FOSFA INTERNATIONAL OFFICIAL METHOD
HANDLING OF LABORATORY SAMPLES

This procedure shall be used by all laboratories carrying out analysis of samples drawn from commodities traded under FOSFA terms. It is for use in conjunction with the Federation's Standard Contractual Methods of Analysis.

1 Sequence of procedures

The laboratory shall have a stipulated sequence for all the procedures listed below (plus any others) in order to ensure that the handling of each sample follows a clearly defined and co-ordinated programme for its administration and analysis.

2 Physical receipt of samples

On receipt of a sample for analysis, the following visual checks should be carried out.

2.1 Check the labelling to ensure that the sample is fully identifiable.

2.2 Check that the seals are intact. If tampering is suspected, request a second sample.

2.3 Check sample visually and any sample odour to assess condition and whether it is contaminated. Any unusual condition should be noted at this stage and further action taken accordingly. If any microbiological or insect activity is noted, the sample should be segregated to prevent contamination of other samples.

2.4 Check that sufficient quantity of the sample has been received for analysis taking into account of the possible need to repeat analysis. The quantity should conform to minimum requirements for the analytical methods for all determinations requested as per the FOSFA current method for the packaging of oilseed samples or other commodities.

2.5 Check that the sample received conforms to the description given in the instructions for analysis.

2.6 When a second or duplicate sample is received, check that it satisfactorily conforms to the above visual checks and the original samples.

If any of the above criteria are not fulfilled, further samples should be requested from the parties to the contract. Laboratories may find it useful to compose a physical check list based on the above items.

3 Sample labelling

All samples must be clearly labelled (including the labelling of both jar and lid if appropriate).

4 Identification during analysis

Identification of each individual sample must be ensured throughout each stage of analysis. This identification might consist of simple number referencing of the sample and accompanying worksheets and does not need to carry full details of the sample's origin.

5 Worksheets

Specially designed worksheets or notebooks should be used to enable information to be recorded at each stage of any determination, including details of the calculation of results. The design should also facilitate close supervision by senior personnel. The date of each stage of a determination should always be shown on the worksheet, as should the date the sample was received and analysis commenced.
6 Supervision

Analysis should be under the supervision of technically qualified senior personnel at all times. Only such personnel should be responsible for checking results and signing certificates. Additionally in signing certificates, they are responsible for ensuring that the correct methods are used in accordance with the FOSFA Analysis Certificate requirements and that, when analysis is being carried out under the terms of FOSFA contracts, these methods conform to the FOSFA Standard Contractual Methods of Analysis.

7 Handling of samples

Laboratory staff should take great care over the handling of samples at all times in order that they remain, as far as possible, in the same condition as they were when they arrived at the laboratory. All samples should be returned to the correct sample store when not in use in accordance with FOSFA Official Method of Storage. The melting and solidification of oil and fat samples should be kept to a minimum. Samples should not be allowed to stand on laboratory benches where they are subjected to direct sunlight or under heat, as this can lead to changes in the sample’s properties (e.g. moisture and volatile matter, peroxide value or free fatty acid content of the oil).

8 Oilseed samples

When oil content analysis is requested, if no request for admixture separation is also received for the same sample, it is none the less recommended that this should still be done if visual inspection of the sample suggests that admixture exceeds 2 %. If it is more than 2 %, report this to the principal to the contract. The oil content is then calculated on a pure basis, with back calculation to a tale quale basis, taking account of the oil content of the admixture if necessary. This operation must be performed when using infra-red equipment. An approximate sorting test can be used to estimate whether admixture analysis is necessary.

9 Analysis in duplicate

Results of duplicate analyses are only acceptable if they are within the acceptable repeatability range quoted in the method, when it is normal to average the results to show the final result. If the duplicates are outside the acceptable repeatability range, then the entire determination should be repeated, preferably using a second analyst, to obtain two further results.

10 Time span of analysis

With several determinations (e.g. moisture, peroxide value), differences can arise in the results if there are delays in carrying out analysis. Duplicate determination should be carried out in rapid succession.

11 Dating of certificates

Analysis should be completed as near to the date of drawing a sample as practicably possible. The certificates should show the date of analysis, any re-test and the date that they were issued.

12 Storage conditions

Samples shall be stored prior to examination and during the post-analysis retention period in a location that is dark and cool (less than 20 °C), dry and free of pests and rodents. It is recommended that the atmosphere of the store should contain a mild insecticide, when being used for the storage of oilseeds.

