T.20/Doc. No 33)

- Revision of the method for *Spectrophotometric investigation in the ultraviolet* (COI/T.20/Doc. No 19/Rev. 3)

- Reports on campesterol and delta-7-stimgastenol and submission of a proposal to CODEX, with the agreement of the Council, to amend its standard on the basis of these two reports and to initiate full harmonisation of all the divergent parameters

- Revision of the trade standard for olive oils and olive pomace oils (COI/T.15/NC No 3/Rev. 7) to lower minimum linoleic acid content to 2.5%

- Revision of the method for the organoleptic assessment of virgin olive oil (COI//T.20/Doc. No 15/Rev. 6)
- Lists of chemical and sensory testing laboratories awarded IOC recognition for 2014/15

b. Outcome of the 24th session of the Codex Committee on Fats and Oils (CCFO), Malaysia, February 2015

The Executive Director reported the conclusions of the above CCFO session. Specifically, he informed the experts that the CCFO had agreed to request CAC38 to give its approval for the revision of Section 3 of the standard for olive oils and olive pomace oils (CODEX STAN 33-1988) as new work in order to find an answer to the problem of genuine virgin olive oils with a campesterol content above the current limit.

It also proposed creating an electronic working group (EWG) led by Argentina and co-chaired by Australia and Italy, open to all Members and Observers and working in English only, subject to approval by the Codex Alimentarius Commission. The objective of the EWG is to present a proposed draft revision of Section 3 to the Codex Commission for circulation at Step 3 and consideration at its next session. If no agreement is reached or work is not completed, the current campesterol limit will remain as it is.

Dr Ben Ammar and Dr Faberi (who will be representing Italy, the co-chair of the EWG) were also present at the CCFO session and corroborated the information provided by the Executive Director.

The Executive Director mentioned that he had suggested to Argentina, Australia and Italy that the meetings of the IOC chemistry experts could serve as a meeting point to support the work of the EWG and that the IOC would pay for the travel expenses of the leader and co-chairs of the EWG. This suggestion had met with a positive response.

Dr Guillaume, the observer from Australia, confirmed that Australia was willing to act along these lines and to build on the work performed on campesterol in conjunction with Argentina.

c. Ring-test held in 2014 on **methods of analysis to determine copper pyropheophytin in olive pomace oils** (**Annex 2**): The test coordinator (Dr Lacoste) outlined the results of the ring-test in which the IOC had collaborated. She explained that crude olive pomace oil contains naturally high concentrations (50 mg/kg) of this compound while refined olive pomace oil may contain levels between 50 and 300 μg/kg. The ring test revealed that the HPLC/UV method is more precise than the HPLC/MS/MS method, which would need to be reviewed before conducting another ring test to enable a feasible limit of not less than 100 μg/kg to be fixed. However, since no representatives were present from Taipei, the experts were unaware of their opinion and did not know if they would be available for such a ring test. It was therefore decided to postpone work until this matter could be agreed.

Dr Cobo presented chlorophyll testing data for olive oils shipped to destinations other than Taiwan which confirmed the conclusions of the study. He wondered whether it would be advisable to include a limit fixed by national health authorities to detect fraudulent mixtures among IOC parameters instead of applying the IOC standard and methods. In his opinion, the issue with the Taiwan authorities had been resolved and the exports tested complied with the 50 ppb limit. In the case of pomace oils and grapeseed oils that are over this limit, exporters could use the document proposed by the Taipei authorities stating that copper chlorophylls are not employed in the product.

By way of a conclusion, several experts expressed concern over establishing a limit in this case since it would have to be applied to all olive oils in other markets. Moreover, it would set a precedent for imposing parameters of this type which seem to be technical barriers more than real protection for producers and/or consumers.

d. The Executive Secretariat reported that the **restricted group for the optimisation of testing methods** had met the day before (8 April). In line with the remit drawn up for the group when it was created in April 2011, the purpose of the meeting was to examine proposals in the light of established priorities and to organise the necessary collaborative tests. Any proposals framed by the restricted group, currently coordinated by Dr Lacoste, are presented to the broader general group which also arranges ring tests involving larger numbers of participant laboratories. Membership of the restricted group has been voluntary since it was set up. Depending on the subject area concerned, the addition of new members is proposed but they must be committed to cooperating and performing the necessary collaborative tests. It was agreed that the proposals of the restricted group would be included under agenda item 3.

3. Study and update of the methods of analysis cited in the IOC trade standard: conclusions of the restricted group on the optimisation and review of COI-referenced testing methods

At the previous chemists' meeting in October 2013, it was considered necessary to revise a number of COI-referenced methods and to draft COI methods to determine peroxide value and free acidity. To do so, it was proposed setting up a restricted voluntary working group. However, the composition of the group was not sufficiently representative. Consequently, the Executive Secretariat decided to invite Members to designate an official representative if they wished.

This restricted working group met for the second time at IOC headquarters on 8 April 2015. The outcome of their work was presented under agenda item 3 for the meeting of the chemistry experts.

The list of participants is given in the document referenced T.20/Doc. No 71-5.

a. Methods for the determination of phenolic compounds: At recent meetings, the experts placed top priority on revising the current method for the determination of phenols, comparing a colorimetric method with an HPLC method and comparing two methods of extraction (SPE and liquid/liquid).

The experts decided to use iso-octane as a solvent instead of hexane. They also decided to use a hydrolysis method for nutritional claims and to study the methods proposed (Prof. Conte and Drs Moreda and Rovellini). Dr Servili proposed selecting a reference method which each laboratory would compare with the method it usually employs. **It was agreed that this method would be selected** by Drs Moreda, Rovellini, Lacoste and Servili and Prof. Conte. Dr Lacoste reported that there was a coefficient of correlation between hydroxytyrosol and tyrosol that needed to be checked in each laboratory for the colorimetric method (Folin). A collaborative test will be organised on 5–6 samples at different concentrations. Three experienced laboratories (Drs Lacoste, Moreda and Servili) will analyse the first samples, following which the test will be enlarged to the rest of the laboratories listed in the relevant annex to the report on the October 2014 meeting. It was agreed that the results would be expressed in tyrosol equivalent.

b. Quantitation of erythrodiol (TLC and HPLC)

At previous meetings, Prof. Conte proposed revising the calculation of erythrodiol+ uvaol (E+U) because he considered it advisable to measure absolute as opposed to percentage content, which is not real because sterols decrease more than E+U during refining.

It would be necessary to examine the validation data and check the correlation between E+U and the internal standard. The first step would be to study the changes in samples, including samples outside the limits, and then to arrange a collaborative test.

Dr Moreda also proposed reviewing the alcohol compounds method and including the revision in the compendium. It was decided that Dr Moreda and Prof. Conte (who was not present at the meeting) would agree on the test samples for the restricted and general collaborative test, as well as on the timing of both. It was agreed that the general collaborative test would be held ahead of the next meeting.

c. New IOC method for the determination of free acidity: Dr Lacoste was in charge of drafting the final version of this method. She pointed out that since samples with an acidity between 0.2 and 6% had not been available for the first test, it would be advisable to coordinate a second collaborative test on samples containing a free acidity of 0.5%, 0.8% and 2%. It was agreed to organise the collaborative test with the laboratories included in the list appended to the previous meeting report**.**

d. New IOC method for the determination of peroxides: The first collaborative test did not give satisfactory results. It needs to be checked whether the two methods [chloroform (EU) and iso-octane (ISO)] give the same results. The method will be revised and circulated for comment. It was agreed that a reference sample would be sent before the summer to allow each participant laboratory to practise. A new collaborative test will be run under the coordination of Dr Lacoste. Depending on their skill, laboratories will use one or other of the solvents (or both) in order to check whether the

two protocols for this new IOC method give the same results. The test will be held in September to avoid the hottest months of the year and labs will be committed to performing the analyses within 3–4 days. The list of participants is appended to the report on the October 2014 meeting.

e. Inclusion of the new method for the determination of minor compounds in the method for the determination of alkyl esters and waxes

Dr Faberi organised the scheduled collaborative test on this method. As it had proved complicated to interpret, he suggested adding qualitative and quantitative chromatograms. He proposed revising the method together with Dr Mariani at the end of April; this proposal was accepted. The IOC will then circulate it for comment within one month and a small ring test will be held, coordinated by Dr Faberi.

Dr Mariani gave a very enlightening presentation demonstrating the importance of detecting these minor components. This showed that many of the fats and oils available to the present-day food industry do not contain sterols. To detect them in virgin olive oil, it is crucial to add the qualitative identification of minor components to the determination of sterols.

f. Global method:

The Executive Director summed up the discussions held on this matter the day before at the meeting of the restricted group at which, after extensive discussion, the experts unanimously agreed to propose leaving the method as it is, but amending its scope to limit it to the detection of hazelnut oils in olive pomace oil and lampante virgin olive oil, and to work subsequently on enlarging its scope.

Drs Moreda and Buedo reported that they had been working on the method and that the Argentine samples had been analysed. The first expert pointed out that the method does not require the usual kind of validation but qualitative validation. He explained that it has proven to be useful for the detection of specific types of fraud such as the admixture of hazelnut oil, desterolised high-oleic sunflower oil, second-centrifugation oil and olive kernel oil; however, the detection threshold needs to be checked. He added that he was going to try to modify the software so that it gives not only a Yes/No type of reply (qualitative) but also a numerical value (quantitative) justifying the Yes/No answer. This would enable better appraisal of the result.

Dr Ollivier proposed drawing up a decision tree.

The EU representative confirmed that the object was to bring the EU regulation into line with the IOC standard.

Dr Diaz was worried if this method identified the presence of second-centrifugation oil in virgin olive oils because his interpretation of the EU regulations is that this would not be prohibited. In his opinion, it would be a cause for concern if a method were to fix limits that went beyond the standard. Dr Mariani commented that until 1997, numerous samples of second-centrifugation oil had been studied. The method had been ruled out because it was impossible to identify whether the oil was lampante virgin olive oil or olive pomace oil. Dr Moreda added that in theory, if second centrifugation is performed at a low temperature there is no problem. If it is not, there is more probability that the oil will be extracted from the kernel of the olives.

After intense debate (over two days), unanimous agreement was finally reached on recommending not to delete the method but to leave its status as it was since it was adopted for entry into force on 1 January 2014. The method will continue to be official but its application will not be compulsory for the time being. The results that were published will be modified to add the numerical values justifying the results, which are currently limited to Yes/No. It was recommended that Dr Moreda continue research in order to extend the method to the detection of the presence of other oils, over and above the mere detection of hazelnut oils for which it had been validated and continues to be so. Australia pledged to ship samples for inclusion in the study.

4. Possible new work (Annex 3)

a. Method for the determination of methanol and ethanol

Dr Moreda presented a method for the determination of methanol and ethanol (**Annex 3**), which he put forward as a laboratory tool as opposed to a testing parameter in the IOC standard. The experts discussed the different methods they used for this purpose and whether or not the proposed method should be validated since it will not be included in the standard nor will any limit be fixed. They finally decided to validate it at a later stage. The possibility was broached of the posting a library of such reference methods on the IOC website once they have been validated by the IOC, even although they are not included in the IOC standard.

b. Determination of isotope ratios

Dr Faberi proposed provisionally adopting this method on which he has kept a database for more than 15 years. He commented that it gives very good results in identifying the geographical area of production of genuine oils and could be included in the library of methods mentioned in the preceding section so that countries could contribute data to help to expand the database. He confirmed that it is useful for PDO oils but not for blends of oils from different geographical origins.

Dr García referred to the Foodintegrity Project and Dr Iosifidou mentioned that C_{13} is used in Greece for honey and other products; she added that she could only provide data for this isotope. Dr Faberi answered that this would not be sufficient because three parameters are needed. Drs Ollivier and Di Giacinto expressed potential interest in collaborating on this subject. The Executive Director told the experts that this matter had been put to the Advisory Committee, but had not generated any interest.

By the end of the discussions on this point, the experts had not reached any agreement on either adopting the method provisionally or including it in a compendium of methods published in a library on the IOC website.

c. Determination of stigmastadienes (direct method)

A. Faberi said that in his opinion this method should be improved (as regards the solvent used), but not withdrawn. It was agreed to hold a restricted collaborative test (Drs Lacoste, Di Giacinto, Faberi, Appruzzesse and Cobo) to check and exchange chromatograms, after which the test will be opened up to more participants.

d. Other

Dr Moreda proposed modifying the method for the determination of alcohol compounds to combine aliphatic and triterpenic alcohols (absolute values) and adding HPLC. After it has been revised, it will be circulated for comment and subsequent validation.

5. Ongoing research

a. Research on changes in quality parameters

At the close of discussions on this item, it was agreed that its importance warranted a separate report. In the final analysis, the question is whether the experts recommend the Technical Committee and the Council to leave the ethyl ester limit at 35 mg/kg as of 1 October 2015 or to lower it as planned to 30 mg/kg. The detailed report is contained in **Annex 4** along with the presentations given by the various delegations while the abridged report is given below. The Executive Secretariat wishes to emphasise that solely the experts from one single Member (EU) expressed an opinion in the debate on this matter in which two opposing stances emerged. The experts from the other Members did not express an opinion except for Dr Ben Ammar who highlighted it was important to take a decision before the start of the next crop year. In the case of the observers from non-member countries, the Australian observer supported the request made by the Spanish experts while the Canadian observer mentioned that the oils entering Canada have a content of less than 30 mg/kg.

"ABRIDGED DRAFT REPORT OF THE DISCUSSIONS HELD BY THE IOC CHEMISTRY EXPERTS ON THE ETHYL ESTER LIMIT SPECIFIED IN THE IOC TRADE STANDARD AS OF 1 OCTOBER 2015

Introduction: This matter was discussed under item 5(a) of the agenda for the chemistry experts meeting held on 10 April. It has been considered advisable to draft a separate report on this part of discussions in order to inform the Technical Committee specifically on this subject in case it wishes to request the Council to decide on 19 June to postpone the automatic amendment of the ethyl ester limit which otherwise will be lowered to 30 mg/kg as of 1 October 2015. It should be noted that this parameter is a quality parameter like free acidity, peroxide value, spectrophotometric values and sensory analysis.

Summary:

The chemistry experts from Spain began by presenting the results of four studies on the basis of which they requested the non-reduction of the ethyl ester limit of virgin olive oils to 30 mg/kg as of 1 October 2015.

The other experts who expressed an opinion on this matter all belonged to the same IOC Member, i.e. the European Union. These experts, who were from France, Greece and Italy, based their statements on the findings of research already reported in October 2014 (Italians) or as yet unpublished (French and Greeks).

No expert from any of the other IOC Members attending the meeting presented other results.

The expert from Australia was the only observer to speak. She suggested leaving the limit unchanged until the experts reached an agreement on this matter.

In short, there is disagreement among the EU experts with the Spanish experts on one side and French, Greek and Italian experts on the other. In the opinion of the Spanish experts, which is not shared by the others, the ethyl ester content of virgin olive oils is influenced by the following:

- Method of oil extraction (two- or three-phase). In their opinion, low levels of ethanol (precursor of ethyl esters) are produced as a result of enzymatic processes that occur during ~~malaxation~~ crushing as well as of the choice of extraction method (two-phase); the final concentration depends on the quantity of water used in the process. The other experts believe that ethyl ester production is independent of the extraction method and is explained primarily by fermentation caused by poor working conditions that could be improved

- Ethyl ester content increases with time. This increase depends on the initial ethanol content, storage conditions and acidity

- Variety (the experts from Italy and Greece have not observed this effect).

The Spanish experts do not believe a correlation exists between ethyl ester content and sensory analysis, contrary to what the other experts think.

In the opinion of the Spanish experts, the reduction of the limit to 30 mg/kg would lead to the downgrading of 9% of the Spanish oils considered to be extra virgin on the basis of the other quality parameters, a situation that would not take into account the significance of Spanish production. The other experts take the view that extra virgin olive oils will be downgraded to the virgin category in all countries but this will send out a signal to the industry to continue making efforts to improve product quality, as it has done in the past."

5.b. Selection and amount of solvents

For safety and environmental reasons, Dr Ollivier proposed deleting hexane whenever possible and cited some examples of methods in which this had been possible (phenols: iso-octane, ethyl acetate; stigmastadienes: iso-octane; 2MP: iso-octane, ether; sterols: iso-octane, acetone; alky esters and waxes ...).

It was agreed that she would send a document to the IOC containing protocols and a concrete proposal for consideration at the next meeting of the restricted group on testing methods.

5.c. Amendment of K_{270} limit

Speaking on behalf of Turkey, Dr Tibet presented proposals (**Annex 5**) for the amendment of various limits (free acidity for lampante oil, K_{270} for olive oil and refined olive oil) as well as decision trees for lampante oils (for campesterol, beta-sitosterol and delta-7-stigmastenol) and a change of designations.

After the experts proposed submitting it to the composition working group, the Executive Secretariat explained the protocol that had to be followed (shipment of three

samples plus testing certificate and questionnaire). The Executive Secretariat will then send the samples to another two laboratories in different countries, following which the results will be examined by the expert group. With respect to the proposed change of designations, it was emphasised that the Working Group on the Future of the IOC had decided to take charge of this matter; hence, Turkey should raise this matter at the meeting of this WG in May.

5.d. The Iranian expert gave a presentation on proteomics (**Annex 6**). He will keep the expert group informed on any developments in this work in which several experts expressed interest.

5.e. Other

Dr Cobo presented a proposal regarding ΔK (**Annex 7**) which showed in graph form (page 1) that this parameter really does indicate peak height at the maximum wave length (λmax) from the line joining K λmax-4 and K λmax+4. Page 2 of the same proposal shows that in the case of extra virgin olive oils the graph in this range is concave and the value is negative. He reminded the group that both Dr Christopoulou and he had already commented on this fact at the October 2014 meeting. Consequently, it did not make sense to obtain the absolute value of this parameter. Dr Di Giacinto rejected this proposal, which will be examined at the next meeting.

6. Update on IOC activities

a. 2nd international workshop on chromatogram interpretation (Seville, Spain, 25–29 May 2015)

The Executive Director informed the group about this workshop, which is directed at technical officers from recognised control laboratories in IOC member countries. The IOC will defray the participation of 20 attendees as it did in 2014.

The workshop programme will cover the methods currently being studied:

- Sterols (HPLC);
- Phenols;
- Triacylglycerols;
- Global method;
- Methanol/ethanol;
- Minor components;
- Stigmastadienes.

The draft programme and provisional list of participants are attached (**Annex 8**).

7. Relations with other standards organisations and participation in meetings

The Executive Secretariat reported on the status of IOC relations with ISO TC 34 SC 11, which last met in Berlin (Germany) on 2–3 April 2014. The next meeting is to be held in Amsterdam (Netherlands). The IOC will attend in its capacity as a liaison organisation and will present an activity report besides collaborating in ongoing work.

12. 14. Priorities and future work; other business

Ms Valentin informed the group about the seminar being organised by the EU and its Joint Research Centre (JRC) on the organoleptic assessment method. The seminar will be held in Milan (Italy) on 2 October 2015 and will be open to all countries, which is why the IOC has applied to attend.

Dr Faberi announced that the Italian ministry would also be at EXPO MILAN. It planned to take a stand at which a panel would teach about organoleptic defects.

The Executive Secretariat ended by thanking the experts for their cooperation and inviting them to attend the next meeting, scheduled to take place at IOC headquarters on 15 and 16 October 2015.

