No L 239/2 Official Journal of the European Communities 28.9.68

REGULATION (EEC) No 1470/68 OF THE COMMISSION

of 23 September 1968

on the drawing and reduction of samples and the determination of the oil content, impurities and moisture in oil seeds

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to Council Regulation No 136/66/EEC[1] of 22 September 1966 on the establishment of a common organisation of the market in oils and fats, and in particular Articles 26 (3) and 27 (5) thereof;

Having regard to Council Regulation No 162/66/EEC[2] of 27 October 1966 on trade in oils and fats between the Community and Greece, and in particular Article 8 thereof;

Having regard to Council Regulation No 142/67/EEC[3] of 21 June 1967 on export refunds on colza, rape and sunflower seeds, and in particular Article 6 thereof;

Whereas in application of Commission Regulation No 282/67/EEC[4] of 11 July 1967 on detailed rules concerning intervention for oil seeds, of Commission Regulation No 284/67/EEC[5] of 11 July 1967 on certain detailed rules for the application of export refunds on oil seeds and of Commission Regulation (EEC) No 911/68[6] of 5 July 1968 on certain detailed rules concerning the subsidy for oil seeds, as last amended by Regulation (EEC) No 1469/68[7] uniform methods should be laid down, for use throughout the Community, for drawing samples and reducing contract samples to samples for analysis and determining the oil content, impurities and moisture in seeds;

Whereas it is appropriate, for each of these operations, to adopt the method generally employed in international trade; whereas in order to obtain consistent results the methods chosen should be precisely defined;

Whereas the measures provided for in this Regulation are in accordance with the Opinion of the Management Committee for Oils and Fats;

HAS ADOPTED THIS REGULATION:

Article 1

1. Without prejudice to the subsequent paragraphs, the drawing of samples, the reduction of contract samples to samples for analysis and the determination of impurities and moisture, required under Article 4 of Regulation No 282/67/EEC, Article 2 of Regulation No 284/67/EEC and Article 17 of Regulation (EEC) No 911/68, shall be carried out according to the methods defined in Annexes I, II, III and IV to this Regulation.

2. By way of derogation from the provisions of Annex I (6.1), sampling superintendents shall be designated by the Member States.

3. By way of derogation from the provisions of Annex I (6.3), at least three contract samples shall be drawn for analysis and arbitration.

4. By way of derogation from the provisions of Annex I (6.2.1), primary samples must be drawn from at least 2% of the bags forming the lot.

5. By way of derogation from the provisions of Annex III (3.2) and (6.3.2), the determination of the moisture and volatile matter shall be carried out on material as received.

[1] OJ No 172, 30.9.1966, p. 3025/66.
[2] OJ No 197, 29.10.1966, p. 3393/66.
[3] OJ No 125, 26.6.1967, p. 2461/67.
[4] OJ No 151, 13.7.1967, p. 1.
[5] OJ No 151, 13.7.1967, p. 6.
[6] OJ No 15 8,6.7.1968, p. 8.
[7] OJ No L 239, 28.9.1968, p. 1.

Article 2

The determination of oil content required under Article 4 of Regulation No 282/67/EEC shall be carried out according to the method defined in Annex V to this Regulation.

By way of derogation from the provisions of Annex V (3.2, 6.3.7, 7.3.1, 7.3.3 and 7.3.5), determination of the oil content shall be carried out on material as received.

Article 3

This Regulation shall enter into force on the third day following its publication in the *Official Journal of the European Communities*.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 23 September 1968.

For the Commission

The President

Jean REY

ANNEX I

ISO Recommendation R 542 (January 1967)

OILSEEDS

SAMPLING

Foreword

Correct sampling is a difficult process which requires most careful attention. Emphasis cannot therefore be too strongly laid on the necessity of obtaining a properly representative sample of oilseeds for analysis.

Most oilseeds are sold on the basis of a sample and on the result of an analysis of the sample, and disputes are invariably settled by reference to the sample, so that careless or inaccurate sampling will lead to misunderstanding, delay and unnecessary financial adjustments.

The procedures given in this ISO Recommendation are recognised as good practice and it is strongly recommended that they be followed whenever practicable. It is recognised that it is difficult to lay down fixed rules to be followed in every case, and particular circumstances may render some modification of the method desirable.

1. Scope

This ISO Recommendation describes methods for sampling consignments of oilseeds. It also describes apparatus in use for this purpose.

2. General

2.1 The purpose of this ISO Recommendation is to specify general conditions relating to sampling for the assessment of the quality of oilseeds purchased as industrial raw materials. Total consignments should be considered in lots of not more than 500 t[1] for large and medium-sized seeds and not more than 100 t for small seeds.

2.2 Samples should be fully representative of the lots from which they are drawn. For this purpose, starting from a lot limited to a maximum of 500 t (or 100 t, as appropriate), a number of primary samples should be drawn and carefully mixed, thus giving a bulk sample from which is obtained, by successive divisions, the contract sample for analysis.

2.3 Special care is necessary to ensure that all sampling apparatus is clean, dry and free from foreign odours.

Sampling should be carried out in such a manner as to protect the samples of oilseeds, the sampling apparatus and the containers in which the samples are placed from adventitious contamination such as rain, dust, etc.

Material adhering to the outside of the sampling apparatus should be removed before the contents are discharged.

3. Definitions

Terms relating to the lot and to the samples have the following definitions:

3.1 *Consignment*

The quantity of seed dispatched at one time and covered by a particular contract.

3.2 *Lot*

A stated proportion of the consignment which will allow the quality to be assessed.

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[1] *Metric tons* (1 t = 1000 kg).

3.3 *Primary sample*

A small quantity of seed taken from a single position in the lot.

A series of primary samples should be drawn, from different positions in the lot, which when bulked and mixed will be representative of the lot.

3.4 *Bulk sample*

The quantity of seed formed by combining and blending the primary samples.

3.5 *Contract sample*

A small sample representing the quality of the lot, obtained from the bulk sample and intended for analysis or other examination.

4. Apparatus

The apparatus required falls under the following headings; examples are given under each heading (see also Figures 1 to 9 in Annex A).

4.1 *Sampling from bags*

Sack-type spears or triers, cylindrical samplers, conical samplers and hand-scoops.

4.2 *Sampling from bulk*

Shovels, hand-scoops, cylindrical samplers, conical samplers, mechanical samplers and other apparatus for drawing small periodical samples from a flow of oilseeds.

4.3 *Mixing and dividing*

Shovels, quartering irons, riffles and other dividing apparatus.

