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DRAFT TANZANIA STANDARD

Methods for determination of organic preservatives in foodstuffs – Part 3: Sorbic acid and its salts

TANZANIA BUREAU OF STANDARDS

0. Foreword

For protecting food from microbial deterioration, a number of methods as application of heat or cold, dehydration, fermentation, irradiation or addition of certain chemicals are employed. Besides extending the periods of use of food a chemical preservative should be safe for human consumption, should not impart undesirable organoleptic changes, be economical in use and be capable of being analyzed. While the use of preservative to be safe under conditions of use is governed by law, it is considered necessary to prescribe methods for their analysis. The use of these methods would not only ensure repeatable and reproducible results for their correct interpretation, but would also facilitate inter-laboratory comparisons

There are two classes of preservatives, class I and class II. Class I preservatives include common salt, sugar, dextrose, glucose (syrup), wood smoke, spices, vinegar honey, etc. class II preservatives include inorganic substances such as sulphurous acid including salts thereof, nitrates of sodium or potassium and organic substances like benzoic acid including salts thereof, sorbic acids and including its sodium, potassium and calcium salts and sodium and calcium propionate.

This standard, covering the determination of organic preservatives, is being issued in three parts. This part (Part 3) covers the determination of sorbic acid and its salts in foodstuffs. The part 1 covers benzoic acid and its salts and part 2 covers propionic acid and its salts. In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with TZS 4

In the preparation of this Tanzania Standard assistance was drawn from IS 12014 (Part 3):1986, Methods for determination of organic preservatives in foodstuffs – Part 3: sorbic
acid and its salts published by Bureau of Indian Standards (BIS)

1.0 Scope

This standard prescribes the methods for determination of sorbic acid and its salts used as preservatives in foodstuffs

2.0 Normative references

2.1 The following referenced standards referred to in the text in such a way that some or all of their content constitutes requirements of this standards. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies

TZS 59: Water for analytical laboratory use – Specification and test method

TZS 4: Rounding off numerical values

TZS 672: Automotive gasoline (premium motor spirit)

3. Quality of reagents

3.1 analytical grade chemicals and distilled water conforming to TZS 59 shall be employed in tests.

4. General

4. 1 this standard specifies two methods for determination of sorbic acids and its salt, viz colorimetric and spectrophotometric methods. Colorimetric method is applicable to cheese and flour confectionary products only. Whereas the spectrophotometric method is applicable to fresh dairy products like cheese, sour cream, yoghurt and flour confectionary products

4.2 Principle

sorbic acids are isolated from food by extraction with diethyl ether and successive partitioning into aqueous NaOH and CH_2Cl_2. Acids are converted to trimethylsilyl (TMS) ester and determined by gas chromatography (GC). Caproic acid is used as internal standards for sorbic acid

5. Colorimetric Method

5.1 Reagents

5.1.1 Sulphuric Acid - 0.3N and 2N

5.1.2 Potassium Dichromate Solution - dissolve 147mg potassium dichromate in distilled water and dilute to 100mL

5.1.3 Thiobarbituric acid solution (0.5 percent) - dissolve 250mg thiobarbituric acid in 5mL of 0.5N sodium hydroxide solution in a 50mL volumetric flask by swirling under hot water. Add 20mL distilled water, neutralize with 3mL of 1N hydrochloric acid (HCl) and dilute to volume with distilled water. This solution should be freshly prepared before analysis

5.1.4 Crystalline magnesium sulphate - $MgSO_4.7H_2O$

5.1.5 Standard Sorbic Acid Solution - accurately weigh 134mg potassium sorbate (equivalent to 100mg sorbic acid) and dilute to 1 litre with distilled water. 1mL of solution corresponds to 0.1mg of sorbic acid. This solution is stable for several days when refrigerated

5.2 Equipment and apparatus

5.2.1 Steam distillation apparatus

5.2.2 Volumetric flask - 50mL and 1litre capacity

5.2.3 Pipette - 5mL and 20mL capacity.

5.2.4 Test Tubes - 15mL capacity.

5.2.5 Spectrophotometer with 10mm Cells

5.2.6 Flask - 250mL.

5.3 Procedure

5.3.1 Preparation of the Sample

5.3.1.1 Cheese- Cut the cheese very finely and mix thoroughly

5.3.1.2 Flour confectionary products

a) All types of bread and cakes not containing fruits - take 1 or half loaf of bread or cake and cut it into slices of 2-3 mm thickness. Spread the slices on the paper and let them dry in a warm place or at room temperature until sufficiently crisp and brittle to grind well. Grind entire sample to pass through 850-micron sieve, mix well and keep in an air-tight container before proceeding for analysis

b) Bread and cake containing raisins and fruits - proceed as in 5.3.1.2 (a) except comminute by passing twice through food chopper and dry the sample in an oven at 70˚C under pressure of less than 0.06666bars

5.3.2 Test portion - weigh 1.5 to 2.0g prepared sample into a distillation tube containing silicon chips. Add 10mL of 2N sulphuric acid and 10g magnesium sulphate. Steam distill the contents maintaining 20-30mL volume in distillation tube with small burner. Avoid charring. Collect 100-125mL distillate in 250mL volume flask within 45 minutes. Rinse condenser with distilled water and dilute the distillate to volume and mix thoroughly.

