

**IOC STUDY ON AUTHENTIC OLIVE OILS DISPLAYING OFF-LIMIT
PARAMETERS: CAMPESTEROL**

The composition of edible vegetable oils in terms of their fatty acids and other components is dependent on the plant from which the oil is extracted. It differs according to the variety of the plant and to the soil and climatic conditions in the producing area. The average composition is known and stable, lying within a specific interval for each compound, and permits identification of the botanical origin of the oil. In some ways it is the by-line of the oil.

This average composition is very important in fighting fraud and ensuring that the olive oil that consumers buy for its health or sensory properties has not been mixed with other, cheaper vegetable oils. The International Olive Council (IOC), the UN-brokered intergovernmental organisation mandated to administer the International Agreement on Olive Oil and Table Olives, has been assigned several roles by its membership of 16 producing countries plus the European Union which together account for more than 96 pc of world production, one of which is to fix the average composition of olive oil products in a trade standard.

In specific soil and climatic conditions a variety of olive may sometimes produce oil in which an authenticity or quality marker may fall outside the established interval; as a result, the oil does not comply with the IOC trade standard on which the CODEX standard is very largely modelled. In such cases, a balance can be sought between allowing the necessary flexibility to recognise that the oil is compliant with the standard and preventing such flexibility ó open to all business operators ó from increasing the likelihood of fraud by permitting mixtures with other vegetable oils because they cannot be identified with the same precision. To find such a balance, the IOC has developed what are known as decision trees. The thinking behind these trees is that when a specific parameter in an oil falls outside the established interval, its composition has to meet tougher limits for other parameters in order to make allowances for its special profile and to rule out the risk of the addition and non-detection of other vegetable oils.

Clearly, this is a balancing act where flexibility has to be juggled against risk and where the volume of non-compliant production also has to be taken into account.

Against this background, the IOC Executive Secretariat wishes to make a number of stringently investigated, objective comments regarding campesterol, one of the components used to certify the purity of olive oil for which a decision tree has recently been adopted.

In 2003, the Codex Alimentarius Commission adopted the revised Codex standard for olive oils and olive pomace oils at its 26th session (Rome, 30 June-7 July 2003), since when the campesterol limit fixed in the IOC trade standard remained unchanged until May 2013 when a decision tree was adopted. No general, across-the-board amendment of this limit was planned because this could jeopardise the authenticity of the large majority of oils produced in the world, with the ensuing negative repercussions for consumers.

Nevertheless, the IOC realised that the values of specific parameters in genuine oils could fall outside the limits fixed in the IOC trade standard. Its Members therefore considered it essential to collect swift, reliable varietal data from producing countries in order to build an IOC reference database and so avoid the problems caused by erroneous data or data of unknown origin. Consequently, it decided to mandate an IOC expert group to conduct an exhaustive study on this issue in order to ascertain the general state of play (not just regarding campesterol), with the cooperation and participation of the producing countries.

From the outset, the IOC experts stressed one fundamental aspect: the parameters at issue concerned product authenticity. A cautious, rigorous approach to the study was therefore called for; the subject area needed to be clearly demarcated and the parameters for review and the range of fluctuation in the limits needed to be clearly identified. They believed that a study of this type was needed to demonstrate that the solution for genuine olive oils with off-limit values was to apply the independent decision trees or decision tables proposed by the experts in order to guarantee them market access without benefiting fraud.

The study was carried out for three years (2009-2012). As agreed at the Codex session, the IOC Executive Secretariat invited member and non-member countries of the IOC to provide input for the survey. Requests for samples were circulated every year but some countries sent only a few samples or none at all, despite the agreement reached at the Codex meeting. A follow-up study is underway to examine other parameters besides campesterol and delta-7-stigmastenol. Therefore, the Executive Secretariat continues to receive samples in which other characteristics are off-limit.

The following picture emerges from the results of the testing conducted so far by the IOC chemistry expert working group.

The IOC has received 198 samples (the list of countries is reported in Table 1), 133 of which have been analysed; the remaining 65 have not been tested because they did not record any off-limit parameter. The following tables show that 121 of the samples were deviant, 37 (from Argentina and Australia) for campesterol and 47 (mainly from Syria) for delta-7-stigmastenol. Eighty-four of the 121 samples had one deviant parameter, 15 had two off-limit parameters and 22 had more than three.

All the information about the number of samples submitted by olive oil producing countries from all over the world, the number of samples analysed, the number of deviant samples and the kinds of deviations per country and category etc are presented in the following tables (166) and Figure 1.

Table 1. Number of samples submitted

| | Country or continent | Number of samples submitted | | | | Number of samples analysed | Number of deviant samples |
|--------------|----------------------|-----------------------------|-----------|-----------|------------|----------------------------|---------------------------|
| | | 1st year | 2nd year | 3rd year | Total | | |
| 1. | ALGERIA | | | 3 | 3 | 0 | 0 |
| 2. | ARGENTINA | 8 | 10 | 16 | 34 | 34 | 34 |
| 3. | AUSTRALIA | 8 | | | 8 | 5 | 4 |
| 4. | CYPRUS | 3 | | | 3 | 0 | 0 |
| 5. | GREECE | 3 | | | 3 | 0 | 0 |
| 6. | ISRAEL | 13 | 24 | | 37 | 0 | 0 |
| 7. | MOROCCO | 3 | 4 | | 7 | 7 | 7 |
| 8. | PORTUGAL | | 3 | | 3 | 3 | 3 |
| 9. | SLOVENIA | | | 3 | 3 | 0 | 0 |
| 10. | SPAIN | 6 | 6 | 7 | 19 | 19 | 19 |
| 11. | SYRIA | 6 | 17 | 16 | 39 | 38 | 36 |
| 12. | TUNISIA | | | 19 | 19 | 19 | 11 |
| 13. | TURKEY | 12 | 12 | 12 | 36 | 8 | 7 |
| TOTAL | | 65 | 76 | 57 | 198 | 133 | 121 |

