

COMMISSION DIRECTIVE 1999/79/EC
of 27 July 1999
amending the third Commission Directive 72/199/EEC of 27 April 1972 establishing Community
methods of analysis for the official control of feedingstuffs
 (Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European 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 the Act of Accession of Austria, Finland and Sweden ⁽²⁾, and in particular Article 2 thereof,

(1) Whereas Directive 70/373/EEC stipulates that official controls of feedingstuffs for the purpose of checking compliance with the requirements arising under the laws, regulations and administrative provisions governing their quality and composition must be carried out using Community methods of sampling and analysis;

(2) Whereas the third Commission Directive 72/199/EEC of 27 April 1972 establishing Community methods of analysis for the official control of feedingstuffs ⁽³⁾, as last amended by Commission Directive 98/54/EC ⁽⁴⁾, sets out methods of analysis for, *inter alia*, the determination of starch by polarimetric method;

(3) Whereas Commission Directive 86/174/EEC of 9 April 1986 fixing the method of calculation for the energy value of compound poultryfeed ⁽⁵⁾, stipulates that for the calculation of the energy value of compound poultry feed, in application of Article 10 Council Directive 79/373/EEC of 2 April 1979 on the marketing of compound feedingstuffs ⁽⁶⁾, as last amended by Commission Directive 1999/61/EC ⁽⁷⁾, the starch content must be determined by the polarimetric method as set out in Commission Directive 72/199/EEC;

(4) Whereas Council Directive 96/25/EC of 29 April 1996 on the circulation of feed materials, amending Directives 70/524/EEC, 74/63/EEC, 82/471/EEC and 93/74/EEC

and repealing Directive 77/101/EEC ⁽⁸⁾ as last amended by Directive 1999/61/EC requires the quantities of some analytical constituents, including starch, to be compulsorily declared on some feed materials;

(5) Whereas in the light of the advances of scientific and technological knowledge, the polarimetric method is no longer appropriate to determine the starch content for other purposes than those of the above mentioned Commission and Council Directives; whereas therefore it is appropriate to limit the purpose and scope of the polarimetric method for the determination of starch;

(6) Whereas some feed materials give rise to interferences, whereby the polarimetric method for the determination of starch could yield false results; whereas it is therefore appropriate to mention these feed materials explicitly;

(7) 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 Commission Directive 72/199/EEC is hereby amended in accordance with the Annex to this Directive.

Article 2

Member States shall bring into force, not later than 31 December 1999, the laws, regulations or administrative provisions necessary to comply with the provisions of this Directive. They shall immediately inform the Commission thereof.

They shall apply the measures from 1 January 2000.

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

⁽¹⁾ OJ L 170, 3.8.1970, p. 2.

⁽²⁾ OJ C 241, 29.8.1994, p. 1.

⁽³⁾ OJ L 123, 29.5.1972, p. 6.

⁽⁴⁾ OJ L 208, 24.7.1998, p. 49.

⁽⁵⁾ OJ L 130, 16.5.1986, p. 53.

⁽⁶⁾ OJ L 86, 6.4.1979, p. 30.

⁽⁷⁾ OJ L 162, 26.6.1999, p. 67.

⁽⁸⁾ OJ L 125, 23.5.1996, p. 35.

Article 3

This Directive shall enter into force on the 20th day following its publication in the *Official Journal of the European Communities*.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 27 July 1999.

For the Commission
Franz FISCHLER
Member of the Commission

ANNEX

Section 1 (Determination of starch) is replaced by the following:

1. DETERMINATION OF STARCH

POLARIMETRIC METHOD

1. Purpose and scope

This method makes it possible to determine the levels of starch and of high molecular weight starch degradation products in feedingstuffs for the purpose of checking compliance with Commission Directive 86/174/EEC and Council Directive 96/25/EC.

2. Principle

The method comprises two determinations. In the first, the sample is treated when hot with dilute hydrochloric acid. After clarification and filtration the optical rotation of the solution is measured by polarimetry.

In the second, the sample is extracted with 40 % ethanol. After acidifying the filtrate with hydrochloric acid, clarifying and filtering, the optical rotation is measured as in the first determination.

The difference between the two measurements, multiplied by a known factor, gives the starch content of the sample.

3. Reagents

3.1. 25 % (w/w) hydrochloric acid, $d: 1,126 \text{ g/ml}$.

3.2. 1,128 % (w/v) hydrochloric acid.

The concentration must be checked by titration using a sodium hydroxide solution 0,1 mol/litre in the presence of 0,1 % (w/v) methyl red in 94 % (v/v) ethanol. 10 ml = 30.94 ml of NaOH 0.1 mol/litre.

3.3. Carrez solution I: dissolve 21,9 g of zinc acetate $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ and 3 g of glacial acetic acid in water. Make up to 100 ml with water.

3.4. Carrez solution II: dissolve 10,6 g of potassium ferrocyanide $[\text{K}_4(\text{Fe}(\text{CN})_6)] \cdot 3\text{H}_2\text{O}$ in water. Make up to 100 ml with water.

3.5. 40 % (v/v) ethanol, $d: 0,948 \text{ g/ml}$ at 20 °C.

4. Apparatus

4.1. 250 ml erlenmeyer flask with standard ground-glass joint and with reflux condenser.

4.2. Polarimeter or saccharimeter.

5. Procedure**5.1. Preparation of the sample**

Crush the sample until it is fine enough for all of it to pass through a 0,5 mm round-meshed sieve.

