

Yellow Pigment in Durum Wheat Flour and Semolina

(according to EN ISO 11052 and German Food and Feed Code §64 LFGB 16.01-3)

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 ISO 11052 ^[1] and the German Food and Feed Code §64 LFGB 16.01-3 ^[2].

Method

The content of yellow pigment is an essential quality marker of raw materials for pasta manufacturing. It is defined as the extractable content of carotenoids of the endosperm. It is defined as milligram β -Carotene per 100-gram dry substance.

The yellow pigment content in wheat products is determined after extraction of the sample followed by photometric measurement at 440 nm.

This method is based on the official methods EN ISO 11052 ^[1] and the German Food and Feed Code §64 LFGB 16.01-3 ^[2] and describes the determination of the yellow pigment content in durum wheat flour and semolina.

Measuring range

Method 2541

Yellow Pigment EN ISO 11052

0.000 – 1.250 mg/100g

Sample material

Durum wheat flour and semolina



Instruments, 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
101990	n-Butanol for analysis EMSURE [®]
100921	Diethyl ether for analysis EMSURE [®]
PHR1239	β -Carotene Pharmaceutical Secondary Standard; CRM

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

Additional needs

- Grinder
- Drying cabinet
- Erlenmeyer flasks 100 mL, 200 mL
- Funnel
- Folded filter
- Watch glass
- Volumetric flasks 100 mL, 250 mL
- Platinum dishes, 80 mm diameter
- Graduated cylinders, 25 mL
- Volumetric flasks, 10 mL, 50 mL, 100 mL
- Standard laboratory glassware (e.g. glass beakers) and pipettes
- Analytical balance

Preparing the solutions

- **n-Butanol, water saturated:**
The solution must be prepared according to EN ISO 11052 ^[1] resp. German Food and Feed Code §64 LFGB 16.01-3 ^[2]

Procedure

Sample Preparation

- Sample material with particle size > 0.5 mm needs to be ground with a grinder

Determination of moisture content

- Follow the procedure according to ISO 712 ^[3].
- Note the moisture content

Extraction

- Place 10 g of the prepared sample into a 200 mL Erlenmeyer flask and follow the extraction procedure according to EN ISO 11052 ^[1] resp. German Food and Feed Code §64 LFGB 16.01-3 ^[2]
- Note the sample weight

Reagent blank

- n-Butanol, water saturated

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. 2541 „Yellow Pigment EN ISO 11052“.
- 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 gram (g), accurate to 0.001 gram (g), confirm with **<OK>**.

Note

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

- A window with an input field to enter the moisture content pops up.
- Enter the moisture content of the sample in % accurate to 0.01 %), confirm with **<OK>** and click on **<START>** to switch to the measurement procedure.

Note

It is possible to enter a moisture content in a range of 0.00 to 100.00 %

- 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/100 g 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:

Yellow pigment [mg/100 g]

Absorption of reagent blank A_{RB}

Absorption of sample A_{Sample}

Method control

- The method can be checked using Cat. No. PHR1239 β -Carotene Pharmaceutical Secondary Standard; Certified Reference Material
- Prepare a stock solution of 25.0 mg β -Carotene in 100 mL **Diethyl ether** in a 100 mL volumetric flask (= 250 mg/l β -Carotene).
- Dilute this solution to 1.0 mg/l β -Carotene. Place 1.00 mL of the stock solution into a 250 mL volumetric flask and fill up to the mark with **n-Butanol**, water saturated.
- Measure this solution versus a reagent blank as described in the section „Measurement“. Hereby enter a sample weight of 10 g and a moisture of 0 %.

Note

Due to the different sample preparation procedure and determination procedure of the 1.0 mg/l β -Carotene standard solution (no extraction step) compared to a sample analysis (extraction) 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/100g] * 2

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

F2 = 5 = Factor sample extraction for real sample (dilution through extraction)

Adjustment

- In case of significant deviations in the method control procedure the preprogrammed factor of 5.612 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 2541 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 2541 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. Durum wheat flour and semolina – Determination of yellow pigment content; ISO 11052:1994
2. German Food and Feed Code §64 LFGB 16.01-3:2007 Bestimmung des Gehaltes an gelben Pigmenten von Hartweizenmehl und Hartweizengrieß
3. Cereals and cereal products - Determination of moisture content - Reference method; ISO 712:2009

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