

II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE 92/89/EEC

of 3 November 1992

amending Annex I to Fourth Directive 73/46/EEC establishing Community methods of analysis for the official control of feedingstuffs

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 70/373/EEC of 20 July 1970 on the introduction of Community methods of sampling and analysis for the official control of feedingstuffs⁽¹⁾, as last amended by Regulation (EEC) No 3768/85⁽²⁾, and in particular Article 2 thereof,

Whereas Fourth Commission Directive 73/46/EEC⁽³⁾, as amended by Directive 81/680/EEC⁽⁴⁾, prescribes the method to be used for the determination of crude fibre;

Whereas there are grounds to adapt this method in the light of advances in scientific and technical knowledge; whereas it is advisable, in particular, to take account of the provisions of Council Directive 80/1107/EEC of 27 November 1980 on the protection of workers from the risks related to exposure to chemical, physical and biological agents at work⁽⁵⁾, as amended by Directive 88/642/EEC⁽⁶⁾, and, in particular, the measures taken to prevent exposure to asbestos;

Whereas it is from now on necessary to replace asbestos by glass containers in order to carry out the filtration procedures;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Feedingstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Annex I to Directive 73/46/EEC is amended in accordance with this Directive.

Article 2

Member States shall bring into force not later than 1 October 1993 the laws, regulations and administrative provisions necessary to comply with the provisions of this Directive. They shall forthwith inform the Commission thereof.

When Member States adopt these provisions, these shall contain a reference to this Directive or shall be accompanied by such reference at the time of their official publication. The procedure for such reference shall be adopted by the Member States.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 3 November 1992.

For the Commission

Ray MAC SHARRY

Member of the Commission

⁽¹⁾ OJ No L 170, 3. 8. 1970, p. 2.

⁽²⁾ OJ No L 362, 31. 12. 1985, p. 8.

⁽³⁾ OJ No L 83, 30. 3. 1973, p. 21.

⁽⁴⁾ OJ No L 246, 29. 8. 1981, p. 32.

⁽⁵⁾ OJ No L 327, 3. 12. 1980, p. 8.

⁽⁶⁾ OJ No L 356, 24. 12. 1988, p. 74.

ANNEX

Annex I, point 3, 'Determination of crude fibre' is replaced by the following:

'DETERMINATION OF CRUDE FIBRE**1. Purpose and scope**

This method makes it possible to determine fat-free organic substances in feedingstuffs which are insoluble in acid and alkaline media and are conventionally described as crude fibre.

2. Principle

The sample, defatted where necessary, is treated successively with boiling solutions of sulphuric acid and potassium hydroxide of specified concentrations. The residue is separated by filtration on a sintered-glass filter washed, dried, weighed and ashed within a range of 475 to 500 °C. The loss of weight resulting from ashing corresponds to the crude fibre present in the test sample.

3. Reagents

- 3.1. Sulphuric acid, $c = 0,13$ mol/l.
- 3.2. Anti-foaming agent (e.g. n-octanol).
- 3.3. Filter aid (Celite 545 or equivalent), heated at 500 °C for four hours (8.6).
- 3.4. Acetone.
- 3.5. Light petroleum boiling-range 40 to 60 °C.
- 3.6. Hydrochloric acid, $c = 0,5$ mol/l.
- 3.7. Potassium hydroxide solution, $c = 0,23$ mol/l.

4. Apparatus

- 4.1. Heating unit for digestion with sulphuric acid or potassium hydroxide solution, equipped with a support for the filter crucible (4.2) and provided with an outlet tube with a tap to the liquid outlet and vacuum, possibly with compressed air. Before use each day preheat the unit with boiling water for five minutes.
- 4.2. Glass filter crucible with fused sintered glass filter plate pore size 40-90 μm . Before first use, heat to 500 °C for a few minutes and cool (8.6).
- 4.3. Cylinder of at least 270 ml with a reflux condenser, suitable for boiling.
- 4.4. Drying oven with thermostat.
- 4.5. Muffle furnace with thermostat.
- 4.6. Extraction unit consisting of a support plate for the filter crucible (4.2.) and with a discharge pipe with a tap to the vacuum and liquid outlet.
- 4.7. Connecting rings to assemble the heating unit (4.1), crucible (4.2) and cylinder (4.3) and to connect the cold extraction unit (4.6) and crucible.