13 Retention of samples

In order to re-check samples and, in the event of any dispute arising, it is necessary to keep samples three months after the issue of certificates. When an arbitration or appeal is involved and this is notified to the analyst, the sample must be kept until further notice. In other circumstances, the analyst may also be requested to keep samples for longer than three months.
FOSFA INTERNATIONAL OFFICIAL METHOD

DETERMINATION OF SEA WATER CONTAMINATION IN OILS

1 Scope

The method is applicable to all oils, with the exception of those (such as acid oils) which are known to have been treated with hydrochloric acid.

2 Principle

The oil is examined for the presence of chloride ions in the aqueous contaminant. If chloride ions are detected and the amount of aqueous phase permits, quantitative determinations of chloride and other ions may be carried out.

3 References


3.2 Analysis of Raw, Potable and Waste Waters. HMSO, 1972.


4 Reagents

4.1 Silver nitrate solution, approximately 0.1 M.

4.2 Nitric acid, concentrated, AR.

5 Procedure

5.1 Sample preparation

5.1.1 If the nature of the sample permits, separate the aqueous and oil components by centrifugation, or by allowing the sample to stand. Determine the volume of aqueous contaminant in the oil.

5.1.2 If the amount of the contaminant is small, extract it by shaking the oil with water. Dilution of the oil with petroleum ether may assist the separation of the aqueous layer. If this procedure is unsatisfactory the oil should ashed at a temperature of not more than 450°C and the tests carried out on an aqueous solution of the ash.

5.2 Qualitative test

Acidify a portion of the aqueous phase with a few drops of concentrated nitric acid, and add a few drops of silver nitrate solution. A white precipitate which darkens upon exposure to light, especially sunlight, (or turbidity if the sample solution is very dilute) indicates the presence of chloride and suggests that the sample may have been contaminated with sea water.
6 Further examination

If a more detailed examination of the aqueous contaminant is required, quantitative determination of chloride and sodium should first be carried out. Determinations of other major constituents of sea water, such as sulphate, magnesium, potassium and calcium may then be carried out as further confirmatory tests.

The principal inorganic constituents of typical sea water are:

<table>
<thead>
<tr>
<th>% m/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloride</td>
</tr>
<tr>
<td>Sodium (as Na)</td>
</tr>
<tr>
<td>Sulphate</td>
</tr>
<tr>
<td>Magnesium (as Mg)</td>
</tr>
<tr>
<td>Calcium (as Ca)</td>
</tr>
<tr>
<td>Potassium (as K)</td>
</tr>
<tr>
<td>Bicarbonate</td>
</tr>
<tr>
<td>Bromide</td>
</tr>
</tbody>
</table>

The methods of analysis will depend on the quantity of the aqueous contaminant available. Some sources of suitable analytical methods are given in Section 3 (References).

7 Test report

The Analyst's report shall indicate the method(s) of analysis employed.
FOSFA INTERNATIONAL OFFICIAL METHOD

DETERMINATION OF SOAP IN ACID OILS

1 Scope
A non-aqueous titration method is described for the determination of soap in all types of acid oils.

2 Definition
The soap content is expressed as sodium oleate as a percentage by mass calculated from the total alkalinity of the oil.

3 Principle
The total alkalinity is determined by titration in neutral solvent solution of the oil using standard ethanolic hydrochloric acid and bromophenol blue indicator solution. For dark coloured and/or very low expected soap contents (less than 0.01 % by mass) the end-point is measured electrometrically.

4 Reagents
Use only reagents of recognised analytical quality.

4.1 Ethanolic hydrochloric acid solution 0.01N and/or 0.1N accurately standardised.

4.2 Bromophenol blue indicator. Solution of 49 g per litre of 95 % (m/m) ethanol.

4.3 Solvent mixture. 1 volume ethanol/2 volumes acetone neutralised by adding a few drops of indicator solution (4.2) and titrating with hydrochloric acid solution (4.1) or with ethanolic potassium hydroxide solution until just yellow.

5 Apparatus
Use laboratory apparatus and in particular the following.

5.1 Burette 10 ml graduated in 0.02 ml.

5.2 pH meter fitted with automatic temperature compensation and equipped with a suitable electrode system. The electrode assembly should be washed with neutral solvent (4.3) before use.

NOTE It is essential that the pH equipment be very carefully checked for both repeatability and reproducibility at the required operational temperatures. Some equipment has been found to give variations in excess of 0.6 pH units in day-to-say measurements on the same solvent mixture and to require several minutes to give a constant reading after agitation has ceased.

5.3 pH electrode suitable for work in non-aqueous solvent mixture is recommended.

5.4 Suitable means of heating the solvent mixture and oil solution such as a water bath thermostatically controlled at about 46°C.

