

Determination of Low Sulphur content according to modified Monier-Williams method

Reference: Internal Method – Sulphites in Foods – Modified Monier-Williams Method

Tested with **VELP Scientifica UDK Distillation Units**



Introduction

Sulphites agents are compounds that are capable to release sulphur dioxide SO₂, which helps to preserve the food. Sulphites were used by many ancient cultures like Romans or Greeks to prevent spoilage, discoloration of food, to sanitize wine vessels, etc. Their use became an issue of concern when certain sensitive individuals exhibited adverse reactions as asthmatic reactions. For this reason, the European Union and other countries have regulated the use of these compounds in food and beverages.

Sulphur dioxide Determination

One of the most commonly used methods for this analysis is the **sulphur dioxide distillation from complex matrixes followed by iodine titration**. This steam distillation performed with an automatic apparatus shortens the required time considerably and ensures reliable results.

The described method shows comparable results to the Optimized Monier-Williams method (OMW).

Chemicals

- Starch solution
- Iodine solution 0.005 N
- Hydrochloric acid (HCl) 33 %

Sample

Sample FAPAS	Dehydrated coconut
Assigned value	40.0 mg/kg
Satisfactory range	38.48 – 41.52 mg/kg

Sample FAPAS	White Beans
Assigned value	30.0 mg/kg
Satisfactory range	29.13 - 30.87mg/kg

Standard FAPAS	Seedless Raspberry Jam
Ref. Number	T27188QC
Assigned value	49.3 mg/kg
Satisfactory range	47.43 - 51.17 mg/kg

Sample Preparation

Homogenize the sample avoiding unnecessary exposure to air. Weigh sample quantity depending on the expected quantity of SO₂ into Test tube Ø80x300 mm, 1000ml (cod. A00001083).

Expected SO ₂ ppm	Sample g
0 - 50	15
50 - 200	10
200 - 500	5
>500	2

If the SO₂ content is unknown, weigh 10 g of sample.

Distillation and Titration

Prepare the receiving solution in a 250 ml Erlenmeyer flask, adding 75 ml of distilled water, 10 drops of starch solution and 1 ml of iodine solution 0.005 N. The receiving solution color is light blue.

Distill the samples according to the following parameters set in a customizable method:

- H₂O (dilution water): 50 ml
- HCl (33%): 30 ml*
- Distillation time: 6 minutes
- Steam power: 50 %
- At the end of the distillation, avoid automatic distillation residues discharge.

* added manually. It is possible to use lower HCl concentration adding it with Acid pump kit.

Prepare some blanks with all chemicals and without the sample.

Titrate the distillate with iodine solution 0.005 N in order to get the solution back to light blue, when SO₂ arrives, the solution turns colorless and the color persists for about 30-40 seconds.

It was found that using always the same amount of iodine reagent (1 ml) in the starting solution, titration is facilitated and accuracy is increased.

Typical Results

Record the ml of titration solution for the calculation using the formula:

$$\text{mg/kg SO}_2 = \frac{(\text{ml sample} - \text{ml blank}) * M_{\text{SO}_2} * N * 1000}{m_{\text{sample}} * 2}$$

ml sample: titrant volume used for sample
 ml blank: average of titrant volume used for blanks
 M_{SO₂}: SO₂ molecular weight (64.06 g/mol)
 N: normality titrant solution (0.005 N)
 m_{sample}: sample quantity (g)

Dehydrated Coconut

Sample quantity (g)	ml Titrant	mg/kg SO ₂
40.10	10.00	39.9
41.16	10.10	39.3
40.15	10.35	41.2
Average ± SD%		40.13 ± 0.79

Expected SO₂ range: 38.48 – 41.52 mg/kg

White beans

Sample quantity (g)	ml Titrant	mg/kg SO ₂
56.20	10.90	31.0
55.25	10.55	30.6
55.12	10.25	29.8
Average ± SD%		30.47 ± 0.50

Expected SO₂ range: 29.13 – 30.87 mg/kg

Seedless Raspberry Jam

Sample quantity (g)	ml Titrant	mg/kg SO ₂
5.06	1.55	49.0
5.07	1.60	50.5
5.12	1.55	48.4
Average ± SD%		49.3 ± 0.88

Expected SO₂ content: 47.23 – 51.17 mg/kg

The complete procedure was verified by dosing 10 ml of sodium bisulphite standard solution containing 23.57 mg SO₂.

Conclusion

The obtained **results are reliable and reproducible** in accordance with the expected values meaning **high repeatability** of the results.

The described method shows comparable results to the Optimized Monier-Williams method (OMW).

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VELP solutions

Tested with:

UDK 149 Automatic Kjeldahl Nitrogen Protein Analyzer

Other VELP UDK Series solutions:

UDK 129 Distillation Unit

UDK 139 Semi-Automatic Kjeldahl Distillation Unit

UDK 159 Automatic Kjeldahl Nitrogen Protein Analyzer

UDK 169 Automatic Kjeldahl Nitrogen Protein Analyzer with Autosampler connection

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