INTERNATIONAL OLIVE COUNCIL

GUIDE FOR THE DETERMINATION OF THE CHARACTERISTICS OF OIL-OLIVES

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1. INTRODUCTION

This guide refers to the fruits of the cultivated olive tree (*Olea europaea* L.) intended for the production of virgin olive oil. Its purpose is to determine the characteristics of oil-olives, olive pomace and olive oil mill wastewater, to calculate the approximate processing yield and to define the parameters for identifying the most suitable harvest time for the purposes of this calculation.

2. DETERMINATION OF THE MATURITY INDEX AND OPTIMUM HARVEST TIME TO OBTAIN QUALITY VIRGIN OLIVE OIL

2.1. INTRODUCTION

Stone hardening is the first stage in the biological formation of the oil in olive fruits. From that point onwards, the oil forms and increases in volume as the fruit undergoes vegetative development until it peaks at a maximum level characteristic of each variety. The genuine sensory characteristics of the oil become apparent before the start of the ripening process. Regular samplings before the traditional ripening period provide useful information for obtaining the largest possible quantity of premium oil. Such sampling is designed to calculate the best time to start harvesting on an analytical basis and to learn about fruit development, oil content, extraction capacity, sensory properties and a number of analytical characteristics.

2.2. DEFINITION

The ripening process can be observed visually in olive varieties as they gradually change colour. The skin usually turns from deep green to violet and black. The colour and texture of the flesh also change during these stages as do the colour and sensory characteristics of the oil (Fig. 1).

Fig.- 1

Physico-chemical influence of ripening on the olive and olive oil

The following formula was developed at the *Venta del Llano* Experimental Station, IFAPA, Mengíbar (Jaén, Spain) to quantify the stages of ripening in olive. As can be seen from Figure 2, it is based on a scoring system for each stage of colouring of the skin and flesh.

MATURITY INDEX

CATEGORY 0:	SKIN COLOUR DEEP GREEN
CATEGORY 1:	SKIN COLOUR YELLOW-GREEN
CATEGORY 2:	SKIN COLOUR GREEN WITH REDDISH SPOTS ON < HALF THE FRUIT SURFACE. START OF COLOUR CHANGE
CATEGORY 3:	SKIN COLOUR WITH > HALF THE FRUIT SURFACE TURNING REDDISH OR PURPLE. END OF COLOUR CHANGE
CATEGORY 4:	SKIN COLOUR BLACK WITH WHITE FLESH
CATEGORY 5:	SKIN COLOUR BLACK WITH < HALF THE FLESH TURNING PURPLE
CATEGORY 6:	SKIN COLOUR BLACK WITH NOT ALL THE FLESH PURPLE TO THE STONE
CATEGORY 7:	SKIN COLOUR BLACK WITH ALL THE FLESH PURPLE TO THE STONE

THE MATURITY INDEX IS OBTAINED BY APPLYING THE FOLLOWING FORMULA WHERE A, B, C, D, E, F, G, AND H ARE THE NUMBER OF FRUITS IN EACH OF THE COLOUR CATEGORIES 0, 1, 2, 3, 4, 5, 6, AND 7 RESPECTIVELY:

$$\text{M.I.} = \frac{A0 + B1 + C2 + D3 + E4 + F5 + G6 + H7}{100}$$

This formula establishes the degree of maturity or maturity index of the sample. Further analyses are needed to determine the percentage moisture content; partial content of oil obtained by centrifugation on a fresh matter (f/m) basis; total oil content on a fresh matter (f/m) and dry matter (d/m) basis when obtained by solvent extraction, oil/solvent density or physical methods; extraction capacity; free acidity and sensory characteristics of the oil.

These data permit numerical identification of the stage of ripening and provide information on the composition and processing performance of the olives and the sensory characteristics of the oil. The sample data are plotted on a graph where the sampling dates are shown on the x-axis and the percentage d/m oil content is shown on the y-axis.

It takes at least four rounds of sampling to assemble the necessary information to identify the optimal time of ripening. It is advisable for sampling to start about one month before harvest traditionally begins and for it to be performed at intervals of 7–10 days.

In normal climatic conditions, d/m oil content will increase over time. Trace a line along the graph dots. When oil content increases less than in the preceding sampling and the line starts to dip, the olives can be considered to have reached their maximum oil content. Additionally, if the sensory characteristics of the olives display an intense fruity aroma reminiscent of fresh green olives, other green fruits, recently mown grass, artichokes etc. and a fresh fruity taste with a balance between bitterness, pungency and astringency, the extraction capacity is over 70% and the free acidity lies between 0.1 and 0.3%, this is the optimum point of ripening. Normally almost all the varieties have a maturity index between 3 and 4 at this point, making it advisable to start harvesting in order to obtain distinctive premium oils.

2.3.   OLIVE SAMPLING IN THE ORCHARD

The objective is to obtain a good indication of yield and to collect sufficient oil to allow weekly comparisons of fruit weight, yield and oil characteristics on the basis of a representative sample of olives from a specific orchard.

2.3.1. CHOICE OF A HOMOGENEOUS SET OF TREES

When walking through the plot, choose several trees with a comparable crop load of homogeneous maturity (colour). Choose at least ten trees if the trees on the plot are large; if they are small, choose at least twenty with the same crop load.

2.3.2. NUMBER OF OLIVES FOR COLLECTION PER TREE AND NUMBER OF TREES

To ensure that the sample is representative, the number of olives taken from each tree must be as small as possible whereas the number of trees must be as large as possible. As the tendency would be to reduce the number of trees and to increase the number of olives per tree on practical grounds, try to find a compromise. This job is made easier if you work with a large number of trees with a homogeneous crop load.

The rule is that the number of olives collected by the end of all the weekly collections must be insignificant for the tree.

The number of olives must be proportional to the size of the trees. If the trees are of comparable size (modern orchards) an identical number of olives can be taken from each one. If they vary a lot in size, as occurs in many old orchards, choose a different number of olives from each tree. However, in such cases, make sure to collect the same number of olives from each tree in each weekly collection.

The quantity of olives chosen will vary depending on the crop load, variety and degree of fruit ripening. For instance, one kilogram is sufficient if the olives are high-yielding and quite ripe. If the variety is low-yielding and the olives are very green, collect approximately 2 kg. The average is around 1.5 kg.

2.3.3. COLLECTION

For collection to be reproducible, harvest the olives at random, moving regularly around the tree. Pick the olives solely from the band lying at a comfortable height. Grasp a shoot and collect the fruits that fit into your hand, regardless of whether you pick only one or several fruits. Avoid taking the olives always from the same place (for instance always from the tip of the shoots). If you do not have enough olives after one round, repeat the exercise.

2.3.3.1.   *First sampling*

Once you are sure about the type of crop load for collection, start picking a specific quantity of olives (see below) from a tree located near the entrance to the plot. Mark the tree. If the crop load, size and fruit maturity of the trees are almost identical and they are all beside each other (for instance in the same row), move on to the next tree. If, on the contrary, the trees are scattered and a different number of olives has to be collected from each one, it is essential to note down the number of the tree and the number of olives to be taken from it (do this by painting the details on the tree or writing them down on a weather-proof label or in a notebook). Another option is to paint an arrow on the trunk pointing to the next tree for collection from the series.

Move on to the next tree and repeat the exercise.

Weigh the olives once you have collected what you roughly guess to be the desired quantity. If necessary, collect more olives from one or more trees.

2.3.3.2.   *Weekly collections*

The quality of the operation basically depends on making sure the weekly collections are similar. Always pick the olives in a very similar manner; collect the same quantity as in the preceding collection and in the same way from the trees. Preferably, the same person should always do the collections.

