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

Date: 24–25 June 2013
(09.30 – 15.00 hours)

Place: International Olive Council
Príncipe de Vergara, 154
Madrid

MEETING REPORT

The Executive Secretariat of the International Olive Council (IOC) convened a meeting of the chemists officially designated by the IOC member countries to cooperate in studying methods of analysis for olive oils and olive pomace oils. The meeting took place on 24 and 25 June at IOC headquarters in Madrid (it closed at 13.30 hours on the 25th, earlier than scheduled, because it coincided with the IOC *Mario Solinas* 2013 prize giving ceremony).

The Executive Secretariat was represented by Executive Director Jean-Louis Barjol, Deputy Director Ammar Assabah, and 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 explained that the delay in closing the 100th session of the Council and in adopting the budget for 2013 had in turn delayed the chemists' meeting, which was restricted solely to the official representatives of each member country; consequently, no observers were present.

After running through the business on the meeting agenda, the Executive Director urged IOC Members to participate more actively in Codex meetings as well as in the IOC survey of oils exhibiting off-limit values for specific parameters. He also commented on the Executive Secretariat's proposal to the World Customs Organisation (WCO) regarding the definition of the lampante category and thanked the chemists for their input.

The Executive Secretariat complimented the experts on the excellent job they were doing while stressing it was important for them to deliver any meeting documentation at least one month in advance to allow it to be circulated and properly examined beforehand. It reminded participants that a private user group had been created on the IOC website to facilitate password-enabled access to the documentation for each meeting.

The list of attendees is given in the document referenced T. 20/ Doc. No 67-4.

The following business listed on the provisional agenda (T.20/Doc. No 67-1) was discussed at the meeting:

1. Adoption of the report of the meeting held on 9–10 October 2012 (T.20/Doc. No 66-3)

The expert group adopted the report on their previous meeting after incorporating the following change under item 2 (sixth paragraph of the section on stigmastadienes on page 5), at the suggestion of Carlo Mariani:

- Replace “(squalene is followed by three peaks corresponding to hydroxyperoxide fatty acids)” by “(squalene is followed by three peaks corresponding to squalene epoxide)”.

2. Update on the documents adopted at the 100th session of the Council of Members

The Executive Secretariat reported on the documents proposed by the expert group which had been adopted at the 100th session of the Council on 27 May 2013 (**Annex 1**):

- **Method for the determination of sterols and triterpene alcohols by capillary column gas chromatography** (COI/T.20/Doc. No 30): This method was provisionally adopted at the 97th session of the Council, held in November 2009. The experts designated by the member countries had stressed the need to delete the method for the determination of erythrodiol and uvaol (IUPAC No 2.431, "Determination of the erythrodiol content") by combining it with the sterols method (COI/T.20/Doc. No 10, "Determination of the composition and content of sterols by capillary column gas chromatography") in order to eliminate potential commercial problems, and had put forward a unanimous proposal to the Technical Committee to present the method for definitive adoption at the 99th session of the Council of Members. In line with the agreement reached at the 17th extraordinary session of the Council, the method is applied to olive oils since 1 January 2012 and to olive pomace oils since 1 January 2013.

In addition, ISO Subcommittee TC 34/SC11 plans to adopt this method because it considers it to be more reliable than the existing methods.

The Council of Members adopted all the business submitted to it for approval, listed below together with relevant comments:

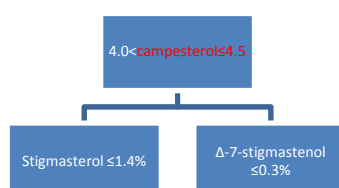
- **Definitive adoption of the global method for the detection of extraneous oils** in olive oil: entry into force as of 1 January 2014.
- **Reduction of the myristic acid limit to 0.03%:** all the limits for the other fatty acids are given to two decimal places.
- **Reduction of the stigmastadiene limit to 0.05 mg/kg:** for extra virgin and virgin olive oils.
- **Reduction of the wax limit (C42+C44+C46) to 150 mg/kg:** for extra virgin and virgin olive oils.
- **Revision of the method for the organoleptic assessment of virgin olive oil.**
- **Approval of the list of laboratories and tasting panels awarded IOC recognition for 2012/13.**
- **Calculation of solely ethyl esters instead of total alkyl esters:** approval of following limits and application timing:

≤ 40 mg/kg in 2013/14
 ≤ 35 mg/kg in 2014/15
 ≤ 30 mg/kg from the 2015/16 crop year
FAEE + FAME < 75 mg/kg during the 2012/13 crop year

