CHLORIDE

PRINCIPLE

Inorganic chloride is precipitated by titration with standard silver nitrate solution, and the end point is detected potentiometrically using a silver-silver chloride indicator electrode (Note 1).

SCOPE

The method can be applied to crude and refined sugars (Note 2),corn syrups and other starch hydrolyzates prepared by acid or enzyme conversion and combinations thereof.

SPECIAL APPARATUS

1. Potentiometer: pH meter with ± 1-mv sensitivity.

2. Electrode

REAGENTS

1. Silver Nitrate Solution, 0.05 *N*: Standard. Dissolve 8.5 g of reagent grade silver nitrate ($AgNo_3$) in purified water. Dilute to 1 L volume and mix thoroughly. Standardize against reagent grade sodium chloride using either the Mohr or Volhard methods.

2. Silver Nitrate Solution, 0.01 *N*: Standard. Transfer 200.0 mL of standard 0.05 *N* silver nitrate solution to a 1 L volumetric flask, dilute to volume and mix thoroughly.

3. Silver Chloride Saturated Potassium Chloride Solution, 4 *M*: Dissolve 298 g of reagent grade potassium chloride (KC1) in purified water, add a few drops of 0.05 *N* silver nitrate solution, dilute to 1 L volume and mix thoroughly.

4. Ammonium Nitrate Solution, 10%: Dissolve 10 g of reagent grade ammonium nitrate (NH_4NO_3) in 100 mL of purified water.

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5. Nitric Acid, Concentrated: Reagent grade (sp g 1.42)

SAFETY

Person(s) performing this method should wear appropriate protective equipment. Analysts should be familiar with the use and disposal of acids. All operations of transfer of concentrated acid to water and of evaporation should be carried out under a fume hood. Glassware should be carefully inspected for defects before use. Analysts should also review the Manufacturer's Safety Data Sheets (MSDS) for all of the chemicals required; however this review is not a substitute for adequate laboratory safety training.

PROCEDURE

Weigh 15 to 50 g (± 0.1 g) of sample (Note 6) into a 250 mL beaker, add 100 mL of warm purified water and dissolve. Place the beaker on a magnetic stirrer, add stirring bar, 1 mL of concentrated nitric acid, and immerse the electrode in the sample solution. While stirring, titrate the sample with standard silver nitrate solution (Note 6) adding in increments of 1 mL. Record the potential and titer after each addition of titrant. When the potential begins to change, add titrant in increments of 0.2 mL. After the potential "break" has been passed, resume titrating in 1 mL increments for three more additions.

CALCULATION

Plot the titration curve of emf versus titrant volume. Read the volume of titrant corresponding to the equivalence point (point of maximum inflection) from the curve.

$$\%\ \text{Chloride, (as is)} = \frac{\text{mL } AgNO_3 \times \text{Normality} \times 0.0355 \times 100}{\text{Sample Wt. (g)}}$$

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NOTES AND PRECAUTIONS

1. Results of equivalent precision and accuracy are obtained with automatic analyzers which generate silver ion coulometrically and detect the end point amperometrically.

2. Crude and refined sugars include crystalline dextrose (anhydrous and hydrate), dextrose solutions, high D.E. hydrolyzates and Corn Sugar Molasses (greens and/or hydrol).

3. Optimum sample size depends on chloride level. For materials containing more than 0.03% chloride use 15 g and 0.05 *N* silver nitrate titrant. For materials containing less than 0.03% chloride use 50 g and 0.01 *N* silver nitrate titrant.

METHOD HISTORY

Combined the Chloride methods for Corn Syrup (E-15) and Corn Sugar (F-10) on 4-15-2010.

Corn Syrup, Chloride (E-15), Date of Acceptance 3-30-1971, Revised 2-27-1996.

Corn Sugar, Chloride (F-10), Date of Acceptance 11-12-1962, Revised 3-08-2004.