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IOC meeting of chemists on methods of analysis for olive oils and olive pomace oils

21 and 22 April

Minutes taken by Claudia Guillaume

1) Approved the report of the meeting from October 2015
2) Institutional update
    a. Several points: was approved the FFA method, premio Mario Solinas, reconocimiento de los laboratorios por el COI, Sistema organoleptico, etc.
    b. eWG update: Alejandra presented the position. Efi (Greece) stated that was very happy to hear that Australia removes the 1.9 in stigmasterol and adopted the level of Argentina. The person from Codex highlighted the importance of preserve the no adulteration of the EVOO. Regards to the calendar states that is not a big deal to decrease a few weeks the date of the final report from the 30/09 to 15/10. **Nobody else make any comments.**
    c. IOC is going to present another proposal to the Codex, this proposal will be presented in the Feb-17 meeting. Codex member states IOC as an observer can present a proposal but for them to do a eWG it has to be presented by a member country. Date for comments finish on 15/05.
3) Results of colaboratives studies
    a. Determination of peroxide. The proposal is to use chloroform and thiosulfate 0.01N. Florence Lacoste made the presentation and only Dr Cobos from Spain was not happy with the RSD of the method, he thought that was too low.
    b. Study collaborative test on pesticides. Dr Izquierdo made a point regards that the export to USA, Japan, and other countries is being very complicated because many pesticides are not with a limit in the Codex and/or EU, so by consequence the imported countries consider that the value should be 0 (not even accepting traces). IOC proposes that a eWG be formed to copilate data about which are the pesticides used, the limits that they are using, which regulations are following and which method is used to determine the value. The eWG will be chaired by Italy (Dr Generalite), several countries (turquie, france, spain, Portugal, iran, etc will be part of the group) also FDA will be invited. I think that would be good for Australia to be part of the group, because we do have the same issue when exporting to USA, but I did not put my hands up because wasn't sure if we want to give information about the pesticides used and the limits. If we are interested we could send an email to confirm our participation.
4) Proposed work
    a. Determination of Stigmastadienes. Dr Faberi presents a new method with a detection limit lower than the actual method (< 0.01 ppm). A WT will be organized to validate the method. Australia will participate.
    b. Determination of minor components with the method of FAAE and Wax. Dr Faberi presents a new method. Did not have much acceptance and there is not voluntary labs to try/validate this method. Dr Faberi asked Spain and France to collaborate and they have accepted.
    c. Determination of copper chlorophylls in table olives and olive oils. Dr Faberi presented a qualitative method. It is a method to determine fraud in table olives, because copper is

added to the table olives to make them shinier and look more eatable, but it is not allowed. Dr Servilli pointed that he has tried the method and it works. The only issue is that the method is qualitative so only mention absence or presence and has been seen that the some processes could develop copper from a natural way, so he think that a limit should be important for this method, which make the difference between natural copper and addition of this one. Dr Faberi requested to the IOC to put this method as an official method.

d. Review of the document T.20/Doc 42. A new eWG was formed leaded by Italy (Dr Luciana).
e. Other. Dr Cobos proposed to unify the method of FAAE and Wax, due that everyone already is using the cleaning with hexane before the FAAE elution (so to review the method T.20/Doc 28). Dr Conte made the comment about that FAAE is only for EVOO and the method of Wax is designed for all the OO grades. Dr Cobos will review the method T.20/Doc 28 and make the proposal to the IOC for the next meeting.

5) Discussion on ongoing studies
    a. Research on volatiles compounds. Dr Conte and Dr Izquierdo commented that they could not give any answer about the researches because they could not finish the job as was planned. They will bring the data for the next meeting.
    b. Choice and quantities of solvents in IOC methods. Dr Ollivier (France) mentioned about the need to reduce the amount of toxic solvent used. Dr Ollivier proposed an order of IOC methods to be reviewed to minimize or replace toxic solvents for other with less toxicity to prevent the health of the lab technician as priority number one but also the environment. IOC made the commitment that will review and consider.
    c. Was discussed within 3d.
    d. BBD: Mercedes (IOC) wants that the group proposes a date for the BBD, as per the labelling document T.15/NC 3. One of the works proposed is about PPP, the variation of the PPP during time. It is a work done by Dr Aparicio “Does BBD embody EVOO freshness”. Dr Conte mentioned that his group is working in a mathematic formula including several simple tests that could predict the BBD. Dr Luciana proposes that the BBD be limited to 12 months (the whole group ignore this comment). The IOC president urge about the need to have a solution and the group should come up with a maximum date. I have made comments about what happens in the Australian standard, regards the maximum in BBD and also tools that may help to the producer how to predict it.
6) Other information
    a. Assistance and training to recognized laboratories. Mercedes solicited that a protocol be done to determine the way and who will be the experts that could assist to the different laboratories to train/assist them. It will be for chemical and sensory laboratories.

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Príncipe de Vergara, 154 – 28002 Madrid – Spain Tel.: +34 915 903 638 Fax: +34 915 631 263 - e-mail: iooc@internationaloliveoil.org - http://www.internationaloliveoil.org/

MEETING OF CHEMISTS TO STUDY METHODS OF ANALYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

Date: General group
25 and 26 September 2017
(9.00 h – 17.00 h)

Place: International Olive Council
Príncipe de Vergara, 154
Madrid

MEETING REPORT

The meeting of chemists to study methods of analysis for olive oils and olive pomace oils was held on 25 and 26 September 2017. Invitations to the meeting were extended, in addition to the representatives of the member states, to observers from non-member countries, to standardisation bodies, and to five representatives of the Advisory Committee.

The meeting was chaired by the Executive Secretariat of the IOC.

The list of participants is contained in document T.20/Doc. No 75-4.

1. Adoption of the report of the meeting held on 6 – 7 February 2017

The report of the meeting held in February was adopted without objections.

2. Institutional agenda

2.1 CODEX ALIMENTARIUS:

Juan Ramón Izquierdo explained that he was the representative of Spain in the Codex Alimentarius Working Group, the chair being held by Spain in coordination with the IOC, as proposed by the EU, while the vice chairs were held by Argentina and Canada. The group should be coordinated without consideration of national interests. The IOC was part of the electronic working group and held observer status in the Codex Commission.

He further more indicated that each country had one single spokesperson. The group was open to all interested parties, and its work was conducted through a platform created by the CODEX, which required registration in order to be able to participate. Although the deadline to register had passed, CODEX was open to allowing new countries to join. Croatia and the IOC were present as observers and there were 22 member states, but not all of those were in the electronic working group for the revision of standard and would not have access to its documents or observations.

A number of reference standards had been selected (Argentina, Australia, California, EU, South Africa, USA, IOC). Although the national standards were very similar, definitions had been identified as potentially problematic. The definitions and parameters were included in some 30 tables. The group would work on the basis of consensus, whereby opposition to any proposal, would require a reasoned justification. Once a point had been concluded it would not be reopened for lack of time. The document should be ready for September 2018, since the group had indicated that it had to be presented four months prior to the meeting.

The group's remit did not include the review of limits, given that it could cause a divergence between various countries. Its work therefore concerned the harmonisation of the standard itself. The review of the standard's limits was the responsibility of the IOC working group.

Jaime Lillo said that a working group would be created to make the relevant observations. Certain delegations were reticent with regard to the IOC, especially the CODEX members that were not Members of the IOC.

The IOC would act as an observer. The IOC had accordingly resumed its institutional visits to a number of countries, such as the USA (end of July), and observers from the USA and Australia were present at that meeting. It had also attended a meeting in Switzerland to coordinate the functions of the various international bodies (IOC, ISO, etc.), organised by the CODEX secretariat.

2.2 Meeting of the Council of Members

The following countries had ratified the 2015 Agreement: Argentina, EU, Lebanon, Montenegro, Morocco and Tunisia.

Mercedes Fernández reported on the revision of the act for the recognition of laboratories, and on the situation of the revision of methods for the detection of extraneous oils, which had been examined but had not been adopted.

2.3 WCO

The IOC had participated in the last meeting of the WCO in May, at which Australia and Russia made a number of objections. The IOC would participate in the next meeting in December 2017 and would attend the meeting of the Scientific Sub-Committee of the WCO in January 2018. The IOC had been requested to present its standard and that of the Codex, but the latter only referred to edible oil. That was the reason why the IOC standard had been used for inedible oils. A first phase would run to 2019, and the second phase would run to 2022. Ordinary virgin olive oil only figured in the IOC trade standard, where it was only included in terms of its market volume. The document would act as the basis for the document that could potentially be adopted by the WCO.

The European Commission wished to study the document before giving its opinion.

- On the second day of the meeting, a new document was distributed. It included the indications made by Mr Ben-Amar and the EU delegation, and referred to the

colour of refined olive oil, which were included in the explanatory notes of Chapter 15.

2.4 Sub-committee ISO 34 SC11

Florence Lacoste said that the group of experts of the ISO had discussed Standard T20 on aliphatic alcohols and triterpene dialcohols. It had indicated that the benzene-acetone mixture should continue to be used for the purification of aliphatic alcohols. However, benzene was no longer kept in many laboratories.

Precision values should be included for triterpene dialcohols.

A proposal by Carlo Mariani, supported by Efi Christopoulou, had been approved in the meeting of chemistry experts in 2014. It provided access to information on oils from the second centrifugation stage. While it was an interesting proposal, it did not establish limits.

It made no sense not using the method for triterpene dialcohols. Methods were sometimes adopted too soon, leading to small errors.

The validation could not be carried out before the Council meeting in November.

Hermenegildo Cobo confirmed that no one used benzene. Furthermore, the method was contained in Regulation No 2568, without having been adopted by the IOC. It was however about to be adopted as an ISO method, requiring its discussion in relation to the IOC standard, adding the precision values. It contained no limits for oils.

He proposed that the revision should be carried out by the Italian delegation, since it had been the one to propose it.

Lanfranco Conte said that if another mixture was to be used, the method would have to be validated in reference to the new mixture.

Wenceslao Moreda said that triterpene dialcohols served no purpose (the proposal had been presented by the Italian delegation). The tendency was towards unifying the methods and it made no sense to have different methods for sterols and for triterpene dialcohols when the methodology was the same. The only difference lay in the band selected. They did not have time to draw up a unified method as the meeting was in November and, for it to be adopted, they would have to have the document ready in the course of the week.

Angelo Fabieri said that he was in favour of eliminating benzene. Regarding the proposal made by Moreda, the methods needed to be unified, especially those using the same methodology. Standard ISO 17025 accredited part of a method.

He volunteered to conduct the revision of the method.

Mercedes Fernández asked who would conduct the modification of the method for its presentation to the Council in November 2017. She recalled that a group had been set up to streamline methods and that specific experts were in charge of the revision of each language.

The experts would wait for the document revised by Fabieri.

Maurizio Servili said that unifying the method had both advantages and disadvantages, and expediting the unification was not a good idea due to the errors that could be made.

Florence Lacoste said that the second document was the method on diglycerols (Project leader: Moreda), diglyceride and triglyceride compounds by gas chromatography (Project leader: Lanfranco Conte), to be considered for validation. Australia had proposed a method on phthalates and pesticide residues that was very similar to the French proposal. An electronic working group on organoleptic assessment had been created (Project leader: Miller).

The next meeting of the ISO group would be held on 19–20 September 2018 in Belfast.

Claudia Guillaume (Australia) proposed a method on pesticides similar to the French proposal, inviting the laboratories to participate in a collaborative test.

3. Results of collaborative tests and studies in 2017

3a. Simplified method for the determination of stigmastadienes

Angelo Fabieri presented the results of the collaborative test. He had not received the modifications indicated by Florence Lacoste. In that regard, he indicated that ultra pure solvents had been used, after verification, but proposed that the maximum limit should be set at 0.0001, since it would otherwise have no sense. He highlighted the importance of treating the silica by pre-heating it and adding water, which was part of the method and not as an option.

In answer to the intervention by Juan Ramón Izquierdo, Angelo Fabieri indicated that the subject of stigmastadienes was very delicate. The change was not a priority and the official method was long and costly, whereby preference should be given to using a shorter method. It could be a screening method, even for laboratories with high values, and should be adopted as a provisional method to allow laboratories to start using it and resolve the issue of the target. The methods should be compared and the laboratories should be allowed to gain more experience using them.

Florence Lacoste said that she had submitted a number of modifications on the text. There were no criteria with regard to the target. In the event of a spike in the reagents in the stigmastadiene region, it should be subtracted from its contents, or the laboratories should be required to use high purity reagents. The method was used as the substitution method for the current method, or as a screening method, so that, should any problems arise, they could resort to the original method.

Hermenegildo Cobo said that the scope of the method was not established and the IOC methods did not indicate the limits of their scope. The methods were being developed in research laboratories, in such a way that the limits established are not determined by the validations values. At the end, a collaborative test should be carried out to evaluate the functionality of the method at critical points.

The laboratories were not trained for that type of method and the results were accordingly not entirely adequate.

Wenceslao Moreda indicated that ultra pure solvents should be included. High purity hexane and even distilled hexane were used, conducting a purity test on these.

The method was of greater use for companies than for testing laboratories. The next collaborative test should set the scope of application more appropriately, for example by analysing samples close to the limit.

Juan Ramón Izquierdo said that the method gave CV values that were far higher than the values obtained with the official method. In the case of official testing laboratories, in a choice between a precision method and a fast method, the official and more precise method should be used. He made no objection to using it as a screening method.

Diego Luis García said that, in view of the precision values of the method, no assurance could be given that one method was more precise than another, whereby a comparison would be needed of the precision levels of both methods.

Mercedes Fernández said that a restricted working group should be created to develop the provisional method. It appeared that all the members of the group were in agreement to adopt it as a provisional method in November. Adoption by the group did not have to be done in person as the documents could be circulated electronically and, if no objections were raised, the method could be adopted.

Angelo Fabieri said that he considered it necessary to study the method in greater depth. They should check the precision values of the method with samples close to the limits established in the various samples, and through a collaborative test. Mercedes Fernandez proposed that Angelo Fabieri should coordinate the test with the collaboration of the IOC.

Angelo Fabieri indicated that he would be delighted to participate in and coordinate the test, but that it depended on his Ministry. He would however try to ensure that it was carried out before the next meeting.

Denis Olivier asked Angelo Fabieri whether he had sufficient samples to compare the samples with both methods.

3b. Quantification of erythrodiol and uvaol (TLC, HPLC)

The Executive Secretariat indicated that a note verbale had been sent in April to recalculate erythrodiol and uvaol contents with a document by Lanfranco Conte indicating the calculation formulas.

Lanfranco Conte said that, in the case of refined oils, there was a fall in sterol content, which distorted the calculation of erythrodiol and uvaol. This had been seen in the data provided by Spain, Turkey, Croatia and Argentina. In some cases the data had been sent in PDF format, while others appeared as a JPG image included in a Word document; some data had even been sent in paper format. It was important to know the number of samples that had been subject to statistical analysis, in the formats sent.

Lanfranco Conte therefore highlighted the importance of receiving the results in Excel and accordingly urged the Executive secretariat to request data in that format.

The method had been developed in collaboration with Wenceslao Moreda and the data obtained through high-performance liquid chromatography (HPLC) had been considered.

Wenceslao Moreda said that the name of the method for the determination of the composition and contents of alcohols by gas chromatography (GC) would have to be discussed. The method for obtaining fractions by HPLC and thin-layer liquid chromatography (TLC) had been unified. In case of doubt, the official method used TLC. The results obtained in the collaborative tests indicated that the HPLC and TLC methods were very similar and the values were very favourable. The conditions used did not necessarily have to be those indicated in the method, but rather those that would obtain the best results.

In relation to the suggestion made by Florence Lacoste, he indicated that the methods could be separated, for example into 2A and 2B. With answer to Lanfranco Conte, he indicated that it was included in the scope, whereby the title should be amended. It was decided that sterols, aliphatic alcohols, erythrodiol and uvaol should be added.

He indicated that he would conduct the modifications as indicated and would send the method to the Executive Secretariat for its circulation. He did not know how the experts wished to draw the difference between the two methods since the preparation of the unsaponifiable matter was exactly the same, while the separation of sterols (by TLC or HPLC) was different, having the analysis of sterols by HPLC in common in both methods.

Mercedes Fernández requested the experts to send all their comments before the circulation of the method and indicated that the IOC would distribute the final version.

Florence Lacoste indicated that in case of discrepancies, the TLC method should be adopted. The information should be included in the section on the scope of application. She furthermore indicated her personal preference for two separate methods, or in any event for separating the method into two parts.

Lanfranco Conte considered that the two methods could be separate but be contained within the same document. He however considered that the title was a bit ambiguous.

Juan Ramón Izquierdo was in agreement with L. Conte with regard to the general title and in the title of section 3, which had not been specified. The method using TLC should be preferred to HPLC because it was the method that had been used by laboratories over the last 30 years. All the bibliographic data had been obtained using the TLC method. It should however be indicated that, in case of doubt, the method using TLC should be given preference.

Kamel Ben-Amar said that, in the case of dispute, in so far as the determination concerned three parameters, even where the dispute only concerned one parameter, the TLC method should be used.

Hermenegildo Cobo said that in the event of dispute, it should figure in the standard and not in the method. Problems relating to discrepancies should be deleted from the method. He did not understand why one method should prevail over another one.

Angelo Fabieri said that there were alternatives for solvents. Some clarifications should be made in the text, indicating which was the reference method as part of the method document, as an annex.

Maurizio Servili clarified that the method would ultimately substitute the three current methods.

3c. Study on pesticides

Tiziana Generali said that the EU regulations were stricter than the CODEX regulation. Document 3(c) confirmed as much.

Mercedes Fernández recalled that those products were not included in the CODEX olive oil standard but rather in horizontal standards.

Juan Ramón Izquierdo said that the Commission should contact the Directorate-General for Health and Food Safety (DG-SANTE), which had a working group on the subject, and should be the group to liaise with CODEX.

Caroline Jeandin concurred with J.R. Izquierdo and indicated that she would take the necessary steps in that regard.

4. Other studies

4a. Method for the determination of methanol and ethanol in virgin olive oil

Wenceslao Moreda said that in the days prior to the meeting he had received the list of the possible laboratories for participation in the collaborative test. He undertook to carry out a collaborative test after the meeting; a reference sample would be sent to the laboratories to enable them to fine-tune the method before sending the samples of the collaborative study, in order to obtain reliable data.

Regarding Lanfranco Conte's observations, the correct wording would be "when the ethanol/methanol balance is reached". The method was based on the biodiesel method. In principle, it would not be necessary to indicate either temperature (T), or time. In the procedure it could however be indicated that the sample was heated to a T and the amount of time.

Angelo Fabieri indicated that an alternative method had been presented in previous meetings. It used an aqueous medium and a used column, given that the aqueous medium spoils the columns. He wondered whether that method should be included in Moreda's method or in a separate method; but as the equipment used was different, it would be more appropriate to leave them as different methods. In the collaborative test, that method could be included for information purposes and, if laboratories wanted to use it, that would be positive. The preparation of the sample was different to

Moreda's method. It should be indicated that the sample was heated to a T over a certain amount of time.

Lanfranco Conte considered that the beginning of the method by W. Moreda should be modified to indicate "until a balance is reached".

Tullia Gallina said that, in answer to Conte, it was uncertain whether a balance was possible, and a more appropriate wording should be found.

Maurizio Servili said that it was a question of balance, and how to find a stable equilibrium. He was in agreement with Angelo Fabieri's suggestion of a simple sentence indicating T and time.

Hermegildo Cobo said that the problem of an indication on balance was that the auditors could require proof that such balance had been achieved.

4b. Global method

The Executive Secretariat indicated that Wenceslao Moreda had revised the method and that no comment had been received. In the last meeting it had been decided that, should no comments be received, it would be sent to the Council of Members for its approval.

Wenceslao Moreda said that, following the last meeting, it had been decided to change the title of the method. The method was an extension of the method for the determination ECN42, since ECN44 and a triglyceride of ECN46 were calculated. The theoretical and the calculated triglyceride composition of an olive oil was very similar but varied greatly when seed oils were added.

The method was not included in the European Regulation, and its application did not appear in the IOC standard. The method was only mentioned and an oil could not be declassified as a result. Were the method to be eliminated, the presence of hazelnut oil in amounts of less than 35% could not be detected. For example, the Turkish hazelnut did not have the same composition as the common hazelnut. In the event of obtaining positive values, in other words, of detecting the presence of hazelnut, the possible consequences should be considered. The second centrifugation and the presence of pit oils and olive pomace oils were detected using the method. Since this method had been developed in 2003, no other method had been proposed for the detection of hazelnut. Synthetic lampantes contained second centrifugation oils, high-oleic de-esterised sunflower oil, and avocado and palm oil.

Regarding coherence, the theoretical composition of the triglycerides should be very similar to the calculated composition. In checking against another method, the ECN42 method could be included, but if the hazelnut oil content was lower than 35% it went undetected. When palm oil was added, it could be detected through its composition of myristic acid. Accordingly, the complete analysis of all the determinations should be continued. If they were all correct it would be impossible to indicate the cause of the incoherence.

Regarding the comment made by Claudia Guillaume concerning the IOC collaborative test, two samples had been analysed. One of the samples was a second centrifugation oil: 80% said no and 20% said yes. There was no method for the detection of second

centrifugation oils among synthetic lampante oils, only the global method. He insisted that said method did not have any legal implications. He continued working on the method and was collaborating with an expert in neural networks with the data on triglycerides and fatty acids. All the criticism he had received made him feel doubted and he did not have any desire to continue working on the method.

The coherence of the composition of the olive fruit was the basis of the ECN42. The method should not be used to declassify any oil. Accordingly, in the case of incoherence, the rest of the methods should be applied to verify the result. He requested the experts to make any comments in writing in order to be able to modify it.

Mercedes Fernández suggested that a restricted group should be created to draw up the comments to be submitted to the Council.