5. Limitation of the size of lot

5.1 *Transport by ship*

Most of the oilseeds are received from ocean-going vessels or from river transport. In both cases sampling normally takes place at transfer from the vessel. Each lot should be 500 (or 100) t or part thereof.

5.2 *Transport by road or rail*

In the case of transfer from vessel to road or rail wagons, sampling may take place prior to the loading of the wagons. Each lot should be 500 (or 100) t or part thereof.
If sampling is carried out from laden wagons, each lot should comprise a number of wagons containing a total of 500 (or 100) t or part thereof.

5.3 *Silo or warehouse*

Where seed is unloaded direct to silos or warehouses from a vessel the samples should be drawn as in clause 5.1. Where there is no provision for such sampling, this may take place as in clause 5.2 prior to, or during, transfer to silo or warehouse. Each lot should be 500 (or 100) t or part thereof.

6. Method of drawing samples

6.1 *General*

Sampling should be carried out by sampling superintendents appointed by buyers and sellers.

As the composition of the lot is seldom or never uniform, a sufficient number of primary samples should be drawn to provide a representative bulk sample. Seed which is sea-damaged or otherwise damaged in transit or out of condition, as well as loose collected[2] and sweepings, should be sampled separately from the sound seed. The damaged material should not be blended with the sound material, but should be assessed separately.

[2] This term is used to designate material which has leaked from its original container, but is not unduly contaminated.

6.2 **Drawing of primary samples**

According to circumstances, the primary samples should be drawn from products in bulk or in bags by means of sampling apparatus mentioned in section 4 and used in accordance with clauses 6.2.1 and 6.2.2.

6.2.1 *From bags*

Unless otherwise specified in the contract or unless the practice at a port requires otherwise, primary samples should be drawn from 2 per cent of the bags forming the lot.

When the bags are opened, the primary samples may be drawn by hand-scoop, cylindrical samplers or conical samplers.

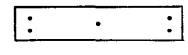
When samples are drawn from the closed bags, sack-type sampling spears (or triers) may be used.

6.2.2 *From bulk*

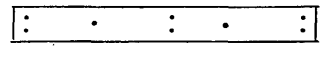
6.2.2.1. When sampling takes place while the product is in motion, primary samples should be drawn through the whole section of the seed and at time intervals depending on the rate of flow.

6.2.2.2 When bulk seed is sampled in the holds of craft during discharge, primary samples should be drawn from as many places as possible and at intervals determined by the rate of discharge.

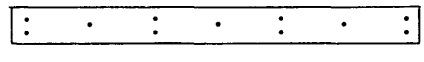
6.2.2.3 If sampling takes place from laden wagons, primary samples should be drawn at three levels, with a cylindrical or conical sampler depending on the seeds, at the following number of points:



Wagons or lorries up to 15 t: 5 sampling points (middle and approximately 50 cm from sides).



Wagons from 15 to 30 t: 8 sampling points



Wagons from 30 to 50 t: 11 sampling points

Note: If the type of wagon does not allow samples to be drawn in this manner, the method of sampling should be as described in clause 6.2.2.1.

6.2.2.4 If sampling takes place from weigh hoppers; primary samples should be drawn by means of cylindrical samplers, shovels or mechanical samplers, in accordance with the practice of the port.

6.2.2.5 The procedure for silos or warehouses is necessarily dependent on local conditions.

6.3 *Contract samples*

The bulk sample should be mixed and divided down to obtain the required number of contract samples by use of apparatus mentioned in section 4. The number of contract samples for analysis and arbitration should be specified in the contract or otherwise agreed between buyer and seller.

For some seeds (e.g. copra, groundnuts in shell), it is advisable to screen the bulk sample before dividing and then to add the fines to the contract samples in the correct proportion. This is to ensure that the samples contain the same percentage of low-quality fines.

6.4 *Size of samples*

The following sizes of samples are usually suitable. Larger or smaller samples may be required in some cases according to the tests to be carried out.

Seed	Lot	Primary sample	Bulk sample[1]	Contract sample
	t	kg	kg	kg
Large seeds (e.g. copra)	Up to 500	1	Up to 200	6
Medium-sized seeds (e.g. groundnut kernels)	Up to 500	0·5	Up to 100	5
Small seeds (e.g. poppy seeds)	Up to 100	0·1	Up to 20	2

[1] Whatever the size of the bulk sample, it should be representative of the lot.

7. Packaging and labelling of samples

7.1 *Packaging of samples*

The contract samples should be packed in bags of closely woven cloth or strong paper, or in paperboard containers, polyethylene bags, sheet metal boxes, glass bottles or glass jars.

Samples for the determination of moisture or for any analysis that may be influenced by a change of moisture content should be packed in moisture-proof containers fitted with airtight closures. The containers should be completely filled and the closures should be sealed to prevent a change of the original moisture content

7.2 *Labels for samples*

If paper labels are used for oilseed samples, it is recommended that their quality and size should be suitable for the purpose. The eyelet hole on the label should be reinforced.

The information on the labels should include at least the following:

(1) Ship or wagon
(2) From
(3) To
(4) Arrived
(5) Quantity
(6) Bulk/bags
(7) Designation of product
(8) Mark[2] or lot number
(9) Bill of lading number and date, or contract number and date
(10) Date of sampling
(11) Place and point of sampling
(12) Sampled conjointly by...

The recorded information on the label should be permanent.

8. Dispatch of samples

Contract samples should be dispatched as soon as possible, and only in exceptional circumstances later than forty-eight hours after sampling has been completed, non-business days excluded.

9. Sampling Report

If a sampling report is prepared, it should indicate:

— the condition of the seed sampled,

— the technique applied if this is different from that described in this ISO Recommendation, and

— all circumstances that may have influenced sampling.

[2] For identification.

ANNEX A

EXAMPLES OF SAMPLING APPARATUS

Note: Many different types and variations of apparatus are available, and dimensions are included solely as a guide.

