SULFUR DIOXIDE (Iodometric)

PRINCIPLE

The sample is diluted and treated with sodium hydroxide to release sulfur dioxide. The solution is then acidified and the sulfurous acid determined by titration with a standard iodine solution using starch as an end point indicator.

SCOPE

This method applies to corn syrups, corn sugars and other water soluble starch hydrolyzates that contain sulfur dioxide. Use the low level procedure for sulfur dioxide levels below 20 ppm.

SPECIAL APPARATUS

1. Microburet: 5 mL capacity (Note 1) with 0.01 mL subdivisions and a tolerance of ± 0.01 mL. Fisher Catalog No. 20-105A, Fisher Scientific Company, 1600 West Glendale Avenue, Itasca, Illinois 60143.

2. Microburet: 10 mL capacity with 0.02 mL subdivisions and a tolerance of 0.02 mL. Fisher Catalog No. 20-105B, Fisher Scientific Company, 1600 West Glendale Avenue, Itasca, Illinois 60143

3. Stirring Apparatus: A magnetic stirrer is recommended.

REAGENTS

1. Sodium Hydroxide Solution, 1.5 *N*: Dissolve 60 g of reagent grade sodium hydroxide (NaOH) in 500 mL of purified water in a 1000 mL volumetric flask. Mix, cool and dilute to volume.
2. Sodium Hydroxide Solution, 1.0 *N*: Dissolve 40 g of reagent grade sodium hydroxide (NaOH) in 500 mL of purified water in a 1000 mL volumetric flask. Mix, cool and dilute to volume.

3. Sulfuric Acid Solution, 2.0 *N*: Dilute 55.5 mL of concentrated sulfuric acid (96% H_2SO_4, sp g 1.84) to 1 liter with purified water. Cool and adjust

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concentration to obtain a titer of 15.0 ± 0.2 mL against 20.0 mL of the 1.5 *N* sodium hydroxide solution, using a phenolphthalein indicator end point.

1. Sulfuric Acid Solution, 1.0 *N*: Add 27.8 mL of concentrated sulfuric acid (96% H_2SO_4, sp g 1.84) into a 1000 mL volumetric flask containing 500 mL of purified water. Mix, cool and dilute to volume.

4. Iodine Solution, Stock, 0.1 *N* (Note 2): Dissolve 40 g of potassium iodide (KI) in 200 mL of purified water in a 1000 mL volumetric flask. Let the solution come to room temperature, add 12.7 g of resublimed crystalline iodine (I_2), stir until completely dissolved, add 3 drops of concentrated hydrochloric acid (37% HCl, sp g 1.19) and dilute to volume with purified water. Mix thoroughly and store in an actinic glass bottle. Standardize as frequently as necessary, so that approximately 25 mL of the iodine solution is equivalent to 25 mL of 0.1 *N* standard sodium thiosulfate solution using starch indicator for end point detection.

5. Iodine Solution, Working Standard, 0.005 *N*: Using a 5 mL Class A pipet, transfer 5.0 mL of the 0.1 *N* stock iodine solution into a 100 mL volumetric flask. Dilute to volume with purified water and mix well. Make fresh daily.

6. Iodine Solution, Working Standard, 0.05 *N*: Using a 50 mL Class A pipet, transfer 50.0 mL of the 0.1 *N* stock iodine solution into a 100 mL volumetric flask. Dilute to volume with purified water and mix well. Make fresh at least weekly, and store in an actinic glass bottle.

7. Starch Indicator Solution, 1%: Slurry 10 g of soluble starch (Lintner Starch, available from Sigma Chemical, P. O. Box 14508, St. Louis, Missouri 63178) in 50 mL of cold purified water. Transfer quantitatively to 1 L of boiling purified water and stir until completely dissolved. Cool and add 1 g of salicyclic acid preservative. Discard after one month.

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PROCEDURE

Weigh accurately 100 g of sample into a 400 mL Erlenmeyer flask. Add sufficient purified water to bring total weight to 200 g (Note 3). Mix the sample and water until the solution is homogenous.

For low level samples: (less than 20 ppm)

Cool to 10 °C or below (Note 4). Place cold sample on magnetic stirrer and stir at a rate sufficient to produce a small vortex at the solution surface. Add 10 mL of cold 1.5 *N* sodium hydroxide solution and stir for 15 to 20 seconds. Add 10 mL of starch indicator solution (Note 5) and 10 mL of cold 2.0 *N* sulfuric acid solution; titrate immediately with 0.005 *N* standard iodine solution until a light blue color persists for one minute.

For high level samples: (above 20 ppm)

Add 20 mL of 1.0 *N* sodium hydroxide solution and stir 5 minutes. Add 25 mL of 1.0 *N* sulfuric acid solution, 10 mL starch indicator solution, and titrate with 0.05 *N* standard iodide solution until a light blue color persists.

Perform a blank titration using 200 mL of purified water and all reagents.

CALCULATION

Sulfur Dioxide, ppm, as is =

$$= \frac{(\text{Sample Titer, mL - Blank Titer, mL}) \times N \text{ Iodine} \times 0.032 * \times 1{,}000{,}000}{\text{Sample Wt., (g)}}$$

$$* \text{ Milliequivalent Weight of Sulfur Dioxide} = \frac{64.071}{2 \times 1000}$$

NOTES AND PRECAUTIONS

1. It is recommended that a 10 mL microburet with 0.02 mL subdivisions and a tolerance of ± 0.02 mL be used when analyzing samples containing more than 8 ppm sulfur dioxide. (Fisher Catalog No. 20-105B)

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2. The 0.1 *N* stock iodine solution may be standardized against arsenious trioxide as described in the "Reagents and Indicators" section, Method R-10.

3. To provide the best potential for detecting the visual end point of volumetric titration, constant volume is recommended. This eliminates variation due to inability to detect the end point consistently.

4. Starch indicator sensitivity may be lost and conversion reactions may occur under alkaline conditions. Therefore, it is necessary to cool the sample solution and keep all reagents at a temperature of 10 °C or below, in order to maintain stability.

5. Some sources recommend as little as 1 mL of starch indicator solution; 10 mL is recommended in this procedure to assist end point detection.

METHOD HISTORY

Combined the Sulfur Dioxide (Iodometric) methods for Corn Syrup (E-67), Corn Syrup (E-67a) and Corn Sugar (F-55) on 11-09-2010.

Corn Syrup, Sulfur Dioxide (Iodometric) (E-67) Date of Acceptance 10-18-1988.

Corn Syrup, Sulfur Dioxide (Iodometric:Low Level) (E-67a) Date of Acceptance 10-18-1988.

Corn Sugar, Sulfur Dioxide (Iodometric) (F-55) Date of Acceptance 10-18-1988.