

Testing olive oil quality: chemical and sensory methods

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Introduction

Olive oil producers must verify the quality of their products by chemical and sensory testing. These tests ensure the olive oil product meets the customer's specifications as well as the quality standards set by the International Olive Council (IOC), formerly the International Olive Oil Council (IOOC).

Meeting IOC standards allows Australian producers to compete in the export market. Australian olive oil sales are increasing in traditional olive oil markets such as Italy and Spain as well as Germany, UK and the USA.

Olive oil is a unique product. Understanding the chemical profile of the oil is important to understanding the sensory characteristics and vice versa. Quality profiles of varieties can be valuable when you look at how crop management affects the quality of olive oil. It is also possible to profile varietal oils produced in different environments where there can be large differences between groves within a single region.

In addition to tests for oil quality, there are a range of other tests designed to ensure olive oil is authentic. This is to ensure that the oil has not been adulterated with seed oils or refined olive oil. The tests can also indicate if the oil has been processed by means other than mechanical extraction.

International standards

Olive oil is expected to meet some basic standards which differentiate it from other oils. It should also be harvested and processed to maintain acceptable quality. For example, some of the basic quality parameters that extra virgin olive oil (EVOO) should meet are described in Table 1.

Table 1. IOC Standards for extra virgin olive oil.

Free fatty acid $\leq 0.8\%$
Peroxide value < 20 mEq O₂ /kg oil

Fatty acid profile	Carbon no.	Content %
Myristic	C14:0	≤ 0.5
Palmitic	C16:0	7.5 – 20
Palmitoleic	C16:1	0.3 – 3.5
Heptadecanoic	C17:0	≤ 0.3
Heptadecenoic	C17:1	≤ 0.3
Stearic	C18:0	0.5 – 5.0
Oleic	C18:1	55 – 83
Linoleic	C18:2	3.5 – 21
Linolenic	C18:3	≤ 1
Arachidic	C20:0	≤ 0.6

In addition to these basic tests there are several other categories which olive oil is expected to comply to. These include:

- sterol composition
- stigmastidiene content
- wax content
- UV absorption
- equivalent carbon number (ECN)
- trans fatty acids
- triacylglycerol composition.

This second group of tests are generally only used to determine if the oil has been adulterated.

More details on the chemistry of olive oil can be found in Primefact 227, *Chemistry and quality of olive oil*.



Grades of olive oil

There are nine grades of olive oil, although most Australian production effort is focused on extra virgin olive oil.

Virgin	Refined
Extra virgin	Refined
Virgin	Olive oil
Ordinary	Crude pomace
Lampante	Refined pomace
	Olive pomace

Tests growers need to do

There are many tests that laboratories carry out on olive oil to determine the quality, shelf life and overall characteristics of the oil. In many cases they are to detect adulteration of the oil. These tests are often time-consuming and expensive, but necessary to ensure that olive oil quality relates to the label. It would be pointless, however, for growers to undertake such tests on their own oil when they know the oil has not been adulterated.

However, there are some tests that are important for growers to do.

Oil content

The amount of oil produced is a characteristic of the cultivar and the environment in which it is grown. It is therefore logical that a grower would want to know which trees were more productive or less. Three types of tests can be done.

- **Solvent extraction** removes all traces of oil from the olive material. The extracted oil does not meet the requirements of extra virgin oil, but it is the only way of determining the absolute amount of oil in the fruit. The method gives a result higher than you would expect to get from an olive press, but it provides an accurate comparison between trees.
- **Cold press method** extracts less oil. It is difficult to compare the oil production from two trees from a cold press test as the result varies slightly each time. However, it gives a better indication than solvent extraction of the oil that might be extracted by an olive press. In addition, the oil extracted is extra virgin oil and can be used for subsequent tests.
- **NIR (Near infra-red analyser)** can be used in the laboratory or processing mill. NIR technology can measure oil and moisture in milled olives and fatty acids and moisture in oil. The instrument works by passing near infra-red light through oil samples and measuring absorbed energy which relates to the concentration of oil, moisture and fatty acids.