Table 2. Distribution of the samples (n=121) according to the number of deviations per sample

| Deviations/per sample | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | Total |
|-----------------------|-----------|-----------|----------|----------|----------|----------|----------|----------|------------|
| Argentina | 5 | 8 | 5 | 5 | 4 | 3 | 3 | 1 | 34 |
| Australia | 4 | | | | | | | | 4 |
| Morocco | 6 | 1 | | | | | | | 7 |
| Portugal | 3 | | | | | | | | 3 |
| Spain | 19 | | | | | | | | 19 |
| Syria | 33 | 3 | | | | | | | 36 |
| Tunisia | 9 | 2 | | | | | | | 11 |
| Turkey | 5 | 1 | 1 | | | | | | 7 |
| TOTAL | 84 | 15 | 6 | 5 | 4 | 3 | 3 | 1 | 121 |

Figure 1 Percentage of each country's samples with more than one deviant parameter/sample

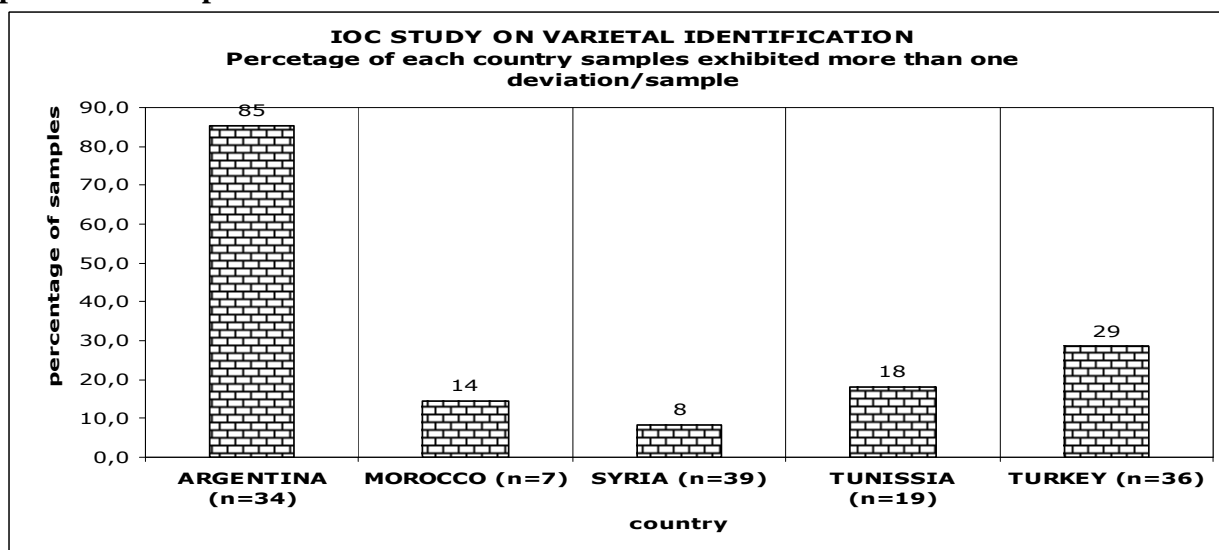


Table 3. Number of deviant samples per parameter in each country, in descending order (n=number of samples submitted)

| Parameter | ARGENTINA (n=34) | AUSTRALIA (n=4) | MOROCCO (n=7) | PORTUGAL (n=3) | SPAIN (n=19) | SYRIA (n=36) | TUNISIA (n=11) | TURKEY (n=7) | TOTAL |
|---------------------------------------|------------------|-----------------|---------------|----------------|--------------|--------------|----------------|--------------|-------|
| Campesterol | 34 | 3 | | | | | | | 37 |
| 7-stigmastenol (extra & virgin) | | | | | 8 | 25 | | 4 | 37 |
| Waxes | 20 | | | | | | | | 20 |
| Oleic acid | 20 | | | | | | | | 20 |
| ECN42 | 14 | | 1 | | | 1 | | | 16 |
| Linoleic acid | 8 | | | | 3 | | | | 11 |
| 7-stigmastenol (lampante) | | | | | | 8 | | 2 | 10 |
| Erythrodiol+uvaol extra & virgin | | | | 3 | 6 | | | 1 | 10 |
| Palmitic acid | 10 | | | | | | | | 10 |
| Apparent -sitosterol (extra & virgin) | 9 | | | | | | | | 9 |

| | | | | | | | | | |
|-------------------------------------|---|---|---|--|---|---|---|---|---|
| 7-stigmastenol (olive pomace) | | | | | | | 8 | | 8 |
| Linolenic acid | 1 | | 7 | | | | | | 8 |
| Apparent -sitosterol (olive pomace) | | | | | | | 5 | | 5 |
| Palmitoleic acid | 5 | | | | | | | | 5 |
| Apparent -sitosterol (lampante) | | | | | | 3 | | 1 | 4 |
| Total sterols | | | | | 2 | 2 | | | 4 |
| Erythrodiol+uvaol (lampante) | | | | | | | | 1 | 1 |
| Gadoleic acid | | 1 | | | | | | | 1 |
| Lignoceric acid | 1 | | | | | | | | 1 |
| 2-glycerol-monopalmitin | 1 | | | | | | | | 1 |

