SLOVENSKI STANDARD
SIST EN 12135:1998

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Sadni in zelenjavni sokovi - Določevanje dušika - Kjeldahlova metoda

Fruit and vegetable juices - Determination of nitrogen content - Kjeldahl method

Frucht- und Gemüsesäfte - Bestimmung des Stickstoffgehaltes - Kjeldahlverfahren

Jus de fruits et de légumes - Dosage de l'azote - Méthode de Kjeldhal

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ICS:

67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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English version

Fruit and vegetable juices - Determination of nitrogen content - Kjeldahl method

Jus de fruits et de légumes - Dosage de l'azote - Méthode de Kjeldahl

Frucht- und Gemüsesäfte - Bestimmung des Stickstoffgehaltes - Kjeldahlverfahren

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 174 "Fruit and vegetable juices - Methods of analysis", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by March 1998, and conflicting national standards shall be withdrawn at the latest by March 1998.

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

1 Scope

This European Standard specifies a method for the determination of the nitrogen content of fruit and vegetable juices and related products by the Kjeldahl method.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1131:1994	Fruit and vegetable juices - Determination of the relative density
EN ISO 3696:1995	Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)
ISO 5725:1986	Precision of test methods - Determination of repeatibility and reproducibility for a standard test method by inter-laboratory tests

3 Definition and symbols

3.1 Definition

For the purposes of this European Standard the following definition applies :

3.1.1 total nitrogen content

The quantity of nitrogen present in the product and corresponding to the ammonia produced and determined under the conditions specified in this European Standard, expressed in milligrams per litre or milligrams per kilogram.

3.2 Symbols

For the purposes of this European Standard the following symbols apply :

c substance concentration
ρ mass concentration
ϕ volume fraction

4 Principle

Digestion of a test portion with concentrated sulfuric acid, using a catalyst, to convert organic nitrogen into ammonium sulfate ; adjustment of the diluted solution to alkaline pH, distillation of the liberated ammonia into an excess of boric acid solution, titration with sulfuric acid or hydrochloric acid to determine the ammonia bound by the boric acid, and calculation of the nitrogen content of the sample from the amount of ammonia produced.

5 Reagents

Use only reagents of recognized analytical grade and only water in accordance with at least grade 3 of EN ISO 3696:1995.

5.1 Sulfuric acid, ρ_{20} (H_2SO_4) = 1,84 g/ml.

WARNING : Concentrated sulfuric acid is corrosive and causes burns ! Wear non-permeable gloves. Avoid contact with skin.

5.2 Sulfuric acid standard volumetric solution, $c(H_2SO_4)$ = 0,05 mol/l.

5.3 Hydrochloric acid standard volumetric solution, c(HCl) = 0,1 mol/l, alternative to 5.2.

5.4 Potassium sulfate (K_2SO_4).

5.5 Copper (II) sulfate ($CuSO_4$), anhydrous.

5.6 Copper (II) sulfate pentahydrate ($CuSO_4 \cdot 5\ H_2O$), alternative to 5.5.

5.7 Catalyst mixture

100 g of potassium sulfate (5.4) and 10 g of Copper (II) sulfate (5.5) or 15,64 g of Copper (II) sulfate pentahydrate (5.6) are thoroughly mixed in a mortar (6.8).

5.8 Selenium dioxide (SeO_2)

5.9 Sodium hydroxide solution

Dissolve 400 g of sodium hydroxide (NaOH) in water and dilute to 1 000 ml.

5.10 Boric acid solution

Dissolve 20 g of boric acid (H_3BO_3) in water and dilute to 1 000 ml.

5.11 Indicator solution : mixed indicator, prepared by dissolving 100 mg of methyl red and 50 mg of methylene blue in 100 ml of ethanol, ϕ (CH_3CH_2OH) = 95 %.

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 Kjeldahl flask of 100 ml capacity.

