Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed (Text with EEA relevance)

Changes to legislation: *There are currently no known outstanding effects for the Commission Regulation (EC) No 152/2009, Division I.. (See end of Document for details)*

ANNEX III

METHODS OF ANALYSIS TO CONTROL THE COMPOSITION OF FEED MATERIALS AND COMPOUND FEED

I.DETERMINATION OF CRUDE FIBRE

1. Purpose and scope

This method makes it possible to determine fat-free organic substances in feed 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 ^{o}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 ^{o}C for four hours (8.6).

3.4. Acetone.

3.5. Light petroleum boiling-range 40 to 60 ^{o}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 and 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 ^{o}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 1 g of the prepared sample to the nearest 1 mg 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 ^{o}C. After each drying cool in the desiccator and weigh rapidly. Place the crucible in a muffle furnace and ash to constant weight (loss in weight between two successive weightings must be less than or equal to 2 mg) at 475 ^{o}C to 500 ^{o}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:

$$X = \frac{(m_0 - m_1) \times 100}{m}$$

where:

m	=	weight of sample in g,
m_0	=	loss of weight after ashing during the determination, in g,
m_1	=	loss of weight 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:

— 0,6 % in absolute value for crude fibre contents lower than 10 %,

— 6 % relative to the higher result, for crude fibre contents equal to or greater than 10 %.

8. Observations

8.1. Feed 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. Feed 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 feed contains 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 shall not be higher than 500 ^{o}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.

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