3.   SAMPLING PROTOCOL FOR THE DETERMINATION OF THE CALCULATED OIL YIELD

PURPOSE AND SCOPE

The purpose of this procedure is to specify the sampling method for determining the characteristics of oil-olives that are necessary to estimate their calculated oil yield. It is applicable to olives presented in packages as defined below or presented loose.

3.1.   REFERENCES

- ISO 7002/1986: Agricultural food products – Layout for a standard method of sampling from a lot.

- NF V 03-200/1966: *Produits de l'agriculture – Fruits et légumes en l'état : échantillonnage* (Agricultural products – Fresh fruits and vegetables: sampling).

- CAC/GL 50-2004: General guidelines on sampling.

- Commission Regulation (EC) No 1148/2001 of 12 June 2001 on checks on conformity to the marketing standards applicable to fresh fruits and vegetables.

3.2.   DEFINITIONS

- **Consignment:** quantity of olives delivered at one time. It may consist of one or more lots or parts of lots.

- **Lot:** definite quantity of olives, delivered at one time, presumed to be of the same characteristics (same variety, same ripeness, same size, same type of packing, etc.).

- **Sampling plan:** planned procedure for the selection, collection and separation of samples from a lot in order to obtain the information needed to take a decision thereon.

- **Sampling:** procedure used to draw or form a sample.

- **Representative sampling:** sampling where the sample is collected in such a manner as to reflect as accurately as possible the lot properties concerned.

- **Primary sample:** package of olives taken from the lot, or, in the case of bulk produce, a quantity taken from a point in the lot.

- **Package:** individually packaged part of a lot, including contents. The packaging is conceived so as to facilitate handling and transport of a number of sales packages or of products, loose or arranged, in order to prevent damage by physical handling and transport.

- **Global sample**: quantity of olives formed by collecting primary samples from the lot.

- **Reduced sample:** quantity of olives taken from a global sample by reduction without changing its composition.

- **Laboratory sample:** definite quantity of olives taken from a global sample or reduced sample and sent to the laboratory.

- **Test sample:** sample prepared from the laboratory sample according to the procedure specified in a testing method and from which test portions are removed.

3.3. GENERAL PROVISIONS

3.3.1. Sampling is performed for laboratory verification of certain characteristics of the olives delivered. Samples should be taken at random. The aim of sampling should be established beforehand by identifying the characteristics intended for inspection.

3.3.2. Sampling should be performed in such a way that the primary samples represent all the characteristics of the lot. After setting aside the damaged parts of the lot (crates, sacks, etc.), each of the parts, damaged and undamaged, should be sampled.

3.3.3. Sampling should be performed by persons with training in sample collection techniques.

3.3.4. A sampling report should be written upon the completion of sampling (see section 3.4.2.4).

3.4. METHODS OF SAMPLING

3.4.1. Identification and preparation of the lot for sampling

A separate sample is taken from any lot that is to undergo analysis. The lot is identified from its marking or from accompanying documents.

The lot should be prepared for sampling in such as way as to enable smooth, swift collection. Samples should be taken by the parties concerned or by a representative authority.

Each lot should be sampled separately; however, if the lot has been damaged during transport, the damaged parts of the lot (crates, sacks, etc.) must be set apart and sampled separately from the undamaged parts. Likewise, if the consignee does not consider the consignment to be homogeneous, even if the dispatcher has not notified so, it should be divided into homogeneous lots and each lot will be sampled after agreement by the seller and vendor unless the latter decides otherwise.

Care should be taken during sampling and sample preparation to prevent any alteration that might modify or affect the tests or the representativity of the global sample.

3.4.2. Sampling plan

The sampling procedure applied should ensure that the global sample is representative of the lot intended for inspection.

3.4.2.1 *Primary samples*

Primary samples should be taken at random from different places and at different levels of the lot.

For olives which can be assumed to be distributed uniformly inside a lot, it is sufficient to take three primary samples per lot to form the global sample. The lot number should be stated. The weight of the primary sample may range from 500 to 700 g in order to obtain a global sample of at least 1.5 kg.

Table I specifies the number of packages or units that has to be collected to form the global sample if the lot is made up of separate packages (for instance, crates).

TABLE 1

Number of primary samples to be taken to form the global sample when the lot is made up of separate packages

Number of packages or units in the lot or sub-lot	Number of primary samples to be taken
From 1 to 25	1
From 26 to 100	approximately 5%, at least 2
More than 100	approximately 5%, 10 at the most

The weight of the primary sample varies according to the number of primary samples forming the global sample, which must weigh 1.5 kg. As a guideline, the primary sample may weigh from 200 to 1 500 g.

3.4.2.2 *Preparation of the global or reduced sample*

The global sample is formed by assembling and mixing the primary samples. The reduced sample is obtained, if necessary, by reducing the global sample. The laboratory sample is drawn from the global sample or the reduced sample.

3.4.2.3 *Laboratory samples*

Three identical samples should be taken from the homogenised global sample for inspection, appeal or arbitration purposes.

The size of the laboratory samples will depend on the laboratory tests that are to be performed.

Each sample should be placed in a clean container made of an inert material that provides suitable protection from changes in moisture, the risk of contamination and transport damage.

Each sample should be sealed and identified (label) at the sampling site. Identification should be legible and permanent and should contain the following information in particular:

(a) Product name (variety, if any);

(b) Name of dispatcher;

(c) Place of sampling;

(d) Date of sampling;

(e) Lot and sample identification mark (shipment note, transport vehicle number, storage site);

(f) Number of sampling report;

(g) Name and signature of sampler;

(h) List, if any, of the laboratory tests required.

After it is formed, the laboratory sample should be dispatched as quickly as possible to its end destination.

The laboratory sample should be stored and transported in conditions preventing any modification of the product. It is therefore desirable for testing to be performed as soon as possible after sampling. No more than 48 hours should elapse between sampling and laboratory testing. If this is not the case, the sample should be kept refrigerated.

3.4.2.4 *Sampling report*

A report should be drawn up for each sample collection. This report should permit clear identification of each lot and should state the following in particular, where appropriate:

(a) Product name (and variety, if any);

(b) Lot consignee;

(c) Place and date of dispatch and receipt;

(d) Name and address of dispatcher;

(e) Place, duration and conditions of lot storage and specification of transport vehicle;

(f) Date and time of sampling;

(g) Atmospheric conditions during sampling;

(h) Lot size or number of packages;

(i) Purpose of sampling and time limit between sampling and testing in normal conditions;

(j) Apparent homogeneity of the lot, proportion of parts displaying other damage;

(k) Surname and first name of the parties present at the sampling;

(l) Number of laboratory samples formed;

(m) Surname and first name of the sampler(s).

The report should also state whether a method other than the one laid down in this procedure has been used.

4. DEFINITIONS

Olive: fruit of the olive tree (*Olea europaea* L.).

Olive paste: product obtained after crushing the olives at the mill. Crushing is one of the processes performed prior to oil extraction. Olive paste is made up of a solid fraction, a fatty or oily fraction, an aqueous fraction and a fraction of volatile components.

Virgin olive oil: Product obtained at the mill from the fruit of the olive tree (*Olea europaea* L.) solely by mechanical or other physical means under conditions, particularly thermal conditions, that do not lead to deterioration of the oil, and which has not undergone any treatment other than washing, decantation, centrifugation and filtration.

