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British Standard Methods for

Chemical analysis of cheese

Part 6. Determination of total phosphorus content. Molecular absorption spectrometric method

[ISO title: Cheese and processed cheese products — Determination of total phosphorus content — Molecular absorption spectrometric method]

Méthodes d'analyse chimique du fromage

Partie 6. Dosage du phosphore total. Méthode par spectrométrie d'absorption moléculaire

Verfahren für die chemische Analyse von Käse

Teil 6. Bestimmung des Gesamtposphorgehaltes. Molekularabsorptionsspektrometrieverfahren

NOTE. This Part should be read in conjunction with Part 1 'General introduction', published separately.

National foreword

This revision of Part 6 of BS 770 has been prepared under the direction of the Dairying Standards Committee. It supersedes BS 770 : Part 6 : 1976, which is withdrawn.

This Part of BS 770 is identical with ISO 2962-1984 'Cheese and processed cheese products — Determination of total phosphorus content — Molecular absorption spectrometric method' in technical content and in presentation.

The method described in the international standard was developed jointly with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists, USA (AOAC).

The method differs from that described in BS 770 : Part 6 : 1976 in two important respects.

- (a) The spectrometric measurement of the phosphomolybdate complex is carried out at a wavelength of 820 nm instead of 700 nm.
- (b) Ascorbic acid is used as the reducing agent in place of hydrazine sulphate.

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Where the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-reference. There is no corresponding British Standard to ISO/R 707 but with reference to clause 7, the method for sampling given in clause 7 of BS 809 : 1974, which is related to ISO/R 707, may be employed. It is expected that the revision of ISO/R 707 will be published, when available, as an identical revision of BS 809.

Additional information

Preparation of the test sample. The procedure for preparation of the test sample described in 8.1 is technically equivalent to the general procedure described in 4.1 and 4.2 of BS 770 : Part 1 : 1976.

Test report. The requirement of clause 9 means that the test report includes the same items as those listed in clause 6 of BS 770 : Part 1 : 1976.

NOTE. *Typographical error.* In 4.3.3, line 1, '5.3.1' should read '4.3.1'.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

1 Scope and field of application

This International Standard specifies a molecular absorption spectrometric method for the determination of the total phosphorus content of cheese. The method is applicable to all kinds of cheese and to processed cheese products.

2 Reference

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

3 Principle

Digestion of the cheese by sulfuric acid and hydrogen peroxide.

Formation of molybdenum blue by addition of molybdate-ascorbic acid solution. Molecular absorption spectrometric measurement, at a wavelength of 820 nm, of the blue colour formed.

NOTE — Dry ashing may be used, provided that the procedure gives the same results as the wet digestion.

4 Reagents

All reagents shall be of recognized analytical grade. The water used shall be distilled or deionized water, free from phosphorus compounds.

4.1 Concentrated sulfuric acid (H_2SO_4), $\rho_{20} = 1,84$ g/ml.

4.2 Hydrogen peroxide, solution containing about 30 g of H_2O_2 per 100 ml.

4.3 Molybdate-ascorbic acid solution.

4.3.1 Sodium molybdate solution.

Dissolve 12,5 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 5 mol/l sulfuric acid solution, dilute with this sulfuric acid solution to 500 ml, and mix.

4.3.2 Ascorbic acid, solution.

Dissolve 10 g of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in water, dilute to 200 ml, and mix.

NOTE — This solution cannot be stored.

4.3.3 Mixed solution.

Immediately before use, mix 25 ml of solution 5.3.1 with 10 ml of solution 4.3.2, dilute to 100 ml with water, and mix.

4.4 Phosphorus, standard solution corresponding to 100 μg of P per ml.

Dry for at least 48 h about 1 g of potassium dihydrogenorthophosphate (KH_2PO_4) in a desiccator over an efficient desiccant, for example concentrated sulfuric acid.

Dissolve 0,439 4 g of the previously dried phosphate in water, dilute to 1 000 ml, and mix.

5 Apparatus

All glassware shall be thoroughly cleaned with a phosphorus-free detergent and rinsed with distilled water.

5.1 Analytical balance.

5.2 Device for grinding or grating cheese, capable of being easily cleaned.

5.3 Boiling water-bath.

5.4 Digestion flasks (Kjeldahl flasks or digestion tubes), of capacity 25 ml.

5.5 Heating apparatus.

5.5.1 Micro gas burners, for heating Kjeldahl flasks.

NOTE — Electric heaters may also be used.

5.5.2 Heating block, for heating digestion tubes.

5.6 Glass beads.

5.7 Graduated cylinders, of capacities 5 and 25 ml.

5.8 One-mark volumetric flasks, of capacities 50 and 100 ml.

5.9 One-mark pipettes, to deliver 1, 2, 3, 5 and 10 ml.

5.10 Spectrometer, suitable for measurements at a wavelength of 820 nm, equipped with cells of optical path length 10 mm.

6 Sampling

6.1 See ISO 707.

6.2 Store the sample in such a way that deterioration and change in composition are prevented.

7 Procedure

7.1 Preparation of the test sample

Remove the rind, smear or mouldy surface layer of cheese, in such a way as to provide a sample representative of the cheese as it is usually consumed. Grind or grate the sample by means of an appropriate device (5.2). Mix the ground or grated mass quickly, and if possible grind or grate a second time and again mix thoroughly. If the sample cannot be ground or grated, mix it thoroughly by intensive stirring and kneading.

Transfer the test sample to an airtight container to await analysis, which should be carried out as soon as possible after grinding or grating. If delay is unavoidable, take all precautions to ensure proper preservation of the sample and to prevent condensation of moisture on the inside surface of the container.

Clean the device after grinding or grating the sample.