GUIDANCE ON

OBJECTIVE TESTS TO DETERMINE QUALITY

OF FRUITS AND VEGETABLES AND DRY AND DRIED PRODUCE

In recent years there has become an increased awareness of the need for the consumer to have fruit available to eat which has reached a satisfactory state of ripeness and which exhibits the true organoleptic characteristics of the produce and of the variety concerned.

In the framework of the Scheme, internal quality of fruit is defined as: “The degree, measured with objective criteria, to which a commodity has reached a sufficient stage of development such as to enable its quality, after harvesting and post harvest handling (including ripening, where required) to be at least the minimum acceptable to the final consumer”.

This document describes those methods of objective testing of fruits that have emerged as beneficial to both Inspection Services, and the fruit industry in general in determining acceptable levels of ripeness and quality.
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TAKING THE SAMPLES

Sampling

The sample has to be taken in accordance with the “OPERATIONAL GUIDELINES FOR THE CONTROL OF THE QUALITY OF PRODUCE EXPORTED UNDER THE “SCHEME” (published in document C(99)10/FINAL – Annex II as amended).

Quality control takes place by assessing bulk samples taken at random from different points in the lot to be inspected. It is based on the principle of presumption that the quality of the bulk sample is representative of the quality of the lot.
DETERMINATION OF TOTAL SOLUBLE SOLIDS OR SUGAR (TSS) BY REFRACTOMETER

During the development of the flesh of a fruit, in many species, nutrients are deposited as starch, which during the ripening process is transformed into sugars. The progression of the ripening process leads to increasing sugar levels.

This document describes an objective test to determine the total content of soluble solids (TSS) or sugar in a fruit by means of the refractometer. The method is especially suitable for ripe and juicy fruit, with significant sugar content, as the determination of TSS is based on the capacity of sugars in a juice to deviate light.

Material

A refractometer measures TSS as °Brix in 0.1% graduations. There are hand-held refractometers as well as digital battery/mains-operated models available. All models apply similar principles. However, the manufacturers’ instructions must always be followed.

Some refractometers automatically compensate for changes in temperature, whereas others may be calibrated to read accurately at a fixed temperature (usually 20°C). To obtain accurate readings at temperatures other than 20°C it is necessary to refer to the International Temperature Correction Table (1974) which is usually supplied with the instrument or ISO standard 2173 - (edition 2003).

Refractometers should not normally require re-calibration, however, the following calibration instructions may prove useful. If there is any doubt as to the accuracy of any reading it is important to consult the manufacturer’s instructions.

1 These instruments are presented for information only. The OECD does not recommend the usage of any particular make.

2 The text describes the calibration and method of operation for the more traditional hand-held refractometer. When using digital battery/main-operated models similar principles apply, however, the manufacturers’ instructions must always be followed.
Use of the refractometer

Depending on the purpose of the analysis, several drops of distilled water, sucrose solution or juice are placed on the prism surface. The liquid on the prism plate should be free from bubbles or floating particles of pulp or other matter.

- **Hand-held model:** The prism lid is closed. To get proper readings, the instrument is turned towards the light. If necessary the eye piece is focused until a clear image appears. The position at which the demarcation line between the light and dark regions crosses the vertical scale gives the percentage soluble solids reading.

- **LCD Digital model:** Push the button to get the soluble solids reading in percent.

Checking and re-calibration to zero

Requirements:

- A bottle of distilled water.
- A small bottle of 6% sucrose solution. The solution should be stored in a bottle, kept away from daylight and used within 48 hrs of preparation.

Several drops of distilled water are placed on the prism surface.

Distilled water should give a reading of zero. If not and where possible, the refractometer must be adjusted to read zero.

The prism plate is wiped dry with a soft tissue free from fluffs.

Several drops of 6% sucrose solution are placed onto the clean and dry prism plate.

The refractometer should give a reading of 6%. If the reading is not accurate:

a) A new fresh solution of accurate 6% sucrose may be required.

b) The refractometer may need to be repaired or replaced.

Taking care of the refractometer

Optical glass is relatively soft and damage can easily occur to prism surfaces. Care should be taken not to scratch the prism and therefore metal and glass objects should be kept away from the prism surface.

Samples should be washed off the instrument as soon as possible with distilled water. A prism is susceptible to alkalis and acids if left in contact for any length of time. They should be washed clean with a suitable solvent before being rinsed with distilled water and dried off with a soft tissue.

Periodically it is an advantage to wipe the prism plate with alcohol to remove any oils which may adhere. Alcohol must not be used on battery/mains operated models.

It is always advisable to keep any liquids confined to the prism end of the refractometer.
Sampling

To evaluate the lot selected for inspection, take a sample of at least 10 fruits of each size at random from the reduced sample. In case of small fruits packed in sales packages (e.g. strawberries, cherries) take 10 sales packages and at least five fruits of each package or 10 primary samples if fruits are packed in bulk in the package.

However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

Sample preparation

It is important that the juice sample used for measuring soluble solids is extracted in a uniform way and to take into account natural differences in the distribution of soluble solids within the fruit for the species concerned.

Although it is not possible to lay down precise guidelines for all produce which could be tested. The overriding criteria is that the juice sample must be as far as possible representative of the whole fruit. Dry fruit should be used, as any external moisture mixing with the juice will lower the reading.

Where specific methods for sample preparation or juice extraction are given in marketing standards or OECD brochures, it should be followed. In absence of such guidelines, sample preparation and the juice extraction should be done in following way:

| Apples, Pears, Peaches and Nectarines | From each fruit two longitudinal slices (from stem end to calyx-end) are taken, one from the most coloured side and one from the opposite. The core is removed. The slice is squeezed longitudinally to get a mixture of juice from all regions. |
| Apricots, Plums | Cut the fruit in half. Each half is measured to get a mixture of juice from all regions. |
| Kiwifruit | Cut the stem and blossom ends at a distance of 15 mm from each end of the fruit and squeeze the two slices separately. |
Melons

Using a small diameter metal borer (1 – 4 mm) a core of melon should be extracted from the equatorial axis area. Each end of the core should be discarded i.e. the skin and the flesh area immediately beneath it and also the soft pulpy seed area. The remaining flesh should be used to extract the juice for testing.

