# First Commission Directive of 25 October 1985 on methods of analysis for edible caseins and caseinates (85/503/EEC)

## FIRST COMMISSION DIRECTIVE

## of 25 October 1985

## on methods of analysis for edible caseins and caseinates

## (85/503/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 83/417/EEC of 25 July 1983 on the approximation of the laws of the Member States relating to certain lactoproteins (caseins and caseinates) intended for human consumption[(1)], and in particular Article 9 (b) thereof;

Whereas Article 9 (b) of Directive 83/417/EEC requires that Community methods of analysis be determined for checking the composition of certain edible caseins and caseinates;

Whereas it is possible to adopt an initial series of methods in respect of which studies have been completed;

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

HAS ADOPTED THIS DIRECTIVE:

*Article 1*

Member States shall take all measures necessary to ensure that the analyses necessary for verification of the criteria set out in Annex I are carried out in accordance with the methods described in Annex II.

*Article 2*

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive by 1 May 1987 at the latest. They shall forthwith inform the Commission thereof.

*Article 3*

This Directive is addressed to the Member States.

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Done at Brussels, 25 October 1985.

*For the Commission*

COCKFIELD

*Vice-President*

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ANNEX I

SCOPE OF THE FIRST COMMUNITY METHODS OF ANALYSIS DIRECTIVE FOR EDIBLE CASEINS AND CASEINATES

I. **General Provisions**

II. **Determination of moisture in:**
- — acid caseins using method 1, Annex II
- — rennet caseins using method 1, Annex II
- — caseinates using method 1, Annex II

III. **Determination of protein content in:**
- — acid caseins using method 2, Annex II
- — rennet caseins using method 2, Annex II
- — caseinates using method 2, Annex II

IV. **Determination of titratable acidity in:**
- — acid caseins using method 3, Annex II

V. **Determination of ash (including $P_2O_5$) in:**
- — acid caseins using method 4, Annex II
- — rennet caseins using method 5, Annex II

VI. **Determination of pH in:**
- — caseinates using method 6, Annex II

ANNEX II

METHODS OF ANALYSIS RELATING TO THE COMPOSITION OF EDIBLE CASEINS AND CASEINATES

**GENERAL PROVISIONS**

1. PREPARATION OF THE ANALYSIS SAMPLE

1.1. **General**

The mass of the sample presented to the laboratory for analysis shall be at least 200 grams.

1.2. **Preparation of the sample for analysis in the laboratory**

1.2.1. Thoroughly mix and break down any lumps, etc., in the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity (twice Volume of sample) to allow this operation to be carried out).

1.2.2. Transfer a representative portion of the sample, i.e. about 50 grams of the thoroughly mixed laboratory sample (1.2.1) to the test sieve (3.3).

1.2.3. If the 50 gram portion completely or almost completely passes (at least 95 % by weight) through the sieve (3.3), use for the determination the sample as prepared in 1.2.1.

1.2.4. Otherwise, grind the 50 gram portion, using the grinding device (3.4), until it satisfies the sieving criterion (1.2.3). Immediately transfer all the sieved sample to an air-tight container

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of sufficient capacity (twice volume of sample) and mix thoroughly by repeated shaking and inverting. During these operations, take precautions to avoid any change in the moisture content of the product.

1.2.5. After the test sample has been prepared, any determination should be proceeded with as soon as possible.

1.3. **Containers**

The sample shall always be kept in an air-tight and moisture-tight container.

2. REAGENTS

2.1. **Water**

2.1.1. Wherever mention is made to water for solution, dilution or washing purposes, distilled water, or de-mineralized water of at least equivalent purity, shall be used.

2.1.2. Wherever reference is made to ‘solution’ or ‘dilution’ without further indication, ‘solution in water’ or ‘dilution with water’ is meant.

2.2. **Chemicals**

All chemicals used shall be of recognized analytical reagent quality except where otherwise specified.

3. EQUIPMENT

3.1. **Lists of equipment**

The lists of equipment contain only those items with a specialized use and items to a particular specification.

3.2. **Analytical balance**

Analytical balance means a balance capable of weighing to at least 0,1 mg.

3.3. **Test sieve**

The test ieves to be used are to be fitted with a lid, to be of diameter 200 mm, to be constructed of wire cloth with a nominal aperture size of 500 urn. The aperture tolerances and wire diameters to be permitted are as given in ISO 3310/1. (Test sieves — Technical requirements and testing — Part 1: Metal wire cloth. ISO 3310/1 — 1975).

The sieves are to be fitted with a receiver.

3.4. **Grinding device**

For grinding the laboratory sample if necessary (see 1.2.4), without development of undue heat and without loss or absorption of moisture, a hammer mill shall not be used.

4. EXPRESSION OF RESULTS

4.1. **Results**

The result stated in the analytical report is to be the mean value obtained from two determinations which satisfy the repeatability criterion for that method.

4.2. **Calculation of percentage**

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Except where otherwise specified, the result shall be calculated as a percentage by mass of the sample.

5. TEST REPORT

The test report shall identify the method of analysis used as well as the results obtained. In addition, it shall mention all details of procedure not specified in the method of analysis or which are optional, as well as any circumstances that may have influenced the results obtained. The test report shall give all the information necessary for the complete identification of the sample.

METHOD 1

**DETERMINATION OF MOISTURE CONTENT**

1. SCOPE AND FIELD OF APPLICATION

This method determines the moisture content in:

— acid caseins
— rennet caseins
— caseinates

2. DEFINITION

The moisture content of caseins and caseinates: the loss of mass as determined by the method specified.

3. PRINCIPLE

The residual mass of a test portion is determined after drying at atmospheric pressure in an oven at 102 $^{o}$C ± 1 $^{o}$C to constant mass. The loss of mass is calculated as a percentage by mass of the sample.

4. APPARATUS

4.1. **Analytical balance**

4.2. **Dishes,** flat-bottomed and of material non-corrodible under the conditions of test e.g. nickel, aluminium, stainless steel or glass. The dishes must have lids which fit tightly but which can readily be removed. Suitable dimensions are: diameter 60 to 80 mm and depth about 25 mm.

4.3. **Atmospheric pressure drying oven,** well ventilated, thermostatically controlled with temperature regulation (at 102 $^{o}$C ± 1 $^{o}$C). The temperature should be uniform throughout the oven.

4.4. **Desiccator,** containing freshly activated silica gel with a water content indicator or an equivalent desiccant.

4.5. **Suitable device for handling dishes,** e.g. laboratory tongs.

5. PROCEDURE

5.1. **Preparation of the test sample**

As described in Section 1.2 of the General Provisions.

5.2. **Preparation of the dish**

5.2.1. Heat the uncovered dish and its lid (4.2) in the oven (4.3), controlled at 102 $^{o}$C ± 1 $^{o}$C, for at least one hour.