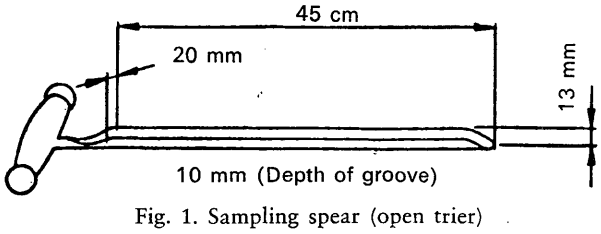


Fig. 1. Sampling spear (open trier)

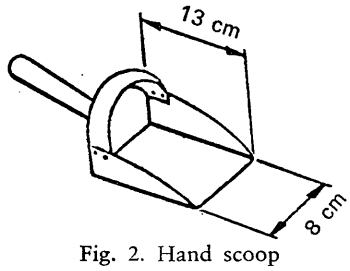


Fig. 2. Hand scoop

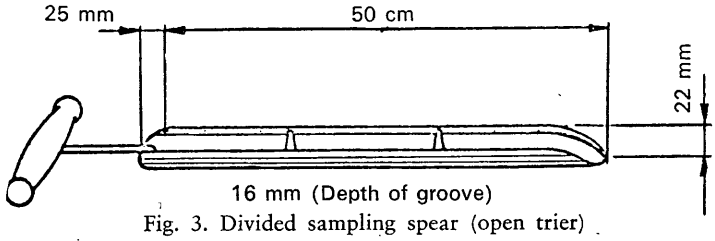


Fig. 3. Divided sampling spear (open trier)

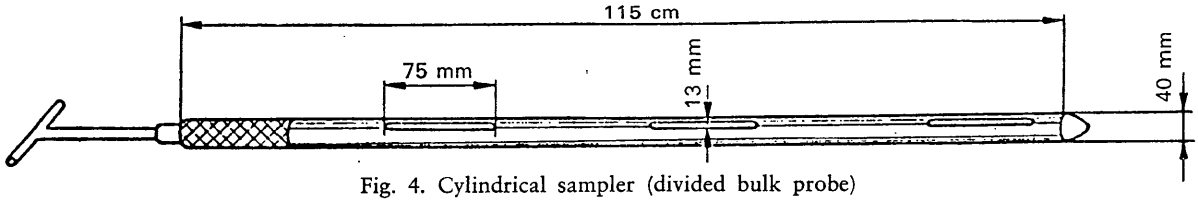


Fig. 4. Cylindrical sampler (divided bulk probe)

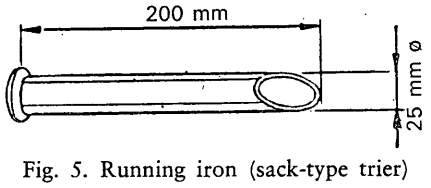


Fig. 5. Running iron (sack-type trier)

Fig. 6. Falling stream sampler (Pelican type)

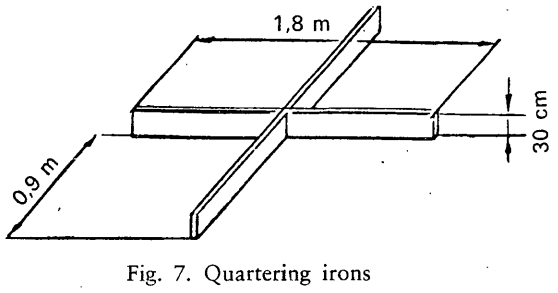


Fig. 7. Quartering irons

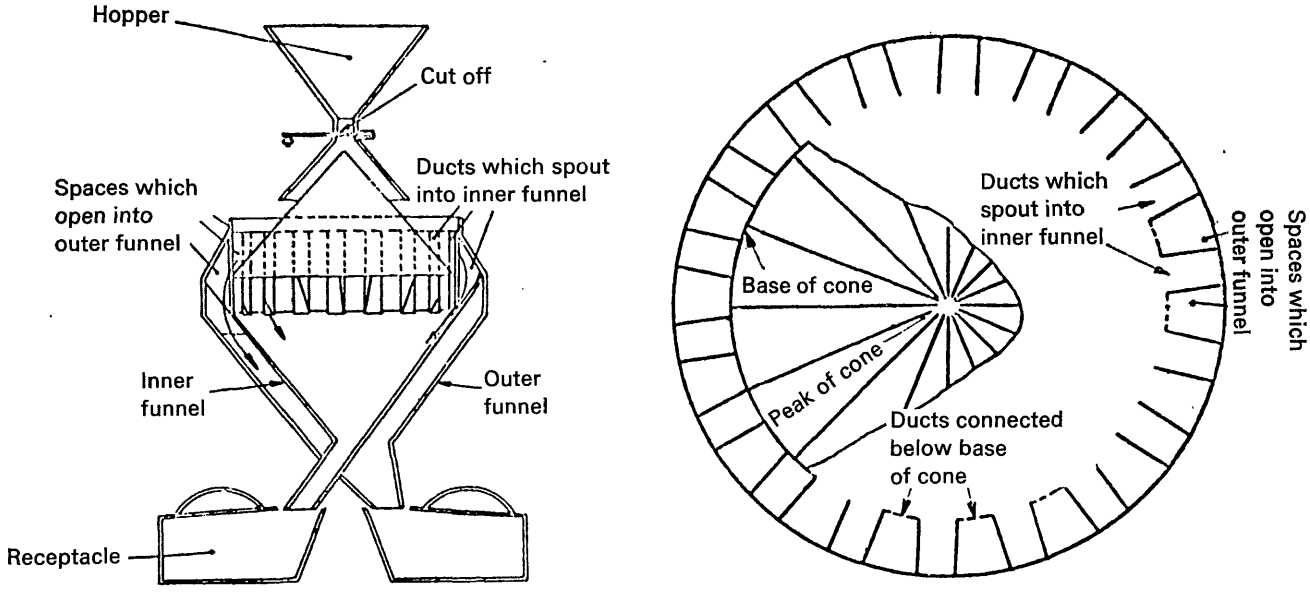


Fig. 8. Conical divider (Boerner type)

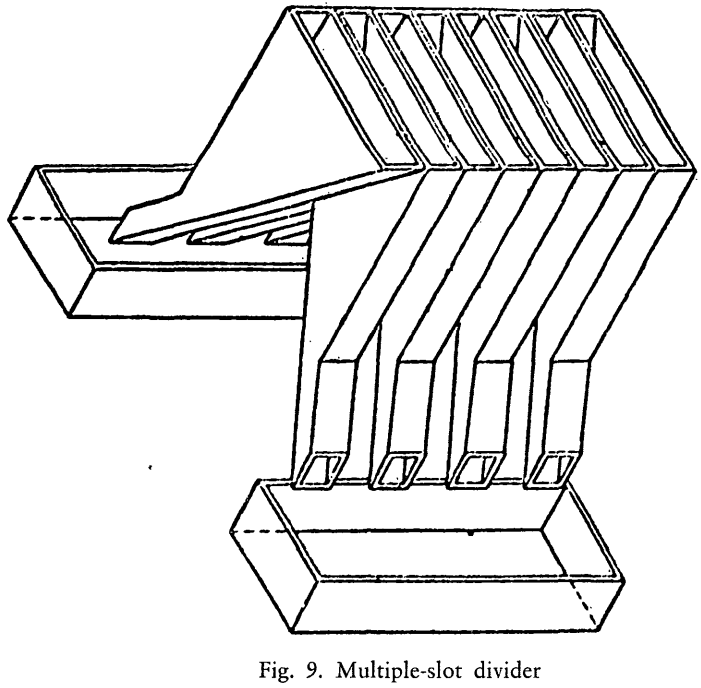


Fig. 9. Multiple-slot divider

ANNEX II

ISO Recommendation R 664 (February 1968)

