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

Madrid, 9–10 October 2012

MEETING REPORT

The Executive Secretariat of the International Olive Council (IOC) convened a meeting of the chemists designated by the IOC member countries to cooperate in studying methods of analysis for olive oils and olive pomace oils. The meeting took place from 09.30 to 15.00 hours at the IOC headquarters in Madrid on 9 and 10 October 2012.

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

More than 14 countries represented IOC Members on the first day of the meeting. Official observer representatives from member country associations, the Advisory Committee, non-member countries (USA, Australia) and standards agencies (ISO, AOCS, Codex) were invited to attend on the second day.

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 in order to promote trade, prevent fraud and protect consumers.

A representative of the United States Department of Agriculture (USDA) attended for the first time as an official observer. Observers representing two associations, ASOLIVA and UNAPROL, were also present. The representative designated by the Advisory Committee was not able to attend because there was no provision for IOC defrayal of his travel expenses. The Executive Secretariat has referred this matter to the Council with a view to future meetings. The representative of Brazil was also invited to take part but was unable to do so.

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 anomalous values for specific parameters in preparation for the meeting scheduled in February 2013. He added that since the IOC held observer status at the Codex Alimentarius, it was for the the IOC Members who were also Codex Members to take a political stance at Codex meetings.

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.

Most of the experts invited to attend were able to do so. The list of attendees is given in the document referenced T. 20/ Doc. No 66-4.

The following business listed on the provisional agenda (T.20/Doc. No 66-1) was discussed at the meeting:

**1. Adoption of the report of the meeting held on 22–23 March 2012 (T.20/Doc. No 66-3)**

The Executive Secretariat took note of the comments made by the experts after the release of the report on the last meeting held on 22–23 March 2012 (ref. T.20/Doc. No 65-3).

This report was then adopted complete with the amendments made by the experts.

• **Scrutiny of the documents intended for submission to the Council of Members for adoption at its 100th session**

At the 17th extraordinary session of the International Olive Council, Members decided to adopt the *Method for the determination of sterols and triterpene alcohols by capillary column gas chromatography* for application to olive oils as of 1 January 2012. It was also decided to study the delta-7-stigmastenol content of Tunisian olive pomace oils and restrictive parameters (C18:3, campesterol and  $\Delta$ ECN<sub>42</sub>) according to the proposal framed by Tunisia (see item 7 of this report), and to apply the method to olive pomace oils as of 1 January 2013 after testing the samples shipped by Tunisia.

After the analysis of the samples, the following decision tree was proposed:

For olive pomace oils (all categories: crude olive pomace oil, refined olive pomace oil and olive pomace oil):

**delta-7-stigmastenol value between 0.5% and 0.7% if:**

**delta ECN 42  $\leq$  |0.4| % and stigmasterol  $\leq$  1.4%**

The IOC trade standard will be revised to incorporate this method for olive oils and olive pomace oils (**Annex 1**).

## **2. Study and update of the methods of analysis included in the IOC trade standard**

The coordinator, Ms Florence Lacoste, outlined the results obtained in the collaborative study undertaken by 20 laboratories in July 2012 on alkyl esters, waxes and stigmastadienes (Doc. 5.3 - **Annex 2**). The IOC supplied the laboratories with six oils and the accompanying data. Two of the oils tested had been entered for the Mario Solinas Quality Award and had been produced by the same producer in Spain but in different seasons (2009 and 2012). This was meant to allow appraisal of possible variations over time (few changes were noted in the two oils).

The raw data showed:

- wide variation in the samples with low levels of methyl esters (samples 5 and 6 – see page 2) ;
- wide variation in the samples with low levels of ethyl esters (page 4);
- more reasonable variation in the samples with a content of 50 mg/kg.

