
International Standard



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Caseins and caseinates — Determination of scorched particles content

Caséines et caséinates — Détermination de la teneur en particules brûlées

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5739 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in November 1982.

It has been approved by the member bodies of the following countries:

Australia	Germany, F. R.	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Sri Lanka
Canada	Iraq	Turkey
China	Ireland	United Kingdom
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Czechoslovakia	Kenya	Venezuela
Egypt, Arab Rep. of	Netherlands	Yugoslavia
Ethiopia	New Zealand	
France	Philippines	

No member body expressed disapproval of the document.

NOTE — The method specified in this International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, USA). The text as approved by the above organizations will also be published by FAO/WHO (Code of principles concerning milk and milk products and associated standards), by the IDF and by the AOAC (Official Methods of Analysis).

The method is derived from procedures developed by the United States Department of Agriculture (USDA) for the determination of scorched particles in dried milk.

Caseins and caseinates — Determination of scorched particles content

1 Scope and field of application

This International Standard specifies a method for the determination of the scorched particles content of caseins and caseinates.

2 References

ISO 707, *Milk and milk products — Methods of sampling*.¹⁾

ISO 3310/1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

3 Definition

scorched particles content of caseins and caseinates: The amount of coloured residue, per 25 g or 10 g of sample, which is insoluble in a sodium carbonate, sodium polyphosphate, disodium ethylenediaminetetraacetate or sodium hydroxide solution, as determined and classified by the procedure specified in this International Standard.

4 Principle

Dissolution of a test portion in hot sodium carbonate, sodium polyphosphate or disodium ethylenediaminetetraacetate solution, or in sodium hydroxide solution at room temperature or at 60 °C, filtration of the solution through a filtering disk and visual comparison of the dried disk with scorched particle standard disks.

5 Reagents

The reagents shall be of recognized analytical quality.

The water used in the preparation of the reagent solutions and in the determination shall be distilled water or water of at least equivalent purity, filtered before use.

5.1 Sodium carbonate solution (for acid caseins and for caseinates).

Dissolve 100 g of anhydrous sodium carbonate (Na_2CO_3) in water, dilute to 1 000 ml and filter.

5.2 Sodium polyphosphate solution (for rennet caseins).

Dissolve 20 g of a sodium polyphosphate [tripolyphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$) or a higher polyphosphate as used in the manufacture of processed cheese] in water, dilute to 1 000 ml and filter.

5.3 Disodium ethylenediaminetetraacetate solution (for calcium caseinates).

Dissolve 100 g of EDTA, disodium salt, dihydrate ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$) in water, dilute to 1 000 ml and filter.

5.4 Sodium hydroxide, approximately 1 mol/l solution (for caseins and caseinates which do not dissolve completely in the other reagents).

Dissolve 40 g of sodium hydroxide in water, dilute to 1 000 ml and filter.

5.5 Ethanol, 95 ± 2 % (V/V) (for caseinates).

6 Apparatus

Usual laboratory equipment, and

6.1 Balance, accurate to 0,1 g.

6.2 Conical flask, of capacity 600 ml.

6.3 Measuring cylinders, of capacities 100 and 500 ml.

6.4 Water bath, capable of being controlled at 60 ± 1 °C.

1) At present at the stage of draft. (Revision of ISO/R 707-1968.)

6.5 Filtering disks, of cotton lintine, diameter 32 mm (1.25 in), suitable for use in the filtering device (6.6). The cotton lintine shall be of approximately 135 g/m² (4 oz/yd²) No. 1 filter cotton fibres. The disks shall be tested by the method specified in annex A and shall meet the requirements specified therein.

6.6 Filtering device, aspirator or pressure type, with a filtering area of diameter 28,6 mm (1.125 in).

6.7 Grinding device, for grinding the laboratory sample if necessary (see 8.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

6.8 Test sieve, of metal wire cloth, diameter 200 mm and nominal aperture size 500 µm with receiver, complying with the requirements of ISO 3310/1.