OLEAGINOUS SEEDS

REDUCTION OF CONTRACT SAMPLES TO SAMPLES FOR ANALYSIS

1. Scope

This ISO Recommendation describes the procedures for obtaining, from a contract sample of oleaginous seeds, a sample for analysis.

Notes

1. Procedures for obtaining representative contract samples from a consignment of oilseeds are described in ISO Recommendation R 542, Oilseeds — Sampling[1].

2. Some contracts for the trading of oilseeds call for analysis of the sample as drawn, i.e. including any impurities that may be present. On the other hand, some contracts call for the preliminary quantitative separation of impurities and analysis of the pure seed separated. Analysis of the impurities may also be required.

2. Principle

Division of the contract sample by suitable means, if necessary after the removal of impurities of large size, using one or other of the dividing instruments specified and taking care that the final analysis sample truly represents the bulk of the contract sample.

The analysis sample, either in its original state or after separation of impurities, is prepared for analysis by the procedure specified in the relevant method.

3. Apparatus

Dividing apparatus.

Quartering apparatus, conical divider, multiple slot divider, or other dividing and sorting apparatus which will ensure uniform distribution of the components of the contract sample in the analysis sample.

4. Division of contract sample

After separating and weighing the impurities of large size, if necessary, mix the contract sample carefully in order to make it as uniform as possible and, by means of dividing apparatus appropriate to the nature of the seed, reduce it successively until there is obtained approximately the mass of material shown in Table 1,

[1] See Annex I.

TABLE 1

Species of seed		Minimum mass of each analysis sample (grammes)
Copra	Cocos nucifera, Linnaeus	1000
Castor	Ricinus communis, Linnaeus	600
Palm kernel	Elaeis guineensis, N. J. Jacquin	600
Groundnut	Arachis hypogaea, Linnaeus	600
Shea nut	Butyrospermum paradoxum (C. F. Gaertner) Hepper	500
Pumpkin	Cucurbita pepo, Linnaeus	500
Sunflower	Helianthus annuus, Linnaeus	500
Soya bean	Glycine max, Linnaeus Merril	500
Cottonseed	Gossypium sp	500
Hemp	Cannabis sativa, Linnaeus	200
Linseed	Linum usitatissimum, Linnaeus	200
Rape	Brassica napus, Linnaeus	200
Rubsen	Brassica rapa, Linnaeus	200
Poppy	Papaver somniferum, Linnaeus	200
Mustard white	Sinapis alba, Linnaeus	200
Mustard black	Brassica nigra, Linnaeus W. D. J. Koch	200
Sesame	Sesamum indicum, Linnaeus	200

Note: For seeds not included in the above Table, the minimum quantities should be the same as prescribed for species of similar size.

5. Separation of impurities

If separation of impurities is required, follow the procedure described in ISO Recommendation R 658, Oleaginous seeds — Determination of impurities[1].

[1] See Annex II.

ANNEX III

ISO Recommendation R 665 (February 1968)

OLEAGINOUS SEEDS

DETERMINATION OF MOISTURE AND VOLATILE MATTER

1. Scope

This ISO Recommendation describes a method for the determination of moisture and volatile matter in oilseeds.

2. Definition

By moisture and volatile matter is meant the loss in mass under the experimental conditions specified below

3. Principle

3.1 Determination of the moisture and volatile matter content of the material as received (pure seed and impurities), by drying at a temperature close to 103 °C, in a temperature-controlled oven at atmospheric pressure, until practically constant mass is reached.

3.2 If required, the moisture and volatile matter content of the pure seed alone may be determined.

4. Apparatus

4.1 Analytical balance.

4.2 Mechanical mill, easy to clean, suitable for the kind of seed and allowing the latter to be ground without heating and without appreciable change in moisture and oil content.

4.3 Mechanical grater or, if not available, hand grater.

4.4 Vessel, of metal resistant to attack, with flat bottom, provided with a well fitting lid and allowing the test sample to be spread to about 0·2 g/cm^2 (e.g. diameter of vessel 70 mm, height 30 to 40 mm). Glass vessels with ground closures may also be used by agreement between buyer and seller.

4.5 Temperature-controlled, electrically heated oven with good natural ventilation, regulated so that the temperature of the air and of the shelves in the neighbourhood of the samples lies between 101 and 105 °C in normal operation.

4.6 Desiccator containing an efficient desiccant such as phosphorus pentoxide, silica gel, activated alumina, etc, and provided with a metal plate which allows the vessels to cool rapidly.

5. Procedure

5.1 *Preparation of sample*

5.1.1 Use a sample for analysis, obtained as described in ISO Recommendation R 664, *Oleaginous seeds — Reduction of contract samples to samples for analysis*.[1] If large non-oleaginous foreign bodies have been separated before the reduction of the laboratory sample, make allowance for this in the calculation (see clause 6.3.1). According to the requirements of the contract, use an analysis sample as received or after separation of the impurities.

[1] See Annex II.

5.1.2 For copra, grate the product by hand or, preferably, by use of a mechanical grater (4.3) which allows the whole sample to be treated. With hand operation, which does not allow all the analysis sample to be grated, endeavour to obtain a sub-sample which is as representative as possible and, to this end, take account of the size and colour of different fragments.

The length of the particles may exceed 2 mm, but should not be greater than 5 mm. Mix the particles carefully and carry out the determination without delay.

5.1.3 For seeds of medium size (e.g. groundnut, soya, etc.), except sunflower seed and cottonseed with adherent linters, grind the analysis sample in the mechanical mill (4.2) which has previously been well cleaned, until the major dimension of the particles obtained is not greater than 2 mm. Reject the first particles (about 1/20 of the sample), collect the rest, mix carefully and carry out the determination without delay.