5.5 Conical Erlenmeyer flasks (250 ml) having no alkaline reaction.

6 Preparation of sample
Melt the oil sample and mix thoroughly.
7 Procedure

7.1 Oils which yield a clear colourless solution in the solvent mixture (4.3)

7.1.1 Test solution

Accurately weigh a suitable mass of sample (6) directly into a 250 ml conical (Erlenmeyer) flask then add 100 ml of solvent mixture (4.3) and dissolve at 40°C to 45°C.

7.1.2 Titration

Add a few drops of indicator solution (4.2). The formation of a green colour indicates the presence of soap. Titrate the test solution (7.1.1) with ethanolic hydrochloric acid (4.1) until the solution is just yellow at its initial temperature (40°C to 45°C). Allow adequate time (5 min) for reaction of calcium soaps etc.

7.2 Oils which yield a dark solution in the solvent mixture (4.3) and/or are expected to have a very low (less than 0.01 % by mass) soap content

7.2.1 pH of solvent mixture

Warm 100 ml solvent mixture (4.3) to 40°C to 45°C, Measure the temperature accurately and adjust the pH meter (5.2) for operation at this temperature and measure the pH. Determine end-point by reference to a calibration curve.

NOTE Choose the vessel to contain the solvent mixture which will subsequently permit the insertion of the pH electrodes (5), such as a beaker or wide-necked conical flask.

8 Expression of results

8.1 Method of calculation

Calculate the soap content, expressed as a percentage (mass/mass) of sodium oleate in the original sample (6) from the formula.

\[
\text{% Soap content} = \frac{V \times N \times 30.4}{W}
\]

(as sodium oleate)

Where:

V is volume in ml of ethanolic hydrochloric acid used (7.1.2 or 7.2.2) to reach equivalence
N is normality of ethanolic hydrochloric acid (4.1)
W is mass of sample used to prepare test solution (7.1.1 or 7.2.2)
DETERMINATION OF SOAP IN ACID OILS

Figure 1: Curve of change in scale reading per unit volume of acid added against volume of acid added and from this measure the equivalence volume corresponding to the curve maximum.
FOSFA INTERNATIONAL OFFICIAL METHOD
PACKAGING AND STORAGE OF OILSEEDS SAMPLES

1 Scope
This FOSFA method for the labelling, packaging, transport and storage of oilseeds samples is applicable to all samples drawn under the terms of FOSFA International contracts for analysis, arbitration or standards purposes.

2 Label design
2.1 Labels must comply with ISO 21294:2017 Section 9.

2.2 Labels should be completed in legible handwriting, preferably in capital letters, or typed. Labels should be securely attached to the samples they represent.

NOTE 1 It is important that principals' instructions to superintendents make it quite clear whether the samples are being drawn for analysis and arbitration purposes.

3 Packaging
3.1 Samples of all oilseeds (other than palm kernels and other lauric seeds) sent to laboratories for analysis should be packed in water-tight plastic jars with screw caps of the same materials or in glass jars with plastic screw caps, of not less than 500 ml, which shall be filled to the top and sealed.

3.2 Palm kernels, illipe nuts, sheanuts, groundnuts – to be packed in a woven polypropylene bag closed and/or sealed then packed in a strong cotton or linen bag, which is then sealed – bags of plastic sheet must not be used.

4 Storage
4.1 Oilseed samples shall be stored at not more than 20°C. Palm kernel, illipe, sheanuts, copra (and other lauric seeds) should be cold stored at minus 15°C.

4.2 Where the sample is not packed and stored in accordance with this recommendation, the oil content at re-test should be adjusted in relation to the variation in moisture between the original test and the re-test.
FOSFA INTERNATIONAL OFFICIAL METHOD

SAMPLING OF EDIBLE GROUNDNUTS FOR AFLATOXIN TESTING
(Not applicable for groundnuts imported into the European Union)

1 Definitions

1.1 lot
An identifiable quantity of a food commodity delivered at one time and determined by an official to have common characteristics, such as origin, variety, type of packaging, packer, consignor or marking.

1.2 sublot
Designated part of a large lot in order to apply the sampling method on that designated part. Each sublot must be physically separate and identifiable.

1.3 incremental sample
A quantity of material taken from a single place in the lot or sublot.

1.4 aggregate sample
The combined total of all incremental samples taken from a lot or sublot.

1.5 laboratory sample
Sample intended for the laboratory (= subsample).

2 General provision

2.1 Material to be sampled
Each lot which is to be examined must be sampled separately. In accordance with the provisions in point 4, large lots should be subdivided into sublots and sampled separately.

2.2 Precautions to be taken
In the course of sampling and preparation of the laboratory samples precautions must be taken to avoid changes which adversely affect the aflatoxin content and the analytical determination or make the aggregate samples unrepresentative.

