Application VAPODEST

C.8.1 Sulphur Dioxide in Food - Alkalimetric method

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1 Principle

Sulphur dioxide and sulphites are popular preservatives and antioxidants. They are used in numerous food groups. The Acceptable Daily Intake (ADI) is 0.7 mg sulphur dioxide equivalent per kilogram of body weight per day. Due to the allergenic effect, labelling is mandatory in the European Union for concentrations of 10 mg/kg and above. Different limit values are set for different food groups.

All the sulphur dioxide is distilled out by adding phosphoric acid. With the help of the water steam, the gaseous sulphur dioxide is distilled into the hydrogen peroxide receiver. In the process, sulphuric acid is formed.

$$H_2O_2 + SO_2 \rightarrow H_2SO_4$$

The amount of sulphuric acid is determined by titration using sodium hydroxide. The SO_2 content is directly related to the sulphuric acid formed.

$$H_2SO_4 + 2\,NaOH \rightarrow Na_2SO_4 + 2\,H_2O$$

The analysed sample should not contain volatile compounds, such as volatile acids. Otherwise, please have a look at our applications D.2.7 or C.8.2.

2 Method

This application note is meant to be a guideline for the operation of your C. Gerhardt analysis system and has to be adapted to your sample matrix and the local circumstances in your laboratory.

This document is based on:

- DIN EN 1988-1:1998-05, Lebensmittel - Bestimmung von Sulfit - Teil 1: Optimiertes Monier-Williams-Verfahren
- AOAC 990.28, Sulfites in Foods – Optimized Monier-Williams Method, 1994

3 Chemicals and material

Quality grade p. a.

3.1. Water: demineralised or distilled
3.2. Paper weighing boats, weighing paper (Art. 1004939)
3.3. Phosphoric acid, w(H_3PO_4) = 60 %
3.4. Hydrogen peroxide, w(H_2O_2) = 3 %
3.5. Methyl red indicator
3.6. Sodium hydroxide, c(NaOH) = 0.01 mol/l
3.7. Sodium disulfite ($Na_2S_2O_5$) (standard substance)

4 Instruments

- Knife blender
- Analytical balance (accuracy 0.1 mg)
- VAPODEST steam distillation system
  VAPODEST 200 to 450 are without titrator. The titration has to be performed by means of a manual burette (class A, according to ISO 385), 50 ml nominal volume, with volume scale in 0.05 ml steps, or a titrator, with an indicator solution or with a pH electrode.
  The titration is performed automatically in case of VAPODEST 450 with external titrator or VAPODEST 550/550C with integrated titrator.

5 Procedure

5.1 Sample preparation

5.1.1 Solid samples

A representative sample quantity is grinded and homogenised.

The sample should be analysed as quickly as possible after grinding.

The sample is weighed in a weighing paper (3.2) and then put into in the tube. If necessary, the sample residues on the tube wall should be rinsed back into the tube using distilled water.

5.1.2 Liquid samples

The sample should be as representative and homogeneous as possible. A sufficient volume is pipetted or weighed into the tube.

The sample quantity is based on the sulphur dioxide content. The following quantities are recommended:

SO_2 content [mg/kg] or [mg/l]	ceq(NaOH) [mol/l]	Sample weight [g] or [ml]
≤ 10	0.01	≥ 50
10 - 20		50
20 - 50		25
50 - 100		15
100 - 200		10
200 - 500		5
500 - 1500		2
≥ 1500		1

It is recommended to use BS-400 digestion tubes (Art. 12-0308), especially for high sample weights.

The phosphoric acid is automatically added by the VAPODEST. Otherwise, 30 ml phosphoric acid (3.3) are added manually before that the tube is clamped in the VAPODEST. If the phosphoric acid is added manually, the inlet tube of the reagent must be closed and the distillation is performed immediately afterwards to avoid loss of sulphur dioxide.

For the automatic addition of phosphoric acid with the VAPODEST 200 to 450, an acid-resistant pump must be installed. Please contact your service technician.

