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Kmetijski pridelki in živilski proizvodi - Splošna navodila za določanje dušika s Kjeldahlovo metodo

Food and feed products -- General guidelines for the determination of nitrogen by the Kjeldahl method

Produits alimentaires et aliments des animaux -- Lignes directrices générales pour le dosage de l'azote selon la méthode de Kjeldahl

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INTERNATIONAL STANDARD

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Food and feed products — General guidelines for the determination of nitrogen by the Kjeldahl method

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1871 was prepared by Technical Committee ISO/TC 34, *Food products*.

This second edition cancels and replaces the first edition (ISO 1871:1975), which has been technically revised.

Introduction

The analysis of products of animal or plant origin, such as those used in food and feed products, often includes determining their nitrogen content according to the Kjeldahl method.

This method can be standardized in principle, as it is generally accepted that different apparatus or operating methods are equivalent if their results are similar.

The purpose of this document is to describe the various stages of the method, the associated critical points and the minimum objectives to be achieved to ensure that the method is applied correctly.

This document provides general guidelines; it is not intended to replace existing International Standards which are in use.

Food and feed products — General guidelines for the determination of nitrogen by the Kjeldahl method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard provides general guidelines for the determination of nitrogen by the Kjeldahl method. It applies to food and feed products containing nitrogenous compounds that can be directly determined by the Kjeldahl method.

NOTE This measurement principle does not take into account the nitrogen from nitrates and nitrites.

2 Principle

Digestion of a test portion with concentrated sulfuric acid in the presence of catalysts to convert the organic nitrogen into ammonium sulfate. Excess sodium hydroxide is added to the cooled digest to release the ammonia. The released ammonia is distilled into an excess of boric acid solution and then titrated with a standard solution of sulfuric or hydrochloric acid. The nitrogen content is calculated from the quantity of ammonia produced.

NOTE In the following text, the term “nitrogen” refers to organic nitrogen.

3 Reagents

Use only reagents of recognized analytical grade, *unless otherwise specified*, and distilled or demineralized water or water of equivalent purity.

3.1 Sulfuric acid, virtually free from nitrogenous compounds and of mass density ρ_{20} = 1,83 g/ml to 1,84 g/ml.

3.2 Catalysts (see 5.2.1).

3.3 Boric acid solution (10 g/l to 40 g/l depending on the apparatus used). If using the colorimetric end-point titration, boric acid solution shall contain indicator (the pH or colour of this mixed solution shall be adjusted before use).

3.4 Standard hydrochloric acid (0,02 mol/l to 0,50 mol/l) or **sulfuric acid solution** (0,01 mol/l to 0,25 mol/l). The titre of the solution, c_t, shall be known to at least within 0,001 mol/l.

3.5 Indicators, which should change colour between pH 4 and pH 5.

NOTE Various indicators are available. A methyl red and bromocresol green mixed indicator is most commonly used. Ready-to-use boric acid solutions containing mixed indicators are available.

3.6 Hydrogen peroxide (H_2O_2), min. 30 % mass fraction.

3.7 Sodium hydroxide solution, min. 30 % mass fraction.

3.8 Antifoaming agents.

EXAMPLE Silicone, liquid paraffin.

3.9 Ammonium sulfate or **ammonium chloride** (minimum purity 99,9 %).

Immediately before use, dry the ammonium sulfate or ammonium chloride at 104 °C ± 4 °C for at least 2 h. Allow it to cool at ambient temperature in a desiccator.

NOTE Solutions of known concentration can be used.

3.10 Tryptophan or **acetanilide** or **lysine hydrochloride** (minimum purity 99 % mass fraction).

These reagents should be kept away from humidity.

WARNING — Do not dry these reagents in an oven before use.

3.11 Sucrose, with nitrogen content less than a mass fraction of 0,002 %.

WARNING — Do not dry sucrose in an oven before use.

4 Apparatus and materials

Usual laboratory apparatus and, in particular, the following.

4.1 Analytical balance, capable of weighing to the nearest 0,001 g.

4.2 Digestion, steam distillation and titration systems.

They are used to perform the operations described in Clause 5 and to ensure that the performance objectives described in 5.5.3 and 5.5.4 are met.

4.3 Boiling point regulators (if needed), for example pumice grains, glass beads, aluminium oxide (corundum) or silicon carbide.

4.4 Weighing paper or **medium**, free from nitrogenous compounds and suitable for the test portion and type of product.

5 Operating method

NOTE According to the nature of the sample, it may be necessary to prepare the test portion in advance to obtain a homogeneous sample (grinding, homogenization, etc.).

5.1 Test portion

The test portion, the quantity of which depends on the presumed nitrogen content determined by the Kjeldahl method, shall be representative of the sample and contain between 0,005 g and 0,2 g of nitrogen.

The test portion can be obtained by weighing with the analytical balance (4.1), to give mass, m, in grams or by using a pipette, to give volume, V_t, in millilitres.

The test portion can be inserted into the tube directly or via a support (4.4).

The quantity of test portion can be adjusted according to the composition of the product under test and the quantity of sulfuric acid (see 5.2.2).

5.2 Digestion

5.2.1 Catalysts

It is important to differentiate between the substances used to raise the boiling point of the liquid during digestion and the catalysts themselves that facilitate digestion. The former are usually potassium sulfate or, possibly, sodium sulfate. They are introduced in sufficient quantity to raise the boiling point of the acid to between 380 °C and 430 °C. The most commonly used catalyst is copper in the form of copper sulfate alone or mixed with titanium oxide.

The optional addition of hydrogen peroxide (3.6) on the basis of 3 ml to 5 ml per tube prior to heating accelerates digestion, but should be used with the utmost care to ensure that no nitrogen is lost in the form of vapour. Moreover, great care should be taken when adding hydrogen peroxide to the tubes, as this causes a strong exothermic reaction.

The quantity of potassium sulfate provided by the catalyst should not be less than 7 g.

Depending on the sectors of activity, various compositions are used. They should meet the requirements of the blank test (5.5.2) and the control tests (5.5.3 and 5.5.4).

Operators should handle selenium-based catalysts and waste conditions with care.

NOTE Ready-to-use composite catalysts are available on the market (for example in tablet or pellet form).

5.2.2 Addition of acid

It is important to use a sufficient quantity of sulfuric acid to ensure digestion after:

— acid consumption by the organic matter of the sample, bearing in mind the fact that 1 g of fat consumes 10 ml of sulfuric acid, 1 g of protein consumes 5 ml of sulfuric acid, 1 g of carbohydrate consumes 4 ml of sulfuric acid;

— acid consumption by the reagents (salts);

— acid losses by evaporation.

The addition of 20 ml to 25 ml of acid (3.1) is generally sufficient for good digestion and to maintain excess acid at the end of the reaction.

5.2.3 Heating

WARNING — The following operations should be performed under a very well ventilated fume hood.

The manufacturer's instructions relating to the use of the equipment should generally be followed. The digestion system should be made homogeneous, for example by creating a thermal or digestion efficiency process diagram (5.5.3).

Foam-producing agents should be brought to boiling point by increasing the temperature gradually or in steps. Three to four drops of antifoaming agent per tube (3.8) can also be used.

For “dry” products (i.e. with no visible wetness), the tubes can be placed directly in a preheated unit.