Alternatively, two longitudinal slices (from stem end to calyx-end) are taken, one from the side that touched the ground during growth and one from the opposite. From the middle of the slice a piece of fruit flesh is cut off, with the core and peel removed. The remaining flesh is squeezed to extract the juice for testing.

Table grapes

At least 5 berries are taken from each bunch or sales package at different places of the bunch or sales package. These berries can be squeezed and tested individually or all together to get a mixture of juice from these berries. However, it is possible to squeeze the whole bunch.

Water melons

Two longitudinal slices (from stem end to calyx-end) are taken, one from the side that touched the ground during growth and one from the opposite. From the equatorial section a piece of fruit flesh is cut off, with the core and peel removed. The piece of fruit-flesh is squeezed.

Citrus fruit

Cut each fruit in half crosswise and squeeze to extract all the juice.

Small fruits e.g. Strawberries, Cherries

At least 5 fruits are taken from each primary sample or sales package at different places of the package. These fruits can be squeezed and tested individually or all together to get a mixture of juice from these fruits.

Tomatoes

From each fruit two longitudinal slices (from stem end to calyx-end) are taken. The slice is squeezed longitudinally to get a mixture of juice from all regions.
Measurement

An equal number of drops from the prepared fruit juice or the prepared fruit are placed onto the refractometer prism plate. The reading on the prism scale is noted to one decimal place. After each test the prism plate must be cleaned with (distilled) water and wiped dry with a soft tissue.

Results

It is important to record the results, to one decimal place, as well as all the details concerning method, variety and stage of maturity and ripeness of the produce being tested.

Each reading for the individual fruit, bunch, primary sample or sales package is noted. The sum total of all readings are averaged (rounded to one decimal place) to give a mean figure.

If the juice is taken from two parts of the fruit (e.g. longitudinal slices, equatorial axis area) in a first step, the two readings for each individual fruit are averaged. In a second step the sum total of these readings should be averaged (round to one decimal place) to give a mean figure. The same procedure is possible in case of small fruit or table grapes where single fruits or berries may be measured.

If the average readings of all fruit are equal to or greater than the limit specified in the standard, the lot has reached the minimum maturity level.

If the average readings of 3 or more of the 10 fruits, bunches or sales packages are at least 10 per cent below the limit specified in the standard, a second sample needs to be taken and analysed with other fruits of the reduced sample or from a new sample. If the average of the two samples is at least 10 per cent below the limit specified in the standard, the lot fails the minimum maturity level and needs to be rejected. No tolerance is applied.
DETERMINATION OF FIRMNESS OF A FRUIT BY PENETROMETER

The firmness of a fruit is linked to the state of maturity and ripeness and may be influenced by the variety as well as the region of production and the growing conditions. This document describes an objective test to determine the firmness of fruit by means of a penetrometer.

The penetrometer is used by producers, packers and distributors to help to determine the stage of ripeness of a fruit and by the retail trade to determine palatability for the consumer and shelf life for their own records.

The determination of firmness of a fruit by means of the penetrometer is based on the pressure necessary to push a plunger of specified size into the pulp of the fruit up to a specific depth.

Material

Penetrometers are available with dial gauges calibrated in both metric (kg) and imperial (lbs) measurements and can be obtained to cover different ranges of pressure suitable for measuring either soft or harder types of fruit, depending on the variety and the stage of ripeness of the produce to be tested.

Three detachable plungers are available:

- One of 8 mm (½ cm²) diameter generally suitable for use in testing softer produce (e.g. peaches, nectarines, plums),
- one of 11 mm (1 cm²) diameter generally suitable for use in testing harder fruit (e.g. apples, pears) and
- a pointed plunger for use in testing avocados.

Ideally the penetrometer should be bench-mounted on a fixed, rigid drill stand to ensure that pressure is applied at a steady controlled rate and at a constant angle to the fruit i.e. vertically downwards. This is more difficult to achieve when using a hand-held penetrometer.

If it is not practical to use a stand mounted penetrometer and it is necessary to use a hand-held one as in the field or market place - then particular care must be taken to ensure a smooth and uniform application of pressure when taking readings. The method is the same for both the hand-held and the mounted penetrometer and must be identical for each item of produce tested in order to obtain consistent results.

If testing is done in a laboratory, a stand-model penetrometer should be used.

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1. These instruments are presented for information only. The OECD does not recommend the usage of any particular make.
Sampling

To evaluate the lot selected for inspection, take a sample of at least 10 fruits of each size at random from the reduced sample. However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

Sample Preparation

From two opposite sides of the equatorial area of the fruit a disc of peel (only skin depth) of up to 2 cm² (¾ sq. ins) is removed.

Where fruit is of mixed colour, e.g. apples, the tests should be carried out where possible between the highest and the lowest coloured portion of the surface.

Measurement

Hold the fruit firmly with one hand, rest it on a rigid surface, such as a table top or the plate at the base of the stand.

The choice of plunger size and scale range used will depend on the type and the variety of the produce being tested and its stage of maturity and ripeness.

If the size of plunger is mandatory in marketing standards, the given size has been used.

It is recommended that the size of the plunger chosen and the particular scale used should be such as to give readings in the middle range of the scale.

Zero the penetrometer and place the plunger head against the flesh in the peeled area of the fruit. Apply steady downward pressure until the plunger has penetrated the flesh of the fruit up to the depth mark (half way up) on the plunger. Slow, steady pressure is essential as sharp uneven movements may give unreliable results. Remove the plunger and note the reading on the penetrometer dial, to one decimal place.

Repeat the process on the opposite side of the same fruit after first zeroing the penetrometer.

It is very important to conduct all tests as uniformly and carefully as possible in order to allow an accurate comparison of results.

Results

It is important to record the results, to one decimal place, as well as all the details concerning plunger size, scale range used, variety and stage of maturity and ripeness of the produce being tested.

As a first step the two readings for each individual fruit are averaged. In a second step the sum total of these readings should be averaged (to one decimal place) to give a mean figure.