The statistical data revealed the following:

### Methyl esters:

- Sample 1 (page 13): acceptable results (50±25 ppm; Horrat value = 1.96)
- Sample 2 (page 14): unacceptable results (14±14 ppm; Horrat value = 3.39)
- Sample 3 (page 15): 59±45 ppm; Horrat value = 3.16

### Ethyl esters:

- Sample 1 (page 19): 50±25 ppm; Horrat value = 1
- Sample 2 (page 20): 21±17 ppm; Horrat value = 1.8. With this uncertainty, there would a a risk if the limit were to be lowered
- Sample 3 (page 21): 87±60 ppm; Horrat value = 3.05 (this indicates a problem)
- Sample 4 (page 22): satisfactory results (151±46 ppm; Horrat value = 1.5)
- Samples 5 + 6 (pages 23 and 24): problems at low values (Horrat value = 5)

Although the results were somewhat better for the sums of the methyl+ethyl esters, there was still a problem overall.

The laboratories had problems with the alkyl esters (blank, eluted triglycerides, etc) which need to be resolved in further testing of the method. The performance of the method is similar to that of the IOC method but the borderline values were higher. The data for the FAEE/FAME ratio have been disregarded because this ratio is to be deleted from the method.

### **Waxes:**

The statistical data for the waxes are presented from page 37 of the collaborative study report:

- Sample 1 (page 37): satisfactory results but 3 laboratories were eliminated
- Sample 2 (page 38): this sample caused problems (2 laboratories were eliminated and the Horrat value >2)
- Sample 3 (page 39): satisfactory results but 3 laboratories were eliminated
- Sample 4 (page 40): wide variation in results (200–750 ppm; mean 564±400; Horrat value = 4) indicating possible problems with integration, etc.
- Samples 5 + 6 with contents < 50 (pages 41 and 42): Horrat value = 2.8 and 3.2, respectively

**Conclusion:** The laboratories did not report any specific problem at the current limits specified in the IOC standard; however, a risk could arise if the limit were to be dropped.

### **Stigmastadienes:**

The data were not statistically processed; a summary is available on page 12 of the collaborative study report. Problems were encountered in detection sensitivity/blank chromatograms at low contents. There were also problems with the quantity or quality of the elution solvent. The method is not adapted to the detection of low stigmastadiene contents.

The experts had the following comments to make in the light of these results:

Mr Carlo Mariani pointed out that the results obtained in the previous restricted ring test had been relatively good. He did not understand, therefore, why problems had occurred now. Moreover, the method contained a note stating that that quantity of solvent had to be adjusted according to the type of material used.

Mr Arturo Cert said that as far as the alkyl esters were concerned, the ratios were sometimes very different due to interference from the silica gel (which contains hydrocarbons from the air used in its production), contamination from the cotton used in the column and contamination from the hexane used for the HPLC, which made it necessary to redistill the solvent. In the case of the waxes, there was no possibility of improving the precision at contents < 50 mg/kg, because it was already the result of the addition of four rounded peaks which in turn were the aggregate of smaller peaks. The waxes sometimes caused problems in repeatability conditions, which gave rise to differing data. This was perhaps due to the action of a

completely saturated acid. Consequently, it would be advisable to use a more polar unsaturated acid. As far as the stigmastadienes were concerned, the detection limit was close to 0.01; hence, there might be problems if the limit was dropped too low.

Mr Mariani explained that distilled solvent gave better results. Heating the silica to more than 400 °C made it possible to eliminate most of the organic substances.

Ms Nadia Maata commented on the wax content of sample 4, for which her laboratory had reported a low content, and enquired about the source of this anomaly. Mr Mariani said it was necessary to check the elution and that Sudan dye was the best for this purpose (squalene is followed by three peaks corresponding to hydroxyperoxide fatty acids).

Mr Wenceslao Moreda urged pursuing research on this method because it uses less solvent.

Ms Lacoste highlighted the two questions at issue. If the aim was to lower the limits, the existing method would have to be used because the draft method only worked at the current limits. It was a question of either changing the limits or changing the method. One solution would be to delete all reference to the stigmastadienes from the draft method and to propose it for provisional application but chromatograms and interpretations would have to be inserted, above all for alkyl esters.

Mr Lanfranco Conte agreed with what the previous two experts had just said. Before holding a further collaborative test, a meeting should be held to perform an HACCP analysis of the method, section by section, in order to make it work properly.

Mr José Ramón García Hierro said it was necessary to abide by the regulations. The method could not be changed without demonstrating the equivalence of the two methods.