It was decided that the experts would meet after the meeting to discuss the point and reach a consensus.

Pierangela Rovellini said that coherence results should be checked against another method. The biosynthesis of olive oil was specific, so that when it was compared to the composition of the fatty acids of other types of vegetable oils, the results were different.

Tullia Gallina indicated that the study of the global method was part of the Oleum Project.

Claudia Guillaume insisted that consensus was rarely achieved on the analyses conducted in the IOC, either because the laboratories did not have sufficient experience or because the method did not work. It only worked in cases in which hazelnut had been added up to 8%.

Kamel Ben-Amar said that other methods should be used to verify, not confirm.

Angelo Fabieri said that the global method should be modified and improved. The problem of false positives had resulted in the decision to modify the method, but it was nevertheless a good method and would be maintained.

The method would be used as a screening method for the detection of extraneous oils. Accordingly, in the event of obtaining positive results, the other determinations should be conducted to verify the results.

Mercedes Fernández said that the second to last paragraph in Annex II referred to Table 3, which did not appear in the document.

Wenceslao Moreda indicated that he did not recall whether Table 3 referred to Project MEDEO or the results of an IOC collaborative test. He would look into it.

4c. Evolution of the analytical characteristics and best before date

The Executive Secretariat indicated that nothing had been received in that regard.

Tullia Gallina gave a presentation on the results of Oleum Project for best before dates and indicated her wiliness to coordinate the group. On the basis of past experience, she knew that a three-person coordination was not ideal.

Maurizio Servili said that some clarity was needed. Project Oleum sought a means of predicting the life span of oils suggesting that the IOC could develop a document referring to the concept. The term "expiry" should not be used in relation to olive oil, the idea of a "best before date" being more appropriate. The IOC document could provide large companies with instructions on indicating best before dates for oils.

One of the problems was the effect of light. That meant that if an olive oil was, for example, left out on a supermarket isle for three years, its quality would be affected. Other vegetable oils that did not contain chlorophyll would be less affected by exposure to light on a supermarket isle.

He considered that Tullia Gallina should be the group coordinator, given that she also coordinated the Oleum Project.

Diego Luís García said that he would include information in the document about the effect of light on oil and other factors that provided guidance for producers/distributors and he would separate the discussion on establishing a best before date. He recalled the need to create a document to incorporate the recommended conditions for transportation and storage of oils. He considered that the document should be created, incorporating the scientific studies that were underway and those that had been completed. He nevertheless considered that the three coordinators should be maintained.

Mercedes Fernández observed that the matter had been a recurrent subject of discussion. She insisted that someone needed to coordinate the group and that a document should be drawn up within a reasonable amount of time, incorporating the conclusions of studies as they were conducted.

The members of the working group would be: Tullia Gallina (coordinator), Lanfranco Conte, Anna Cane, Maurizio Servili, Raffaelle Sacchi, Claudia Guillaume, Styliani Iosifidou, Diego Luis García, Wenceslao Moreda, Nadia Maata, Kamel Ben Ammar, Fahri Yemiscioglu, Carlos Machado, Florence Lacoste and Ina Willenberg.

4d. Substitution of dangerous solvents

Denis Ollivier presented Document 4.d.1.

The IOC collaborative test had been applied for waxes, ethyl esters, stigmastadienes, monopalimitate and the difference of ECN42. It was validated with the reference solvent. The conclusions of the study were contained in the document.

Luciana di Giacinto asked why the statistical treatment had not been conducted with the official method. She would have conducted the statistical study with the 26 laboratories that had used both of the methods.

The outlier laboratories were eliminated, and the group agreed to conducting the test in that way.

Hermenegildo Cobo indicated that using the IOC examination to carry out tests to validate methods was not ideal, given that using an alternative solvent would simply mean repeating the analysis until positive results were obtained.

Mercedes Fernández explained that the IOC examination had been conducted as per the decision made in the last meeting, asking whether everyone agreed to introduce a note to include the alternative solvents, which would require the introduction of the data for the validation of the new solvents.

Wenceslao Moreda said that the accredited laboratories required validation with the new solvents.

Angelo Fabieri proposed to include in a note the precision results obtained with hexane seeking to achieve equivalent results.

Mercedes Fernández said that the document was being drawn up for distribution among the experts and for its approval at the next meeting.

Wenceslao Moreda said that when changing the solvents, the amounts were exactly the same as in the official method, given that the recovery tended to be different.

Denis Ollivier indicated that it had been conducted with the same amounts, the only variation being the boiling temperature for the elimination of the solvent.

4e. Update on recent studies on the relationship between volatile compounds and sensory defects

Two groups within the IOC group of experts on organoleptic assessment were currently studying the question.

Diego Luis García indicated that the Oleum Project had selected 180 samples, of which 60 had been evaluated by 6 tasting panels, using various analysis techniques. The objective was to fine-tune a method of analysis to obtain volatile compounds in virgin olive oil. The analysis of volatile compounds clearly identified lampante oils. Difficulties arose in respect of oils that fell between categories. The project was very interested in the oils that fell into that grey area, and it wanted to develop a method that provided a method of analysis that was quantifiable, standardised and easy to apply.

Juan Ramón Izquierdo asked whether the panels used in the Oleum Project were officially recognised or belonged to companies.

The study that was being conducted by the Ministry was based on official tasting panels. The focus lay on the oils that lay between two categories. The point of view of the official panels differed. For example, in the EU it was considered that there were two blurred zones given that there were three categories, while the IOC method established three areas of uncertainty given that it considered four categories. The study carried out by the Ministry was trying to develop a verification system: a tool to assist panels in verifying a category. For example, if an olive oil was described as extra virgin and was found to be otherwise, the inspection bodies were not concerned

with the category that it actually belonged to, but with the fact that it did not belong to the declared category.

Diego Luís García indicated that panels with different levels of certification had been used and their coordination was being observed. Some of the panels were official, while others belonged to universities. At the end of the study, they expected to reach the conclusion that if the degree of certification had an influence, it was important. Any differences between panels were due to the level of training, situation, etc.

Tullia Gallina presented the relationship between the reference samples and sensory aspects.

Lanfranco Conte confirmed Tullia Gallina's intervention and indicated that a conference would be held in San Remo at the end of September on the coordination between sensory analysis and instrumental methods.

Raffaelle Sacchi says that a project had been carried out in Naples over the last two years, looking at the borderline areas between virgin and extra virgin and between virgin and lampante. Some interesting results had been obtained and were being analysed, leading to the development of models for the fruity, bitter and pungency attributes. One of the areas of possible interference was the presence of phenolic substances. It had been observed that they presented differences in volatility.

4f. Determination of phenolic compounds: revision of IOC method

Pierangela Rovellini presented the method for the determination of glycols using HPLC, for the determination of tyrosol and hydroxytyrosol.

Wenceslao Moreda indicated that a number of collaborative tests had been carried out with acceptable results. Laboratories were not accustomed to the use of solid-phase extraction (SPE) for the separation of the phenolic fraction. With a view to the revision of the method, a workshop to test the three methods had been envisaged. Results had been obtained with the available means of extraction of the phenolic fraction, obviating the need to choose one of the two techniques.

A decision should be taken on whether to carry out the workshop and whether the creation of an official method for the determination of the phenolic compounds was a priority. The IOC method did not provide good data and he proposed obtaining data on the response values of the HPLC method, which was thought to be more accurate. The problem was the identification of spikes, the extraction with SPE, which was the reason for conducting the workshop, to obtain response factor values. The current IOC method and the method using HPLC gave very similar values. When the real values were obtained, response values could be obtained for all three methods.

Mercedes Fernández said that a workshop on the subject would be of great interest not only to the IOC but also to many countries. The invitation of the *Instituto de la Grasa* in Seville was still open, so the 2018 spring meeting could be held there, subject to approval by the IOC.

Tullia Gallina proposed coordination between the experts to reach a conclusion, with the task leader of the group on phenolic compounds of the Oleum project.

Wenceslao Moreda had sent all the information held by the IOC to the task leader.

Maurizio Servili recalled that data had been obtained using HPLC, whereby hydrolysis might or might not work.

Caroline Jeandin said that in February 2017 the European Commission had undertaken to get in touch with DG-SANTE. The representatives of the Directorate General indicated that they had not presented any objections at the time of the approval of the Regulation. Regarding the harmonisation of the method, the Directorate General indicated that the Members were responsible for the adoption of an appropriate method, and it would be very positive if harmonisation was achieved at a European level.

Diego Luís García indicated that there was some doubt as to the parameters to be tested.

Angelo Faberi explained that the subject was getting complicated. The solution was however simpler since the IOC developed methods.

5. Varietal studies

5a. Review of the analytical characteristics of virgin olive oils displaying anomalous parameters

Mercedes Fernández indicated that two decision trees had been developed in 2013, for delta-7-stigmastenol and campesterol. The decision tree for campesterol had been presented to CODEX and approved.

Turkey had requested the decision tree for delta-7-stigmastenol in lampante oil and refined olive oil. Palestine requested the decision tree for lampante oil.

Wenceslao Moreda indicated that no decision tree existed for delta-7-stigmastenol in lampante oil or refined oil, where levels would be high. The lampante and refined oils from Syria could not be refined or added to an olive oil due to their high delta-7 levels. The matter should therefore be addressed.

Lanfranco Conte agreed with W. Moreda, since lampante could be used to obtain refined olive oil. The Italian standard contained the decision trees for lampante and refined olive oil to protect the Italian companies that used those types of oils.

Mercedes Fernández said that Turkey had sent samples and that various studies had been conducted. Accordingly, although decision trees were previously only presented for edible virgin oils, Syria and Tunisia had expressly requested a decision tree for olive pomace oils. In that light, the subject needed only be resumed.

Juan Ramón Izquierdo recalled that, according to the established protocol, the country should send information for three crop years. That was appropriate for lampante, but refined oil was obtained using refineries and was therefore an industrial product. He was not opposed to studying it, but it should be done through the appropriate channels and taking into consideration the volume of those oils that did not meet those parameters.

Lanfranco Conte said that the data he had for Syria and Tunisia was quite old. He did not remember the three crop year requirement. The problem raised by the anomaly in lampantes was that they needed to be sold upon leaving the olive mill. Such anomalies should only concern oils sold in bulk, not bottled oils. Studies should be resumed with the data available. The issue of quantities was a different matter and should a country have a problem, it should be addressed.

Mercedes Fernández said that the volume of samples was taken into consideration and three crop years were studied. Three decision trees were currently being studied for lampante oil, not for refined oil.

Nadia Maata said that some samples had been analysed containing 23% of anomalies. A request had been sent to the IOC without having received any feedback.

Mercedes Fernández confirmed that the samples had reached the IOC but indicated that no request had been received. The anomalous parameter in that type of oil was linoleic acid.

Diego L. García said that the anomalies were exceptions and should be treated with caution. The scale of the problem should be studied, not only in Turkey but in all countries. Adulteration also had to be taken into consideration, given that the standard was intended to protect oil from that type of problem.

Fahri Yemiscioglu said that climate change was creating variations in the composition of food products. The phenomenon had been identified two years previously in Syria, but it was not happening in Turkey. It was a persistent problem, which did not depend on variety, and the countries affected had sent a lot of data in that regard.

Maurizio Servili said that, apart from the environmental factors, some varietal factors did come into play. New producer countries encountered problems with certain varieties, calling for an adequate study. It should check whether the anomalies appeared over various crop years. For example, the anomalies identified in Argentina were tracked over various crop years.

Mercedes Fernández recalled that the proposal was for delta-7 and two decision trees already existed (for edible virgin oils and pomace oils), and that a request already existed to extend it to lampantes. The Codex only covered edible oils, whereby the subject of lampante virgin oil had not been addressed in its day.

Juan Ramón Izquierdo indicated that when the delta-7 study was carried out the quality of the oil was unknown, which was why it was now proposed to extend it to the other virgin oils.

Luciana di Giacinto indicated that the decision trees concerned bulk oils, which was not specified in the request.

Wenceslao Moreda recalled that the Syrian oils that had been analysed, were virgin oils but that they were, organoleptically-speaking, lampantes. Consideration should be given to refined oils, given that oil from a lampante with the problem would result in a refined oil with the same problem.

Mercedes Fernández asked again whether the proposal was to apply the decision trees to all the virgin oils. A study had not been conducted for refined oil, whereby the subject would have to be addressed.

Lanfranco Conte said that the same decision tree could not be applied to extra virgin oil since it included the contents of stigmastadienes, and in lampantes the upper limit of that parameter was 0.05 ppm.

Mercedes Fernández said that the documents would be distributed and the restricted group would submit a new proposal.

5b. Other anomalies not covered by decision trees

Nadia Maata said that in the previous point she had indicated the problem relating to linoleic acid in Morocco and proposed studying a decision tree.

Mercedes Fernández recalled that the parameter had already been the subject of two studies that the CCFO had requested the IOC to conduct and that CODEX had debated the parameter for years at the request of Australia. If the group considered it appropriate to propose it to the Council again, it could.

Juan Ramón Izquierdo said that there was no objective reason not to study the problem. The subject had been pending for a long time. The IOC had a working group that studied varieties.

Mercedes Fernández recalled that the problem had appeared before delta-7. Morocco had sent samples that needed to be analysed in order to comply with the procedure for anomalous parameters. It was unclear whether the restricted group wanted to take the subject up again and send the samples for analysis. For that reason it was being proposed again now.

Luciana Di Giacinto did not understand why Morocco had not applied the IOC's protocol on anomalies.

Juan Ramón Izquierdo insisted that Morocco was a very important producer country. The previous study had been conducted ten years earlier and situation could have changed as a result of climate change. The group should therefore reopen the study.

6. Miscellaneous information

6a. Presentation of the H2020 Oleum Project

Tullia Gallina presented the information included in Doc 6(a).

Angeli Faberi indicated his surprise at the request for samples received from Sweden, enquiring as to the type of sample that was requested.

Tullia Gallina indicated that commercial samples and legal blends had been sent. The feasibility of analysing them was being considered.

Caroline Jeandin indicated that a questionnaire (No. 2) had been sent out but not many replies had been received.

Tullia Gallina observed how difficult it was to receive answers to questionnaires. They would however just send the questionnaire out again.

Lanfranco Conte explained that the questionnaire was online. It would however be sent again. The questionnaire contained open questions, requiring full answers.

6b. Results of eWG for the revision of COI/T.20/Doc. no 42-2/Rev.1. - Precision data of the methods (Dr Di Giacinto)

Mercedes Fernández recalled that Luciana Di Giacinto had been appointed as the group coordinator at the meeting before last.

Luciana di Giacinto indicated that Florence Lacoste had done all the work, not the group. She had taken the document presented by Luciana di Giacinto and worked on it directly as the coordinator had not been in touch with the members of the group. The tables of the provisional methods were missing, as well as the table of the method for phenolic compounds, which was not provisional. Florence Lacoste had incorporated all the precision tables, applying an ISO document to conduct the calculation. The precision values for the provisional methods, which were frequently used, should be added in document 42.

Kamel Ben-Amar said that he considered, as Florence Lacoste would have pointed out had she been there, that the precision values of the provisional methods should be included.

The corresponding modifications were included in each of the methods.

Denis Ollivier transmitted a message from Florence Lacoste, who was not able to attend the second day of discussions, indicating that the organisers of collaborative tests should send the data in an Excel table to the IOC, to be able to use them subsequently. Florence Lacoste had had to add each of the results by hand.

Luciana Di Giacinto said that the IOC trade standard had to be taken into consideration in relation to the precision values. Modifying Doc No 42 and the significant figures contained therein, would require the modification of the IOC trade standard.

Hermenegildo Cobo proposed changing the title of the two methods, on fatty acids (Method Doc. No 33), and on acidity (Method Doc. No 34).

Kamel Ben-Amar indicated that there were two methods for the analysis of waxes: the method for waxes and the method for waxes, fatty acids methyl esters and fatty acids ethyl esters. The significant figures were indicated in one method but absent in the other, whereby it was proposed to eliminate the decimals for waxes in both methods.

Luciana di Giacinto explained that Regulation No 2568 already provided the removal of the decimal figures for waxes.

Hermenegildo Cobo explained that it was necessary to ensure coherence between the method and the standard. Furthermore, the method for peroxides provided that when the peroxide content was above 10, the decimal was not included.

Mercedes Fernández thanked the experts for their collaboration and asked them to indicate the documents that would be presented to the meeting of the Council.

- Luciana di Giacinto would revise Doc. No 42
- Kamel Ben-Amar would also revise the methods, since Doc. No 42 was not enough.

6c. Determination of the composition of triacylglycerols and composition and content of diacylglycerols by capillary gas chromatography

Mercedes Fernández explained that when the ISO method was adopted, it would not be included in the standard, which was provisional, but it would be posted online.

6d. Presentation of a revised version of method IOC / T20 / Doc. No. 28 for discussion

Hermenegildo Cobo said that he had introduced the 60 ml of hexane and proposed to eliminate the word stigmastadiene.

Wenceslao Moreda said that unification would be ideal and the elution with 60ml of hexane helped to avoid problems of co-elution.

Angelo Faberi said that merging the methods would change the standard and it would be necessary to update it with new methods. A new method should be validated, regardless of whether it was given a new number of assigned an old number.

Luciana di Giacinto said that the alternative instruments should be included.

Mercedes Fernández concluded that all the experts appeared to be in agreement to merge the two methods and introduce 60 ml of hexane. A group made up by Luciana Di Giacinto (modification of Doc. No 42), Denis Ollivier (solvents), Hermenegildo Cobo (Doc. No 28), Angelo Faberi, Kamel Ben Ammar and Florence Lacoste (to revise the rest of the methods) was created. The group would be given until 6 October 2017 to finish its work, with a view to presenting the outcome at the session of the Council.

6e. Improved method for TAG analysis in olive oils: a comparison of the current method (DeltaECN42) and "UHPLC + CAD"

Lanfranco Conte indicated that the subject was already closed and should not be included in the agenda.

6f. Assistance and training for recognised laboratories or laboratories applying for IOC recognition

Mercedes Fernández asked the experts to indicate whether they were interested in being part of the group and, if so, preferably not take part in the IOC proficiency test.

Luciana Di Giacinto, Wenceslao Moreda, Carlo Mariani, Claudia Guillaume and Kamel Ben-Ammar put themselves forward.

6g. Determination of MOSH – MOAH

Wenceslao Moreda indicated that, with regard to the determination of saturated and aromatic hydrocarbons, pomace oils contained a certain amount of those hydrocarbons due to the means of extraction of the olive pomace. It resulted from the contamination of sunflower oil from Ukraine. The study had been organised by the Joint Research Centre (through DG-SANTE). The EU was setting up a task force in that regard. There were three dates for the submission of data: February 2018, February 2019 and February 2020. It would consider olive-pomace oils.

The methodology was based on the online method and there were plans to incorporate the off-line method. The quantification was conducted for C20 to C40, but as it was going to be developed for all kinds of food and containers (paper with ink), instead of asking for the total contents, it would be conducted for C10-C13, C20-C40 and C40-C50. The hydrocarbons group gave an indication of the container in question due to migrations from the container to the oil, which would be the data considered by the working group. The predicted figures were around 2mg/kg , highlighting the need for data.

The only expert in the EU working group with a link to olive oil was Wenceslao Moreda. The other members came from the packing sector. Furthermore, there was a limited amount of time, and decisions could be taken that would affect the olive oil sector.

The only limit was for sunflower oil from Ukraine, at 50mg/kg.

Lanfranco Conte confirmed W. Moreda's indications. An association of sweet products existed in Italy. It was studying lipophilic contaminants, which had important implications for oil.

Mercedes Fernandez indicated that the Executive Secretariat had written to the group coordinator, who had confirmed that that the information would be transmitted to the IOC.

6h. Determination of 3MCPD

Wenceslao Moreda explained that the palm oil lobby was slowing down progress on the subject of 3MCPD, a compound that appeared in refined oils. The chlorine could come from the water, the bleaching earth activated with hydrochloric acid, salt contents, and the chlorines in the pesticides used. The established maximum limit was 0.8 mg/kg per day. The limit of 3MCPD in vegetable oils was established at 2 mg/kg, but nothing had been indicated for 2MCPD. The olive pomace sector was unaware of the contents of the compound. Its determination was very complicated: there were three methods for the preparation of the sample, including an ISO method.

The compounds were toxic when freed, even though they could be esterified with fatty acids. Information would be needed of those contents. 3MCPD and 2MCPD were carcinogenic. Glycidol was carcinogenic and genotoxic (damaging DNA). Refined and olive-pomace oils could accordingly be qualified as carcinogenic. The subject had received a lot of traction in the palm oil sector. It could create problems in relation to the use of extra virgin oil for frying. In general, it would suggest that no vegetable oil should be used for frying.