If the average readings of all fruit are equal to or greater than the limit specified in the standard, the lot has reached the minimum maturity level.

If the average readings of 3 or more of the 10 fruits are at least 10 per cent below the limit specified in the standard, a second sample needs to be taken and analysed with other fruits of the reduced sample or from a new sample. If the average of the two samples is below the limit specified in the standard, the lot fails the minimum maturity level and needs to be rejected. No tolerance is applied.
DETERMINATION OF FRUIT ACIDS BY TITRATION AND CALCULATION OF THE SUGAR/ACID RATIO

It is the sugar/acid ratio which contributes towards giving many fruits their characteristic flavour and so is an indicator of commercial and organoleptic ripeness. At the beginning of the ripening process the sugar/acid ratio is low, because of low sugar content and high fruit acid content, this makes the fruit taste sour. During the ripening process the fruit acids are degraded, the sugar content increases and the sugar/acid ratio achieves a higher value. Overripe fruits have very low levels of fruit acid and therefore lack characteristic flavour.

Titration is a chemical process used in ascertaining the amount of constituent substance in a sample, e.g. acids, by using a standard counter-active reagent, e.g. an alkali (NaOH).

Once the acid level in a sample has been determined it can be used to find the ratio of sugar to acid.

There are two methods specified for the determination of the titratable acidity of fruits:

- Method using a coloured indicator;
- Potentiometric method, using a pH meter, which should be used for very coloured juices.

Material:

- A laboratory burette of 25 or 50ml capacity or an automatic burette is used. A 10ml pipette, beaker (250ml), a filter (muslin cloth or fine filter) and an extractor or homogeniser.
- A bottle of distilled water.
- Sodium Hydroxide (NaOH): The Standard Laboratory solution of 0.1M which is used in the actual titration is considered to be dilute, and can readily be purchased in this form.
- Phenolphthalein: This is a 1% w/v solution of phenolphthalein in 95% v/v ethanol which is flammable and toxic if ingested. This is only required for the method using a coloured indicator.
- Indicator stripes To check the exact point of neutrality an indicator stripe should be used. Not necessary if pH Meter is used.

Sampling

To evaluate the lot selected for inspection, take a sample of at least 10 fruits of each size at random from the reduced sample. However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.
Sample preparation

Depending upon the type of produce, either cut the fruit in half and squeeze out the juice with an extractor or a juice-press e.g. citrus fruits, or homogenise the flesh into a pulp. The juice of all squeezed fruits is mixed.

The skin and solids should not be included; the solids being filtered out through muslin cloth or fine filter extracting as much juice as possible.

Use a clean and dry safety 10ml pipette. Draw up 10ml of juice and discharge it into a 250ml beaker. Using another clean and dry pipette draw up 50ml of distilled water and add to the juice in the beaker.

Measurement

Method using a coloured indicator

Add 3 drops of phenolphthalein to the juice/water solution in each beaker from a dropping pipette which is specifically kept for that purpose.

Ensure the tap on the burette is shut and using a funnel pour the 0.1M solution of NaOH into the burette until it reaches the zero mark. Do not spill the solution onto the skin.

Slowly titrate the NaOH into the juice/water solution (with a 25ml burette or an automatic burette). Care must be taken that the NaOH is dropped directly into the solution and does not adhere to the glass, otherwise the reading may be false. While titrating care must be taken to continually swirl the solution in the beaker to keep it thoroughly mixed. This is essential, particularly when the solution nears neutrality. It is important to determine the point of neutrality or the end point of titration very exactly. The phenolphthalein indicator changes very rapidly from colourless to pink and the end point can easily be missed, which will give an inaccurate reading for the test. It is important therefore that towards the end of the titration the NaOH is added a drop at a time.

Using phenolphthalein as an indicator, the point of neutrality is reached when the indicator changes from colourless to pink. The indicator colour must remain stable (persisting for 30 seconds) and be light pink when viewed over a white background. However, the shade can vary depending on the type of juice being tested. If the point of neutrality is missed, i.e. the colour of the indicator is too dark, the test is not acceptable and must be repeated. An indicator stripe should be used to avoid the neutral point of pH 8.1.

Read off the amount of the amount of NaOH used (titre) on the burette and record this figure.
- Re-fill the burette for each subsequent test.
- Clean the equipment thoroughly and rinse with distilled water. Detergents must not be used.

Note: When testing very acidic juices e.g. lemons and limes a larger amount of NaOH is required. Therefore, when the NaOH reaches the 25ml mark on the scale the burette tube should be recharged as described above. When the end point is reached the various readings are added together and recorded to produce a figure of NaOH used for each titration.
Method using a pH meter

The point of neutrality i.e. the end point of titration may also be determined using a pH meter. The precise method used will depend on the manufacturer instructions, but the following will provide a general guide.

Checking the pH meter

- Make sure the pH meter has warmed up before use - allow about 30 minutes.
- Remove the electrode from the distilled water in the storage beaker and dry.
- Place the electrode into the beaker containing a buffer solution of pH 7 and calibrate the meter to the same figure.
- Whenever readings are taken, ensure that the electrode is not in contact with the sides or base of the beaker.
- Remove the electrode and - after rinsing in distilled water - place in the solution to be tested; the electrode should not have any contact with the glass.

Measurement

Ensure the tap on the burette is shut and using a funnel pour the 0.1M solution of NaOH into the burette until it reaches the zero mark. Do not spill the solution onto the skin.

Slowly titrate the NaOH into the juice/water solution. Care must be taken that the NaOH is dropped directly into the solution and does not adhere to the glass, otherwise the reading may be false. While titrating care must be taken to continually swirl the solution in the beaker to keep it thoroughly mixed. This is essential, particularly when the solution nears neutrality. It is important to determine the point of neutrality or the end point of titration very exactly. The end point can easily be missed, which will give an inaccurate reading for the test. It is important therefore that towards the end of the titration the NaOH is added a drop at a time.

Using a pH meter, while titrating the digital readout will be seen to climb from around 4 or 5. When the reading reaches 7 proceed carefully. The point of neutrality or the end point of titration is reached at pH 8.1. If this figure is exceeded the test is not acceptable and must be repeated.

