Supelco®
Analytical Products

Application Note

Sulfite in Dried Fruits: Reflectometric Determination After Reaction with Potassium Hexacyanoferrate, Zinc Sulfate, and Sodium Nitroprusside

Introduction

This application note provides a comprehensive overview of the reflectometric determination of sulfite levels in dried fruits utilizing the Reflectoquant® Sulfite Test. Sulfites are frequently employed as preservatives in dried fruits to enhance color retention and prevent spoilage. However, their presence can pose health risks to sensitive individuals, making precise measurement crucial for ensuring food safety and meeting regulatory standards. In the EU, the permissible sulfite levels in dried fruits are specified in the EU Food Additives Regulation (EC) No 1333/20081, which details the allowable limits for these additives in products intended for direct consumption.

The procedure detailed in this note encompasses sample preparation and analysis using the Reflectoquant® Sulfite Test strip in conjunction with the portable RQflex® 20 reflectometer. It also includes calculations for determining sulfite content in mg/kg. This method offers a rapid, cost-effective, and reliable approach to assessing sulfite concentrations while also allowing for comparisons with traditional titrimetric and photometric methods to ensure the accuracy and validity of results.

By adhering to the outlined procedure, users can effectively monitor sulfite levels in dried fruits, thereby enhancing food safety practices and protecting consumer health.

Experimental

This application note describes the reflectometric determination of sulfite in dried fruits.

Method

Sulfite ions react with a mixture of potassium hexacyanoferrate(II), zinc sulfate, and sodium nitroprusside to form a red compound that is determined reflectometrically.

Measuring range

Test kit: 10–200 mg/L Sulfite

Method: 0.05–4.00 g/kg Sulfite (considering the dilution factor of 5 resp. 20)

Applicable Sample

Dried fruits, e.g. dried apple, dried apricot

Influence of Foreign Substances

Foreign substances in the sample solution can

- increase the measurement value because of an amplification of the reaction
- lower the measurement value because of a prevention of the reaction

A quantification of these effects is stated in tabular form in the respective package insert for the most important foreign ions and substances. The tolerance limits have been determined for the individual ions and substances; they may not be evaluated cumulatively.

In the case of samples with a complex, in many cases inexactly known composition (matrix) it is particularly difficult to estimate the potential influence of the foreign substances on the analysis (matrix effect). The following instructions describe a method by means of which the user can test whether a matrix effect is present or not.

- **Interference check by dilution**
- **Interference check by spiking**

Reagents, Instruments, and Materials

Test/Reagents Kit(s)

- Sulfite Test, Reflectoquant® **(1.16987)**

Instrument(s) & Devices

- Reflectometer RQflex® 20, Reflectoquant® **(1.17246)**

MilliporeSigma

Other Reagents and Accessories

- Water suitable for UV/Vis spectroscopy **(1.16754)** or distilled water
- Sodium hydroxide solution, c(NaOH) = 1 mol/L (1 N), Titriplex® **(1.09137)**
- Hydrochloric acid 1 mol/L Titripur® **(1.09057)**
- pH-indicator strips, pH 0-14 Universal indicator **(1.09535)**

For analytical quality assurance:

- RQcheck set for RQflex® 20 Reflectometer **(1.17247)**
- Recalibration Set for RQflex® 20 Reflectometer **(1.16954)**
- Sodium sulfite anhydrous for analysis EMSURE® **(1.06657)**
- Titriplex® III GR for analysis **(1.08418)**
- Buffer solution pH 9.00 Certipur® **(1.09461)**

Analytical Procedure

Reagent Preparation

- 0.01 mol/L NaOH solution
- Pipette 5 mL sodium hydroxide solution 1 mol/L into a 500 mL measuring flask. Fill up to the mark using distilled water or water suitable for UV/Vis spectroscopy.

Sample Preparation

- Weigh approx. 10 g of chopped sample exactly into an Erlenmeyer flask.
- Add 0.01 mol/L sodium hydroxide solution to the sample depending on the expected sulfite content according to the dilution table below.