Virgin olive oil is classified into two large groups according to its physico–chemical and organoleptic characteristics:

- Virgin olive oil *fit for consumption* as it is:

  o *Extra virgin olive oil*: virgin olive oil which has a free acidity, expressed as oleic acid, of not more than 0.8 grams per 100 grams and the other characteristics of which correspond to those laid down for this category in the trade standard.

  o *Virgin olive oil:* virgin olive oil which has a free acidity, expressed as oleic acid, of not more than 2.0 grams per 100 grams and the other characteristics of which correspond to those laid down for this category in the trade standard.

  o *Ordinary virgin olive oil*: virgin olive oil which has a free acidity, expressed as oleic acid, of not more than 3.3 grams per 100 grams and the other characteristics of which correspond to those laid down for this category in the trade standard.

- Virgin olive oil *not fit* for consumption as it is:

  o *Lampante virgin olive oil:* virgin olive oil which has a free acidity, expressed as oleic acid, of more than 3.3 grams per 100 grams and/or the organoleptic characteristics and other characteristics of which correspond to those laid down for this category in the trade standard.

Olive pomace: solid or dough-like by-product of the extraction of virgin olive oil generated at the mill. It is made up of residual olive paste containing a percentage of water and residual oil which varies depending on whether the processing method entails pressing, two or three-phase centrifugation or optional second centrifugation, the operating methods employed and the expertise of the personnel operating the extraction machinery.

It is usually used by the extraction industry to obtain crude olive-pomace oil or for other purposes.

Olive rinse water: water from the closed-circuit washers used to separate solid impurities (mud, sand, stones, etc.) from the olives.

Wastewater: liquid by-product of the extraction of virgin olive oil generated at the mill. It is made up of the vegetation water contained in the olive fruits and the water added during oil processing. It contains a percentage of solid matter from the paste, which varies depending on whether the production method entails pressing, two or three-phase centrifugation or optional second centrifugation. It may contain a small percentage of residual oil depending on the extraction technology employed.

Centrifuge rinse water: liquid by-product of the extraction of virgin olive oil generated in the two-phase system comprising a small percentage of vegetation water contained in the olive fruits and the rinse water used in the vertical centrifuge. It contains small percentages of solid matter from the paste and residual oil.

Paste moisture (Mst): fraction of the olive paste comprising vegetation water and volatile components of the olives, determined as a percentage:

$$Moisture\ (\%) = \left| \frac{weight\ of\ (water + volatile\ matter)}{weight\ of\ paste} \right| \times 100$$

$$Weight\ of\ paste = weight\ of\ oil + weight\ of\ solids + weight\ of\ (water + volatile\ matter)$$

Dry matter (DM): fraction of the olive paste comprising the solid and oil fractions, determined as a percentage:

$$Dry\ matter\ (\%) = \left| \frac{(weight\ of\ solids + weight\ of\ oil)}{weight\ of\ paste} \right| \times 100$$

When the moisture content is known, a very simple calculation is required to calculate the dry matter content:

$$DM = 100 - Mst$$

Oil-free dry matter (OFDM): fraction of the olive paste comprising the solids, determined as a percentage:

$$OFDM\ (\%) = \left| \frac{weight\ of\ solid\ fraction}{weight\ of\ paste} \right| \times 100$$

When the moisture content and total oil content are known, it is also very simple to calculate the oil-free dry matter:

$$OFDM = 100 - Mst - TOC$$

Total oil content (TOC): fraction of the olive paste comprising the oily fraction, determined as a percentage:

$$TOC(\%) = \left| \frac{weight\ of\ oil}{weight\ of\ paste} \right| \times 100$$

Oil content on a dry matter basis (DMO): ratio of the oily fraction of the olive paste to the dry matter.

This parameter is used extensively because it eliminates the distortion created by moisture, which fluctuates greatly according to the variety of olives, the water content and the extraction method employed. It is determined as a percentage:

$$DMO = \left[\frac{weight\ of\ oil}{(weight\ of\ paste - weight\ of\ water)} \right] \times 100 \quad (1)$$

If we take the formula for total oil content,

$$TOC = \left| \frac{weight\ of\ oil}{weight\ of\ paste} \right| \times 100 \quad (2)$$

and divide equation (2) by (1):

$$\frac{TOC}{DMO} = \frac{(weight\ of\ paste - weight\ of\ water)}{weight\ of\ paste} \times 100$$

we obtain:

$$\frac{TOC}{DMO} = 1 - \left| \frac{weight\ of\ water}{weight\ of\ \quad paste} \right| \quad (3)$$

However, the term

$\frac{weight\ of\ water}{weight\ of\ paste}$ is *Mst*, the moisture expressed relative to one. Hence,

$$TOC = DMO(1 - Mst) \quad \text{or}$$

$$DMO = \frac{TOC}{(1 - Mst)}$$

Processing losses: fraction of the oil content not obtained directly during the extraction process. This fraction is chiefly found in the olive pomace, and to a much lesser extent in the wastewater. The percentage of oil contained in these by-products depends on:

- Variety of olives
- Fruit water content
- Extraction method and working conditions or operating techniques
- Application of second centrifugation
- Technical expertise of personnel

Processing yield (PY): fraction of the oil content obtained directly during the extraction process. It is arrived at by deducting losses from the total oil content:

$$PY = TOC - processing\ losses$$

Processing yield depends on the mill and varies according to:

- Raw material (variety of olive, moisture content and degree of ripeness)
- Type of extraction process
- Efficiency of the extraction process
- Technical expertise of personnel

It is worked out easily by the mill at the end of each operation and/or each season in the following manner:

$$PY\ (\%) = \left| \frac{kg\ of\ oil\ obtained}{kg\ of\ olives\ crushed} \right| \times 100$$

Hence, it is private mill information.

Calculated processing yield (CPY): theoretical estimate of mill processing yield. As we have already seen, the largest percentage of losses occurs in the olive pomace. The equation can therefore be simplified by eliminating the losses that occur in the wastewater, in which case the CPY is obtained as follows:

$$CPY = TOC - pomace\ losses - wastewater\ losses$$

Pomace losses: oil occluded in the oil-free dry matter. It represents the oil lost in the olive pomace and is expressed as a percentage:

$$Pomace\ losses = \left| \frac{OC_{pomace}}{OFDM_{pomace}} \right|$$

The amount of oil lost in the oil-free dry matter will be:

$$Pomace\ losses = \left| \frac{OC_{pomace}}{OFDM_{pomace}} \right| \times OFDM_{paste}$$

As $OFDM_{pomace}$ is ($100-Mst_{pomace}-OC_{pomace}$), it follows that:

$$Pomace\ losses = \left| \frac{OC_{pomace}}{(100 - Mst_{pomace} - OC_{pomace})} \right| \times OFDM_{paste}$$

Hence, the calculated processing yield is:

$$CPY = TOC - \left| \frac{OC_{pomace}}{(100 - Mst_{pomace} - OC_{pomace})} \right| - OCW \times OFDM_{paste}$$

The data for OC_{pomace} and Mst_{pomace} should be consulted in the relevant tables because they depend on the variety of olive, the crop year, the method of extraction employed and extraction management.

Wastewater losses: oil-free dry matter in the wastewater.

Oil content of wastewater: residual oil in the wastewater. This value is used to check the centrifuge is working and is expressed as a percentage:

$$OCW\ (\%) = \left| \frac{weight\ of\ oil}{weight\ of\ wastewater} \right| \times 100$$

Total solids in wastewater: solid, insoluble fraction in suspension in the wastewater. This value is used to check the centrifuge is working and is expressed as a percentage:

$$TSW\ (\%) = \left| \frac{weight\ of\ solids}{weight\ of\ wastewater} \right| \times 100$$

Valuate (to): To place a value on something. To judge or estimate the amount a person deserves for their work, and to pay them the corresponding amount.