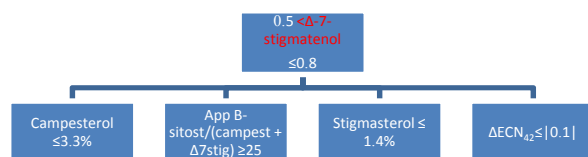
- **Composition of olive oils obtained from various varieties:** it was decided to adopt **three decision trees for campesterol (with values between 4.0 and 4.5%) and delta-7-stigmastenol (with values between 0.5 and 0.8%) in virgin and extra virgin olive oils, and for delta-7-stigmastenol in olive pomace oils (with values between 0.5 and 0.7%).**

The rest of the parameters must comply with the limits fixed in the standard, particularly the new stigmastadiene limit.

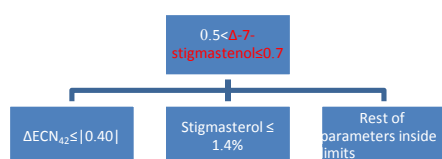
Campesterol decision tree for virgin and extra virgin olive oils:



Delta-7-stigmastenol decision tree for extra virgin and virgin olive oils:



Delta-7-stigmastenol decision tree for crude and refined olive pomace oils:



3. Conclusions of the 23rd meeting of the Codex Committee on Fats and Oils (CCFO)

The Executive Secretariat reported on the CCFO meeting held in Malaysia from 25 February to 1 March 2013 (**Annex 2**). The experts unanimously decided to request the United States and Australia to provide off-limit samples, with a view to the next meeting scheduled for February 2015.

Kamel Ben Ammar, who had been present at the CCFO meeting, remarked on the importance attached to the IOC survey and to the joint position of the IOC member countries.

Arturo Cert and Carlo Mariani were of the opinion that the limits should not be raised more than necessary because otherwise the limit for apparent β -sitosterol would not be met. They also highlighted the usefulness of campesterol as the sole parameter for detecting avocado oil, which is being produced extensively in the southern hemisphere, given that it does not contain stigmasterol which is therefore of no use for its detection.

4. Study and update of the methods of analysis cited in the IOC trade standard: conclusions of the restricted group on testing method optimisation

When it was decided to set up this restricted working group in April 2011 it was agreed that its chief tasks would be to examine proposals in the light of priorities and to arrange the necessary ring tests.

At its earlier meeting in October 2012, the group established its working priorities for the optimisation of methods of analysis. When outlining priorities at the meeting, Florence Lacoste insisted on:

- studying the method for the determination of unsaponified stigmastadiene with more than 3 g of silica (direct method);
- applying the old method for the determination of FAAEs to determine stigmastadienes and circulating a training sample with a content on the borderline of the limit (0.05mg/kg) together with chromatograms (column method, 15 g, unsaponified – see item 5);
- revising the current method for phenols determination. In the experts' opinion, this method does not give real values for hydroxytyrosol and tyrosol, nor is the title of the method very appropriate. The term biophenols should be replaced by a more suitable term such as phenolic compounds. At the next meeting, Lanfranco Conte will present a hydrolysis method with a 1,3-hydroxytyrosol standard. The standard used for the Folin colorimetric method (1,2-hydroxypropanol) will have to be found. Diego Luis García said it would be very helpful to add some chromatograms of old oils.

5. Current status of the testing methods available for the detection of olive oil adulteration; method for the determination of triglycerides

After hearing the inconclusive results of the diglycerides ring test conducted in 2011 on three different methods (NGD–SSIG, DGF–ISO and SPE–IG), the experts had agreed at previous meetings that triglycerides determination was of potential interest for the detection of certain types of adulteration given that the NGD method permitted determination not only of diglycerides but also of triglycerides.

At those meetings the experts had agreed it was necessary to run a ring test to validate the determination of triglycerides. Mr Mariani had explained that this method had undergone extensive testing. Formerly, C57 was used as the internal standard. However, it appeared to raise triglyceride content due to thermal degradation. Conversely, C60 triglyceride degradation did not exceed 15%, which meant they could be used for this kind of analysis. The method appeared to permit detection of high oleic sunflower oil, palm olein, groundnut oil, avocado oil, etc. Sensitivity was 1% in the case of groundnut oil and C58 recorded an enormous increase. Mr Mariani had further commented that the limits could range from 0.15 for C48 to 0.30–0.32 for C58.