Table 4. Number of deviant parameters of each country

| Country | DEVIANT PARAMETER | Number of deviant parameters |
|------------------|--|------------------------------|
| ARGENTINA (n=34) | campesterol, apparent -sitosterol (extra & virgin), waxes, palmitic acid, palmitoleic acid, oleic acid, linoleic acid, linolenic acid, lignoceric acid, ECN42, 2-glycerol-monopalmitin | 11 |
| AUSTRALIA (n=5) | campesterol, gadoleic acid | 2 |
| MOROCCO (n=7) | linolenic acid, ECN42 | 2 |
| PORTUGAL (n=3) | erythrodiol + uvaol | 1 |
| SPAIN (n=19) | 7-stigmastenol, total sterols, erythrodiol + uvaol, linoleic acid | 4 |
| SYRIA (n=39) | 7-stigmastenol (extra, virgin and lampante), apparent -sitosterol (lampante), total sterols, ECN42 | 4 |
| TUNISIA (n=19) | 7-stigmastenol (olive pomace), apparent -sitosterol (olive pomace) | 2 |
| TURKEY (n=36) | 7-stigmastenol (extra, virgin and lampante), apparent -sitosterol (lampante), erythrodiol + uvaol | 3 |

Table 5. Deviant parameters per category of olive oil

| Category | Parameter |
|---------------------------|--|
| Extra & virgin | campesterol, 7-stigmastenol, apparent β -sitosterol, total sterols, waxes, palmitic acid, palmitoleic acid, oleic acid, linoleic acid, linolenic acid, gadoleic acid, lignoceric acid, ECN42, 2-glycerol-monopalmitin, erythrodiol + uvaol |
| Lampante | 7-stigmastenol, apparent β -sitosterol, total sterols, erythrodiol + uvaol |
| Olive pomace | 7-stigmastenol, apparent β -sitosterol |

Table 6. Deviant parameters by country

| Parameter | Country |
|---|-------------------------|
| Campesterol | Argentina, Australia |
| 7-stigmastenol (extra & virgin) | Spain, Syria, Turkey |
| 7-stigmastenol (lampante) | Syria, Turkey |
| 7-stigmastenol (olive pomace) | Tunisia |
| Apparent β -sitosterol (extra & virgin) | Argentina |
| Apparent β -sitosterol (lampante) | Syria, Turkey |
| Apparent β -sitosterol (olive pomace) | Tunisia |
| Total sterols | Spain, Syria |
| Erythrodiol+uvaol | Portugal, Spain, Turkey |
| Waxes | Argentina |
| Palmitic acid | Argentina |
| Palmitoleic acid | Argentina |
| Oleic acid | Argentina |
| Linoleic acid | Argentina, Spain |
| Linolenic acid | Argentina, Morocco |
| Gadoleic acid | Australia |

| | |
|-------------------------|---------------------------|
| Lignoceric acid | Argentina |
| ECN42 | Argentina, Morocco, Syria |
| 2-glycerol-monopalmitin | Argentina |

The results for the samples tested were divided into subgroups by deviant parameter and oil category. Only one result was needed for each parameter and sample for data processing purposes. Consequently, the results were evaluated on the basis of the **worst case** as opposed to the mean value of the samples analysed. In other words, the highest value recorded per sample was taken into account for the parameters with a maximum limit (campesterol, 7-stigmastenol, etc.) and the lowest for the parameters with a minimum limit (apparent α -sitosterol). Hence, data processing took into account all the samples for which even just one laboratory detected a deviation.

The results underwent further processing as follows:

A. Effectiveness of the decision tree in the detection of olive oil fraud, i.e. the risk of adulteration when a decision tree is applied due to a permitted increase in the official limit of a parameter.

This is considered a very important step before the adoption of a decision tree because the first and foremost concern is to protect olive oil from fraud. This procedure comprised two steps:

1. Firstly, a bar-line combination chart was created to compare the effectiveness of a parameter vis-à-vis a deviant parameter in the detection of olive oil adulteration. This step permits identification of those parameters which can replace the deviant parameter.
2. Secondly, the percentage of seed oil detectable in olive oil was calculated by using not only the purity criteria that proved effective in the first step, but additional criteria as well and applying them at the official or even stricter limits. This step allowed us to conclude whether there are other parameters (aside from the deviant one) that are effective in the detection of fraud, or whether the deviant parameter is irreplaceable.

B. Effectiveness of the application of the decision tree to the deviant samples analysed

This entailed calculating the statistical data for the deviant samples and the percentage of samples tested that conformed to the proposed limit for each parameter.

The most suitable parameters were selected on the basis of sample conformity and a decision tree was created for each deviant parameter and category of virgin olive oil.

The results of the above evaluation for the deviant parameter **campesterol** are reported below:

