COMMISSION REGULATION (EEC) No 3519/84

of 14 December 1984

amending Regulation (EEC) No 1470/68 on the drawing and reduction of samples and the determination of the oil content, impurities and moisture in oilseeds

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 of 22 September 1966 on the common organization of the market in oils and fats (1), as last amended by Regulation (EEC) No 2260/84 (2), and in particular Article 27 (5) thereof,

Whereas Commission Regulation (EEC) No 1470/68 (3), as last amended by Regulation (EEC) No 1223/81 (4), lays down the method for determining the oil content of oilseeds, namely that set out in Annex V thereto; whereas a new method, using nuclear magnetic resonance spectrometry, was recently developed; whereas Member States should be authorized to use that method;

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

Regulation (EEC) No 1470/68 is hereby amended as follows:

1. Article 2 (1) is hereby replaced by the following:

'1. The determination of oil content required under Article 4 of Regulation No 282/67/EEC and Article 32 of Commission Regulation (EEC) No 2681/83 (1) shall be carried out in accordance with the method defined in Annex V to this Regulation.

Member States may, however, in the case of colza and rapeseed, also use the method referred to in Annex VII to this Regulation. In such cases, where the results of the tests are in dispute, the method referred to in Annex V shall be used and its results shall prevail.

(1) OJ No L 266, 28. 9. 1983, p. 1.'

2. A new Annex VII is added, the text of which is as set out in the Annex to this Regulation.

Article 2

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, 14 December 1984.

For the Commission

Poul DALSAGER

Member of the Commission

(1) OJ No 172, 30. 9. 1966, p. 3025/66.
(2) OJ No L 208, 3. 8. 1984, p. 1.
(3) OJ No L 239, 28. 9. 1968, p. 2.
(4) OJ No L 124, 8. 5. 1981, p. 10.

ANNEX

'ANNEX VII

DETERMINATION OF THE OIL CONTENT OF COLZA AND RAPESEED BY NUCLEAR MAGNETIC RESONANCE SPECTROMETRY

1. SCOPE AND FIELD OF APPLICATION

The method described is for the determination of the oil content of colza and rapeseed.

2. PRINCIPLE

Determination by low resolution nuclear magnetic resonance (NMR) of the response of liquid compounds containing hydrogen in seed which have been previously dried. Calculation of the oil content from the NMR response value obtained after making allowance for the response due to the non-fatty matter contained in the seed.

3. APPARATUS

3.1. Nuclear Magnetic Resonance Spectrometer, continuous wave low-resolution type, installed in a temperature-controlled room.

3.2. Nessler cylinders, 100 ml, calibrated at 40 ml (approximately 50 mm from the bottom of the tube) suitable for use with the NMR spectrometer (3.1).

3.3. Oven, regulated at 103 ± 2 °C.

3.4. Teflon stopper with holder to fit Nessler cylinders (3.2).

3.5. Desiccator.

4. SAMPLING

4.1. Draw the sample as described in Annex I to Commission Regulation (EEC) No 1470/68.

4.2. Reduce the drawn sample for analysis as described in Annex II to Commission Regulation (EEC) No 1470/68.

5. PROCEDURE

5.1. **Preparation of reference standards of rapeseed oil and extracted rapeseed residue**

5.1.1. Select a reference sample of rapeseed (clean and free from impurities) in which the fatty acid composition of the oil corresponds as closely as possible to that of the rapeseed to be analyzed for oil content.

Dry a weighed quantity of the seed selected as the reference (at least 250 g) at 103 ± 2 °C for 17 hours ± 1 hour according to the method laid down in Annex III to Regulation (EEC) No 1470/68. Cool in a desiccator (3.5).

5.1.2. Extract the oil from the dried reference seed (5.1.1) according to the method laid down in Regulation (EEC) No 1470/68.

Transfer the extracted oil to a clean, dry, glass container which can be readily and effectively sealed. Retain the defatted residue, dry it in an oven (3.3) at 103 ± 2 °C for 17 hours ; cool the residue in a desiccator (3.5), weigh and store it in an airtight container.

5.2. **Calibration of the spectrometer**

5.2.1. Weigh accurately (to within 10 mg) 5 g, 10 g, 20 g and 30 g quantities of the dry extracted reference oil (5.1.2) into individual Nessler cylinders (3.2), taking care to prevent any droplets of oil adhering to the walls of the cylinders above their operative part.

5.2.2. Introduce an empty Nessler cylinder (3.2) into the NMR spectrometer (3.1) and adjust the response of the instrument to zero following the manufacturer's instructions.

5.2.3. Measure the response (R_5, R_{10}, R_{20}, R_{30}) for each of the tubes containing the reference oil standards calculating the mean value of three repetitive readings for the response of each oil standard.

5.2.4. Plot a graph with the mean of the three readings for each of the reference oil standards as coordinates and the mass of the standards as abscissae. A straight line passing through the origin should be obtained ; if not, the adjustment of the spectrometer should be checked.