5.3.3 Determination - Pipette 2mL of test portion and 2mL of distilled water (for blank) into separate 15mL test tubes. Add 1mL of 0.3N sulphuric acid and 1mL of potassium dichromate solution and heat in a boiling water-bath exactly for five minutes. Immerse tube in icebath and add 2mL of thiobarbituric acid solution. Place it in boiling water-bath and boil it for 10 minutes. Cool and determine optical density of solution at 532nm against blank using matched 10mm cells.

5.3.4 Plotting of the calibrating curve - pipette 5, 10, 15, 20, 25mL of sorbic acid standard solution into separate 500 mL volumetric flasks. Dilute each to volume and mix thoroughly and proceed as prescribed in 5.3.3. Plot the optical density against µg sorbic acid/mL

5.3.5 Calculation - calculate the sorbic acid content in the sample after reading the corresponding sorbic acid value of the optical density.

$$\text{Percent sorbic acid in the sample} = \frac{125\ x\ A\ x\ 100}{M}$$

Where

A = Sorbic acid contents obtained from calibration curve, and

M = mass of sample taken

Percent of sodium sorbate= percent sorbic acid x 134

6. SPECTROPHOTOMETRIC METHOD

6.1 Reagents

6.1.1 Metaphosphoric Acid Solution – dissolve 5g phosphoric acid in 250mL distilled water and dilute to 1 litre with absolute ethanol

6.1.2 Mixed Ethers – petroleum ether and anhydrous diethyl ether (1:1)

6.1.3 Potassium Permanganate Solution - dissolve 15g potassium permanganate in distilled water, dilute to 100mL and filter through glass wool.

6.1.4 Sorbic Acid Solution (stock)- dissolve 200mg of sorbic acid in 200mL of mixed ether

6.1.4.1 working solution - dilute 10mL stock solution to 200mL with mixed ethers. This solution corresponds to 0.05mg/mL

6.1.5 Reference solution - shake 100mL mixed ethers with 10mL metaphosphoric acid solution and dry supernatant ether fraction with 5g anhydrous granular sodium sulphate and decant to obtain clear reference solution

6.2 equipment and **Apparatus**

6.2.1 High-Speed Blender

6.2.2 Separating Funnels - 500mL.

6.2.3 Volumetric Flasks

6.2.4 Graduated Pipette

6.2.5 Ultraviolet Spectrophotometer - provide with a 0.5mm monochromator with silica cells of thickness 20mm, fitted with ground lids

6.3 Procedure

6.3.1 Preparation of the Sample

6.3.1.1 cheese – cut the cheese sample very finely and mix thoroughly

6.3.1.2 Flour confectionary products

a) All types of bread and cakes not containing fruits - take 1 or half loaf of bread or cake and cut it into slices of 2-3 mm thickness. Spread the slices on the paper and let them dry in a warm place or at room temperature until sufficiently crisp and brittle to grind well. Grind entire sample to pass through 850-micron sieve, mix well and keep in an air-tight container before proceeding for analysis

b) Bread and cake containing raisins and fruits - proceed as in 5.3.1.2 (a) except comminute by passing twice through food chopper and dry the sample in an oven at 70°C under pressure of less than 0.06666bars

6.3.2 Test Portion – Accurately weigh 10g of the prepared sample in the high-speed blender cup. Add enough metaphosphoric acid solution to yield a total of 100mL liquid in mixture. Blend for 1 minute and immediately filter through Whatman No. 3 paper or equivalent filter paper with the same pore size. Transfer 10mL filtrate to 250mL separating funnel containing 100mL mixed ethers and shake for 1 minute. Discard aqueous layer and dry the ether extract with 5g anhydrous sodium sulphate.

6.3.3 Determination – place the ether solution in a silica cell with a ground lid of thickness 20mm and measure the absorbance of the solution at 250nm with respect to reference solution in a similar silica cell.

6.3.4 Plotting of the Calibrating Curve – into a series of a four 100mL volumetric flasks, add 1,2,4 and 6mL of working standard sorbic acid solution and dilute to volume with mixed ether. Determine absorbance of the solution at 250nm. Plot the absorbance against mg of sorbic acid/mL.

6.3.5 Calculation – calculate the sorbic acid content in the sample after reading the corresponding sorbic acid value from the calibration curve.

Percent sorbic in the sample

$$Percent\ sorbic\ acid = ({}^{mg\ sorbic\ acid}/_{g\ sample}) \times \left({}^{1}/_{1000mg}\right) \times 100$$

$$= \frac{mg\ sorbic\ acid}{10}$$

Percent sodium sorbate = percent sorbic acid x 1.34