Iodometric Determination of Sulfite in Prebath PB-6
ECN-1315
ECP-1315

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INTRODUCTION
The sample is added to an excess of iodine, formed by acidifying standard potassium iodide. Part of the iodine is reduced to iodide by the sodium sulfite and sodium metabisulfite in the sample; the remaining iodine is measured by titrating it with standardized sodium thiosulfate using starch indicator. Since the quantity of sulfite is equivalent to the quantity of reduced iodine, and the quantity of sodium thiosulfate used in the titration is equivalent to the quantity of remaining iodine, the difference between the total iodine and the volume of sodium thiosulfate is a measure of the sodium sulfite and sodium metabisulfite total.

This method requires handling potentially hazardous chemicals. Consult the Material Safety Data Sheet for each chemical before use. MSDS’s are available from your chemical supplier.

Note: Use pipets and volumetric glassware meeting the “Class A” definition by the National Institute of Standards and Technology (NIST).

RELIABILITY
The method is reliable in that it measures the true total amount of sulfite in the mix. In some mixes containing a relatively small amount of sulfite, an appreciable portion of the sulfite is oxidized during mixing, leading to low sulfite results.

Reagents
Use ACS Reagent Grade reagents unless otherwise specified.
- 0.1 N Potassium Iodate, KIO₃ (standardized to 4 decimal places)
- 7.0 N Sulfuric Acid, H₂SO₄
- 0.60 M Potassium Iodide, KI
- 0.1 N Sodium Thiosulfate, Na₂S₂O₃ (standardized to 4 decimal places)
- Starch Indicator
PROCEDURE

Sample Treatment
1. Pipet, wipe the pipet before leveling, 50.0 mL of standardized 0.1 N potassium iodate into a 250-mL conical flask.
2. Add 25 mL of 7.0 N sulfuric acid from a tip-up pipet.
3. Add 25 mL of 0.60 M potassium iodide from a tip-up pipet.
4. Pipet (wipe) 10.0 mL of sample into the flask.

Titration
1. Titrate with standardized 0.1 N sodium thiosulfate to a light yellow color.
   Note: Highly seasoned samples may not turn light yellow but an observable color change will take place.
2. Add, from a tip-up pipet, 5 mL of starch indicator and continue the titration until the iodine-starch (dark blue or black) color disappears.
   Note: The final color of the solution will be clear or tinted depending upon the degree of seasoning.

Calculations

\[
Na_2SO_3, \text{g/L} = \frac{\left[ (N \text{ KI}_3)(mL \text{ KI}_3) - (N \text{ Na}_2\text{S}_2\text{O}_3)(mL \text{ Na}_2\text{S}_2\text{O}_3) \right] \left( \text{eq wt Na}_2\text{SO}_3 \right)\left(1000\right)}{\left( \text{mL sample} \right)\left(1000\right)}
\]

\[
= \frac{\left[ (N \text{ KI}_3)(50.0) - (N \text{ Na}_2\text{S}_2\text{O}_3)(mL \text{ Na}_2\text{S}_2\text{O}_3) \right] \left(63.03\right)\left(1000\right)}{(10.0)(1000)}
\]

\[
= 6.303\left[ (N \text{ KI}_3)(50.0) - (N \text{ Na}_2\text{S}_2\text{O}_3)(mL \text{ Na}_2\text{S}_2\text{O}_3) \right]
\]