Fair price: Value assigned to something after valuating it.

According to the last two definitions, the agreed selling price should be fair to both parties. Consequently, the following factors should be taken into account:

- Olive quality
- Physical, hygiene and health status of the olives
- Moisture content of olives
- Total oil content
- Processing yield quoted by the processor
- Calculated processing yield quoted by the olive grower
- Oil quality
- Processor costs
- Market price of virgin olive oil

5. CALCULATIONS

The moisture content of the olive paste is defined as:

$$Paste\ moisture(\%) = \frac{Water\ weight}{Paste\ weight} \times 100$$

where the weight of the paste is:

$$Paste\ weight = Oil\ weight + Solids\ weight + Water\ weight$$

The oil content of the olive paste on a fresh matter basis is determined as a percentage as:

$$Fresh\ matter\ oil\ content\ (FMO)\ \% = \frac{Oil\ weight}{Paste\ weight} \times 100$$

It is determined as follows on a dry matter basis:

$$Dry\ matter\ oil\ content(DMO)\ \% = \frac{Oil\ weight}{Paste\ weight - Water\ weight} \times 100$$

When both formulas are divided, we obtain:

$$\frac{FMO}{DMO} = \frac{Paste\ weight - Water\ weight}{Paste\ weight} = 1 - \frac{Water\ weight}{Paste\ weight} = 1 - Mst$$

Where Mst is the moisture expressed relative to one:

$$FMO = DMO \times (1 - Mst)$$

The oil-free dry matter (weight of solids) is defined as:

$$OFDM \ (\%) = 100 - Moisture - FMO$$

The oil content of the pomace is the main processing loss. When referred to moisture contents of 25% (pressing), 55% (three-phase centrifugation) and 75% (two-phase centrifugation), it must not exceed 6% oil of the fresh matter when the olives are pressed, 3.5% when they undergo three-phase centrifugation and 4% when they undergo two-phase centrifugation.

Consequently, in such conditions, the OFDM of the pomace ($OFDM_{pomace}$) is:

Pressing : $OFDM = 100 - 25 - 6 = 69$
Three-phase centrifugation: $100 - 55 - 3.5 = 41.5$
Two-phase centrifugation: $100 - 75 - 4 = 21$

Hence, the percentage of oil in the oil-free dry matter is:

Pressing: **6/69 = 0.087**
Three-phase centrifugation: 3.5/**4.1 = 0.08**
Two-phase centrifugation: 4/**21 = 0.19**

Hence, the approximate processing yield will be:

Pressing:

$$APY_{pres \, \sin g} = TOC - 0.087 \times OFDM$$

Three-phase centrifugation:

$$APY_{3\,ph} = TOC - 0.08 \times OFDM$$

Two-phase centrifugation:

$$APY_{2\,ph} = TOC - 0.19 \times OFDM$$

It is termed 'approximate' processing yield because, as we have seen, it depends on the oil and moisture content of the pomace.

It could be generalised as follows:

$$APY = TOC - \left(\frac{OY_{pomace}}{100 - Mst_{pomace} - OY_{pomace}} \right) \times OFDM_{paste} - ocw$$

The actual processing yield is:

$$PY_{actual} \, (\%) = \frac{kg \; oil \; obtained}{kg \; olives \; crushed} \times 100$$

6.   ANALYTICAL METHODOLOGY

The analytical determinations performed most frequently to monitor and assess the composition of olives intended for oil production are:

- Maturity index;
- Moisture content;
- Partial oil content;
- Total oil content on a dry matter (d/m) basis;
- Total oil content on a fresh matter (f/m) basis;
- Oil-free dry matter;
- Emulsions;
- Extraction capacity;
- Calculated processing yield.

6.1.   Maturity index

Follow the methodology specified in section 2 for this determination.

6.2 .   Moisture content (Mst)

To determine the moisture content, crush the sample of olives in a laboratory hammer mill (Fig. 3) fitted with a screen with hole openings of 3.5–4.0 mm Ø. Homogenise the crushed olive paste without separating the oil. Place 60–70 g of the paste in a pre-calibrated dish, place the sample in a hot air circulation oven at 105 ºC. Dry for approximately 8–10 hours to constant weight. Cool the sample in a desiccator and calculate the moisture loss by applying the appropriate formula.

6.3.   Emulsions

Emulsions are the interface that occurs between the water and oil phases through the action of an emulsifier.

Pectins are the usual emulsifiers in olives. They exert this effect when the fruits are sound and at the first stage of ripening, they have a high moisture content, usually above 55%, and they are crushed straight away.

Emulsions develop and/or increase during crushing depending on the factors mentioned above, as well as on crusher speed and screen aperture diameter. In almost every case, the degree of emulsification is directly related to the degree of moisture.

An emulsified olive paste is fluid in texture and has a very uniform appearance (Fig. 17). As a rule it shows little resistance to the movement of the malaxator (mixer) blades. It looks like a fruit purée. The oil does not float on the surface during malaxation and the blades are always coated with olive paste.

The basic reason for the small quantity of oil separated is that the droplets of oil released during crushing are immediately enveloped by the lipoproteins in the vegetation water which prevent them from coalescing (Fig. 18).

Fig. – 17

Emulsified olive paste

Fig. – 18

The lipoproteins in the vegetation water prevent the droplets of oil from coalescing.

When this kind of emulsion occurs, the olive paste is said to be "difficult" or emulsified. In rheological terms it is a non-Newtonian fluid or Bingham body. When stress is applied to the emulsified olive paste it behaves like a plastic flow semi-solid. This kind of olive paste tends to cause problems in all the oil production processes. Less oil is separated, which lowers the oil processing yield, and the olive pomace contains more oil.

The values for the following analytical determinations are affected when this type of olive paste occurs: partial oil content, which is usually lower due to centrifugation; total oil content of the pomace, wastewater and rinse water, which tend to be higher because of poorer extraction; extraction capacity, which tends to be lower; and calculated processing yield, which does not tend to coincide with the actual yield because it is generally based on total oil content methods that do not detect or assess the degree of emulsion.

Research into ways of reducing or eliminating emulsion formation without affecting oil characteristics have been conducted at the experimental oil mill of the Seville Fats & Oils Institute (*Instituto de la Grasa*, C.S.I.C., Seville, Spain). The best research results have been obtained when using microtalc as a processing aid at the start of malaxation in accordance with the existing rules and regulations.