6.2 Digestion apparatus with an effective suction device, for acid fumes evolved during the digestion.

6.3 Distillation apparatus, e.g. Parnas-Wagner type [1].

6.4 Pipette of 10 ml capacity.

6.5 Measuring cylinder of 25 ml capacity.

6.6 Burette, graduated in 0,1 ml.

6.7 Conical flask

6.8 Mortar

6.9 Boiling aids

7 Procedure

7.1 Preparation of the test sample

Normally products shall not be pre-treated, however dilution may be necessary and their analysis by this method shall be on a volumetric basis, results being expressed per litre of sample. The analysis of concentrated products may also be carried out on a volumetric basis, after dilution to a known relative density. In this case, the relative density shall be indicated. Based on a weighed sample and taking the dilution factor for analysis into account, the results may also be expressed per kilogram of product. In products with a high viscosity and/or a very high content of cells (for example pulp), a determination on the basis of a weighed test sample is the usual procedure.

7.2 Determination

7.2.1 Digestion

Pipette 10 ml of test sample(V_s) into a Kjeldahl flask (6.1) and transfer to the flask a few pieces of boiling aids (6.9). Heat gently in or on the digestion apparatus (6.2) until the test sample becomes dark but do not evaporate fully to dryness. Allow to cool.

Calculate the soluble solids content of the test sample from the relative density determined using EN 1131. Use 4 ml of concentrated sulfuric acid (5.1) per gram of soluble solids, most of which should be sugar.

Transfer into the Kjeldahl flask (6.1) after cooling, the calculated amount of concentrated sulfuric acid (5.1) plus 1 ml excess. Add 0,9 g of catalyst mixture (5.7) and a granule of selenium dioxide (5.8).

> NOTE : For example 10 ml of test sample with a relative density of 1,040 and a soluble solid content of 1 g requires 5 ml of concentrated sulfuric acid (i.e. 4 ml plus 1 ml).

Heat gently again on or in the digestion apparatus (6.2), boil until the solution is clear, and rotate the flask frequently until no particles of material are left in the neck of the flask. Then keep the liquid boiling for another 60 min.

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[1] Parnas-Wagner is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this product.

Allow to cool to room temperature. Carefully add about 35 ml of water. Mix and cool again.

7.2.2 Distillation

Transfer into a conical flask (6.7) 30 ml of the boric acid solution (5.10) and one or two drops of the indicator solution (5.11). Mix. Place the conical flask under the condenser so that the tip of the outlet tube is immersed in the boric acid solution.

Transfer the content of the Kjeldahl flask quantitatively to the distillation apparatus (6.3). Add 20 ml of the sodium hydroxide solution (5.9) by means of a measuring cylinder (6.5).

Distil so that about 100 ml of distillate are collected in approximately 4 min to 5 min. Make sure that the distillate is cooled effectively and the content of the conical flask does not exceed 25 °C during the distillation. About 30 s before the end of the distillation, lower the conical flask so that the tip of the outlet tube is no longer immersed in the acid solution and rinse the tip with a small amount of water.

7.2.3 Titration

Titrate the distillate in the conical flask using the standard volumetric solution (5.2 or 5.3) until the indicator turns to pink. Record the volume (V) of sulfuric or hydrochloric acid solution required.

8 Calculation

Calculate the total nitrogren content ρ_N of the test sample in milligrams per litre using the following equation :

$$\rho_N = 1{,}4 \cdot V \cdot \frac{1\,000}{V_s}$$

where :

V is the volume in millilitres of sulfuric (5.2) or hydrochloric acid solution (5.3) required for the determination

V_S is the volume in millilitres of the test portion

Take into account the dilution factor and the relation of the value to mass or volume. If a concentrated product has been diluted to single strength, report the relative density of the single strength sample.

Express the nitrogen content ρ_N of the test sample without a decimal place.

9 Precision

Details of the interlaboratory test on precision of the method are summarized in annex B. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than given in annex B.