Ms Efi Christopoulou said that, in principle, she disagreed with adopting the method provisionally because the same situation could occur as with the sterols method (1 band/2 bands), leading to distortion of the results.

Mr Denis Ollivier stressed that the adoption of the method could give rise to problems if the stigmastadiene limit were to be lowered to 0.05 ppm because it does not work properly at low stigmastadiene contents.

After wide ranging discussion, Ms Lacoste said that the method could be adopted without the stigmastadienes.

The experts unanimously agreed to place a proposal before the Technical Committee **for the provisional adoption of the method for the determination of alkyl esters and waxes (excluding stigmastadienes) using 3 g of silica (Annex 3).**

**3. Study on the limits specified in the IOC standard for fatty acid methyl esters and ethyl esters (Annex 3)**

The Executive Secretariat received few answers to its Note Verbale requesting data on this question. At their previous meeting, the experts had agreed to lower the limit for ethyl ester content and to take solely this parameter into consideration pending the outcome of several studies under way on oil ageing in order to have a better understanding of the variations in alkyl esters. After lengthy debate, the experts representing the member countries put this matter to a vote in the absence of a consensus: seven countries voted for an ethyl ester limit of 40 mg/kg and seven for a limit of 30 mg/kg. A limit of 40 mg/kg for ethyl esters would coincide with 90% of the figures notified by Italy, which equates with an alkyl ester limit of 50 mg/kg, which had already been proposed.

Mr Conte and Mr Moreda stated the logical step would be to lower the ethyl ester limit and to disregard the methyl esters. However, it would be wise to wait to see the outcome of several ongoing shelf life surveys to have a better idea of the ways in which alkyl esters can change.

Mr Angelo Faberi presented results forwarded by Italy and explained that the data showed that the limit was too high. Solely ethyl esters should be taken into consideration.

In the opinion of Mr Maurizio Servili, the experts should not backtrack to avoid adopting new limits.

Mr Cert pointed out that the limit for extra virgin olive oils had been arrived at through correlation with the sensory evaluation. If the limit was lowered, oils might be demoted to other categories on the grounds of their alkyl ester content and not of their organoleptic assessment.

Mr Mariani outlined the background to the development of these criteria. Initially a limit of 50 ppm had been proposed; this had later been raised to 75 ppm and a FAEE/FAME ratio had been inserted. According to the data obtained for extra virgin olive oils sold in Italy, the limit could be lowered to 50 ppm.

Ms Christopoulou pointed out that this quality parameter also served to detect deodorised oils and proposed decreasing this limit to 50 ppm. After a general discussion on changing the limits, she reported that a study conducted on more than 200 Greek samples had revealed a poor correlation of 0.4 (compared with an optimum of 0.99) between organoleptic appraisal (median of defects) and alkyl ester content.

Mr Ollivier explained that if an oil was graded as extra virgin in a sensory assessment but considered borderline at 75ppm when tested for alkyl esters, the opinion of the tasting panel could be invalidated if a second expert opinion were requested. Consequently, lowering the limit to 50 ppm would reinforce sensory testing. He added that oils that were initially extra virgin olive oils did not

necessarily remain so for the whole of their lifetime and could evolve into virgin olive oils. Consequently, it was not necessarily useful to await the completion of the shelf life studies to take a decision on this matter. Although marketers might not wish to do so, virgin olive oils can be sold at retail.

Mr Servili stated that methyl esters developed naturally whereas ethyl esters were the result of fermentation. The survey conducted in Italy had examined retail oils in which the parameters evolved normally.

Mr García Hierro and Mr Moreda asked for the limit not to be lowered for the time being, but to eliminate the second option between 75 and 150 ppm and to delete the FAEE/FAME ratio. They considered it necessary to study the changes that occur in oils before taking a decision, for instance content is higher in unfiltered oils than in filtered oils.

Mr Ariel Buedo said that Argentine oils complied with the current limits; however, if these were changed and the FAEE/FAME ratio was deleted, they might have problems.

Ms Christopoulou proposed fixing the alkyl ester limit at 50 ppm. If the content was higher, the FAEE/FAME ratio would have to be = 1.

