

Saccharine in table-top Sweeteners

(according to EN 1376 and German Food and Feed Code §64 LFGB 57.22.99-2)

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 EN 1376 [1] and German Food and Feed Code §64 LFGB 57.22.99-2 [2].

Method

Sweeteners like saccharin are often used in calorie-reduced food and beverage products due to their low nutritional value whilst having at the same time a high sweetener power. Saccharine is the oldest and best-known sweetener with a sweetener power of approx. 200 – 400 times of sucrose. The chemical name for saccharine is Benzoic sulfimide [3].

The Saccharin content is determined photometrically in the absorbance maximum at 270 nm. This method is based on the official method EN 1376 [1] and German Food and Feed Code §64 LFGB 57.22.99-2 [2] and describes the determination of Saccharine in table-top sweeteners.

Measuring range

Description		
Method 2536	Saccharine EN 1376	0.0 – 1200.0 mg/g

Sample material

- Table-top sweeteners



Reagents and auxiliaries

Cat. No.	Description
1.73027	Spectroquant® UV/VIS Spectrophotometer Prove 300 plus
1.73028	Spectroquant® UV/VIS Spectrophotometer Prove 600 plus
100784	Rectangular cells quartz 10 mm
814114	Saccharin sodium salt dihydrate for synthesis (≥99.0%)
109141	Sodium hydroxide solution 0.1 mol/l (0.1N) Titripur®

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

Additional needs

- Mortar and pestle
- Analytical balance
- Drying cabinet (optional)
- Volumetric flasks, 50-mL, 100-mL, 250-mL, 500-mL
- Standard laboratory glassware (e. g. glass beakers) and pipettes
- Analytical balance.

Sample preparation

- Pulverize sample finely in a mortar

Procedure

Saccharine determination

Reagent blank

Sodium hydroxide solution 0.1 mol/l (0.1 N).

Sample

Weigh pulverized sample to a volumetric flask and dissolve it according to EN 1376 ^[1] resp. German Food and Feed Code §64 LFGB 57.22.99-2 ^[2].

Note

The sample weight should approx. be equivalent to an amount of 30 mg Saccharin sodium salt resp. 35 mg Saccharin sodium salt dihydrate.

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. 2536 "Saccharine EN 1376".
- 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 milligram (mg), accurate to 0.1 milligram (mg), 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 1.0 to 3000.0 mg.

- 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/g, the absorption for the reagent blank (A_{RB}) and the sample (A_{Sample}) and the ratios A_{255}/A_{270} and A_{285}/A_{270} for the sample measurement from the display.
- Tap the <START> button to start the measurement procedure for the next sample.

Note

The absorption ratios A_{255}/A_{270} and A_{285}/A_{270} for the sample measurement are useful to check the plausibility of the measurement. With these ratios it is possible to identify if the measurement is influenced by interfering substances.

Typical values for pure saccharin sodium salt in Sodium hydroxide solution 0.1 mol/l are

Ratio $A_{255}/A_{270} = 0.83$

Ratio $A_{255}/A_{270} = 1.55$

For further details see also EN 1376 ^[1] resp. German Food and Feed Code §64 LFGB 57.22.99-2 ^[2].

Evaluation

Statement of the results:

- Saccharine-Na [mg/g]
- Absorption of reagent blank A_{RB}
- Absorption of sample A_{Sample}
- Ratio A_{255}/A_{270}
- Ratio A_{285}/A_{270}

Note

- To recalculate the result to mg/g Saccharine sodium salt use recalculation factor 1.175.
- To recalculate the result to mg/g Saccharine use recalculation factor 0.893.

Method control

- The method can be checked using **Cat. No. 814114** Saccharin sodium salt dihydrate for synthesis (≥99.0%) as standard substance
- Prepare a stock solution with 1000 mg/l **Saccharin sodium salt** by dissolving 280.0 mg **Saccharin sodium salt dihydrate** in approx. 220 mL Sodium hydroxide solution 0.1 mol/l. Transfer the solution completely to a 250-mL volumetric flask and fill up to the mark with Sodium hydroxide solution 0.1 mol/l.
- Dilute the stock solution to 120 mg/l **Saccharin sodium salt** with Sodium hydroxide solution 0.1 mol/l (30 mL stock solution ad 250 mL in a 250-mL volumetric flask).

- Analyze the prepared standard as described in section “Measurement”.
- Hereby enter a sample weight of 30.0 mg.
- The target value is 1000 mg/g

Adjustment

- In case of significant deviations in the method control procedure the preprogrammed factor 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 value standard)

- To edit the preprogrammed factor, select method 2536 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 2536 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”.

For more information visit,
SigmaAldrich.com/photometry

Literature

1. Foodstuffs - Determination of saccharin in table top sweetener preparations - Spectrometric method; EN 1376:1996.
2. German Food and Feed Code §64 LFGB 57.22.99-2:1998 Bestimmung von Saccharin in Tafelsüßen
3. Matissek R., Steiner G., Fischer M.; (2010): Lebensmittelanalytik, 4. Auflage, Springer-Verlag, Berlin Heidelberg.

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