

Sulfuric dioxide determination in food

KjelFlex K-360: sulfuric dioxide determination according to the national standard of China

The presented method for sulfuric dioxide (SO_2) determination is based on the chemical degradation of sulfites into volatile SO_2 . SO_2 is separated from the sample matrix by steam distillation, with the distillation unit KjelFlex K-360, into a lead acetate solution. The amount of sulfuric dioxide is determined by means of titration with an I₂ 0.005 M standard solution.

1. Introduction

Sulfuric dioxide is an important and universally permitted food preservative extensively used in the processing and preservation of foods. Foods that are commonly preserved using SO_2 or sulfites are fruits and vegetables, fruit juices and concentrates, syrups, wines, jams and to a lesser extent prawns, fish, minced meats, sausages, and mushrooms.

Nevertheless, due to the suspected adverse effects on human health, the residue level of SO_2 is subject to regulatory legislation requiring analytical methods to determine the levels present [1, 2].

Here, a reliable method for the determination of SO_2 in foods employing the automatic distillation unit KjelFlex K-360, according to the national standard of China GB/T5009.34 -2003 is presented.

2. Experimental

Equipment: KjelFlex K-360 (including acid resistant pump) with Metrohm 877 Titrino plus, Mixer B-400.

Samples: Commercial dry cherry tomato and hot peper powder.

Determination: The samples were homogenized with Mixer B-400. 5 g were weighted into a 500 mL sample tube and the method was verified by using a sodium sulfite stock solution.

Table 1: Method parameters for the distillation and titration with the KjelFlex K-360.

| KjelFlex K-360 | | Metrohm 877 titrin | o plus |
|--|------------|--------------------|----------------------------|
| H ₂ O volume | 0 mL | Pause | 20 s |
| NaOH volume | 0 mL | Titration solution | l ₂ 0.005 mol/L |
| Reagent (hydrochloric acid [1:1] or 85 % H₃PO₄) | 10 mL | Sensor type | Redox electrode |
| Reaction time | 5 s | Measuring mode | End point |
| Steam Power | 100 % | Titration rate | Optimal |
| Distillation time | 420 s | Stop Criterion | 2 mV |
| Tiration Start | 420 s | Stop volume | 100 mL |
| Titration type | Boric acid | Stop EP | 3 |
| Stirrer speed distillation | 7 | Stop Time | Off |
| Stirrer speed titration | 10 | Filling Rate | max. mL/min |
| | | | |

Before starting distillation, 25 mL lead acetate (conc. 20 g/L) solution was added into the receiver vessel and the sulfuric dioxide was released by the reaction of sample

with hydrochloric acid (1:1). Next, the sulfuric dioxide was distilled into the lead acetate solution by steam distillation. After distillation, 10 mL hydrochloric acid 37 % and 1 mL indicator were added to the receiving vessel solution before being titrated with an I_2 0.005 mol/L standard solution (see Table 1).

3. Results and comparison to BUCHI method

In Table 2, the sodium sulfite recoveries according to the Chinese standard are compared to the recoveries of the BUCHI method [4]. The sulfuric dioxide contents of the samples were determined according to the Chinese standard are listed in Table 3.

Table 2: The recovery results of SO_2 with sodium sulfite (n=4).

| Sodium sulfite | Recovery Chinese standard method [%] | Recovery BUCHI method [%] |
|----------------------|--|------------------------------|
| Sample 1 | 95.0 | 96.9 |
| Sample 2 | 94.1 | 95.0 |
| Sample 3 | 95.7 | 92.8 |
| Sample 4 | 97.3 | 98.4 |
| Average recovery [%] | 95.5 | 95.8 |
| RSD [%] | 1.2 | 2.2 |

Table 3: Determined sulfuric dioxide contents in samples (the result is averaged, n=3; rsd in brackets).

| Sample | SO ₂ [g/kg] |
|-------------------|------------------------|
| Dry cherry tomato | 4.59 (2.7 %) |
| Hot peper powder | 0.0269 (5.6 %) |

4. Conclusion

Determination of sulfuric dioxide in dry cherry tomato and paper powder was performed using the KjelFlelx K-360. According to Chinese standard of limits on the residual of sulfuric dioxide in different types of food, the content of SO_2 in dry cherry tomato is much higher than the residual of sulfuric dioxide limit [3]. The recovery for the sodium sulfite standard was above 95 % for both the BUCHI application notes and Chinese standard method [4,5]. Following the national standard of China, the KjelFlex K-360 is appropriate instrument for the distillation of SO_2 in foods.

5. References

[1] AOAC official method 990.28 Sulfites in Foods (1998).

[2] The National standard of China GB/T 5009.34 – 2003 Determination sulphite in foods.

[3] The National standard of China GB/T 2760 – 2014 National Food Safety Standard: Standards for Uses of Food Additives.

[4] BUCHI application note No. 090/2012 Determination of Total SO_2 in Certified Potato Powder Reference.

[5] BUCHI short note No. 169/2014 Sulfur dioxide determination in shrimps.

For more detailed information and safety conside-rations please refer to the Application Note No. 216/2015.