- When the pH meter reads 8.1 read off the amount of NaOH used on the burette and record.
- Remove the electrode and rinse it in distilled water ready for the next test. Do not allow it to become contaminated.
- Re-fill the burette for each subsequent test.
- Clean the equipment thoroughly and rinse with distilled water. Detergents must not be used.

Note: When testing very acidic juices e.g. lemons and limes a larger amount of NaOH is required. Therefore, when the NaOH reaches the 25ml mark on the scale the burette tube should be recharged as described above. When the end point is reached the various readings are added together and recorded to produce a figure of NaOH used for each titration.
Calculation of the Sugar/Acid Ratio

The °Brix value of the fruit concerned must also be obtained before calculation of the sugar/acid ratio is possible.

The calculations for determining the sugar/acid ratios of all produce are the same, but as some products contain different acids the appropriate multiplication factor must be applied to each calculation. Some products may contain more than one type of acid, it is the primary acid that is tested. A list of these acids and multiplication factors are found below.

Factor for:  
- citric acid : 0.0064 (Citrus fruit)  
- malic acid : 0.0067 (Apples)  
- tartaric acid : 0.0075 (Grapes)

*Using citric acid as an example, 1ml 0.1M NaOH is equivalent to 0.0064g citric acid.*

**Results expressed as percentage acid:**

Percentage acid = \( \frac{\text{Titre} \times \text{acid factor} \times 100}{10 \text{ (ml juice)}} \)

The sugar acid ratio = \( \frac{\°\text{Brix value}}{\text{Percentage acid}} \)

**OR**

**Results expressed as acid in gram/litre:**

g/l acid = \( \frac{\text{Titre} \times \text{acid factor} \times 100 \times 10}{10 \text{ (ml juice)}} \)

The sugar acid ratio = \( \frac{\°\text{Brix value} \times 10}{\text{g/l acid}} \)

E.g.: In case of citric acid the result would be expressed as:

<table>
<thead>
<tr>
<th>Percentage citric acid</th>
<th>Gram/ litre acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage Citric Acid = ( \frac{\text{Titre} \times 0.0064 \times 100}{10\text{ml juice}} )</td>
<td>g/l Citric Acid = ( \frac{\text{Titre} \times 0.0064 \times 100 \times 10}{10 \text{ (ml juice)}} )</td>
</tr>
<tr>
<td>This formula can be simplified to: Percentage Citric Acid = Titre x 0.064</td>
<td>This formula can be simplified to: g/l Citric Acid = Titre x 0.64</td>
</tr>
<tr>
<td>The sugar acid ratio = ( \frac{\°\text{Brix value}}{\text{Percentage acid}} )</td>
<td>The sugar Acid Ratio = ( \frac{\°\text{Brix value} \times 10}{\text{g/l Citric Acid}} )</td>
</tr>
</tbody>
</table>
Results

It is important to record the results, to one decimal place, as well as all the details concerning method, variety and stage of maturity and ripeness of the produce being tested.

If the result achieves the limit specified in the standard, the lot has reached the minimum maturity level.

If the result is at least 10 per cent below/above the limit specified in the standard, a second sample needs to be taken and analysed with other fruits of the reduced sample or from a new sample. If the average of the two samples is below/above the limit specified in the standard, the lot fails the minimum maturity level and needs to be rejected. No tolerance is applied.

Health and Safety Guidelines

Sodium Hydroxide in its undiluted form is extremely corrosive to body tissue. Skin contact causes irritation almost immediately and continued contact causes burns. The 0.1 Molar solution used in this test is much safer. However, it is recommended that protective coats are worn when using, and that it is used only in a well ventilated room.

Phenolphthalein is highly flammable and should be used with care. It should be stored and used away from naked flames or other sources of ignition. It is toxic if ingested.

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DETERMINATION OF THE JUICE CONTENT

The juice content is an essential parameter to determine the quality of different fruits, especially for citrus fruit.

Material

- Extractor or juice press (simple household press, citrus press, household centrifuge)
- Filter (muslin cloth, fine filter or strainer)
- Scale
- Beaker

Sampling

To evaluate the lot selected for inspection, take a sample of at least 2kg of fruits each size at random from the reduced sample.

However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

Sample preparation

Cut the fruit in half crosswise and squeeze each half to extract all the juice with an extractor or a juice press. The squeezing of each half should be done as thorough as possible in order to extract all the juice.

The extracted juice is then filtered through muslin cloth, fine filter or strainer.

Measurement

Determine the total weight (1g scale) of

- fruit
- beaker
- extracted juice

Calculation of juice content

The juice content is calculated in following way:

\[
\text{Juice percentage} = \frac{\text{Total weight of juice (in g)} - \text{beaker weight (in g)}}{\text{Total weight of fruit (in g)}} \times 100
\]
Results

It is important to record the results, to one decimal place, as well as all the details concerning method, variety and stage of maturity and ripeness of the produce being tested.

If the result is equal or greater than the limit specified in the standard, the lot has reached the minimum juice content.

If the result is at least 10 per cent below the limit specified in the standard, a second sample needs to be taken and analysed with other fruits of the reduced sample or from a new sample. If the average of the two samples is below the limit specified in the standard, the lot fails the minimum juice content and needs to be rejected. No tolerance is applied.

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DETERMINATION OF DRY MATTER CONTENT
BY LABORATORY REFERENCE METHOD OR
MICROWAVE-OVEN QUICK METHOD

Introduction:

The accepted method for determination of percent dry matter is drying the sample in a (vacuum) oven at 70 °C until consecutive weighings made at 2 h intervals vary by less than 3 mg (AOAC Methods 1980). Although several samples can be dried at any one time, this method has the disadvantage of usually requiring samples to be dried overnight to complete the test.

Microwave drying technology has its merits due to its speed, simplicity, low cost, and repeatability, but it results in localised drying and gives a high variability in drying times dependant on power settings and sample type.

The laboratory reference method shall be used in case of rejection and dispute.

DETERMINATION OF DRY MATTER CONTENT BY LABORATORY REFERENCE METHOD

This method allows to determine the loss of mass during the process of desiccation of the fruit.