For processing purposes, a dose of 0.5–3.0% is used depending on the moisture content and difficult characteristics of the olives. Centrifugal determination of the partial oil content is the method of analysis that permits identification and quantification of the flow problems in "difficult" pastes by comparing untreated pastes with pastes treated (Fig. 19) with different amounts of talc. The test results are applied in the production process and largely help to correct processing problems and to obtain excellent results in the determination of the oil yield and in the control of the pomace, wastewater and rinse water.
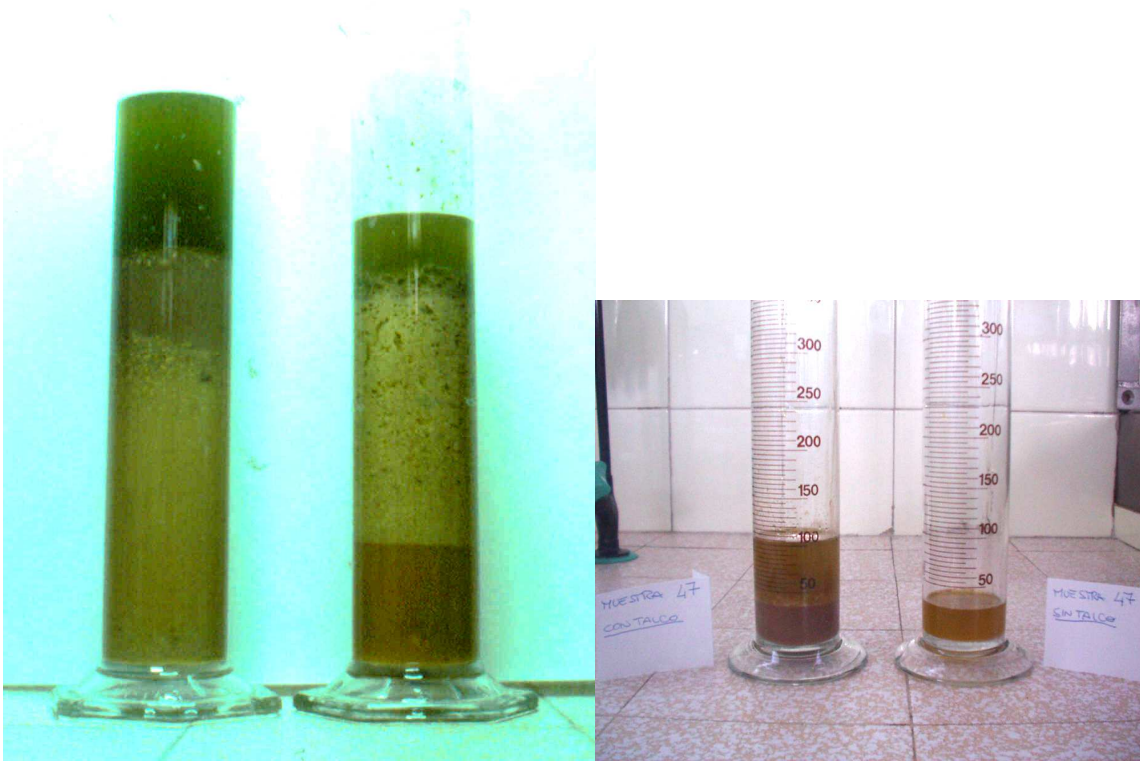
Fig.- 19

**Effect of talc treatment in the laboratory
(left: paste treated with talc; right: paste not treated with talc)**

6.4. Partial oil content (POC)

A laboratory centrifugation facility is needed for this determination, made up of a crusher (Fig. 3), a thermo-malaxator (Fig. 4), a basket centrifuge (Fig. 5) and 500-ml test tubes.

Fig. - 3

Fig. 3 – Hammer crusher

Fig. - 4

Fig. 4 – Thermo-malaxator

Fig. 5 - Centrifuge

Start by grinding a sample of at least 1 kg of olives in the crusher. Homogenise the paste without separating the oil. Weigh 600 g into the malaxator tub, insert in the thermo-malaxator at a bath temperature of 30 ºC and mix for 20 minutes. If the paste emulsifies when malaxation begins, add 1.7% talc* . Repeat if the emulsion persists. After 20 minutes, add 200–300 ml of water at 100 ºC depending on the texture of the paste. Mix for a further 10 minutes.

Pour the paste into the centrifuge. Centrifuge at 3 500 rpm for 2 minutes and collect the separated liquids in a test tube. Add 100 ml of water at 100 ºC to the centrifuge. Centrifuge the paste for a second time to remove any residual oil. Add any separated liquids to those already contained in the test tube and leave to settle for 1 hour.

Next measure the volume of oil, convert the reading to weight and determine the ratio between the weight of the oil and the weight of the paste. After the appropriate calculations, the end result is the percentage partial oil content.

6.5.   Oil content on a dry matter basis (DMO)

This can be calculated by the **Soxhlet** (Fig. 6) and **Nuclear Magnetic Resonance (NMR)** methods (Fig. 7). The Soxhlet method is a standard reference method for other methods and is applied to crushed, dried olive paste .

———————————

*N.B. If inert processing aids are not used, significant differences may be noted in the actual yield at the mill.

After obtaining this piece of data, place the dried paste in a covered blade crusher to break up the solids quickly without separating the oil. Weigh 20–30 g of the paste and place it in an extraction cartridge. Place the cartridge in the extractor. Use n-hexane as the solvent. Extract the sample by reflux for a minimum of 5–6 hours. Extraction should never last less than 6 hours if the olives have been picked at the start of the season. Next distil the oil–solvent mix in the same equipment and remove the residual solvent from the oil by placing it in a hot air circulation oven for at least 3 hours.

Fig. - 6

Fig. 6 – Soxhlet apparatus

Calculate the extracted oil contained in the solvent-free flask and apply the appropriate formulas to work out the total oil in dry matter. This method can be applied for the same determination in olive pomace, olive oil wastewater and centrifuge water.

$$DMO = \left| \frac{weight\ oil}{weight\ drypaste} \right| \times 100$$

The **NMR** method entails exciting the proton content of the sample. On being subjected to impulses from a magnetic field, the energy emitted is compared with the energy produced by a series of patterns for known quantities of oil. The system interprets the comparison plotted by the different calibration curves, determines the quantity of oil and relates it to the percentage of sample taken. The sample must be practically dry if the measure is to be very reliable; only a small residual amount of moisture is allowed.

Fig. - 7

Nuclear Magnetic Resonance (NMR)

Take between 14 and 40 g from a sample of crushed olives and place on a pre-calibrated dish or plastic film (Fig. 8) resistant to the oven temperature. Process in the oven in the same way as for the determination of the moisture content. By the end of this operation the fresh and dry weight of the sample will be known.

Remove the whole of the dry sample from the dish, if used. Wrap it in the plastic film and mould it into a malleable ball shape (Fig. 9).

Place the sample in the measuring tube. Make sure it is no more than 3 cm high (size of the measurement port) so that the whole of the sample is analysed. Insert the tube in the measuring apparatus (Fig. 10) and enter the reference and dry weight of the sample. The apparatus indicates the percentage oil content on a dry matter basis. If the fresh weight of the sample is entered at this point, the apparatus gives the percentage oil content on a fresh weight basis.

This method can be used to perform the same determination on olive pomace (with the appropriate calibration curve).

N.B. In principle, NMR data for oil content tend to be slightly higher than Soxhlet data.

Fig. - 8

Sample of crushed olives

Fig. - 9

Dry sample

Fig. – 10

The sample is placed in the measuring tube

6.6. – Total oil content on a fresh matter basis (FMO)

Two methods are currently available to determine this content in fresh, crushed olive paste. The first uses the **Autelec** system (Fig. 11), which performs the determination by comparing the densities of the oil/solvent mix and the solvent at a controlled temperature. Heptane is the solvent used.

Fig. – 11

Autelec system

The system is made up of an apparatus like that shown in Figure 11 for preparing and measuring the sample. Weigh 40 g of paste in the cylindrical beaker. Measure a specific amount of anhydrous sodium sulphate as a dehydratant. Add a set amount of heptane solvent. Next insert the cylinder inside the apparatus to break up and mix the olive paste with the solvent. Agitate for two minutes (Fig. 12).

Fig. – 12

Agitator

Fig. - 13

Filtration of the oil/solvent mix from the solids in the agitator

Then separate the oil/solvent mix from the solids through a filter placed on the upper part of the agitator (Fig. 13).

Place the filtered solvent/oil mix in the chamber of the measuring equipment where its density is calculated and compared with established calibration curves. The percentage total oil content on a fresh matter basis is obtained after each measurement.
This method can also be applied to the olive pomace and dried paste.

