

Kjeltec™ 9 Distillator

Determination of SO₂ (Sulphite) in Food and Beverages with Steam Distillation Method according to AOAC 962.16^[1]



FOSS Kjeltec™ 9 Distillator

Kjeltec 9 is an automated distillation unit designed primarily for protein analysis following the Kjeldahl method but can be widely used for many different distillation processes. The solution is perfect for laboratories running a low to medium volume of daily samples and using many different distillation methods.

Sample Types

The method described in this Application Note covers the following sample types:

- Liquid samples: wine, soft drinks, fruit juice, beer, must
- Solid and semi-solid samples: dried apricot, celery, jams, syrups, sausages, burgers

Detection limit: 5mg/L

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1 Application Information

1.1 Introduction

SO₂ is used as a preservative in the food industry, especially in beverages such as fruit juice and wine and dried fruit and vegetables. Due to severe health risks, the maximum amount of SO₂ is limited by law. The EC guidelines require labeling SO₂ at concentrations no more than 10 mg/kg or 10 mg/L.

This Application Note guides using Kjeltex 9 Distillator to analyse SO₂ in food and beverages. This method is modified according to AOAC method 962.16^[1] and 990.28^[2]. It also conform to 2676/EEC^[3] and OIV-MA-AS323-04A : R2012^[4].

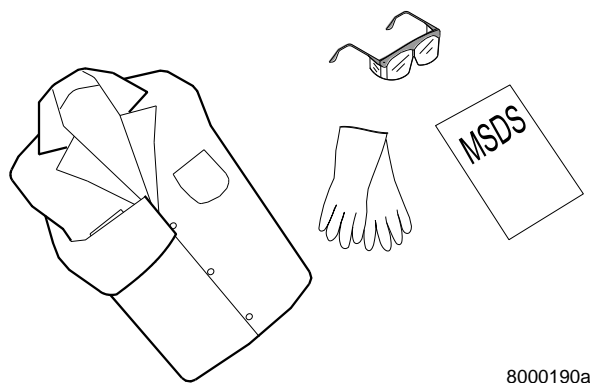
1.2 Principle

Total sulphur dioxide is liberated by acidic steam distillation and is fixed and oxidized by hydrogen peroxide. The sulphuric acid formed is determined by titration with standardized sodium hydroxide.

1.3 Safety Precautions

Personal protection equipment (PPE) such as gloves, a lab coat, and eye protection should always be worn while handling phosphoric acid, hydrogen peroxide, and sodium hydroxide.

Follow safety instructions of instrument User Manual and Material Safety Data Sheets.



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Fig. 1 Personal protection equipment

1.4 Apparatus

- Milling, grinding equipment or Homogenizer
- Analytical balance, accurate to 0.1 mg
- FOSS Kjeltex 9 Distillator

Note: If sulphite determinations are commonly performed, the silicone tubing from the splash head to the condenser should be replaced with Viton tubing (article number 1582-0001)

- 750 ml digestion tube

- E. Titration burette, 50 ml or equivalent
- F. Erlenmeyer flask (Receiver flask, up to 500 ml)

1.5 Reagents

During the analysis, use only reagents of recognized analytical grade.

- A. 60% w/v phosphoric acid
 - Weigh 1042 g of deionised water into a 4L plastic beaker. Add 2.5 L 85% w/v phosphoric acid.

Note: For safety and environmental reason, phosphoric acid can be replaced by 50% m/v citric acid.
- B. Titrant (Standardised) - 0.01 N or 0.05 N sodium hydroxide

Note: The normality of the titrant is required to 4 decimal places.
- C. Receiver solution - Hydrogen peroxide solution
 - To prepare the hydrogen peroxide solution, add 200 ml 35% H₂O₂ to 1 L distilled H₂O. Add 10 ml indicator solution (100 mg methyl red, 50 mg methylene blue in 100 ml ethanol)

Note: The receiver solution should be freshly prepared.

1.6 Analytical Details

Model	Dilution	60% H ₃ PO ₄	Steam Power	SAfE	Receiver Solution	Distillation End
Kjeltec 9 Distillator	50 ml	30 ml	90%	5s	30 ml H ₂ O ₂ solution	5 min, no tube empty

Table 1 General procedure for distillation

1.7 Procedure

1.7.1 Sample Preparation

1. Adjust sample to room temperature
 - For liquid samples, dissolved CO₂ is removed by shaking vigorously or using an ultrasonic bath for degassing.
 - For solid samples, cut the sample into small pieces with a sample size of 0.5 cm. Avoid unnecessary exposure to air. The sample must be prepared fresh before the test.

