

1985 No. 194

AGRICULTURE

**The Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations
(Northern Ireland) 1985***Made* 26th July 1985*Coming into operation* 16th January 1986

The Department of Agriculture, in exercise of the powers conferred on it by sections 66(1), 75(1), 76(1), 77(4), 84 and 86 of the Agriculture Act 1970(a) and of every other power enabling it in that behalf, after consultation with such persons or organisations as appear to it to represent the interests concerned, hereby makes the following regulations:—

Title, commencement and interpretation

1.—(1) These regulations may be cited as the Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1985, and shall come into operation on 16th January 1986.

(2) In these regulations “the principal regulations” means the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982(b).

(3) The Interpretation Act (Northern Ireland) 1954(c) shall apply to these regulations as it applies to a Measure of the Northern Ireland Assembly.

Amendment of the principal regulations

2.—(1) In Schedule 1 to the principal regulations for the words “the final sample” in Part IV, paragraph 3(b)(i) there shall be substituted the words “the reduced sample”.

(2) In Schedule 2 to the principal regulations—

(a) for items 3a and b of paragraph 3 (Methods of Analysis) there shall be substituted “3. Crude oils and fats.”;

(b) for the methods of analysis 3a. (OIL — IN THE PRESENCE OF MILK POWDER) and 3b. (OIL — IN THE ABSENCE OF MILK POWDER) there shall be substituted the method of analysis for crude oils and fats set out in the Schedule to these regulations;

(c) for the words “20 ml saturated solution of ammonium ferric sulphate” in paragraph 5.2 of the method of analysis 14. (WATER-SOLUBLE CHLORIDES) there shall be substituted the words “2 ml saturated solution of ammonium ferric sulphate”;

(d) for the formula for calculating the weight of chlorine given in paragraph 6 of the method of analysis 14. (WATER-SOLUBLE CHLORIDES) there shall be substituted the following formula:

$$“[W = 5.845 \times (V_1 - V_2)]”;$$
 and

(a) 1970 c. 40; amended by the Agriculture Act 1970 Amendment Regulations 1982 (S.I. 1982/980)

(b) S.R. 1982 No. 338; previously amended by S.R. 1984 No. 26

(c) 1954 c. 33 (N.I.)

(e) for the words "sulphuric acid solution (3.5)" on their first appearance in paragraph 5.1.1. of the method of analysis 17. (IRON, COPPER, MANGANESE AND ZINC) there shall be substituted the words "hydrochloric acid solution (3.2)".

Sealed with the Official Seal of the Department of Agriculture for Northern Ireland on 26th July 1985.

(L.S.)

R. E. Aiken

Assistant Secretary

3. CRUDE OILS AND FATS

1. Scope and Field of Application

This method is for the determination of the content of crude oils and fats in feeding stuffs. It is not applicable to the analysis of oil seeds or oleaginous fruit(a). The method involves two procedures which are to be applied according to the nature of the feeding stuff to be analysed:—

Procedure A is applicable to straight feeding stuffs of plant origin, with the exception of those which are known to contain oils and fats which cannot be totally extracted with light petroleum without prior hydrolysis. Among these are gluteins, yeasts, soya and potato proteins. This procedure is also applicable to compound feeding stuffs, with the exception of those which contain milk powder, or from which oils and fats cannot be totally extracted with light petroleum without prior hydrolysis.

Procedure B is applicable to straight feeding stuffs of animal origin as well as to the feeding stuffs mentioned above as exceptions to the application of procedure A.

2. Principle

In procedure A the sample is extracted with light petroleum. The solvent is removed by distillation and the residue dried and weighed.

In procedure B the sample is pre-treated by heating with hydrochloric acid. The mixture is cooled and filtered. The residue is washed, dried and submitted to the determination according to procedure A.

3. Reagents

- 3.1 Light petroleum, boiling range 40-60°C. The bromine value must be less than 1 and the residue of evaporation less than 2 mg/100 ml.
- 3.2 Sodium sulphate, anhydrous.
- 3.3 Hydrochloric acid 3N.
- 3.4 Filtration aid, e.g. Kieselguhr, Hyflo-supercel.

4. Apparatus

- 4.1 Extraction apparatus. If fitted with a siphon (Soxhlet apparatus), the reflux rate should be such as to produce about 10 cycles per hour; if of the non-siphoning type, the reflux rate should be about 10 ml per minute.
- 4.2 Extraction thimbles, free of matter soluble in light petroleum and having a porosity consistent with the requirements of 4.1 above.
- 4.3 Drying oven, either a vacuum oven set at $75 \pm 3^\circ\text{C}$ or an air-oven set at $100 \pm 3^\circ\text{C}$.