5. Procedure

Weigh out to the nearest 0,001 g, 1 g of the prepared sample and place it in the crucible (4.2), (see observations 8.1, 8.2 and 8.3) and add 1 g of filter aid (3.3).

Assemble the heating unit (4.1) and the filter crucible (4.2), then attach the cylinder (4.3) to the crucible. Pour 150 ml of boiling sulphuric acid (3.1) into the assembled cylinder and crucible and if necessary add a few drops of anti-foaming agent (3.2).

Bring the liquid to the boil within 5 ± 2 minutes and boil vigorously for exactly 30 minutes.

Open the tap to the discharge pipe (4.1) and, under vacuum, filter the sulphuric acid through the filter crucible and wash the residue with three consecutive 30 ml portions of boiling water, ensuring that the residue is filtered dry after each washing.

Close the outlet tap and pour 150 ml boiling potassium hydroxide solution (3.7) to the assembled cylinder and crucible and add a few drops of anti-foaming agent (3.2). Bring the liquid to boiling point within 5 ± 2 minutes and boil vigorously for exactly 30 minutes. Filter and repeat the washing procedure used for the sulphuric acid step.

After the final washing and drying, disconnect the crucible and its contents and reconnect it to the cold extraction unit (4.6). Apply the vacuum and wash the residue in the crucible with three consecutive 25 ml portions of acetone (3.4) ensuring that the residue is filtered dry after each washing.

Dry the crucible to constant weight in the oven at 130 °C. After each drying cool in the desiccator and weigh rapidly. Place the crucible in a muffle furnace and ash to constant weight at 475 °C to 500 °C for at least 30 minutes.

After each heating cool first in the furnace and then in the desiccator before weighing.

Carry out a blank test without the sample. Loss of weight resulting from ashing must not exceed 4 mg.

6. Calculation of results

The crude fibre content as a percentage of the sample is given by the expression :

$$\frac{(b-c) \times 100}{a}$$

where

a = mass of sample in g ;

b = loss of mass after ashing during the determination, in g ;

c = loss of mass after ashing during the blank test, in g.

7. Repeatability

The difference between two parallel determinations carried out on the same sample must not exceed :

— 0,3 in absolute value for crude fibre contents lower than 10 %,

— 3 % relative to the higher result, for crude fibre contents equal to or greater than 10 %.

8. Observations

8.1. Feedingstuffs containing more than 10 % crude fat must be defatted prior to analysis with light petroleum (3.5). Connect the filter crucible (4.2) and its contents to the cold extraction unit (4.6) and apply vacuum and wash the residue with three consecutive 30 ml portions of light petroleum, ensuring that the residue is dry. Connect the crucible and its contents to the heating unit (4.1) and continue as described under 5.

8.2. Feedingstuffs containing fats which cannot be extracted directly with light petroleum (3.5) must be defatted as shown in 8.1 and defatted once more after boiling with acid.

After boiling with acid and the subsequent washing connect the crucible and its contents to the cold extraction unit (4.6) and wash three times with 30 ml acetone followed by three further washings with 30 ml portions of light petroleum. Filter under vacuum until dry and continue the analysis as described under 5, beginning with potassium hydroxide treatment.

8.3. If the feedingstuffs contain over 5 % of carbonates, expressed as calcium carbonate, connect the crucible (4.2) with the weighed sample to the heating unit (4.1). Wash the sample three times with 30 ml hydrochloric acid (3.6). After each addition let the sample stand for about one minute before filtering. Wash once with 30 ml water and then continue as described under 5.

8.4. If an apparatus in the form of a stand is used (several crucibles attached to the same heating unit) no two individual determinations on the same sample for analysis may be carried out in the same series.

8.5. If after boiling it is difficult to filter the acidic and basic solutions, use compressed air through the discharge pipe of the heating unit and then continue filtering.

8.6. The temperature for ashing should not be higher than 500 °C in order to extend the lifetime of the glass filter crucibles. Care must be taken to avoid excessive thermal shock during heating and cooling cycles.