Another system known as Near Infrared Transmission or **NIT** is based on the spectrum produced by the oil dispersed in the olive paste when it is subjected to radiation at wavelengths in the near infrared range.

The determinations are compared against a wide range of spectra for paste containing differing correctly quantified amounts of oil which are used as calibration models.

The system uses a monochromator–detector (Fig. 14) inside which the sample of crushed paste is placed on a dish. The dish is made of a transparent material to allow the infrared rays to cross through the sample. The apparatus is fitted with a crown gear allowing 360º rotation (Fig. 15) so that measurements can be taken at different parts of the sample to obtain an average value.

A computer is used to enter the sample reference, to give commands and to collect the analytical data generated (Fig. 16). The system determines the moisture content and the oil content on a fresh matter basis. The possibility of incorporating the calculation of the acidity is also being explored. The same determinations can be performed on olive pomace if the proper calibrations are used.

Fig. - 14

Monochromator–detector

Fig. - 15

Crown gear permitting measurement of different areas of the sample

Fig. – 16

Computer

To obtain the **fresh matter oil content (FMO)** according to the testing methods outlined in section 6.6. it is necessary to know the moisture content (Mst) and to apply the formula:

$$FMO = \frac{(100 - Mst) \cdot DMO}{100}$$

To obtain the **dry matter oil content (DMO)** according to the testing methods outlined in section 6.5 it is necessary to know the moisture content (Mst) and to apply the formula:

$$DMO = \frac{100 \cdot FMO}{100 - Mst}$$

6.7. Oil-free dry matter (OFDM)

To calculate this value it is necessary to know the moisture content (Mst) and the fresh matter oil content (FMO) and to apply the following formula:

OFDM = 100 – Mst – FMO

6.8. Extraction capacity (EXT)

This determination is intended to quantify the extent to which the oil contained in the olive fruits can be extracted according to their ripening and compositional characteristics. It therefore gives useful information on the progress in ripening and the extent to which the oil can be extracted in the oil production process.

For this calculation it is necessary to determine the ratio of the partial oil content to the total fresh matter oil content, as determined by any of the methods cited, by applying the following formula:

$$EXT = \frac{POC}{FMO} \times 100$$

7. DETERMINATION OF THE CALCULATED PROCESSING YIELD OF OIL-OLIVES

One of the most important features of the world olive oil industry from both the economic and social standpoint is the system of making virgin olive oil. Three major factors central to the obtention of a quality product have to be considered to understand the full scope of this question:

- Raw material, i.e. olives;
- Extraction system and technology;
- Extraction management.

The raw material is the major primary determinant of the quality of virgin olive oil because it is impossible to produce a good product if the source olives are not sound. Consequently, the potential quality of the olive fruits is the first point to consider when studying the quality of virgin olive oil.

Numerous factors affect the olive fruits and should be controlled by olive growers to the extent possible. Growers should be aware of the fact that potential quality will depend on factors such as:

- Climate;
- Pests and diseases;
- Harvesting methods;
- Moisture content;
- Transport to the mill;
- Form and duration of pre-extraction olive storage.

This potential quality will also be a decisive factor in setting a fair price for the olives, which is also influenced by their total oil content.

Setting a fair price for the olives sold by the olive grower to the oil oprocessor is one of the most prominent, social balancing mechanisms in the olive sector. The agreement reached by growers and oil processors clearly has economic repercussions and must therefore be fair and take into account the interests of both parties. Objective criteria are needed to evaluate those interests, one of which is obviously and perhaps most importantly the oil contained in the olives. For the olive grower, this means the total oil content of the olives while for the processor it means the content that can actually be extracted.

The volume of oil that can be extracted from each batch of olives depends on the moisture content and how the olive oil producer manages the processing system as well as on several other factors that have to be controlled. Irrespective of the processing method employed, the common starting point for the agreement between growers and processors is the total oil content of the olives. However, the price of the olives cannot be based absolutely on this content because losses occur during extraction and have to be evaluated and reflected in the price.

The maximum volume of oil obtainable at the mill is known as the processing yield. This parameter is unique to each mill because it depends on the efficiency of the extraction equipment, the operating method employed, the professional expertise of the equipment operators and, naturally, the raw material. It is only known to mill managers at the end of the extraction process or at the end of the production season when they know the amount of oil obtained and the quantity of olives crushed. Olive growers do not know this value in advance. They therefore have to estimate mill processing yield by performing specific analyses on the olives to determine their macroscopic characteristics and by tabulating and drawing on data from preceding crop years for the mean estimated losses during processing. Using this information, a very simple theoretical model can be applied to calculate approximately the processing yield at the mill. This calculated processing yield serves as a guide for olive growers and olive oil processors when reaching an agreement over a specific batch of olives.

The purpose of this document is to list the analytical determinations applicable to olive fruits and to the by-products generated in the production process and to provide guidance on two points:

- The evaluation of a fair price for batches of olives;

- Very useful specific parameters to enhance the efficiency of processing plants.

Calculated processing yield (CPY) according to the Abencor method and the total oil content method

If an Abencor centrifugal system is available, at least two methods of analysis have to be applied to assess in advance or at any other time the amount of oil that can be extracted from a batch of olives and to evaluate their extraction performance:

(1) The Abencor–MC2 centrifugal system, which provides data on olive performance during crushing, emulsification, addition of processing aids, malaxation, centrifugal solid–liquid separation, liquid–liquid separation via settling and partial oil content.

(2) Any system determining total oil content on a fresh matter basis according to the following formulas.

$$CPY = FMO \times 0.82 - 0.2 \times (Mst - 56)$$

and

$$CPY = FMO - (0.085 \times OFDM - (0.035 \times Mst)$$

Comparative results for processing yields obtained at facilities equipped with the Abencor–MC2 system have revealed that the yield obtained according to this system is always between 1.5 and 2 points lower than the actual processing yield.

When the olive paste forms an emulsion but is not treated appropriately with processing aids, the FMO is not correct and must not be used.

When the olives have minor emulsion problems and are treated with processing aids (1.7%), the calculated FMO is 0.5–1 points lower than the actual FMO.

When the olives have medium emulsion problems and are treated with processing aids (3.4%), the calculated FMO is around 0–0.5 points below the actual FMO.

When the olives have serious emulsion problems and are treated with processing aids (more than 3.4%), the calculated FMO is lower than or equal to the actual FMO.

All these experimental findings indicate that, to estimate processing yield, the above amounts have to be added to the FMO value according to the degree of emulsification, which is directly linked to the level of moisture.

To collect information on the composition of the olive fruits and to ensure more accurate data it is advisable to determine the FMO according to any of the existing physical and chemical methods. It should be remembered that according to experimental results, any of the methods applied gives values that are considerably higher than the values for actual processing yield and which usually lie between 2.5 and 5.5 points depending on the method used.

To arrive at the calculated processing yield, it will be necessary to deduct the experimental value obtained by the method concerned from the FMO value.

The most rational approach to estimate the calculated processing yield would therefore appear to be to use the two proposed methods, to apply the suitable correction factors for each one and to work out the average of the two data.

However, as this could cause problems in terms of method availability, time and cost, it is proposed applying the following formula to compute the calculated processing yield from the total oil content

$$CPY = FMO \times 0.82 - 0.2 (Mst - 56)$$

Experiments have demonstrated that this formula is to be recommended when the olives have a moisture content of more than 50%. It has been obtained experimentally on the basis of the **oil content on a fresh matter basis (FMO)** and normal processing losses due to the oil contained in the by-products. It also allows for the losses generated by extreme moisture contents deviating from the optimal content, which has been set at 56 per cent.