Pierangela Rovellini reported that ISO was conducting a collaborative test on frying oil.

Fahri Yemiscioglu said that there was no limit for those compounds. The European Food Safety Authority recommended reducing daily intake from 2 ppb to 0.8 ppb. The palm oil used in Nutella was carcinogenic. A hundred samples of different types of vegetable oils had been analysed. Among those samples, palm oil had the highest content of those compounds as it had to be used in a refined form. In the case of virgin olive oils, the environmental chlorine could be eliminated. They group on contaminants should begin to study the issue.

Wenceslao Moreda said that the study had made further progress with respect to palm oil. Chlorine was found in the fruit because it was transmitted from the soil. Chemical refining produced lower levels of those products, as it applied lower temperatures (two processes at 150º C), while physical refining used a single process at 274ºC.

7. Other business

Jaime Lillo reported on the protocol for arbitration drawn up by the IOC, the Executive Secretariat was not legally competent in matters of arbitration that ran counter to the regulations in the countries involved. The subject would be presented in the group of experts on organoleptic assessment.

Hermenegildo Cobo said that in the EU, the Member State in question appointed an authority to monitor imported and exported oils and those on the domestic market. No external body could intervene alongside the competent authorities in each country. The document was accordingly full of exceptions. Exporters did not intervene in the importation of a product to a country.

Caroline Jeandin observed that the restricted meeting of the previous day had discussed not including the system in the standard, given that it could be the subject of litigation in the case of discrepancies between the oils.

Hermenegildo Cobo indicated that matter was mention in Regulation No 2568 but not in the method.

Jaime Lillo said that the objective was to resolve a problem that arose international trade. The restricted group should continue working for its presentation in the group of experts on organoleptic assessment.

In bringing the meeting to a close, the Executive Secretariat thanked the participants for their collaboration and invited them to the next meeting, which would be held in

the same week as the seminar for the study of the methods of analysis for the determination of phenolic compounds at the headquarters of the *Instituto de la Grasa* in Seville (Spain) from 12 to 16 March 2018.

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IOC

MEETING OF CHEMISTS ON METHODS OF ANALYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

15 – 16 March 2018

Minutes taken by Claudia Guillaume

1- Adoption of the report of the meeting of 25 – 26 September 2017 and of the draft agenda

2- Institutional update

a. 106th session of the council of members
b. Documents adopted and status of the documents pending adoption. Documents to be presented in the 107th session.

As per last meeting, the global method is still pending, as per Tunisia experts the last phrase in the method should be removed (which states that if the result is not conclusive should be confirm with other method), if not, there is no point to have this method, if should be confirmed with other method. He mentioned that a method should give results, not interpretation of results. The interpretation should be in the norm, not in the method.

Ariel Buedo mentioned that does not agree to put in the std because it has been shown that some oils could have anomalous results and is genuine.

Moreda mentioned that the problem is when the oil pass all the tests except the global method, when an oil already fails other parameters that is covered in the std, that's it.

EU representative mentioned that she is happy with the method and should be included in the body of norm. she proposes to remove the last phrase as per Tunisia and make sure that the scope of the method is clear. This method indicates if the olive oil TAG composition is coherent or non-coherent with the theoretical TAG composition. It shall only serve as a screening method to detect suspicions of bad or fraudulent practices that would require to be further investigated.

Mercedes confirmed that this is an official method, but without juridical property, so no oils could be disqualified from the grade because of this method. This method is only for screening. The name, the scope and the calculation were changed but the method still is official. Argentina and Tunisia are not in agreement.

CG comments: this could affect Australian oils, mainly from warm weather, where linolenic and linoleic acid content is higher, giving a higher ECN42.

c. eWG on the revision of codex standard for olive oil and olive pomace oils (CODEX STAN 33-1981)

Juan Ramon Izquierdo presented an update of how this discussion is going. He mentioned the 'friction' between countries. Refined olive oil to be sold as such, fatty acid profile (mainly linolenic and decision tree), definitions of the grades and panel test.
He said very clear as personal opinion that 'each country' only should focus in the fundamental parameters and have more flexibility in the points that are not fundamentals.
In Oct 2018 should be ready the final document with the agreements and disagreement, to be approved in Feb2019.
Mercedes mentioned that IOC has given green light to Morocco to start a study about linolenic and a potential decision tree. Previously this work was done electronically, Morocco has sent all the

samples to all the labs from the IOC members and no response was received. Mercedes mentioned that again and what the group wants to do.
Dr Cristopolous (Greece member) has sent to IOC a document with some data and a proposal decision tree.
The Morocco representative (Nadia Matta) mention that from 365 samples 77 have problems, they have tested all the parameters but only have sent the fatty acid profile but could send all the other parameters.
Mauricio Servilli from Italy, mentioned that have been discussed before when Australia implemented the std and decision tree was created, so he recommends reviewing that data and include the data from Morocco and see if applicable. Also mentioned that previously Codex didn't accept a decision tree and wanted to change the absolute value of linolenic. So, if that hasn't change, the discussion is different.
Mercedes reaffirm that is necessary to bring together the group again and discuss. In 1998 was when the Codex removed the value of linolenic because was not agreement and since then is like that.
Mercedes wants to confirm that the idea is of a decision tree instead of changing the absolute value.
Izquierdo confirms that Codex wants the decision tree, that already has accepted this with campesterol.
Representative from Tunisia (Camel Benamar), he asked to Juan Ramon Izquierdo if the E+U expression result could be included in the eWG.
Izquierdo, highlight that the function of the eWG is to be harmonized the Codex with all the countries, not only with IOC. As in the case of E+U, all the national norms are consistent, so will not be part of this working group to attend this issue. Mercedes mentioned that IOC will present to the group this when ready.

d. IOC/Codex collaboration
e. New proposal by the IOC to the WCO for the revision of the explanatory notes for the heading 15.09 and 1.10

This was a proposal that IOC has done to the customs authorities. It was designed to make sure that the people that is checking the oil while entering in the country be sure what they are checking. The first proposal (2.e.1) that IOC did 2 years ago was rejected because it was so complicated and the 'normal' people not very familiar with the industry were not able to understand. So, the IOC has done another proposal (2.e.2) which was directed by Angelo Fabieri and the new proposal was sent to all the member and he has received the data, will process and send the new proposal.
Juan Ramon Izquierdo suggested to present the document to customs from different countries to see what they think, because they will be the ones using this document.
Maria del Mar from Customs laboratory mentioned that the last proposal is too vague that went from a very complicated to a one with only FFA in the document. She mentioned that customs don't do FFA either, that need to be sent to a lab to confirm if the oil is ok or not, so she doesn't see the need to do it so simple.
Juan Ramon Izquierdo suggest giving 15 -20 days to make comments and find out with customs instead of making a decision today. Mercedes has agreed and was decided that the document will be open until end of March for comments.

3. Results of collaborative tests and studies scheduled in 2018

a. Simplified method for the determination of stigmastadienes

Faberi has commented about the new method which used less solvent than the actual method. In 2015 a collaborative study done hasn't show a good performance, last year another collaborative study with some adaption of the method. This study works better, the results have been better but

still the r and R were not good enough to validate the method. Other changes have been made and another collaborative study will be done to be finished by October 2018. Modern Olives will participate in this study.

b. Quantification of E+U – TLC, HPLC (HPLC method – Dr Moreda, GC method – Dr Conte).

Mercedes remind to all the countries to send information about E+U. Dr Moreda remind that the method is the same, only pretend to change the expression of the result in mg/kg instead of % as is actually. He also mentioned about sending data from results of E+U in all different grades (VOO, EVOO, ROO, OO and pomace oil). Dr Faberi mentioned that there is also a modification of the method about reporting only E instead of the sum of both (E+U).

Moreda made the comments about the separation of the phase by HPLC instead of TLC. The collaborative study was done by IOC last year and the results were acceptable. So, could be proposed both option to separate the phases, then the quantification will be done by GC anyway. Dr Lacoste and Dr Cobos mentioned that is very important to highlight that TLC/GC will be the official method. Dr Moreda will put the method all together and will send to the group.

c. Study on MRLs of pesticides (Dr Generali).

Dr Generali explained about the collaborative study, where 34 laboratories have participated, 24 have used QuEChERS. 23 labs were out of 34 analysed all compounds corresponding at 68% of participants. 3 labs have not achieved the sufficient scope, none z-score > 5 were obtained. 2 false negatives and no false positives.

Dr Teresa Perez Millan from Interprofesional Aceite de Oliva de Espana (3.c.2), has done a presentation about MRLs where they analysed the world situation about phytosanitary in olive oil. The studiy was in 2 phases: 1) Review of MRL for world olive oil situation and 2) Identification of the potential problems for the olive oil export regards phytosanitary products. Australia is within the countries studied.

www.oliveoilfromspain.org / www aceitedeolivadeespana.com / info@interprofesionalaceitedeoliva.com

Dr Generali continues saying that the EU is updating the list of MRL permitted (3.c.1).

Mercedes suggests that Australia could direct to the right person to be able to access to the data of pesticides in our country. Will discuss with Dr Generali later to know where this has been sent.

It was mentioned about the need in method harmonization.

4. Ongoing studies

a. Method for the determination of methanol and ethanol in VOO (Drs Moreda and Faberi).

Moreda state that the method is in place and would like to do collaborative study. Juan Ramon Izquierda asked that the definitive method be circulated between the group.

It has been approved to do a collaborative study, Modern Olives will participate. One sample will be sent to work and set up the method before June. After that blind samples will be sent in Sept, to have the data ready by next IOC meeting.

b. Update on analytical characteristic and the BBD (COI T.15/NC N.3, section 10.1.7.1)

Dr Tullia Gallina has presented the changes collected (4.b.3) and asked for consensus. It was debated a lot between which were the parameter that have more impact in the storage of the oil: light, temperature, inert gas, etc. The temperature was confirmed that the range of storage would be 13-25 C. Dr Gallina will review and collect all the comments and modification presented and circulate a new version by end March.

c. Update on recent studies on the relationship between volatile compound and sensory defects (Prof Conte and Dr Izquierdo).

Dr Izquierdo made the comment that still working in a software and some statistical formulas to get a correlation between sensory defects and volatile compounds.

d. Determination of phenolic compounds: revision of IOC method. Conclusions of the workshop held at the Fat and Oil Institute, Seville.

In this workshop was analysed phenols by HPLC-DAD, Modified Folin and phenols by hydrolysis. The conclusion was that the method should not be around the health claim, it should be vice versa, also being concluded that the expression of results could be changed from mg/kg to mmols, to avoid the transformation of complex phenols to simple phenols.

The SPE extraction is easier and simpler than liquid-liquid, the 3 wavelengths are needed to improve the identifications of the peaks, also was discussed the need to find a simpler method to identify total phenols for the companies that want to do health claims. The phenol by hydrolysis method seems to be reproducible and could be used for that. Still needs to find a conversion factor to the HPLC method.

Moreda proposed to re-write the HPLC method with more information about chromatograms, data, retention time, etc to make sure that everyone understands how to be carried out the method properly.

5. Varietal studies

a. Application of decision trees of D7 stigmasterol in lampante VOO

(5) Comments on Turkish data from Efi Christopoulou. It was no comments about the decisional tree, so it will be passed to the chemical meeting on 2nd Oct 2018.

Dr Nadia Matta (from Morocco), Morocoo in the south area have a lot of problem with this compound being higher than IOC limit, mainly in the variety Picholine Marroqui. Lately also has been seen that in the north of the country also present some anomaly in this parameter.

In the south represents the 40% of the production from this area, it is very important to highlight hat all the samples testes were EVOO.

They have found value up to 1.3%. by consequence, the decisional tree proposed by Greece was not be enough and adequate for Morocco.

b. Application of decision tree to linolenic acid

(5.f) from Efi Christopoulou

Mercedes requests samples from Australia to include in the collaborative study. I said that there is no problem, as soon as I received the petition from IOC.

6. Miscellaneous information

a. Presentation of the H2020 OLEUM research project (Dr Tullia Gallina Toschi)

A set of 180 samples of EVOO, VOO and lampante were collected by 6 sensory panels and exchanges among all the involved partners for their sensory and volatiles. A huge difference was found and was really hard to identify all the defects detected by the sensory. So only defect like fusty and winey were considered for this study. Reference std has been done and sent to the labs for determination of volatiles to see if any coherence is found. Diego Garcia is the head of this project and by end of the year should be some data of this research.

b. Assistance and training for recognised laboratories or laboratories applying for IOC recognition

c. Determination of MOSH-MOAH (Dr Moreda)

The oils more affected will be the pomace olive oil. The EU is asking for data to give an opinion in 2019. Dr Moreda mentioned that is super important that producer countries presented data to avoid ridiculous limits. Now, EU is thinking in MOSH less than 2ppm and MOAH less than 1ppm, which could be a problem because there is not method at the moment to be reproducible at those levels.

Dr Servilli commented that Germany is the country putting more problems. Italy is having problem to export to Germany not only for pomace olive oil but also for EVOO.

The main thing to discuss also means to study the daily intake that could be dangerous, because this is not studied yet.

Dr Lacoste mentioned that her lab has tested many EVOO and they have found presence of 10-12 ppm of MOSH and MOAH. She considered that could be a contamination from pesticides, petrochemical from motors or any other transport associated to the process, also could be through the processing aid like talc.

Dr Lacoste have done a study about the method and the limit of detection of 10 ppm. To decrease this limit, is needed to do another isolation step to purify more the sample portion. A new study will be done.

Dr Izquierdo suggested that the group of contaminants should oversee this issue.

d. Determination of 3MCPD (Dr Lacoste)

There is a new regulation just published which stated some limits for those esters of glycidols.

Those esters are formed in the deodorisation of the oil, so those limits are only for refined olive oil and pomace olive oil not applicable for virgin olive oils.

7. Other business.

a. ISO meeting in Belfast (Dr Lacoste)

Gretel Bescovish is the person now in charge of ISO.

pending works:

-revision of FFA (remove the phenolphthalein because of its toxicity and replace the indicator)

-revision of FAP (to include in the method the short chain FA for detection of animal fat)

-method for phthalates (will start the interlab for method validation this year).

- Mario Solinas award, is working with reference materials to train the sensory judging.

- looking for a project leader for the project of added antioxidants

- review of the aliphatic alcohols

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NSW GOVERNMENT | Department of Primary Industries

Minutes from notes taken by Jamie Ayton

International Olive Councils' Chemists Meeting on methods of analysis for olive oils and olive pomace oils.
4-5th October 2018, Madrid, Spain

1. **Adoption of the report from 15-16th March 2018 meeting and draft agenda.**

Accepted

2. **Institutional update**

- The IOC is continuing to work with other organisations to harmonise standards.

- At the 107th session of the Council of members of the IOC:
  - The merger of the sterols and alcoholic compounds was accepted.
  - those labs requesting type B recognition will be required to also obtain type A recognition first.
  - IOC Document no 14, the training for organoleptic panels, has been submitted for revision.
  - The organoleptic evaluation method has been reviewed and will be submitted for approval.
  - Document no.18 – determination of wax content will be removed from the website. The web page is being rebuilt, all new documents will be on the new website.

- The electronic working group (EWG) on the revision of the Codex Standard for Olive Oils and Olive Pomace Oils (CODEX STAN 33-1981) is progressing well. There is agreement on some of the standards and others are not agreed upon. There will be a meeting in February 2019 between the EWG representatives, IOC and CODEX to discuss outcomes. The outcomes from the working group include:
  - Agreement that the organoleptic characteristics of olive oils are important for the trade classification of the oil.
  - Majority agreement to remove ordinary virgin classification
  - No consensus on the limits for some of the fatty acids
  - No agreement on linolenic acid. Some countries requested the limit to be increased to 1.5%. IOC to decide if decision tree could be used. If no agreement can be reached, national limits will remain.
  - No consensus on inclusion of 2-glycerolmonopalmitate. Committee will continue to discuss in the hope that a consensus can be found.
  - Fatty acid ethyl esters has been proposed as an official test. In order to reach a consensus it is proposed to move FAEE to the appendix of the trade standard. This was not accepted by the committee.

- No consensus on including DAGs in the trade standard. Opportunity for those countries proposing the inclusion of the method to send in further data to try to get the decision changed.

- IOC is keen to increase collaboration with Codex.
- Amendments have been made to nomenclature for Chapter 15 of the World Customs Organisation document (specifically Headings 15.09 and 15.10 – Olive oils and olive pomace oils), which have been submitted for approval.

3. Results of collaborative tests and studies scheduled in 2018.

- Simplified method for the determination of stigmastadienes
    - The modified method will use 3g of silica instead of 15g.
    - There is a systematic tendency to obtain higher results using the simplified procedure. The difference between the regular method and simplified method was about 0.34 mg/kg on a sample with a result of approximately 2 mg/kg. Reproducibility and repeatability is significantly different between the methods.
    - There was general agreement that more representative samples need to be sent out for ring tests e.g. something near the limit rather than very high results
    - An email will be sent to participants to confirm their participation and then new samples will be sent out. Previously there have been 26 labs which asked to participate, with 19 labs providing results.

- Quantification of Erythrodiol and Uvaol
    - Calculations are based on sterols at the moment (e.g. diols/total sterols). The problem has been that when oil is refined, sterols are removed, therefore giving a lower denominator. Hence, there is a false high result for diols in refined oils.
    - The new method will use 5-α-cholestanol as the internal standard.
    - 22 labs were invited to do a ring test. Repeatability and reproducibility have been calculated from those results.
    - The IOC has asked all countries to send in data, or limits may be set and oil from some countries may not meet the limits.
    - The IOC is very keen to get as much information regarding erythrodiols as they can get. They need information on EVOO, pomace and refined oils.
    - Need to use erythrodiol and not Uvaol in calculations. The limit will be changed to mg/kg (from the current % calculation). Professor Conte will send out reminders for labs to send data for this.
    - Figure 4 in the method will be removed and replaced with a true lampante oil chromatogram.

- MRLs of pesticides in olive oil
    - Not much discussion. IOC needs as much information on pesticides in olive oil as possible
    - Australia to provide data on pesticide residues in olive oil – **Modern Olives**?

4. Ongoing studies

- Method for the determination of methanol and ethanol in virgin olive oils

- Dr Moreda and Dr Faberi confirmed that the participants list for ring test is complete and labs still want to take part. Samples were distributed at meeting (**including a sample for Modern Olives**).
- Dr Moreda and Dr Faberi require that chromatograms be sent as well as data.
-

- Update on analytical characteristics and the best before date (COI T.15/NC N.3, section 10.1.7.1).
    - There is agreement within the IOC on the good practices guide and storage instructions (10.1.7.2), however there was some comment that it could be more detailed. There is no agreement on best before date.
    - Lengthy discussions about producers/packers being blamed for issues if oils do not meet limits. Consensus that there is a need to engage distributors and retailers to encourage them to improve practices so that oil meets the trade standard.
    - Discussions regarding storage during transportation. Needs to be good conservation of the sample along the supply chain. Temperature and light have significant impact.
    - IOC secretariat will make a proposal at a forthcoming meeting regarding this point.

- Update on recent studies on the relationship between volatile compounds and sensory defects.
    - This work is continuing in Spain and Italy.
    - Lanfranco Conte is working on compounds looking for associations between sensory and volatile compounds.

- Determination of phenolic compounds: revision of IOC method.
    - Colourmetric method for is complicated. Hydrolysis method is less expensive/time consuming.
    - Dr. Moreda is coordinating laboratories to participate in ring test. **NSW DPI agreed to participate and received a sample at the meeting**.
    - Current method on website – consensus at meeting that it needs to be revised. Lengthy discussion about which is the best method to replace the current method. No consensus between Spanish and Italians.
    - Proposal is to use either of two methods; (a) isolating the phenolic fraction or (b) direct hydrolysis.

- Absolute value of delta K
    - The absolute value of delta K will be removed from the method.
    - Fresh oils can sometimes be -0.02, so when using the absolute value, they fall outside the limits.
    - Proposal – to remove the absolute value and include a third decimal place for delta K limit. After lengthy discussion, the final proposal is to remain at 2 decimal places until consensus has been reached, then possibly change to 3 decimal places.

5. Varietal studies

- Application of decision trees to delta-7- stigmasterol for lampante olive oils
    - Proposed by Turkey and Palestine.
- Application of decision tree to linolenic acid
    - Proposed by Turkey and Morocco.

- The IOC requested more information from the countries concerned before any decisions are made. They will discuss again at the next meeting.