5.1.4 Small seeds (e.g. linseed, colza, hemp, etc) as well as safflower seed, sunflower seed and cottonseed with adherent linters, are analysed without previous grinding.

5.2 *Test portion*

5.2.1 Weigh the vessel (4.4) with its cover, after leaving it open for at least thirty minutes in the desiccator (4.6) at laboratory temperature.

5.2.2 Weigh into the vessel to the nearest 0·001 g,

either $5 \pm 0{\cdot}5$ g of the grated product (see clause 5.1.2) for copra, or meal (see clause 5.1.3) for medium-sized seeds other than sunflower seed or cotton seed with adherent linters,

or 5 to 10 g of whole seed for sunflower seed, cottonseed with adherent linters and small seeds.

Spread the material evenly over the whole base of the vessel and close the vessel by means of its cover. Weigh the whole.

5.2.3 Carry out these operations as quickly as possible, to avoid any appreciable change in moisture content

5.3 *Determination*

Put the vessel containing the test portion, with the cover removed, in the oven (4.5) which has previously been set to operate at 103 ± 2 °C. Close the oven. After three hours (twelve to sixteen hours for cottonseed with adherent linters), reckoned from the time when the temperature returns to 103 °C, open the oven, immediately close the vessel by means of its cover, and place the assembly in the desiccator. As soon as the vessel has cooled to laboratory temperature, weigh it.

Return the vessel, with cover removed, to the oven for one hour, take it out again after closing it, allow it to cool and weigh as before.

If the difference between the two weighings is equal to, or less than 0·005 g (for a test portion of 5 g), regard the operation as finished. If not, subject the sample to successive one-hour periods in the oven, until the difference between two successive weighings is equal to, or less than 0·005 g. Make all weighings to the nearest 0·001 g.

Carry out two determinations on the same prepared sample.

6. Expression of results

6.1 *Method of calculation and formula*

Calculate the moisture and volatile matter content to one place of decimals, as a percentage by mass of the material as received, by means of the formula

$$\text{moisture and volatile matter, per cent by mass} = \frac{M_1 - M_2}{M_1 - M_0} \times 100$$

where

M_0 is the mass, in grammes, of the vessel,

M_1 is the mass, in grammes, of the vessel and test portion before drying,

M_2 is the mass, in grammes, of the vessel and test portion after drying.

Take as the result the arithmetic mean of two parallel determinations, if the conditions of repeatability are satisfied. Otherwise, repeat the determination on two other test samples. If this time the difference again exceeds 0·2 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out, provided that the maximal difference between the individual determinations does not exceed 0·5 g per 100 g of sample.

Report the result to one decimal place.

6.2 *Repeatability*

The difference between the results of two determinations carried out simultaneously or in rapid succession should not exceed 0·2 g of moisture and volatile matter per 100 g of sample.

6.3 *Remarks*

6.3.1 If, before the analysis, large non-oleaginous foreign bodies have been separated from the sample (see clause 5.1.1), correct the result found above (see clause 6.1) by means of the formula

$$\text{moisture and volatile matter, per cent by mass} = h \times \frac{100 - X}{100}$$

where

h is the percentage, by mass, of moisture and volatile matter in the sample calculated from the formula given in clause 6.1,

X is the percentage, by mass, of large impurities previously separated, in the initial material as received.

6.3.2 If the determination of moisture and volatile matter has been carried out on pure seed, calculate also the moisture and volatile matter content by means of the formula given in clause 6.1.

7. Note on procedure

Never put moist products in the oven together with products that are nearly dry, as this will result in the latter being partially rehydrated.

8. Test report

The test report should show the method used and the result obtained, indicating clearly whether this represents the moisture and volatile matter content of the material as received or that of the pure seed. It should also mention all operating conditions not specified in this Recommendation, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report should also include all details required for complete identification of the sample.

ANNEX IV

ISO Recommendation R 658 (February 1968)

OLEAGINOUS SEEDS

DETERMINATION OF IMPURITIES

1. Scope

This ISO Recommendation describes a method for the determination of impurities in oleaginous seeds used as primary industrial materials, and defines the various categories of impurities as usually understood.

2. Definitions

2.1 By impurities are meant all foreign matter, organic and inorganic, other than seeds of the basic species.

2.2 By fines are meant the particles passing the sieves shown in Table 1 (see clause 5.2.1), according to the species being analysed.

In the case of groundnut, meal from the seeds contained in the fines is not regarded as an impurity.

2.3 By non-oleaginous impurities are meant non-oleaginous foreign bodies (bits of wood, pieces of metal, stones, seeds of non-oleaginous plants), fragments of stalks, leaves and all other non-oleaginous parts belonging to the oleaginous seed analysed (for example bits of shell, loose or adhering to palm kernels), retained by the sieves with holes of the diameters given shown in Table 1. In the case of seeds sold in their shell, for example sunflower seeds (*Helianthus annuus* Linnaeus) pumpkin seed (*Cucurbita pepo* Linnaeus), the loose shells are regarded as impurities only if their proportion is larger than that of the corresponding kernels present in the same sample.

2.4 By oleaginous impurities are meant foreign oleaginous seeds.

3. Principle

Separation of the impurities by sieving and sorting, into three categories:

— fines,

— non-oleaginous impurities,

— oleaginous impurities.

Determination of the mass of each category.

4. Apparatus

4.1 Sieves (see Table 1)

4.2 Tweezers or other suitable instruments

4.3 Analytical balance.

5. Procedure

5.1 *Test portion*

The test portion is the analysis sample obtained by reduction of the contract sample according to ISO Recommendation R 664, *Oleaginous seeds — Reduction of contract samples to samples for analysis.*[1]

Weigh the test portion with a precision of at least 0·1%.

[1] See Annex II.

5.2 *Determination*

The determination of impurities should be carried out sufficiently quickly to avoid any appreciable change in the moisture content of the seed.

5.2.1 *Separation of fines*

Separate the fines quantitatively by sieving the test portion, using a sieve with circular holes of the diameter shown in Table 1. Collect the fines and weigh them to the nearest 0·01 g.

TABLE 1

Diameter of holes of sieves

Name of product	Aperture diameter millimetres
Copra, medium-sized seeds	2·0
Small seeds (*Papaver somniferum* Linnaeus, *Brassica* sp., *Sinapis* sp., *Nicotiana* sp.)	0·5
All other oleaginous seeds	1·0

5.2.2.1 In the case of groundnut, collect the total fines thus obtained, which include non-oleaginous fines and fines from the seed, weigh these to the nearest 0·01 g and determine their oil content. Determine also the oil content of the pure seeds, in order to calculate the content of non-oleaginous fines.

