SLOVENSKI STANDARD
SIST EN ISO 5378:1998

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Škrobi in škrobni derivati - Določevanje dušika po Kjeldahlovi metodi - Spektrofotometrijska metoda (ISO 5378:1978)

Starches and derived products - Determination of nitrogen content by the Kjeldahl method - Spectrophotometric method (ISO 5378:1978)

Stärke und Stärkederivate - Bestimmung des Stickstoffgehalts nach dem Kjeldahl-Verfahren - Spektrometrisches Verfahren (ISO 5378:1978)

Amidons, fécules et produits dérivés - Dosage de l'azote selon la méthode de Kjeldahl - Méthode spectrophotométrique (ISO 5378:1978)

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English version

Starches and derived products - Determination of nitrogen content by the Kjeldahl method - Spectrophotometric method (ISO 5378:1978)

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This European Standard was approved by CEN on 1994-08-22. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard ISO 5378:1978, prepared by ISO/TC 93 "Starch", was submitted to the formal vote and was approved by CEN as EN ISO 5378:1994 on 1994-08-22 without any modifications.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 1995, and conflicting national standards shall be withdrawn at the latest by February 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Endorsement notice

The text of the International Standard ISO 5378:1978 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD 5378

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Starches and derived products — Determination of nitrogen content by the Kjeldahl method — Spectrophotometric method

Amidons, fécules et produits dérivés — Dosage de l'azote selon la méthode de Kjeldahl — Méthode spectrophotométrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 5378 was developed by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, and was circulated to the member bodies in March 1976.

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

Australia	Germany	Romania
Austria	Hungary	Spain
Chile	Ireland	Turkey
Czechoslovakia	Mexico	United Kingdom
Finland	Netherlands	Yugoslavia
France	Philippines	

The member body of the following country expressed disapproval of the document on technical grounds :

Poland

Printed in Switzerland

Starches and derived products — Determination of nitrogen content by the Kjeldahl method — Spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination, by the Kjeldahl method, of the nitrogen content of starch and its derived products whose presumed nitrogen content is less than 0,025 % (*m/m*).[1)]

NOTE — In starches and their derived products to which nitrogenous materials have not been added, the nitrogen is present essentially in the form of protein and/or amino acids.

2 REFERENCES

ISO 1227/Add. 2, *Starch, including derivatives and by-products — Vocabulary, Addend.*

ISO 1871, *Agricultural food products — General directions for the determination of nitrogen by the Kjeldahl method.*

ISO 3188, *Starches and derived products — Determination of nitrogen content by the Kjeldahl method — Titrimetric method.*

3 DEFINITION

nitrogen content : The value found using the procedure specified. It includes the nitrogen content of free amino acids, of compounds producing amino acids on hydrolysis and of ammonium compounds. It does not include the nitrogen of nitrate and nitrite radicals, the nitrogen attached directly to another nitrogen atom or the nitrogen attached to an oxygen atom.

4 PRINCIPLE

Destruction of organic matter by sulphuric acid in the presence of a compound catalyst[2)], alkalization of the reaction products, distillation of the liberated ammonia and collection in a sulphuric acid solution, followed by spectrophotometry, of the ammonium salt formed after the addition of the Nessler reagent.

5 REAGENTS

The reagents shall be of recognized analytical quality. Ammonia-free distilled water or water of at least equivalent purity shall be used.

5.1 Sulphuric acid, concentrated, ρ_{20} 1,84 g/ml [96 % (*m/m*)].

5.2 Sodium hydroxide, solution 30 % (*m/m*), ρ_{20} 1,33 g/ml.

NOTE — This solution may be more concentrated.

5.3 Compound catalyst[3)], consisting of, for example

— potassium sulphate : 97 g;

— copper(II) sulphate, anhydrous : 3 g.

5.4 Ammonium sulphate, ammonium oxalate or **ammonium chloride.**

5.5 Sulphuric acid, approximately 0,1 N standard volumetric solution.

5.6 Nessler reagent, prepared as follows at least 2 days before use :

Dissolve 100 g of mercury(II) iodide and 70 g of potassium iodide in 100 ml of water. Dissolve 224 g of potassium hydroxide in 700 ml of water in a 1 000 ml one-mark volumetric flask and allow to cool to ambient temperature. Add the mercury(II) iodide/potassium iodide solution slowly, with stirring, to the potassium hydroxide solution. Dilute to the mark with water and mix. Allow to stand for at least 2 days before using.

