Application VAPODEST

C.8.2 Sulphur Dioxide in Food – Iodometric 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.

The iodometric method is used for samples with other water vapour volatile components, such as volatile acids. All sulphur dioxide is expelled by adding acid. The gaseous sulphur dioxide is distilled into the sodium hydroxide receiver through the water steam and bound as sodium sulphite.

$$SO_2 + 2\,NaOH \rightarrow Na_2SO_3 + H_2O$$

The distillate is acidified and a defined iodine solution is added. The excessive iodine is titrated back with a sodium thiosulphate solution.

$$SO_3^{2-} + I_2 + H_2O \rightarrow SO_4^{2-} + 2\,I^- + 2\,H^+$$

$$I_2 + 2\,S_2O_3^{2-} \rightarrow 2\,I^- + S_4O_6^{2-}$$

(only in neutral or slightly acidic solution)

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 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 Sodium hydroxide solution, c(NaOH) = 0.1 mol/l (or 0.5 mol/l depending on the content ranges of the sample)
3.5 Hydrochloric acid, w(HCl) = 5 %
3.6 Iodine solution c(I_2/l) = 0.05 mol/l
3.7 Starch solution: 500 ml H_2O + 1 g soluble potato starch, boil for 5 minutes, then filter and cool
3.8 Sodium thiosulphate solution c($Na_2S_2O_3$) = 0.1 mol/l
3.9 Sodium metabisulphite ($Na_2S_2O_5$) (standard substance)
3.10 Pipette, different volumes

4 Instruments

- Knife blender
- Analytical balance (accuracy 0.1 mg)
- VAPODEST steam distillation system
  VAPODEST 200 - 400. The titration is carried out with a manual burette (class A, according to ISO 385), 50 ml nominal volume, with volume scale in 0.05 ml steps.

5 Procedure

5.1 Sample preparation

5.1.1 Solid sample

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 (3.1).

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.

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

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

For the automatic addition of phosphoric acid with the VAPODEST 200 to 400, 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
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	-	-	-	-
Suction receiver solution	-	-	-	-
Titration	-	-	-	-
Calculation	-	-	-	-
Reading pH value, fixed enpoint or automatic endpoint	-	-	-	-
Titration online	-	-	-	-

✓ = Automatic • = Manual - = Not specified

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

The distillate is collected in a 300 ml Erlenmeyer flask into which 5 ml of the sodium hydroxide solution (3.4) and 50 ml of distilled water (3.1) are placed.

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 the distillate should be approx. 250 ml.

Note: For some samples it may be useful to replace the phosphoric acid with 5 ml hydrochloric acid (w = 5%).

5.3 Titration

After completion of the distillation, acidify the distillate with approx. 5 ml hydrochloric acid (3.5) (check pH) and add 10 ml of the iodine solution (3.6). In addition, add a few drops of the starch indicator (3.7). Then titrate with sodium thiosulphate solution (3.8) until the solution is colourless. Since this is an indirect titration, the blank value must be determined beforehand.

5.4 Blank value

The iodometric titration is an indirect titration. This means that the consumption of the iodine solution is not determined. Instead, a certain amount of iodine is added at the beginning and the remaining iodine is determined at the end. Therefore, the consumption of the sodium thiosulphate solution of the blank sample, which does not contain sulphur dioxide, must be determined. The amount of iodine consumed is then the difference between the blank value and the amount of sodium thiosulphate solution consumed in the sample.

5.5 Performance check

To check the analytical performance of the water steam distillation system, the recovery rate of a standard solution of sodium metabisulphite with 100 ppm SO_2 is determined. For this purpose, 0.1484 g sodium metabisulphite (3.9) is weighed in and dissolved in 1000 ml distilled water (3.1). The solution should always be prepared freshly. Take an aliquot 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 w of SO_2, expressed in mg/kg, is calculated as follows:

$$\omega = \frac{64{,}06 * (V_0 - V_1) * 1000 * c}{m * 2}$$

ω = Mass fraction of SO_2 in the sample [mg/kg]
64,06 = Molar mass sulphur dioxide [g/mol]
V_0 = Volume of sodium thiosulphate solution of the blank value [ml]
V_1 = Volume of sodium thiosulphate solution for the sample [ml]
c = Concentration of the sodium thiosulphate solution [mol/l]
m = Sample weight [g]

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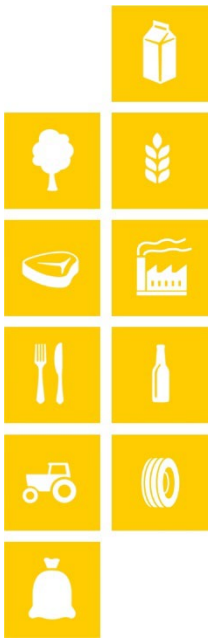

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