

Total Phosphorus in Milk and Dairy Products

(according to German Food and Feed Code §64 LFGB 01.00-92)

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 method of the German Food and Feed Code §64 LFGB 01.00-92 ^[1].

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.

The total phosphorus content in milk and dairy products is determined after wet digestion of the sample under acidic and oxidative conditions or after dry ashing digestion. The prepared sample reacts with a molybdate and ascorbic acid reagent to form phosphomolybdenum blue that is measured photometrically at 820 nm.

This method is based on the official method of the German Food and Feed Code §64 LFGB 01.00-92 ^[1] and describes the determination of the total phosphorus in milk and dairy

Measuring range

Method 2532

Phosphorus Milk §64 LFGB 01.00-92

0 – 2000 mg/100g P

Sample material

Milk and dairy products



Reagents and auxiliaries

Cat. No.	Description
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
100731	Sulfuric acid 95-97% for analysis EMSURE [®]
160315	Diethyl ether for analysis EMSURE [®]
107210	β-Carotene Pharmaceutical Secondary Standard; CRM
109057	Hydrochloric acid 1 mol/l (1N) Titripur [®]
106521	Sodium molybdate dihydrate for analysis EMSURE [®]
255564	L-Ascorbic acid ACS reagent
119898	Phosphate standard solution traceable to SRM from NIST KH ₂ PO ₄ in H ₂ O 1000 mg/l PO ₄ Certipur [®]

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

Additional needs

- Platinum, quartz or porcelain dishes, 55-mm diameter (dry ashing only)
- Watch glass (dry ashing only)
- Muffle furnace (dry ashing only)
- Crucible tongs (dry ashing only)
- Exicator (dry ashing only)
- Bunsen burner / Heating block (wet digestion only)
- Digestion flask or digestion tubes (wet digestion only)
- Glass beads (wet digestion only)
- Cheese grater
- Water bath
- Graduated cylinders, 5 mL, 25 mL
- Volumetric flasks, 20 mL, 50 mL, 100 mL
- Standard laboratory glassware (e. g. glass beakers) and pipettes
- Analytical balance

Preparing the solutions

- **Sodium molybdate solution:**
The solution must be prepared according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].
- **Ascorbic acid solution:**
The solution must be prepared according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].
- **Sodium molybdate / Ascorbic acid reagent:**
The reagent must be freshly prepared before use according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].

Sample Preparation

- according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].

Procedure

Wet digestion resp. dry ashing

- Weigh sample to a digestion flask or tube for the wet digestion step or to a dish for the dry ashing step and follow the procedure according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].
- Note the sample weight.

Phosphorus determination

- **Reagent blank**
- Place 10 mL of Sodium molybdate / Ascorbic acid reagent into a 20 mL volumetric flask and fill up to the mark with distilled water.
- Incubate this solution according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].

• Sample

- Place 1 mL of the prepared sample into a 50 mL volumetric flask, add approx. 25 mL of distilled water, add 25 mL of the Sodium molybdate / Ascorbic acid reagent, mix and fill up to the mark with distilled water.
- Incubate this solution according to German Food and Feed Code §64 LFGB 01.00-92 ^[1].

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. 2532 „Phosphorus Milk §64 LFGB 01.00-92“.
- 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 weight pops up.
- Enter the weight of the sample in grams (g), accurate to 0.001 grams (g), confirm with **<OK>** and click on **<START>** to switch to the measurement procedure.

Note

It is possible to enter a sample weight in a range of 0.010 to 10.000 g.

- 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/100g P and the absorption for the reagent blank (ARB) and the sample (ASample) from the display.
- Tap the **<START>** button to start the measurement procedure for the next sample.

Evaluation

Statement of the results:

Total phosphorus [mg/100 g 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 50 mg/l P with water for analysis or distilled water.
- Dilution: 1000 mg/l PO_4 = 326.1 mg/l P → 50 mg/l P
Place 3.067 mL Cat. No. 119898 Phosphate standard solution 1000 mg/l PO_4 into a 20 mL volumetric flask and fill up to the mark with distilled water.
- Place 1 mL of this solution into a 50 mL volumetric flask, add approx. 25 mL of distilled water, add 25 mL of the Sodium molybdate / Ascorbic acid reagent, mix and fill up to the mark with distilled water.
- Incubate this solution according to German Food and Feed Code §64 LFGB 01.00-92^[1].
- Measure this solution versus a reagent blank as described in the section „Measurement“. Hereby enter a weight of 1.00 g.

Note

Due to the different sample preparation procedure and phosphorus determination procedure of the 50 mg/l P standard solution compared to a sample analysis it is necessary to recalculate the displayed result manually as follows:

Measured Concentration standard [mg/l] =

Displayed result [mg/100g] * F1 / F2 =

Displayed result [mg/100 g] x 10 / 100 =

Displayed result [mg/100 g] / 10

F1 = 10 = recalculation mg/100 g to mg/l

F2 = 100 = Factor sample preparation for real sample

Adjustment

- In case of significant deviations in the method control procedure the preprogrammed factor of 58.24 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 2532 from <Methods>.
- Close the window for the “Zero adjustment” by clicking on <X>.
- Close the input field for the sample weight 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 2532 from <Methods>.

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

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

Click <Settings> and select the list “FACTORS”.

Literature

1. German Food and Feed Code §64 LFGB 01.00-92:2016 Bestimmung des Gesamtphosphorgehaltes in Milch und Milchprodukten

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To place an order or receive technical assistance

Order/Customer Service: SigmaAldrich.com/order

Technical Service: SigmaAldrich.com/techservice

Safety-related Information: SigmaAldrich.com/safetycenter

SigmaAldrich.com