6.9 Scorched particle standard disks, indicating increasing scorched particles contents by the classification letters A, B, C and D respectively.

NOTES

- 1 Prints of these standard disks are available from the United States Department of Agriculture, Agriculture Marketing Service, Dairy Division, Washington, D.C. 20250, USA.
For illustrative purposes only, a black-and-white reproduction of these standard disks is given below. **These illustrations shall not be used for official classification of scorched particle contents.**
- 2 Instructions for the preparation of these standard disks, if required, are given in annex B.

7 Sampling

See ISO 707.

8 Procedure

8.1 Preparation of the test sample

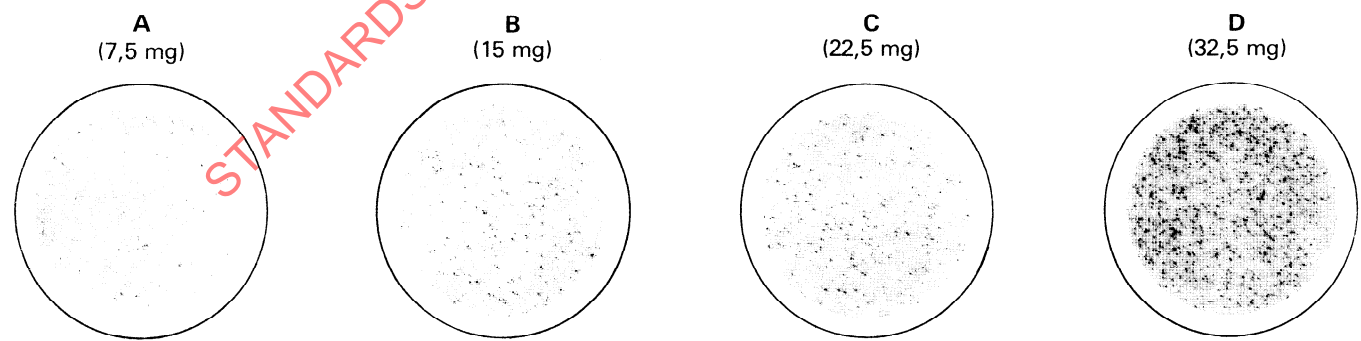
8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an airtight container of sufficient capacity to allow this operation to be carried out).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.8)

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1. Otherwise, grind the 50 g portion, using the grinding device (6.7), until it passes the sieve. Immediately transfer all the sieved sample to an airtight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.4 After the test sample has been prepared, proceed with the determination as soon as possible.

Scorched particle standard disks



8.2 Test portion

8.2.1 Spray-dried caseinates or edible acid caseins

Weigh $25,0 \pm 0,1$ g of the test sample (8.1) and transfer to the flask (6.2).

8.2.2 Roller-dried caseinates, rennet caseins or industrial acid caseins

Weigh $10,0 \pm 0,1$ g of the test sample (8.1) and transfer to the flask (6.2).

8.3 Determination

8.3.1 Addition of reagent

8.3.1.1 Acid caseins

Add to the 25 g test portion of edible casein or to the 10 g test portion of industrial acid casein, as the case may be, 300 ml or 150 ml, respectively, of filtered water. Allow to stand for at least 10 min, swirling at frequent intervals until all the casein is dispersed. Then add, respectively, 30 ml or 15 ml of the sodium carbonate solution (5.1), and swirl. Take care to wash down any of the casein adhering to the walls of the flask.

8.3.1.2 Rennet caseins

Add to the test portion 100 ml of filtered water, shake and allow to stand for 3 h. Then add 200 ml of the sodium polyphosphate solution (5.2). Take care to wash down any of the casein adhering to the walls of the flask.