5.3. **Measurement**

5.3.1. Dry the test sample (clean and free from impurities) (4.1) as described for the reference standard (5.1.1).

Note :

After cooling, the dried test samples should be transferred to the room in which the NMR spectrometer is installed, at least one hour before analysis.

5.3.2. Weigh 25,00 g of the dried test sample into a Nessler tube (3.2) and push the teflon stopper down by the holder (3.4) towards the surface of the seed.

Note :

A 25-g sample of rapeseed occupies a volume of about 40 ml and makes the most effective use of a standard 40-ml capacity instrument. Test samples of more than 25 g may be analyzed using instruments equipped with a sample assembly designed to allow the analysis of test samples occupying a volume greater than 40 ml.

5.3.3. Transfer the tube containing the dried test sample to the NMR spectrometer (3.1) and measure the response (R_s) where R_s is the mean of three repetitive readings.

5.3.4. Repeat the measurement with a furhter dried test sample of 25,00 g.

5.3.5. Fill a tared Nessler cylinder (3.2) to the 40 ml calibration mark with the reference dried defatted seed residue (5.1.2). Weigh the cylinder and its contents and calculate the mass of seed residue (in the cylinder). Transfer the Nessler tube to the NMR spectrometer and measure the response (R_r) following the same procedure as for the test sample.

6. EXPRESSION OF RESULTS

6.1. **Calculation of oil content on dry weight basis**

The apparent oil content of the dry seeds, (0) as per cent (w/w) is given by the formula :

$$O = \frac{R_s \cdot m_x \cdot 100}{R_x \cdot m_{ds}}$$

where :

R_s is the NMR response of a sample as determined in 5.3.3 ;

R_x is the NMR response of x g crude oil (20 or 30 g) as determined in 5.2.3 ;

m_x is the mass in g of the sample of x g crude oil (20 or 30 g) ;

m_{ds} is the mass in g of the dry seed sample (25,00 g).

The contribution of the residue, (C) as per cent (w/w) to the apparent oil content (0) is given by the formula :

$$C = \frac{R_r \cdot m_x \cdot m_t \cdot 100}{R_x \cdot m_{ds} \cdot m_r}$$

where :

R_r is the NMR response of the dry residue as determined in 5.3.5 ;

m_r is the mass in g of the test portion of dry residue ;

m_t is the mass in g of the residue equivalent to that from 25,00 g dry seeds as determined in 5.1.

The oil content of the dry seeds, as per cent (w/w) is thus given by the formula :

$$\text{oil \% (dry seed)} = O - C = \left(R_s - R_r \frac{m_t}{m_r}\right) \cdot \frac{m_x \cdot 100}{R_x \cdot m_{ds}}$$

6.2. **Calculation of oil content on basis telle quelle**

The oil content of the fresh seeds, as per cent (w/w) is given by the formula :

$$\text{oil \% (telle quelle)} = \left(R_s - R_r \cdot \frac{m_t}{m_r}\right) \cdot \frac{m_x \cdot 100}{R_x \cdot m_{ds}} \cdot \left(1 - \frac{a}{100}\right)$$

where :

a is the moisture content of the seeds in per cent (w/w) as determined according to Annex III to Commission Regulation (EEC) No 1470/68.

6.3. **Repeatability**

6.3.1. The difference between oil determinations of the two test samples must not exceed 0,4 g of oil per 100 g of sample.

6.3.2. If the difference between the two test samples exceeds 0,4 g of oil per 100 g of sample, the measurement of the two samples must be repeated.

6.3.3. If the difference between the two test samples still exceeds 0,4 g of oil per 100 g of sample, new samples shall be drawn from the sample for analysis, and the whole procedure is repeated.

6.3.4. If the difference between the determinations of the new sample set exceeds 0,4 g of oil per 100 g of sample, the oil content of the sample in question shall be determined in accordance with the method described in Annex V of Commission Regulation (EEC) No 1470/68.

7. NOTES

7.1. **Accuracy of oil determination**

The accuracy of the determination of oil content by NMR spectrometry shall be checked at least once a month by comparing the NMR results with results obtained by extraction with n-hexane in accordance with the method described in Annex V of Commission Regulation (EEC) No 1470/68.

Routinely this is done simultaneously with the monthly preparation of the oil reference sample (5.1).

The difference between the results obtained by NMR and extraction with n-hexane must not exceed 0,4 g of oil per 100 g of sample.

If the difference between the results obtained by NMR and extraction with n-hexane exceeds 0,4 g of oil per 100 g of sample, the extraction is repeated and a new calibration of the NMR spectrometer is performed as described in clause 5.2.

7.2. **Interference by ferrous metals**

Trace amounts of ferrous metals interfere with the results. It is recommended to remove such compounds with a magnet.

If the presence of ferrous metals constitutes a problem, it is recommended that the oil content of the sample in question is determined in accordance with the method described in Annex V of Commission Regulation (EEC) No 1470/68.'