TC 34

International Standard 5511

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Oilseeds — Determination of oil content — Low resolution nuclear magnetic resonance spectrometric method

Graines oléagineuses — Détermination de la teneur en huile — Méthode par spectrométrie de résonance magnétique nucléaire à basse résolution

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Oilseeds — Determination of oil content — Low resolution nuclear magnetic resonance spectrometric method

0 Introduction

A reference method for the determination of the oil content of oilseeds is specified in ISO 659.

1 Scope and field of application

This International Standard specifies a rapid method for the determination of the oil content of oilseeds.[1)]

Under normal conditions of use, it does not apply to oilseeds which do not yield oil which is completely liquid at 20 °C (shea, palm, illipe, cocoa, etc.).

2 References

ISO 659, *Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content".*

ISO 665, *Oilseeds — Determination of moisture and volatile matter content.*

ISO 771, *Oilseed residues — Determination of moisture and volatile matter content.*

3 Definition

oil content: All organic substances which are liquid at the temperature of measurement (in principle 20 °C) contained in the oilseeds, determined by the method specified in this International Standard.

4 Principle

Determination by low resolution nuclear magnetic resonance (NMR) spectrometry of the content of liquid components containing hydrogen which are present in oilseeds which have been previously dried at 103 ± 2 °C, and taking into account the effect of solids (oilseed residue).

5 Materials

5.1 Crude oil from seeds of the same botanical species, and of similar geographical origin and chemical composition to those of the seeds for analysis, extracted in the laboratory carrying out the analysis, in accordance with the method specified in ISO 659, less than 1 month previously.

Store under conditions protecting the oil from oxidation.

5.2 Oilseed residue from seeds of the same botanical species, and of similar geographical origin and chemical composition to those of the seeds for analysis, from which the oil has been extracted in the laboratory carrying out the analysis, in accordance with the method specified in ISO 659, less than 1 month previously.

Dry the residue at 103 ± 2 °C in accordance with the method specified in ISO 771.

6 Apparatus

Ordinary laboratory equipment, and in particular

6.1 Apparatus required for the drying method (see ISO 665).

6.2 Continuous wave, low resolution NMR spectrometer.

NOTE — The use of a pulsed low resolution NMR spectrometer is permitted, provided that it is capable of operating on a test portion of at least 10 g.

6.3 Measuring tubes, preferably with closures, suitable for use with the NMR spectrometer (6.2) and of the largest possible capacity, made from a non-conducting non-magnetic material which does not contain hydrogen [for example glass, polytetrafluoroethylene (PTFE)].

6.4 Desiccator.

1) This method has been successfully tested on the following: colza, soya, sunflower seed and groundnuts.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, that quantity of seed, which shall be at least 20 g, necessary to fill the measuring tube (6.3) sufficiently.

NOTE — Some instruments, however, cannot accommodate 20 g of some species of seed, which means that the test portion is less representative.

7.2 Drying

The presence of particles of ferrous metals falsifies the results; they shall be removed using a magnet.

Dry the test portion and determine its moisture content in accordance with the method specified in ISO 665.

Place the desiccator containing the dried seeds in immediate proximity to the NMR spectrometer in a room in which the temperature (generally about 20 °C) does not show sudden variations and leave for several hours (at least 3 h) before carrying out the determination so that the seed and the apparatus are at the same temperature.

NOTES

1 If possible, use a temperature-controlled room.

2 The effect of temperature variation on the results obtained is very large: the oil response decreases by approximately 0,3 % per degree Celsius. Consequently, all operations for calibrating the instrument and all measuring operations should be carried out strictly at the same temperature. It is recommended, if the necessary apparatus is available, to control thermostatically the "effective" part of the instrument and to place the seeds, before measurement, in insulated metal blocks at the measuring temperature. Otherwise, carry out a simplified calibration of the apparatus every 30 min.

7.3 Calibration of the instrument

Adjust the NMR spectrometer (6.2) in accordance with the manufacturer's instructions, then adjust to zero after placing an empty measuring tube (6.3) in the magnetic field.

Into four measuring tubes identical to that used for the adjustment (i.e. also giving a reading of zero when they are placed empty in the magnetic field), weigh, to the nearest 0,01 g, 5; 10; 20; and 30 g, if the apparatus so permits, of the crude oil (5.1), taking care that no drop of oil adheres to the sides of the tube above its effective part.