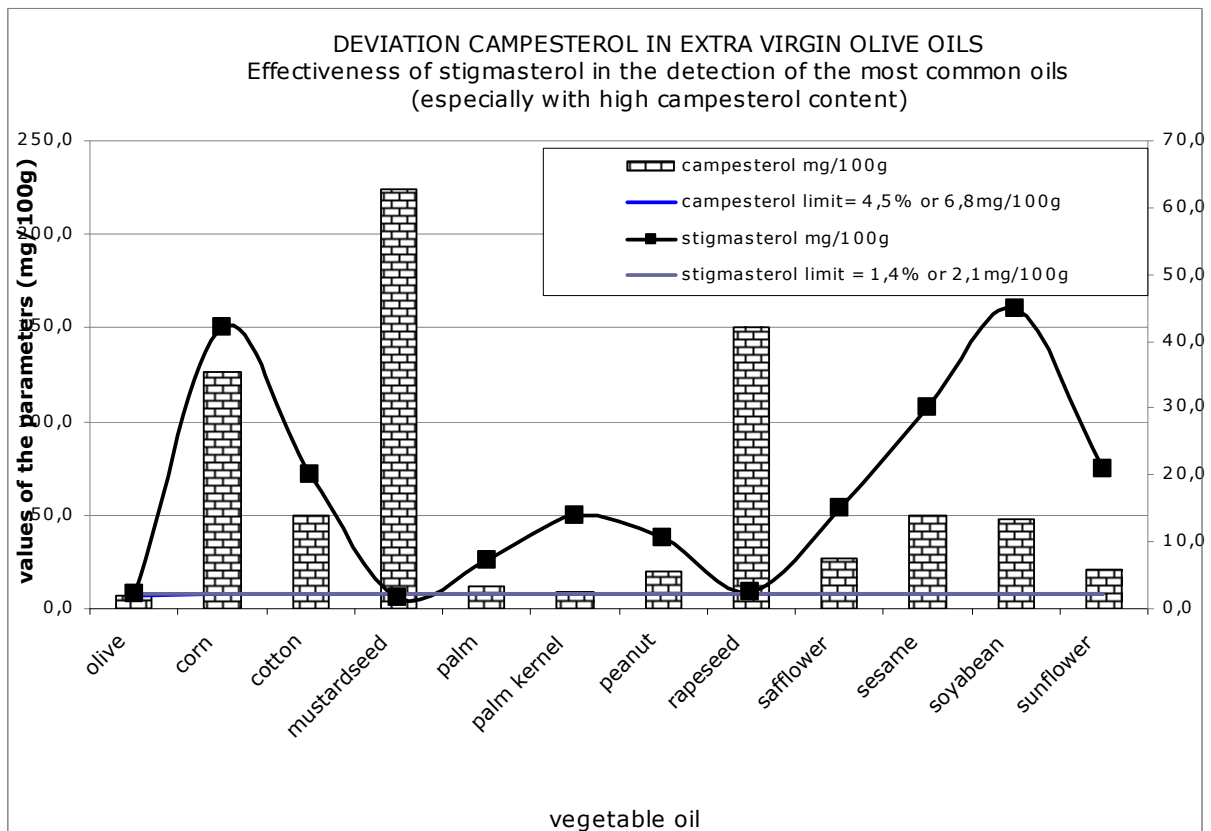
DEVIATION: CAMPESTEROL IN EXTRA VIRGIN AND VIRGIN OLIVE OIL

A. Effectiveness of the decision tree in the detection of olive oil fraud

1. Except for olive oil, the most common vegetable oils have a high content of campesterol (corn, cotton, mustard, palm, palm kernel, peanut, rapeseed, safflower, sesame, soyabean, sunflower) and stigmasterol (except rapeseed and mustard). These two components are therefore used as evidence of the presence of seed oils.

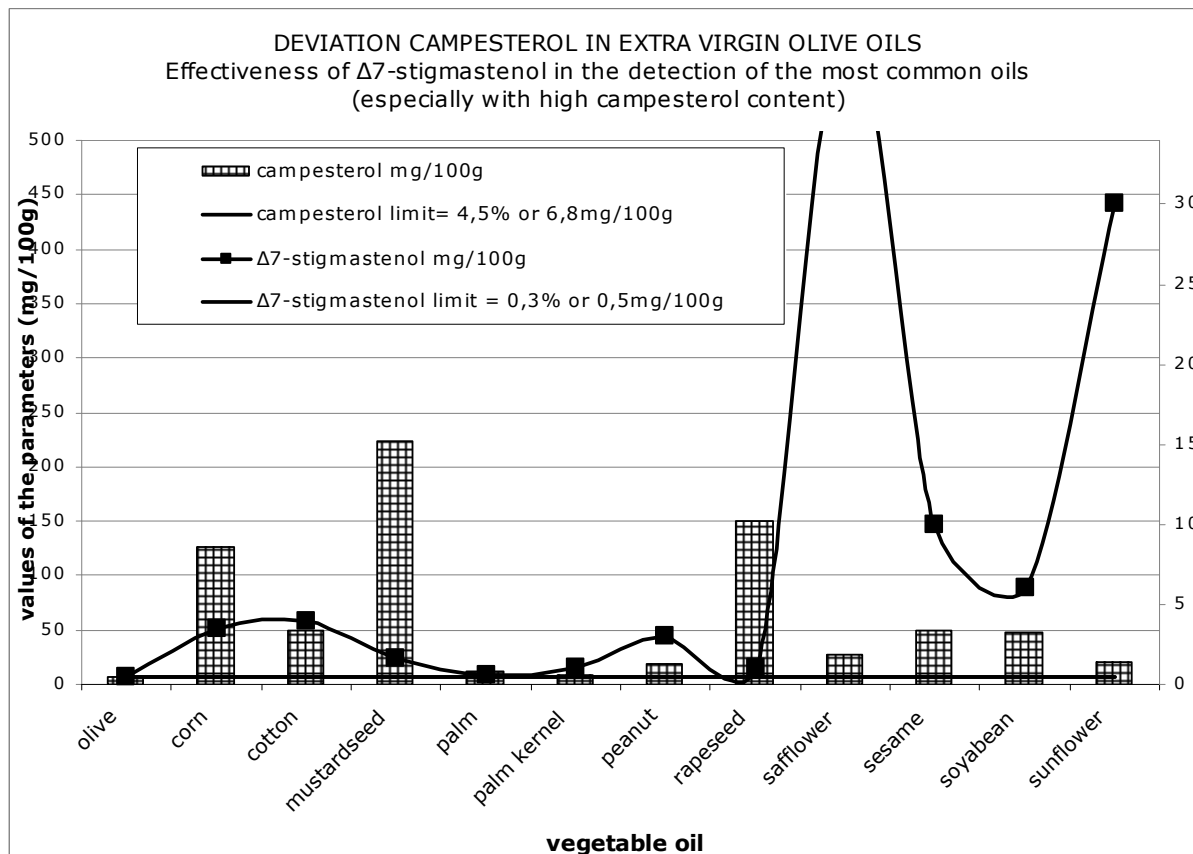
Stigmasterol and 7-stigmastenol are compared separately with the deviant parameter (campesterol) in the following two bar-line combination charts in order to assess their effectiveness in the detection of olive oil adulteration. Total sterols content has also been taken into account to enhance the reliability of the conclusions; consequently, the values of the parameters plotted on the y-axis are expressed in mg/100g.