6. Miscellaneous information

- Presentation of the OLEUM project
    - Consortium includes EU, China, US and Argentina
    - Objectives include development of new methods for quality and authenticity of olive oils; the development an integrated quality assurance infrastructure for methods of analysis and the development of a technical support network as a worldwide network of analytical laboratories involved in the analysis.
    - To determine the presence of refined oils, including soft-deodorised oils in olive oil, samples are being analysed to test the sensitivity of the methods. Analysis includes DAGs, PPPs, FAEE by HPLC, FAEE by TDR, volatile compounds by SPME-GC-MS and volatile compounds by GC-IMS.
    - To determine blends between olive oils and other vegetable oils, samples are being analysed for TAGs using HPLC-IR, TAGs by HESI-UHMRS, FAMES, sterols, tocopherols, volatile compounds by SPME-GC-MS and bioactive compounds.
    - New in-house method for sterols has been developed. The method consists of isolation using SPE (1g of silica gel) of the previously silanised oil, followed by separation using GC-FID.
    - New method for the quantification of volatile compounds has been proposed. 180 samples have been analysed using SPME. Eighteen compounds have been identified as important for the relationship between volatile compounds and sensory defects. Prediction is that up to 60% of oils could be correctly classified using this method, so these oils would not need to go on to a sensory panel test. Information will be made available on a cloud so that information can be uploaded and classifications of oil can be made.
    - Further information available at http://www.oleumproject.eu

- Assistance and training for recognised laboratories or laboratories applying for IOC recognition.
    - Lengthy discussions regarding auditing of labs applying for IOC accreditation. General consensus was that there is significant confusion about what this project will achieve. Auditing costs will be substantial.
    - There will be “surprise” audits of accredited laboratories to ensure that each lab meets the requirements of the IOC.
    - There will definitely be some changes to the accreditation process. Much more discussion to take place prior to the implementation of the process.

- Determination of MOSH and MOAH
    - Discussion regarding the best method of analysis.
    - Only minor technical details of analytical methods discussed. No decisions made.

- Determination of 3MCPD
    - Discussion regarding the best method of analysis.
    - Only minor technical details of analytical methods discussed. No decisions made.

- Determination of Glycidol
    - Discussion regarding the best method of analysis.

- Only minor technical details of analytical methods discussed. No decisions made.
- It is recommended that processing temperatures are kept low to reduce glycidol

Next IOC chemists meeting - proposal for it to be held on 4-5th April 2019.

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Príncipe de Vergara, 154 – 28002 Madrid – Spain Tel.: +34 915 903 638 Fax: +34 915 631 263 - e-mail: iooc@internationaloliveoil.org - www.internationaloliveoil.org

MEETING OF EXPERTS ON METHODS OF ANALYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

Madrid, 4-5 April 2019

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MEETING REPORT

Chairs: Jaime Lillo (Deputy Executive Director); Mercedes Fernández Albaladejo (Head of Standardisation and Research Unit); Nicola Caporaso (Head of Olive Oil Chemistry Department).

The group of experts on methods of analysis for olive oils and olive pomace oils met on 4 and 5 April 2019. The meeting was held by the Executive Secretariat (ES) and took place at the headquarters of the International Olive Council (IOC) in Madrid between 9am and 3pm on 4 March and between 9am and 3pm on 5 April. On 4 April a meeting was held from 4pm to 7pm for the restricted group on the composition of olive oils with abnormal parameters.

The ES opened the meeting with the first item of the draft agenda, asking if all were in favour of adopting the report of the previous meeting (October 2018). The meeting report was approved, as well as the proposed agenda for the current meeting.

The ES informed the attendees that three documents were adopted by written procedures at the 108th session in November 2018:

i) the accreditation guide for organoleptic laboratories;
ii) the revision of the decision for physico-chemical laboratory recognition; and
iii) the agreement with the University of Navarra to create a scientific portal to collect information related to olive oil and health (olive health information system).

The following documents have been proposed to be presented at the next session of the Council of Members in Marrakesh, Morocco, in June 2019: IOC Documents 26, 19, 42 and trade standard. Document 28 will be discussed at this meeting, and it is likely to be proposed at the next IOC session.

J.R. Izquierdo asked whether corrections to the methods are still possible.

The ES commented that if there are minor modifications, the experts are invited to send comments on them and make proposals.

Electronic working group on the revision of the Codex Standard for Olive Oils and Olive Pomace Oils (CODEX STAN 33-1981) (Dr J.R. Izquierdo)

The ES proposed creating a specific group to work on the revision of the Codex standard.

Despite the IOC having only Observer status at Codex, the participation of IOC member countries is important to harmonise national norms.

The Codex physical meeting at Kuala Lumpur saw the participation of few IOC members, only the EU and Morocco. Seven delegations, led by Morocco, were in favour of keeping the ordinary category. However, the chair of the group decided to remove the ordinary category as consensus had been reached. This opens a debate among IOC members about what to do with the ordinary category.

The ES commented that if IOC members want to keep this category, they could debate it with Codex and its chair. On the contrary, if IOC members are in favour of global harmonisation, the IOC meeting should be the place to discuss the matter.

The median for virgin olive oil is not harmonised, which further weakens standardisation worldwide.

J.R. Izquierdo, as the coordinator of the eWG on the Codex revision, delivered a presentation on this matter.

The Codex norm is universal but not compulsory. It aims to obtain high quality, safe products. Documents must be structured into a main body (essential to keep the principles) and the annex (not strictly important when rejecting a product).

The decision-making process is based on consensus, which does not mean unanimity.

The two most important points of the Codex standard are based on protecting consumers and facilitating trade.

Only sections 3, 8 and the Annex of the Code document on olive oil and olive pomace oil can be discussed/changed.

Izquierdo reiterated that he worked in a neutral way, not as a Spanish delegate but as the chair of the eWG, because Spain had its own representation and because of his mandate.

During the work of the eWG, the position was to reach consensus, when possible. Countries are asked to give reasons for or against a modification, in an objective and justified way.

J. Lillo commented that harmonisation with national norms, therefore *de facto* the IOC norm, was excluded because it is an international norm. The majority of national norms do not have the ordinary category. At the Codex meeting, attendees discussed what happens with IOC members when the IOC norm is applied entirely, including the ordinary category.

J.R. Izquierdo commented that the IOC norm was taken into consideration, and therefore as were the positions of these countries as they apply the IOC standard in full (example of Tunisia).

The Codex standard is applied for edible olive oils, while the IOC standard is currently applied for non-edible olive oils on an international level. There could therefore be barriers to trade. The best possible way to avoid this would be to first decide at the level of the IOC, and then propose changes at the level of Codex.

J. Lillo commented on the action of the Codex chair, and the definition of consensus.

J.R. Izquierdo commented that his work was technical work. These questions are outside his mandate.

J. Lillo reiterated that several countries expressed their opposition to removing the ordinary category, but the chair decided that consensus was reached. The discussion should be held at the IOC within our

member countries. The commission will hold a meeting in July, so that countries might have the possibility to complain for the Codex procedure.

J.R. Izquierdo discussed keeping one decimal place for expressing analytical results. He discussed the uncertainty range of the analytical results, the significant digits of a value (values are usually expressed as the number of significant digits + 1). For values close to the legal limit, uncertainty shall be taken into account. When a value is within the uncertainty region, because of the presumption of innocence principle, it should not be considered. This is the reason why only significant digits should be used.

L. Di Giacinto asked for clarifications regarding legal limits and the presumption of innocence. Why *trans* fatty acids are kept, even if precision margins are not optimal for a legal limit (considering the significant digits).

J.R. Izquierdo replied that a second digital digit was added for acidity and for fatty acid composition. If the group had proposed changes for all other parameters, this could have led to confusion.

In the physical meeting, consensus was reached on the definition of refined olive oil.

A third topic refers to the note that refined olive oil and refined olive-pomace oil cannot be sold, except in the case where the country allows it.

Consensus was not reached on the following points:

- The definition of olive-pomace oil;
- The limit for the median of defects;
- Changing the limit for palmitic acid content, oleic acid, adopting the decision trees for Δ-7-stigmastenol (some countries need more information and data on this), FAEEs, etc.

Definition of lampante olive oil: the Codex standard only applies to edible oils so lampante oil could not be added. However, the eWG will try to add an indication for oils that do not comply with standards for edible oils.

The new eWG has the mandate to revise section 3, 8 and Appendix V, in aspects where members expressed strong concerns.

- To collect data on DAG's, PPPs, FAEE's;
- To review data on linolenic acid content by the IOC and propose limits;
- To discuss other issues raised by other countries;
- To prepare a report for the next physical meeting.

The Codex Secretariat said that they should not discuss items for which there has been clear agreement.

J. Lillo insisted that the Codex standard, despite not being compulsory, is very important and we should therefore aim to achieve complete harmonisation for all standards.

M. Fernández commented that it is important to conduct studies and investigations within the IOC, so that members and institutions can debate with other member countries and experts. She asked the opinion of the IOC experts.

J.R. Izquierdo commented that some parameters are still in the norm, e.g. the iodine value, because it is possible that some countries do not have the facilities to perform more complex analytical

determinations. Matters like these can be proposed within the eWG. These methods are also in the Appendix, so they are not compulsory. A complete harmonisation would not be possible, because some of the compulsory parameters found at the level of the IOC standards will need to be placed in the Codex Appendix (which should be kept in any way, following the Codex documents structure).

A. Faberi commented that all values are inter-related, therefore proposed changes should be carefully evaluated, e.g. limits for oleic acid content. A scientific approach is needed for any change, including the iodine value, to justify keeping or changing some methods or limits.

Olive oil has the biggest and most complex standard, therefore maintaining obsolete methods or parameters makes little sense. If nobody can carry out studies on these parameters, it might be time for the IOC to propose its removal.

L. Conte commented that new methods exist, but we should understand the extent to which obsolete methods are still applied. For example, for the iodine value there is a method by the AOCS, which might be added to the standard. Density is maintained because it is an important parameter for customs in international trade.

F. Lacoste said that there is an annex to the ISO standard 3961, which includes the option to calculate the iodine value starting from fatty acid composition, so this is already in the standard.

B. Ammar thanked Dr Izquierdo for the work of the eWG. He apologised for the absence of the Tunisian delegation at the Kuala Lumpur meeting. He thought that the ordinary category was an easy prediction. He asked how consensus could be reached at the Codex meeting if positions were not common within IOC member countries?

The debate is still open about this category, at the level of the IOC.

He suggested the ES open the debate about removing the ordinary category.

E. Christopoulou also thanked J.R. Izquierdo. She commented on the expression of results, which is different to expressing limits. Limits must not be affected by uncertainty. She agrees with Dr Izquierdo's position on this matter, which was proposed to be discussed by the IOC experts.

An eWG within the IOC expert group was created. The idea is to deal with the aspects commented by Dr Izquierdo in relation to the Codex standard, taking into consideration the Codex procedures (including a bibliographical study).

A. Faberi commented that some of the topics are almost political, and certainly economical, and so several of the chemical experts might not be competent to give opinions on this matter. He also commented that in the past the group was asked to provide technical (chemical) opinions, which were then not taken into account during the IOC session, where members asked for further justification. Thus, it is not clear to what extent the recommendations and opinions given by this eWG will be taken into consideration and applied. The mandate for this eWG should be clear, and the ES should defend the position and outcomes of this group.

Di Giacinto asked whether decisions taken within the Codex working group will affect IOC standards.

J. Lillo commented that if the eWG proposes changes that would affect the IOC standards, the changes should be discussed at the expert meeting (full), and then proposed at the IOC member session. The mandate is to discuss matters that are currently on the table.

The following experts expressed their will to participate in the work of this eWG on Codex:

A. Faberi, N. Mata, B. Ammar, A.P. Buedo, A. El Antari will be invited; J.R. Izquierdo was invited; F. Yemiscioglu asked his government and will inform the ES. Argentine and Uruguay were invited.

A note will be sent to IOC member countries to officially invite these countries and others that had no representation at the current expert meeting. The ES will send a note verbal to these countries.

It was suggested that the Egyptian representative to be contacted, as well as other countries that did not have representative at the current expert meeting.

M. Fernández commented that we experienced that in some cases the written comments of the eWG for certain countries were different from the opinion expressed at the Codex physical meeting.

Participation in this eWG is not limited to this expert group but members can express their will from other groups.

Point 1(f) – Proposal for IOC/WCO collaboration

M. García (Spain) and **A. Faberi** (Italy) have worked on this proposal for the last 3 years. The last meeting was held at the end of March in Brussels.

The final report will be shared by the ES as soon as it is released. Some 30 (2/3) voted in favour and 15 (1/3) voted against, so it was approved provisionally. We await the notification of its definitive approval.

Point 8 - Any other business and close of meeting
Revision of Method 28 (Dr. Cobo Martínez)

H. Cobo Martínez commented on the proposed method COI/T.20/DOC. 28/Rev. 2. Issues with quantifying the 70 ml of hexane used to elute the extract were highlighted. He confirmed this issue with several laboratories. The modification was to elute 70 ml of hexane from the sample to remove hydrocarbons when in the column. The issue would be to lose some internal standard and thus affect the final quantification.

The method was validated in 2010; it was proposed to add a footnote explaining that the volume can be optimised by each laboratory.

The ES commented that it might be useful to invite him to the next meeting, as he also sent a new method proposal.

F. Lacoste commented that that everyone applied the EU standard, pre-fractioning the sample to remove hydrocarbons. She did not check the interference caused by removing them or not, and it was suggested to do so.

W. Moreda commented that they might encounter issues with determining ethyl esters if the fraction is not cleaned to remove hydrocarbons. This was not an issue for extra virgin olive oil, but it might be an issue now because the method measures other oils as well.

The fraction to be eluted should be specified.

L. Conte said that there are no issues with ethyl esters because it is only applied to extra virgin. We might have two separate methods, with different application ranges, one for olive-pomace oil and another for virgin and extra virgin olive oil.

A. Faberi commented on unifying the methods: if accreditation is requested, the two methods implies that both have to be accredited, so he proposed unifying the protocols in the same method, and adding an option for the two ways of carrying out the purification.

H. Cobo Martínez agreed with Dr Faberi, because if the method is modified, the new method will have to be validated.

The ES proposed starting a study on the method instead of sending it now, and having it ready for October.

F. Lacoste commented that the method does not mention olive-pomace oils at all, so the method is not valid for olive-pomace oil. If we want to cover olive-pomace oil as well, a paragraph could be added stating that it would be best to have hexane cleaning beforehand.

The current method works well with virgin and extra virgin olive oils.

H. Cobo Martínez commented on the update of the precision values of T28. The EU has not adopted this document yet, but the EU has methods 4 and 20.

L. Di Giacinto commented that not all the precision values are there. In the first part of Document 42-2 she pointed out that a mistake was found, in which some tables refer to Doc. 18 by mistake, instead of COI T20/Doc. 28. This needs to be corrected.

F. Lacoste commented that ring tests have not been done using 70 mL hexane, and so full validation is needed.

The ES proposed to start an eWG to revise this method and to validate it, carried out by H. Cobo Martínez, A. Faberi and C. Mariani. The aim is to provide the modified method and propose it at the next meeting.

A Faberi suggested taking the original method, which includes some mistakes, because it was validated with hexane. He agreed with creating a working group.

H. Cobo Martínez discussed the original version of the method.

W. Moreda commented that we cannot apply the method without washing it with hexane.

H. Cobo Martínez clarified that it is important to validate the method and understand the range (maximum concentration) at which the measurement can be done.

L. Conte clarified that the method is to check whether olive oil is mixed with olive-pomace oil, therefore it should not be assumed that the category is known *a priori*.

W. Moreda commented that the group need to reach a milestone before the next meeting.

H. Cobo Martínez agreed with Conte, because the nature of the sample is not known, otherwise there is a risk to duplicating the method and thus the need to re-analyse the sample. The method should be of general applicability.

The ES commented that the final proposal will be shared with everyone when it is ready.

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Point 4(c) – Update on recent studies on the relationship between volatile compounds and sensory defects (Dr Gallina Toschi)

T. Gallina Toschi presented an update on the Oleum study in relation to the link between the sensory profile of extra virgin olive oils and instrumental methods.

They are in the pre-validation phase. The SPME-GC/MS method was used to study volatile compounds in EVOO, because it is a method that is both widely available and used. Flash-GC nose was also tested. For the E-nose, there is a strong chemometric component, and it is already used in the industry.

GC-IMS is a non-targeted approach which showed promising results. She suggested collaborating in order to perform a ring test and check the performance of this approach.

At least one method will be fully validated, however she suggested testing more than one method to be validated, so that results can be compared.

The full validation will be carried out in September, but pre-validation is needed.

D. García-González presented the validation of the SPME-GC-MS method for the analysis of volatile compounds in virgin olive oils. The preliminary data were shown and discussed.

Two detector types were tested, FID and MS. The same matrix was used to build the calibration curve. Repeatability of the area and the quantification protocol were discussed.

Some compounds have limited recovery, with exceptions. The method with internal standard seems to perform the best. The limits of detection and quantification were studied. He highlighted that this protocol has been agreed with many Oleum partners.

T. Gallina Toschi commented that they are trying multiple ways. The method is considered as a mixed method, in terms of compromising the robustness, the repeatability and the ease of analysis.

The idea is to provide mixtures of volatile compounds in the future, to be available to laboratories.

L. Conte commented that it will be extremely important to strictly follow the method, due to the high number of volatiles and variables. He is in favour of not make the chemical approach too complicated, because the profile of what will be present in the headspace strictly depends on their relative concentration in the liquid phase.

Regarding the FID or MS, he developed the GC-MS method but the industry asked to modify it as GC-FID.

T. Gallina Toschi commented that selecting about 20 compounds and using a mixture of compounds will help greatly.

D. García-González specified that the internal standard will be provided together with the mixture of ~20 compounds to be analysed, in mixtures that are proposed to be commercially available in the future.

P. Rovellini asked about whether the objective includes comparing the performance of the instrumental method in relation to the response of the panel test.

T. Gallina Toschi replied that it desirable to use the volatile compounds as a reference, or as an additional variable. The difficult part is reaching an agreement to obtain a robust and applicable protocol.

J.R. Izquierdo spoke about the privately funded studies that are being conducted in Spain. The data will be shared when the project is finished. He commented that the algorithms reach up to 90% of positive results (successful classification).

The implementation needs time, and some issues still need to be fixed in order to apply it on a pilot plant in public laboratories.

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Point 3(a) - Simplified method for the determination of stigmastadienes (Dr Faberi)

A. Faberi reported an update for this study but informed that analysis was not possible because of technical issues. The ES has added this information as additional data within the annual proficiency test for the IOC recognition of physico-chemical testing laboratories.

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Point 3(b) - Quantification of Erythrodiol and Uvaol (Prof. Conte)

A survey was carried out in many countries starting 3 years ago. Data has also been taken from the industry.

L. Conte collected and analysed the data, which he then presented and discussed at the meeting. He pointed out that the erythrodiol and uvaol content as currently calculated (%) are related to the sterol content.

The absolute content of erythrodiol and uvaol is commonly used, giving good results. Therefore, the method was validated again by taking into account this calculation.

He highlighted that there is often a mistake in identifying uvaol, as technicians mistakenly select the first peak eluted after erythrodiol which it is not uvaol.

The data received, even if expressed as mg/kg, were often the sum erythrodiol+uvaol. Taking this into account, as an overestimation of erythrodiol, all data were used as erythrodiol only.

Data are missing from Australia and the USA. The group has been waiting for these data since 2017.

Some oils with sterol content below 1000 mg/kg were found, as well as others whose category does not agree with the erythrodiol content.

He thinks that if different units of measurement are used for different categories of oil, there is a risk of generating confusion when asking the absolute content for a category and the relative content (%) for others.

He reflected on the fact that the compositional range will always be higher when expanding the number of samples, and that we will always have very few samples that will be considered outliers.

The proposal could be: i) only for lampante and olive-pomace oil, expressing it as absolute content; ii) for virgin and extra virgin expressing it as a percentage.

The frequency distribution in all data sets should be evaluated, to understand whether the amount of oil that would be outside the limits is representative of the market and production of a country or location.

W. Moreda asked whether detection power could be lost when it is expressed as an absolute value instead of a percentage.

L. Conte replied that it is important and should be evaluated.

E. Christopoulou asked why the data are not available to all experts for processing. She proposed that all experts have access to the data.

She asked Conte for some clarifications on his report.

L. Conte replied that the report can now be found in .pdf format. He was in charge of the work by the ES, and his reports were sent to the ES in good time so that they could be disseminated to all chemists.

A. Pablo Buedo commented on the limits in relation to Argentinian samples. He suggested studying the topic and not keeping a very restrictive value.