Materials and instruments

- Analytical scale with gradation of 0.01g (error factor of around 0.1 - 0.3% dry matter)
- (Vakuum) Oven with air flow of 60°C - 105°C
- Desiccator
- Spatula or spoon
- Petri dishes (8 cm in diameter)
- Knife
- Slicer, vegetable peeler or grater
- Food processor with chopping blade or cheese grater
- Calculator, data sheet

SAMPLING

To evaluate the lot selected for inspection, take a sample of at least 15 fruits from the reduced sample.

However, fruits should be firm and free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

PROCEDURE

Each time that samples are weighed, they must be controlled until the nearest centigram.

Weight and number each Petri dish (one for each chosen fruit) and take note of the weight (A).

Preparation of the fruit:

Take a sample of 10 gram of flesh using the slicer / peeler / grater. The sample will be taken by removing thin slices of flesh (thickness 1.5 – 2mm) from all around the cut.
In case of avocados or kiwifruit the fruit has to be prepared in following way:

- **Avocados**: Cut the fruit along the longitudinal diameter, eliminate the seed and the seminal tegument and remove the outer skin without peeling the flesh. The thickness of the slice should be 2mm. A total of 20 gram of fresh weight should be sampled per fruit. The peeling should be taken from the face of one quarter.

- **Kiwifruit**: Cut the fruit along the equatorial diameter. The thickness of the slice should be 3mm. A total of 10 - 20 gram of fresh weight should be sampled per fruit.

Place the slices on the Petri dish and cut the slices into smaller pieces in order to facilitate the drying process. Write down the total weight of the fresh sample + Petri dish (B). The weighing should be done immediately after placing the sample on the dish in order to avoid loss of water from it.

The oven should be warmed to the required temperature before the samples are put inside. (An accurate thermometer placed in a cup filled with vegetable oil should be placed inside the oven to achieve the most accurate temperature readings).

To reduce the effect of the maillard reaction, the oven should be operated by relatively low temperature.

Dry the sample in the oven with an air flow of 70°C for until constant weigh is reached (around 4 – 6 hours).

In case of avocados or kiwifruit drying should be done in following way:

- **Avocados**: 60°C until constant weigh is reached (around 18 hours),
- **Kiwifruit**: 65°C until constant weigh reached (around 8 hours).

After the drying, weigh the sample and note the total weight of the dry sample + Petri dish (C). Weighing should take place within 15 minutes from taking the samples out of the oven.

**CALCULATION**

The percentage of dry matter is calculated by:

\[
\text{Percentage dry matter} = \frac{(C-A)}{(B-A)} \times 100
\]

A = weight of Petri dish
B = total weight of fresh sample + Petri dish
C = total weight of dry sample + Petri dish

**RESULTS**

It is important to record the results, to one decimal place, as well as all the details concerning method, variety and stage of maturity and ripeness of the produce being tested.

If the average readings of all fruit are equal to or greater than the limit specified in the standard, the lot has reached the minimum maturity level.

If the average readings of at least 30% of the fruits are at least 10 per cent below the limit specified in the standard, a second sample needs to be taken and analysed with other fruits of the
reduced sample or from a new sample. If the average of the two samples is below the limit specified in
the standard, the lot fails the minimum maturity level and needs to be rejected. No tolerance is applied.

DETERMINATION OF DRY MATTER CONTENT QUICK METHOD USING THE
MICROWAVE-OVEN

This method allows to determine the loss of mass during the process of desiccation of the fruit.

Materials and instruments

- Analytical scale with gradation of 0.01 g
- Microwave-oven, capable of reaching a power of 800 W
- Spatula or spoon
- Petri dish (8 cm in diameter)
- Knife, vegetable peeler,
- Slicer, food processor with chopping blade or cheese grater
- Calculator, data sheet

SAMPLING

To evaluate the lot selected for inspection, take a sample of at least 10 fruits of each size at
random from the reduced sample. However, fruits should be firm and free from defects such as sun
scorch and pest or disease damage, which may have affected the normal ripening process.

PROCEDURE

Weigh an empty dish and record its weight as the tare weight (A).

Cut the fruit longitudinally in two parts, eliminating the seed and the seminal tegument.

From one part of the fruit, 1.5 mm-thick slices must be cut with the help of the slicer (around
5 -10 gram).

Deposit the slices without overlap on a numbered Petri dish. For each fruit a separate Petri dish is
needed.

Weigh each glass-slide which contains the sample and record the weight (B).

Put the glass-slides into the microwave oven. It must be checked beforehand, for this thickness of
the sample slice, that the desiccation is constant and that no brown coloration due to burning will
appear. Establish a power of 800 W and after 4 minutes, weigh the sample directly, without allowing it
to cool in the desiccator. Return the sample into the microwave for 1 minute and weigh it again.
Repeat the process until the weight is constant or the difference of the mass between two consecutive
weighings is not greater than 0.5 mg. The total time of desiccation ranges between 4 and 7 minutes.
The final weight will be (C).
CALCULATION

Calculate the dry matter content as following:

\[
\text{Percentage dry matter} = \frac{(C-A) \times 100}{(B-A)}
\]

A = weight of Petri dish

B = total weight of fresh sample + Petri dish

C = total weight of dry sample + Petri dish

RESULTS

It is important to record the results, to one decimal place, as well as all the details concerning method, power settings, variety and stage of maturity and ripeness of the produce being tested.

If the readings of all fruit are equal to or greater than the limit specified in the standard, the lot has reached the minimum maturity level.

If the readings are below the limit specified in the standard, a second sample needs to be taken with other fruits of the reduced sample or from a new sample and analysed by the laboratory reference method before the lot will be reject.

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DETERMINATION OF TOTAL SOLUBLE SOLIDS BY VIS-NIR

To determine the optimum harvest date as well as fruit quality development in post-harvest period, rapid non-destructive sensing methods are available. Optical spectroscopy in visible (VIS) and near infrared (NIR) spectrum range have been successful for many years to study quality properties of agricultural products.