Mr Cert agreed with this proposal and insisted on taking into account both the physico-chemical parameters and sensory appraisal.

When the ethyl ester limit was put to the vote, the result was: seven countries in favour of 40 mg/kg (France, Argentina, Israel, Portugal, Spain, Morocco and Algeria) and seven in favour of 30 mg/kg (Italy, Slovenia, Tunisia, Egypt, Greece, Iran and Turkey).

Ms Lacoste stated that an ethyl ester limit of 40 mg/kg would coincide with 90% of the figures notified by Italy. This equated with an alkyl ester limit of 50 mg/kg, which had already been proposed.

Mr Cert pointed out that in the case of the EU, the amendment of the regulation would have to be envisaged for application as of October 2013.

After a discussion among the experts from Italy, Spain and France, Mr Conte proposed the following course of action with the agreement of the other experts:

In the light of the technology available to produce extra virgin olive oil, a limit of **40 mg/kg could be adopted for the time being for ethyl esters. This would then be lowered in 2013–2014. Between 2014 and 2016 it would be fixed at 35 mg/kg, and then at 30 mg/kg in 2016 to allow economic operators to adapt gradually.** These limits could change (come into effect earlier) depending on the results available by the scheduled dates. The countries pledged to supply data.

#### **4. Current status of the methods of analysis available to detect adulterations of olive oil and possible revision of certain limits: myristic acid, stigmastadienes**

As a feature of its cooperation with standards agencies, the IOC strives to harmonise the testing methods cited in the trade standard in order to safeguard the quality of olive oil products, to stimulate trade and to prevent commercial obstacles.

Mr Faberi presented the results of the collaborative test conducted on the determination of triglycerides (**Annex 4, Doc. 5.2**). A very small number of laboratories took part in the test, for which five samples were furnished:

- Sample 1: extra virgin olive oil
- Sample 2: olive oil + avocado oil + palm oil
- Sample 3: extra virgin olive oil
- Sample 4: olive oil + palm oil + avocado oil + sunflower oil
- Sample 5: old olive oil + refined oil

The C48 percentages were low for the extra virgin olive oils. Testing for C48 and C58 proved to be significant for the detection of the addition of sunflower oil and avocado oil.

Determination of diglycerides: the 1-2:1-3 ratio is important for extra virgin olive oils (samples 1 and 3) and fluctuated according to laboratory and sample. With regard to the method, the columns used were 5 m and 15 m (too long, risk of degradation). Laboratories were allowed to use a PTV or on-column injector; most used the second. The importance of using a short 5-m column should be mentioned in the method. Mr Faberi said he had used a 4.65-m column and mentioned that the solubility of the internal standard had posed a problem. The ITERG had used toluene to solubilise this standard.

It was decided to revise the method and to hold a further ring test as of January 2013, coordinated by Mr Faberi. The list of the laboratories that will participate in principle is appended in **Annex 8**.

#### **Possibility of proposing the global method to the Council: study on oils of different origins mixed with extraneous oils (Annex 5).**

Mr Moreda presented document 4.6 reporting the detection ability of the method depending on the type of extraneous oil and the varieties: sunflower oil (1–5%), hazelnut oil (2.5–12%) and corn oil (1–2%). In Table 2 of his report he gave the data for oils produced from varieties of olive from different countries.

Mr Buedo commented that when statistically processed, the results for Argentine oils (document 7.1) showed anomalies between delta ECN<sub>42</sub> and the global method.



Ms Pierangela Rovellini did not consider it useful to apply this technique to oils exhibiting deviations from the official limits. She also questioned the use of solely propionitrile in the Spanish method and proposed a revision of the global method (document 4.10.4, also including the Exempla program).

Except for the expert from Argentina, the experts agreed with the definitive adoption of the provisional global method while the Italian method would be a line for further improvement.

Mr Moreda pointed out that the precision data were specified for the global method (provisionally adopted since 2007) but not for the part of the Italian method. This would raise problems as regards definitive adoption if these data had to be included

The Executive Secretariat therefore asked Ms Rovellini and Mr Moreda to team together in putting the finishing touches to the method.