Figure 2. Effectiveness of stigmasterol in the detection of the most common oils



Conclusion: Stigmasterol is more effective than campesterol in the detection of all other oils except rapeseed and mustard.

Figure 3. Effectiveness of Δ^7 -stigmastenol in the detection of the most common oils



Conclusions:

- Δ^7 -stigmastenol is very effective for the detection of sunflower and safflower oil
- Δ^7 -stigmastenol is more effective than campesterol for the detection of cotton, peanut, sesame and soyabean oils
- Δ^7 -stigmastenol is useless for the detection of mustard and rapeseed oil

2. Calculation of the percentage of detectable seed oil in olive oil using various purity criteria in order to ascertain whether some other parameters (aside from campesterol, the deviant parameter) are effective in the detection of fraud or whether the deviant parameter is irreplaceable.

In all cases, the percentage of detectable seed oil in olive oil was calculated by using the official limit for campesterol as well. This calculation indicates how the effectiveness of campesterol will change if its limit is raised from 4.0% to 4.5%.

The following tables present the results of this exercise. The parameters that are more effective than the deviant parameter detection-wise are shaded in grey.

Table 7: Detection of olive oil adulteration with high campesterol oils

| | Fraud detection parameter | Parameter limit applied | Percentage of seed oil detectable in olive oil | Value used for | |
|------------------------|---------------------------|-------------------------|--|----------------|----------|
| | | | | olive oil | seed oil |
| Corn | Campesterol % | 4.0 | é1.5% | 3.0 | 18.0 |
| | Campesterol % | 4.5 | é2.4% | 3.0 | 18.0 |
| | Stigmasterol % | 1.4 | é1.5% | 1.1 | 6.0 |
| | 7-stigmastenol % | 0.3 | é12.0% | 0.2 | 0.5 |
| | Apparent -sitosterol % | 93.0 | é1.5% | 94.0 | 73.0 |
| | total sterols | | | 1500 | 7000 |
| | Linoleic acid% | 3.5-6.0 | é3.5% | 4.5 | 45.0 |
| ECN42 (absolute value) | 0.2 | é1.0% | 0.1 | 0.9 | |
| Cotton | Campesterol % | 4.0 | é5.0% | 3.0 | 10.0 |
| | Campesterol % | 4.5 | é7.5% | 3.0 | 10.0 |
| | Stigmasterol % | 1.4 | é3.5% | 1.1 | 4.0 |
| | 7-stigmastenol % | 0.3 | é5.5% | 0.2 | 0.8 |
| | total sterols | | | 1500 | 5000 |
| | Linoleic acid % | 3.5-6.0 | é3.5% | 4.5 | 48.0 |
| | ECN42 (absolute value) | 0.2 | é1.0% | 0.1 | 1.2 |
| Mustard seed | Campesterol % | 4.0 | <1.0% | 3.0 | 28.0 |
| | Campesterol % | 4.5 | é1.2% | 3.0 | 28.0 |
| | Stigmasterol % | 1.4 | not detected | 1.1 | 0.2 |
| | 7-stigmastenol % | 0.3 | not detected | 0.2 | 0.2 |
| | Apparent -sitosterol % | 93.0 | é1.0% | 94.0 | 64.0 |
| | total sterols | | | 1500 | 8000 |
| | Linoleic acid % | 3.5-6.0 | é12.0% | 4.5 | 17.0 |
| | Linolenic acid % | 1.0 | é2.0% | 0.7 | 10.0 |
| | Erucic acid % | 0.0 | é0.1% | 0.0 | 37.0 |
| ECN42 (absolute value) | 0.2 | é4.5% | 0.1 | 30.0 | |
| Palm | Campesterol % | 4.0 | é14.0% | 3.0 | 20.0 |
| | Campesterol % | 4.5 | é20.0% | 3.0 | 20.0 |
| | Stigmasterol % | 1.4 | é4.0% | 1.1 | 12.0 |
| | 7-stigmastenol % | 0.3 | >15.0% | 0.2 | 1.0 |
| | total sterols | | | 1500 | 600 |
| | Myristic acid % | 0.03 | é1.0% | 0.02 | 1.0 |
| | Linoleic acid % | 3.5-6.0 | not detected | 4.5 | 2.0 |
| Palm kernel | Campesterol % | 4.0 | >15.0% | 3.0 | 9.0 |
| | Campesterol % | 5.0 | >15.0% | 3.0 | 9.0 |
| | Stigmasterol % | 1.4 | é3.5% | 1.1 | 14.0 |
| | 7-stigmastenol % | 0.3 | >15.0% | 0.2 | 1.0 |
| | total sterols | | | 1500 | 1000 |
| | Lauric acid % | 0.0 | é0.1% | 0.0 | 45.0 |
| | Myristic acid % | 0.03 | é0.1% | 0.02 | 14.0 |

| | | | | |
|-----------------|---------|--------------|-----|-----|
| Linoleic acid % | 3.5-6.0 | not detected | 4.5 | 2.0 |
|-----------------|---------|--------------|-----|-----|