A. Vander Stappen commented that it would be better to wait for additional data. In the context of the Codex meeting, we should make sure that Australia and the USA are invited to send data. It does not matter if they do not provide the data, but it would be useful to include them.

L. Conte proposed: i) to start data collection, particularly for the USA and Australia, giving a deadline; ii) if data are not provided, they should accept the limits established in the study.

He said that it is important for the absolute erythrodiol content to also indicate the amount of the batch, if possible (for example, the percentage of production in terms of national production). Data should be provided in Excel, for the ease of data processing. He also asked to also include data about waxes.

He will calculate the distribution data (Gaussian) of all the data available.

He also asked for feedback about the limit he proposed.

It was suggested to set the deadline to 14 July 2019

E. Christopoulou presented a proposal on the data for erythrodiol and uvaol.

She commented that it is important to understand the percentage of these compounds (which depends on the sterol content), and their absolute content.

She commented that we cannot have different limits for this parameter (erythrodiol+uvaol) depending on the sterol content. However, it is possible to have different limits for different olive oil categories.

She presented a summary table where the results of the study are present, with some statistics related to the number of samples complying or not with the current limit for erythrodiol and uvaol expressed as a percentage, and the samples that comply when the proposed limit of 50 mg/kg is applied.

She does not agree with Conte's point of view, because a high number of deviant samples are obtained when applying the new proposed parameter.

She would like to process all the data and understand the real figures and the influence of this new parameter on the current situation.

L. Conte commented that the first peak is sometimes integrated as uvaol, however this is a mistake as it is a different compound, and this is clear when looking at retention times.

Uvaol is found at concentrations of only 9-13%.

For poor quality lampante oils, some sterols are lost during refining; the sterol content then decreases with a consequent apparent increase of erythrodiol content, which causes issues.

As some sterols are lost, being sterols at the denominator, the final value of the calculation is different.

He highlighted that samples should ideally represent the market situation, rather than very small producers, because the aim was to look at the market situation.

The erythrodiol content will continue to be a dependent variable to the sterols.

There are different limits for each category of olive oil and olive-pomace oil.

As indicated in his report, he removed the samples with sterols outside the limits (1000 mg/kg), and erythrodiol and uvaol above 4.5%.

He believes that too much time has been spent on this issue, given the high amount of data currently available. We should however reach a decision on the limit and the parameter to be used.

K. Ben Ammar said that we have been talking about changing this parameter for two years. He proposed sending a questionnaire to experts from all countries with the following questions:

i) Would you like to change from % to mg/kg;
ii) If yes, would this apply to virgins only, or for refined and mixtures as well;
iii) If yes for virgins, will this be one limit only for virgin+extra and lampante, or 2 limits;
iv) Should we consider only erythrodiol, or erythrodiol+uvaol,
v) Please propose a limit.

Based on these responses, a decision will be made.

L. Conte commented that he believes that experts should acquire the opinion of the industry, because he is aware of complaints from producers, refineries, etc.

Another question would be whether to link the new expression of this parameter to other parameters (e.g. waxes).

As a member of the restricted committee for laboratory monitoring, he is aware of cases where mistakes are commonly made, e.g. *trans* isomers, and uvaol.

J.R. Izquierdo agreed with Ben Ammar's proposal and Conte's opinion. It is important to get information from the refining industry as well.

E. Christopoulou agreed with Ben Ammar's proposal, but she is worried that chemists do not have experience on this new parameter so they may not be able to answer these questions.

K. Ben Ammar said that the experts will be able to answer, because they will see all the data, the reports and comments available so far.

M. Servili commented that the data collection should continue, and - in parallel - we should ask experts' and the industry's opinion to understand whether this issue is in fact important or not.

W. Moreda agreed with Conte's proposal. In his experience, this parameter might allow the detection of desterolised oils.

J.R. Izquierdo commented that he agrees on getting information from the refining industry, to better understand the situation, and work with real contents rather than theorical ones.

A. Pablo Buedo commented that, as this issue is only relevant for refined olive oils, he suggested keeping the current expression of limits for other oils.

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RESTRICTED MEETING ON COMPOSITION
(OLIVE OILS WITH ABNORMAL PARAMETERS)

TURKEY: DELTA-7-STIGMASTENOL
MOROCCO: LINOLENIC ACID

Point 3(b) – Application of decision trees to Δ7-stigmastenol lampante virgin olive oils

The study on Δ-7-stigmastenol has been sponsored by Turkey, as well as countries such as Palestine, Syria, Italy, etc.

E. Christopoulou presented the data. She commented that this compound is effective in the detection of fraud in terms of mixtures of olive oils with sunflower oils.

The campesterol limit at <3.3% is more effective than Δ-7-stigmastenol. Stigmasterol has no official IOC limit, but its concentration in virgin olive oil is lower or equal to campesterol, while in lampante oil it is not lower than campesterol. A limit of 1.8% for stigmasterol would not be effective.

She presented the proposal to solve the issue of lampante oils with high Δ-7-stigmastenol content by proposing three alternative decision trees, which were discussed by the experts.

She suggested that – if it is proposed to apply the decision tree – it should only be applied to the deviations and not for the samples that comply with Δ-7-stigmastenol and other compounds within the current limits.

The campesterol content of other vegetable oils is very high compared to virgin olive oil, so the proposed parameter includes three major sterol compounds (apparent β-sitosterol / campesterol + Δ-7-stigmastenol). It is expected to be higher than 21. In Greek samples it was higher than 26.

The compositional results of about 1,800 samples was taken into account. 20.3% is expected to be the limit for samples complying with the current Δ-7-stigmastenol limit, while the minimum value should be 22.2% for oils not complying with the current Δ-7-stigmastenol limit.

She proposed the limit of 25, which would cover almost 95% of the deviant samples tested and will exclude possible frauds.

F. Yemiscioglu thanked Christopoulou for her hard work, and commented that the issue of oils with high Δ-7-stigmastenol has been under attention for the last 10 years. For three years they have been trying to put forward ideas to solve the issue of deviations from lampante oils.

A decision tree exists for virgin and extra virgin olive oils and olive pomace oils. There is none for lampante.

E. Christopoulou commented that she believes the most effective decision tree will be the n.2. The value for the new parameter (apparent β-sitosterol/sum of campesterol + Δ-7-stigmastenol). She believes that the value of 22 would be effective as well, but she asked the others for their opinion.

K. Ben Ammar commented that we should limit the application of the decision tree to samples outside the current limits. He believes the issue is with the apparent β-sitosterol, not only with Δ-7-stigmastenol.

L. Conte commented that he would be in favour of decision tree n.2, because the percentage of sterols we measure bring some degraded information, as some sterols are found in esterified form in sunflower oils. He is in favour of using a more restrictive value.

A. Faberi commented that he tested decision tree n. 3 and the value is always reached, which makes this parameter redundant if these values are kept.

F. Yemiscioglu mentioned that a value of 22 is a comfortable value for virgin and extra virgin olive oils. If such values are adequate for virgins, why is the group proposing to increase it for lampante?

E. Christopoulou explained that only samples showing one deviation were used, not those showing more deviations. She applied the decision tree excluding other samples.

F. Yemiscioglu agrees with this approach.

F. Lacoste asked for clarifications regarding decision tree n.2, as it uses a value of 25 for the new parameter.

E. Christopoulou replied that the value of 22 might increase the risk of potential adulteration, however she left the choice of the value (22 or 25) to the other experts.

L. Conte commented that for Turkey, a value of 0.7 or 0.8 would guarantee the best performance. He asked about how important this deviation is in terms of the amount of oil that is affected for Turkey.

F. Lacoste said that on the value of decision tree n.2, the value of 25 will not be reached.

K. Ben Ammar commented that mathematically, one cannot reach the value of 25 for this parameter.

L. Conte commented that the calculation brings to approximately 23.

E. Christopoulou: with a value of 22, there are 87% of non-deviated samples, while with a value of 25 there are 79% of non-deviated samples.

M. Servili commented that we cannot accept a lampante oil with a campesterol content below 3.3%. We can discuss setting the value for the ratio at 23 or above, but 22 seems too low because it will not have discriminating power.

A. Faberi commented that mathematically, 22 is low but a value of 25 is too high.

The experts debated the calculations in the formula.

M. Servili commented that we need to decide between 0.7 or 0.8. The ratio makes sense if we put 23 or below as a limit.

K. Ben Ammar commented that in the case of 23 this parameter would not be useful.

F. Yemiscioglu would be comfortable with using a value of 0.8, or even 0.7, as the group will decide.

L. Conte was not in favour of using a strict limit now that may be changed later on to a higher limit as this does not give a good image. He is in favour of 0.8.

E. Christopoulou said that she is in favour of a value of 25.

J.R. Izquierdo commented that if we accept 0.8 as a value, we should use 23 as the value of the formula.

All experts agreed with a value of 0.8, and Christopoulou commented that there is a small difference between 0.7 and 0.8.

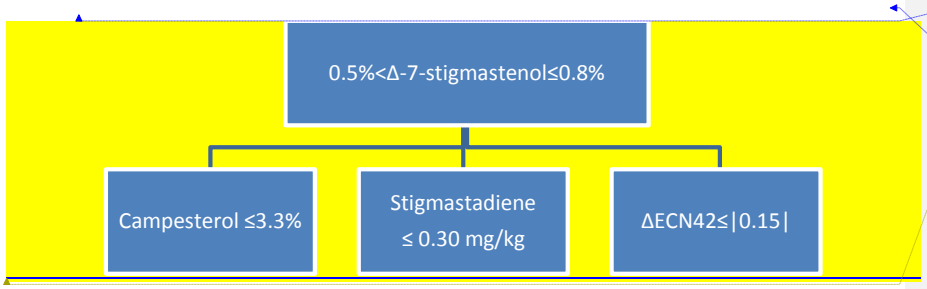
J.R. Izquierdo also commented that in reality the use of this ratio is not needed, if we use the value of 22.

Argentina commented that a higher value would be more valuable.

K. Ben Ammar said that a high value of β-sitosterol is needed to reach a ratio above 23, which would cause issues.

Decision tree n.3 with modifications was unanimously proposed by the group, based on the wide range of data and calculations. It was therefore agreed to use the following decision tree:

- ~~ΔECN42 ≤ | 0.15 |~~
- ~~Delta-7-stigmastenol ≤ 0.8~~
- ~~Campesterol ≤ 3.3~~
- ~~Apparent β-sitosterol / (campesterol + Δ-7-stigmastenol) ≥ 23~~
- ~~Stigmastadiene ≤0.30~~



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Con formato: Resaltar

K. Ben Ammar asked if it was necessary to revise the decision tree for virgin and extra virgin olive oils.

The ES commented that we could discuss the matter to check whether or not it is useful.

A. Faberi commented that it is a useful debate and the decision tree should probably be revised.

A. Pablo Buedo proposed keeping 25 for extra virgin and virgin olive oils.

F. Lacoste said that it might be a problem having a parameter for virgin olive oils but not for lampante.

L. Conte commented that when calculating the decision tree for virgin olive oils the value can be reached with difficulties.

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Point 5(b) - Application of decision trees to linolenic acid

The IOC Council of Members adopted the Moroccan proposal for a decision tree on linolenic acid content. The ES commented that samples from Morocco had been received for analysis, but they could not be analysed because there were not enough of each sample to be sent to other laboratories.

N. Mata pointed out that, over a three-year period, between 30 and 40% of the oils had issues with their linolenic acid content. She highlighted the importance of this issue for the national economy, also considering increases in production over the last few years.

E. Christopoulou conducted a study on the data provided by Morocco, which has the greatest deviations of linolenic acid content. This fatty acid has been defined as a "critical" fatty acid, because it is a good indicator of fraud. She commented on the data provided by Morocco (Doc 5.b.1.2). The issue of linolenic acid has been examined since 1998. Morocco has the greatest deviations related to this parameter. However, she does not have a solution for this issue at the moment.

L. Conte commented that the data came from 54 Moroccan samples, of which 14 did not comply with the proposed campesterol limit. Out of 54, 25 were above the limit for ECN42. He commented that we must be careful with stigmasterol because some other oils like rapeseed oil do not have stigmasterol, which can lead to fraud.

J.R. Izquierdo commented that the group might think about a decision tree to define the minimum amount of refined olive oil added to virgin olive oil.

E. Christopoulou said that rapeseed (*Brassica* in general) has a high brassicasterol content so it is a very sensitive parameter for detecting these oils in olive oils. However, it is not a good parameter for other types of vegetable oils.

She would like to combine these parameters as using only ΔECN42 would not be effective either.

Stigmastadiene could be a good parameter but not for all vegetable oils.

K. Ben Ammar understands the position of Morocco. However, it is difficult to decide the limit, and there are other restrictive parameters to consider in the decision tree. He does not want to make any changes to ΔECN42, but he is in favour of discussing which sterol compound to use.

N. Mata said that there was an error in the results for campesterol and stigmasterol; she will communicate the correct results to Dr Antari, who was unable to attend the meeting.

L. Conte said that when adulterating with other oils, these oils will alter the sterol composition depending on the ratio of added oil, and the initial sterol composition.

It is possible to calculate the minimum amount of oil that could be added without fraud being detected. The use of partially desterilised oil was also taken into consideration for the calculations.

He suggested taking these data and checking the calculations so that we could verify and simulate the potential data.

M. Servili said that desterolised oils exist, but that they should also consider the commercial side of the issue, such as the cost of this process. He believes that Conte's suggestion about the decision tree is the only possible solution.

E. Christopoulou said that to check for possible adulteration, the total sterol content must be taken into account. She also commented that brassicasterol is six times more sensitive than campesterol in detecting the presence of rapeseed oil in olive oil.

The ES proposed sending a survey to all IOC member countries.

We need more samples. Dr Antari knows the protocol and it should be applied in order to allow proper data collection.

The proposed action is to ask for more data to carry out another wider study.

J.R. Izquierdo suggested the ES send a letter to the USA and Australia to ask them to provide data for the study.

F. Yemiscioglu said that the linolenic acid issue has a 10+ year history. These countries should make a more detailed plan for these studies. The committee should share the details of what we want to see. The issue is clear, but a clear plan is needed, e.g. decision tree with specific compounds.

E. Christopoulou said that Algeria, Argentina, Australia, France, Israel, Lebanon, Morocco, New Zealand, and even Spain, all have this issue.

L. Conte commented that Argentina, Australia and a few other countries had highlighted this issue in the past. The data published in Australia showed that no oil had value higher than 1.1% linolenic acid, so Australian scientists did no further study into the issue.

A. Pablo Buedo said that linolenic acid deviation is not common. It exists but it is not frequent so it is not an important issue.

N. Mata commented that Dr Antari has not yet made a proposal but he is working on this and he will send a proposal in due course.

The ES commented that it will send the request for the survey in the next few weeks. The data should be provided before 30 June.

The data should be provided in Excel.

Samples will also be analysed by the IOC if needed, following the usual protocol.

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Point 3(c) – Study of MRLs of pesticides (T. Generali)

T. Generali presented the report of the work she had coordinated and analysed the results of the survey. She highlighted that some difficulties were found in getting all the answers. The work can be considered as finished.

She suggested the ES redact a unified document, that can be updated periodically with new information.

She presented and discussed the results of the annual proficiency test to evaluate the residues of pesticides in olive oils.

Commercial olive oils from the market were sampled and found to be contaminated by 27 pesticides, at a known amount.

Some 38 laboratories participated, 5 of which were EU national reference laboratories for pesticides.

Out of the 38 laboratories, 32 analysed all compounds.

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Point 4(a) - Method for the determination of methanol and ethanol in virgin olive oils (Dr Moreda and Dr Faberi)

W. Moreda provided samples for ring tests for polyphenols and methanol quantification. He presented the status of this point at the last meeting of 4-5 October. Training samples were sent to laboratories in order to check if they encountered difficulties and to receive any comments or feedback they may have.

The samples for the actual ring tests were shared during the current meeting.

SPE-HPLC-DAD and hydrolysis with HPLC-DAD were the two methods tested.

It was requested to strictly follow the protocol shared to laboratories willing to participate in the ring test. The proposed deadline to send in results was the end of May.

He asked participants to inform him of any issues or questions they may have on this matter.

It is known that the current provisional method underestimates the real content of phenolics.

It is important to propose a universal method which gives the most "real" value.

The method that applies hydrolysis is important because of how easy it is to use in the industry.

T. Gallina Toschi said that they will send some feedback on the method, but they will ask if they could fix some of the issues they had before the actual ring test. If there are different columns or conditions, the results cannot be compared.

She asked Dr Moreda to set a stable method.

W. Moreda said that two extraction methods are proposed, so that every laboratory can decide which one to apply. If every laboratory applies both, that would mean they could compare them both and see which protocol is more efficient. In this case, everyone should apply both methods and strictly follow the protocol provided.

L. Conte asked for clarifications on Table 1 of Dr Moreda's report presenting the results of total phenols, and whether each laboratory calculated their response factor when analysing the ethanol content.

It was clarified that they are just the sum the hydroxytyrosol and tyrosol; each laboratory has to calculate their response factor.

M. Servili commented that for hydrolysis, we should define a conversion factor of the results, only related to the bioactive secoiridoids. Lignans and phenolic acids should be excluded.

W. Moreda replied that we will only consider the secoiridoid derivatives, which have the highest antioxidant activity (orto-diphenols).

L. Di Giacinto commented that several years ago the two extraction protocols compared about 40 samples and the results were perfectly comparable.

T. Gallina Toschi commented that the ring test will give confidence on which method is more valid, but if the final decision will be to keep both protocols they should be harmonised.

P. Rovellini commented that they carried out both ring tests, by strictly following Dr Moreda's method. It should be stressed that each laboratory should calculate the response factor.

They have difficulties in applying the calculation tool and she asked for explanations from Dr Moreda.

When they applied the calculation as tyrosol, they obtained identical results to Dr Moreda's method.

During the last meeting, it was agreed for her to ask for updates on ISO. Argentina, Canada, China, Germany, the USA, Italy, etc will join the working group. At the next meeting in February 2020 in Sydney, the method will be proposed within the SC11 sub-committee. She suggested that it would be good to validate the method within the IOC as the IOC is in the SC11..

The method was sent to the ES, which will upload it in the restricted area of the website.

W. Moreda replied that the samples do not have secoiridoid compounds, which is where the most mistakes are made when applying the method (because their molecular weight is assumed to be the same as tyrosol). He suggested that differences will be found in the new ring test.

For the hydrolysis issue, the aim is to find an easy method. He believes that extracting the compounds to carry out HPLC analysis and then hydrolysing them would not make sense.

The values of the area of the IS they used (Rovellini) is very high, and he cannot understand why.

P. Rovellini wanted to correlate the biophenol content analysing the current method with the new proposed method. This is why they carried out the test with and without analysis.

W. Moreda asked who is interested in the ring test (22 samples for phenols and 20 for methanol are available, with a wide composition range).

Deadline: 31 May 2019.

This will be used as the rapid method, in addition to the full profile.
His opinion is that it makes little sense hydrolysing after extracting the phenols.
The issue of using the EFSA health claim is the underestimation of the content.
He said that if in the future new methods will be found, the group can implement them in the official method, but it is important to finalise this step.
The following experts expressed their interest in participating to the ring tests:

Stigmastadiene:	**Phenols:**	**Metanol:**
1. A. Faberi	1. Unaproliva, M.D. Gómez	1. J.R. Izquierdo
2. J.R. Izquierdo	2. L. Di Giacinto	2. H. Cobo Martínez
3. W. Moreda	3. S. Iosifidou	3. R. Luque (2)
4. M.M. García González	4. Z. Kerem	4. F. Lacoste
5. B. Butinar	5. J.R. Izquierdo	5. A. Galli
6. F. Lacoste	6. M.M.García	6. P. Rovellini
7. D. Ollivier		7. F. de Paula

8. A. Gali 9. S. Iosifidou 10. P. Rovellini 11. M. Monfreda 12. M. Servili 13. T. Gallina Toschi 14. Chemiservice 15. M.F. Costa 16. A. Alegre 17. J. Ayton 18. L. Ravetti 19. A. Sheridan 20. N. Mata 21. K. Ben Ammar (3) 22. S. Okan 23. C. Dologh 24. A. Stenvik 25. G. Estévez 26. R. Silva 27. Unaproliva– M.D. Gómez	González 7. H. Cobo Martínez 8. R. Luque 9. F. Lacoste 10. D. Ollivier 11. K. Ben Ammar 12. T. Gallina Toschi 13. J. Ayidou 14. M. Servili 15. C. Machado 16. P. Rovellini 17. F. de Paula (2) 18. B. Butinar 19. E. Perri 20. I. Willenberg	Rodriguez (2) 8. Mª Dolores Gómez 9. T. Gallina Toschi 10. H. Alegre 11. L. Ravetti 12. N. Maata 13. K. Ben Ammar

W. Moreda will send instructions by email to participants to the ring tests for biophenols and methanol.

L. Di Giacinto proposed also analysing it by hydrolysing the extracted fraction.

It was debated whether to also test this way, as an additional protocol.