The spectral signature of fresh fruit and vegetables shows in the 400 to 1100 nm region two dominant absorption bands. The first one is the absorption band of chlorophyll at about 670 nm in the VIS region (400 to 750 nm), additional absorption between 500 and 600 nm is caused by red blush pigments. The second absorption band is due to water at about 970 nm in the short wave NIR region (750 to 1100 nm), close to the water absorption band, sugar and other carbohydrates contribute to additional light absorption in NIR region.

There exist many studies on food quality by using VIS-NIR spectroscopy analysing the reflectance and transmittance spectra of fresh fruit. It was concluded that it would be possible to predict simultaneously the contents of soluble solids, sugars and chlorophyll with a single spectrum from 400 to 1000 nm, which would allow to develop multidimensional predictors of consumer acceptance.

To determine quality parameters of fresh fruit, specific adaptation of sensor probe to fruit species as well as to desired quality information is necessary. Ground colour which is caused due to chlorophyll content in fruit skin and adjacent cell tissue could be determined by using a sensor probe for diffuse reflectance measurement on fruit surface. However, determination of internal quality parameters like TSS content requires the use of a sensor probe for partial or complete light transmission through fruit. The light path length through fruit cell tissue as well as the morphological fruit characteristics affect the measure spectral signature.

For the wavelength range from 400 to 1100nm very promising low-cost miniaturised spectrometer modules are commercially available with attached photometric sensors based on silicon. Therefore, this spectrum range is expected to be attractive for agricultural applications.

As the method described above is an indirect method to determine quality parameters, it is necessary to establish for each species, growing area, season and eventually variety a calibration curve adjusted to results obtained by appropriate laboratory reference methods.
DETERMINATION OF THE STARCH CONTENT OF APPLES AND PEARS USING AN IODINE SOLUTION

During the development of the flesh of a fruit, nutrients are deposited as starch which during the ripening process is transformed into sugars. The progression of the ripening process leads to decreasing starch levels.

This document describes an objective test to determine the amount of starch in the flesh of a fruit using an iodine solution. Iodine turns a blue-black colour when it comes into contact with starch. As a fruit ripens more starch is converted to sugar, and the blue-black area becomes less prominent. Ripening usually takes place from the core of the fruit towards the skin. Ripening fruit will generally show an increasing white ring around the core, if treated with iodine. But other patterns (i.e. an increasing star around the core) are possible depending on variety.

This test is particularly suitable for fruit such as apples, and to a lesser extent to pears. But it is useful only to determine the ripeness of fruit at harvest time. At the subsequent stages of marketing the starch content – even of underdeveloped and unripe fruit may have decreased without sufficient increase of ripeness.

Material

- Iodine solution

  The iodine solution is prepared by dissolving 10g of potassium iodide in 30ml of distilled water and then adding 3g of iodine. Once the iodine has dissolved the mixture is made up to 1 litre by adding distilled water at 10° - 30°C. This solution can be stored for up to 6 months in a cool (4 to 7°C) dark place.

  Note: The chemicals and the prepared solution will stain, so should be kept away from the skin and from fabrics.

Sampling

To evaluate the lot selected for inspection, a sample of 10 fruits minimum of each size is taken at random from the reduced sample. However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

Sample preparation

Using a sharp knife each fruit is sliced in half. It is very important that the surfaces are cleanly cut, without any additional damage occurring to the flesh of the fruit or the skin. Additional damage of this type may cause further starches to be released, from the damaged cells, leading to an inaccurate result.
Measurement

One half of each freshly cut surfaces of the fruit is evenly coated with iodine solution. This can be applied using a dropper bottle, pipette or spray bottle.

The cut sections are left for one minute before the results are recorded.

The stage of ripeness (percentage surface of the fruit changing to a blue-black colour) must be recorded.

The amount of blue-black colour present on a tested sample may be directly related to the ripeness of the fruit.

Results

It is important to record the results as well as all the details concerning method, variety and stage of maturity and ripeness of the produce being tested.

The stage of ripeness (Code-No. of starch conversation chart) for each individual fruit is noted.

If the readings of all fruit are equal or less than the suitable ripe index, the lot has reached the minimum maturity level.

If 3 or more of the 10 fruits do not reach the suitable ripeness index, a second sample needs to be taken and analysed with other fruits of the reduced sample or from a new sample. If the average of the two samples does not reach the suitable ripe index, the lot fails the minimum maturity level and needs to be rejected. No tolerance is applied.

Note: Care should be taken when interpreting the results of this test as many varieties of apples and pears ripen in different ways, and produce differing starch patterns. Varieties are suitable for eating at different stages of maturity and ripeness, dependent on individual consumer preference.

Health and Safety Guidelines

Pure Iodine can cause severe irritation to the respiratory system, skin and eyes, and the solid will burn the skin. It is recommended that gloves, goggles and protective clothing be worn when handling.
Starch conversion chart for apples

Radial type (R)

1 (R) Slight central discolouration

2(R) – 3(R) – 4(R): Increasing radial discolouration

5(R) – 6(R) – 7(R): Increasing central discolouration with peripheral cracks

8(R) – 9(R) -10(R): Increasing peripheral discolouration
**Starch conversion chart for apples**

**Circular type (C)**

1 (C) - Slight central discolouration

2(C) – 3(C) – 4(C): Central discolouration, from “coin” to “5-leaver clover”

5(C) – 6(C) – 7(C): Increasing central discolouration with peripheral spots

8(C) – 9(C) -10(C): increasing peripheral discolouration
DETERMINATION OF SKIN COLOUR BY OECD COLOUR GAUGES

The colour of fruit skin is a good indicator to describe the ripeness of a fruit or uniformity concerning presentation.

The use of colour gauges permits to define a colour stage or a range of colour stages to describe a certain grade of ripeness/maturity.

To get an objective result different colour gauges are elaborated.

Material

The OECD SCHEME has elaborated Colour Gauges for Use by the Trade in Gauging the Skin Colouring of

- Apples (Golden Delicious) and
- Tomatoes.

![Colour gauge for tomatoes (open)](image1) ![Colour gauge for apples](image2)

Note: To prevent discolouration, the colour gauges should not be exposed to daylight permanently. In case of discolouration the colour gauges have to be replaced.