Moisture content

Moisture content of olives changes dramatically from day to day. If the moisture content in the olive is high, the fruit will be heavy and the oil concentration will be diluted. High moisture content also means oil extraction may be restricted. There may also be a loss of flavour and reduced levels of antioxidants including polyphenols. Although the oil content does not change, the percentage of oil will vary from day to day based on the moisture content. It is therefore important to test the moisture content of the olives at the same time that oil content is tested so that samples can be accurately compared with each other and over time.

Fruit moisture content is often 55% or higher in developing fruit but then drops to 50% or less with the commencement of ripening. This will vary between varieties and seasonal conditions and some varieties will not follow this trend. Manzanillo will tend to maintain a high moisture level throughout the ripening period. By monitoring fruit moisture levels from the beginning of ripening period, growers can utilise moisture content to indicate optimum harvest timing and determine if it is conducive to good extraction and separation efficiency.

Free fatty acids (FFA)

Free fatty acids is the simplest test and indicative of good harvesting and handling processes.

FFA are fatty acids which have broken away from oil molecules or triacylglycerols (TAGS – Fig. 1). Their presence indicates that degradation has occurred in the oil through poor handling during processing. Free fatty acids can influence the organoleptic value of the oil. Additionally, free fatty acids are water-soluble and may be lost during processing. Every batch of oil should be tested for free fatty acids. The IOC standard for free fatty acids in extra virgin olive oil is a maximum of 0.8%.



Figure 1. Process of oil hydrolysis (breakdown of TAG molecules) and release of free fatty acids by lipase enzymes present in the fruit.



Figure 2. Test kit for home testing of free fatty acid content (available from Wagga Wagga Agricultural Institute).

In samples analysed by NSW DPI during 2005, the average FFA was 0.2%, indicating that most Australian producers have no trouble meeting the IOC standard of $\leq 0.8\%$ for extra virgin olive oil. If the FFA is high, e.g. over 0.8%, it indicates that there has been fruit damage (frost, bruising), delays between harvest and processing or harvesting of over-ripe fruit. High FFA oils can be blended with low FFA oil to bring the FFA under a desired level. If FFA is high, there are likely to be other problems with the oil and blending may not be advisable. This may be the case if the fruit is frost damaged, as the oil will have a characteristic flavour as well as high FFA.

Setting a FFA level lower than 0.8% can provide a useful standard to ensure that growers provide high quality undamaged fruit that is not harvested too late (late harvest fruit will tend to have higher FFA).

Peroxide value

Peroxide value (PV) is an important test which growers should also carry out on every batch of their oil. This test relates to storage of the oil and measures chemical products that are produced through reaction with oxygen to ultimately cause rancidity.

Once the process of oxidation that leads to rancidity starts, it happens very quickly. There is a snowball effect wherein oxidation takes place and the oil then becomes rancid.

The IOC standard is ≤ 20 mEq O_2 /kg oil. In general, peroxide levels higher than 10 may mean less stable oil with a shorter shelf life. High levels at

the bottling stage are not a good start for long shelf life.

If low peroxide oil is blended with high peroxide oil it will result in the whole batch going rancid very quickly.

Factors that increase the peroxide value include high temperature, visible light and oxygen. Contact with metal surfaces, such as copper, can also catalyse oxidation of the oil. Damage to fruit and delays between harvest and processing are also a risk. Oil should be stored in cool, inert vessels, such as stainless steel or glass, away from the light. Oxygen should be removed by sparging with nitrogen prior to sealing the vessel.

Setting a low peroxide value standard will encourage producers to take care with fruit quality, harvest time, harvesting operations and processing to ensure that the oil meets the IOC standard. It also means that oils will be more stable and that if they are stored correctly, shelf life will be extended. (reasonable levels of antioxidants in the oil will also contribute to oil stability).

