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Analytical Products

Application Note

Total Phosphorus in Fruit and Vegetable Juices

(according to EN 1136 and German Food and Feed Code §64 LFGB 31.00-6)

Note

Pursuant to the valid copyright regulations this application note contains only a rough description of the content of the official method followed by a detailed description of the specific measurement procedure with the Spectroquant® Prove Spectrophotometers. A detailed description of the method specific handling steps can be found in the official methods EN 1136 [1] and the German Food and Feed Code §64 LFGB 31.00-6 [2].

Method

Most foods containing phosphorus compounds. Especially foods with rich content of proteins like milk and dairy products or meat and poultry are sources of phosphorus compounds but also fruit and vegetable juices containing phosphorus compounds.

The total phosphorus content in fruit and vegetable juices is determined in the ash of the sample. The prepared sample reacts with ammonium heptamolybdate and ascorbic acid to form phosphomolybdenum blue that is measured photometrically at 720 nm (according to the cited standards).

This method is based on the official methods EN 1136 [1] and the German Food and Feed Code §64 LFGB 31.00-6 [2] and describes the determination of the total phosphorus in in fruit and vegetable juices.

Measuring range

Method 2534	Phosphorus Juice EN 1136	0.0 – 300.0 mg/l P

Sample

Fruit and vegetable juices

Reagents and auxiliaries

Cat. No.	Products
1.73026	Spectroquant® VIS Spectrophotometer Prove 100 plus or
1.73027	Spectroquant® UV/VIS Spectrophotometer Prove 300 plus or
1.73028	Spectroquant® UV/VIS Spectrophotometer Prove 600 plus
114946	Rectangular cells 10 mm
109063	Hydrochloric acid 2 mol/l (2N) Titripur®
160313	Sulfuric acid 1 mol/l (2 N) Titripur®
101182	Ammonium heptamolybdate tetrahydrate GR for analysis
255564	L-Ascorbic acid ACS reagent
119898	Phosphate standard solution traceable to SRM from NIST KH_2PO_4 in H_2O 1000 mg/l PO_4 Certipur®

Also first generation Prove instruments are compatible and preprogrammed with this method.

MERCK

Additional needs

- Platinum dishes, 80 mm diameter
- Muffle furnace
- Crucible tongs
- Exicator
- Water bath
- Graduated cylinders, 25 mL
- Volumetric flasks, 50 mL, 100 mL
- Standard laboratory glassware (e. g. glass beakers) and pipettes
- Analytical balance.

Preparing the solutions

- **Ammonium heptamolybdate solution** – The solution must be prepared according to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2].
- **Ascorbic acid solution** – The solution must be prepared according to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2].

Sample preparation

According to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2] and EN 1135 [3].

Procedure

Ashing

- Pipette 25.0 mL sample into a platinum dish and follow the procedure according to EN 1136 [3].
- Dissolve the pure white ash in 2 – 3 mL Hydrochloric acid 2 mol/l and transfer it to a 50 mL volumetric flask. Fill up to the mark with distilled water.

Phosphorus determination

- **Reagent blank**
- Place 50 mL of dist. water into a 100 mL volumetric flask, add 20 mL Sulfuric acid 1 mol/l, 2 mL Ammonium heptamolybdate solution, 2 mL Ascorbic acid solution, mix and fill up to the mark with distilled water.
- Incubate this solution according to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2].

Sample

- Place an appropriate volume of the prepared sample into a 100 mL volumetric flask, add approx. 50 mL of distilled water, 20 mL Sulfuric acid 1 mol/l, 2 mL Ammonium heptamolybdate solution, 2 mL Ascorbic acid solution, mix and fill up to the mark with distilled water.
- Note the volume of the prepared sample
- Incubate this solution according to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2].

Note

The recommended volume of the prepared sample depends on the phosphorus content of the juice sample. Experience has shown that it is advisable to use a volume of 2.0 mL for grape juices, 2.0 mL for orange juices and 5.0 mL for apple juices.

Measurement

Note

It is advisable to measure the reagent blank and the sample using the same cell as the one used for the zero adjustment or else a cell with identical optical characteristics and an identical absorption (matched pair).