5.2 Distillation

The VAPODEST unit has to be put into operation according to the operating instructions. The following program parameters are recommended for the different unit versions of the VAPODEST. These can only serve as guidelines for the analysis and must be adapted to your own conditions if necessary.

		VAP 200	VAP 300	VAP 400	VAP 450	VAP 550 / 550C
H_2O Addition	100 ml	•	✓	✓	✓	✓
Reagent Addition **Phosphoric acid (3.3)**	30 ml	✓	✓	✓	✓	✓
Reaction time	0 s	✓	✓	✓	✓	✓
Distillation time	360 s	✓	✓	✓	✓	✓
Steam power	100 %	✓	✓	✓	✓	✓
Sample suction*	30 s	-	✓	✓	✓	✓
Receiver Addition **Hydrogen peroxide (3.4)**	80 ml	•	•	✓	✓	✓
Suction receiver solution	30 s	-	-	-	✓	✓
Titration	-	•	•	•	✓	✓
Calculation	-	•	•	•	•	✓
Reading pH value, fixed enpoint or automatic endpoint	-	•	•	•	•	✓

✓ = Automatic • = Manual - = Not specified

*Note: Depending on the sample type, the sample tube must be emptied manually if it contains solids.

The opening of the outlet tube of the distillation apparatus must be immersed in the receiver solution. At the end of the distillation, the total volume of distillate should be approx. 250 ml.

Two methods are saved in the VAPODEST 550/550C for the sulphur dioxide determination. Only the unit of the sample amount is different (g or ml).

5.3 Titration

3 - 4 drops of methyl red indicator (3.5) are first added. Then, sodium hydroxide (3.6) is added until the colour changes from red to yellow. If the end point is determined with a pH meter or a titrator, the addition of the indicator is omitted. In this case, the distillate is titrated to a fixed end point, for instance pH 4.5.

It is important that the pH-endpoint is higher than the pH reached after the distillation of a blank.

5.4 Blank value

For the blank value determination, perform the analysis just with the indicated chemicals.

5.5 Performance check

To check the analytical performance of the water steam distillation system, the analysis of a standard with a know content of sulphur dioxide is carried out.

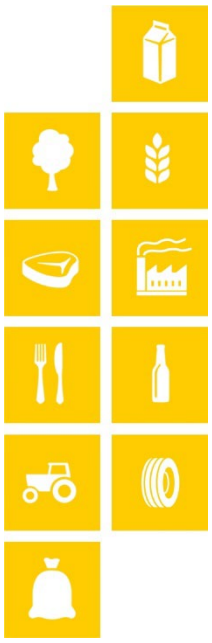
For this purpose, a standard solution of sodium disulfite with 100 ppm SO_2 (3.7) is prepared. 0.1484 g of sodium disulfite (3.7) are weighed out and dissolved in 1000 ml of distilled water. The solution should always be prepared fresh. An aliquot is taken on the basis of the measuring range. 10 ml contain 1 mg SO_2. The recovery rate should be at least 80 %.

6 Calculation

The mass fraction of SO_2, expressed in mg/kg, is calculated as follows:

$$\omega = \frac{32{,}03 * (V_1 - V_0) * 1000 * c_{eq} * t}{m}$$

ω = Mass fraction of SO_2 in the sample [mg/kg]
32,03 = Milliequivalent weight of SO_2 [g/mol]
V_1 = Volume of standard solution used for the sample [ml]
V_0 = Volume of standard solution used for the blank test [ml]
1000 = Factor to convert milliequivalents to microequivalents
c_{eq} = Concentration of the standard solution [mol/L]
t = Titre of the standard solution
m = Weight of the sample [g]



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- Nitrogen in food and feed samples according to Kjeldahl and Dumas
- Crude fibre, ADF and NDF in feed
- Fat in food and feed
- Alcohol determination
- Total cyanide in water
- Trace metal in soil and sludge
- COD determination in water
- Total nitrogen determination in water, soil and plants
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