Fatty acid profile

The Fatty acid profile (FAP) is a measure of the proportions of individual fatty acids in the oil and is therefore an important part of the oil quality. The proportions of the different fatty acids can influence the stability of the oil as well as determining the nutritional value of the oil. Some fatty acids are considered better than others. For example, oleic acid – which acquired its name from olive oil – is the most desirable nutritionally. Linolenic acid, with three double bonds, is the most chemically reactive and therefore undesirable from the view of stability. Palmitic acid is a saturated fatty acid and is also undesirable. The IOC standard for fatty acids is shown in Table 1. The fatty acid profile is influenced by cultivar and environment.

Stability is influenced by the degree of saturation, with polyunsaturated fats such as linoleic and linolenic acid being many times more prone to oxidation than the mono-unsaturated fat, oleic acid. There is an inverse proportional relationship between oleic acid and linoleic acid. Oil with high oleic acid of 80%, will be low in linoleic acid (4–8%) while if oleic acid is low, say 65% then linoleic will be high (10–18%). Olive oil with high oleic acid is nutritionally preferable and potentially more stable than low oleic olive oil. The IOC standard for oleic is 55–83% but setting a standard in your area that promotes or rewards high oleic acid is not so simple. The reason for this is that climate, season and variety all influence the fatty acid profile. In general, cooler, high altitude environments will produce higher oleic acid levels than hot, low altitude environments. It is worthwhile to observe the range of oleic acid across a specific region

before setting standards for FAP. It is also worth having the FAP test done to detect cases where linolenic acid may exceed the 1.0% limit for EVOO.

Polyphenols

Polyphenols (PP) are strong antioxidants and are important for stability as well as the flavour characteristics of bitterness and pungency in olive oil. The amount of PP in the oil can vary from 0 to 1000 ppm or more. Usually the range is from 60 to 400. Oils can be categorised into 'Low', around 50–200; 'Mild', 200–400; and 'High', > 400 ppm. There are no international standards for PP and you will need to interpret test results for yourself. Caution is also needed as results can vary between laboratories. There are many different phenols in olive oil and high PP in one oil sample can cause excess bitterness but a similar level in another can give robust and balanced oil. In general oils with moderate levels of PP (200–400 ppm) can be easier to manage and market than very strong flavoured robust oils. Polyphenols are not essential for high quality olive oil. Delicate fruity oils with very low PP levels can be reasonably stable and highly marketable. The variety Leccino often has very low levels of PP but high levels of tocopherols which are also antioxidants. Early harvest of this variety can ensure a reasonable level of fruitiness and good stability despite low pungency and bitterness. Delaying harvest can lead to bland, flat oils with poor stability.

It could be worthwhile monitoring polyphenols levels as a guide to flavour intensity particularly early in the harvest period to make sure the levels are not too high. PP levels can also be used to determine a harvest period when PP are within a required range. The cost of testing is relatively high and it is not essential for IOC standards.

UV absorption

The UV absorption test is used by industry to identify oils which are old or which have been refined. The test measures changes in the structure of fatty acids, something which occurs during ageing or heating of oil. The test may be used by people who are purchasing the oil to ensure it is fresh and not adulterated with refined olive oil.

Other tests

In addition to the tests described above, there are other tests which may be required. If the oil is being purchased by somebody who does not know the background of the oil they may require a full set of tests to determine that the oil is authentic.

Authenticity testing

- Fatty acid profile
- Sterol composition
- Trans fatty acids – for refined oil
- Wax content – identifies pomace oil
- 2-position palmitic acid – for adulteration
- Triacylglycerol.

Tests for export

If the oil is being exported to Europe or Asia, there may be a requirement for any or all of the following additional tests as determined by the relevant customs departments.