#### **Reduction of the stigmastadiene limit (Annex 6)**

Following the proposal to lower the stigmastadiene limit to 0.05 ppm, a pertinent Note Verbale was addressed to the Members. No negative feedback was received.

Mr Michel Blanc said that the current standard referred to a value of 0.01 at a coefficient of variation (reproducibility) of 98%. Consequently, statistical data would be needed for a level of 0.05 ppm.

Ms Lacoste pointed out that according to the IOC standard, the limit for each criterion included the precision values of the attendant method; however, these had not been determined for this level. She asked for a sample with this content to be included in the next proficiency test for IOC recognition.

Mr Cert asked whether the method was capable of determining a stigmastadiene content of 0.05 ppm.

Mr Faberi and Mr Conte reported that numerous analyses of oils with very low contents had demonstrated that the limit could be lowered to this level. This was very important for subsequently putting in place the decision trees.

Mr García Hierro also agreed with dropping the stigmastadiene limit to 0.05 mg/kg.

**Consensus was reached on lowering the limit for stigmastadiene content to 0.05 mg/kg and on including a sample (with a content of 0.04 mg/kg) in the next laboratory proficiency check test organised in 2013 to earn for IOC recognition for 2013/14.**

### **Reduction of the wax limit (Annex 7)**

Mr Moreda presented document 4.5. He reported that tests had been conducted in which C40 had been disregarded and he proposed a limit of 125 mg/kg. However, Arbequina oils had been found to have higher wax contents. Consequently, to avoid problems, the limit would have to be raised to 150 mg/kg.

Mr Mariani highlighted the relevance of this study. He acknowledged that some oils would exceed the limit and that there were certainly problems with the chromatographic separation since most of the laboratories did not manage to separate phenyl benate from C40. Higher wax levels were observed at C44 and C46 in Arbequina oils with high contents of C40 waxes and phytyl close to C42. Usually, C46 was not found in good quality oils or only at very small levels. He stressed the importance of chromatographic separation, which influenced C40 separation, as well as of correct peak interpretation and said that an interlaboratory test would be necessary.

Mr Cert said that although Mr Mariani's research was very interesting, a ring test was needed. He therefore proposed adopting Mr Moreda's proposal (C42–C46) and working further on the method. The EU decision tree allowed determination of erythrodiol content first before testing for wax content, which might allow a larger proportion of Argentine oils to be considered extra virgin. He added that this determination was not very important for extra virgin olive oils.

Ms Lacoste reported that the ring test she had organised demonstrated that some of the laboratories did not achieve the level of chromatographic separation requested by Mr Mariani. She would use these test data to calculate the reliability of the two alternatives: C40–C46 and C42–C 46.

Mr Faberi pointed out that the precision data would have to be recalculated if the method was changed (C42– C46). He was seconded on this point by Mr Kamel Ben Ammar who asked for a ring test to be conducted to compare the precision values.

Mr Buedo said that the application of the new limits would not really improve matters for Argentine oils. He therefore urged caution over the limits and proposed taking into account the erythrodiol + uvaol values.

Mr Moreda pointed out that the proposal benefited oils produced in Argentina irrespective of the limit.

Ms Lacoste reported on the ring test undertaken that year (method using 3 g of silica) to compare the precision data depending on the sum of the waxes taken into account, i.e. from C40 to C46 or from C42 to C46. The test revealed that the reproducibility was similar in both cases. Hence, the precision and accuracy were equivalent irrespective of whether the sum of the C40–46 or C42–46 waxes was calculated and the Horrat values were comparable. She clarified that the test had

been carried out to compare the mathematical approaches and that the precision data stated in the official method had not been altered. This remained to be done using the data obtained in the previous study.

**It was agreed to take into account the sum of C42 to C 46.**

There were problems in reaching an agreement on the limit. However, after lengthy discussion, a consensus was reached on proposing a limit of **150 mg/kg for the virgin and extra virgin categories.**

Since the Council had adopted the **reduction of the limit for myristic acid to 0.03%** at its 18<sup>th</sup> session, the other fatty acids would also be given to two decimal places.

