



SLOVENSKI STANDARD

SIST EN 1134:1996

01-avgust-1996

Sadni in zelenjavni sokovi - Ugotavljanje vsebnosti natrija, kalija, kalcija in magnezija z atomsko absorpcijsko spektrometrijo (AAS)

Fruit and vegetable juices - Determination of sodium, potassium, calcium and magnesium content by atomic absorption spectrometry (AAS)

Frucht- und Gemüsesäfte - Bestimmung der Gehalte an Natrium, Kalium, Calcium und Magnesium - Atomabsorptionsspektrometrisches Verfahren (AAS)

Jus de fruits et de légumes - Dosage du sodium, du potassium, du calcium et du magnésium par spectrométrie d'absorption atomique (SAA)

<https://standards.iteh.ai/catalog/standards/sist/a3ee2089-a1c4-47a4-a6c7-35c4385b1efb/sist-en-1134-1996>

Ta slovenski standard je istoveten z: EN 1134:1994

ICS:

67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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en

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EUROPEAN STANDARD

EN 1134

NORME EUROPÉENNE

EUROPÄISCHE NORM

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Descriptors: food products, beverages, fruit and vegetable juices, chemical analysis, determination of content, sodium, potassium, calcium, magnesium, atomic absorption spectrophotometry, flame photometry

English version

**Fruit and vegetable juices - Determination of
sodium, potassium, calcium and magnesium
content by atomic absorption spectrometry (AAS)**

Jus de fruits et de légumes Dosage du sodium,
du potassium, du calcium et du magnésium par
spectrométrie d'absorption atomique (SAA)

Frucht- und Gemüsesäfte - Bestimmung der
Gehalte an Natrium, Kalium, Calcium und
Magnesium - Atomabsorptionsspektrometrisches
Verfahren (AAS)

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This European Standard was approved by CEN on 1994-09-29. 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.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

Foreword

This European Standard has been prepared by the 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 April 1995, and conflicting national standards shall be withdrawn at the latest by April 1995.

Annexes designated "informative" are given only for information. In this standard annexes A and B are informative.

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.

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1 Scope

This European standard specifies a method for the determination of sodium, potassium, calcium and magnesium content of fruit and vegetable juices and related products by atomic absorption spectrometry.

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.

- | | |
|---------------|---|
| ISO 5725:1986 | Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests |
| ISO 3696:1987 | Water for analytical laboratory use - Specification and test methods |

3 Symbols

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For the purposes of this standard, the following symbols apply:

- | | | |
|--------|--------------------------|--|
| c | Substance concentration. | SIST EN 1134:1996 |
| ρ | Mass concentration. | 35c4385b1efb/sist-en-1134-1996 |

4 Principle

Sodium, potassium, calcium and magnesium content is determined by atomic absorption spectrometry (AAS) on the diluted test sample. In samples containing a high proportion of pulp, the calcium content is determined after ashing. In order to prevent a partial ionization of the metals in the flame, caesium chloride is added when determining sodium and potassium, and lanthanum, as a matrix modifier, when determining calcium and magnesium.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and only water in accordance with at least grade 2 of ISO 3696:1987.

Standard and calibration solutions shall be kept in polyethylene bottles.

5.2 Sodium standard solution, ρ (Na⁺) = 1 g/l

A commercially available sodium standard solution is used. This solution may also be prepared by dissolving 2,542 g of dried sodium chloride (NaCl) in water and making up to 1 litre in a volumetric flask.

5.3 Potassium standard solution, ρ (K⁺) = 1 g/l

A commercially available potassium standard solution is used. This solution may also be prepared by dissolving 4,813 g of potassium hydrogen tartrate (C₄H₅KO₆) in water and making up to 1 litre in a volumetric flask (20 °C).

5.4 Calcium standard solution, ρ (Ca⁺⁺) = 1 g/l

A commercially available calcium standard solution is used. This solution may also be prepared by dissolving 2,5 g of calcium carbonate (CaCO₃) in a sufficient quantity of hydrochloric acid (5.8) and making up to 1 litre with water in a volumetric flask.

5.5 Magnesium standard solution, ρ (Mg⁺⁺) = 1 g/l

A commercially available magnesium standard solution is used. This solution may also be prepared by dissolving 8,3646 g of magnesium chloride (MgCl₂·6H₂O) in a sufficient quantity of hydrochloric acid (5.8) and making up to 1 litre in a volumetric flask.

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<https://standards.iteh.ai/catalog/standards/sist/a3ee2089-a1c4-47a4-a6c7-55c438581cb/sist-en-1134-1996>**5.6 Caesium chloride stock solution, ρ (CsCl) = 40 g/l**

Dissolve 40 g of caesium chloride, CsCl in water and make up to 1 litre in a volumetric flask.

5.7 Lanthanum stock solution, ρ (La³⁺) = 50 g/l

Moisten 58,6 g of lanthanum oxide, La₂O₃ with water in a 400 ml beaker and carefully add 100 ml hydrochloric acid ρ (HCl) = 370 g/l until all of the La₂O₃ is dissolved. Transfer to a 1 litre volumetric flask and make up to the mark with water. Lanthanum chloride may also be used to prepare this solution. In this case, dissolve 134 g of LaCl₃ · 7 H₂O in water and make up to 1 litre in a volumetric flask.