5.2.2 *Separation of oleaginous and non-oleaginous impurities*

5.2.2.1 *General case* (copra, seeds of medium size)

In the material retained by the sieve shown in Table 1, separate by means of tweezers (or any other suitable instrument) the non-oleaginous impurities (2.3) on the one hand, if necessary detaching bits of shell adhering to the seeds (e.g. palm kernels), and the oleaginous impurities (2.4) on the other hand.

Weigh separately, to the nearest 0·01 g, each category of impurities.

If specified in the contract, note the nature of the oleaginous impurities in order that this may be recorded in the test report.

5.2.2.2 *Small seeds*

Transfer the residue from the sieve shown in Table 1 to a second sieve so as to retain impurities larger than the seeds (or separate these impurities by means of tweezers or any other suitable instrument).

Sort this fraction into non-oleaginous impurities (2.3) and oleaginous impurities (2.4).

Weigh separately, to the nearest 0·01 g, the fines and the two fractions of impurities (non-oleaginous and oleaginous) larger than the seeds, and also the partially sorted seeds.

Using an aliquot portion of the latter fraction of seeds (at least 10 g, weighed to the nearest 0·01 g), separate by sorting, on the one hand the non-oleaginous impurities of about the same size as the pure seeds, and on the other hand the small foreign oleaginous seeds. Weigh these two fractions of impurities to the nearest 0·001 g.

5.2.3 If required, the foreign oleaginous seeds may be grouped and weighed according to species, in order to show in the test report the percentage of each species.

5.2.4 Carry out two determinations on the same prepared sample.

6. Expression of results

6.1 *Method of calculation and formulae*

6.1.1 Show the content of each category of impurities as a percentage by mass of the seed as received. The sum of these represents the percentage of total impurities.

6.1.2 When the determination of impurities has been carried out on the whole test portion (see clause 5.2.2.1) the percentages are calculated as follows:

Fines, per cent $$P = M_1 \times \frac{100}{M_0}$$

Non-oleaginous impurities, per cent $$I_n = M_2 \times \frac{100}{M_0}$$

Oleaginous impurities, per cent $$I_0 = M_3 \times \frac{100}{M_0}$$

Total impurities, per cent $$I_t = P + I_n + I_0$$

where

M_1, M_2, M_3 are the masses, in grammes, of each category of impurities

M_0 is the mass, in grammes, of the test portion.

6.1.3 When only a part of the impurities is separated from the whole test portion and the other parts from an aliquot portion of the remainder (see clause 5.2.2.2), the percentages are calculated as follows:

Fines, per cent $$P = M_1 + \frac{100}{M_0}$$

Non-oleaginous impurities, per cent $$I_n = \left(M_{2a} + M_{2b} \times \frac{M_a}{M_b} \right) \times \frac{100}{M_0}$$

Oleaginous impurities, per cent $$I_0 = \left(M_{3a} + M_{3b} \times \frac{M_a}{M_b} \right) \times \frac{100}{M_0}$$

Total impurities, per cent $$I_t = P + I_n + I_0$$

where

M_1 is the mass, in grammes, of the fines,

M_{2a} is the mass, in grammes, of the fraction of non-oleaginous impurities larger than seeds of the basic species and separated from the whole test portion,

M_{3a} is the mass, in grammes, of the fraction of oleaginous impurities larger than seeds of the basic species and separated from the whole test portion,

M_{2b} is the mass, in grammes, of the fraction of non-oleaginous impurities of approximately the same size as seeds of the basic species and separated from the aliquot portion of the residue obtained by eliminating, from the test portion, fines and impurities larger than seeds of the basic species,

M_{3b} is the mass, in grammes, of the fraction of oleaginous impurities of approximately the same size as seeds of the basic species and separated from the aliquot portion of the residue obtained by eliminating, from the test portion, fines and impurities larger than seeds of the basic species.

M_0 is the mass, in grammes, of the original test portion,

M_a is the mass, in grammes, of the residue obtained by eliminating, from the test portion, fines and impurities larger than seeds of the basic species,

$$(M_a = M_0 - M_1 - M_{2a} - M_{3a})$$

M_b is the mass, in grammes, of the aliquot portion of the residue M_a, from which have been separated impurities of approximately the same size as the basic species.

6.1.4 In the case of groundnut the percentages are calculated as follows:

Total fines, per cent $$P = M_1 \times \frac{100}{M_0}$$

Foreign fines, per cent $$P_s = \frac{M_1 \times 100}{M_0}\left(1 - \frac{h}{H}\right)$$

Non-oleaginous impurities, per cent $$I_n = \frac{M_2 \times 100}{M_0}$$

Oleaginous, impurities, per cent $$I_o = \frac{M_3 \times 100}{M_0}$$

Total impurities, per cent $$I_t = P_s + I_n + I_0$$

where

M_1, M_2, M_3 are the masses, in grammes, of each category of impurities,

M_0 is the mass, in grammes, of the test portion,

H is the oil content, per cent by mass, of the pure seed,

h is the oil content, per cent by mass, of the fines.

6.1.5 Take as the result the arithmetic mean of two parallel determinations, if the conditions of repeatability are satisfied.

6.1.6 Show the results to two decimal places for contents not exceeding 0·5% and to one decimal place for contents above this limit.

6.2 *Repeatability*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed the percentage shown in Table 2.

TABLE 2

Permissible difference between results of two parallel determinations

Content of impurities %	Maximum permissible difference %
Up to and including 0·5	0·2
Over 0·5 to 1·0	0·4
Over 1·0 to 2·0	0·6
Over 2·0 to 3·0	0·8
Over 3·0 to 4·0	1·0
Over 4·0 to 5·0	1·2
Over 5·0 to 6·0	1·4
Over 6·0	1·6

If the difference is greater than the limit indicated in Table 2, obtain two other test portions, analyse one as above and keep the other for a fourth determination if necessary. In this case, take as the result the arithmetic mean of the result obtained at the third analysis and the nearest result obtained in the previous analyses, provided that the difference does not exceed the limit allowed.

Failing this, analyse also the fourth test portion and take as the result the mean of the four determinations.

7. Test report

The test report should show the method used and the results obtained. If the product contains foreign oleaginous seeds, and if stipulated by the contract, show not only their total percentage but also their nature.