2.3 Incremental samples
As far as possible incremental samples should be taken at various places distributed throughout the lot or sublot, see Table 1. Departure from this procedure must be recorded in the record referred to in point 2.7.

2.4 Preparation of the aggregate sample and laboratory samples
The aggregate sample is made up by combining and sufficiently mixing the incremental samples.

2.5 Replicate samples
Replicate samples for enforcement, trade (defence) and referee purposes are to be taken from the homogenised laboratory samples.
Packaging and transmission of laboratory samples

Each laboratory sample must be placed in a clean inert container offering adequate protection from contamination and against damage in transit. All necessary precautions must be taken to avoid any change in composition of the laboratory sample which might arise during transportation or storage.

2.6 Sealing and labelling of laboratory sample

Each sample taken from official use shall be sealed at the place of sampling and identified. A record must be kept of each sampling, so that each lot can be identified unambiguously, with the date and place of sampling together with any additional information likely to assist the analyst.

3 Explanatory provisions

3.1 Sampling frequency

Without prejudice to the specific provisions as laid down in point 4, the following formula can be used as a guide for the sampling of lots traded in individual packings (sacks, bags, retail packings, etc).

\[
\text{Sampling frequency (SF)} = \frac{\text{Weight of the lot} \times \text{weight of the incremental sample}}{\text{Weight of the aggregate sample} \times \text{weight of individual packing}}
\]

Weight: in kg
Sampling frequency (SF): every \( n \)th sack or bag from which an incremental sample must be taken (decimal figures should be rounded to the nearest whole number).

3.2 Weight of the incremental sample

The weight of the incremental sample should be about 300 grams, unless otherwise defined in Table 1. For retail packings, the weight of the incremental sample depends on the weight of the retail packing.

4 Specific provisions

4.1 Lots should be subdivided into sublots not exceeding 25 tonnes in weight. The minimum number of incremental samples and aggregate sample size to be taken from each sublot are given in Table 1.

a. each sublot must be sampled separately;
b. samples shall be taken as randomly as possible from throughout the consignment;
c. each sample taken shall be ground finely and mixed thoroughly using a process that has been demonstrated to achieve complete homogenisation;
d. samples of nuts that are “in shell” may include the shell in the final homogenate;
e. the formal samples for enforcement, trade (defence) and referee purposes shall be taken from the homogenised material. In the case of the formal enforcement samples a minimum of three independent subsamples (each a minimum weight of 50 g) shall be removed from the mixed slurry sample for analysis and the size of the sample shall be sufficient to allow for this.

The result shall be taken to be the mean of the analytical results of the three independent subsamples.

4.2 Acceptance of a sublot

a. accept if the mean of the independent subsamples conforms with the maximum limit;
b. reject if the mean of the independent subsamples exceeds the maximum limit.

<table>
<thead>
<tr>
<th>Nuts and nut products</th>
<th>Minimum no. of incremental samples</th>
<th>Approximate incremental sample size (g)</th>
<th>Minimum aggregate sample size (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Groundnuts shelled raw/roasted</td>
<td>30</td>
<td>330</td>
<td>10</td>
</tr>
<tr>
<td>Groundnuts in shell</td>
<td>30</td>
<td>660</td>
<td>20*</td>
</tr>
<tr>
<td>Peanut butter</td>
<td>24</td>
<td>200</td>
<td>5</td>
</tr>
</tbody>
</table>

* weight of nuts in shell

© FOSFA International Copyright 2018

April 2018
1 Scope

The method described is applicable to all groundnuts but is usually used for Hand Picked & Selected groundnuts.

2 Principle

The groundnut kernels are categorised into whole kernels, half kernels (brissures) and broken and damaged kernels and expressed as groundnut kernels to one ounce avoirdupois.

3 Procedure

3.1 Draw a portion of 500 g of the sample and record the weight to the nearest gram.

3.2 Separate into whole kernels, half kernels and broken and damaged kernels and damaged half kernels.

3.3 Record the weight of each portion to nearest gram.

3.4 Count the whole kernels and record the result.

3.5 Count the half kernels and record the result.

3.6 Perform in duplicate test.

4 Calculation and expression of results

Groundnuts kernels, to one ounce avoirdupois = \( \frac{(N + n/2) \times 28.35}{W + w} \)

Also count to 100 g = \( \frac{(N + n/2) \times 100}{W + w} \)

Where:

- \( N \) is the number of whole kernels;
- \( n \) is the number of half kernels;
- \( W \) is the mass, in grams, of the whole kernels;
- \( w \) is the mass, in grams, of the half kernels.

Calculate the mean of the duplicates and quote the count to the nearest whole number.