Sampling

To evaluate the lot selected for inspection, take a sample of at least 10 fruits at random from the reduced sample. However, fruits should be free from defects such as sun scorch and pest or disease damage, which may have affected the normal ripening process.

Measurement

The colour of the fruits (typically background colour of the individual fruit) will be compared against the different colour steps of the colour gauge.

The tests should be done by daylight or under fluorescent white light.

Results

The colour code which matches the skin colour is noted.

If the colour of the fruit skin lies between two colour codes, both codes are noted.

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DETERMINATION OF THE MOISTURE CONTENT FOR DRIED FRUITS

METHOD 1 - LABORATORY REFERENCE METHOD

1. Scope and application

This reference method serves to determine the moisture for dried fruits, as dried or desiccated apricots, figs, prunes, dates, grapes, apples, pears, etc.

2. Reference

This method is based on the method prescribed by AOAC: AOAC Official Method 934.06 - Moisture in Dried Fruits.

3. Definition

Moisture content for dried fruits: conventionally, loss in mass measured under the operating conditions specified in AOAC Official Method 934.06. The moisture content is expressed as percentage by mass (grams per 100 grams).

4. Principle

Determination of the moisture content of a test portion by drying in an oven 6 h at 70 ± 1 °C under pressure < 100 mm Hg (13.3 kPa).

5. Apparatus (see AOAC Official Method 934.06)

5.1 Analytical balance sensitive to 1 mg or better.

5.2 Mechanical mill or food chopper.

5.3 Non-corrosive metal dish, provided with well-fitting lid, about 8.5 cm of diameter, allowing the test portion to be spread to about 0.2 g/cm² or less.

5.4 Electric vacuum oven with thermostatic control capable of being regulated in normal operation at 70 ± 1 °C under pressure < 100 mm Hg (13.3 kPa.).

5.6 Desiccator containing an effective desiccant.

5.7 Steam-bath

1 Annex I of standard layout for UNECE standards concerning the marketing and commercial quality control of dry and dried produce.
6. Procedure

Follow the operating conditions as specified in AOAC Official Method 934.06 for Moisture in Dried Fruits, with the following additional specifications, concerning the preparation of the test sample:

Homogenize the laboratory sample and take a minimum of 100 g of dried fruits as a test sample. With non-pitted stone fruits (apricots, prunes, peaches, dates, etc), remove the stones using the rest as a test sample.

Grind or chop the test sample to obtain small particles, using either a mechanical mill or food chopper, without overheating the product, or cut and grind by hand if necessary, using knife, scissors, mortar and pestle or similar.

Use 5.0 to 10 g of the ground or chopped product as a test portion. Mix the test portion with circa 2 g of finely divided glass fiber filter or of washed sand, with the help of a spatula, and weigh to the nearest 0,001 g.

When necessary, moisten the test portion and the glass fiber filter or the washed sand with a few millilitres of water, mix thoroughly with the help of the spatula, and heat the open dish on the steam-bath to near dryness, before complete the drying in the vacuum oven.

Carry out two determinations on the same test sample.

7. Expression of results and test report

The moisture content, W, as percentage by mass of the sample (grams per 100 grams), is equal to:

\[ W = \frac{M_1 - M_2}{M_1 - M_0} \times 100 \]

where

- \( M_0 \) is the mass, in grams, of the dish and lid.\(^1\)\(^2\)\(^3\)
- \( M_1 \) is the mass, in grams, of the dish and lid, and the test portion before drying.\(^1\)\(^2\)
- \( M_2 \) is the mass, in grams, of the dish and lid, and the test portion after drying.\(^1\)\(^2\)

Take as a result the arithmetic mean of the results of the two determinations, if the difference between the results is smaller than 0.2%. The result has to be reported to one decimal place.

The test report shall show the method used and the results obtained. It shall mention any operating details not specified or optional, and incidents, likely to have influenced the results. It shall also include all the information necessary for the complete identification of the sample.

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\(^1\) Weigh to the nearest 0.001 g
\(^2\) In case, plus the glass fibre or washed sand, and spatula.
\(^3\) After heating, on the oven for 2 hours and cooling in the desiccator.
8. **Repeatability**

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst, using the same apparatus and in the same laboratory, should not be greater than 0.2 g of moisture per 100 g of sample.

**METHOD 2: RAPID METHOD**

1. **Scope and application**

This rapid method serves to determine the moisture for dried fruits. ¹

2. **Reference**

This method is based on the method prescribed by AOAC: AOAC Official Method 972.20 - Moisture in Prunes and Raisins (Moisture Meter Method). This method is also commonly used as unofficial method for the determination of moisture content in other kinds of dried fruits.

3. **Definition**

Moisture content for dried fruits: conventionally, correlation between moisture content and conductance-temperature measured under the operating conditions specified in AOAC Official Method 972.20. The moisture content is expressed as percentage by mass (grams per 100 grams).

4. **Principle**

Determination of the conductance and temperature of a test portion by the moisture tester meter and under the operating conditions specified in AOAC Official Method 972.20. The moisture tester meter has to be calibrated according to the laboratory method, for each kind of dried fruit, taken into account the variety or commercial type and the type of presentation (whole, pitted, slabs, dices, etc) and, when necessary, the crop year and/or the origin.

5. **Apparatus (see AOAC Official Method 972.20)**

   5.1 Moisture tester meter type A series.

   5.2 Thermometer (if not incorporated to the moisture tester meter).

   5.3 Mechanical mill or food chopper.

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¹ It is also possible to employ other rapid methods based on different conductance methods, or on the principle of loss of mass by heating with apparatus including an halogen or infra-red lamp and a built-in analytical balance, always at condition that the method and the apparatus has to be calibrated according the laboratory method.
6. **Procedure**

Follow the operating conditions as specified in AOAC Official Method 972.20 - Moisture in Prunes and Raisins (Moisture Meter Method).

Carry out the determination on two test portions

7. **Expression of results and test report**

7.1 **Result**

The result should be the arithmetic mean of the two determinations. Report the result to one decimal place.