Table 8: Detection of olive oil adulteration with high campesterol oils

| | Fraud detection parameter | Parameter limit applied | Percentage of seed oil detectable in olive oil | Value used for | |
|------------------------|---------------------------|-------------------------|--|----------------|----------|
| | | | | olive oil | seed oil |
| Peanut | Campesterol % | 4.0 | ≤10.0% | 3.0 | 13.0 |
| | Campesterol % | 4.5 | ≤15.0% | 3.0 | 13.0 |
| | Stigmasterol % | 1.4 | ≤5.0% | 1.1 | 7.0 |
| | 7-stigmastenol % | 0.3 | ≤5.5% | 0.2 | 2.0 |
| | Apparent -sitosterol % | 93.0 | ≤7.0% | 94.0 | 80.0 |
| | total sterols | | | 1500 | 1500 |
| | Linoleic acid % | 3.5-6.0 | ≤10.0% | 4.5 | 20.0 |
| | Behenic acid % | 0.2 | ≤3.0% | 0.1 | 3.0 |
| Rapeseed | Campesterol % | 4.0 | ≤1.5% | 3.0 | 30.0 |
| | Campesterol % | 4.5 | ≤1.7% | 3.0 | 30.0 |
| | Stigmasterol % | 1.4 | not detected | 1.1 | 0.5 |
| | 7-stigmastenol % | 0.3 | not detected | 0.3 | 0.2 |
| | Brassicasterol % | 0.1 | ≤0.1% | 0.01 | 9 |
| | Apparent -sitosterol % | 93.0 | ≤1.0% | 94.0 | 54.0 |
| | total sterols | | | 1500 | 5000 |
| | Linoleic acid % | 3.5-6.0 | >10.0% | 4.5 | 16.0 |
| | Linolenic acid % | 1.0 | ≤3.0% | 0.7 | 10.0 |
| | Erucic acid % | 0.0 | ≤0.5% | 0.0 | 3.0 |
| ECN42 (absolute value) | 0.2 | ≤4.0% | 0.1 | 0.33 | |
| Safflower | Campesterol % | 4.0 | ≤10.0% | 3.0 | 9.0 |
| | Campesterol % | 4.5 | ≤15.0% | 3.0 | 9.0 |
| | Stigmasterol % | 1.4 | ≤4.0% | 1.1 | 5.0 |
| | 7-stigmastenol % | 0.3 | <0.5% | 0.2 | 14.0 |
| | Apparent -sitosterol % | 93.0 | ≤1.0% | 94.0 | 52.0 |
| | total sterols | | | 1500 | 3000 |
| | Linoleic acid % | 3.5-6.0 | ≤2.5% | 4.5 | 76.0 |
| | ECN42 (absolute value) | 0.2 | ≤1.0% | 0.1 | 5.3 |
| Sesame | Campesterol % | 4.0 | ≤5.0% | 3.0 | 10.0 |
| | Campesterol % | 4.5 | ≤8.0% | 3.0 | 10.0 |
| | Stigmasterol % | 1.4 | ≤2.0% | 1.1 | 6.0 |
| | 7-stigmastenol % | 0.3 | ≤2.0% | 0.2 | 2.0 |
| | Apparent -sitosterol % | 93.0 | ≤1.0% | 94.0 | 69.0 |
| | total sterols | | | 1500 | 5000 |
| | Linoleic acid % | 3.5-6.0 | ≤13.0% | 4.5 | 16.0 |
| | ECN42 (absolute value) | 0.2 | ≤1.5% | 0.1 | 4.3 |

Table 9: Detection of olive oil adulteration with high campesterol oils

| | Fraud detection parameter | Parameter limit applied | Percentage of seed oil detectable in olive oil | Value used for | |
|------------------|---------------------------|-------------------------|--|----------------|----------|
| | | | | olive oil | seed oil |
| Soyabean | Campesterol % | 4.0 | €4.0% | 3.0 | 16.0 |
| | Campesterol % | 4.5 | €6.2% | 3.0 | 16.0 |
| | Stigmasterol % | 1.4 | €1.5% | 1.1 | 15.0 |
| | 7-stigmastenol % | 0.3 | €3.0% | 0.2 | 2.0 |
| | Apparent -sitosterol % | 93.0 | €2.0% | 94.0 | 65.0 |
| | total sterols | | | 1500 | 3000 |
| | Linoleic acid % | 3.5-6.0 | €3.5% | 4.5 | 48.0 |
| | ECN42 (absolute value) | 0.2 | €1.0% | 0.1 | 2.2 |
| Sunflower | Campesterol % | 4.0 | >15.0% | 3.0 | 7.0 |
| | Campesterol % | 4.5 | >15.0% | 3.0 | 7.0 |
| | Stigmasterol % | 1.4 | €2.5% | 1.1 | 7.0 |
| | 7-stigmastenol % | 0.3 | €0.5% | 0.2 | 10.0 |
| | Apparent -sitosterol % | 93.0 | €2.0% | 94.0 | 68.0 |
| | total sterols | | | 1500 | 3000 |
| | Linoleic acid % | 3.5-6.0 | €3.5% | 4.5 | 45.0 |
| | ECN42 (absolute value) | 0.2 | €1.0% | 0.1 | 0.5 |

Conclusions:

- Stigmasterol (limit 0.4%) is equally or more effective than campesterol (limit 4.0%) in the detection of corn, cotton, palm, palm kernel, peanut, safflower, sesame, soyabean and sunflower oil.
- The official limit for apparent -sitosterol (×93.0%) is effective in the detection of corn, mustard seed, rapeseed, safflower, sesame, soyabean and sunflower oil.
- The official limit for CN42 (0.2) is very effective for the detection of the addition of corn, cotton, safflower, sesame, soyabean and sunflower oils to olive oil.
- The official limit for brassicasterol (0.1%) protects olive oil from adulteration with rapeseed oil.
- The official limit for behenic acid (0.2%) protects olive oil from adulteration with peanut oil, that for linolenic acid (1.0%) protects it from adulteration with mustard and rapeseed oil and that for myristic acid (0.03%) protects it from adulteration with palm and palm kernel oil.
- Stigmastadiene (limit 0.05 mg/kg) is very effective for the detection of the adulteration of virgin olive oil with all refined oils.