At the same meetings, Wenceslao Moreda had concurred with the rest of the experts that it would not be advisable to fix a limit for diglycerides because extra virgin olive oil reaches thermodynamic equilibrium after a year and this parameter would be of no use. He had added that triglyceride determination offered interesting possibilities although it was limited by certain factors such as thermal degradation. He considered further testing to be necessary. Much work

remained to be done to establish a limit, particularly for palm olein, the different fractions of which would have to be analysed.

Mr Cert had proposed first organising a ring test to investigate potential degradation and any other relevant aspect and then fixing definitive limits on the basis of the proposal put forward. He had added that the method enabled detection of certain types of oil (palm, high oleic, etc.) but only when they were added singly; if more than one extraneous oil was added, the method was not effective.

In May 2013, the Council of Members definitively adopted this *Global method for the determination of extraneous oils* (COI T.20/Doc. No 25) for entry into force as of 1 January 2014; until then, it had been provisionally adopted since 2006. Following this adoption, Mr Moreda presented data on the application of the global method to 29 samples from Argentina (**Annex 3**). As regards its legal validity, he proposed that it be a prolongation of the ΔECN_{42} method, i.e. if an oil complies with ΔECN_{42} , it complies with the global method; if not, it has to be investigated.

Mr Moreda announced that he would re-circulate the method after including the precision data obtained under the MEDEO project and stating figures to two decimal places in the revised software.

At the proposal of the experts, the Executive Secretariat has included this method for application in the annual laboratory proficiency check test for IOC recognition for the 2013/14 crop year.

In addition, José Garcia-Mesa (IFAPA) presented a study conducted by his team on the determination of β -carotene isomers (**Annex 4**).

ITERG and the *Instituto de la Grasa* have carried out research on this subject. Although they have not published results, the first has mentioned the research in its activity report and the second in a thesis. Deodorisation temperature has been observed to be the most critical issue while the *cis/trans* ratio does not appear to be significant. The *cis* form is less stable than the *trans* form and reversion may occur, thus aggravating matters. Season-to-season differences were also mentioned in addition to the difficulty of finding a marker for oils deodorised at low temperatures.

6. Survey of ethyl ester data and of the changes in this parameter

The experts agreed that this method had helped to enhance oil quality in general. The Executive Secretariat requested countries for official data on fatty acid methyl esters (FAMES) and fatty acid ethyl esters (FAEEs).

Since most of the data supplied reported low EE content, the experts had submitted a proposal to the IOC Council of Members, which was accepted, to recalculate the limit solely for FAEEs and to propose a gradual reduction while

research continues underway in several countries (**Annex 5**) to pinpoint the changes in this parameter.

Mr Conte presented data on the changes in FAEEs. The samples used were borderline oils that were not optimal extra virgin olive oils and had been analysed to determine the FAEEs and methanol/ethanol; they had also undergone organoleptic assessment. Greater variability was observed in retail oils. The experts agreed that filtered oils were more stable. Participants were also informed about other studies carried out in Perugia, Greece, France (*Centre Technique de l'Olivier*) and Morocco.

Maurizio Servili suggested it would be advisable to establish a common protocol. It was pointed out that such a protocol had already been developed in Spain and it was made available to participants.

Mr Moreda commented that he had found no explanation for the increase in FAEEs or for the decrease in ethanol content noted in some cases.

7. Presentation of the final report on the determination of pesticides (COI PT-12)

The test coordinator, Tiziana Generali, presented the final report (**Annex 6**) on the outcome of proficiency test COI PT-12 in which 40 laboratories had participated although only 14 had analysed all the compounds. The samples for proficiency test COI PT-13 will be shipped shortly.

José Ramón García Hierro said that the results were better than those obtained in the European collaborative test and stressed it was important to allow for false positive and false negatives.

The Executive Director informed the group about the situation in the United States where the list of permitted pesticides is different and much shorter than in the European Union (EC Regulation 396, Annex 6).

It was mentioned that in the EU the limit for olive oils is obtained by applying a transfer factor of 5 to the limits for authorised substances for olives. The Executive Secretariat asked for information about the situation in non-EU countries.

8. Organisation of collaborative tests in 2013: determination of triglycerides, determination of mineral oils

Angelo Faberi, the coordinator of the collaborative test on the determination of triglycerides, reported on the arrangements for the test which had been agreed upon at the previous meeting. He announced that the samples would be shipped to the participants listed in **Annex 7** before the summer together with the stigmastadiene training sample. The test results will be presented at the next chemists' meeting.

Ms Lacoste informed participants that the ring-test organised by ISO Subcommittee TC/34 SC11, for which she was the coordinator, would be presented at the next meeting.