7.2 **Test report**

The test report must state the method used and the results obtained. The report must contain all the information necessary for the complete identification of the sample.

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DETERMINATION OF THE MOISTURE CONTENT FOR DRY FRUIT

METHOD 1 - LABORATORY REFERENCE METHOD

1. Scope and application

   This reference method serves to determine the moisture and volatile matter content for both inshell nuts and shelled nuts (kernels).

2. Reference

   This method is based on the method prescribed by ISO: ISO 665-2000 Oilseeds - Determination of moisture and volatile matter content.

3. Definition

   Moisture content and volatile matter content for dry produce (inshell nuts and shelled nuts): loss in mass measured under the operating conditions specified in ISO 665-2000 for oilseeds of medium size (see point 7.3 of ISO 665-2000). The moisture content is expressed as mass fraction, in percent, of the mass of the initial sample.

   For whole nuts, when moisture content is expressed both on the whole nut and on the kernel, in cases of dispute between the two values, the moisture content value of the whole nut takes precedence.

4. Principle

   Determination of the moisture and volatile matter content of a test portion by drying at 103 ± 2°C in an oven at atmospheric pressure, until practically constant mass is reached.

5. Apparatus (see ISO 665-2000 for more details)

   5.1 Analytical balance sensitive to 1 mg or better.

   5.2 Mechanical mill.

   5.3 3 mm round-holes sieve.

   5.4 Glass, porcelain or non-corrosive metal containers, provided with well-fitting lids, allowing the test portion to be spread to about 0.2 g/cm² (approximately 5 mm height).

   5.5 Electric oven with thermostatic control capable of being regulated between 101 and 105°C in normal operation.

   5.6 Desiccator containing an effective desiccant.

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¹ Annex I of standard layout for UNECE standards concerning the marketing and commercial quality control of dry and dried produce.
6. **Procedure**

Follow the operating conditions as specified in ISO 665-2000 for oilseeds of medium size (point 7 and 7.3 of ISO 665-2000), but with the following specific modifications, concerning the preparation of the test sample.

Although ISO 665-2000 sets up one initial period of 3 hours in the oven set at 103 ± 2° C, for nuts it is recommended one initial period of 6 hours.

6.a **Determination of the moisture and volatile matter content of kernels:**

For shelled nuts, homogenize the laboratory sample and take a minimum of 100 g of kernels as a test sample.

For inshell nuts, take a minimum of 200 g and, using a nutcracker or hammer, remove the shells and fragments or particles of shell, using the rest as a test sample. The kernel skin (cuticle or spermoderm) is included in the test sample.

Grind and sieve the test sample until the size of the particles obtained is no greater than 3 mm. During the grinding operation, care should be taken to avoid the production of a paste (oily flour), the overheating of the sample and the consequent loss of moisture content (for example, if using a mechanical food chopper, by successive very short grinding and sieving operations).

Spread evenly over the base of the vessel about 10 g of the ground product as a test portion, replace the lid, and weigh the whole vessel. Carry out two determinations on the same test sample.

6.b **Determination of moisture and volatile matter content on whole nuts (shell plus kernel):**

Remove all the foreign matter (dust, stickers, etc.) from the test sample. Homogenize the laboratory sample and take a minimum of 200 g of nuts as a test sample.

Grind the whole nuts using either a Rass Mill, a Romer Mill or a Brabender apparatus or similar, without overheating the product.

Spread evenly over the base of the vessel about 15 g of the ground product as a test portion, replace the lid, and weigh the whole vessel. Carry out two determinations on the same test sample.

7. **Expression of results and test report**

Follow all the instructions as specified in ISO 665-2000 (point 9 and 11) for method of calculation and formulae, and for test report, without any modification.¹

8. **Precision**

For conditions of repeatability and reproducibility apply specifications of ISO 665-2000 (point 10.2 and 10.3) for soya beans.

¹ The main points specified are as follows:

- Moisture and volatile matter content is expressed as mass fraction, in percent, of the mass of the initial sample.
- The result is the arithmetic mean of the two determinations; the difference between the two determinations should not exceed 0.2% (mass fraction).
- The result has to be reported to one decimal place.
METHOD 2: RAPID METHOD

1. Principle

Determination of the moisture content using a measuring apparatus based on the principle of loss of mass by heating. The apparatus should include a halogen or infra-red lamp and a built-in analytical balance, calibrated according to the laboratory method.

The use of apparatus based on the principle of electrical conductivity or resistance, as Moisture Meters, Moisture Testers and similar, is also allowed always at condition that the apparatus has to be calibrated according with the laboratory reference method for the tested product.

2. Apparatus

   2.1 Mechanical mill or food chopper.

   2.2 3 mm round-holes sieve (unless indicated otherwise by the instructions for use of the apparatus).

   2.3 Halogen or infrared lamp with built-in analytical balance sensitive to 1 mg or better.

3. Procedure

   3.1 Preparation of sample

       Follow the same instructions as given for the laboratory reference method (points 6.a and 6.b), unless indicated otherwise by the instructions for use of the apparatus, particularly with regard to the diameter of the fragments.

   3.2 Determination of moisture content

       Carry out the determination on two test portions of approximately 5 to 10 g each, unless indicated otherwise by the instructions for use of the apparatus.

       Spread the test portion over the base of the test receptacle, thoroughly cleaned in advance, and note the weight of the test portion to within 1 mg.

       Follow the procedure indicated in the instructions for use of the apparatus for the product to be tested, in particular with regard to the adjusting of temperatures, the duration of the test and the recording of the weight readings.

4. Expression of results

   4.1 Result

       The result should be the arithmetic mean of the two determinations, provided that the conditions of repeatability (4.2) are satisfied. Report the result to one decimal place.
4.2  Repeatability

The difference in absolute value between the respective results of the two determinations performed simultaneously or one immediately after the other by the same operator, under the same conditions on identical test material, must not exceed 0.2%.

5.  Test report

The test report must state the method used and the results obtained. The report must contain all information necessary for the full identification of the sample.
**LIST OF STANDARDS WITH SPECIFIC MINIMUM/MAXIMUM PARAMETERS:**

**State of year 2005**

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