If required, show the percentage of each species of foreign oleaginous seeds.

The test report should also mention all operating details not specified in this ISO Recommendation, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report should also include all details required for the complete identification of the sample.

ANNEX V

ISO Recommendation R 659 (February 1968)

OLEAGINOUS SEEDS

DETERMINATION OF OIL CONTENT

1. Scope

This ISO Recommendation describes a method for the determination of the oil content of oleaginous seeds used as primary industrial materials.

2. Definition

By oil is meant the total quantity of substances extracted under the operating conditions given below.

3. Principle

3.1 Determination of the oil content of the material as received (pure seed plus impurities) by extraction in a suitable apparatus, with a suitable solvent, n-Hexane or light petroleum.

3.2 It is possible, if required, to analyse the pure seed and the impurities separately.

3.3 In the case of groundnuts it is possible, if required, to analyse the pure seed, the total fines, the non-oleaginous impurities and the oleaginous impurities separately.

4. Reagents

4.1 n-Hexane; failing this, light petroleum distilling between 40 and 60 °C and having a bromine value below 1. For either solvent the residue on complete evaporation should not exceed 0·002 g/100 ml.

4.2 Sand, washed with hydrochloric acid and calcined.

4.3 Pumice-stone, in small granules, previously dried.

4.4 Hydrochloric acid concentrated, d = 1·19.

5. Apparatus

5.1 Suitable extraction apparatus (capacity of flask, 200 to 250 ml).

5.2 Electric heating bath (sand-bath, water bath, etc.).

5.3 Analytical balance.

5.4 Electric oven with temperature regulation.

5.5 Mechanical mill, easy to clean, corresponding to the nature of the seeds and allowing grinding to be done without heating or appreciable loss of moisture and oil.

5.6 Mechanical grater or, failing this, hand grater.

5.7 Mortar and pestle, of porcelain, iron or bronze, or, preferably, suitable microgrinder.

5.8 Extraction thimble and cotton wool, free from matter soluble in n-Hexane or light petroleum.

5.9 Metal vessel, flat-bottomed, diameter about 100 mm, height about 40 mm.

5.10 Porous vessel of ceramic material, cylindrical, internal diameter 68 mm, external diameter 80 mm, height 85 mm, thickness of walls and base 6 mm.

5.11 Fumigation oven, with temperature control.

5.12 Pipette, 2 ml, graduated in tenths of a millilitre.

5.13 Watch glass, diameter 80 to 90 mm.

6. Procedure

6.1 *Preparation of sample*

6.1.1 Carry out the operations described below on the analysis sample obtained according to ISO Recommendation R 664, *Oleaginous seeds — Reduction of contract samples to samples for analysis*[1]. If, before the reduction of the contract sample, large non-oleaginous foreign bodies have been separated, this should be taken into account in the calculations (see clause 7.3.3.)

According to the requirements of the contract, use the analysis sample without, or after, separation of the impurities.

6.1.2 For copra, grate the product with a hand grater or preferably with the mechanical grater (5.6), which can deal with the whole sample for analysis. Hand operation does not allow the whole sample for analysis to be grated. Endeavour to obtain a sub-sample which is as representative as possible. The thickness and the colour of the different pieces should be taken into account.

The length of the grated particles may exceed 2 mm, but should not be greater than 5 mm.

Mix the particles carefully and carry out the determination without delay.

6.1.3 For medium-sized seeds (e.g. sunflower, groundnut, soya) with the exception of cottonseed with adherent linters, crush the sample for analysis, in the previously well cleaned mechanical mill (5.5), until particles are obtained with a major dimension not greater than 2 mm. Reject the first grindings (about 1/20 of the sample) and collect the rest. Mix carefully and carry out the determination without delay.

6.1.4 For cottonseed with adherent linters, weigh to the nearest 0·01 g in the tared metal vessel (5.9) about 60 g of the sample for analysis without separation of impurities. Put the vessel and seed in the oven (5.4) previously heated to 130 °C and leave to dry for two hours at 130 ± 2 °C, then remove the vessel from the oven and allow to cool in air for about thirty minutes. Transfer the dried seed to the porous

[1] See Annex II.

ceramic vessel (5.10), the inside walls and base of which have been previously moistened with 1·5 ml of concentrated hydrochloric acid (4.4) by means of the pipette (5.12), taking care that the acid is completely absorbed without forming adherent drops. Close the vessel with the watch glass (5.13) and put in the fumigation oven (5.11). Heat so as to reach 115 °C in thirty minutes; do not heat beyond this temperature and maintain it for another thirty minutes.

Take the vessel out of the oven, allow to cool for one hour in air, reweigh the treated seed to the nearest 0·01 g, then grind the seed in the mechanical mill (5.5) and continue as described in clause 6.1.3.

6.1.5 Small seeds (e.g. linseed, colza, etc) are analysed without previous mechanical grinding.

6.2 *Test portion*

6.2.1 The test portion should be representative of the analysis sample.

6.2.2 Weigh, to the nearest 0·01 g, about 10 g

— of the grated product (6.1.2), as soon sa it has been grated, in the case of copra,

— of the ground product (6.1.3) as soon as it has been ground, in the case of medium-sized seeds except cottonseed with adherent linters,

— of the ground product (6.1.4) as soon as it has been ground, in the case of cottonseed with adherent linters,

— of the previously well mixed sample, in the case of small seeds.

6.3 *Determination*

6.3.1 In the case of copra and medium-sized seeds, including cottonseed with adherent linters, put the test portion in the thimble (5.8) and plug it with a wad of cotton wool.

6.3.2 For small seeds, grind the test sample in the mortar or microgrinder (5.7), taking care not to leave any seeds intact. Transfer, without loss, the ground seeds to the thimble (5.8), using a spatula. Wipe the mortar and pestle, or the bowl of the micro-grinder and the spatula, with a wad of cotton wool (5.8) soaked with solvent (4.1) and plug the thimble with this wad.

6.3.3 In the case of groundnuts, a test portion of about 10 g may be put into the extraction thimble (5.8), this comprising the separated fractions of pure seeds, non-oleaginous impurities, oleaginous impurities and total fines, in quantities proportional to the quantities of these different constituents in the analysis sample.

6.3.4 When the seed is very damp (moisture content above 10%), put the filled thimble for some time in an oven at a temperature not higher than 80 °C to reduce the moisture content to less than 10%.

6.3.5 Weigh, to the nearest 0·001 g, two flasks A and B, each containing one or two granules of pumice-stone (4.3), previously dried at 103 ± 2 °C and cooled for at least one hour in a desiccator.