- Moisture and volatiles
- Unsaponifiable matter
- Insoluble impurities
- Flash point
- Trace metals – iron (Fe), copper (Cu)
- Trace metals – lead (Pb), arsenic (As)
- Pesticides
- Halogenated hydrocarbons.

Sensory (organoleptic) tests

Sensory quality is the most important test to ensure the oil is acceptable for consumption.

The **sensory components** of olive oil are

- Aroma
- Flavour
- Pungency
- Bitterness.

Sensory assessment is based on the positive and negative descriptors of **olive oil sensory components**. Positive descriptors are fruity, green, citrus, spicy, fragrant, tropical, soft, over-ripe, bitter and pungent, and they are determined by the quality of fruit produced on the tree. Negative descriptors are caused by human error and include, fusty, musty, muddy, winey, metallic, rancid, burnt and others.

According to the IOC standards, extra virgin olive oil, for example, should not have any sensory defects and should have some fruitiness. It will be necessary to define the sensory quality of olive oils in more detail than this. To do this properly, the assessor will need to develop the capability to determine sensory quality consistently and to have a reasonably good knowledge of olive oil sensory qualities and styles. At the most basic level this would be a knowledge and ability to detect defects and faults. At a more advanced level it could mean an ability to identify oil for specific markets and the

capability to blend oils to meet customer specifications.

Sensory assessment begins with stipulated tasting conditions, then assessment of the aroma by sniffing followed by tasting to determine flavour, pungency and bitterness.

Panel. Because human perceptions of tastes and smells can be personal and subjective, sensory assessment is usually done by a panel of 8–12 people who are trained to IOC standards and led by a panel leader. The panel leader coordinates activities and educates members in perception, descriptors, etc.

Summary – tests that are important to growers

Free fatty acids. A low FFA level provides reasonable (though not absolute) assurance that the fruit was in good condition at processing time. Do it yourself: the test can be done outside an accredited laboratory if the correct FFA Kit equipment is used by trained personnel (see Fig. 2).

Fatty acid profile. Provides some assurance that the oil is EVOO and there is value in monitoring Oleic acid levels.

Peroxide value. Measure of the active oxygen in the oil and the potential to go rancid. High starting levels of PV are a bad sign.

Sensory. Essential.

Polyphenols. Not an essential test but highly useful; tells a lot about quality, stability and sensory character.

Exporting. It is necessary to have the full range of IOC and adulteration tests done on oils being exported to ensure they meet IOC standards. Besides the five tests above, when exporting, sterol composition (table 2) should also be tested. The recent discovery that the variety Barnea has naturally high levels of the sterol campesterol, exceeding the IOC level of 4 %, highlights the need to test for sterols.

Important – sampling protocols. Before taking samples for testing, obtain and follow accredited laboratory guidelines for sampling, packaging and dispatch of samples for testing.

Commercial testing service

The NSW DDPI NATA-accredited Olive Oil Testing Laboratory at Wagga Wagga specialises in the research and analysis of olive oil products and offers a commercial service to industry and service providers.

The Laboratory's analysis service is playing an increasingly important role by providing both chemical tests and sensory evaluation to verify the quality of Australian products. These tests underpin

Sterols – fingerprint	Content %
Cholesterol	≤0.5
Brassicasterol	≤0.1
Campesterol	≤4.0
Stigmasterol	< campesterol in edible oils
Delta-7-stigmasterol	≤0.5
β-sitosterol	} ≥93.0
delta-5-avenasterol	
delta-5-23-stigmastadienol	
clerosterol	
sitostanol	
delta-5-24-stigmastadienol	

Table 2. Allowable sterol content

the success of Australian producers, who are marketing Australian olive oil products into many overseas destinations, and ensure that olive oil products meet international standards in a global marketplace.

For more details on analysis service, contact the Customer Service Officer at DPI Wagga Wagga, Tel: 02 6938 1957

Growers must ensure their oil meets international standards. These quality standards, developed by the IOC, are detailed on the IOC Website, www.internationaloliveoil.org

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