For olives with a moisture content of less than 50%, it has been found that the following formula is to be recommended:

$$CPY = FMO - (0.085 \times OFDM) - (0.035 \times Mst)$$

This formula was tested at the experimental oil mill over four olive crop years (2005/06; 2006/07; 2007/08; 2008/09). The next chart plots the data obtained in the 2005/06 crop year for 85 batches of 2–12 tonnes of olives processed in a two-phase production line with a capacity of 5 tonnes per hour. Each lot underwent separate checks.

The graph shows the moving average for the nine-data-point subsets of a full set of 86 data for the moisture content (M), partial oil content (POC), fresh matter oil content (FMO), actual processing yield (APY_l) and calculated processing yield (CPY) of the lots that were processed.
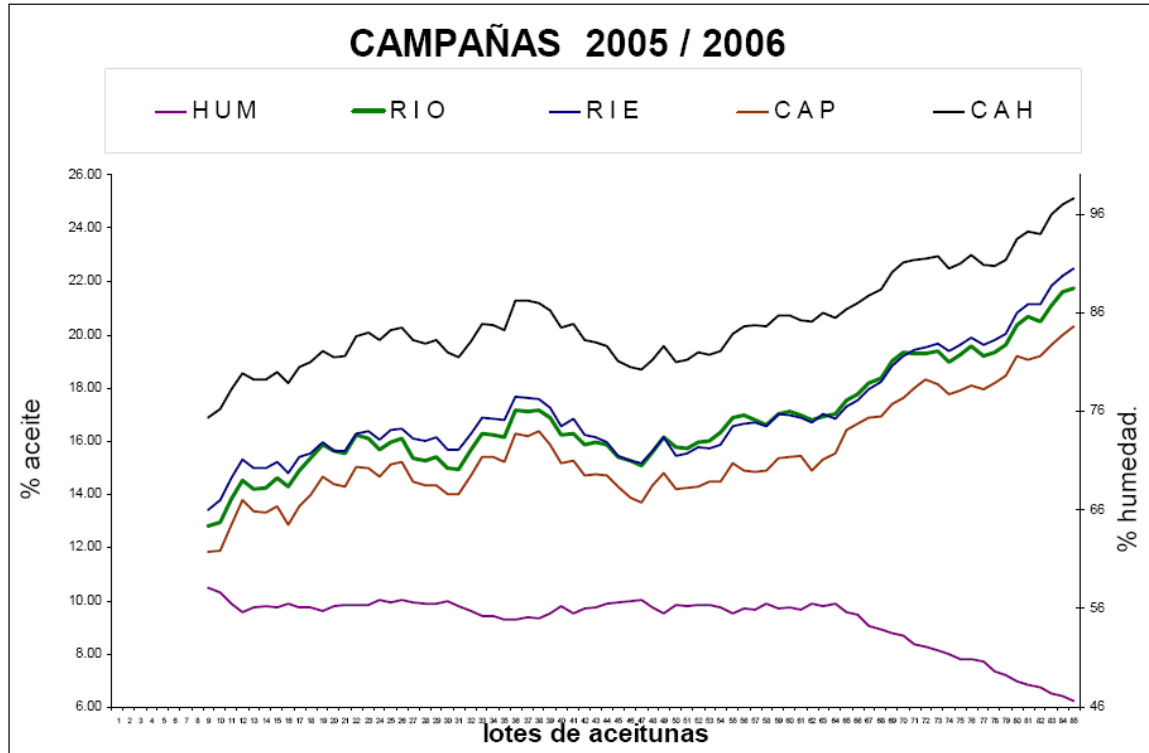
The keys to the English equivalents of the Spanish-language acronyms in these graphs are:

HUM = Mst
CAP = POC
CAH = FMO
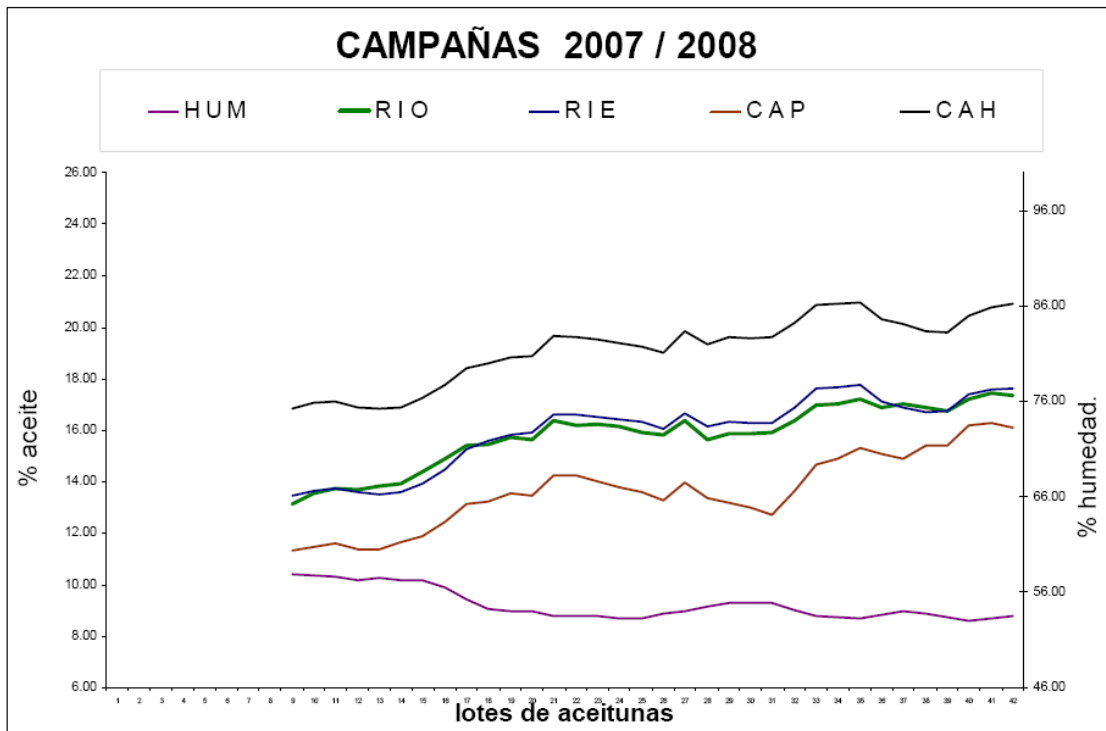RIO = APY
RIE = CPY
Lotes de aceitunas = olive lots
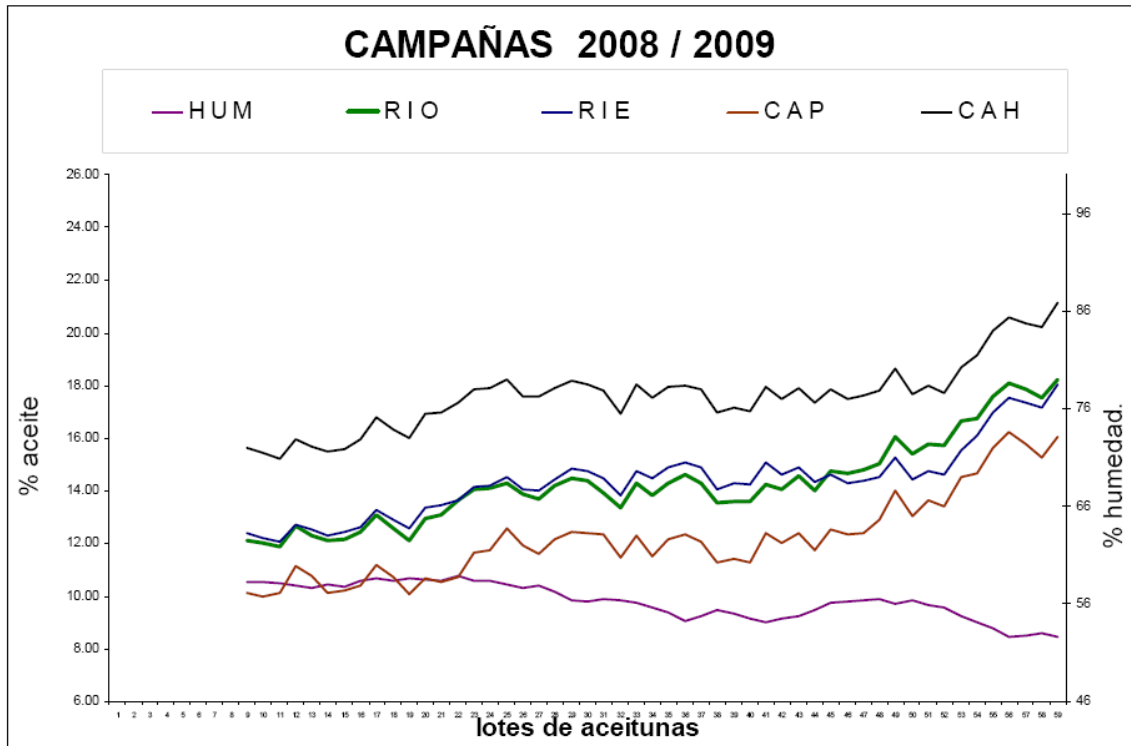% aceite = % oil
% humedad = % moisture