Table 1. NaOH volume and dilution factor for expected sulfite concentration

Expected concentration [g/L SO_3^{2-}]	Recommended amount of 0.01 mol/L NaOH	Dilution factor
0.05–0.80	50 mL	5
0.80–4.00	200 mL	20

Note: The suggested quantity of sodium hydroxide solution may need to be adapted depending in the swelling properties of the sample to be analyzed.

- Slowly stir or shake gently for 5 minutes.
- If necessary, adjust the pH to 9–10 using sodium hydroxide solution 1 mol/L or hydrochloric acid 1 mol/L
- Allow the sample to be deposited and use the supernatant for further analysis.

Measurement

- Press the START button of the reflectometer and this is imperative - at the same time immerse both reaction zones of the test strip in the pretreated sample (5–30 °C) for 2 sec.
- Carefully allow excess liquid to run off via the long edge of the strip onto an absorbent paper towel.
- Immediately insert the strip all the way into the strip adapter with the reaction zones facing the display.
- After the end of the reaction time (30 s), read off the result from the display in mg/L SO_3^{2-}. The result is automatically stored.

Notes on the measurement:

- If the measurement value exceeds the measuring range (HI is shown on the display), repeat the measurement using fresh, with sodium hydroxide diluted samples until a value of less than 200 mg/l SO_3^{2-} is obtained. Concerning the result of the analysis, the dilution (see also section calculation) must be taken into account.
- If the test strip is inserted into the adapter after the reaction time has expired, renewed depression of the START button may produce a false result.

Calculation

The result in g/kg sulfite or g/kg sulfur dioxide is calculated using the following calculation formula:

$$\text{Sulfite content [g/kg]} = \frac{\text{Measured value [mg/L]} \cdot \text{Vol.NaOH [mL]}}{\text{Weight of sample [g]} \cdot 1000}$$

$$\text{Sulfur dioxide [g/kg]} = \frac{\text{Measured value [mg/L]} \cdot \text{Vol.NaOH [mL]}}{\text{Weight of sample [g]} \cdot 1280}$$

Analytical Quality Assurance

Analytical quality assurance (AQA) is recommended before each measurement series. Check the instrument using the RQcheck. If RQcheck failed, perform a recalibration using the recalibration set and repeat the RQcheck. For more details see RQflex® 20 manual.

To check test strips, measurement device, and handling (recommended before each measurement series):

- Dissolve 0.157 g of anhydrous sodium sulfite and 0.040 g of Titriplex® III in distilled water, make up to 100 mL with distilled water, and mix. SO_3^{2-} content: 1000 mg/L.
- Take 10.0 mL of this solution, add 10 mL of buffer solution pH 9.00, make up to 100 mL with distilled water, and mix. Subsequently analyze as described in section 7. The content of SO_3^{2-} determined should be 100 mg/L.

Results and Discussion

Here in this study, three different dried fruits were used as test samples for measurement and comparison. The results were verified using titration and photometry as orthogonal method to Reflectoquant® Test kit. The results are shown in the following table.

Table 2. Results - comparison with titrimetric and photometric method:

Sample	Reflectoquant® [mg/kg]	Titration [mg/kg]	Photometry [mg/kg]
Apricot 1	1280	---	1310
Apricot 2	2520	2450	2520
Apple	450	396	405

Conclusion

- Rapid testing of sulfite content in dried fruits can be performed with Reflectoquant® Sulfite Test with RQflex® 20 reflectometer in a quick and easy way.
- Orthogonal methods using titration and photometry has provided evidence that experimental results of the rapid test are comparable to titration and photometry.

References

1. European Commission. (2008). Regulation (EC) No 1333/2008 on food additives. Official Journal of the European Union.

Featured Products

Description	Cat. No.
Instruments & Test Strips	
Sulfite Test Method: reflectometric with test strips 10–200 mg/L SO_3^{2-} Reflectoquant®	**1.16987**
Reflectometer RQflex® 20 Reflectoquant®	**1.17246**

For more information visit us at
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