Put the thimble (5.8) containing the test portion into the extraction apparatus (5.1). Transfer to flask A the necessary quantity of solvent (4.1). Fit the flask to the extraction apparatus on the heating bath (5.2). Carry out the heating in such a way that the rate of reflux is at least three drops per second (moderate, not vigorous, boiling).

After extracting for four hours, allow to cool. Remove the thimble from the extractor and place it in a current of air in order to remove the greater part of the solvent contained.

Empty the thimble into the mortar (5.7), add about 10 g of sand (4.2) and triturate as finely as possible (if a micro-grinder is used, grind without adding sand). Replace the mixture in the thimble and the latter in the extractor, and continue the extraction for two hours, using the same flask A.

Leave to cool, remove the thimble again, eliminate the solvent and repeat the trituration as above (without further addition of sand). Carry out a third extraction for two hours, collecting the product this time in flask B.

Remove the greater part of the solvent, by distillation on a boiling water bath, from flask A and B.

Remove the last traces of solvent by heating the flasks for twenty minutes at 103 ± 2 °C. Assist the elimination either by blowing in air at intervals or by using reduced pressure. Leave the flasks to cool in a desiccator, for at least one hour, and weigh to the nearest 0·001 g.

Heat again for ten minutes under the same conditions, cool again and weigh.

The difference between these two weighings should not exceed 0·010 g. If it does so, heat again for ten minutes, until the difference in mass is not greater than 0·010 g. Keep a note of the final mass of flask A.

If the mass of oil in flask B does not exceed 0·010 g, the operation is complete. If it does so, carry out a fresh extraction for two hours, using flask B, until the mass of oil from the last extraction is not more than 0·010 g. Keep a note of the final mass of flask B.

6.3.6 The oil extracted should be clear. If it is not, determine the content of impurities. For this purpose dissolve the fatty matter in the solvent used for extraction, filter through a filter paper, previously dried at 103 ± 2 °C, to constant mass, wash the filter several times with the same solvent to remove the oil completely, dry again at 103 ± 2 °C to constant mass (to cool and weigh the filter paper, use a suitable vessel provided with a lid). Correct the result accordingly.

6.3.7 If it is required to determine the oil content of the pure seed, analyse the seed separated from the impurities, proceeding as for the material as received.

6.3.8 To ascertain the oil content of the impurities, carry out the analysis in the same way as for the pure seed, with the following differences:

— the test portion may be less than 10 g, without, however, going below 2 g,

— a single four hours extraction may be used, the small error in obtaining less than the true content of the material as received being negligible.

6.3.9 Carry out two determinations on the same prepared sample.

7. Expression of results

7.1 *Method of calculation and formula*

The oil content, as a percentage by mass of the material as received, is calculated from the formula

$$\text{Oil content, per cent by mass} = \frac{M_1}{M_0} \times 100$$

where

M_1 is the sum of the masses, in grammes, of oil found in flasks A and B at the last weighing.

M_0 is the mass, in grammes of the test portion extracted.

Take as the result the arithmetic mean of two parallel determinations, if the conditions of repeatability are satisfied. If not, repeat the analysis on two other test portions. If the difference still exceeds 0·4 g per 100 g of sample, take as the result the arithmetic mean of the four determinations.

Express the result to the first decimal place.

7.2 *Repeatability*

The difference between two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0·4 g of oil per 100 g of sample.

7.3 *Remarks*

7.3.1 The same formula (see clause 7.1) serves for calculating the oil content of pure seeds and also that of the impurities when the pure seeds and the impurities are analysed separately.

In this case the oil content, as a percentage by mass of the material as received (pure seeds and impurities), may be calculated from the formula

$$\text{Oil content, per cent by mass} = H_1 - \frac{P}{100}(H_1 - H_2)$$

where

H_1 is the percentage, by mass, of oil in the pure seeds,

H_2 is the percentage, by mass, of oil in the impurities,

P is the percentage, by mass, of impurities in the material as received.

7.3.2 In the case of cottonseed with adherent linters, the oil content, as a percentage by mass of the material as received, is equal to

$$\text{Oil content, per cent by mass} = \frac{M_1}{M_0} \times 1\,00 \times \frac{M''_0}{M'_0}$$

where

M_0 and M_1 have the same meaning as in clause 7.1,

M'_0 is the mass, in grammes, of the test portion (about 60 g) before pre-treatment (see clause 6.1.4),

M''_0 is the mass, in grammes, of the same test portion after pre-treatment (see clause 6.1.4) and before grinding.

7.3.3 If, before the analysis, the large non-oleaginous foreign bodies have been separated (see clause 6.1.1.) the result obtained above (see clauses 7.1, 7.3.1 or 7.3.2) for the oil content of the material as received should be corrected according to the formula

$$\text{Oil content, per cent by mass} = \frac{H_0}{100} \times (100 - X)$$

where

H_0 is the percentage, by mass, of oil in the material analysed (calculated according to clauses 7.1, 7.3.1 or 7.3.2 as appropriate)

X is the percentage content, by mass, of large non-oleaginous foreign bodies previously separated in the original material as received.

7.3.4 In the case of groundnut, the oil content, as a percentage by mass of the material as received, is equal to

$$\text{Oil content, per cent by mass} = H_1 - \frac{P + I_0 + I_n}{100} \times (H_1 - H_2)$$

where

P is the percentage, by mass, of total fines,

I_0 is the percentage, by mass, of oleaginous impurities,

I_n is the percentage, by mass, of non-oleaginous impurities,

H_1 is the percentage, by mass, of oil in the pure seeds,

H_2 is the percentage, by mass, of oil in the impurities.

If the extraction has been carried out in a single thimble, calculate the oil content according to clause 7.1.

7.3.5 If required, the oil content may be expressed on the dry matter and calculated from the formula

$$\text{Oil content, per cent by mass} = H_0 \times \frac{100}{100 - U}$$

where

H_0 is the percentage, by mass, of oil in the material as received,

U is the moisture content, per cent, by mass.

8. Note on procedure

In the case of semi-drying oils, it is preferable to remove traces of solvent by drying at reduced pressure.

9. Test report

The test report should show the method used and the result obtained, indicating clearly whether this analysis represents the oil content of the seed as received, or the oil content of the pure seed related to the dry matter. The solvent used should also be stated. The report should also mention all operating details not specified in this ISO Recommendation, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report should include all details required for the complete identification of the sample.