This graph shows the moving average for the nine-data-point subsets of a full set of 46 data for the moisture content (M), partial oil content (POC), fresh matter oil content (FMO), actual processing yield (APY$_l$) and calculated processing yield (CPY) of the lots that were processed.
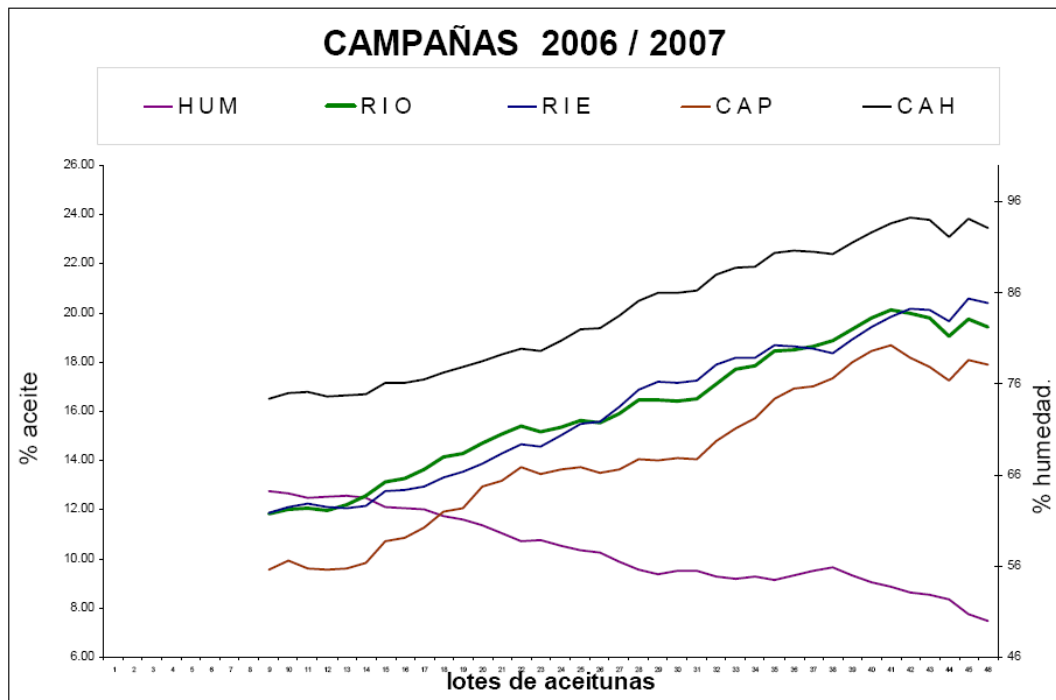
The next graph shows the moving average for the nine-data-point subsets of a full set of 42 data for the moisture content (M), partial oil content (POC), fresh matter oil content (FMF), actual processing yield (APY$_1$) and calculated processing yield (CPY) of the lots that were processed.

This graph shows the moving average for the nine-data-point subsets of a full set of 59 data for the moisture content (Mst), partial oil content (POC), fresh matter oil content (FMO), actual processing yield (APY) and calculated processing yield (CPY) of the lots that were processed.

This graph shows the moving average for the nine-data-point subsets of a full set of 232 data for all four crop years for the moisture content (Mst), partial oil content (POC), fresh matter oil content (FMO), actual processing yield (APY_1) and calculated processing yield (CPY) of the lots that were processed.
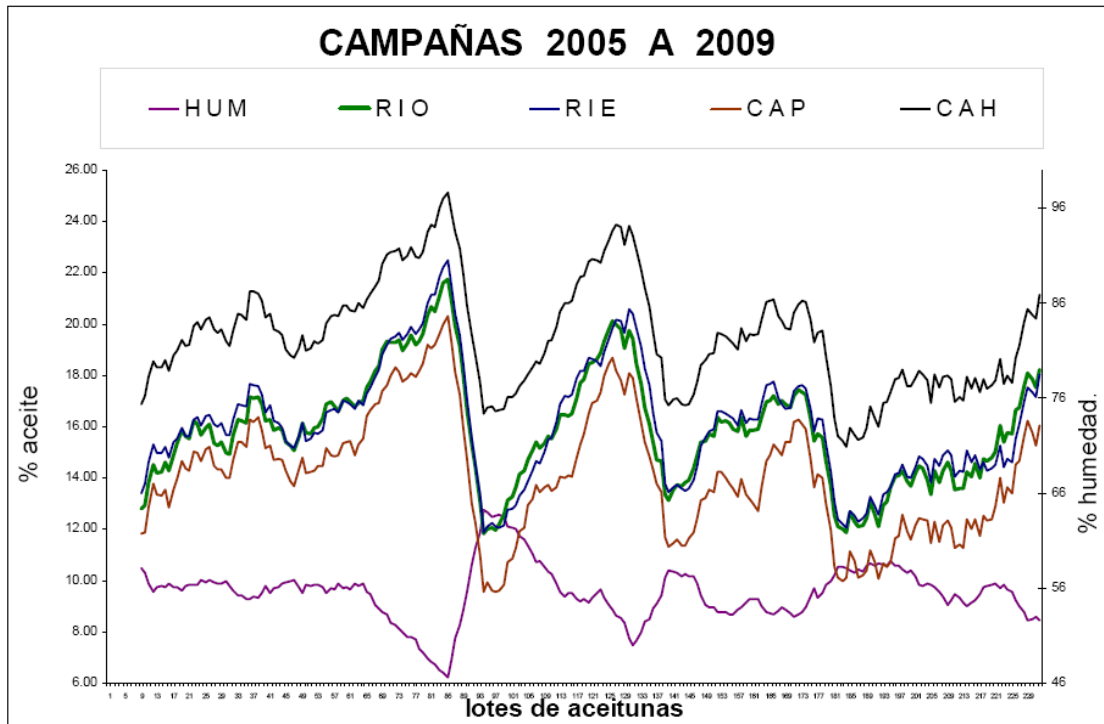
N.B. Photographs courtesy of José Alba Mendoza.