5.8 Hydrochloric acid solution, ρ (HCl) = 100 g/l**6 Apparatus**

Usual laboratory apparatus and, in particular, the following :

- 6.1 Atomic absorption spectrometer with burner head for air/acetylene.
- 6.2 Hollow cathode lamp for sodium
- 6.3 Hollow cathode lamp for potassium
- 6.4 Hollow cathode lamp for calcium
- 6.5 Hollow cathode lamp for magnesium
- 6.6 Enzyme test pipettes graduated along the stem only, with long ungraduated delivery tip.
- 6.7 Pipettes with accuracy equivalent to 6.6 (alternative to 6.6) e.g. positive displacement capillary pipettes.

7 Procedure

7.1 Preparation of the test sample

Normally products shall not be pretreated and their analysis by this method shall be on a volumetric basis, results being expressed per litre. 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 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 high viscosity and/or very high content of cells (for example pulp), determination on the basis of a weighed test sample is the usual procedure.

Clear and cloudy test samples are used without prior centrifugation. Test samples containing a high proportion of pulp are ashed beforehand for the determination of calcium. The ash is absorbed in 2 ml of hydrochloric acid, $c(\text{HCl}) = 4 \text{ mol/l}$, transferred quantitatively into a 50 ml graduated flask and made up to the 50 ml mark with water. The subsequent dilution of the sample is chosen in such a manner that the expected concentration of Ca^{++} lies within the linear range of the calibration curve. The lanthanum content in the solution to be measured amounts to 0,5 g/100 ml.

7.2 Test procedure

7.2.1 General

Special attention is to be paid to the cleanliness of the apparatus used. All glassware shall be rinsed beforehand with water of Grade 2, according to ISO 3696:1987.

For measuring the standard and sample solutions enzyme-testing pipettes (6.6) or their equivalent (6.7) are to be used and also suitable glassware for handling small volumes.

When diluting cloudy juices, a sample of at least 1 ml shall be taken.

Calibration of the instrument is to be carried out for each series of measurements.

When carrying out the determination, make sure that the atomic absorption spectrophotometer is carefully adjusted to the optimum wavelength and perform the measurements according to the instrument manufacturer's instructions.

If separate stages of the determination are carried out in different laboratories (e.g. general laboratory and instrument room) it is essential that the same batch of water be used for preparing both the sample solutions and the reference solutions.

7.2.2 Sodium determination (wavelength 589,0 nm)

7.2.2.1 Preparation of the solution for the zero setting

Prepare a solution of caesium chloride, ρ (CsCl) = 0,4 g/100 ml, from the stock solution (5.6).

7.2.2.2 Preparation of the reference solutions

According to the expected sodium contents of the samples, 3 to 5 dilutions are prepared from the sodium standard solution for calibration purposes. Add cesium chloride stock solution to the solutions to be measured, such that ρ (CsCl) = 0,4 g/ 100 ml.

7.2.2.3 Preparation of the sample solution

The dilution of the sample is chosen in such a manner that the expected sodium content lies within the linear range of the calibration curve. Add cesium chloride stock solution to the solutions to be measured, such that ρ (CsCl) = 0,4 g/ 100 ml.

7.2.3 Potassium determination (wavelength 766,5 nm or 769,9 nm)

7.2.3.1 Preparation of the solution for the zero setting

Prepare a solution of caesium chloride, ρ (CsCl) = 0,4 g/100 ml from the stock solution (5.6).

7.2.3.2 Preparation of the reference solutions

According to the expected potassium contents of the samples, 3 to 5 dilutions are prepared from potassium standard solution for calibration purposes. Add cesium chloride stock solution to the solutions to be measured, such that ρ (CsCl) = 0,4 g/100 ml.

7.2.3.3 Preparation of the sample solution

The dilution of the sample is chosen in such a manner that the expected potassium content lies within the linear range of the calibration curve. Add caesium chloride stock solution to the solutions to be measured, such that $\rho(\text{CsCl}) = 0,4 \text{ g}/100 \text{ ml}$.

7.2.4 Calcium determination (wavelength 422,7 nm)

7.2.4.1 Preparation of the solution for the zero setting

Prepare a solution of lanthanum, $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$, from the stock solution (5.7).

7.2.4.2 Preparation of the reference solutions

According to the expected calcium contents of the samples, 3 to 5 dilutions are prepared from calcium standard solution for calibration purposes. Add lanthanum stock solution (5.7) to the solutions to be measured, such that $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$.

7.2.4.3 Preparation of the sample solution

The dilution of the sample (if necessary after sample preparation, see 7.1) is chosen in such a manner that the expected calcium content lies within the linear range of the calibration curve. Add lanthanum stock solution (5.7) to the solutions to be measured, such that $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$.

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7.2.5 Magnesium determination (wavelength 285,2 nm)

7.2.5.1 Preparation of the solution for the zero setting

Prepare a solution of lanthanum, $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$, from the stock solution (5.7).

7.2.5.2 Preparation of the reference solutions

According to the expected magnesium contents of the samples, 3 to 5 dilutions are prepared from the magnesium standard solution for calibration purposes. Add lanthanum stock solution (5.7) to the solutions to be measured, such that $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$.

7.2.5.3 Preparation of the sample solution

The dilution of the sample is chosen in such a manner that the expected magnesium content lies within the linear range of the calibration curve. Add lanthanum stock solution (5.7) to the solutions to be measured, such that $\rho(\text{La}^{3+}) = 0,5 \text{ g}/100 \text{ ml}$.