In addition, the percentage detection threshold of seed oil in olive oil has been calculated in order to determine how the effectiveness of campesterol in fraud detection is affected by raising its limit from 4.0% to 4.5%, 4.6%, 4.7%, 4.8%, 4.9% and 5.0%. The results obtained are presented in Table 10.

Table 10. Approximate percentage detection threshold of seed oil in olive oil on applying various campesterol limits (data from CODEX composition tables and obtained in IOC official laboratories).

| Type of oil | Campesterol limit applied | | | | | | | Value used for fraud detection | |
|-------------|---------------------------|------|------|------|------|------|------|--------------------------------|---------------------|
| | 4.0 | 4.5 | 4.6 | 4.7 | 4.8 | 4.9 | 5.0 | CAMPESTEROL % | TOTAL STEROLS mg/kg |
| MUSTARD | 0.8 | 1.2 | 1.3 | 1.4 | 1.4 | 1.5 | 1.6 | 28.0 | 8000 |
| RAPESEED | 1.3 | 1.7 | 1.9 | 2.0 | 2.1 | 2.2 | 2.3 | 30.0 | 5000 |
| CORN | 1.5 | 2.4 | 2.5 | 2.7 | 2.9 | 3.0 | 3.2 | 18.0 | 7000 |
| SOYABEAN | 4.0 | 6.2 | 6.6 | 7.0 | 7.5 | 7.9 | 8.3 | 16.0 | 3000 |
| COTTON | 5.0 | 7.6 | 8.2 | 8.8 | 9.4 | 10.1 | 10.7 | 10.0 | 5000 |
| SESAME | 5.0 | 7.6 | 8.2 | 8.8 | 9.4 | 10.1 | 10.7 | 10.0 | 5000 |
| PEANUT | 10.0 | 15.0 | 16.0 | 17.0 | 18.0 | 19.0 | 20.0 | 13.0 | 1500 |
| SAFFLOWER | 10.0 | 14.6 | 15.5 | 16.5 | 17.5 | 18.8 | 20.0 | 9.0 | 3000 |
| PALM | 14.0 | 20.0 | 21.0 | 22.0 | 23.0 | 24.0 | 25.0 | 20.0 | 600 |
| SUNFLOWER | 15.0 | 23.0 | 25.0 | 27.0 | 29.0 | 31.0 | 33.0 | 7.0 | 3000 |
| PALM KERNEL | 24.0 | 33.0 | 35.0 | 37.0 | 39.0 | 41.0 | 43.0 | 9.0 | 1000 |
| OLIVE OIL | | | | | | | | 3.0 | 1500 |

As can be seen from the above table, campesterol by itself is very sensitive in the detection of mustard, rapeseed and corn oil but not so in the case of the other oils.

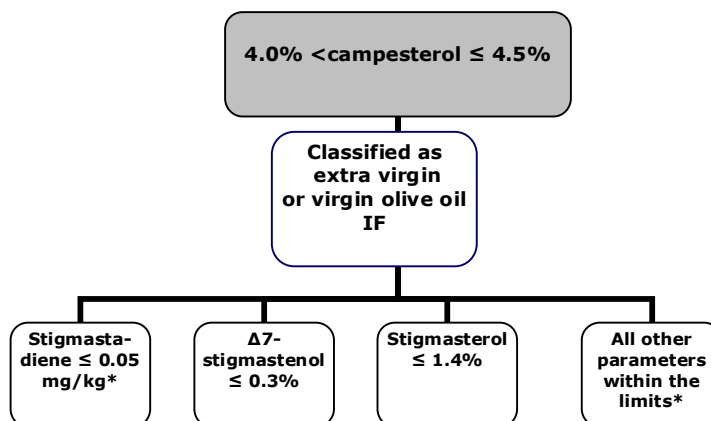
In the light of these results, it is concluded that raising the campesterol limit from 4.0 to 5.0% almost **doubles the percentage detection threshold of seed oil, thus halving the effectiveness of the measurement of campesterol in the detection of fraud.**

In all the above cases there are other effective parameters besides campesterol for detecting adulteration. It could therefore be argued that the adoption of a high campesterol limit in the decision tree (e.g. 6.0%) does not increase the risk of adulteration or that campesterol can be replaced by stigmasterol. However, this is absolutely wrong because:

- ✓ Increasing the campesterol limit reduces the **detection threshold of seed oil (see Table 10)**.
- ✓ The purity parameters are complementary in detecting fraud; hence, all the purity criteria are necessary for the detection of adulteration. Campesterol in particular is a useful tool in the case of fraud since the most common vegetable oils (except olive oil) have a high campesterol content.
- ✓ Introducing a large increase in the limit of a purity parameter might have dramatic consequences for fraud detection because olive oils differ in composition. Moreover, all the above calculations are based on the theoretical detection of the adulteration of olive oil by admixture with one vegetable oil, but what happens when more than one seed oil is added to olive oil?
- ✓ Replacing the campesterol parameter by stigmasterol at a limit of 1.9% does not ensure the authenticity of olive oils because at this limit stigmasterol is not effective in the detection of fraud. Moreover, its adoption would lead to further deviations in some olive oils.