8.3.1.3 Caseinates

Add to the 25 g test portion of spray-dried soluble caseinate or to the 10 g test portion of roller-dried soluble caseinate, as the case may be, 50 ml or 20 ml, respectively, of the ethanol (5.6) and shake until all the caseinate has been moistened. If necessary, add more ethanol. Then add 300 ml or 150 ml, respectively, of filtered water. Allow to stand for at least 10 min, swirling at frequent intervals until all the caseinate is dispersed. Then add 30 ml or 15 ml, respectively, of the sodium carbonate solution (5.1), and swirl. Take care to wash down any of the caseinate adhering to the walls of the flask.

In the case of calcium caseinates or other insoluble caseinates, add, before the addition of the sodium carbonate solution, 30 ml or 15 ml of the disodium ethylenediaminetetraacetate solution (5.3). Allow to stand for at least 10 min, swirling at frequent intervals until all the caseinate is dispersed. Take care to wash down any of the caseinate adhering to the walls of the flask. Then add the 30 ml or 15 ml of the sodium carbonate solution (5.1).

8.3.2 Heating and filtration

8.3.2.1 Swirl the flask, cover, and heat in the water bath (6.4), controlled at 60 °C, swirling the flask at intervals, until all

the test portion is dissolved; the time required shall not exceed 45 min.

8.3.2.2 Swirl the flask once more and then filter the contents through one of the filtering disks (6.5) mounted in the filtering device (6.6). Rinse the flask with two successive 100 ml portions of filtered water at 60 °C, and pass the rinsings through the filtering disk in such a way that the rinsings are allowed to run down the walls of the filtering device and the filtering disk is thoroughly washed.

8.3.2.3 Remove the filtering disk and allow it to dry, or dry it at 30 to 40 °C, protected from dust.

8.3.2.4 If difficulties are encountered in passing the solution through a disk, or if a significant quantity of gelatinous material appears on the disk, repeat the determination using the procedure specified in 8.3.3 and 8.3.4, using the sodium hydroxide solution (5.4) instead of the sodium carbonate solution (5.1) or the disodium ethylenediaminetetraacetate solution (5.3).

8.3.3 Alternative addition of reagent (when the casein or caseinate dissolves unsatisfactorily according to 8.3.1 and 8.3.2)

8.3.3.1 Acid caseins

Proceed as specified in 8.3.1.1, but, instead of adding 30 ml or 15 ml of the sodium carbonate solution (5.1), add, respectively, 30 ml or 15 ml of the sodium hydroxide solution (5.4).

8.3.3.2 Rennet caseins

Add to the 10 g test portion of rennet casein, 150 ml of filtered water, swirl and allow to stand for at least 10 min. Add 15 ml of filtered sodium hydroxide solution (5.4) and swirl.

8.3.3.3 Caseinates

Proceed as specified in 8.3.1.3, but instead of adding 30 ml or 15 ml of the sodium carbonate solution (5.1), add, respectively, 30 ml or 15 ml of the sodium hydroxide solution (5.4).

8.3.4 Heating and filtration when sodium hydroxide solution is added (alternative to 8.3.2)

8.3.4.1 Cover the flask and allow to stand at room temperature, swirling at frequent intervals, until all the test portion appears to be dissolved. Proceed as specified in 8.3.2.2 and 8.3.2.3.

8.3.4.2 If the test portion is not completely dissolved, or if filtering is difficult, heat the covered flask in the water bath (6.4), controlled at 60 °C, for 20 min, swirling the flask at intervals, until all the test portion is dissolved. Proceed as specified in 8.3.4.1.

9 Expression of results

9.1 Evaluation

Compare the test disk (8.3.2) with the scorched particles standard disks (6.9), viewing from directly above in uniform indirect light, and assign the appropriate classification letter to the test disk.

A test disk falling between two standard disks shall be assigned the classification letter corresponding to the higher scorched particles content.