5.2. Determination of the total optical rotation (P or S) (see observation 7.1)

Weigh 2,5 g of the crushed sample to the nearest mg and place in a 100 ml graduated flask. Add 25 ml of hydrochloric acid (3.2), shake to obtain even distribution of the test sample and add a further 25 ml of hydrochloric acid (3.2). Immerse the flask in a boiling water bath shaking vigorously and steadily for the first three minutes to prevent the formation of agglomerates. The quantity of water in the water bath must be sufficient for the bath to remain at boiling point when the flask is introduced into it. The flask must not be taken out of the bath whilst being shaken. After exactly 15 minutes, remove from the bath, add 30 ml of cold water and cool immediately to 20 °C.

Add 5 ml of Carrez solution I (3.3) and shake for one minute. Then add 5 ml of Carrez solution II (3.4) and shake again for one minute. Make up to volume with water, mix and filter. If the filtrate is not perfectly clear (which is rare), repeat the determination using a larger quantity of Carrez solutions I and II, for example 10 ml.

Measure the optical rotation of the solution in a 200 mm tube with the polarimeter or saccharimeter.

5.3. Determination of the optical rotation (P' or S') of substances soluble in 40 % ethanol

Weigh 5 g of the sample to the nearest mg, place in a 100 ml graduated flask and add about 80 ml of ethanol (3.5) (see observation 7.2). Leave the flask to stand for 1 hour at room temperature; during this time, shake vigorously on six occasions so that the test sample is thoroughly mixed with the ethanol. Make up to volume with ethanol (3.5), mix and filter. Pipette 50 ml of the filtrate (= 2,5 g of the sample) into a 250 ml erlenmeyer flask, add 2,1 ml of hydrochloric acid (3.1) and shake vigorously. Fit a reflux condenser to the erlenmeyer flask and immerse the latter in a boiling water bath. After exactly 15 minutes, remove the erlenmeyer flask from the bath, transfer the contents to a 100 ml graduated flask, rinsing with a little cold water, and cool to 20 °C. Clarify using Carrez solutions I (3.3) and II (3.4), make up to volume with water, mix, filter and measure the optical rotation as indicated in the second and third paragraphs of 5.2.

6. Calculation of results

The starch content (%) is calculated as following:

6.1. Measurement by polarimeter

$$\text{Starch content (\%)} = \frac{2000 (P - P')}{[\alpha]_{\text{D}}^{20^\circ}}$$

P = total optical rotation in angle degrees

P' = optical rotation in angle degrees of the substances soluble in 40 % (V/V) ethanol

$[\alpha]_{\text{D}}^{20^\circ}$ = specific optical rotation of pure starch. The numerical values conventionally accepted for factor are the following

- + 185,9°: rice starch
- + 185,4°: potato starch
- + 184,6°: maize starch
- + 182,7°: wheat starch
- + 181,5°: barley starch
- + 181,3°: oat starch
- + 184,0°: other types of starch and starch mixtures in compound feedingstuffs

6.2. Measurement by saccharimeter

$$\text{Starch content (\%)} = \frac{2000}{[\alpha]_{\text{D}}^{20^\circ}} \times \frac{(2N \times 0,665) \times (S - S')}{100} - \frac{26,6 N \times (S - S')}{[\alpha]_{\text{D}}^{20^\circ}}$$

S = total optical rotation in saccharimeter degrees

S' = optical rotation in saccharimeter degrees of the substances soluble in 40 % (V/V) ethanol

N = weight (g) of saccharose in 100 ml of water yielding an optical rotation of 100 saccharimeter degrees when measured using a 200 mm tube

16,29 g for the French saccharimeters

26,00 g for the German saccharimeters

20,00 g for mixed saccharimeters.

$[\alpha]_{\text{D}}^{20^\circ}$ = specific optical rotation of pure starch (see 6.1)

6.3. Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed 0,4 in absolute value for a starch content lower than 40 % and 1,1 % relative for starch contents equal or higher than 40 %

7. Observations

7.1. If the sample contains more than 6 % of carbonates, calculated in terms of calcium carbonate, they must be destroyed by treatment with an exactly appropriate quantity of dilute sulphuric acid before determination of the total optical rotation.

7.2. In the case of products with a high lactose content, such as powdered milk serum or skimmed milk powder, proceed as follows after adding 80 ml of ethanol (3.5). Fit a reflux condenser to the flask and immerse the latter in a water bath at 50 °C for 30 minutes. Leave to cool and continue the analysis as indicated in 5.3.

- 7.3. Following feed materials, in case they are present in significant amount in feedingstuffs, are known to give rise to interferences when determining the starch content by polarimetric method and whereby incorrect results could be yielded:
- (sugar) beet products such as (sugar) beet pulp, (sugar) beet molasses, (sugar) beet pulp-molassed, (sugar) beet vinasse, (beet) sugar,
 - citrus pulp,
 - linseed; linseed expeller; linseed extracted,
 - rape seed; rape seed expeller; rape seed extracted; rape seed hulls,
 - sunflower seed; sunflower seed extracted; sunflower seed, partially decorticated, extracted,
 - copra expeller; copra extracted,
 - potato pulp,
 - dehydrated yeast,
 - products rich in inulin (e.g. chips and meal of Jerusalem artichokes);
 - greaves.
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