NOTE — The reagent should be kept in a brown glass bottle. If it is kept in an air-tight bottle and in the shade, it can be used even after 1 year if care is taken to leave the sediments undisturbed when it is re-used.

1) For products whose presumed nitrogen content is greater than 0,01 % (*m/m*), see ISO 3188.

2) See ISO 1871.

3) See ISO 1871, sub-clause 5.2.

6 APPARATUS

Usual laboratory equipment and, in particular :

6.1 Kjeldahl flask, of suitable capacity, usually 500 to 800 ml, preferably with a ground glass joint, and provided with a pear-shaped glass bulb fitting loosely in the top of the neck of the flask.

6.2 Digestion stand, on which the Kjeldahl flask (6.1) can be heated in an inclined position in such a way that heat is applied only to that part of the flask wall which is below the liquid level at all stages.

6.3 Distillation or steam distillation apparatus, with a 200 ml graduated dropping funnel and an efficient splash head, the latter connecting the Kjeldahl flask (6.1) to the condenser.

Any apparatus that satisfies the control tests given in ISO 1871 is permitted.

6.4 Conical flasks, capacity 100 ml.

6.5 One-mark volumetric flask, capacity 200 ml, with a plain neck, complying with the requirements of ISO 1042, class A.

6.6 Pipettes, of suitable capacity, complying with the requirements of ISO 648, class A.

6.7 Spectrophotometer, capable of being adjusted at a wavelength of 430 nm and provided with appropriate cells whose optical path length shall be stated in the test report.

6.8 Mechanical grinder or **mortar.**

6.9 Sieve, with a nominal mesh opening of 0,6 mm, complying with the requirements of ISO 565.

6.10 Analytical balance.

7 PROCEDURE

7.1 Preparation of the test sample

Mix the sample thoroughly and rapidly by shaking or stirring with a spatula in the sample container[1]. If the sample container is too small for this purpose, transfer the entire sample to another predried container of a suitable size to facilitate mixing.

It may be necessary to grind the sample, in which case it must pass through the sieve (6.9) without leaving any residue.

7.2 Test portion

Weigh, to the nearest 0,001 g, 2 to 5 g (mass m_0) of the test sample (7.1), according to the presumed nitrogen content, and transfer to the predried Kjeldahl flask (6.1), taking care that none of the product adheres to the inner wall of the neck of the flask.

In the case of a viscous liquid or a product in paste form, the test portion may be weighed in a small glass container or on a sheet of aluminium, paper or plastics which does not yield nitrogen, or whose nitrogen content is known, and which is left in the flask. In the case of a container which yields nitrogen, this should be taken into account in the blank test (7.8).

7.3 Destruction of organic matter

Add 3 g of the compound catalyst (5.3) and, using a suitable measuring cylinder, add the appropriate volume, in millilitres, of the concentrated sulphuric acid (5.1), calculated by the formula $20 + 4\,m_0$, in such a way that the acid rinses the inner wall of the neck of the flask.

Mix the contents of the flask by swirling the flask gently until the mixture is free from lumps and the test portion is completely wetted. In order to avoid super-heating, add a boiling aid (for example glass beads). Insert the pear-shaped glass bulb (see 6.1) in the neck of the flask and place it in an inclined position on the digestion stand (6.2).

Heat with care until the liquid in the flask boils gently. Continue to heat for 1 h after the liquid becomes clear. In the case of digestion apparatus heated by gas, ensure that the flame does not extend beyond the part of the flask filled with liquid, in order to avoid loss of nitrogen.

7.4 Distillation

Allow the contents of the flask to cool and rinse the pear-shaped glass bulb and the inner neck of the flask with a few millilitres of water, allowing the rinsings to run into the flask. Add, with care, between 50 and 150 ml of water (according to the apparatus used), whilst swirling the contents of the flask. Connect the flask to the distillation or steam distillation apparatus (6.3), previously freed from ammonia by steaming.

Adjust the lower end of the condenser so that it just touches the bottom of the one-mark volumetric flask (6.5), containing 25 ml of the sulphuric acid solution (5.5). Render the digestion liquid alkaline by slowly adding, through a graduated separating funnel (see 6.3) placed in the neck of the flask, 120 ml of the sodium hydroxide solution (5.2), ensuring that the neck of the funnel does not become empty. Mix well, then turn on the condenser water and start heating; the ammonia then begins to be carried over.

1) In the case of glucose syrup, remove the surface layer (about 5 mm) before mixing.