Examine the disk for hair, wood, metal, dust or insect fragments. If there are indications of their presence, determine the extraneous matter content of the sample.¹⁾

9.2 Repeatability

If the test method is carried out in duplicate, the duplicate test disks shall indicate the same classification. Otherwise, repeat the determination in duplicate.

10 Test report

The test report shall show the method used and the result obtained and shall record any extraneous matter observed. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all the details required for the complete identification of the sample.

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1) A method for the determination of extraneous matter content will form the subject of a future International Standard.

Annex A

Testing of filtering disks

(derived from *Standard Methods for the Examination of Dairy Products*, 14th Edition 1978 and 11th Edition 1960, American Public Health Association)

A.1 Materials

A.1.1 Wetting agent, 1 % solution.

Use an aerosol solution or any other suitable wetting agent.

A.1.2 Gum solution.

Add 0,75 g of carob bean or other suitable gum to 100 ml of water, while stirring in a blender.

Remove air bubbles by treating the solution under vacuum or by heating. Boil, cool, and then add 2 ml of 40 % formaldehyde solution (this causes separation of insoluble vegetable fragments and permits the use of clear supernatant solution).

To facilitate dispersion in water in the absence of a blender, suspend 0,75 g of carob bean in a few millilitres of ethanol in a 100 ml volumetric flask. Dilute to the mark with water and mix thoroughly. Then proceed as described above.

A.1.3 Sucrose solution.

Dissolve 750 g of sucrose in 750 ml of water.

A.1.4 Fine sediment mixture, 0,2 g/l dispersion.

Prepare the mixture from oven dried (at 100 °C) ground cow manure, garden soil and charcoal. Sieve the materials separately and collect the fractions passing through a sieve of aperture size 106 µm [diameter 203,2 mm (8 in)] and retained on a sieve of aperture size 75 µm, proceeding as follows:

Place not more than 100 g of cow manure or soil, and not more than 50 g of charcoal, on the sieve of aperture size 106 µm nested over the sieve of aperture size 75 µm. Cover, and position the receiver. Shake the nest of sieves by hand, at a rate of about 120 strokes per minute, for 5 min. In maximum batches of about 20 g, resieve the fractions retained on the 75 µm sieve as above for 5 min. Use the fractions retained from this second sieving operation and mix them uniformly in the following proportions:

— cow manure	66 %
— garden soil	28 %
— charcoal	6 %

Place 2 g of this mixture in a 100 ml one-mark volumetric flask and moisten with 5 ml of the wetting agent (A.1.1). Add 46 ml of gum solution (A.1.2), bring the liquid level just into the neck

of the flask by adding the sucrose solution (A.1.3), and allow to stand for at least 30 min. Add a few drops of ethanol, dilute to the mark with the sucrose solution, and mix thoroughly.

Pour the solution into a 250 ml beaker or screw-cap bottle and, allowing minimal incorporation of air, stir with a small mechanical stirrer at a frequency of rotation of 200 to 300 min⁻¹ until the sediment is uniformly distributed as observed under bright reflected light. Position the blade of the stirrer so that fine particles do not accumulate in small eddies at the bottom of the beaker. While stirring, transfer a 10 ml portion (corresponding to 200 mg of fine sediment mixture), by means of a graduated pipette (outlet of diameter 3 mm), to a volumetric flask and make up to 1 000 ml with water.

A.2 Apparatus

Usual laboratory equipment, and

A.2.1 Balance, accurate to 0,000 1 g.

A.2.2 Desiccator, provided with an efficient desiccant.

A.2.3 Oven, capable of being controlled at 100 °C.

A.2.4 Filtering device (see 6.6).

A.2.5 Filtering disks (see (6.5)).

A.2.6 Filter papers, of diameter 7 or 9 cm, high quality and medium grade.

A.3 Procedure

A.3.1 Wash a filter paper (A.2.6), placed in a Buchner funnel, with about 200 ml of water, dry it to constant mass at 100 °C, cool in a covered dish in a desiccator and weigh.