5. Procedures

5.1 *Procedure A* (see 8.1 below)

Weigh approximately 5g of the sample to the nearest mg, transfer it to an extraction thimble (4.2) and cover with a wad of fat-free cotton wool.

Place the thimble in an extractor (4.1) and extract for six hours with light petroleum (3.1). Collect the light petroleum extract in a dry, weighed flask containing fragments of pumice stone. (Where the oil or fat has to undergo subsequent quality tests, use glass beads in the place of fragments of pumice stone.)

Remove the solvent by distillation. Dry the residue, maintaining the flask for one and a half hours in the drying oven (4.3). Leave to cool in a desiccator and weigh. Dry again for 30 minutes to ensure that the weight of the oils and fats remains constant (loss in weight between two successive weighings must be less than 1 mg).

(a) As to which see Council Regulation No. 136/66/EEC of 22 September 1966

5.2 Procedure B

Weigh approximately 2.5 g of the sample to the nearest mg (see 8.2), place in a 400 ml beaker or a 250 ml conical flask and add 100 ml of hydrochloric acid 3N (3.3) and fragments of pumice stone. Cover the beaker with a watch glass or fit the conical flask with a reflux condenser. Bring the mixture to a gentle boil over a low flame or a hot-plate and maintain under these conditions for one hour. Do not allow the product to stick to the sides of the container.

Cool and add approximately 1-2 g of filtration aid (3.4) sufficient to prevent any loss of oil and fat during filtration. Filter through a moistened, fat-free, double filter paper, such as a Whatman 541, of 18.5 cm diameter. Wash the residue with cold water until a neutral filtrate is obtained. Check that the filtrate does not contain any oil or fats. Their presence indicates that the sample must be extracted with light petroleum, using procedure A, before hydrolysis.

Place the double filter paper containing the residue on a watch glass and dry for one and a half hours in the oven at $100 \pm 3^\circ\text{C}$.

Place the double filter paper containing the dry residue in an extraction thimble (4.2) and cover with a wad of fat-free cotton wool. Place the thimble in an extractor (4.1) and proceed as indicated in the second and third paragraphs of 5.1.

6. Expression of result

Express the weight of the residue as a percentage of the sample.

7. Repeatability

The difference between the results of two parallel determinations carried out on the same sample by the same analyst should not exceed—

0.2%, in absolute value, for contents of crude oils and fats lower than 5%;

4.0%, related to the highest result, for contents of 5 to 10%;

0.4%, in absolute value, for contents above 10%.

8. Observations

8.1 For products with a high content of oils and fats, which are difficult to crush or unsuitable for drawing a homogeneous test sample, proceed as follows:

Weigh approximately 20 g of the sample to the nearest mg and mix with 10 g or more of anhydrous sodium sulphate (3.2). Extract with light petroleum (3.1) as indicated in 5.1. Make up the extract obtained to 500 ml. Take 50 ml of the solution and place in a small, dry, weighed flask containing fragments of pumice stone. (When the oil or fat has to undergo subsequent quality tests, use glass beads in the place of fragments of pumice stone.) Distil off the solvent, dry and proceed as indicated in the last paragraph of 5.1.

Eliminate the solvent from the extraction residue left in the thimble, crush the residue to a fineness of 1 mm, return it to the extraction thimble (do not add sodium sulphate) and proceed as indicated in the second and third paragraphs of 5.1.

Calculate the content of oils and fats as a percentage of the sample by using the following formula:

$$(10a + b) \frac{100}{w}$$

where—

a = mass in grams of the residue from the aliquot part of the extract from the first extraction,

b = mass in grams of the residue after the second extraction,

w = mass of sample in grams.

8.2 For products low in oils and fats the test sample may be increased to 5 g.

EXPLANATORY NOTE

(This note is not part of the Regulations.)

These regulations amend for the second time the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982, ("the regulations of 1982") in partial implementation of the Commission Directive 84/4/EEC (O.J. No. L 15, 18.1.84, p. 28), which among other provisions amends the Directive 71/393/EEC establishing Community methods of analysis for the official control of feeding stuffs, (O.J. No. L 279, 20.12.71, p. 7).

The regulations substitute a new method of analysis of feeding stuffs for the determination of crude oils and fats for the methods originally provided in Schedule 2 to the regulations of 1982, (regulation 2(2)(a) and (b)).

In addition to the implementation described above the regulations correct certain errors which appeared in the methods of sampling and analysis prescribed in Schedules 1 and 2 to the regulations of 1982.