F. Lacoste said that the Italian method was proposed at the ISO meeting. It would be an issue to have two different methods, one applied at the ISO and another at the IOC.

We need to compare three methods, in order to prove that there are differences and that we cannot state that the results are equivalent (comparable or not).

L. Conte commented that IOC non-member countries will probably apply the ISO method when evaluating the health claim. It is therefore important to compare the ISO method and this new method. We should avoid dichotomy among ISO and IOC.

P. Rovellini commented that there is no ISO comment distributed to the experts.

The ES commented that it will be shared on the restricted section of the website.

T. Gallina Toschi and **M. Servili** commented that they believe it might be a good solution to test three methods, in order to definitely end this issue.

The ES commented that future work should be aimed at finding solutions and avoiding working without advancing and reaching an outcome.

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Point 6(a) – Legal and illegal blends between olive oil and other vegetable oils – OLEUM (Dr. Moreda)

W. Moreda delivered a presentation on a method to detect illegal mixtures of olive oils with other vegetable oils.

A. Faberi asked whether experiments have been carried out using the global method also for oils that have anomalous composition, particularly those with high linolenic acid content.

W. Moreda replied that he had run experiments for those oils, as well as oils with anomalous steel composition. For the latter, no issues were detected, while for the high linolenic content it depends on the other parameters. It will be interesting to check this with the Moroccan samples.

The ES commented that the ES does not have enough oil from Morocco. N. Mata was asked to send more oil and she asked how much exactly. Moreda replied that 15-20 grams are enough for him.
The IOC study requires three bottles of 250 ml, because one bottle is stored at the ES.

W. Moreda said that if someone would like to analyse samples with abnormal sterol composition, they can send them to him for analysis.

Z. Kerem commented that the samples sent to Chemiservice had very good results but they showed a 0.29 difference on Picual. He asked whether it is a case of anomalous composition.

About the legal blends, they mix at least 50% of olive oil with other oils. The aim was to evaluate the minimum amount of olive oil.

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Point 6(c) – Progress of the expert group on contaminant residues

N. Caporaso gave updates on the work on contaminants.

A Faberi informed that the private industry has been carrying out analysis for 3-MCPD, as well as information related to the refining condition (these data have also been provided by SISSG).

Data are split into olive oil, refined olive oil, and extra virgin olive oil. The EU Commission will shortly update the regulation 1881 by introducing the limit for 3-MCPD.

The industry is not in favour of a double limit, but the EU was.

For virgin and extra virgin olive oil, which are virtually absent of this compound, he said it might be convenient to propose an even stricter limit for this category.

W. Moreda presented his results about the presence of 3-MCPD in olive oils and olive-pomace oils. He also commented that the position of the EU is to keep virgin olive oils out of these classifications, and only include refined oils with a limit, due to the practical absence of 3-MCPD in unrefined olive oils.

- 1.25 ppm for coconut, corn, plan, sunflower, rapeseed, soybean (and their blends).
- 2.50 ppm for the rest

As there was not enough data for olive oil, it was placed in the second category.

It is impossible for olive-pomace oils to be within 1.25ppm. Their content is high (e.g. 3-5ppm), therefore this category will need to be included in the 2.5 ppm category. On the other hand, many

extra virgin olive oils are below LOQ. Even olive oil (mixture) is clearly below 1.25 ppm, therefore all oils of this category comply with the stricter limit.

MOSH and MOAH is also a worrying topic, because contamination can be found at many stages of the production chain.

Second centrifugation or olive-pomace oil (but also organic VOOs) contain MOSH and MOAH.

For MOSH, the group has until 2020 to send data. Otherwise, the EFSA will release an opinion without enough data if they are not provided for the olive sector.

The data discussed by Dr Moreda showed that:

- Organic EVOO had 350 ppm MOSH, and 30 ppm MOAH.
- ROO had MOAH of 16 and MOAH 9 ppm.
- Olive-pomace oils had 150-200 ppm MOSH and 40-160 ppm MOAH.

It was also highlighted that the potential for error when analysing the oil, especially when manually integrating the peaks, can be very high.

F. Lacoste carried out some screening 4 years ago and she knows that some oils have more issues than others. She has seen some samples with a content of a few dozen mg/kg, but she has never seen values of 300 mg/kg for virgin olive oils (virgin and extra virgin).

She is not convinced that VOO can have such a high content.

In oils extracted by solvent from residues, the contents of MOSH can be high, and these are the only oils in which high contents can be found.

W. Moreda said that it is important to include other industries, such as producers of pesticides, herbicides, additives, etc that could bring organic matrices, even the lubricant sector which represents contamination sources.

There are sunflower oils which do not show or show very low MOSH and MOAH.

F. Yemiscioglu commented that the group needs to look at the formation mechanism. Mono and diglycerides are the precursors of these compounds, if high temperatures are applied. There is no condition of formation of these compounds in unrefined olive oils, i.e. 3-MCPD and glycyl esters.

For refined olive oils, the limit for glycidyl esters is 1ppm. Oils containing a higher amount of monoglyceride and diglycerides are risky; there is different lipase activity between seeds and fruits (e.g. coconut, olive).

30% of Turkish production has a high amount of diglyceril esters due to the deodorisation process.

There are 2 accredited methods, one of which is of crucial importance.

In olive-pomace oil, in Turkey there is experience in the preparation of table olives: if they are kept in unsalted water and then used for lampante oil production, the brine (containing chlorine) might make it produce 3-MCPD at high temperature.

He believes that chemical refining is safer because high temperature is not needed.

A. Vander Stappen commented that the extension given by the EU to allow IOC members to collect data from the ES is short. She suggested sending them as soon as possible, in terms of weeks, not months.

The ES commented that the data have been collected and the ES will probably send an official response within a few weeks.

W. Moreda commented that his data for 3-MCPD have been presented, and only data from other countries are missing at the moment.

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The ES commented on a proposal to write a protocol for laboratories, in order to support them in terms of training, potential issues they might encounter and give suggestions to improve their analytical performance, as well as to arrange visits to verify when doubts arise about their activity. This activity is organised by the ES, in collaboration with some experts, and it will be carried out in collaboration with the company that carried out the proficiency testing for the IOC recognition. At the next meeting, this protocol will be presented to the experts.

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Point 6(1) – Presentation of the research project H2020 OLEUM (Prof. Gallina Toschi)

T. Gallina Toschi presented updates on the OLEUM project.

The next steps of the project will be to present a report on emerging frauds, as well as presenting the four methods to be fully validated together with the reference material.

The work on reference material for defects of winey-vinery and for rancid has progressed and samples will be ready soon.

The ring tests will be performed on 8-12 laboratories. Two methods have been identified for full validation.

They are in the pre-test phase so a complete action will be completed in June.

Other work is in progress regarding the study of the effect of storage conditions (light, temperature), to produce a predictive software, which will need validation.

A method for phenolic compound analysis has been validated, the results have been published on Molecules. It was decided to bring the phenolic analysis method to a validation step.

Tullia asked the OLEUM partner to wait for the start of the ring test organised by Dr Moreda, because we all agreed that one official method is needed. Despite the lack of wide distribution of UHPLC needed for the OLEUM method, Gallina Toschi noted that in the next few years the UHPLC instrument will be diffused widely. Therefore, the final suggestion would be to propose this method as a parallel one.

Scheduled timing: October 2019 they will have a review meeting in Bruxelles; June/July 2020 in Bologna; at the end of the meeting it will be at the IOC (fall/winter 2020).

The ES commented that the IOC is happy to serve as a forum of scientific and technical discussions on these topics, and that the ES is happy to host the OLEUM coordinators to share the project conclusions.

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The next meeting will be hold on 3-4 October.

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SUMMARY OF THE MAIN POINTS OF DISCUSSION

Electronic working group on the revision of the Codex Standard for Olive Oils and Olive Pomace Oils

J. R. Izquierdo coordinated the Codex eWG on the revision of the Codex standard.

The ES proposed the creation of a specific group aimed to work on the Codex standard revision.

During the Codex meeting, despite the opposition of a few countries, it was considered that consensus was reached, and the ordinary category was removed.

A new eWG was created to revise section 3, 8 and Appendix V of the Codex standard. The main tasks are:

- To collect data on DAG's, PPPs, FAEE's;
- To review data on linoneic acid content and propose limits;
- To discuss other issues raised by other countries;
- To prepare a report for the next physical meeting.

Experts suggested opening a debate among IOC members about removing the ordinary category in the IOC standard.

An eWG within the IOC expert group was created. The following experts will participate, but the invitation is open to other experts as well:

A.Faberi, N. Mata, B. Ammar, A. Vander Stappen, A. El Antari will be invited, J.R. Izquierdo was invited, the representative of Turkey asked his government and will inform the ES. Argentina and Uruguay were invited

The IOC ES will send an invitation to other experts and countries that were not present during the meeting.

Revision of Method 28

H. Cobo Martínez proposed revising Method COI/T.20/DOC. 28/Rev. 2 for the volume to be used to elute the column.

The ES will invite Dr Mariani to the expert group, as he is the author of the original version.

The ES also proposed starting an eWG to revise the method and propose it for the October meeting.

Some experts commented that a full validation is needed.

When the eWG has a final proposal, the ES will share it with all experts.

Update on recent studies on the relationship between volatile compounds and sensory defects (OLEUM project)

Three different approaches for volatile compound analyses were studied (SPME-GC/MS, Flash-GC, E-nose). At least one method will be fully validated in September, after a pre-validation step.

Quantification of Erythrodiol and Uvaol

This study started three years ago, coordinated by Prof Conte.

Data are missing from Australia and the USA. The expert group has been waiting for these data since 2017.

The coordinator suggested, for lampante and olive-pomace oil, to express the value as absolute content, while for virgin and extra virgin to use percentage.

Other experts asked the value to be available to all experts in Excel format in order for them to run other calculations or mathematical tests.

It was proposed to start a data collection, particularly for the USA and Australia, giving a deadline; if data are not provided, they should accept the limits established in this study.

Data should be provided in Excel format, with possible indication of the amount of the lot, i.e. the significance of the production on a national level, in order to understand the impact of that specific sample/variety. It was also suggested to include data about waxes.

Prof Conte will calculate the compositional distribution of the samples when he has received all the data. He also asked for feedback about the proposed limit.

The deadline is set to **14 July.**

It was proposed, in order to progress on this issue, to share the following questionnaire to all experts:

i) Would you like to change from % to mg/kg;
ii) If yes, would this apply to virgins only, or for refined and mixtures as well;
iii) If yes for virgins, will this be one limit only for virgin+extra and lampante, or 2 limits;
iv) Should we consider only erythrodiol, or erythrodiol+uvaol,
v) Please propose a limit.

Experts suggested also acquiring the opinion of the industry (refineries).

Application of decision trees to Δ7-stigmastenol lampante virgin olive oils

Turkey has important issues for lampante oils with abnormal Δ7-stigmastenol concentration.

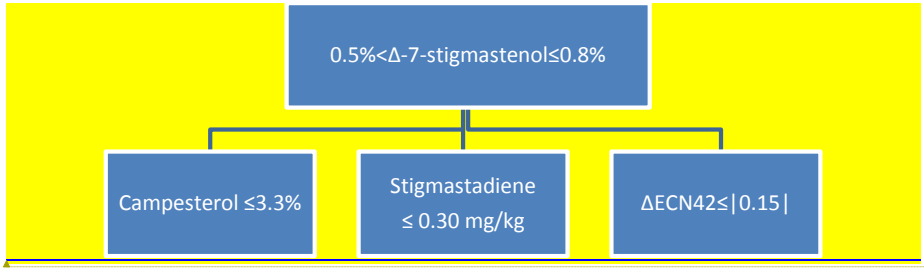
Dr Christopoulou presented a study on this aspect, and proposed three decision trees to overcome the issue of these lampante oils, on a dataset of approximately 1,800 samples.

~~After some discussion and calculations to understand the impact of each decision tree in terms of false positive, the group unanimously agreed on proposing decision tree n.3 with modifications: the deltaECN42 ≤ | 0.15|, campesterol % ≤3.3, delta7 stigmastenol % ≥23 and stigmastadiene ≤0.30 (%).~~

After an extensive debate and analysis of the data provided, three decision trees were presented to solve the problem of lampante virgin olive oils with a delta-7-stigmasterenol content that is not standard:

category : lampante virgin olive oils	decisional tree 1		decisional tree 2		decisional tree 3	
	limits	conformity %	limits	conformity %	limits	conformity %
ΔECN42	≤0,20	100	≤0,15	100	≤0,15	100
campesterol %	≤3,0	55	≤3,3	91	≤3,3	91
delta-7-stigmasterol %	≤0,7	80	≤0,7	80	≤0,8	87
ap. β-sito/(campe+Δ7-stigma)	≥25	79	≥25	79	≥22	99
Stigmastadiene mg/kg	≤0,30	100	≤0,30	100	≤0,30	100

The tree unanimously proposed by the chemistry group:



Application of decision trees to linolenic acid

Morocco asked for a decision tree on linolenic acid content, as their oils have issues with high linolenic content in genuine oils.

Dr Christopoulou carried out a study on the data provided by Morocco.

Changing the limits is not easy when the aim is to detect fraud, because there are partially de-sterolised oils for which fatty acid composition gives good indications.

Experts suggested defining a decision tree.

Con formato: s6, Revisar la ortografía y la gramática

Con formato: Resaltar

Dr Antari will send a proposal to solve the issue of oils with high linolenic content.

The ES proposed sending a survey to all IOC member countries in order to get more data. It was suggested to invite the USA and Australia. The expected deadline to provide data is **30 June.**

Study on MRLs of pesticides

Dr Generali presented and discussed the results of the annual proficiency testing done to evaluate the residues of pesticides in olive oils.

Some 38 laboratories participated, 5 of which are EU national reference laboratories for pesticides.

She suggested the ES redact a unified document and share it.

Method for the determination of methanol and ethanol in virgin olive oils, and Determination of phenolic compounds: revision of IOC method

Dr Moreda presented the status of this point in the October meeting. Training samples were given to laboratories in order to check whether they encountered difficulties, and to ask for their comments and feedback. Samples to perform the ring tests were distributed at the current meeting.

The deadline set to provide results of the ring test is **31 May.**

It was asked to strictly follow the protocol provided. Two alternative extraction methods are proposed, so that everyone can decide which one to apply. However, it would be better to test both, so that results could be compared.

The method with hydrolysis is important because of the ease of use for the industry.

It was commented that ring tests give a strong confidence on which method is more valid, but if the final decision will be to keep both protocols they should be harmonised. Also, the response factor should be calculated by each laboratory

Dr Rovellini is working on a different method at the level of ISO sub-committee C11, in which a method will be proposed in February 2020.

The ES will upload Rovellini's method to the restricted area of the website, for all experts.

Some 21 laboratories are participating in the ring test for phenolic compound analysis.

Some 15 laboratories are participating in the ring test for methanol analysis.

Dr Moreda will send instructions by email to the participating laboratories.

It was eventually agreed to run three methods, i.e. the two proposed by Dr Moreda and the one proposed by Dr Rovellini, so a full comparison will be available.

Legal and illegal blends between olive oil and other vegetable oils

Dr Moreda presented an update on this work.

Laboratories that have oils with anomalous composition, either in terms of linolenic acid content or abnormal sterol composition, can send their samples to him for analysis.

Updates on the OLEUM project

Prof. Gallina Toschi presented the updates related to the OLEUM project.

Among others, at least one method will be validated for the support of the panel test, as well as two protocols (GC-MS and GC-FID) for the quali-quantitative analysis of 18 volatile compounds.

They are in the pre-test phase so that a complete action will be finished in June.

Other work is in progress regarding the study of the effect of storage conditions (light, temperature), to produce a predictive software, which will need validation.

A method for phenolic compounds analysis has been validated; the results have been published on Molecules.

Progress of the expert group on contaminant residues

The work on 3-MCPD limit is a priority within this group, as the EU will shortly set a limit, and given the lack of data provided for the different grades of olive oils and olive-pomace oil, the risk was to put all of them in the higher limit category, damaging the image of olive oil as a healthy fat. It was agreed to delay the decision by a few weeks in order for the IOC to provide data, therefore the members participating in the eWG on 3-MCPD are collecting and sending the data to the ES.

The limits for MOSH and MOAH have been also discussed, in terms of the presence of these compounds in different grades of olive oils and olive-pomace oil, as well as the formation mechanism. The group on possible contaminants in olive oil and olive-pomace oil will have a physical meeting on 28 May.

Final remarks

The ES commented on a proposal to write a protocol for laboratories, in order to support them in terms of training, potential issues they might encounter and suggestions to improve their analytical performance.

***

Plan for the chemistry expert group

Electronic Working Groups (eWG) and Actions

Group	Members	Objectives	Action	Deadline
eWG0 Codex discussion	**E. Christopoulou** *A. Faberi, N. Mata,* *B. Ammar,* *A.P. Buedo,* Invited: *A. El Antari* *J.R. Izquierdo* *F. Yemiscioglu* *Argentine and Uruguay invited.* *Other members are invited by the ES*	-To understand the Codex framework -To reach an agreement and find a unified position within IOC member countries -To debate the opportunity to remove the ordinary category	A: The ES to invite experts and representatives of member countries B: Members to discuss topics related to the Codex norm for olive oil and olive pomace oil C: To present proposals and comments from expert at the next physical meeting D: To continue the debate and discussion	A: 15 May B: 27 Sept C: 4-5 Oct D: 2020
eWG1 Method Doc. 28 revision	**C. Martínez** **A. Faberi** C. Mariani	-To propose a revision of method T.20/DOC. 28/Rev. 2 -To validate the method	A: The ES to invite other members B: Members to discuss a revision (e.g. use of hexane, etc) C: Members to plan a method validation D: To propose the revision at the next meeting	A: 15 May B: 15 July C: 15 Sept D: 4-5 Oct
eWG2 Eryt+uvaol data collection	**L. Conte** **ES** *All countries, IOC members and non- members, are requested to participate*	-To gather data on the levels of erythrodiol and uvaol in olive oils and olive pomace oils -To carry out a survey to gauge opinions on how to express e+u content -To propose a limit	A: The ES to invite countries, especially the ones that have not yet replied (Australia, USA) B: Deadline for countries contacted C: The ES to collect data and share it with coordinator D: Coordinator to analyse the data and share with the ES to be discussed at the next meeting	A: 30 April B: 14 July C: 13 Sept. D: 4-5 Oct
eWG3 Linolenic acid data	**E. Christopoulou** **ES** *All interested experts and countries (including non-members)*	-To collect data on levels of linolenic acid in abnormal samples -To propose a solution to the issue, e.g. a decisional tree	A: The ES to start a survey on this matter, especially for countries that did not provide data B: Deadline reached C: Coordinator and the ES to analyse the data collected, and share it with members D: Members to discuss and propose solutions to solve this issue at the next meeting	A: 30 April B: 30 June C: 30 Aug C: 4-5 Oct
eWG4 Methanol validation	**W. Moreda** J.R. Izquierdo H. Cobo Martínez R. Luque, F. Lacoste A. Galli, P. Rovellini F. de Paula Mª Dolores Gómez T. Gallina Toschi H. Alegre, L. Ravetti, N. Mata, K. Ben Ammar	-To validate the method for the analysis of methanol content	A: The ES to send samples to laboratories B: Laboratories to analyse the samples and send results to coordinator C: Coordinator to analyse the data D: Coordinator to provide a summary report to be discussed at the next physical meeting	A: 15 April B: 31 May C: 31 July D: 27 Sept
eWG5 Biophenol validation	**W. Moreda** M.D. Gómez, L. Di Giacinto S. Iosifidou, Z. Kerem J.R. Izquierdo, M.M.García González H. Cobo Martínez R. Luque, F. Lacoste D. Ollivier, K. Ben Ammar T. Gallina Toschi J. Ayidou, M. Servili C. Machado, P. Rovellini F. de Paula, B. Butinar	- To validate the method for the analysis of phenolic compounds	A: The ES to send samples to laboratories B: Laboratories to analyse the samples and send results to coordinator C: Coordinator to analyse the data D: Coordinator to provide a summary report to be discussed at the next physical meeting	A: 15 April B: 31 May C: 31 July D: 27 Sept