- Open the methods list (<Methods>) and select Method No. 2534 "Phosphorus Juice EN 1136".
- The instrument automatically prompts a "Zero adjustment".
- For the zero adjustment fill a clean and dry 10-mm rectangular cell with distilled water.
- After prompting, insert the filled rectangular cell into the cell compartment. The zero adjustment is performed automatically.
- Confirm the performance of the zero-adjustment procedure by clicking on **<OK>**
- A window with an input field to enter the sample volume pops up.
- Enter the volume of the sample in milliliter (mL), accurate to 0.1 milliliter (mL), confirm with **<OK>** and click on **<START>** to switch to the measurement procedure.

Note

It is possible to enter a sample volume in a range of 0.1 to 20.0 mL.

- Fill the prepared reagent blank into a clean and dry 10-mm rectangular cell. Insert the cell into the cell compartment. The measurement is performed automatically. A (✓) symbol appears behind the cue "Insert Reagent Blank".
- Confirm the measurement by clicking on <OK>.
- Finally fill the prepared sample solution into a clean and dry 10-mm rectangular cell. Insert the cell into the cell compartment. The measurement is performed automatically. A (✓) appears behind the cue "Insert Sample".
- Confirm the measurement by clicking on <OK>.

- Read off the result in mg/l P and the absorption for the reagent blank (A_{RB}) and the sample (A_{Sample}) from the display.
- Tap the **<START>** button to start the measurement procedure for the next sample.

Evaluation

Statement of the results:

- Total phosphorus [mg/l P]
- Absorption of reagent blank A_{RB}
- Absorption of sample A_{Sample}

Method control

- The method can be checked using Cat. No. 119898 Phosphate standard solution traceable to SRM from NIST KH_2PO_4 in H_2O 1000 mg/l PO_4 Certipur®.
- Dilute this solution to 10 mg/l P with water for analysis or distilled water.
- Dilution: 1000 mg/l PO_4 ≙ 326.1 mg/l P →10 mg/l P
- Place 3.067 mL **Cat. No. 119898** Phosphate standard solution 1000 mg/l PO4 into a 100 mL volumetric flask and fill up to the mark with distilled water.
- Place 10 mL of this solution into a 100 mL volumetric flask, add approx. 50 mL of distilled water, 20 mL Sulfuric acid 1 mol/l, 2 mL Ammonium heptamolybdate solution, 2 mL Ascorbic acid solution, mix and fill up to the mark with distilled water.
- Incubate this solution according to EN 1136 [1] resp. German Food and Feed Code §64 LFGB 31.00-6 [2].
- Measure this solution versus a reagent blank as described in the section „Measurement". Hereby enter a volume of 10.00 mL

Note

Due to the different sample preparation procedure and phosphorus determination procedure of the 10 mg/l P standard solution (no ashing step) compared to a sample analysis (ashing) it is necessary to recalculate the displayed result manually as follows:

Measured Concentration standard [mg/l] =

Displayed result [mg/l] / F1 =

Displayed result [mg/l] / 2

F1 = 2 = Factor sample preparation for real sample (dilution after ashing)

Adjustment

- In case of significant deviations in the method control procedure the preprogrammed factor of 2.999 or the current factor used in the calculation of the displayed results can be adjusted by the user.
- The corrected factor must be recalculated as follows:

  Factor corrected = Current factor x (target value standard / measured and recalculated value standard)
- To edit the preprogrammed factor, select method 2534 from <Methods>.
- Close the window for the "Zero adjustment" by clicking on <X>.
- Close the input field for the sample volume by clicking on <X>
- Click <Settings> and select the list "FACTORS".
- Tip on the input field "Factor", enter the corrected factor and confirm by clicking on <OK>.
- Close the window for the "Zero adjustment" by clicking on <X>.
- For the next measurement restart the method by selecting the method anew from <Methods>.

Note

To find the used factor, select Method 2534 from <Methods>.

Close the window for the "Zero adjustment" by clicking on <X>.

Close the input field for the sample volume by clicking on <X>.

Click <Settings> and select the list "FACTORS".

Literature

1. Fruit and vegetable juices – Determination of phosphorus content – Spectrometric method; EN 1136:1994.
2. German Food and Feed Code §64 LFGB 31.00-6:1997 Bestimmung des Phosphorgehaltes in Frucht- und Gemüsesäften.
3. Fruit and vegetable juices – Determination of ash; EN 1135:1995.

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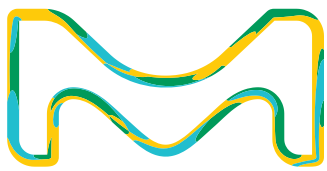

MK_AN8052EN Ver. 1.0
36287
01/2024