**5. Organisation of ring-tests for 2012–2013 (Annex 8)**

**Pesticides:** pending the results due in late 2012, which will be presented at the next chemists' meeting.

**Mineral oils:** Ms Lacoste reported on this **ISO interlaboratory test:** Stage 1: optimisation of laboratory testing conditions (around 40 laboratories); Stage 2 (autumn): dispatch of samples to the laboratories which participated in Stage 1. Mr Moreda will supply one sample of olive pomace oil for inclusion in the test.

**3-MCPDs:** AOCS interlaboratory test.

**Triglycerides:** January 2013, coordinated by Mr Faberi.

**6. Study on the changes in quality parameters during storage (Annex 9)**

The experts presented a variety of information on this important issue.

Ms Luciana Di Giacinto presented data on a study conducted in 1993 (documents 6.2, 6.3 and 6.4) reporting the impact of packing on oil shelf life. More recent surveys reported the same changes over nine-month periods (phenols, tocopherols, etc.).

Mr Moreda reported on a study under way on four different varieties and differing storage conditions (daylight and darkness), the results of which would serve as a basis for drawing conclusions about the shelf life of oils.

Ms Raffaella Sacchi informed the group on a study due to be completed by December 2012 in which shelf life was estimated according to the light conditions (dependent on the levels of triglycerides, antioxidants, carotenoids, chlorophyll, etc.). She reported that there was not a good correlation between current accelerated oxidation tests and actual shelf life.

Mr Diego García González pointed out that the expiry of oil shelf life was determined by quality and that pyropheophytins were used for this purpose in the United States. It would be a question of checking the changes in certain components at temperature peaks.

Mr García Hierro thought it would be useful to estimate shelf life on the basis of the different components of the oil.

Ms Rovellini presented a study on the nutritional quality of oils (document 6.7), acidity, ultraviolet absorbency and antioxidants. The results reported a 60% drop in quality after six months, followed by a further 10% drop six months later.

Some results would be ready for the next meeting of the group in the spring of 2013.

**7. Study on the composition of oils produced from different varieties – decision trees. Current status with a view to the next session of the Codex Committee on Fats and Oils (Malaysia, 2013). (T.20-3/Doc. No 5-3) – Annex 10**

The Executive Secretariat emphasised the key importance attached to the work on olive oil composition, which was being undertaken at the proposal of the IOC Technical Committee.

Participants were told that the Members considered it necessary to collect reliable varietal data as swiftly as possible in order to allow the IOC to build up a reference database and so avoid the problems caused by erroneous data or data of unknown origin. The objective was to ascertain the actual state of play, which could only be achieved with the cooperation and participation of the producing countries. The Executive Director highlighted the relevance of the compositional survey to the work of the Codex Committee on Fats and Oils (CCFO) on which the IOC had observer status and announced that the IOC member countries would be holding a meeting in December 2012 to prepare for the next CCFO session. He stressed it was important for IOC countries to participate in Codex sessions or at least to submit written comments.

The Executive Secretariat thanked the survey participants and collaborating laboratories and complimented the excellent work of the experts (Ms Chistopoulou and Mr Conte) who had processed the survey data and who would draft the report for submission to the Technical Committee and the Codex Alimentarius.

Participants were given a summary of the outcome of meeting held on varietal composition. Of the 198 samples shipped for testing during the three years of the survey, 121 had been found to deviate from the limits (Argentina, Syria, Spain, Turkey, Tunisia, Australia, Morocco, Portugal). Only one parameter had to be off-standard in each sample.

**1<sup>st</sup> decision tree** for extra virgin and virgin olive oils: campesterol value between 4.0% and 5.0% for (Argentine) oils if:

~~Stigmastadiene~~  $\leq 0.05$  ppm and delta-7-stigmastenol  $\leq 0.3\%$  and stigmasterol  $\leq 1.4\%$

The application of this decision tree might be at variance with the Argentina Food Code, which was revised in 2011 and fixes a campesterol limit of  $\leq 4.5\%$ .

In Brazil a standard applicable since 1 August 2012 fixes a limit of  $\leq 4\%$  for campesterol while the voluntary standards of the United States and Australia establish a respective limit of  $\leq 4.5\%$  and  $4.8\%$ .

