SLOVENSKI STANDARD
SIST EN 12133:1998

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Sadni in zelenjavni sokovi - Določevanje klorida - Potenciometrijska titracijska metoda

Fruit and vegetable juices - Determination of chloride content - Potentiometric titration method

Frucht- und Gemüsesäfte - Bestimmung des Chloridgehaltes - Potentiometrisches Titrationsverfahren

Jus de fruits et de légumes - Dosage des chlorures - Méthode de titrage potentiométrique

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67.160.20	Brezalkoholne pijače	Non-alcoholic beverages

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English version

Fruit and vegetable juices - Determination of chloride content - Potentiometric titration method

Jus de fruits et de légumes - Dosage des chlorures - Méthode de titrage potentiométrique

Frucht- und Gemüsesäfte - Bestimmung des Chloridgehaltes - Potentiometrisches Titrationsverfahren

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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of an identical text or by endorsement, at the latest by March 1998, and conflicting national standards shall be withdrawn at the latest by March 1998.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 potentiometric titration method for the determination of the chloride content of fruit and vegetable juices and related products.

2 Normative references

This European Standard incorporates by dated or undated references, 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 ISO 3696:1995	Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)
ISO 5725:1986	Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests

3 Symbols

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

c	Substance concentration
ρ	Mass concentration
ω	Mass fraction
d	Specific gravity
n	The volume in millilitres of the silver nitrate standard solution used to titrate the chloride content of the fruit juice sample using the end potential method
V	Volume of standard solution at the equivalence point
V'	Volume of standard solution added before the potential rise
ΔV_i	Constant volume for addition of the standard solution (e.g. 0,2 ml)
ΔE	First potential difference
$\Delta\Delta E$	Second potential difference
$\Delta\Delta E_1$	Second potential difference before the biggest potential rise
$\Delta\Delta E_2$	Second potential difference after the biggest potential rise

4 Principle

The chloride content is determined by potentiometric titration using a silver/silver chloride electrode.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and only water in accordance with at least grade 2 of EN ISO 3696:1995.

5.2 Potassium chloride

The potassium chloride shall contain no more than 0,005 % (*m/m*) bromide and shall be dried for three days to four days in a desiccator before use.

5.3 Nitric acid, ω (HNO_3) $\geq$ 65 g/100 g, d $\geq$ 1,4

5.4 Potassium chloride solution, ρ (KCl) = 1,8636 g/l.

Dissolve 1,8636 g of potassium chloride (5.2) in 1 l of water (5.1). 1 ml of this solution contains 0,886 mg of chloride.

5.5 Silver nitrate standard solution, c ($AgNO_3$) = 0,025 mol/l

1 ml of this solution reacts with 0,886 mg of chloride.

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 pH-millivoltmeter readable to at least 2 mV.

6.2 Magnetic stirrer

6.3 Ag/AgCl electrode with saturated potassium nitrate solution as electrolyte.

6.4 Micro-burette readable to at least 0,01 ml.

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.

Mix cloudy samples well before dilution.

7.2 Test procedure

Following the manufacturer's instructions for the apparatus used, either titrate to the end potential or plot the potential curve.

7.2.1 Titratation to the end potential

Pipette 5,0 ml of standard chloride solution (5.4) into a 150 ml beaker. Dilute to approximately 100 ml with distilled water (5.1) and add 1,0 ml of nitric acid (5.3). Dip the electrode (6.3) into the solution, and add silver nitrate solution (5.5) from the micro-burette (6.4) while stirring slowly using the magnetic stirrer (6.2).

Titrate a total of 4,00 ml in steps of 1,00 ml, recording the potential in millivolts at each step on the pH-millivoltmeter (6.1). Then titrate a further 2,00 ml, in steps of 0,2 ml. Finally titrate up to a total of 10,00 ml in 1,00 ml steps. After each addition, wait about 30 s before reading the potential in millivolts. Plot the values obtained against the volume (in millilitres) of standard solution added. Hence estimate the end potential from the inflection point on the curve.

Pipette 5,0 ml of standard chloride solution (5.4) into a 150 ml beaker. Add 95 ml of water (5.1), followed by 1,0 ml of nitric acid (5.3). After inserting the electrode, titrate the solution to the equivalence potential obtained previously. This estimation should be repeated until a reproducible figure is obtained. This figure then serves for all the chloride estimations in each particular batch of samples.

Pipette 50,0 ml of a suitably diluted sample into a 150 ml beaker. Add 50 ml of distilled water followed by 1,0 ml of nitric acid, and titrate as above.

7.2.2 Determination of potential curve

For particularly accurate determinations it is possible to obtain the complete titration curve of the test sample against silver nitrate standard solution. The equivalence point can then be obtained graphically or, for the highest accuracy, by calculation.

Pipette 50 ml of the suitably diluted sample into a 150 ml beaker with 50 ml of distilled water (5.1) and 1 ml of nitric acid (5.3). Titrate against the silver nitrate standard solution in 0,5 ml steps and record the corresponding potential at each step. Use this initial titration to give the rough titration volume.

Repeat the estimation under the same conditions. Begin in 0,5 ml steps and reduce these to 0,2 ml steps once within 1,5 ml to 2,0 ml of the expected endpoint. Continue similarly after the endpoint and then raise the steps to 0,5 ml in a symmetrical fashion.

8 Calculation

8.1 Calculation for the end potential technique

If n ml of silver nitrate solution were used, the chloride content ρ_{Cl^-} in milligrams per litre is calculated from the following equation :

$$\rho_{Cl^-} = n \cdot 17{,}72 \qquad (1)$$

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

Report the chloride content for fruit juice in milligrams per litre to one decimal place, and for vegetable juice in grams per litre to one decimal place.

8.2 Calculation for the potential curve technique

The volume of standard solution at the equivalence point is calculated from the following expression :

$$V = V' + \Delta V_i \cdot \frac{\Delta\Delta E_1}{\Delta\Delta E_1 + \Delta\Delta E_2} \qquad (2)$$

The chloride content ρ_{Cl^-} in milligrams per litre is calculated from the following expression :

$$\rho_{Cl^-} = V \cdot 17{,}72 \qquad (3)$$

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

Report the chloride content for fruit juice in milligrams per litre to one decimal place, and for vegetable juice in grams per litre to one decimal place.