It is recommended to use 50 ml liquid sample in one digestion tube for the analysis.

1.7.2 Distillation and Titration

Note: Perform at least three reagent blanks before each batch of samples.

1. Pipette 50 ml of the liquid sample or weigh up to 10 g into a 750 ml distillation tube.

Note: Don't put the weighing paper into the digestion tube for distillation
2. Select the program profile
3. Load digestion tube into the instrument
4. Start to run a sample
5. Remove sample from the distillation unit and titrate the distillate with standardized NaOH (Colour change is from light purple to first hint of light green, note volume of titrant used).

Note: Check recovery rates with sodium sulphite (Na₂SO₃) as standard. Adjust the sample amount and concentration of titrant according to the sulphite concentration. For very low concentrations (< 20 mg/L), it is recommended to use 100 to 200 mL sample, then adjust the dilution to 0 ml.

1.7.3 Recovery Test

Note: Perform at least three reagent blanks before recovery samples.

1. Weigh up to 0.15 g ± 0.1mg Na₂SO₃ (99% Anhydrous Sodium Sulphite) into a 750 ml digestion tube.

Note: Do not dry sodium sulphite (Na₂SO₃) before the test; it can be used from the bottle directly.

2. Select the program profile
3. Load digestion tube into the instrument
4. Start to run a sample
5. Remove sample from the distillation unit and titrate distillate with standardized NaOH (0.01 N or 0.05 N with 4 decimal places)

Then the recovery can be calculated:

$$Recovery = \frac{100 \times (T - B) \times N \times 3.2/M}{SO_2 \text{ theoretical content}}$$

T = Volume of titrant for sample (ml)

B = Volume of titrant for blank (ml)

N = Normality of titrant

M = Weight of Na₂SO₃

SO₂ theoretical content = 50.829 × Purity (i.e. 99%)

1.8 Expression of Results

1.8.1 Calculation

Please calculate the results according to the below calculation:

$$ppm SO_2 = \frac{(T - B) \times N \times 32000}{\text{Volume (mL) or weight (g) of sample}}$$

T = volume of titrant for sample (ml)

B = Volume of titrant for blank (ml)

N = Normality of titrant

1.8.2 Method Performance

Samples

Three different sample types have been tested to check the performance of the Kjeltex 9 Distillator according to the method described in this application note. Below results are based on the test condition listed in the below table. The steam power used for distillation is 90%. The concentration of standard acid used for titration is 0.01 NaOH with 4 decimal places. The receiver solution is prepared fresh daily.

Sample Type	Sample Amount	Dilution (mL)	60% Phosphoric Acid (mL)	Receiver Solution (H ₂ O ₂ Solution, mL)	Distillation End
Dried apricot	10 g	50	30	30	5 min
Dried apple rings	10 g	50	30	30	5 min
Red wine	50 mL	50	30	30	5 min

Table 2 Analytical conditions for sample types used for the method performance data

Results for Distillator

The study was performed at FOSS using Kjeltex 9 Distillator and auto titrator. The study was performed using the procedure described within this Application Note.

Sample Type	Average Obtained Result (ppm SO ₂)	n	SD
Dried apricot	874.14	10	60.25
Dried apple rings	258.24	10	10.33
Red wine	158.16	10	1.89

Table 3 Performance data of Kjeltex 9 Distillator

1.9 References

- [1] AOAC Official Method 962.16 Sulfurous Acid (Total) in Food; Modified Monier-Williams Method
- [2] AOAC Official Method 990.28 Sulfites in Foods; Optimized Monier-Williams Method
- [3] 2676/EEC, Commission Regulation of 17 September 1990 Determining Community Methods for the Analysis of Wine
- [4] OIV-MA-AS323-04A : R2012 Compendium of international Methods of Wine and Must Analysis

2 Questions and Feedback

If you have questions or feedback concerning this Application Note, please contact your FOSS support team.

Joi Chen
Product Specialist, Service

3 Revision History

Rev.	Date of Issue	Revised Material	Approved by
1	2022-06-14	First issue.	JICH