It had not been possible to check whether oils from Australia or the United States complied with this decision tree. Only three samples - unaccompanied by any test certificate - were received from Australia in the 1st year.

The Argentine expert informed the chemists that a limit of  $\leq 4.8\%$  had been proposed in the discussions held by the e-group (USA, Australia and Argentina) set up at the last Codex meeting. Neither the ES nor the other participants had received any information on or invitation to join this e-group.

**2<sup>nd</sup> decision tree** for extra virgin and virgin olive oils: delta 7-stigmastenol value between 0.5% and 0.8% if:

~~Stigmastadiene~~  $\leq 0.05$  ppm et delta ECN 42  $\leq |0.1\%|$  and campesterol  $\leq 3.3\%$  and ratio ( $\beta$ -sitosterol/campesterol+delta 7-stigmastenol)  $\geq 25$  and stigmasterol  $\leq 1.3$  or  $1.4\%$

Professor Conte said that when queried about this decision tree, a Syrian oil importing company had replied that it would work for Syrian oils.

He stressed that these decision trees would be applied to bulk oils of specific origin. In the case of packed oils, blending would be required to meet the standards. He added that from the scientific point of view, these conclusions referred solely to Argentina because they were based on samples received from that country.

The Executive Director said this matter would have to be placed before the Technical Committee.

The documents also contain proposals to address the problem of C18:3 (encountered in Morocco) pending additional analyses.

However, this is not a top priority given the current Codex position on C18:3, for which each country is free to set limits.

#### **8. Current status of olive oil and olive pomace oil standards in the member countries**

The ES reported the replies received from a number of countries to the Note Verbale circulated on this matter and stressed that it was important for the other countries to notify the information requested.

#### **9. Olive oil chemistry priorities**

The experts had already mentioned the following as work priorities: continuation of the study on mineral oils in olive pomace oils, detection of deodorised oils, changes in analytical parameters (physico-chemical and organoleptic) during storage, volatile compounds (correlation between sensory and physico-chemical analysis), application of a decision making system, methods for the detection of fraud and the admixture of seed oils to olive oils, creation of a world database incorporating information and the olive oil registers of all the countries.

Mr Conte broached the issue of nutritional claims (linked to Regulation No 432/2012) and commented that there was a problem in the explanations given in the Regulation. Olive oil is protected from oxidation through its phenolics content. The EU Regulation lays down a content of 5 mg/20 g for hydroxytyrosol and derivatives but this is a mistake because oleuropein and tyrosol are not derivatives of hydroxytyrosol. The polyphenols should be shown in a chromatogram in the current HPLC method and the two methods should be tested (official HPLC and provisional colorimetric) for validation purposes.

Mr Ollivier told the experts that the peroxide value study (according to the solvents used, chloroform and iso-octane) would be examined by the restricted testing group. An absolute value for the absorbancy in the ultraviolet should be envisaged for delta K.

The experts unanimously requested chromatogram training workshops to improve between-laboratory reproducibility.

**8. Other business**

Mr Ollivier mentioned the problems in entering the data for the IOC proficiency test, with the ensuring risks during data transfer.

The Executive Secretariat invited the experts to submit written comments for the next meeting. It also reminded them that it was important to present any 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.

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The draft updated trade standard applying to olive oils and olive pomace oils (COI/T.15/NC No 3/Rev. 6) is attached in **Annex 1**.

**Draft updated standard applying to olive oils and olive pomace oils**  
**(COI/T.15/NC No 3/Rev. 6)**



**Results of ring test on alkyl esters, waxes and stigmastadienes**  
**(F. Lacoste)**

**Study on the limits specified in the IOC standard for fatty acid ethyl esters and methyl esters**

**Results of ring test on the determination of triglycerides**  
**(A. Faberi)**

**Global method: study on oils of different origins mixed with extraneous oils**

**Reduction of the stigmastadiene limit**

**Reduction of the wax limit**

**Organisation of ring-tests in 2012–2013**

**Study on the changes in quality parameters during storage**



**Study on the composition of the oils produced from different varieties –  
decision trees. Current status with a view to the next meeting of the Codex  
Committee on Fats and Oils (Malaysia, 2013) - (T.20-3/Doc. No 5-3)**