Measure the response of the instruments for the four tubes, taking R_5, R_{10}, R_{20} and R_{30} as the mean of five readings of the integrator on each of the four tubes, during the integration time giving the best repeatability of measurement, using a level of radiofrequency energy sufficient for obtaining a satisfactory signal in relation to the background noise, but not producing more than approximately 1 % saturation. The correct selection of the level of saturation may be carried out by the operator if the relation between the radiofrequency energy and the saturation is known. If not, the correct adjustment of the level of saturation may be obtained from the manufacturer of the instrument. State in the test report the name and type of instrument used and all the adjustments carried out.

Draw a straight line representing the values of R as a function of the mass of oil placed in the tubes. This line shall pass through the origin. If it does not, contact the manufacturer to adjust the instrument or make the necessary adjustment if the instrument so permits.

Check the calibration of the instrument regularly (if possible, once each day). If the instrument and the samples are not thermostatically controlled, check the variation in the gradient of the calibration line by measuring the response of a 20 or 30 g sample every 30 min.

7.4 Measurement

Transfer quantitatively and rapidly the dry seeds prepared in 7.2 into a measuring tube (6.3) identical to the one used for the adjustment. If possible, close the tube to prevent absorption of moisture.

Without altering the adjustment of the instrument, measure its response. Let R be the mean value of this response for five readings of the integrator using the integration time previously chosen.

7.5 Correction for oilseed residue

Into a measuring tube (6.3) identical to the one used for the adjustment, weigh exactly the quantity (mass m'_0) of dried oilseed residue from which the oil has been extracted (see 5.2) required to fill the effective part of the tube, and keep it at room temperature.

Without altering the adjustment of the instrument, measure its response and take as the result the mean value of this response for five readings of the integrator using the integration time previously chosen.

Carry out this operation again on the same mass (m'_0), successively with 5 to 10 different samples of the oilseed residue (5.2), taking T as the mean value of the responses obtained. This value needs to be determined only about once per month.

NOTE — As a guide, colza seed data have shown that, without correction, this method gives higher results than the reference method specified in ISO 659 by approximately, on average, 0,30 g of oil per 100 g of sample.

7.6 Number of determinations

Carry out two determinations on test portions taken from the same test sample.

8 Expression of results

8.1 Method of calculation and formulae

8.1.1 The apparent oil content of the seeds, h, expressed as a percentage by mass, is equal to

$$\frac{R \times m_x \times 100}{R_x \times m_0}$$

where

m_0 is the mass, in grams, of the test portion before drying (7.1);

m_x is the mass, in grams, of the sample corresponding to approximately x g of crude oil (20 or 30 g);

R is the response of the instrument for the test portion, determined in 7.4;

R_x is the response of the instrument for x g of crude oil, determined in 7.3 (R_{20} or R_{30}).

8.1.2 The response of the residue, t, conventionally expressed as a percentage by mass, is equal to

$$\frac{T \times m_x \times 100}{R_x \times m'_0}$$

where

m'_0 is the mass, in grams, of the dehydrated residue from which the oil has been extracted, used in 7.5;

T is the mean value of the responses of the instrument for the m'_0 grams of dehydrated residue from which the oil has been extracted, determined in 7.5;

m_x and R_x have the same meanings as in 8.1.1.

8.1.3 The oil content of the seeds, expressed as a percentage by mass, is equal to

$$\frac{h - t(1 - 0{,}01H)}{1 - 0{,}01t}$$

where

H is the moisture content, expressed as a percentage by mass, of the seeds, determined in accordance with ISO 665;

h and t are the values calculated in 8.1.1 and 8.1.2, respectively.

NOTE — For a given type of seed, the response of the residue is generally independent of the samples. For example, in the case of colza (see the note to 7.5), the oil content of the seeds is approximately equal to

$h - 0{,}30$

8.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst, shall not exceed 0,6 g of oil per 100 g of sample.

9 Test report

The test report shall show the method used, the results obtained, the name and type of NMR spectrometer used and, if applicable, all adjustments made. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

The test report shall include all the information necessary for the complete identification of the sample.