A.3.2 Filter 60 ml of the fine sediment mixture (A.1.4), corresponding to 12 mg of sediment, after thoroughly mixing and stirring, through a filtering disk (A.2.5) mounted in the filtering device (A.2.4), using a clean flask to collect the filtrate. Transfer the filtrate to a beaker. Rinse the flask three times with water and add the rinsings to the contents of the beaker.

A.3.3 Refilter the filtrate through the washed, dried and weighed filter paper (see A.3.1) placed in a Buchner funnel.

Rinse the beaker and paper thoroughly with water. Then dry the paper to constant mass at 100 °C (see A.3.1).

A.3.4 Test at least two more disks.

A.4 Evaluation

The average mass of sediment per disk passing through three or more disks should not exceed 2,8 mg.

A standard disk prepared from a fine sediment mixture should not appear to have sediment buried beneath the surface.

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Annex B

Preparation of scorched particle standard disks

(derived from the US Code of Federal Regulations, 7 CFR 58.2676)

B.1 Materials

B.1.1 Sucrose solution.

Dissolve 750 g of sucrose in 750 ml of water.

NOTE — Filter before use.

B.1.2 Charcoal mixture, 1 g/l dispersion.

Prepare a charcoal mixture having the following composition (at least 1 g is required):

20 % (m/m)	200 mesh charcoal;
50 % (m/m)	150 mesh charcoal;
20 % (m/m)	100 mesh charcoal;
10 % (m/m)	65 mesh charcoal;

Transfer 1 g of this mixture to a 1 000 ml one-mark volumetric flask, dilute to the mark with the sucrose solution (B.1.1) and mix thoroughly.

B.1.3 Spray-process, non-fat dry milk solids.

B.1.4 Reconstituted, non-fat dry milk solids.

Dissolve 50 g of non-fat dry milk solids (B.1.3) in 500 ml of water.

NOTE — Filter before use.

B.2 Apparatus

Usual laboratory equipment, and

B.2.1 Balance, accurate to 0,000 1 g.

B.2.2 Petri dish.

B.2.3 Desiccator, provided with an efficient desiccant.

B.2.4 Volumetric flasks, of capacities 200 and 1 000 ml.

B.2.5 Oven, controlled at 119 °C.

B.2.6 Filtering device (see 6.6).

B.2.7 Filtering disks (see 6.5).

B.3 Procedure

B.3.1 Scorched particles

Evenly spread 5 g of the non-fat dry milk solids (B.1.3) in the Petri dish (B.2.2) and heat in the oven (B.2.5), controlled at 119 °C, for 4 h to produce scorched particles. Allow to cool in the desiccator (B.2.3).

B.3.2 Scorched particles dispersion

Mix 0,5 g of the scorched particles gently with approximately 20 ml of the sucrose solution (B.1.1). Transfer the mixture to the 200 ml volumetric flask (B.2.4) and dilute to the mark with the same sucrose solution.

B.3.3 Standard particle dispersions

Prepare standard particle dispersions as follows:

a) dispersion I

Mix:

- 75 ml of the reconstituted milk solids (B.1.4);
- 3,0 ml of the scorched particle dispersion (B.3.2);
- 0,075 ml of the charcoal dispersion (B.1.2).

The particle content of this dispersion is 7,5 mg.

b) dispersion II

Mix:

- 75 ml of the reconstituted milk solids (B.1.4);
- 6,0 ml of the scorched particle dispersion (B.3.2);
- 0,15 ml of the charcoal dispersion (B.1.2).

The particle content of this dispersion is 15,0 mg.

c) dispersion III

Mix:

- 75 ml of the reconstituted milk solids (B.1.4);
- 9,0 ml of the scorched particle dispersion (B.3.2);
- 0,3 ml of the charcoal dispersion (B.1.2).

The particle content of this dispersion is 22,5 mg.