Con formato: Español (España, internacional)

	E. Perri, I. Willenberg			
Δ-7-stigmastenol	**E. Christopoulou** **ES**	-To propose a decisional tree for lampante oil	ES to propose the agreed decisional tree for lampante oils to the IOC session	Next Session
Laboratory support	**ES**	-To propose a protocol for laboratory support	ES to draft a protocol to support and guide laboratories to improve their performance	4-5 Oct

***** Experts indicates in **bold** are coordinators of the eWGs.

Expected results and deliverable by next meeting (October 2019)
on methods of analysis for olive oils and olive pomace oils

Group	Responsible	Achievement	Deliverable
eWG0 Codex discussion	**E. Christopoulou**	Agreement reached among IOC member countries on the common position in relation to the Codex standard	-Report on Codex producers -Report on IOC member positions -Written or verbal expression of commitment to the new proposal by country representatives
eWG1 Method Doc. 28 revision	**C. Martínez** **A. Faberi**	Revision of T.20/DOC. 28 proposed at the expert meeting	-Revision sent to the ES -Revision proposed at IOC session by the ES
eWG2 Eryt+uvaol data collection	**L. Conte** **ES**	-Data obtained from all countries on eryrtrodiol+uvaol; -Agreement reached on how to express the content	-Summary report of all data available -Summary of responses to the survey -Written or verbal expression of commitment to the new proposal by country representatives
eWG3 Linolenic acid data	**E. Christopoulou** **ES**	-Data and survey responses obtained from all countries -Proposed solution to the issues of olive oils with high linolenic content	-Report on the survey and summary of the data collected (analysed statistically) -Proposal to be discussed at the next physical meeting
eWG4 Methanol validation	**W. Moreda**	Method validated for methanol analysis	-Summary report on the results of the validation test
eWG5 Biophenol validation	**W. Moreda**	-Method validated for phenolic compounds analysis -Agreement reached on the method to be officially proposed as the IOC method for biophenols	-Summary report on results of the validation test -Method proposed at the ES (next physical meeting) to be presented at the November IOC session
Δ-7-stigmastenol	**ES**	-Proposed decisional tree accepted by IOC members	-Decisional tree proposed by IOC members
Laboratory support	**ES**	-Draft protocol discussed and agreed at the next meeting	-Draft protocol presented to experts by the ES

International Olive Council

MEETING OF CHEMISTS ON METHODS OF ANALYSIS FOR OLIVE OILS AND OLIVE POMACE OILS

08 October 2020 – 7 pm to 3 am (AUS time)

Attendance through video conference: Claudia Guillaume and Jamie Ayton

Minutes taken by Claudia Guillaume

(1)Results of collaborative studies and tests/surveys scheduled for 2020

a)Simplified method for the determination of stigmastadienes (Dr. Faberi)

The results of this collaborative were collected and analysed resolving that this simplified method will be adopted as an alternative to the current method. However, if any litigation arises, the original stigmastadienes method will need to be used

b)Method for the determination of methanol and ethanol in virgin olive oils (Dr. Moreda)

The collaborative study was completed but the numbers of laboratories that responded were limited (only 10 of 17) so could not be achieved a conclusive decision. Dr Moreda will follow up anomalous results with the labs and encouraged the labs that haven't responded to send the data before end Nov-20. Australia has participated in this collaborative.

c)Determination of phenolic compounds (Dr. Moreda)

The collaborative study didn't work as expected and the results were not conclusive. No decision can be made at this stage.

d)Quantification of erythrodiol and uvaol (Prof. Conte)

Absolute concentration (mg/Kg) of erythrodiol in selected categories of olive oils.
The proposal was made some years ago in order to facilitate the trade of some Lampante oils with erythrodiol + uvaol relative content (%) closely near to the limit (4,5%). It is a well-known issue (earlier papers date back to early '70) that refining process can deplete oil of sterols; the calculation formula for erythrodiol + uvaol relative content (%) presents the sum of sterols at the denominator, this involves that a low content of sterols (the area values of gas-chromatographic peaks is used for this calculation) leads to a false higher content of erythrodiol + uvaol.
Because of this, often refining companies reject or propose a lower market price for those Lampante oils with erythrodiol +uvaol content closely near to the limit, that means to obtain a refined oil with this analytical data exceeding the established limit. Companies in such a case, beside the risk to be penalized because they store an oil out of legislation, are also compelled to mix these refined oils with others, with lower erythrodiol content.
The adoption of the absolute concentration of Erythrodiol content has the purpose to make this analytical parameter a variable quite independent from the sterols content. For this reason, to calculate the concentration of erythrodiol + uvaol content on the basis of percentages is not a solution, as it remains a variable depending on sterols concentration (of course, lower concentration of sterols involves lower values of gas chromatographic peaks areas).
A survey was launched within IOC and several thousands of data were collected for all the categories of olive oils, within at least three years. Some countries sent data, not always in a suitable form to be useful for survey's purposes, while other countries, even if request by IOC Secretariat several times,

never reacts. Considering that the problem arises for refined olive oils and related products (olive oil = mix of refined olive oil and virgin olive oils), my proposal is to adopt a limit for these categories, only, furthermore, considering that often some laboratories erroneously identify as uvaol the peak eluted immediately after erythrodiol that instead is a terpenic alcohols depending on contamination of the TLC band of sterols + erythrodiols from the upper one or, in the past form the presence of derived of sterols depending on strong refining process, and considering the poor contribute of uvaol to the sum, the proposal is also to limit the calculation to erythrodiol, only. Data of refined oils and related mix obtained within the IOC survey are reported in the following tables, as frequency distribution. On this basis, it seems that for refined oils, a limit of 60 mg/kg could include about 100% of samples, while in the case of olive oils (only Greek data were available), the limit should be a bit higher, e.g. 60-80 mg/kg to include 93% of samples.
As a conclusion, my proposal is to add a recall to a footnote in the table of limits for refined and olive oils only, reporting that *"in the case the percentage of the sum of erythrodiol + uvaol should exceed the limit of 4,5%, the oil is considered as fitting the standard if the absolute erythrodiol content expressed as mg/kg is < 75 mg/kg".*

e) Ordinary virgin olive oil (Kamel Ben Ammar from Tunisia)
Presentation about Ordinary virgin olive oil (OVOO) category removed at the Codex Meeting. Tunisia doesn't want to be removed because will become a barrier of trade for countries that still are trading this category. Dr Ben Ammar talked in behalf of the IOC eWG who recommends to maintain the OVOO category in the IOC and Codes standards. The IOC eWG proposes or it is open for discussion to decrease the limit of the sensory defect and acidity of the oil.
Mercedes Fernandez from IOC highlighted and remarked several times about the importance that Codex re-consider the removal because if OVOO becomes not edible for Codex, only IOC will be able to assess this category and incoherence between standards will be raised.
Many other countries didn't agree with this proposal and Australia either.

f) Modifications of oleic acid and palmitic acid in the IOC std
- Oleic acid limit will increase from 83 to 85%
- Palmitic acid limit will decrease from 7.5 to 7%

(2)Other studies in progress

a)Status of the latest studies on the relationship between volatile compounds and sensory defects (Prof. Gallina Toschi).

60 commercial samples chosen EV – 27, VO – 20 and 13 OV. Used SPME-GC-FID/MS to determine volatiles levels for each defect. Protocols being prepared for FID and MS to relate instrumental quantifications with categories/attributes obtained by the panel. This method would be allowed when there is a second test requested from the client to confirm sensory analysis.
The main volatiles identified so far are:
Fusty muddy – octane, ethanol, 3-methyl-1-butanol, propanoic acid, 6-methyl-5-hepten-2-one
Winey/vinegary – acetic acid, ethyl acetate, ethanol
Musty –(E)2-heptenal, 1-octen-3-ol, Propanoic acid
Frostbitten – ethyl propanoate
Rancid – Hexanol, nonanal, (E,E) – 2,4-hexadienol, (E) -2 decenal, pentanoic acid

(3)Study on composition

a)Application of decision trees to C18:3 values greater than 1 for extra virgin olive oils.

Efi Christopoulou(Greece)

The natural variation of fatty acid composition in authentic virgin olive oil is an item under consideration by the IOC e-WG "FATTY ACIDS COMPOSITION" and of the CODEX e-WG "REVISION OF THE STANDARD FOR OLIVE OILS AND OLIVE-POMACE OILS". The issue of fatty acids composition has been a concern for chemists for many many years. This issue is being discussed again and again and no definitive solution was found to address it. It is undeniable fact that olive oils from some countries are out of the official limits regarding fatty acids composition.
The following parameters are very effective in the detection of fraud of virgin olive oil with each high linolenic acid vegetable oil.

✓ for the detection of rapeseed oil: gadoleic acid, erucic acid, brassicasterol, campesterol, apparent β-sitosterol, ap. β-sitosterol/campesterol
✓ for the detection of rapeseed low erucic oil: brassicasterol, campesterol, apparent β-sitosterol, ap. β-sitosterol/campesterol
✓ for the detection of mustard oil: linolenic acid, gadoleic acid, erucic acid, brassicasterol, campesterol, apparent β-sitosterol, ap. β- sitosterol/campesterol
✓ for the detection of soyabean oil: ΔECN42, campesterol, stigmasterol, apparent β-sitosterol, ap. β-sitosterol/campesterol
✓ for the detection of linseed oil: linolenic acid, brassicasterol, campesterol, stigmasterol, apparent β-sitosterol, ap. β- sitosterol/campesterol

The increase of the linolenic limit will have the following consequences :
- The parameter linolenic acid could not be used for the detection of fraud, since the range of its values will be increased. So, the number of the purity criteria will be reduced by one.
- In addition, there will be not an effective parameter for the detection of fraud of an olive oil with de-sterolised rapeseed oil.
- Values of the linolenic acid from 1.0% to 1.5% might have a great influence in the values of the parameter ΔECN42, which is a valuable tool in the detection of fraud and in the effectiveness of the method IOC/T.20/Doc. No 25 in the detection of olive oil fraud.

For the time being, the only solution for facing the deviations regarding linolenic acid limit is the adoption of a decisional tree, effective both in the detection of olive oil fraud and in the deviant virgin olive oils of all olive oil producing countries.
In the decision tree could be used parameters with stricter than the official limits or new parameters of which the effectiveness in the detection of fraud has proven. Such parameters are:

✓ Campesterol
✓ Apparent β-sitosterol
✓ Stigmasterol
✓ Apparent β-sitosterol/campesterol
✓ ECN 42

Taking into account all the before mentioned and recognizing the need to address the difficulties encountered in marketing of authentic olive oils that are high in linolenic acid, the decisional tree for linolenic acid in virgin olive oils could be:

Decisional tree for edible virgin olive oils - 1,00<linolenic acid %≤1,40

Ap. β- sitosterol ≥ 93,5
Stigmasterol ≤ 1,4%
Appar. β-sito /campesterol ≥ 24
All other parameters withing the official limits

The vegetable oils with high linolenic acid content exhibit simultaneously a very high campesterol content (ranging from 15,8% to 38,6%).

However, the campesterol could not be used with lower than 4,0% limit in detecting the addition of these seed oils to virgin olive oils due to the fact that some countries exhibit high campesterol content (e.g. Argentine, Australia).

Dr Ben Ammar (Tunisia) - Overall, I share this proposition with Dr. Christopoulou, however, I do not find it very useful to consider the ratio of apparent b-sitosterol/campesterol ≥ 23.5 since for apparent b-sitosterol ≥ 93.5% and campesterol ≤ 4.0%, the ratio apparent b-sitosterol /camp is always ≥ 23.4; and therefore this 23,5 ratio is not useful and I see that it is more appropriate to replace it with 27.5; so indirectly we force campesterol to be less than 3,4%.

Dr Efi Christopoulou emphasised that the limit remains at <1.0% for EVOO. Decision tree only used when C18:3 is between 1.0 - 1.4 %.

Mercedes Fernandez asked that all countries work with this decision tree and see how oils in their country conform and share with IOC before end of December 2020.

b)Exchange of views on the possible revision of certain parameters in the light of new findings.

South and East of Spain has found that EVOO of high quality and harvested with low Maturity index have deviation in the values of some sterols. Below in the table are the varieties with higher campesterols values of 4% and Total sterols with values lower than 1000 ppm.
Australia has presented this 10 years ago in Picholine and Koroneiki varieties. Greece also highlighted that Koreneiki – over 60% of crop is this variety in Greece. Due to changes in harvest times due to demand, total sterols, campesterol and erythrodiol not conforming in early, mid and late harvest samples.
There is a genetic aspect with low sterol however temperature has the predominant effect. Changing climate is contributing to making this a more serious problem in recent years.
IOC sought agreement to study total sterols to check volumes of affected oil and find the size of the problem. Will study individual sterols at same time to make less work. Deadline set for the end of 2020 for sending samples/data to IOC.

	Studied campaigns	Harvest	Olive degree of ripeness	Total sterols	Campesterol	Other sterols
Picual	6 (2014-2020)	Early	0,7-2,1	771-960 mg/kg	ok	ok
Royal	3 (13/14; 14/15; 16/17)	Early	0,7-2,1	929 - 996 mg/kg	ok	ok
Arbequina	4 (2016-2020)	Early	0,7-2,1	ok	4,1 – 4,3 %	ok
Villalonga	3 (2017-2020)	Pending	Pending	800 - 957 mg/kg	ok	ok

(4)Miscellaneous

a)Accompaniment and support of physico-chemical analysis laboratories.
Fatty acid methyl esters (Dr Cobo)
Incorporate Fatty acid ethyl ester in the Fatty acid methyl ester method. Italy and France asked that a clear note was stated in the method that this would apply **only** if the peaks appeared in the sample. Per example in EVOO most likely these peaks will not be found but in lampante oil yes, so is important to know if the integration needs to be done or not.
Dr Cobo is leading this project, will amend and present to IOC for review.

b)Conclusion on the PT-IOC Pesticide Contaminants Working Group (Dr. Generali).

The Italian National Reference Laboratory for pesticide residues in products of animal origin and commodities with high fat content (NRL-AO), in cooperation with the IOC (International Olive Council) organized a new proficiency test (PT) in olive oil named COIPT-19.
Many European laboratories were invited to participate: NRLs, official and private laboratories. Furthermore, all Italian Official Laboratories (OfLs) involved in the National and European monitoring programs were invited to participate.
The exercise consisted in the determination of six different pesticides in one olive oil sample spiked at a concentration in a definite range (0.050 - 0.350 mg/kg). These pesticides were chosen from a list of twenty-six compounds presented in COIPT-19 Announcement. Forthy laboratories agreed to participate in this PT and all submitted results.
Conclusion: All laboratories submitted results and thirty-five (equal to 88%) analysed all compounds. As consequence of the statistical evaluation of the data five false negative values were calculated: one in the case of Lab 33 for Cypermethrin and Lab 27 for Dimethoate and three false negative values regarding Lab 27, 29, 37 for Phosalone. No false positive z scores have been derived.
The range of Robust RSD values was from 11% to 25 % with a minimum value of 11% for Dimethoate and a maximum value of 25% in the case of Cypermethrin.
Regarding the AZ2 parameter, that expresses the global performance of laboratory, thirty-six out of fourthy laboratories got a good global performance with AZ2 values in the range ≤ 2.0 Two laboratories (29 and 37) obtained a value of just over 3 while 2 other laboratories (27 and 40) achieved a value well above 3.

The majority of participating laboratories supplied mean recoveries in the range 70-120% as specified by the Document SANTE/2017/11813 “Analytical quality control and method validation procedures for pesticide residues analysis in food and feed”.
Furthermore, in some cases the recoveries reported, were not consistent with the results found in the spiked samples when compared to the robust mean values. In the COIPT-19 as in the previous PTs the QuEChERS methodology or methods based on QuEChERS resulted the most used by participants.

c)Presentation of the H2020 "OLEUM" research project (Prof. Gallina Toschi).

- Using software to predict shelf life of olive oil
- Prototype being developed – University of Perugia.
- Parameters being considered, still in development phase.
- Other parameters can be added on later.

d)Information on ISO TC34 SC 11 (Dr F. Lacoste).

Revision of standards

- ISO/CD 6321 - Animal and vegetable fats and oils — Determination of melting point in open capillary tubes (slip point)

New projects

- ISO/WD 18363-4 - Determination of fatty-acid-bound chloropropanediols (MCPDs) and glycidol by GC-MS/MS - Part 4: Method using fast alkaline transesterification and measurement for 2-MCPD, 3-MCPD and glycidol
- ISO/CD 24363 –Determination of Fatty Acid Methyl Esters & Squalene by Gas Chromatography – this is a method proposed by Australia
- ISO/NP 20948 - Animal and vegetable fats and oils — Determination of aflatoxin B1, B2, G1, G2 in vegetable oils by high performance liquid chromatography and immunoaffinity column cleanup
- ISO/PRF TS 22115 - Relative composition of oils and derivatives by capillary gas chromatography (fingerprint method)

- Sensory assessment of oils and fats (meeting in Australia in Feb)
    - Descriptors for sensory analysis – negative vote
    - Preparation of samples for training panels – negative vote

Preliminary Work Item

- ISO/PWI 5132 - Animal and vegetable fats and oils — HPLC analysis of phenolic antioxidants

New publications

- ISO/TS 23942:2020 – Determination of hydroxytyrosol and tyrosol content in extra virgin olive oils - Reverse phase high performance liquid chromatography
- ISO 23349:2020 – Determination of sterols and stanols in foods and dietary supplements containing added phytosterols

Revisions published

- ISO 3657:2020 - Animal and vegetable fats and oils - Determination of saponification value
- ISO 660:2020 - Animal and vegetable fats and oils - Determination of acid value and acidity

e)Presentation about the US standard of identity (J. Profaci).

North American Olive Oil Association (NAOOA) Standard of Identity

Gave history of attempts to develop an olive oil standard in U.S. since 1970's. NAOOA was negotiating with American olive oil producers association (AOOPA) to speak with one voice, and through negotiation to draft an SOI that would represent the greatest possible consesus, but that broke down. Notwithstanding the progress being made and before conducting consumer research, Deoleo S.A, filed a citizen's petition for an SOI in Nov-19 and did so jointly with AOOPA.

NAOOA doesn't agree with Deoleo/AOOPA SOI proposal. NAOOA perceived weaknesses:

- Not based on CODEX as required by FDA regulations
- Provided little to no scientific support for deviations
- Provided no consumer research to establish 'need' for an SOI
- Represented only 25% of the USA market

NAOOA keeps working in its proposal where the key elements are:

- 0.5% for FFA,
- removed PPP's and DAG's from proposal
- harmonised most methods with those of IOC standards on purity
- delete olive oil comprised of' and 'olive pomace oil comprised of' grades and instead required labelling of all component grades in ingredient statements
- added 'safe harbor' based on proof of reasonable shelf life

Next steps for NAOOA:

- Convince FDA to initiate the rulemaking withing 6 months window
- Campaign to get other stakeholders to support the need for an SOI
- Consumer petition signed by over 250 consumers being provided to FDA
- Health expert group letter being prepared
- Scientific expert group letter on need to facilitate revision still needed

Should it agree to initiate rulemaking, FDA will likely take one of the four options:

- publish the proposed NAOOA SOI for public comment
- publish the proposed Deoleo/AOOPA SOI for public comment
- publish a combination of the NAOOA and Deoleo/AOOPA SOIs for public comment
- ask NAOOA, Deoleo and AOOPA to present FDA with a unified SOI

f)Presentation on the application of NMR in the analysis of olive oil (Victor Pidal and Thomas Spengler).

Innovative solution based on Magnetic Resonance for every need in the olive oil value chain presented by Bruker. This technology offers the opportunity to test your olives or olive oil through the whole value chain:
Olive Farmer : Moisture, Oil content and optimum harvest time
Oil mills and cooperatives: Monitor oxidation and shelf life prediction
Distribution: ID check incoming trucks, screening of origin, screening of grade (EVOO, VOO, etc)
Commercial labs: Verification of country of origin, screening of grades, legal blends, antioxidants, etc

g) Review alpha tocopherols limits
Proposal: to review the tocopherols limit to increase to 300 ppm.
Alpha-tocopherol in refined oils currently is allowed up to 200ppm. The proposal of this study is to increase limit to 300ppm alpha tocopherol in refined oil. Need to identify if the limit is the addition of tocopherol to sample or added tocopherol + natural tocopherol in sample.
Proposal to also carry out a study of refined and pomace oils to determine the levels of tocopherols and set a new limit based on those results. IOC will aim to decide in the next meeting.

(9)Priorities for future projects

(10)Other issues and closing of the meeting