In the light of all the above considerations, **the 4.5% limit for campesterol was fixed in the decision tree** as described below:

In cases where a virgin olive oil has a campesterol content between 4.0% and 4.5%, other parameters exist which ensure their authenticity. Hence, the decision tree for $4.0\% < \text{campesterol} \leq 4.5\%$ could include stigmasterol $\leq 1.4\%$, 7-stigmastenol $\leq 0.3\%$ and stigmastadiene $\leq 0.05\text{mg/kg}$, provided all the other purity criteria lie within the established limits.



* The limit for stigmastadiene in the IOC international trade standard was lowered from 0.10 mg/kg to 0.05 mg/kg in May 2013, for extra virgin and virgin olive oils

B. Application of the decision tree to the deviant samples analysed

The decision tree has been verified by applying it to 34 authentic samples with deviant campesterol content.

Table 11. Statistical data on samples deviating from the campesterol limit

| EXTRA VIRGIN OLIVE OILS (n=34) | Mean | Min | Max | Median | Decision tree |
|---|-------------|------------|------------|---------------|----------------------|
| Stigmastadiene content (mg/kg) | 0.01 | 0.01 | 0.10 | 0.01 | Ö0.05 |
| Campesterol % | 4.9 | 4.4 | 5.7 | 4,9 | Ö4.5 |
| Stigmasterol % | 1.0 | 0.6 | 1.7 | 1.1 | Ö1.4 |
| 7-stigmastenol % | 0.3 | 0.1 | 0.5 | 0.2 | Ö0.3 |
| Apparent -sitosterol % * | 93.2 | 91.9 | 94.3 | 93.2 | ×93.0 |
| Ap. -sito/(stigma+ 7-stigma) | 77.2 | 46.0 | 134.7 | 71.85 | ×60.0 |
| C18:2 linoleic acid % ** | 19.4 | 9.1 | 23.5 | 19.7 | Ö6.0 and ×3.5 |
| ECN42 (absolute value) * | 0.2 | 0.1 | 0.6 | 0.2 | Ö0.2 |

Notes:

* Apparent -sitosterol and ECN42 could not be included in the decision tree at their official limits of ×93.0 and Ö0.2], respectively. However, they are shown above to allow scrutiny of sample conformity with these limits.

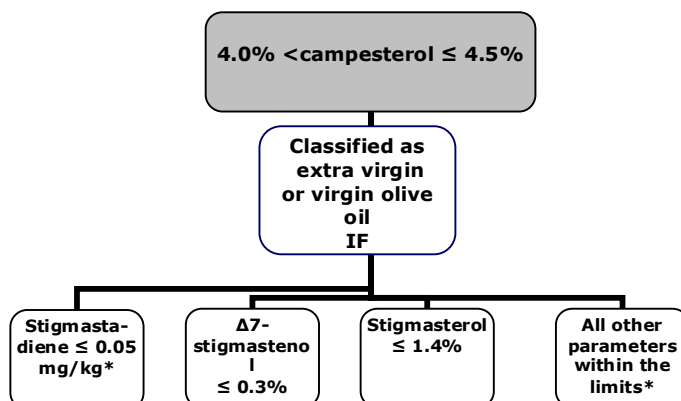
**Linoleic acid was proposed initially for inclusion in the decision tree for campesterol.

Consequently, the decision tree for samples of olive oils in the extra virgin and virgin categories with values **4.0%<campesterolÖ4.5%** should include: stigmasterol (limit Ö1.4%), 7-stigmastenol (limitÖ0.3%) and stigmastadiene (limit Ö0.05mg/kg).

Final conclusions:

The IOC member countries, which account for 97 pc of world export volume, are obliged to apply the provisions of the IOC trade standard in their international trade. In the ultimate analysis, in its trade standard the IOC is duty-bound to stipulate authenticity parameters that provide maximum protection from the risk of fraud, i.e. from the risk of the addition of other vegetable oils. Campesterol is one such important parameter but some of the olive varieties grown in the New World produce oils whose campesterol limit exceeds the level stipulated in the IOC standard. This is no obstacle to their being sold on the domestic market. However, to give them access to the world market and to allow them to abide by the IOC trade standard, it was decided to find a solution for the campesterol limit even although only a very limited volume of product is affected. A straight increase of the existing limit (4.0%) was ruled out for obvious reasons to do with the risk of fraud, as was the idea of introducing a higher limit for certain countries or even regions because this would generate traceability costs for the oils concerned and the likelihood of commercial discrimination. Finally, the IOC opted to introduce the decision tree presented in this paper for oils with a campesterol content between 4.0 and 4.5% by applying more restrictive limits for some purity parameters. There are of course authentic oils with a higher content than 4.5% but this limit helps to contain the risk of fraud in the majority of the oils produced worldwide where campesterol is not a problem. Every decimal point above 4.5% represents a heightened threat of non-detectable admixtures with other vegetable oils. By fixing a limit of 4.5%, the IOC has found the same point of equilibrium as Argentina in its National Food Code and as the United States in its voluntary federal standard. Lastly, if exporters have oils on their hands with a campesterol content of more than 4.5% which they wish to sell on the world market, one legal option is to blend them, prior to export, with olive oils with a lower campesterol content so that the resultant blend abides by the IOC trade standard.

Campesterol decision tree for virgin and extra virgin olive oils:



* The limit for stigmastadiene in the IOC international trade standard was lowered from 0.10 mg/kg to 0.05 mg/kg in May 2013, for extra virgin and virgin olive oils

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