9. Study on the changes in quality parameters during storage

The Executive Secretariat reported on the discussions of the labelling working group (**Annex 8**), which had met for the second time on 12 November 2012 to look into the revision of section 10 of the trade standard. It informed participants that the WG had agreed that the questions of the best before date and storage conditions should be referred to the chemists' group and that the conclusions of the studies on the changes in quality parameters during storage should be taken into account.

10. Current status of standards relating to olive oils and olive pomace oils

The Executive Secretariat reported that new standards were getting ready for adoption in both non-member (South Africa, India) and member countries (Israel, Iran and Turkey). It asked the member countries concerned for working drafts of the new standards and offered them any assistance they might need from the IOC (**Annex 9**).

The Executive Secretariat next informed the group on the work undertaken by the WCO which had requested the IOC to draft a definition of lampante virgin olive oil (**Annex 10**) for possible inclusion in order to thus make a distinction between lampante oil and the current definition of virgin olive oils in the Codex standard.

After lengthy comment, it was agreed that Messrs Cert and Moreda would send the Executive Secretariat a revised wording as soon as possible.

Next, the Executive Secretariat presented the data received from Spain, France, Greece, Italy and Portugal regarding free acidity, peroxide value, K_{270} , K_{232} and waxes in response to the Note Verbale it had circulated to all the member countries requesting data for the possible review of certain parameters proposed by some delegations (**Annex 11**). The Executive Secretariat urged those countries that had not yet supplied data to do so before the next meeting in order to give the experts time to analyse them.

Commenting on the wax data, Mr Mariani pointed out that high C46 values in extra virgin olive oils increased the possibility of adding second-centrifugation oils. He also said that the ISO method was not valid for olive oils.

Luciana Di Giacinto proposed lowering the wax limit for extra virgin olive oils to 100 mg/kg.

France proposed fixing values of:

- $\leq 0.5\%$ for the acidity of extra virgin olive oils
- ≤ 15 meq/kg for the peroxide value of virgin olive oils
- $K_{270} \leq 0.20$ for virgin olive oils.

Spain proposed:

- $\leq 0.5\%$ for the acidity of extra virgin olive oils;
- ≤ 18 meq/kg for the peroxide value of virgin olive oils;
- $K_{270} \leq 0.20$ and $K_{232} \leq 2.20$.

The Executive Secretariat awaits further proposals and data.

11. Priorities and future work on olive oil chemistry

The Executive Director reminded the experts that it was important to forward any written comments as soon as possible after receiving the report in order for them to be circulated.

In view of the many existing topics being investigated, the chief priority identified by the experts was a proposal to provide training in physico-chemical testing methods for the member countries.

Mr Faberi mentioned the traceability study underway in Italy, which he considered relevant and which had been presented to the group of experts. He said he would forward the revised method for consideration at the next chemists' meeting.

12. Seminar on chromatogram interpretation

At the previous meeting of the group, a seminar or workshop on chromatogram interpretation had been scheduled. This had been included in the activity programme of the Executive Secretariat to coincide with a chemists' meeting. At the time, the Executive Secretariat had welcomed the proposal and invitation of the *Società italiana degli oli e dei grassi* to hold this theoretical/practical seminar at a laboratory in Italy (Udine or Milan) at which the experts who developed the methods would be the guest lecturers. This could become a regular activity, focused on waxes, stigmastadienes, alkyl esters, sterols, TG, etc.

The *Instituto de la Grasa* also expressed interest in holding such an event at its centre.

A concrete proposal should be forwarded by early October for consideration and submission to the Council in November 2013 with a view to holding the seminar in 2014.

13. Other business

The Executive Secretariat invited the experts to submit written comments for the next meeting of the Technical Committee. It also reminded them that it was important to present all the documentation by the one-month deadline set by the Council to allow the group to examine it in advance and so ensure more effective discussions at meetings.

The Executive Director and Sandrine Valentin informed the experts on the authentication seminar held at IOC headquarters on 10 and 11 June 2013, which had been co-organised by the Directorate General for Agriculture and the Joint Research Centre with the participation of the IOC. The topics (adulteration and deodorisation) for the call for proposals for research projects at the end of 2013 were identified at the seminar, the proceedings of which will be posted shortly on the websites of the three organisations.

By way of a conclusion, the Executive Director invited the experts to attend the prize giving ceremony and reception for the IOC *Mario Solinas* Quality Award 2013 and called upon them to attend the next meeting, scheduled at IOC headquarters on 8 and 9 October 2013.
